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# ADVANCES IN RENEWABLE NATURAL MATERIALS FOR TEXTILE SUSTAINABILITY

Edited by MOHAMMAD SHAHID, ARANYA MALLICK, and SAYANDEEP DEBNATH



# Advances in Renewable Natural Materials for Textile Sustainability

Advances in Renewable Natural Materials for Textile Sustainability promotes sustainable practices in the textile industry through exploring the use of natural materials in textile production. The book delves into the advantages of using natural materials at every stage of textile processing, from fiber production to textile wet processing (dyeing, printing, finishing), and recycling after end use. The chapters provide critical discussions on natural materials for functional and smart textiles, sustainable methods of their preparation, application, and environmental impact of using biobased materials. It also discusses opportunities and challenges.

- Offers a comprehensive overview of the historical significance of natural fibers in textile production, the environmental impact of textile manufacturing, and the role of natural materials in reducing this impact.
- Provides examples of successfully implemented sustainable production processes.
- Discusses upcycling and repurposing of natural textile materials, sustainable textile waste management and recycling, and the use of natural colorants for dyeing and finishing textiles.
- Covers the use of biobased finishing agents, enzymes, and natural materialbased auxiliaries in sustainable textile production and discusses biopolymers and nanocellulose and their potential in textile applications.
- Explores sustainable textile reinforcement using natural fibers and natural fiber-based composites and their applications.
- Considers the future of sustainable fashion and the role of natural materials in smart textiles for advanced applications like textile packaging, medical applications, textile sensors, and actuators, among others.

With its comprehensive coverage, this book is an essential resource for professionals, researchers, and academics in the textile industry and anyone interested in sustainability in the fashion and textiles. It offers valuable insights for readers who want to make more informed choices and contribute to a more sustainable future.

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# Advances in Renewable Natural Materials for Textile Sustainability

Edited by Mohammad Shahid Aranya Mallick Sayandeep Debnath



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# Preface

In recent years, awareness of sustainability has increased across all industry sectors, driven by the urgent need to address environmental challenges and promote the responsible use of resources. The textile industry, a significant contributor to environmental pollution, actively seeks innovative solutions to reduce its ecological footprint while meeting the increasing demand for high-performance, eco-friendly products. This book, *Advances in Renewable Materials for Textile Sustainability*, comprehensively explores the cutting-edge developments in natural fibers and their applications in creating sustainable textile products. It brings together a diverse array of research contributions that highlight the potential of renewable materials to revolutionize the textile sector.

**Chapter 1** sets the stage with a historical perspective on natural fibers, tracing their evolution and enduring significance in textile manufacturing. This foundation leads into **Chapter 2**, which delves into eco-friendly natural fibers for sustainable agrotextiles, emphasizing their role in packaging agro-products. The journey through innovative natural fibers continues in **Chapter 3**, examining sustainable fibers derived from pineapple leaves, detailing their extraction, properties, and diverse applications. **Chapter 4** highlights hemp's transformation from a traditional fiber to one with modern applications, showcasing its versatility and ecological benefits. **Chapter 5** explores using plant fibers and biomass to produce tissue paper, presenting a sustainable alternative to conventional materials. **Chapter 6** discusses natural fiber-based composites and their properties and wide-ranging applications. **Chapter 7** presents the integration of natural fibers into smart textiles, illustrating advanced applications that merge functionality with sustainability. The comprehensive review in **Chapter 8** examines natural plant fibers as sustainable substitutes for traditional textile materials.

The potential of nanocellulose fibers in textile applications is explored in **Chapter 9**. This is further expanded in **Chapter 10**, which discusses the emerging applications of nanocellulose in textile processing. **Chapter 11** introduces chitosan as a biobased sustainable textile auxiliary, followed by **Chapter 12**, which covers the role of cyclodextrins in sustainable textile processes. **Chapter 13** discusses recent advances in enzyme applications for sustainable textile processing. The challenges, characterization, and sustainable treatment of textile dyeing effluents are addressed in **Chapter 14**. **Chapter 15** examines the use of natural materials in the functional finishing of textiles. **Chapter 16** explores natural plant extracts for UV protective textiles, while **Chapter 17** discusses the insect-repellent properties of natural dyes, highlighting recent advances and prospects.

Each chapter in this book highlights the crucial role of natural fibers and renewable materials in promoting textile sustainability. By incorporating these innovations, the textile industry can progress toward a more sustainable and environmentally friendly future. This book is an essential resource for researchers, industry professionals, and anyone dedicated to advancing sustainability in textiles.

> Mohammad Shahid Aranya Mallick Sayandeep Debnath

# About the Editors

Mohammad Shahid, PhD, is working as Assistant Professor & Head, Department of Chemistry, at Mewar University, Chittorgarh, India. He earned his Master's degree in Chemistry from Shibli National College, Azamgarh, in 2006 and his Ph.D. in Chemistry from Jamia Millia Islamia, New Delhi, in 2014. Before joining DKNMU, Dr. Shahid held positions at several prestigious institutions worldwide. He served as a Postdoctoral Fellow at Soochow University, China, a Marie Curie Postdoctoral Fellow at the University of Glasgow, UK, a Kothari Postdoctoral Fellow at the Institute of Chemical Technology, Mumbai, India, and Assistant Professor & Head, Department of Applied Science, Dr K N Modi University, Rajasthan, India. His research was supported by numerous esteemed fellowships and grants, including the UGC Fellowship, China Postdoctoral Fellowship, EU Marie Curie Fellowship, and Dr. D. S. Kothari Fellowship. His prolific academic career includes over 50 scientific publications in peer-reviewed journals, reviews, reports, patents, and book chapters. He has also edited and authored multiple books with renowned international publishers. His research interests are diverse, spanning textile chemistry, green chemistry, and the development of sustainable materials.

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# 1 Natural Fibers A Look from a Historical Perspective

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## **1.1 INTRODUCTION**

The exploitative nature of humankind has led to the discovery and invention of many items. Fibers are one such entity found in abundance and used to the extent of their potential. Humans have used natural fibers for centuries for clothes, shelter, utility items, textile decorations, etc.<sup>1,2</sup> These fibers have some of the most important qualities, like comfort, durability, and renewability, making them indispensable. Since all fibers need their specific agro-climatic conditions and geographic locations to thrive, different regions of the world have had diversity in producing different natural fibers, which have, in turn, influenced their local clothing habits.<sup>3,4</sup> Completing these basic needs created the next step, adding value and artistry to everyday textiles, which opened a door for crafts based on these fibers' properties. Interestingly, India has had the basket of the most varieties of natural fibers, ranging from cellulosic, and protein, to lignocellulosic fibers, throughout the ages. As a result, the variety of crafts that evolved in the subcontinent is far more diverse than on most other continents.<sup>3,5</sup> The flourishing Indian markets for these homegrown textiles led to a gold mine of crafts and trade with external entities and within the country.<sup>6</sup> In 17th century India, the major cotton products were chintzes, baftas, or finer counterparts like calicoes.<sup>3-6</sup> Major fibers like linen, wool, jute, silk, ramie, sisal, and more have also had their origination as useable fibers in different parts of the world and have since continued to grow in production and use all across the globe. While natural fibers enjoyed their dominance for a considerable period, with the invention of synthetic fibers in the 19th century, an influx of man-made materials into the dressing routine of modern civilization began.<sup>7</sup> While synthetic materials came with desirable qualities, such as affordability, ease of mass production, easy care, and durability, their harm to nature and their non-biodegradability posed a huge threat to natural resources, human health, and environmental and social well-being.<sup>7</sup> Recent reports show that global fiber production per person has increased by 75% from 1975 to 2022. Of the global fiber production of 116 million tonnes in 2022, polyester remains the most widely produced fiber, making up 54% of total production.8 With the increase in global fiber production and the increasingly dangerous threats of synthetic materials, there is a

need to balance the use of natural and synthetic fibers in society. It is important to look at natural fibers in retrograde, study their historical importance, and restore their status in modern society as suitable places of usage. This chapter deals with the insight into the historical perspective of natural fibers, surface, and cross-sectional fine structure, their properties, and suitability for end use, with special references to major natural fibers under three sections viz. seed (cotton), fruit (coir), bast (linen, jute, ramie, hemp, sisal) and animal (silk, wool).

## 1.2 NATURAL FIBERS

Simply put, natural fibers refer to material from natural sources like plant produce or animal pelages. Natural fibers can generally be classified into two types- primary and secondary. When plants are grown primarily for their fibers, they are primary plants, and those that produce fibers as a by-product are called secondary plants.<sup>1,9,10</sup> Kenaf, jute, sisal, hemp, and cotton are examples of primary plants, while pineapple, cereal stalks, agave, oil palm, and coir are examples of secondary plants. The major natural fibers prehistorically are reported as cotton, linen, jute, hemp, sisal, ramie, coir, silk, and wool.<sup>10,11</sup> New natural fibers include bamboo, seaweed, banana, soy, nettle, pineapple, and many more. The classification of major natural fibers from historical perspectives is shown in Figure 1.1.

The natural fibers grown in different areas are different due to the change in agroclimatic conditions as *one spans the* parts of the world. From a historical perspective and present scenario, the leading countries growing natural fibers are depicted in Figure 1.2.

Since these natural fibers have different chemical constituents, they vary in shape, diameter, and other properties. Tables 1.1 and 1.2 show the properties of cellulosic, lignocellulosic, and protein fibers for ease of understanding.



FIGURE 1.1 Classification of natural fibers.<sup>1,11</sup>



**FIGURE 1.2** A visual representation of leading countries that grow cotton, linen, ramie, sisal, hemp, jute, coir, silk, wool, and alpaca.<sup>4,12,13</sup>

## 1.2.1 SEED FIBER

The term 'seed fiber' is vast, including different types of seed extracts like fibers, husks, pods, fruit, and hulls. The single most popular one has been covered in this chapter, namely cotton.<sup>1,30</sup>

#### 1.2.1.1 Cotton

No discussion about natural fibers is complete without mentioning cotton – a fiber so widespread and useful since the beginning of time. The first evidence of cotton cultivation is in approximately 3200 BC, with its initial discovery along the Indus River and other regions, such as Peru and Mexico. The oldest mention of cotton was found in Rig Veda and the writings of European scholar Herodotus.<sup>31,32</sup> Cotton is a single-celled seed hair reported to be one of the purest cellulose forms in nature.<sup>33,34</sup> SEM images of cotton fiber are shown in Figure 1.3.

However, many cotton varieties are found worldwide, branching from the Gossypium family.<sup>37</sup> Depending upon the climatic condition, soil, and region, there are noticeable variations in the staple length, fineness, and crop color.<sup>38</sup> In the eyes of modern spinners who rely solely upon the characteristics of cotton to select their optimum mix, the present-day convenience of global markets allows them to import a variety of cotton worldwide.<sup>39</sup> In older ages, this was not the case. Local crafts were developed using local variations of cotton that the people could access in the vicinity.<sup>31</sup> Though the Gossypium family branches several subgenus plants, species such

# TABLE 1.1Physical, Mechanical, and Chemical Constituents of Cellulosic and Lignocellulosic Fibers14-24

	Physical Properties		Mechanical Properties			Major constituents			
Fiber	Moisture Content (%)	Density (g/cm³)	Diameter (µm)	Tensile Strength (MPa)	Young's Modulus (GPa)	Elongation at Break (%)	Cellulose (%)	Hemicellulose (%)	Lignin (%)
Cotton	8.0	1.5-1.6	12.22	287-597	5.25-8	6.0	94	3	0.0
Linen (raw flax)	12	1.45	12-16	510-910	50-70	2.7-3.5	56.5	15	2.5
Jute	12	1.34	25-200	200-460	20-55	1.16-1.5	45-71.5	13.6-21	12-26
Hemp	6-12	1.4	25-600	550-900	30-60	1.6	77.5	10	6.8
Ramie	8.5	1.55	10	915	23		84.1	3.4	0.0
Sisal	11	1.45	$474.4 \pm 0.065$	100-800	9.0-38.0	$1.74 \pm 0.13$	47-78	10-24	7-11
Coir	13	1.15	142–169	100-200	6	15-30	38.4	24.5	31.8

# TABLE 1.2Physical and Mechanical Properties of Protein Fibers15,23-29

	Physical Properties	sical Mechanical Properties					
Fiber	Moisture Content	Density	Diameter	Tensile Strength	Young's Modulus	Elongation at Break	
Silk	11%	1.32-1.6 g/cm3	5–18 µm	300–600 MPa	14.5–17.0 GPa	15-20%	
Wool	13–18%	1.076–1.307 g/cm3 (depends on medullation) <sup>a</sup>	24.95 to 26.39 μm <sup>b</sup>	1–1.7 MPa	3.1 GPa	25-40%	

Notes:

<sup>a</sup> May vary with medullation.

<sup>b</sup> May vary for finer varieties of wool.



**FIGURE 1.3** Scanning electron microscope longitudinal image  $(a)^{35}$  and cross sectional image  $(b)^{36}$  (Reprinted with permission from Ref. 35; Reprinted with permission from Ref. 36 of cotton fiber.)

as *hirsutum*, *barbadense*, *herbaceum*, and *arboreum* are the only commercially cultivated ones, of which 97% is the *G*. *hirsutum* species.<sup>32</sup> Cotton fibers can be characterized by fiber fineness, fiber staple length, maturity, strength, leaf grade, and color.<sup>33</sup> Different varieties of cotton in India and their properties are listed in Table 1.3.

India is known to grow one of the rarest cotton varieties in history, colloquially known as tree cotton, Khara cotton, Ganges cotton, and more popularly, phuti karpas along the river Ganges, and its major watercourse, river Meghna, in Bangladesh. The world-renowned Dhakai Muslin was made using this cotton breed. With time, the identification of this breed, along with the craftsmanship, fell into oblivion.<sup>41–43</sup> Around 70 authentic Dhaka muslins are preserved in the Victoria and Albert Museum, London. Bangladesh's government is reportedly working to revive the craft by identifying the fiber breed using genetic analysis. A team of highly skilled experts

Different Cotton Varieties in India	Staple Length (mm)	Micronaire	
Short Staple (20.0 mm & below)			
Assam Comilla	_	7–8	
Bengal Deshi	_	6.8-7.2	
Medium Staple (20.5 mm-24.5 mm)			
Jayadhar	21.5-22.5	4.8-5.8	
V-797/ G.Cot.13/ G.Cot.21	21.5-23.5	4.2-6.0	
AK/Y-1/MCU-7/SVPR-2/PCO-2/K-11	23.5-24.5	3.4-5.5	
Medium Long Staple (25.0 mm-27.0 mm)			
J-34 (Raj)	24.5-25.5	4.3-5.1	
LRA-5166/ KC-2	26.0-26.5	3.4-4.9	
F-414/ H-777/ J-34 Hybrid	26.5-27.0	3.8-4.8	
F-414/ H-777/ J-34 Hybrid	27.5–28.5	4.0-4.8	
H-4/ H-6/ MECH/ RCH-2	27.5-28.5	3.5-4.7	
Sankar-6/10	27.5-29.0	3.6-4.8	
Bunny/ Brahma	29.5-30.5	3.5-4.3	
MCU-5 / Surabhi	32.5–33.5	3.2-4.3	
DCH-32	34.0-36.0	3.0-3.5	
Suvin	37.0-39.0	3.2-3.6	

## TABLE 1.3 Different Varieties of Cotton in India and Their Properties<sup>40</sup>

Note:

<sup>a</sup> Micronaire: The fineness of cotton fibers is expressed in terms of mass (microgram) for a specified unit (inch) of length.

searched far and wide for a plant that matched the DNA of the fossilized *phuti karpas* and came closest to it with a 90% match in Titabri in Kapasia.<sup>44</sup> There are other cotton varieties worldwide, with a similar penchant to dazzle the reader, most of which are extinct or endangered.

Certain types of cotton tend to have naturally colored lint, meaning a color other than white. The limited availability of shades, coupled with the dearth of cultivation, make colored cotton a novelty niche crop, with a very small portion of products made of them, like everyday apparel items, shirts, pants, and more. Colored cotton is usually obtainable in light brown, khaki/camel color, brown, dark brown / chocolate color, dirty grey, tan, red, green, and light green shades (Figure 1.4). However, on prolonged exposure to the sun, the color may fade from the bolls of the cotton lint. These varieties have an approximate yield of 11.15 q/ha – 20.01 q/ha in Indian irrigated or rainfed areas. One benefit of using this cotton is that it eliminates many wet processes that consume considerable energy, water, and utility.<sup>46-48</sup>

Though the silk route existed, it is reported the trade of finished goods and the bulk of raw material transportation of fibers like cotton may not have been a feasible option at that time.<sup>31,56</sup> The tropical condition in some of the states of India plays the role of a great host for most of the spinnable cotton species, not only in its original



FIGURE 1.4 Cultivars of colored cotton fiber.<sup>45</sup> (Reprinted with permission from Ref. 45.)

# TABLE 1.4Notable Properties of Premium Cotton Types<sup>39,49–55</sup>

Premium Cotton	Description	Strength (g/tex)	Elongation (%)
Pima	A cross between <b>Sea Island</b> and <b>Egyptian cotton</b>	41.0	7.0
GIZA (G.45)	Giza cotton is a high-quality variety of Egyptian long-staple cotton which has further increased in value due to its cultivars created with selective breeding.	42.3	6.0
SuVin	Cross between <b>Sujata</b> and <b>St. Vincent</b> cotton developed by Central Institute of Cotton Research, India.	37.0	13.2

form but also in the form of cross-breed homegrown species.<sup>57</sup> A few premium ones are also available worldwide alongside these usual cotton species. These usually include cotton from a similar species but with varying factors, like staple length, yield, cultivation methods, etc. Table 1.4 presents comparative details of a few premium cotton types.

Even though only the *hirsutum* and *barbadense* species are utilized commercially, a variety of textiles can be created from them. Cotton fiber can be transformed into muslins, poplin, cambric, chambrays, calicos, and denim, among other fabrics, based on the manufacturing process, the weaving, and other factors. The leading producers and exporters of cotton are shown in Table 1.5.

## 1.2.2 FRUIT FIBER

The main fiber enlisted under this section is coconut fiber or coir. While it is largely considered as a fruit fiber, it can also be segmented under seed fibers by some.<sup>1,60</sup>

	Quantity (MT)	Exporters	Quantity (MT)
China	66,84,000	U.S.A.	27,87,000
India	56,61,000	Brazil	14,49,000
U.S.A.	31,50,000	Australia	13,50,000
Brazil	30,62,000	Greece	2,83,000
Australia	12,64,000	India	2,50,000
	China India U.S.A. Brazil Australia	China66,84,000India56,61,000U.S.A.31,50,000Brazil30,62,000Australia12,64,000	China         66,84,000         U.S.A.           India         56,61,000         Brazil           U.S.A.         31,50,000         Australia           Brazil         30,62,000         Greece           Australia         12,64,000         India

TABLE 1.5	
Major Producers and Exporters of Cotton 2022/2023 <sup>58,59</sup>	

#### 1.2.2.1 Coir Fiber (Coconut)

Coconut fiber has been used to produce ropes and cordage since time immemorial. Extracting coir fiber, a by-product of the coconut industry, involves processing coconut husks to eliminate their organic constituents.<sup>61</sup> While the existence of coconuts in India has been a contention among scholars, it has been substantiated through references in literary works such as the Raghuvamsa of Kalidasa and Sangam literature.<sup>62</sup> SEM images of coir fiber are shown in Figure 1.5.

Historian Shri. P. K. Balakrishnan postulates that the advent of the Portuguese influence marked a pivotal juncture in initiating systematic coconut cultivation within the region of Kerala.<sup>62</sup> Coir's affiliation with Kerala can be traced back to the 19<sup>th</sup> Century when the cocos nucifera tree stood as a prominent tropical species frequently observed in the landscapes of Kerala. In the Malayalam language, "Kera" denotes coconut, while "Alam" signifies land, aptly naming the state "Keralam," which means Land of Coconut. Kerala is the leading state in India for coir production, while India, in turn, is the leading country for the same.<sup>64</sup> The many advantages of coconut fibers include resilience against moths, fungi, and rot, insulative properties for heat and sound, not easily combustible, resistance to flame, resilience against moisture, durable and resilient fiber, good dimensional stability, static-free, and ease of cleaning.<sup>65</sup>



**FIGURE 1.5** Scanning electron microscope longitudinal image (a) and cross-section image (b) of coir.<sup>63</sup> (Reprinted with permission from Ref. 63, Copyright © 2020, Elsevier Ltd.)



FIGURE 1.6 Production of raw coir and export of raw coir 2022.68

However, its high flexural rigidity, large diameter, and low length-to-diameter ratio (L/D) make mechanical processing harder to take place at a higher speed.<sup>66</sup> To solve this, industrial processes like chemical softening, help reduce fineness and flexural rigidity and improve the tensile properties.<sup>66</sup> These properties made coir a popular fiber for geotextiles, which was developed for application towards erosion control to use its attribute of high-water absorption. Indian coir is available in the form of raw fiber, spun yarn, woven mats, rugs, carpets, coir rope, and more.<sup>64</sup> Coir fabric, due to its tensile strength, serves as a support membrane in road edge drains, improves rural road conditions with geotextile-lined trench drains, enhances slope stability through coir netting, and reinforces soft soil stabilization effectively.<sup>64</sup> Coir fiber can also be used in natural fiber composites (bio-composites), which provide environmental gains, reduced energy consumption, lighter weight, insulation, and sound absorption properties. The prospects of manufacturing "high quality" natural fiber composites for lower mechanical loads are very promising.<sup>64</sup> Rubber-coated coir mats are also a widespread use of coir fiber.<sup>67</sup> These mats include rubberized coir mats, felted coir mats, polyurethane foam mats, latex foam mats, and sandwiched rubber coir sheets (SRC).<sup>67</sup> Figure 1.6 provides production and export data of raw coir during year 2022.

#### 1.2.3 BAST FIBERS

Fibers are termed bast fibers when extracted from the outer cell layers of the stem part of the plants.<sup>69</sup> Historically and currently, some of the most important bast fibers include linen, hemp, jute, ramie, and sisal, which have been covered in this section.<sup>69,70</sup> Due to their strength and resilience, bast fibers have been used in geotextiles, automotive parts, sports products, and more.<sup>70</sup> The vitality of these fibers in the early world, their properties, and resultant usages will be discussed in this segment.

#### 1.2.3.1 Linen

Linen, the long, strong fiber from flax, is one of the oldest known textiles in human civilization. The plant is also known for yielding edible seeds and oil. Scientifically known as *Linum usitatissimum*, it is mainly grown in China, France, Belgium, Belarus, and Russia.<sup>71,72</sup> SEM images of linen fiber are shown in Figure 1.7.

Linen was known to the early Indian civilizations through its mention in the ancient texts of Vedas and Upanishads by the term "ksauma," which was described as the fibrous material derived from the bark of the linseed plant.<sup>73,74</sup> Ayurveda and the Sushruta Samhita talk about its medicinal properties and benefits of consumption as food and childcare, including a suggestion that a newborn child should be wrapped in linen along with the bed linens of a child.<sup>74</sup> Aside from literary references, physical evidence has also been found in Upper Paleolithic regions at Dzudzuana Cave, Georgia, and other Neolithic relics. Here, wild flax fibers were woven as fabrics for garments dyed in natural dyes, baskets, and cords for tools.<sup>75,76</sup> The first cultivation of linen was reported to take place around the same time, in circa 8000 BC, within the geographical expanse known as the Fertile Crescent. The ancient Egyptian civilization is reported to have cultivated flax as a major textile fiber crop, as an 'irrigated variety'.<sup>76,77</sup> Besides Egyptians, other civilizations, such as Babylonians, Phoenicians, and others between 5000 and 4000 BC, also cultivated flax. The Egyptians often utilized linen to mummify their royalty, burial shrouds, garments, and even as a form of currency.74 In the 3rd century BC, circa 2000 years ago, a remarkable artifact within the realm of Etruscan writings, the Liber Linteus, also known as the Linen Book of Zagreb, represents the usage of linen to create books, concomitantly being the sole surviving example of a linen book, or libri lintei.<sup>77,78</sup> The excavation of the Mycenaean state of Pylos in the Greek bronze Age around 1200 BC uncovered Linear B tablets containing the documentation of a linen industry managed by royalty, with the term Aivov referring to linen thread, or fabric in Greek.<sup>77</sup> The documented production method at Pylos was inferred to have been a uniform process for historical sites before. Succeeding the initial growing and harvesting, the flax stem was retted.77

After the Middle East and Europe, flax made its way to Great Britain through Phoenician traders. While the English had looked down upon linen as 'effeminate,'



**FIGURE 1.7** Scanning electron microscope longitudinal image (a) and cross sectional image (b) of linen.



FIGURE 1.8 Producers of linen 2022, raw and retted.<sup>71</sup>

their ideologies gradually changed. It started being used to make undergarments, due to its comfort and the beliefs that it kept away cutaneous disease. After subsequent invasions and trading, linen started being manufactured in France, Italy, Ireland, Scotland, Netherlands, Germany, and other parts of the world.<sup>79</sup> Ever since, linen has been widely utilized in various domains, including but not limited to home furnishing fabrics (upholstery, bed linens, living linens, tableware, draperies, wall coverings, and more), apparel (shirts, pants, suiting items, and more), surgical threads, fabrics for aviation purposes, reinforced plastics and composite materials, textured papers, hand-kerchiefs, and luggage fabrics. Furthermore, linen had also found applications in insulation materials and filtration, as seen in hose pipes usually used by firefighters.<sup>80</sup> Figure 1.8 provides details of producers of raw and retted linen in year 2022.

#### 1.2.3.2 Hemp

A versatile plant with a rich history in cultivation, hemp has been utilized for many purposes, such as textiles, medicinal applications, recreational substances, and as a source of nourishment.<sup>81</sup> Sprouting from the plant Cannabis sativa L. Cannabaceae, it is a herbaceous, woody plant that is native to Central Asia and continues to grow in Asian and European areas with ease.<sup>82</sup> Like flax, hemp is also a bast fiber that naturally resists many insect species and does not need excessive water in its cultivation process. Its positive qualities also include its deep root system, which reduces soil loss and erosion, which is ideal for a variety of crop rotations.<sup>83</sup> Figure 1.9 shows SEM image of hemp fiber.

The historical evidence of hemp cultivation in China dates back at least 6000 years. It is worth noting that the scenario of hemp farming and retting has been vividly portrayed as early as the XiZhou Dynasty, which thrived during the 11th to 7th centuries B.C. and was documented in possibly the oldest Chinese agriculture treatise, 'Xia Xiao.<sup>82</sup> The use of Hemp as rope and canvas materials was implemented in shipbuilding in ancient times.<sup>85</sup> Despite the dearth of historical records of hemp in



**FIGURE 1.9** Scanning electron microscope longitudinal image of hemp.<sup>84</sup> (Reprinted with permission from Ref. 84.)

Europe, in 484 B.C. Erodotus postulated that the introduction of hemp to the European continent can be attributed to the migratory movements of the Scythian civilization from Asia at approximately 1500 years B.C.<sup>81</sup> Furthermore, Teutonic tribes played a significant role in disseminating hemp cultivation across various regions of Europe. However, during this period, the hemp experienced a substantial surge. Subsequently, from the 18th to the 19th centuries, hemp cultivation assumed a pivotal and commercial role. The gradual decrease in hemp cultivation in Europe throughout the 20th century can be attributed to the widespread adoption of synthetic fibers and rising labor costs.<sup>85</sup>

In addition to its utilization as a versatile fiber crop, hemp has also been recognized for its potential as a medicinal plant and its historical significance as a ritualistic and intoxicating substance. The hemp plant used for medicinal use differs from those used for extracting fibers.<sup>86</sup> The historical utilization of hemp in paper manufacturing can be traced back over two millennia, establishing a profound connection between hemp and the evolution of paper production.<sup>81</sup> Despite numerous endeavors, the presence of synthetic fibers, alternative natural fibers, and economically advantageous hemp imports from Eastern nations, coupled with the labor-intensive nature of hemp cultivation and its constrained profitability, led to the gradual decline and ultimate disappearance of hemp during the early 1970s.<sup>81</sup> In 1998, the Ministry of Agriculture of Italy allowed the hemp plant to be grown in Italy, after which the world slowly followed.<sup>81,86</sup> Only recently, in 2018, the Government of Uttarakhand, India, issued a letter for the scientific use of hemp and its research and development, therefore facilitating the growth of the crop in 13 districts of the states.<sup>87</sup> These steps are instrumental in reintroducing natural fibers to the world and utilizing them for their desirable properties. Due to hemp properties, as discussed, it is a fiber that has been traditionally used to produce ropes, cordages, apparel, cloth, food, oil, medicines, and a variety of other things. It is also used to manufacture banknotes and



FIGURE 1.10 Producers and exporters of hemp 2022.68

home furnishing items and has carved out a place for itself under the niche market category.<sup>88</sup> While parts of the world still have reservations against hemp, it is a useful, ubiquitous, and renewable fiber that should be fully utilized for the best results in the textile world. Figure 1.10 provides and overview of producers and exporters of hemp during year 2022.

#### 1.2.3.3 Jute

Jute, a multicellular lignocellulosic bast fiber, is one of those natural fibers that came the closest to cotton in terms of production (mainly for packaging) in the demandhungry world of the mid-20<sup>th</sup> century. A herbaceous plant mainly cultivated in the equatorial, tropical, and subtropical zones, the fibers are usually off-white to brown and range from 1–4 meters (3–12 feet) in length.<sup>89,90</sup> Like linen, this fiber material is derived from the stems and leaves of jute plants. Despite a wide variety of fiber-yielding jute, only two varieties are domesticated and cultivable, namely, tossa jute (*Corchorus olitorius*), an African species, and white jute (*Corchorus capsularis*), said to have originated in the Indo-Myanmar.<sup>91,92</sup> Figure 1.11 shows SEM images of longitudinal and cross-sectional jute fiber.

The discovery of Jute artifacts dates back to the Harappan civilization, dated 2200–1900 centuries BCE.<sup>90</sup> Bengali poems of the 16th and 17th enturies also mention jute.<sup>94</sup> Through the British East India Company's victory in the Battle of Buxar in 1764, the states of Bengal, Bihar, and Orissa were acquired by them-- opening up the opportunity to raid the agricultural treasures of India, including jute fibers. This led them to use it for their own gain to export finished goods to the rest of the world.<sup>89,95</sup> However, the flexural rigidity of the jute when it was in a completely dry state made it unspinnable.<sup>96</sup> Oil in the form of an emulsion lubricates the fibers, and the fiber becomes spinnable with suitable machinery and processes.<sup>97</sup> As an emulsion, the oil enhances inter-fiber friction, which aids in regulating relative fiber motions during attenuation.<sup>97</sup> However, Dundee's whaling practice supplied whale



**FIGURE 1.11** Scanning electron microscope longitudinal image of jute (a), cross-section image of jute (b). (Reprinted with permission from reference number<sup>93</sup>, copyright © 2021, Taylor & Francis.)

oil, which, when mixed with water and applied to the fiber in oil in water emulsion form, made it possible to soften the material enough to spin into a yarn.<sup>98</sup> After the gradual curtailment of whale hunting, alternatives to whale oil started to be used.<sup>97,98</sup> Jute batching oil (JBO) is one such oil applied to jute fiber and is used widely. However, the presence of JBO makes the jute replete with a kerosene-like odor and is reportedly unhygienic. The prima facie information states that the JBO is transferred from jute packaging materials to food grains, which are frequently transported and stored in them, making them unfit to consume.<sup>99</sup> Attempts have been made with sulfated castor oil (SCO) in emulsion form to replace the jute batching oil, which is said to be sustainable and water-miscible in nature.<sup>97</sup> 2.5% SCO emulsion gives a relatively more economical way of jute processing. It is an eco-friendly, biodegradable, and non-toxic way of jute conditioning, but yarn imperfections (with respect to thick places, thin places, and neps) are more numerous as compared to jute treated with JBO.<sup>97</sup>

Dundee, with a thriving linen production until the 1830s, saw the introduction and rise of jute, which soon overtook flax by the 1860s.<sup>100</sup> With the passage of time, the manufacturing was passed on to mills in Serampore, India, and, subsequently, Calcutta, with Scottish connections.<sup>101</sup> Jute was the choice of packaging material as it has an impressive weight-to-packaging ratio. One tonne of flour can be packaged in a 15lb hessian bag/ a 4lb twill sack, and one tonne of potatoes can be packaged by using a 22lb hessian bag/ a 46lb twill sack.<sup>102</sup>

Jute is a fiber that has been used for the cheapest and poorest of purposes, and when quality permits, it climbs a rung.<sup>103</sup> Brightness and fineness are two parameters that identify its quality.<sup>104</sup> Brighter fibers, like golden or white-colored ones, are much more preferable in the industry than their dull grey and darker counterparts.<sup>104</sup> When farmers ret it in stagnant and muddy waters, the fiber loses its valuable qualities, unlike when done under more hygienic conditions with gently running clean water.<sup>105</sup> The 'goldenization' of jute, or brightening of jute, is a process carried out by brightening agents that are used to chelate the unwanted particles, which includes



**FIGURE 1.12** (a) Untreated jute fiber and EDTA treated "goldenized jute", (b) Change in jute colour with increase in EDTA.

lightening it, removing iron and tannins (that darken the fiber), and making it fit to meet saleable expectations.<sup>105</sup> Figure 1.12 shows effect of EDTA treatment to brighten the colour of jute.

Jute packaging products enjoyed their historical popularity by being extensively employed in hessian, which comprise 80% of total jute products as of 2005, sacking, ropes, twines, and carpet backing cloth (CBC).<sup>106-110</sup> Hessian, a lightweight material, finds versatile applications in producing bags, wrappers, upholstery, wallpapers, and other home furnishing items.<sup>107</sup> Jute shopping bags have been the most popular product, even when competing with polyester bags.<sup>111</sup> Jute bags are put through stiffening and extrusion lamination processes for stiffness, water-proofing, and to prevent fiber loss. These bags are stiffened using starch/binder/modified starch and extrusion laminated using polypropylene or polyethylene granules, along with a glossy or matte finish. With diversified jute products, a plethora of options exist, including but not limited to floor coverings, home textiles, technical textiles, geotextiles, jutereinforced composites, pulp and paper, particle boards, shopping bags, handicrafts, fashion accessories, and shoes like espadrilles.<sup>111-119</sup> These industries have harnessed the potential of jute and its related fibers, employing advanced 'non-woven' and composite technologies to fabricate a wide range of technical textiles, non-wovens, and composites.<sup>113</sup> The utilization of jute fibers in these areas has recently witnessed a notable surge.<sup>120,121</sup> Through its centuries of presence as a usable fiber, jute has seen its share of ups and downs. With the inception of plastic, jute saw its fall. With the current wave of eco-friendly materials, jute is rising again and supports the livelihood of 12 million farming families in Southeast Asia as of 2020.90,120,122 Table 1.6 lists statistics of largest producers and exporters of raw jute during 2018–2019.

#### 1.2.3.4 Ramie

Ramie, an ancient fiber crop often dubbed 'the king of the fibers,' has been used for around 6000 years. The ramie fiber is widely recognized as one of the strongest and elongated natural textile fibers globally, especially among vegetable fibers. It is a type

	Producers	Quantity <sup>a</sup> (lakh bales)	Exporters	Quantity <sup>a</sup> (lakh bales)
First	India	66.44	Bangladesh	8.33
Second	Bangladesh	93.17	India	1.52
Third	Nepal	0.62	Others	1.11

# TABLE 1.6Production and Export of Raw Jute 2018–19122

of lignocellulosic bast fiber obtained from the phloem, specifically the bast layer, of the stem of the *Boehmeria nivea* (L.) Gaud., a perennial shrub belonging to the nettle family.<sup>90,123,124</sup> After it has been decorticated, the raw state of ramie consists of bundled fibers, necessitating the separation of individual fibers to be spun.<sup>125,126</sup>

While it may sound like an infallible fiber, it does have a few huge drawbacks-the difficulty to spin it due to its gummy remnants, high energy utilization, and the high amount of toxic effluents left by the degumming chemicals that cause cell damage.<sup>127,128</sup> It is widely believed that the plant originated in Asia-mainly in China, India, and Indonesia, deriving its nickname 'China Grass.'129 According to historical records, ramie fibers were purportedly used for adorning burial shrouds in China and wrapping mummies in Egypt. Other literary references include the writings of Kalidasa and Ramayana, which reference and express admiration for ramie, described as a type of 'grasscloth.'<sup>123</sup> The introduction of ramie occurred in a sequential manner across several European countries. The export of Ramie fiber from China to the Western world commenced in the early 18th century, while the establishment of ramie on a commercial scale did not occur until 1930. Holland witnessed the introduction in 1733, followed by France, Germany, England, the United States, and Belgium.<sup>123</sup> Ramie had notable growth during the mid-1980s, coinciding with the prevailing fashion trend that strongly focused on natural fibers.<sup>123</sup> China, particularly the central and southern regions, is the global leader in ramie production. Japan, Taiwan, Brazil, Philippines, Indonesia, Korea, Vietnam, and India are among the other prominent manufacturers of ramie fiber.<sup>123</sup> Ramie fiber has found extensive applications in various industries. This versatile natural fiber produces a wide range of products, like industrial sewing thread, packing materials, fishing nets, and filter cloths. Extending beyond its traditional use, Ramie is also employed in creating textiles for household furnishings, such as upholstery and canvas. Additionally, it finds its way into the realm of fashion, often in combination with other textile fibers, to produce clothing items. Ramie contributes to various apparel items, including dresses, suits, skirts, jackets, pants, blouses, shirts, children's wear, and cotton-blended knitted sweaters. Shorter fibers and Ramie waste material may be used to create various paper products, sometimes bank notes and delicate cigarette papers.<sup>124</sup> Ramie's versatility extends to its use in parachute textiles, woven fire hoses, and thin woven canvas. Ramie cultivation is also reported to show potential in the remediation of phosphorous-contaminated



FIGURE 1.13 Production of raw ramie 2018–19.<sup>122</sup>

environments, such as the Everglades. The ability of ramie plant to effectively uptake phosphorous makes it an ideal candidate for environmental restoration efforts in this ecosystem.<sup>88</sup> Figure 1.13 provides statitiscs of production of raw ramie during 2018–2019.

## 1.2.3.5 Sisal

Sisal is a lignocellulosic hard natural fiber extracted from the leaves of the Agave sisalana plant, and its origins are traced back to Mexico.<sup>130,131</sup> However, its cultivation and subsequent naturalization have extended to various nations across the globe, including Brazil, China, Kenya, Tanzania, Madagascar, and Mozambique.<sup>131</sup> After 2 years of sowing the sisal plant, it can be harvested for 7–10 years or even more, producing 180–240 leaves, depending on factors.<sup>132</sup> Figure 1.14 shows SEM images of Indian sisal.

The historical origins of sisal cultivation in East Africa can be attributed to the introduction of 62 plants in 1893 by R. Hindorf, an esteemed German East Africa



**FIGURE 1.14** Scanning electron microscope longitudinal image (a) and cross-section image (b) of Indian sisal in Orissa.<sup>130</sup>(Reprinted with permission from ref. 130, copyright © 2011, Elsevier B.V.)

0	•			
	Producers	Quantity (tonnes)	Exporters	Quantity (tonnes)
First	Brazil	91,923	Brazil	38,100
Second	Tanzania	36,331.26	Kenya	29,500
Third	Kenya	22,018.23	Madagascar	6,600
Fourth	Madagascar	17,612.51	Mexico	900
Fifth	China (mainland)	14,297.54	Mozambique	100

# TABLE 1.7Production and Export of Raw Sisal 2018–19122

Largest Producers and Exporters of Raw Sisal

Company member.<sup>130,131</sup> This pivotal event marked the inception of sisal production in the region. Furthermore, it is noteworthy that certain statistical data about the sisal industry in East Africa have been documented.<sup>131</sup> This fiber is extensively employed in the manufacturing processes of twines, ropes, sacks, carpets, cordages, marine liners, and fishing nets due to its length, high strength modulus, and durability (especially in saline water). It also serves as a vital source of raw materials for the local hand-spinning and processing mills, thereby fostering supplementary employment opportunities. It is also used to manufacture dartboards, filters, mats, coffee bags, shopping bags, etc. Sisal reinforcing composites have the potential to serve as a viable alternative or supplement to fiberglass in the reinforcement of plastic materials in various applications, such as automotive components, marine vessels, furniture, water storage tanks, and piping systems. They can even enhance the structural integrity of cement mixtures, contributing to the construction of affordable housing.<sup>133,134</sup> Additionally, sisal fibers in reinforced composites can substitute for asbestos in manufacturing roofing materials and brake pads.<sup>134</sup> Moreover, it serves as an insulating material and can be transformed into a fiberboard as a viable alternative to wood. Due to its inherent porosity, this material also finds utility in diverse applications, such as cigarette paper filters.<sup>132</sup> Table 1.7 provides statistics of largest producers and exporters of raw sisal in 2018-2019.

#### 1.2.4 ANIMAL FIBERS

Animal fibers are those that are extracted from animals—either their pelages, like wool, or a product of their processes, like silk. The two most prominent animal fibers covered in this section are silk and wool.<sup>135,136</sup>

#### 1.2.4.1 Silk

A fiber famous for its luster and luxurious feel, silks are protein-based fibers spun by arthropods such as silkworms, spiders, scorpions, mites, and fleas.<sup>137</sup> Silk is popularly known as the 'Queen of Textiles' due to its sensuousness, elegance, comfort, drapability, and more, which makes it the go-to fabric for luxury items.<sup>138</sup>

Silk can be classified into two categories: mulberry silk, made by the Bombyx mori silkworm, and non-mulberry silk, like wild or Vanya silks, made by the Saturniidae



FIGURE 1.15 Classification of silk.<sup>138</sup>

wild silkworms (Figure 1.15).<sup>138</sup> The best silk is produced by the mulberry leaf-fed silkworms, Bombyx Mori, also known as the silk moth.<sup>139</sup>

Among Vanya silks, tasar silk fabrics are produced by handlooms, some of which are gicha-noil, tasar plain, cotton-tasar blend, tasar-mulberry blend, and peduncle fabric. Muga silk cloth is largely used by the Assamese women as mekhela, riha-sador sarees. Eri spun silk is used for dress material, and the coarse variety is used for making scarves, chadors, shawls, and quilts. Trimoulter silk yarn is used as package material for pencils and to make talcum powder puffs.<sup>140</sup> The waste silks are hand spun into matka, katan, feshua (jatan or Jatam, Jhut) and noil yarns.<sup>141</sup> Articles made from waste silk also have a good export market.<sup>140</sup> Figure 1.16 shown SEM image of silk.



**FIGURE 1.16** Scanning electron microscope longitudinal image of silk.<sup>142</sup> (Reprinted with permission from Ref. 142.)

The archaeological findings established the presence of silk in ancient Chinese civilization approximately 5000 years ago.<sup>143</sup> According to Chinese folklore, the practice of sericulture and the intricate art of silk weaving was attributed to the ingenious HsiLing-Shih, the wife of the Yellow Emperor, who reigned over China during the 3<sup>rd</sup> millennium BCE. In the realm of Chinese literature, the empress was commonly denoted as the revered "Goddess of Silk" after she discovered silk when a silkworm cocoon fell into her tea and the fibers unwound from it.143 The archaeological record states that the earliest discernible manifestations of domesticated silk usage in China can be traced back to the 3<sup>rd</sup> millennium BCE. This discovery is supported by the presence of remnants at the site of Qianshanyang, serving as a testament to the ancient Chinese civilization's engagement with silk. The samples from the earliest cultural deposit discovered at the site are attributed to the species Bombyx mori, dated to a period ranging from approximately 3500 to 2700 BCE. Biomolecular evidence of silk fibroin in the Tombs of Jiahu, a Neolithic site in the Henan province, has shed light on the existence of the fiber about 8500 years ago.<sup>143</sup> Silk remnants have been discovered in various archaeological sites, highlighting their historical significance. Notable findings include silk remains retrieved from a 1000 BCE Egyptian mummy, a mid-second millennium BCE burial site in Sapalli-tepe in Uzbekistan, a 7th century BCE German grave, and the 2nd century BCE Delivala Stupa site in Rambukkana in Sri Lanka. The earliest extant documented evidence about the presence of silk in China can be traced back to the oracle bone inscriptions dated to the Shang dynasty period.<sup>143</sup> As per historical accounts, the mastery of silk production techniques was diligently guarded within China until the advent of the Western Han Period, around the 2<sup>nd</sup> century BCE. The techniques of degumming and reeling, which are pivotal processes in silk production, have long been regarded as enigmatic secrets of the Chinese silk industry. Degumming entails the extraction of the outer sericin gum from silk fibers, leaving behind the inner fibroin through the immersion of cocoons in a low-alkaline solution. These long filament fibers, which happen to be the only naturally sourced filament fibers, are then cleaned, inspected, and sent for further processes.<sup>143,144</sup> The process of reeling silk entails the collection of long silk strands, (whether gummed or not) onto a bobbin, eliminating the necessity of twisting them into shorter segments to form a spun thread. This practice of sericulture and making silk textiles was swiftly disseminated to the Korean and Japanese regions, subsequently extending its reach to various European nations and beyond.<sup>143</sup> Silk was traded around the world as a luxurious and expensive fabric for a long time. Besides this, it was also used to heal war wounds. The war surgeon Ambroise Paré (1510-1590) reverted to using vascular ligatures made of silk or fine linen strips. However, only in 1869 did Joseph Lister introduce the first sterile silk suture into clinical practice, which has since been used for tendon tissue engineering, wound ligation, and more.<sup>145–147</sup> Silk is said to have made its way to India around 400 years back and reached its zenith during the medieval period.<sup>148,149</sup> The art of weaving silks flourished in the Mughal period in the court of Akbar.<sup>149</sup> Due to competition from China and Japan's advanced sericulture and production, India's silk industry declined.<sup>148</sup> Today, India not only contributes to global silk production, but it has favorable climatic conditions to grow all four silk types; Eri, Muga, Tasar, and mulberry.148

## TABLE 1.8Production of Raw Silk and Export of Raw Silk, 202168,151

Largest Producers and Exporters of Raw Silk

	Producers	Quantity (tonnes)	Exporters	Value (in thousand US \$)
First	China (mainland)	46,700	USA	68.47
Second	India	33,770	UAE	54.69
Third	Uzbekistan	2,037	China	21.56
Fourth	Vietnam	1,067	UK	19.88
Fifth	Thailand	503	Australia	12.98

Raw silk is used to make clothes, including shirts, suits, ties, blouses, lingerie, pajamas, and jackets. Hand-spun mulberry silk is used for comforters and sleeping bags. Mulberry silk is used to manufacture a range of textured fabrics, including dupions, plain silk, deluxe, satin, chiffon, chignons, crepe, and brocades. Home furnishing and decorative items like carpets, curtains, draperies, cushion coverings, and couch covers can also be made using silk. Knitted items made from silk fibers, such as socks and stockings, are expensive and in high demand as well. Silk glands are utilized to create the silk gut used in surgery for internal suturing. The silk glands are dissected, placed in warm water, and tugged at both ends to produce a thread of equal thickness. This protein is self-absorbable and does not need to be removed during wound healing. Silk grafts have been used successfully to repair damaged arteries.<sup>145,146,150</sup>

Silk has had several industrial and commercial applications. In France, 22–24 denier silk was utilized in tire manufacturing to provide a longer lifespan than rubber tires in bicycle tires and artillery ammunition bags. Parachutes used to be made out of 13–15 denier silk fiber. These parachutes were utilized during World War II.<sup>136</sup> Table 1.8 lists largest producers and exporters of raw silk in 2021.

#### 1.2.4.2 Wool

Wool is referred to as the fleece of various animals, including sheep, goats, rabbits, cashmere, angora, Persian goat hair, alpaca, vicuna, guanaco, fur from rabbit, camel, and even beavers, with sheep wool being the main source.<sup>152,153</sup> Wool is a member of a group of proteins known as keratin and contains about 33% keratin itself, alongside other elements like suint or dried perspiration (28%), burrs or dried vegetable matter (2–10%), fat (12%), dirt (26%) and mineral matter (1%).<sup>154</sup> Viewing wool fibers in a cross-section shows a discernible cortex composed of about 90% keratinous fiber and sometimes vacuoles filled with air known as the medulla, which is found only in coarse wool fiber (Figure 1.17). They also have a cuticle composed chiefly of lipids, carbohydrates, and proteins.<sup>152</sup> On its outer surface, wool fibers have unidirectional scales, which make them coarse and rough and, in turn, unsuitable for apparel due to the irritation caused.<sup>152</sup> Table 1.9 shows characteristics of different types of wool.

Indian wool is usually coarser than that produced in Australia, New Zealand, and other places, due to the different breeds of sheep. A few Indian breeds are Chokla, Nali, Magra, Marwari, Malpura, Kheri, Gaddi, and Rampur.<sup>155,156</sup> India has 44 indigenous


**FIGURE 1.17** Scanning electron microscope cross-section image (a) and longitudinal image (b) of Malpura wool.

TABLE 1.9   Categories of Wool <sup>152</sup>			
Type of Wool	Fine	Medium-Coarse	Coarse
Diameter (µm)	20–24 µm	24–30 µm	above 35 µm
Medullation	Medulla generally absent	Medullation present	Big medulla
Scales	790/cm	700–250/cm	276/cm
Lustre	Least	Moderate	Most

sheep breeds, along with a few crossbreeds. Many crossbred breeds yield wool with a fineness range from 25 to 60 µm.<sup>155</sup> Crossbreeds like Bharat Merino, Avivastra, and Avikalin have evolved by crossbreeding exotic sheep like Rambouillet and Australian Merino with the indigenous sheep, Chokla, Nali, and Malpura.<sup>155</sup>

Sheep had started to be domesticated in about 9000 B.C.<sup>157</sup> The Argali and the Musmon, native to America and Africa or Europe, respectively, were the primordial domestic wool-yielding animals to whom we are indebted for such a large portion of our clothing. The United Kingdom has also given a great variety of sheep to the world. During the Roman occupation of Spain, the area had a great reputation for its fine wool.<sup>153</sup>

The first indication of a fleece comes from a sculpture from Sarab, Iran, which dates back to the 6th millennium B.C. Very interestingly, in this sculpture, the wool is indicated by a series of V-like structures that are said to indicate the staples of a hairy medium fleece. The earliest wool textile remains are dated no earlier than 1500 B.C. These Bronze Age cloths have been preserved in Denmark by waterlogging and in Egypt by desiccation. Changes in the wool fiber diameter distribution during the fleece evolution show that wool has evolved by a change in many of the short, shedding kemps of the hairy medium fleece into long, continuously growing heterotypic hairs.<sup>157</sup>

In ancient times, the Greeks coated their helmets with felt, and Roman legionnaires had wool felt breastplates.<sup>158</sup> Woollen or worsted processing turns raw fiber into yarn. Knitted and woven wool items are made from yarn. To remove contaminants, wool fibers are washed and scoured after extraction. They may be combined with other

wool grades, carded, spun, dyed, woven, knitted, or non-woven to form a surface.<sup>159</sup> Wool is a versatile material that may be knitted, woven, wet, and needle-felted to create a variety of surfaces such as garments, blankets, horse rugs, saddle cloths, carpets, insulation, and upholstery control.<sup>158</sup> Woollen felt is considered the oldest known textile, although this technique is usually only seen in more art and craft objects than other product categories.<sup>160</sup>

Wool felt was used to cover piano hammers and stereo speakers and on the inside of heavy machinery to absorb noise and odor. Wool and cotton have also long been used in the production of diapers. Wool clothing is hydrophobic on the outside and hygroscopic on the inside, making it ideal for concealing soiled diapers. Wool felted with lanolin is water resistant, air permeable, and somewhat antimicrobial, making it ideal for odor control.<sup>158</sup>

Merino wool is used for high-quality carpets, beds, insulators, fertilizers, and firefighters' clothing. Wool and seaweed are used to reinforce bricks, making them stronger and more environmentally friendly. Wool has also been used in operations that require absorbing huge oil spills.<sup>161</sup>

The term 'wool' not only includes categories of sheep wool but also covers fibers like pashmina, alpaca, and more. Pashmina is a fiber famous for its ultra-soft hand and usage in Indian craft. It is the fiber obtained from the down of the Changthangi goat found in Kashmir.<sup>162,163</sup> Its length can range from 25–93mm, and its fineness is  $12-13\mu$ .<sup>163</sup> One of its most intriguing qualities is increasing warmth without increasing weight.

Alpaca fiber is another specialty fiber used to create a variety of garments or items, similar to sheep's wool, like blankets, sweaters, hats, gloves, scarves, coats, a wide variety of textiles, and ponchos in South America and other parts of the world. Figure 1.18 shows SEM image of Alpaca fiber. Alpacas have two breeds – the Huacaya breed, which is widely prevalent in the Australian region, renowned for their dense fleece and highly crimped fibers, and the Suri, which is characterized by its scarcity and coveted status due to its opulent, luxuriant fleece exhibiting silky strands.<sup>165,166</sup> In the mid-1800s, the alpaca populations experienced a notable resurgence, subsequently attaining a state of natural distribution within the regions of Peru, Chile, and Bolivia. Deluxe to the touch, lightweight, warm, and cozy garments



**FIGURE 1.18** Scanning electron microscope of the longitudinal image of Alpaca fiber. (Reprinted with permission from Ref. 164.)

Largest Producers and Exporters of Shorn Wool				
	Producers	Quantity (tonnes)	Exporters	Quantity (tonnes)
First	China (mainland)	3,56,19	Norway	2838.861
Second	Australia	3,28,00	Switzerland	509.134
Third	New Zealand	126,880.08	Brazil	4751.972

# TABLE 1.10Production of Raw Shorn Wool 2022 and Export of Raw Shorn Wool 202368

made from alpaca fiber are quickly catching on as one of the world's best-kept secrets in the clothing and fashion industry.<sup>165,166</sup> Table 1.10 lists largest producers and and exporters of shorn wool and their production quantities.

### **1.3 APPLICATIONS OF NATURAL FIBERS**

Learning more about natural fibers, their properties, strengths, weaknesses, and constituents enables us to include them in products as we deem fit through design processes. In today's world, replacing man-made fibers with natural ones is a viable and necessary solution that must be applied to minimize the waste and destruction that most facets of the textile industry contribute to.<sup>7,167</sup>

As seen in Figure 1.2, the production areas of the major natural fibers were scattered diversely across the world. As per Tables 1.1 and 1.2, variations in the diameter of the different natural fibers have been noted. The finer fibers, such as cotton, hemp, linen, silk, and wools like alpaca and pashmina have been very useful for clothing since immemorial. The scanning electron microscopy of longitudinal and crosssectional views of different natural fibers revealed some useful information. Silk has the smoothest surface, making it one of the most lustrous fibers. Finer in diameter and lustrous in nature, silk has been one of the most sought-after materials for excellent textiles. Wools have noticeable scales on their surfaces, which enable felting. Coarse wools have different degrees of medullation in their cross-section. The coarser wools were mostly used to produce felts, quilts, and other insulation products, due to the presence of hollow medulla. Cotton, a cellulosic fiber, has been one of the most used materials for producing textiles. Finer lignocellulosic fibers such as hemp, linen, and ramie are found to be present in textiles, mainly in apparel. The coarser lignocellulosic fibers, on the other hand, such as jute, sisal, ramie, and coir, were mainly used for packaging and other household purposes, owing to their robust nature. However, their larger diameter posed limitations in usage. Lignocellulosic fibers notably all have polygonal cross sections with uneven surfaces, which gave them unique textures and a slightly rougher feel, depending upon the stage of their life cycle and diameter. The presence of strength and lumen in the fibers made them useful for insulation and composite-based applications. Today, with the understanding of modern instruments, plenty of scientific reports suggest newer and more effective uses to utilize the potential of natural fibers. As seen in Table 1.11, each fiber has the traditional label of where it was used and contemporary uses, which are up-and-coming in the field.

# TABLE 1.11Application of Major Natural Fibers, Their Man-Made Alternatives, and Contemporary Products

Fiber	Products	Alternative Fibers	Diversified Use
Cotton <sup>[31,168–171]</sup>	Apparel (shirts, t-shirts, dresses), Home (bed linens, blankets), Nonwovens (gauze bandages, filters, sanitary pads).	Regenerated fibers (rayon, viscose) and polyester fabrics for apparel, blends of rayon, nylon and more for home, polypropylene, rayon.	Tents, tarps, bank notes, cords, and ropes. Cotton linters (the short fibers that remain on the plant after it is ginned) are also used in mattresses, furniture, automobile cushions.
Coir <sup>[64]</sup>	Biodegradable natural-fiber meshes, erosion control blankets, ropes, cordages, geotextiles	Net, wire mesh and aspen fiber, erosion control mats, polypropylene meshes.	Natural fiber composites (biocomposites), geotextiles as an interface between the sub grade and the sub- base of pavements, pre-fabricated vertical drains (PVD) and as road edge drains.
Linen <sup>[80,172]</sup>	Bed and kitchen linens, suiting, clothing apparel, surgical and sewing threads, sailclothsheets, collars, cuffs.	Acrylic, poly-blends, and viscose are used in bed linens; rayon and polyester used for sewing threads.	Insulation materials, filtration application, fabrics for light aviation purposes, reinforced plastics and composite materials.
Jute <sup>[121,173]</sup>	Hessian sacks, carpet backing cloths (CBC), rugs, mats, home textiles, handicrafts, shoes.	Polythene shopping bags, glass and carbon fiber geotextiles, rubber and foam used in shoes, sandals and espadrilles.	Automobiles, pulp and paper, furniture, bedding, technical textiles, non-wovens, composites, roof coverings, soundproofing insulation materials, and laminated shopping bags, technical textiles, geotextiles, jute-reinforced composites, pulp, paper, particle boards, shopping bag, fashion accessories.
Hemp <sup>[88,174,175]</sup>	Produce rope, cloth, oil lamp wicks, apparel, home furnishing.	Polypropylene, nylon, rayon ropes, poly-blend and regenerated fiber apparel and home furnishing items.	Used as aggregates with lime binders, added in reinforcement, beds for animals, banknotes, and paper.
Ramie <sup>[88,124]</sup>	Sewing thread, packing materials, fishing nets, filter cloths, apparel, curtains, draperies, upholstery, bedspreads, table linens, sheets, dish towels.	Polyester threads, poly-blends, viscose, rayon-made apparel and home furnishing items.	Parachute, woven fire hoses, thin weaving, canvas, filter cloth, paper products, and bank notes and delicate cigarette papers.

(Continued)

# TABLE 1.11 (CONTINUED)Application of Major Natural Fibers, Their Man-Made Alternatives, and Contemporary Products

Fiber	Products	Alternative Fibers	Diversified Use
Sisal <sup>[130,132]</sup>	Twines, ropes, cordages, marine liners, fishing nets, dartboards, filters, mats, coffee bags, shopping bags, buffing cloth.	Nylon, polyester, rayon twines and cordages, plastic shopping bags, shellac and leather buffing cloth, plastic, metal, glass, leather furniture and home décor.	Sisal is a reinforcing material and can be used in place of asbestos and fiberglass in reinforced plastic composite materials, automotive components, marine vessels, water storage tanks, piping systems, roofing materials, brake pads, cigarette paper filters, furniture, and fiber board.
Silk <sup>[140]</sup>	Raw silk was used for clothing like shirts, suits, ties, blouses lingerie, pajamas. Hand-spun mulberry silk used to make comforters and sleeping bags, dupions, plain silk, deluxe, satin, chiffon, chignons, crepe, brocades are made from mulberry silk, carpet, furnishing, curtains, draperies, cushion covers and sofa covers, wall hanging, knitted materials for socks, stocking, silk gut used in surgery for internal suturing is made from silk glands, waste silks are hand spun into matka, katan, feshua (jatan or Jatam, Jhut) and noil yarns powder puffs. Parachutes used to be made from 13–15 denier silk fiber.	Poly-blends and regenerated fibers like viscose, rayon, lyocell used to make apparel, innerwear, ethnic wear, pillowcases, blankets, decorative fabrics.	Trimoulters silk yarn is used as package material in pencil industry and for making talcum powder puffs. The silk gut used in surgery for internal suturing is made from silk glands. Silk grafts have been used successfully to replace cut arteries.
Wool <sup>[158,161]</sup>	Garments, blankets, horse rugs, saddle cloths, carpets, insulation, upholstery, high-quality carpets, beds, insulators, and fertilizers, as well as fire-fighter's clothing.	Poly-blends and acrylic used to make garments and home furnishing, and Nomex and Aramids used in firefighters' uniform.	Cover for piano hammers, stereo speakers, and inside of heavy machinery to absorb noise and odor, used in diapers due to absorbency and wool felted with lanolin is water resistant, air permeable, and somewhat antimicrobial, making it ideal for odor control. Wool and seaweed are used to reinforce bricks, making them stronger and more environmentally friendly. They also have potential for absorbing oil spills.

# 1.4 CONCLUSION

In this chapter, we have discussed the notable natural fibers from the historical perspective regarding their growing areas, documented history of usages, along with their surface and cross-sectional morphology, and the alternative major synthetic fibers developed over time that took a major place in the textile market. Natural fibers are a gift of nature that has shaped human civilization. As a society, we must keep innovating with these fibers and fully use their potential. Because of these early fibers, so many socio-economic human systems fell into place. Trade is one such system that textiles brought the most out of. The popular Silk Route had a large role in disseminating fibers and knowledge about them to different parts of the world. Subsequently, colonization played a major role in the destruction of the textile industries of local and indigenous people in colonial countries. Mechanized techniques of creating textiles came into being along with the industrially-driven modern world we see today. The rise of synthetic fabrics in the 19th century changed clothing habits and notably replaced products made from their natural counterparts. However, looking at the adverse effects of the rampant use of synthetic fibers, researchers started looking for viable alternatives and continue to look for suitable natural fibers that can replace synthetic fibers for clothing and industrial purposes. Coming back in a full circle, natural fibers' durable, renewable, and relatively less harmful qualities made them a viable solution to minimize the destructive effects of synthetic fibers, for a better future. This chapter may help researchers bridge the use of traditional fibers and their possible application in wider fields, given the historical and technical aspects of natural fibers discussed in this chapter.

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# 2 Eco-Friendly Natural Fibers Based Sustainable Agro-Textiles for Packaging of Agro-Products

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# 2.1 INTRODUCTION

Agro-textiles, or agricultural textiles, are specialized fabrics or materials used in the agricultural industry to enhance crop growth, protect plants, and improve overall farming practices. These textiles are designed to address various agricultural challenges, such as weather extremes, pest control, soil erosion, and water conservation. Agro-textiles offer a range of functionalities, including providing thermal insulation to crops during cold weather, shielding plants from excessive sunlight, and reducing water evaporation from the soil. They are often employed as crop covers, shade nets, ground covers, and mulches. In recent years, agro textiles have gained significant popularity due to their eco-friendly nature and ability to optimize crop production while reducing the dependency on chemical inputs. These materials can positively impact crop yield, quality, and sustainability, making them essential tools in modern agricultural practices [1]. The ongoing research and development in agro textiles will lead to innovative solutions that further revolutionize and improve farming practices worldwide. Agro-textiles, specifically designed for agricultural applications, have gained prominence due to the global rise in population and the growing demand for high-quality farm produce. These textiles play a crucial role in enhancing agricultural yields and agro-product quality. The choice of textile fibers is essential in determining agro-textiles' effectiveness in various applications [1]. There are different types of fibers used in agro-textiles. Plant fibers such as wool, jute, coir, sisal, and hemp, as well as synthetic fibers including PP (polypropylene), PE (polyethylene), PET (polyethylene terephthalate), nylon, and regenerated fibers like viscose, lyocell, and PLA (polylactic acid), are commonly utilized in agricultural products. Synthetic fibers are preferred over natural fibers due to their high strength, durability, and other favorable properties for agricultural applications [2]. However, using synthetic fibers has

raised environmental concerns, leading to a demand for more eco-friendly alternatives. Researchers and manufacturers are actively seeking renewable and sustainable fibers with minimal environmental impact. Natural fiber (wool, cotton, cellulose) consumption has declined drastically compared to synthetic fibers over the past few decades. Natural fibers comprised almost 96% of the world's fiber consumption in the 1960s, but has now drastically declined to a mere 27%. Choosing natural fibers can make a huge difference in protecting our earth against pollution [3]. In terms of applications, textile structures in various forms are employed in different agricultural settings. Shade houses, polyhouses, and greenhouses utilize agro-textiles to control environmental factors such as temperature, water, and humidity. These textiles help create a favorable microclimate for plant growth and protect crops from adverse weather conditions. Additionally, agro-textile products such as sunscreens, bird nets, windshields, mulch mats, hail protection nets, and harvesting nets are widely used. Sunscreens provide shade and protection against excessive sunlight, bird nets prevent birds from damaging crops, windshields act as windbreaks, mulch mats control weed growth and soil moisture, hail protection nets safeguard crops from hailstorms, and harvesting nets assist in the collection of fruits or vegetables [4]. Overall, agro-textiles offer innovative solutions for improving agricultural practices and addressing farmers' challenges. The choice of fibers and the application of these textiles contribute to higher crop yields, better product quality, and more sustainable farming methods [4].

# 2.2 CHARACTERISTICS OF AGRO-TEXTILES

- **Resistance to Solar Radiation**: After sowing or planting, agro-textiles are placed over the cultivated areas; therefore, they must withstand solar radiation and temperature variations in the surrounding air. Since the intensity of the radiation directly affects plant growth, all shading techniques aim to control the amount of solar energy that enters the greenhouse and minimize solar radiation [4, 5].
- **Resistance to UV radiation**: UVPF (ultraviolet protection factor) is highly dependent on the chemical structure of the fiber. Compared to synthetic fibers like PET, natural fibers like cotton, silk, and wool absorb UV rays to a lesser extent. For comparable construction, the UPF rises with fabric density and thickness and depends on porosity. Strong morphological impacts caused by UV radiation on plants include changes in the root-shoot ratio, cotyledon curling, leaf thickness, and leaf discoloration [4, 5].
- **Retention of water**: Agro-textiles help to conserve water and retain moisture in the soil, boosting crop output.
- **Protection property**: Agro-textiles should have adequate elasticity, rigidity, flexibility, and dimensional stability.
- **Lightweight**: The weight of the agro-net should be sufficient to support the plant. Lower-weight materials should be chosen to allow crops to grow naturally [4, 5].
- Weather resistance: The agro-textile product must function efficiently in both cold and hot climates. Polyethylene, polypropylene, polyester, and nylon are common synthetic fibers.

- **Resistance to Microorganisms**: Physical, mechanical, and chemical qualities are lost as microbes operating in both aerobic and anaerobic environments break down the fiber polymer. The product should be resistant to microorganisms to guard against fungus and other illnesses brought on by persistent wetness [4, 5].
- **Strength**: An essential feature of agricultural nets is their tensile strength, determining their long-term resilience and lifespan because textiles have a high rip strength, punctures in them do not spread quickly [5].
- Abrasion resistance: This is necessary to protect crops from animal and pest attacks. Environmental concerns about the indiscriminate use of huge nets are growing. However, as aquaculture has grown, new requirements have emerged, such as nets capable of continuous immersion and increased abrasion resistance [5].
- **Flexibility**: Fabric structures are regarded as a subcategory of tensile structures that enable the use of agro-textiles in various settings.
- **Durability**: Agro-textiles can improve agricultural goods' quantity, quality, and safety due to their superior environmental resistance, mechanical capabilities, ease of processing, and durability.

# 2.3 TYPES OF AGRO-TEXTILE PRODUCTS BASED ON THEIR STRUCTURE AND ITS PROPERTIES

Agro-textile products can also be classified based on the fabric production technique used to manufacture them. Here are some classifications of agro-textile products based on fabric production techniques.

# 2.3.1 WOVEN AGRO-TEXTILES

Woven agro-textiles are specialized textiles designed and used in agricultural applications. They are woven fabrics made from synthetic or natural fibers engineered to provide specific functionalities and benefits for the agricultural industry. Here are some key aspects of woven agro-textiles [5].

- **Material Composition**: Woven agro-textiles can be made from various materials, including polypropylene, polyethylene, polyester, and natural fibers such as jute or cotton. The choice of material depends on the specific application and the desired properties of the textile.
- **Strength and Durability**: Woven agro-textiles are known for their strength and durability. They are designed to withstand the harsh conditions of agricultural environments, including exposure to sunlight, moisture, and pests. The woven structure provides structural integrity and enhances the overall strength of the textile [5].
- Weed Control: Weed-control fabrics can be made from woven agro-textiles. They are spread on the ground before planting crops to function as a barrier to weed growth by blocking sunlight and limiting their emergence. This helps to limit crop and weed competition for resources and nutrients [5].

- Erosion Control: Woven agro-textiles are also utilized for erosion control in agriculture. They can stabilize soil on slopes, preventing erosion caused by wind and water. The textiles help retain soil particles and promote vegetation establishment, further aiding erosion prevention [5].
- **Moisture Management**: Some woven agro-textiles are designed to manage moisture levels in the soil. They can help regulate water absorption, retention, and drainage, ensuring optimal moisture conditions for plant growth. This feature is particularly useful in regions with irregular rainfall or greenhouse cultivation [5].
- **Temperature Regulation**: Certain types of woven agro-textiles possess insulating properties that help regulate temperature. They can protect crops from extreme heat or cold, creating a microclimate that promotes healthy growth.
- UV Protection: Woven agro textiles often incorporate UV stabilizers that protect crops from harmful ultraviolet (UV) radiation. This is especially important in regions with intense sunlight, where prolonged exposure to UV rays can damage plants [5].

# 2.3.2 NON-WOVEN AGRO-TEXTILES

Non-woven agro-textiles are specialized fabrics used in agricultural applications. They are made from synthetic fibers that are bonded together using various techniques, such as thermal, mechanical, or chemical bonding, rather than being woven or knitted. These textiles offer several advantages in agriculture due to their unique characteristics [5].

Here are some key features and applications of non-woven agro-textiles:

- Water permeability: Non-woven agro-textiles are typically permeable to water, allowing efficient water penetration and distribution throughout the soil. This feature is essential for irrigation systems, as it helps maintain proper moisture levels and prevents waterlogging [5].
- **Moisture retention**: Agro-textiles can also act as a moisture barrier, reducing water evaporation from the soil surface. This property helps conserve water and promotes optimal growing conditions for plants.
- **Protection against pests and insects**: Agro-textiles can act as a physical barrier, protecting crops from pests and insects. They create a barrier that prevents pests from reaching the plants, reducing the need for chemical pesticides and promoting environmentally friendly pest management practices [5].
- **Erosion control**: Non-woven agro-textiles can help prevent soil erosion. They stabilize the soil by providing a protective layer that holds the soil particles together, reducing the impact of wind and water erosion [5].
- **Crop cover and insulation**: Agro-textiles can be used as a crop cover to protect plants from extreme weather conditions, such as frost, hail, or excessive heat. They provide insulation, retain heat, protect crops from temperature fluctuations, and extend the growing season.

- Seedbed protection: Non-woven agro-textiles can be used to cover seedbeds, protecting seeds from birds and other animals that may eat or disturb them. This helps ensure proper germination and establishment of the seeds [5].
- Nursery and greenhouse applications: Agro-textiles find wide applications in nurseries and greenhouses. They can be used as ground covers, shading materials, or protective covers for potted plants, providing optimal growing conditions, and enhancing plant growth [5].

# 2.3.3 KNITTED AGRO-TEXTILES

Knitted agro-textiles, also known as agricultural textiles or agro textiles, are specialized fabrics used in various agricultural applications. These textiles are produced by knitting techniques, which involve interlocking loops of yarn to create a flexible and porous structure [6].

Here are some common types and applications of knitted agro-textiles:

- **Support net**: Climbing plants such as tomatoes, cucumbers, and beans are supported and trained using support nets. They aid in keeping the plants upright and allow for enhanced air circulation and sunshine exposure, which can lead to increased growth and output [6].
- **Root ball net**: Root ball nets are specialized agricultural and horticultural goods used to support and protect plant roots during various stages of cultivation, transplantation, and growth. These textile materials, consisting of synthetic or natural fibers, are intended to serve various agricultural functions, including erosion control, weed suppression, moisture retention, and root protection. Root ball nets are used to secure and preserve plant root systems during transplantation or relocation [6].
- **Packing sacks for vegetables**: These nets are used for packaging vegetables. They help protect vegetables during transportation and storage. The nets allow air to circulate around the vegetables, which can help prevent moisture build-up and the growth of mold [6].
- **Tubular packing nets for fruits and vegetables**: Tubular packing nets for fruits and vegetables are specialized materials designed to provide protection, support, and transportation solutions for agricultural products. These tubular packing nets are commonly used in the agricultural industry for various purposes, including packaging and transport [7].

# 2.4 DIFFERENT TYPES OF AGRO-TEXTILES FOR PACKAGING OF AGRO PRODUCTS WITH CONSTITUENT FIBER

Agro-textiles are specialized textiles used in agriculture to improve crop yield, protect plants, and optimize growing conditions. These textiles are made from various types of fibers that offer specific benefits. Some common agro-textile products and their constituent fibers are listed in Table 2.1.

Product	Fiber Type
Packing sacks material	Jute plastic, polypropylene, HDPE
Leno net	Co-polymer nylon, PE, PP, HDPE
Tububar Net	HDPE, Nylon, Cotton
Root Ball Net	Cotton, polyester, Cot/polyester blend, HDPE
Support nets	Nylon, HDPE
Plant net	HDPE, PET
Flower & vegetable support net	HDPE, PET
Nets for covering pallets	Polyethylene, nylon, polyester, HDPE

# TABLE 2.1Common Agro-Textile Products and Their Constituent Fibers

As the population increases, the demand for textiles also increases, directly affecting the environment. So, nowadays, people are more concerned about sustainability and durability. Governments across the globe are taking measures by providing environmental guidelines/regulations to control unsustainable consumption of petroleumbased products and increase environmental consciousness and community interest. Natural fibers derived from renewable natural sources are environment-friendly, biodegradable, sustainable, and have inherent benefits [8]. Natural fibers are broadly categorized into three categories based on their origin (Figure 2.1).

- · Plant-based fibers
- Animal-based fibers
- · Mineral-based fibers



FIGURE 2.1 Classification of natural fibers based on their origin.

TABLE 2.2 World Production of Natural Fibers		
Fiber Source	World Production (10,000 Ton)	
Flax	83	
Jute	230	
Kenaf	97	
Hemp	21.4	
Wool	130	
Cotton	2500	
Coir	10	
Abaca	7	
Bamboo	3000	
Sisal	37.5	
Grass	70	

The world production of apparel and textiles in 2018 was recorded as 110 million tons, and only 29% of the total is natural fiber [8]. The worldwide share of different types of natural fibers per 10,000-ton natural fiber production is given in Table 2.2 [9].

In the last decade, the worldwide composite industry situation was that the industry sector reached U\$ 2.1 billion in 2010, and now the current point is that the industrial interest in natural fiber composite will grow quickly worldwide. The usage of NFPCs (Natural *Fiber Reinforced Polymer Composite*) has multiplied appreciably within shopper products as industry sectors have grown significantly over the past few years. As indicated by opinions, over five years (2011–2016), the NFPCs industry was envisioned to develop 10% worldwide [9].

# 2.5 IMPORTANCE/SIGNIFICANCE OF SUSTAINABLE AGRO-TEXTILES PACKAGING PRODUCTS

The importance or significance of sustainable agro-textile packaging products can be viewed from five perspectives, (i.e., benefit to society, import substitution, economic benefits, environmental benefits, and technical benefits).

# 2.5.1 BENEFIT TO SOCIETY

Using natural fibers, such as Indian wool, in agro-textiles would diversify the technical textile industry's product line and motivate the shepherd community to increase output, which will increase the price of raw materials. Producers of cotton, jute, and other natural fibers will profit similarly [9].

# 2.5.2 IMPORT SUBSTITUTION

Most agricultural nets and filters are constructed entirely of imported polyester, nylon, or plastic from different nations. These kinds of textiles are produced using

foreign yarns or occasionally chips made of polyester and plastic. Imported yarns and chips come from the USA, Germany, China, Japan, Taiwan, and other countries. Therefore, natural fiber-based sustainable agro-textile would replace imports for India [10]. The total Value and Volume of Imports in India for Synthetic fibers per year is approximately USD 1,531,721 and total quantity is 62,129, respectively. Considering utilization of natural fibers of a minimum of 30% in agro packaging products would save import of approx. USD 4,59,516 per year [10].

### 2.5.3 ECONOMIC BENEFITS

Pests and diseases harming crops are a global issue. Numerous pests have hurt agricultural crop yield. Many negative consequences have resulted from agriculture's overuse of pesticides, such as higher plant residue levels, insect resistance, and contamination of the air, water, and soil. Ecosystems are greatly impacted when pesticide residues build up and disrupt the food chain [10].

Mice are naturally present in crops in tiny quantities but thrive in advantageous conditions like high crop yields and poor harvesting efficiency [10].

- Early fall rains lead to winter weed seed set.
- Late spring or early summer showers damage mature crops, resulting in a flood of summer weeds.
- Favorable burrowing conditions, such as cracked or light soils.

# 2.5.4 BENEFITS TO THE ENVIRONMENT

Current agricultural packaging is made of polyethylene, which is not biodegradable. When used, sun radiation causes the polyethylene to break down into microplastic, ending in landfills or agricultural soil. It has been demonstrated that these tiny microplastic particles, which concentrate on persistent organic pollutants, represent major threats to different ecosystems [10] because plastic-based mulch heavily contaminates soil, recycling it is difficult, leaving either landfilling or incineration as an environmentally harmful alternative.

Natural fiber-based agro products will be biodegradable, and degraded products will also add nutrients to the soil over a few years.

# 2.5.5 TECHNICAL BENEFITS

Added micro capsule-based finishing could protect against rodents and microorganisms, including bacteria, fungi, and viruses, and it has UV-repellent properties. Due to improper handling and preparation, foods can harbor bacteria, viruses, parasites, fungi, and even prion infections that could cause diseases. This results in foodborne illnesses, which kill 420,000 people a year and impact 10% of the world's population [11]. Preharvest and post-harvest yield loss are significantly impacted by agronomic pests. One of the main pests of crops is thought to be rodents. Rodents store between 0.5 kg and 4 kg of grain in designated compartments in their tunnels. Approximately 10% of rodent species have an impact on agriculture collectively. Rats harm crops by around 30% a year on a global scale. The most frequent cause of problems is the "*Bandicota bengalensis*" kind of rat, widespread in residential and agricultural regions [11]. Hence, sustainable and functional packaging for agro-products with protection against rodents, microorganisms including bacteria, fungi, and viruses, and UV-repellent properties is a need of the hour [11].

As of today, plastics (rigid and flexible) have the largest share of the market in food packaging (37% market), followed by paper and board (34%), glass (11%), and metal (9%) [10]. If we look at the characteristics of the given food packaging materials, paper, and board have a large market share, which can be attributed to their recyclable and renewable source, printable nature, ability to store both wet and dry food after laminating or covering, and overall cost-effectiveness due to their suitability for mass production [11]. In Europe, PP (19.3%), LDPE and LLDPE (17.5%), and PET (7.4%) are the most often used plastics in food packaging. 39.9% of all plastics manufactured (61.8 million tonnes in Europe in 2018) are used in packaging [11]. Sustainable Agro-textiles are crucial in promoting environmentally friendly and efficient agricultural practices. These textiles, also known as agricultural textiles or agro-textiles, are designed specifically for agricultural applications and aim to enhance productivity while minimizing negative environmental impacts. By conserving resources, protecting crops, managing water efficiently, and improving yields, these textiles promote sustainable farming and support the goal of achieving a more sustainable and resilient food production system [12].

# 2.6 FUNCTIONAL FINISHES IN AGRO-TEXTILES FOR PACKAGING OF AGRO-PRODUCTS

Finishing is the process of giving the texture and function required by the final product, depending on the application, while taking advantage of the characteristics of the textile.

### 2.6.1 RODENT REPELLENT FINISHING

Quantitative damage caused by Rodents

- 33 MT losses annually (worldwide) [13]
- 10% post-harvest losses of total produce
- 75% losses to rice and wheat
- 60–70% open sack storage [13]
- 6% loss (out of which 50% due to rodents) [13].

Rodents are the most common vertebrate pests, causing direct harm to crops and commodities through feeding and indirect damage through spoiling, contamination,

and hoarding on farms and after harvest. The study of pre-harvest losses reveals a range of 5–15% harm to cereal crops such as rice and wheat [13]. These damages can be prevented, and crops can be stored safely if effective measures are employed. Also, the numerous diseases caused by rodents, which are transmitted due to contaminated food items, can be avoided and prevented. For instance, in the pre-harvest stage of only the rice crop in India, the damage caused by rodents to the paddy tillers was 13.86% in the State of Punjab, but 8.62 to 9.5%, 8.43 to 9%, and 5.75 to 6.23%) in the districts of Nadia, Hooghly, North 24-Parganas, and Burdwan, respectively. Rat infestation caused paddy crop losses ranging from 4.6 to 54% [14]. Rodent-repellent finishing for agro-textile nets in the packaging can help protect agricultural products from rodent damage. There are several ways to achieve this, and the choice of rodent-repellent finishing depends on the application's specific needs [14].

#### 2.6.2 NANOTECHNOLOGY-BASED FINISHING

Nano-sized particles like silver or copper nanoparticles can be incorporated into textile fibers or coatings. These nanoparticles possess antimicrobial properties and can effectively inhibit the growth of microorganisms. Functional finishes involving nanotechnology can play a significant role in packaging materials to enhance the preservation of fruits, vegetables, and fresh meats by inhibiting the growth of microorganisms, including bacteria, fungi, and viruses. This is achieved through the incorporation of nanoparticles like zinc (Zn), silver (Ag), copper (Cu), silicate (SiO<sub>2</sub>), and zinc oxide (ZnO) into the packaging materials [15]. Nanoparticles like silver, copper, and zinc have well-documented antimicrobial properties. They can release metal ions that are toxic to microorganisms, effectively inhibiting their growth and proliferation. This is particularly important for preventing spoilage and contamination of food products. Food products can have a longer shelf life by creating a barrier to external gases and inhibiting microbial growth. This reduces food waste and maintains the items' nutritional value and quality [15]. The combination of silver (Ag), copper (Cu), silicate (SiO<sub>2</sub>), and zinc oxide (ZnO) nanoparticles in agro-textile products for packaging can offer various benefits. These nanoparticles can impart several functional properties to the textiles used in agriculture [15]. Silver and copper nanoparticles are known for their strong antimicrobial properties. Incorporating these nanoparticles into agro-textiles can help inhibit the growth of bacteria, fungi, and other microorganisms, which can be especially useful for packaging materials in agriculture where spoilage or contamination is a concern [15]. Zinc oxide nanoparticles can provide UV protection to textiles, shielding agricultural products from harmful UV radiation. This is essential for light-sensitive products that may degrade when exposed to the sun. Silicate nanoparticles can help improve the durability and abrasion resistance of textile materials. This is important for packaging materials, as they need to withstand handling and transport without tearing or degrading [15]. Nano clay finishes, such as those based on nano clay particles or nanocomposites, can be used in agro-textiles packaging products for various purposes. These finishes can enhance the performance and durability of agro-textiles, making them suitable for agricultural applications. Functional finishes with nanoparticles can reduce the need for chemical preservatives in food packaging, making it a more environmentally friendly and healthier option [15]. Nanotechnology-based functional finishes in food packaging offer innovative solutions for extending the shelf life of perishable goods, reducing food waste, and enhancing food safety [15].

Nano-biopesticides, derived from various sources like plants, microbes, and their derivatives, represent an eco-friendly approach to pest control. They offer several advantages, including slower release, prevention of degradation, longer activity, and biodegradability. Neem-based pesticides are a prime example of such nano-biopesticides, and they are utilized in various applications, including agro-textile products and packaging [16]. Neem-based pesticides are derived from the neem tree (*Azadirachta indica*) and have been used for centuries in traditional agriculture. The key active component in neem-based pesticides is azadirachtin. These pesticides are considered eco-friendly and are effective against a wide range of pests. They have been widely used in organic farming and are a prime example of natural nano-biopesticides [16]. Neem-based nano-biopesticides can be incorporated into agro-textile products, such as crop covers and packaging materials. These products help protect crops from pests and reduce the need for synthetic chemical pesticides. Neem-based packaging can also provide natural pest resistance to stored agricultural products [16].

# 2.7 IMPORTANCE OF FUNCTIONAL FINISHES IN AGRO-TEXTILE USED FOR PACKAGING PRODUCTS

Agricultural produce quality and safety during storage and transportation are greatly enhanced by functional finishes in agro-textiles used for product packaging. Functional finishes are crucial in agro-textile packaging for the following main reasons:

- **Moisture Management**: The amount of moisture inside the container can be managed using agro-textile materials with moisture management finishes. This is necessary to keep the produce that has been stored from developing mold, rotting, and deteriorating [17].
- **UV Protection**: Certain agricultural goods are susceptible to damage from ultraviolet (UV) light. Functional coatings with UV protection can block UV radiation from damaging the contents and causing deterioration [17].
- **Pest and Insect Resistance**: Some agro-textiles can be treated with finishes that deter pests and insects, reducing the risk of infestation and damage to the stored produce [17].
- **Temperature Control**: Agro-textiles with insulating or temperaturecontrolling finishes can help maintain a stable environment within the packaging, preventing temperature fluctuations that can negatively affect the produce [17].
- **Strength and Durability**: Functional finishes can enhance the strength and durability of agro-textiles, ensuring that the packaging can withstand the rigors of transportation and handling without tearing or breaking [17].

# 2.8 AGRO-TEXTILE PACKAGING PRODUCTS – AVAILABILITY

Some common agro-textile packaging materials available in the market are shown in Figure 2.2:

- **Plant nets**: Plant netting is an enclosed structure made up of woven material or Agro-nets to allow enough sunlight, air, and moisture to pass through the gaps. Apart from extreme sunlight and hot winds, plant netting protects gardens from insects, pests, and birds that can damage soft-ripening fruits and crops.
- **Root ball net**: Root ball nets are biodegradable packaging materials that safeguard root balls during transit and storage. It also aids in the retention of soil around the roots.
- **Support nets**: These nets support climbing vines and vertically growing plants while promoting bloom growth. It allows huge fruit to be held by plants.
- **Packing sacks for vegetables**: These nets are used to pack various farm products. It may be packing sacks or tubular packing nets.
- Net for covering pallets: Pallet nets can assist with minimizing the movement of goods on pallets and help keep items in their position. It is strong, durable, and suitable for use in busy environments.
- Flower and vegetable support net: Flower and Vegetable Support Netting allows plants to receive the maximum light and air while protecting important flower yields.
- Leno bags: Leno bags are used to pack fresh vegetables, fruits, potatoes, onions, etc. They are specially woven and designed to maintain the freshness of the packed goods by allowing air inside the bags to prevent inner moisture that may spoil the goods.
- **Tubular packing nets for fruits and vegetables**: Tubular packing nets for fruits and vegetables are innovative packaging solutions designed to protect and support delicate produce during transportation and storage.



FIGURE 2.2 Some common agro-textile packaging materials.

# 2.9 AGRO-TEXTILE PACKAGING PRODUCTS – TESTING STANDARDS

Some common tests available for agro-packaging products are listed in Table 2.3.

The particular applications for which agricultural and agro-packaging products are intended vary in terms of their mechanical requirements. Support nets for flowers and vegetables, plant nets, root ball nets, support nets, vegetable packing sacks, pallet covering nets, leno bags, and fruit and vegetable tubular packing nets are a few. High-modulus polyethylene (HDPE) is the most widely used material for agricultural nets. Its structure and properties make it an attractive choice for various applications [18]. Alternative natural fiber-based agro-packaging products have a huge market potential.

The way these products are made has a significant impact on how they behave mechanically. Agricultural net structures are primarily fabricated using two methods: (a) knitting (Raschel weave) and (b) weaving (flat or Leno weave). The technical literature contains a dearth of information about the mechanical propulsion characteristics of knitted or woven nets, with no specific mention of agricultural nets [18, 19].

The mechanical properties of available agricultural nets produced and utilized in various agricultural applications can be tested per International standard testing procedures. [18]. Table 2.4 below shows a list of some common test methods.

# TABLE 2.3List of Tests for Available Agro Packaging Products

**Construction Parameters** 

Mesh Size GSM EPI-PPI/WPI-CPI Warp Count/Weft Count Type of Material

#### **Mechanical Parameters**

Tensile Strength Bursting Strength Tensile Strength after UV exposure Color Fastness to UV Color Fastness to Light

# TABLE 2.4List of Mechanical and Performance Test Standards

#### Property

#### Tensile Strength

Bursting Strength Color Fastness to UV Color Fastness to Light

#### Test Method

ASTM D 4632, ISO 5082, ISO 5081, UNI 9405, EN ISO 13934-1 ISO 13938-1 BS EN ISO 105, ASTM G154 CYCLE 2 ISO 105 B02

# 2.10 FUTURE TRENDS OF AGRO-TEXTILES IN PACKAGING PRODUCTS

In the future, agro-textiles are expected to play a significant role in revolutionizing agriculture and addressing some of the challenges faced by the industry. Agro textiles will incorporate smart technologies and functionalities to enhance agricultural practices. These textiles may include sensors to monitor soil moisture, temperature, and nutrient levels, enabling farmers to make data-driven decisions. Functional textiles may also provide controlled-release mechanisms for fertilizers or pesticides, ensuring optimal distribution and minimizing waste [20]. There will be a growing emphasis on sustainable and environmentally friendly materials for agro textiles. Bio-based fibers, such as those derived from agricultural waste or recycled materials, will gain popularity due to their lower carbon footprint [20]. Natural fibers such as cotton, jute, and flax are often sourced from plants grown sustainably. Crop rotation, reduced pesticide and herbicide use, and the possibility of organic farming are all examples. These practices reduce the carbon footprint of agricultural production [20]. Many plants that generate natural fibers are good at removing CO<sub>2</sub> from the environment. Cotton plants, for example, absorb CO<sub>2</sub> during their growth, which helps to reduce greenhouse gas emissions. This carbon sequestration can help to offset some of the emissions from agricultural activities. Compared to synthetic fiber production, processing natural fibers into textiles frequently consumes less energy. Synthetic fibers, such as polyester and nylon, are often manufactured from petroleum-based sources, requiring energy-intensive methods. Natural fibers offer a lower energy footprint, lowering the carbon footprint associated with textile manufacturing [20]. Many natural fibers are biodegradable; they degrade spontaneously without generating hazardous contaminants. Because agro-textiles derived from natural fibers do not contribute to long-term trash in landfills, they have a lower environmental impact. The manufacture of synthetic fibers frequently entails using chemicals such as solvents and dyes. On the other hand, natural fibers can typically be processed with fewer toxic chemicals, lowering the environmental impact [20]. Agro-textiles will find applications in sustainable packaging solutions for agricultural produce. Biodegradable and compostable textiles can replace single-use plastics, providing an eco-friendly alternative for packaging fruits, vegetables, and other agricultural products.

#### 2.10.1 Eco-Friendly Agro-Textile Yarn

Eco-friendly agro-textile yarn for packaging products is an emerging trend in the field of sustainable agriculture and packaging. These yarns are designed to provide environmentally friendly solutions for various aspects of agriculture and packaging, offering benefits such as biodegradability, reduced waste, and resource conservation [21]. Future agro-textile yarns will increasingly be made from biodegradable materials such as organic cotton, hemp, jute, and other natural fibers. These materials will break down naturally, reducing the environmental impact of packaging waste.

As consumers demand greater product transparency and sustainability, agro-textile yarn manufacturers may adopt more sustainable production practices. This includes

reduced water usage, energy-efficient processes, and eco-friendly dyes and chemicals [21]. Agro-textile yarns used for packaging will be supplied from organic and nongenetically modified crops, ensuring low chemical inputs and encouraging biodiversity. This addresses the growing demand for organic and non-GMO (Genetically Modified Organisms) products [21]. Producers will experiment with blending various natural fibers to improve the functionality and robustness of agro-textile yarn. These mixtures could enhance general functionality, strength, and moisture resistance. To improve their adaptability and suitability for a wider range of applications, agrotextile yarns for packaging may include value-added features like UV resistance and antibacterial qualities [21].

### 2.10.2 CIRCULAR ECONOMY ON AGRO-NETS OF PLASTIC

Plastic agro-nets should be designed, produced, used, and recycled to minimize waste, conserve resources, and reduce negative environmental effects. This is known as a circular economy approach to nets. Agro-nets must be manufactured with longevity and durability in mind. This minimizes waste and lessens the need for regular replacements. These nets can be used again by farmers for several growing seasons. When making agro-nets, the use of recycled plastic material results in a decline in the market for virgin plastic, and the recycling sector will also grow [22]. The use of biodegradable or compostable materials for agro-nets that will naturally degrade at the end of their life, hence decreasing the plastic waste problem. Create industry standards for agro-nets to guarantee they are suitable for various agricultural practices and can be reused or recycled easily [22]. Farmers and agricultural communities should be informed of the benefits of a circular economy for agro-nets and how to maintain, repair, and recycle these goods properly. Governments can play an important role in fostering circularity by enacting legislation that encourages using recycled materials, establishes recycling goals, and incentivizes businesses to adopt circular practices. Investigate and develop new materials for agro-nets that are more sustainable and environmentally friendly, such as bioplastics or other novel alternatives [23]. A circular economy approach to plastic agro-nets attempts to reduce waste, conserve resources, and lessen the environmental effects connected with their manufacturing and disposal while still meeting the needs of the agriculture industry.

### 2.11 CONCLUSION

Natural fibers for sustainable agro-textiles exhibit great promise and potential for use as more environmentally friendly alternatives in agro-product packaging. These natural fibers, such as jute, hemp, cotton, and sisal, can offer numerous advantages in sustainability, biodegradability, and reduced environmental impact compared to conventional synthetic materials like plastics. By reducing the dependence on nonbiodegradable plastics, adopting natural fibers in agro-textiles can significantly decrease plastic pollution and its adverse effects on ecosystems. It also lowers the carbon footprint associated with the production and disposal of synthetic materials. Using agro-textiles can enhance crop quality and yield by protecting against pests, weather, and UV radiation, thus promoting sustainable farming practices. Utilizing locally sourced natural fibers can boost the income of small-scale farmers and strengthen rural economies. Additionally, the cost-effectiveness of these materials can potentially reduce packaging expenses for agro-producers. Eco-friendly natural fibers are biodegradable and can be recycled, reducing waste and land pollution while creating opportunities for a circular economy in agriculture. As consumers become more environmentally conscious, eco-friendly packaging can attract a wider customer base and increase market competitiveness for agro products. As a result, lignocellulosic or protein-based agro textiles may prove to be a cost-effective and environmentally benign option and a boon to agriculture.

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# 3 Sustainable Fibers from Pineapple Leaves Extraction, Properties, and Applications

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# 3.1 INTRODUCTION

The growing environmental problems and the diminishing of non-renewable resources have encouraged the use of green materials that are compatible with nature to reduce pollution and environmental impacts. Most of the pollution is caused by chemical processing industries. Certain industrial wastes are hazardous and contribute harmfully to air, soil, and water quality. Today, many research attempts have been reported to convert various industrial and agricultural wastes into value-added products. An increase in the global population causes an increase in the basic needs of people, i.e., food, shelter, and clothing. Cotton is the most exploited natural fiber for apparel production in the textile industry. Due to the increase in the need for cotton and the search for new and alternative natural fibers, researchers exploited other non-conventional natural fibers. In this context, agricultural products and processing residues have been highly exploited for textile applications, such as fiber extraction, dveing, wastewater treatment, etc. (Jose et al., 2019; Jose et al., 2020). For textile applications, many attempts were reported on unconventional natural fibers, such as sugar palm fiber, corn husk fiber, areca nut husk fiber, banana pseudo stem fiber, pineapple leaf fiber, soy hull fiber, etc. However, none could replace cotton; meanwhile, most researchers attempted to blend a certain percentage of these unconventional fibers with cotton. Besides apparel industries, these fibers could find potential applications in various fields, such as composites, paper, and pulp, particle boards, agro/geotextiles, etc. (Gupta et al., 2022; Pandey et al., 2022; Pandey et al., 2020).

One of the most exploited agro-residues for the textile industry is the fibers extracted from residual pineapple leaves. Pineapple is a member of the Bromeliaceae family. Global pineapple cultivation focuses on four major varieties: Smooth Cayenne, Queen, Spanish, and Abacaxi (Pandit et al., 2020). Most pineapple cultivation is concentrated in Asian and Latin American countries. Depending on the variety, a matured pineapple leaf may be 2–3.5 feet in height and 2–3 inches in width (Singha et al., 2020). PALF has a silky, lustrous appearance and is rich in cellulose. It is coarser than cotton but finer than banana pseudostem fiber. Meticulous research attempts have been reported on PALF for developing textiles, paper and pulp, composites, handicrafts, etc. In addition to fiber, pineapple stem is a rich source of the proteolytic enzyme called Bromelain, a key ingredient in many pharmaceutical formulations (Ketnawa et al., 2010). If properly utilized, PALF could provide more income to the pineapple cultivators.

There are a few pros and cons to dealing with PALF. The quantity and inexpensive cost of leaves are unquestionably advantages. The disadvantages include (i) low fiber yield, (ii) storage issues of huge volumes of green leaves, (iii) low volume fiber extraction and processing machinery, (iv) lack of supply study in the market, (v) coarseness of fiber, (vi) huge manpower requirements for extraction, and (vii) high cost. Despite all these disadvantages, researchers are consistently exploring agro-residue fibers for their value addition. This chapter deals with the extraction, properties, and applications of PALF. The prospects and challenges of PALF from the fiber extraction to the end product are also included.

### 3.2 EXTRACTION OF PALF

One of the critical issues for the utilization of leaf fiber starts from its extraction itself. Since the size and shape of each leaf vary from one plant to another, it is difficult to extract the fiber using a single machine. However, custom-made machines were developed to extract many non-conventional fibers (George et al., 2023). The PALF can be extracted manually by retting mechanically, chemically, and biochemically.

#### 3.2.1 MANUEL FIBER EXTRACTION

This method is the simplest method for the extraction of leaf fibers. The fibers are extracted with sharp and flat objects such as shattered crockery. The process is highly laborious, time-consuming, and, thus, not recommended for mass production. On the other hand, the quality of the fibers produced by this approach is outstanding (Rafiqah et al., 2020).

### 3.2.2 MECHANICAL EXTRACTION

Mechanical extraction, often known as decortication, is a rapid method for extracting leaf fiber. Various decorticators have been invented by researchers. In the decortication process, leaves are flattened and beaten by a rotating wheel set with blunt knives. Due to the high beating motion, the biomass attached to the fibers is scraped, leaving the fiber alone. The decorticator is well-suited for large-scale production. However,



**FIGURE 3.1** (a) Fiber decorticator for long fiber extraction, (b) short PALF extraction machine, (c) fibers obtained from long fiber decorticator, (d) fibers obtained from the short fiber extraction machine.

since an intense beating process is involved, the quality of the fiber may be slightly inferior to that obtained by manual extraction or retting.

In large-scale decorticators, whole the leaves are processed. However, in a smallscale decorticator, the operator feeds one side of the leaves by hand, and after scraping, the other side is fed. After decortications, fibers are washed, and a manual brushing is performed to clean the fiber and remove the fiber entanglements. Further, depending upon the quality of the fiber produced, mild water retting may be performed if necessary. The water retting will remove the residual gummy material from PALF and individualize the bunched fibers.

Figure 3.1(a) depicts a long-leaf fiber extractor. This machine is versatile, as it can harvest fibers from pineapple leaves, banana pseudostems, and water hyacinth (George et al., 2023). Figure 3.1(b) is the short fiber (5-6 cm) extraction machine. In the first machine, the whole leaves may be directly inserted manually, whereas in the second machine, before feeding the leaves, they need to be cut in 5–6 cm and



**FIGURE 3.2** Sketch of simple fiber decorticator. (Reprinted with permission from Hulle et al., 2015.)

squeezed to remove the water from the leaves. Figure 3.1(c) and 3.1(d) show the fibers obtained from the respective machines. It is apparent from the figure that the long fiber extraction machine produces long and clean fibers, whereas the short PALF fiber machine produces short and entangled fibers.

Sisal leaves are similar to pineapple leaves in their structure. There are standard decortication machines available for sisal fiber extraction. A schematic representation of the sisal leaf decorticator is shown in Figure 3.2. PALF may be extracted using sisal fiber extraction machines with suitable amendments (Das et al., 2010).

Figure 3.3 shows the extraction machine developed by Alhamdulillah Pineapple Fiber Industries, Bangladesh, as well as the extracted fibers. This decortication machine could process 220 kg of leaves per shift of 8 h, resulting in 2.0 kg fiber. For superior qualities, the PALF fibers need to be washed and dried immediately after extraction. Further, a combing is performed to remove the fleshy components of the leaves present, if any. Combing also removes entanglements and improves the luster of the fiber. The color of the fiber changes from green to lustrous white after washing, drying, and combing (Figure 3.4). Another research attempt has reported on the fabrication of an extractor for chopped PALF (Banik et al., 2011).

### 3.2.3 RETTING

Retting is the most commonly employed extraction process for natural fibers. Dew and water retting are commonly employed; however, the former method is unsuitable for all fibers. In water retting, the fiber sources are completely immersed in running or stagnant water for a prolonged duration. The retting process in running water produces a better quality of fiber. Due to the bacterial action, the fleshy portions of the fibers slowly disintegrate; as a result, the fibers easily come out. Water quality, temperature, and pH are the governing factors during water retting. The water-retting process produces fibers with good uniformity and better quality (Hulle et al., 2015). The duration of retting varies considerably with the sources. The ease of fiber removal should be evaluated regularly to avoid damage caused by over-retting. The major


FIGURE 3.3 (a) PALF decorticator, (b) Extracted PALF (right).



FIGURE 3.4 Decorticated PALF after washing and drying.

advantages of retting are (i) easy process, (2) large quantities can be processed, (iii) good fiber quality, and (iv) suitable for industrial processing. The major drawback is the prolonged duration of the process. For the extraction of PALF, the majority of people adopt decortication. However, retting is also preferred to improve the fiber quality. Figure 3.5 shows the tank retting of PALF.



FIGURE 3.5 Retting of PALF in tank.

# 3.2.4 CHEMICAL EXTRACTION

Chemicals such as strong acids and alkalies can remove the cementing material from plant fibers and thus be employed to extract fibers. Most researchers prefer alkalis to acids for the chemical treatment since the acid treatment may damage the cellulosic fibers. The acid process hydrolyzes hemicellulose and lignin into shorter-chain pentose molecules, while alkali dissolves lignin, hemicelluloses, pectin, and waxes (Jönsson and Martín, 2016). The entire raw material is often subjected to chemical treatment, and on certain occasions, chemical treatment is given to improve the quality of extracted fibers. The first method is employed if the fiber is extremely difficult to remove from the source, either by water retting or mechanical extraction. On the other hand, in many cases, even after mechanical extraction of the fibers from the leaves, the fibers are found to be bunched together due to the presence of certain gummy substances (pectineus matter). This gum may act as an inhibitor for further wet/mechanical processing of the fiber. For the individualization of fiber, a mild treatment using chemicals or enzymes may be given, sometimes called degumming (Pandey et al., 2021). The degumming process removes the non-cellulosic and cementing materials such as hemicellulose, lignin, pectins, and waxes to a certain extent. As a result, the cellulosic content of the fiber is enhanced, and it also provides better strength to the fiber. The nature of the chemical and its concentration, pH, material-to-liquor ratio, treatment temperature, duration, chemical composition of fiber, and gum content are the key factors determining the fiber quality during degumming. It has been observed that alkali degumming often darkens the color of natural fiber. Proper precautions must be taken to avoid fiber damage due to excess chemical treatment.

# 3.2.5 ENZYMATIC FIBER EXTRACTION

Enzymatic extraction is a biochemical process in which the pectins from the surrounding fiber bundles are degraded by suitable enzymes. This process is faster than

natural water retting and produces soft fibers. The process is eco-friendly compared to chemical extraction methods, as it requires a very low concentration of enzymes and auxiliaries. To remove the hemicelluloses and pectins from the leaf fiber, xylanase and pectinase enzymes are commonly used (Hanana et al., 2015; Samant et al., 2020). Pectinases have a primary role in degumming natural fibers by removing interlamellar pectin, which acts as a cementing matter between the fibers. Reports show that combining xylanase and pectinase enzymes effectively removes the gum in the PALF with satisfactory mechanical properties (John and Anandjiwala, 2007). Unlike the chemical method, which indiscriminately attacks the fiber, the enzymes are substrate-specific and attack the target only. The process involves the removal of hemicelluloses and pectins, and the cellulose content remains unaltered. As a result, the enzymatic process produces soft fibers. However, at present, enzyme treatment is more expensive than chemical retting. Table 3.1 shows the chemical composition of important lignocellulosic fibers. Among the listed, it is apparent that the highest cellulosic content is for PALF (70-80%). The hemicellulose and lignin content is also lower than that of other fibers. These properties make PALF a preferred choice for textile scientists.

The authors have conducted a study to estimate the difference in the diameter and tensile strength of PALF extracted from two popular varieties, Mauritius and Giant Q, which originated from India and Bangladesh, respectively. The fibers were extracted through a decorticator, washed, and dried. The fiber diameter and tensile strength from the bottom, middle, and top portions of the PALF were analyzed (Table 3.2).

# TABLE 3.1 Chemical Composition of Leaf Fibers Extracted From Agro Residues (Badanayak et al., 2023)

Fiber	Cellulose (%)	Hemicellulose (%)	Lignin (%)	Pectin (%)	Ash (%)
PALF	70-82	12-20	5-12	2–9	1–4
Banana fiber	60-65	6-19	5-10	3–5	1-1.5
Coconut fiber	32–43	22–25	20-40	3–4	0.2–2.2
Jute fiber	61–70	16–23	11–13	0.2–2.0	0.5–2.0

#### **TABLE 3.2**

#### **Comparative Analysis of Properties of Different PALF Varieties**

Pineapple Variety	Fiber Section	Diameter <sup>a</sup> (µm)	Tensile Strength (g/tex)
Mauritius	Bottom	45.18 (25.7)	41.75
	Middle	43.85 (30.8)	40.23
	Тор	44.06 (29.3)	35.83
Giant Q	Bottom	49.3 (27.1)	50.43
	Middle	44.9 (23.1)	47.91
	Тор	50.87 (25.9)	39.22

Note:

<sup>a</sup> The values in the parenthesis indicate CV%.

This is because the fiber processes non-uniformity from top to bottom. It was found that the diameter at the bottom, middle, and top portions of the PALF obtained from the Mauritius variety was finer than that of Giant Q. However, the tensile strength of the latter was found to be superior. Similar studies have been reported elsewhere (Oliveira Glória et al., 2016).

#### 3.3 APPLICATIONS OF PALF

#### 3.3.1 TEXTILES

Textile experts have yet to discover a natural fiber that is a perfect substitute for cotton, and cotton remains the most exploited fiber in the apparel business. However, many research attempts have been reported to blend cotton with other unconventional natural fibers. Both stem and leaf fibers have been employed for this. The primary concerns related to using agro-residue fibers in conjugation with cotton for apparel-grade fabric are as follows. (1) The agro-residue fibers are generally very coarse and cannot be spun into fine yarn; (2) there is no steady supply in the market; (3) tedious extraction process; (4) often higher cost than cotton fiber, due to poor yield (5) long fiber length, which creates difficulties in short-staple fiber processing machines, (6) brittle and hard, due to high lignin content and (7) non-availability of standard spinning system. Regardless of these limitations, the fibers extracted from plant leaves have been attempted to make textiles in combination with other natural/ synthetic fibers.

Among agro-residue leaf fibers, pineapple leaf fiber is one of the most exploited fibers for textile applications. It is rich in cellulose in comparison with other agro-residue leaf fibers. It is very fine and has good mechanical strength and luster. Plenty of attempts have been reported to prepare yarn and fabric using PALF. In the Philippines, since long ago, the fiber has been extracted manually and used to make very thin apparel. It is known as "Pina" fabric and is a traditional textile material for wedding gowns. The process of making Pina fabric doesn't involve any mechanical processing, either in yarn formation or weaving. Individual fibers are hand-knotted together to form yarns, which are then transformed into fabric in traditional hand-loom using silk yarn in the warp. The Pina fabric is lightweight and transparent. Since considerable manual effort has been involved, the fabric is expensive in the market, ranging from USD 10–15/meter. Figure 3.6 shows the traditional Pina fabric from the Philippines and the union fabric developed using eri silk in the warp direction and mechanically produced PALF in the weft direction (Hazarika et al., 2018).

As previously stated, since no standard spinning system exists for the spinning of pure PALF, several attempts have been made to convert it into yarn through long and short-fiber spinning systems. Due to the inherent coarse diameter ( $30-80 \mu m$ ), PALF cannot be used to develop fine-quality yarn. Attempts have been made to blend PALF with cotton, silk, jute, and ramie fiber.

Jose et al. and his crew have made a considerable effort to transform PALF into textiles. In such a study, they attempted to make fine yarn from PALF. The decorticated and retted PALF were cut into short lengths and carded to form a sliver. This sliver was spun into fine yarn of apparel quality (38 tex). Subsequently, a union fabric was developed using cotton yarn in the warp direction and PALF in the weft direction.



**FIGURE 3.6** (a) Traditional Pina fabric developed using manual method, (b) Union fabric developed from eri silk and PALF through mechanical spinning and weaving.

The developed union fabric was suitable for apparel (Jose et al., 2018). Another study of the same research group reported the extraction of PALF by decortications followed by water retting, degumming, and bleaching. Both the degummed and bleached PALF were converted into yarn (90 tex) in a jute fiber processing system. Further, the fabrics were made out of them. The physico-mechanical properties of the developed fabric showed its suitability for home furnishing textiles (Hazarika et al., 2017). In a similar work, Hazarika et al. (2018) blended eri and mulberry silk waste with degummed and bleached PALF in different proportions to make an apparel-quality yarn. A plain weave fabric was developed using 30-tex cotton as warp yarn and 90-tex PALF–silk-blended yarn as weft in a traditional handloom. The developed fabric was found to have excellent textile properties; thus, it was used to prepare ladies' and gents' garments. Figure 3.7 shows the PALF yarn developed in conjugation with cotton and wool. The same research group published a review article on the production, properties, and applications of the PALF (Jose et al., 2016).

Excellent efforts have been made by a research team led by Jalil et al. to turn the PALF into textiles. In a study, they employed an apron draft ring spinning frame and flyer jute spinning frame to produce 100% PALF yarn. Since the 100% PALF yarn was found to be coarser (120–140 tex), a further attempt was made to produce cotton-PALF blended yarn in a cotton spinning system. They could produce 30 tex fine yarn by blending 20% PALF with cotton (Jalil et al., 2021). Due to the similarities in the properties of PALF with jute, the same group made a blended yarn of PALF with jute fiber in a jute spinning system. A blending of up to 30% PALF was performed, and the resultant yarn was found to have better mechanical properties than bare jute yarn (Jalil et al., 2015). Similar studies on the spinning of PALF in a jute system have been reported elsewhere (Ghosh et al., 1988; Basu and Roy, 2008). Various proportions



**FIGURE 3.7** (a) PALF- cotton blended yarn (30:70), (b) PALF- wool blended yarn (30:70) (right).

of PALF (50, 70, and 30%) were blended with cotton in rotor spinning. The yarn produced from a 50/50 blend has been used for making home furnishing textiles (Surjit et al., 2022). Similar work on the spinnability of PALF in cotton systems has been reported much earlier (Ghosh et al., 1982). In another research, Dey et al. (2005) reported the yarn formation of PALF with ramie fiber. A study reports a competitive study of cow leather with selective PALF blended fabrics. This study concludes that PALF-based textiles are comparable with cow leather in terms of tensile strength, elongation, color, and light fastness properties (Sureshkumar et al., 2012). Debnath (2015) briefed the unconventional textile applications of PALF. Recently, a Philippinesbased industry named "ananas-anam" developed vegan leather from pineapple fiber branded Piñatex® (https://www.ananas-anam.com/).

## 3.3.2 COMPOSITES

Researchers are continuously attempting to fabricate bio/green composites using natural fibers, due to the global community's growing awareness about using sustainable composites for various applications. There is a well-established industry for composites from synthetic fiber reinforcement and matrix. Manmade fibers such as carbon and glass fibers dominate the composite industry, along with synthetic resins such as polyester and epoxy. A composite is referred to as bio-composite if even one of its components comes from biological sources, and green composites are those in which both of their components come from biological sources. Plenty of natural fibers such as flax, bamboo, jute, oil palm fiber, pineapple leaf fiber, wool, coconut fiber, and banana pseudostem fiber have been adopted for the preparation of green composites (Kaewpirom and Worrarat, 2014; Pei et al., 2023; Dong et al., 2014; Jose et al., 2023).

Composites are an emerging and promising area for the utilization of PALF. Composites could be developed from PALF in the form of fiber, yarn, fabric, or non-woven material. However, most researchers favor using it as fiber or non-woven. This excludes the cumbersome tasks of converting the fiber into yarn/fabric. Many research studies have been conducted on the fabrication of composites using PALF and PALF / natural/synthetic fiber blended composites. Whilst both thermoplastic and thermoset polymers were employed for the development of composites, most of the researchers selected epoxy, polyester resin, polyethylene, and polypropylene (Mohamed et al., 2010; George et al., 1998; Devi et al., 1997; Arib et al., 2006).

The benefits of using PALF in the composites include low cost, long fiber length, high strength, and biodegradability. On the other hand, poor compatibility with synthetic resin, the need for a coupling agent, variation in fiber diameter from top to bottom, high flammability, low durability, and high moisture content are the retarding factors (Todkar and Patil, 2019). These drawbacks could be ruled out to a certain extent through surface modification of PALF before composite preparation. Surface modification techniques, such as alkali treatment, peroxide treatment, silination, etc., have been reported to increase the strength of the composites (George et al., 1996; Threepopnatkul et al., 2009; Mishra et al., 2001; Rajeshkumar et al., 2020; Jose et al., 2022a). Another method is to mix PALF with suitable natural/ synthetic fiber. Blending PALF with suitable fibers could significantly increase the mechanical properties of the PALF composites (Panthapulakkal and Sain, 2007; Zin et al., 2019; Jose et al., 2022). Additionally, the use of fillers is also reported to have a considerable effect on the physico-mechanical properties of the PALF composites (Reddy et al., 2020; Pittayavinai et al., 2016). Figure 3.8 shows the composite developed using PALF and natural rubber and its cross-sectional SEM images.

One of the budding areas of natural fiber composites is the automotive industry. Many prime car manufacturers already use carbon and glass fiber composites in their body parts; however, research continues to use natural fiber composites in



**FIGURE 3.8** (a) PALF- natural rubber hybrid composite, (b) SEM images of PALF- rubber composites.

vehicles (Akampumuza et al., 2017; Samant et al., 2022). In this aspect, flax fiber is often exploited due to its high mechanical properties (Samant et al., 2023). The use of natural fiber composites lightens the weight of the vehicle; thus, it has a direct impact on the increase in the mileage of the vehicle. However, due to the lower strength compared to the carbon fiber composites, the use is limited to certain automobile components only. PALF-based composites may find applications in the dashboard, interior door panel, rooftop, seat backs, hat racks, etc. (Friedrich and Almajid, 2013; Dunne et al., 2016).

#### 3.3.3 PAPER AND PULP

A cost-effective method of utilizing agro-residue fibers is in the paper and pulp industries. Undoubtedly, wood pulp is used to make superior-quality paper, but numerous studies have been done on using cellulosic-rich agricultural residues to make various kind of papers. The utilization of agro-residue fibers for paper production not only reduces the cost but also may prevent deforestation. Reports are available on using agro-residue fibers (such as banana, corn stalk, Typha Domingensis, Napier grass, etc.) for paper production (Daud et al., 2014; Pandey et al., 2020). While both handmade and machine-made papers are available in the market, the latter holds the major share. Most of the papers produced from agro-residue fibers are meant for cardboard and paper bags, and their production is preliminarily focused on small and mediumscale industries. Since PALF is rich in cellulose (70-75%), this is one of the best agro-residue fibers that could be used to produce good quality papers. Unlike other lignocellulosic fibers, the high cellulosic content of PALF avoids the excessive lignin removal process from the raw material. Despite these advantages, very little literature is available on producing papers from pineapple leaf fiber. Ter Teo et al. (2020) converted pineapple leaf agro-waste and wastepaper into a new paper using simple processing routes that involve pulping, mixing, sieving, compaction, and drying.

Rattanawongkun et al. (2020) indicated that pulp blending was highly efficient for enhancing the properties of degraded pulp. They also found that the sheets molded from pineapple leaf and rice bran pulp were strong, and their tensile indices were in the range of typical commercial molds. In another study, PALF was blended with cane-bagasse and waste newspaper, and paper was made through the soda pulping method. The physico-mechanical properties of the blended papers were compared with bare PALF paper. The researchers could produce various functionalities by blending cane-bagasse and waste newspaper with PALF in different ratios (Sibaly and Jeetah, 2017). Water and acetone in various proportions were used as pulping agents for PALF fibers (Laftah and Rahaman, 2015). The researchers optimized a 3% acetone concentration for the highest lignin solubility and mechanical properties. The moisture barrier properties and the mechanical properties of the PALF paper were found to be elevated using a poly(lactic acid) coating (Abd Razak et al., 2015). Few research articles on producing paper and pulp are reported elsewhere (Laftah and Rahaman, 2016; Daud et al., 2014; Praveen Kumar, 2020). Figure 3.9 shows the handmade paper and needle-punched nonwoven made from PALF.



FIGURE 3.9 (a) Handmade paper from PALF, (b) needle punch non-woven from PALF.

## 3.3.4 MICROCRYSTALLINE AND NANO CELLULOSE FROM PALF

Recently, considerable interest has been given to extracting Microcrystalline cellulose (MCC) and nano cellulose (NC) from agricultural wastes. They are renewable and sustainable replacements of many materials for diversified applications. MCC and NC are used in the packaging, cosmetics, food, and pharmaceutical industries (Ventura-Cruz and Tecante, 2021). It also acts as a filler in polymer composites (Bharimalla et al., 2019). In addition to the abundance, the researchers prefer to select cellulose-rich agro-residues for the extraction of MCC and NC. Reports are available on the extraction of MCC and NC from various agro-residue fibers (Ventura-Cruz and Tecante, 2021). Since PALF satisfies the above two criteria, it is well accepted in the scientific communities for the aforementioned purposes. Alkali treatment followed by acid hydrolysis is the common method adopted by most researchers for the synthesis of MCC and NC from PALF. The defibrillation methods include enzyme treatment, TEMPO/ultrasonication, and steam explosion. These techniques gradually remove the non-cellulosic materials such as lignin, hemicellulose, pectins, fats, and waxes from PALF and finally depolymerize the high molecular cellulose chains to small ones (Cherian et al., 2010). The resultant MCC and NC will have low molecular weight, small size, and high crystallinity index. The physico-mechanical properties of the MCC and NC greatly depend on the extraction process protocols. Various researchers have reported many research attempts on the synthesis of MCC and NC from PALF (Ravindran et al., 2019; Fareez et al., 2020).

# 3.4 GLOBAL PRODUCTION, BOTTLENECKS, AND FUTURE PROSPECTIVE

The agricultural residues generated from field and processing industries are a matter of concern. Managing huge amounts of biomass from this process sometimes creates a headache for farmers and industries. Many countries outlawed the burning of agro-residues due to the alarming global warming. Thus, an alternative way of disposing of or converting it into valuable products is highly appreciated.

Pineapple is one of the preferred choices of fruit. In the current scenario, Costa Rica is the top producer of Pineapple, followed by Indonesia, Philippines, and Brazil. These counties produce 2.938, 2.886, 2.860, and 2.317 million metric tons of pineapple annually. (https://www.statista.com/statistics/298517/global-pineapple-production-byleading-countries/). In Costa Rica alone, pineapple cultivation generates employment to 23,000 people directly and 92,000 indirectly (Pandit et al., 2020). Depending upon the variety and agro-climatic conditions, approximately 10,000-12,000 plants are cultivated per acre. The plants are uprooted every three years and replaced with new plants. With the increase in the age of the plant, the size of the fruit gradually decreases. Thus, for better production, re-plantation is essential every three years. Now let's see the statistics below. A pineapple plant that has been uprooted could have 40-50 mature leaves. Depending upon the variety of the plant, the weight of the leaves may vary from 50-80 g. The average yield of the fiber is estimated as 1.0-2.0%. According to recent statistics, globally, 1.05 million hectares are under pineapple cultivation (https://www.statista.com/statistics/298540/global-pineapple-area-harvested/). Considering 10,000 plants per acre, 40 matured leaves per plant, a leaf weight of 50 g, and a fiber yield of 1.5%, a hectare of pineapple cultivation could produce a minimum of 200 kg of fiber. This indicates that the global production capacity of PALF, according to the current production statistics, is around 200,000 tonnes. This is a huge amount !!!

Regardless of this huge opportunity, why is a scanty amount of PALF available in the market? Let's see the bottlenecks.

- Storage of green pineapple leaves after cultivation
  - Immediately after the harvesting, the plants used to be uprooted so that, later, the land would be prepared for subsequent planting. The extraction of fibers from the leaves cannot be performed on dry leaves. Therefore, keeping leaves green until the extraction process is crucial. Unlike other plant leaves, pineapple leaves have a waxy coating on both sides, which prevents dehydration. This feature is definitely an advantage since it helps to prevent the dehydration of the leaves to a certain extent. Nevertheless, high relative humidity levels and exposure of leaves to rain speed up the decaying processes. Thus, keeping the leaves away from the above-said factors is essential. A suitable storage room may be selected for this. Pineapple leaves, if properly kept in controlled storage conditions, may remain green even after 20–25 days of harvesting. Another option is cutting the leaves when and where they're needed for decortication.
- · Poor fiber yield

One of the major bottlenecks in the extraction of fibers from agro-residues is the poor fiber yield; PALF is not an exception. Various researchers reported a yield of 1.0–2.0% from the leaves, depending on the plant's variety and the extraction method (Jose et al., 2016). This is much less than a similar type of leaf fiber, sisal, which is commercially cultivated for fiber production. This is certainly a big constraint!

- Large labor requirement
  - A large amount of manpower is required for the various extraction stages, starting with the collection of uprooted plants from the field, transportation to the collection center, separation of leaves from the plant, decortications and washing, drying, and finally, packaging. Most of the above-said processes are still performed manually; thus, the total production is laborintensive. The cost of labor itself heavily influences the price of the PALF.
- Absence of high-volume extraction machinery Pineapple is cultivated for fruits, and PALF is a byproduct. Thus, the major focus is concentrated on fruit only. For high-volume production, decortication is the preferred method. Many small and medium decorticators have been developed worldwide, ranging the PALF production from 1.0 kg to 10 kg per working shift. Most of the decorticators are manually operated and produce long fibers. The majority of the decorticators used to be set in the collection centers, whereas a few mobile decorticators were also developed, which can move inside the field. However, the availability of high-volume decorticators for PALF extraction is scanty, to the best of the author's knowledge. Recently, a China-made decorticator was launched in the market with a processing capacity of 10,000 kg leaves per shift.
- Lack of a standard spinning system to convert into yarn In contrast to other natural fibers like jute, flax, sisal, etc., which are grown primarily for fiber, pineapple is grown exclusively for fruit. Thus, no standard machines are available to convert the PALF into yarn. People used different processing systems to convert the PALF into yarn. Industries are not showing much interest in developing suitable machinery for fiber extraction and processing, maybe due to the following reasons. (1) Exorbitant price of PALF in the market, (2) scarcity of bulk supplies, (3) coarse nature of the fiber, (4) hairiness of the yarn, (5) issues related to the market of PALF textiles
- Management of huge biomass after PALF extraction The extraction of PALF through decortication produces a huge amount of biomass from the fleshy leaves. Considering the yield of the fiber is 1.0%, the rest is a waste. If a decorticator processes 1500 kg of leaves per day, around 1485 kg output is the residual biomass. The PALF producer must find a solution to handle the huge biomass produced during decortication. A possible way is to convert it into manure or compost. Another economical way of managing this enormous waste is to produce bromilin, a therapeutic from biomass (Vilanova Neta et al., 2012).
- Lack of awareness and market The lack of awareness about the PALF is another concern. Most of the pineapple cultivators are either unaware of the value of PALF fiber or not interested in fiber extraction due to high investment and poor fiber yield. In the textile fashion industry, PALF-based fabrics are still scanty. Due to this, very few fashion designers are working on it, and the fabrics that are developed are of premium price. From the customer's point of view, common people do not know about PALF fabric and are not interested. The high price of PALF textiles is another retarding factor.

#### 3.5 CONCLUSION

Regardless of its abundance, pineapple leaf fibers are not exploited to their full economic strength. The extraction of PALF from the leaves offers a valuable fiber to the textile industry and provides an additional income to the farmers. Many research attempts have been made to convert the PALF into value-added products, including textiles, kraft paper, nonwoven composites, and handicrafts. Poor fiber yield and lack of large-scale machinery for fiber extraction are the major obstacles in the extraction of PALF. The lack of standard machinery to convert PALF into textiles is an added issue. Despite all these bottlenecks, PALF is a preferred choice for textile scientists. PALF is now used for composites and luxury textiles, including vegan leather preparation. Thus, it can be concluded that the future of PALF holds promising prospects.

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# 4 Hemp A Traditional Fiber With Modern Applications

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## 4.1 INTRODUCTION

Industrial hemp is one of the most versatile crops cultivated for millennia. The oldest artifact is hemp fabric from 8000 B.C. The hemp plant spread from Central Asia (Mesopotamia) to Europe, Africa, and South America. China has a continuous history of hemp production for over 6000 years. The earliest evidence of hemp products in Europe can be traced back to the Hallstatt culture (800–400 B.C.) (Piotrowski and Carus 2011). Until the sixteenth century, hemp was the primary crop in Europe. In the fifteenth century, hemp cultivation began in South America (Chile) and a century later in North America. Kentucky had a thriving cordage industry in 1775. The uniforms of colonial soldiers were made of hemp. The first U.S. flag and jeans were made from hemp (Fortenbery and Bennet 2004). After thousands of years of usage of hemp seeds for oil, nutrition, and fibers for clothing, shoes, cordages, carpets, and paper, hemp cultivation decreased in the early twentieth century. The importance of hemp fiber declined as the development of motorized ships greatly reduced the demand for sailcloth and ropes.

The intensive development and commercialization of synthetic fibers have significantly reduced the usage of hemp fibers. Competition from cotton and jute also appeared. Besides, hemp cultivation was prohibited worldwide (in many countries) on account of genetic similarity to marijuana (Cannabis indica). However, since the mid-1990s, hemp has experienced a renaissance. By overcoming mistakes made in understanding hemp, since industrial hemp has a low content (0.2–0.3%) of psychoactive ingredient (delta-9-tetrahydrocannabinol, THC), the conditions have been met to lift the prohibitions on hemp cultivation in many European countries and all U.S. states. A huge hemp potential gets meaning with the global trend toward developing a sustainable global economy to improve purchasing power and living standards without wasting resources for future generations. Today, hemp is a sustainable and quality resource for various sectors, including textile, paper, building, automotive, agriculture, pharmaceutics, and energy. This chapter discusses the quality, processing, and sustainability of hemp fiber, with a focus on modern applications.

#### 4.2 HEMP FIBER

Hemp (Cannabis sativa L.), an annual plant, can be either dioecious or monoecious. The hemp plant has a branched stem in which several different morphological regions are distinguished. The matured stem is comprised of several layers: epidermis, cortex, phloem, cambium, xylem, and pith. The epidermis is the outer layer, consisting of color-binging media and stomata. The cortex layer is attached to the phloem layer of bast fiber bundles. The woody stem core comprises the xylem, cambium, and pith layers. This layer occupies most of the stem (about 75%) and consists of short fibers (hurds or shives), which are used for construction (hempcrete, fiberboard) and paper sectors, animal bedding, compost, etc. The bast fibers comprise 20-30% of the stem and are arranged in rings of primary (or technical) and secondary fibers throughout the stem. However, the arrangement of these fibers along the stem is not consistent. The highest concentration of primary fibers is found in the middle part of the stem. The secondary fibers initiated from the cambium can be found in the lower part of the stem, between the primary fiber bundles and the woody core, but typically in a low proportion (Manian et al. 2021). Their high lignification and stiffness result in lower technological value than primary fibers (Cronier et al. 2005; Manaia et al. 2019). Since the height of the stem of a hemp plant grown for fiber (at higher sowing density) varies from 150 cm to 400 cm and even more, the length of hemp technical fibers ranges from 80 to 300 cm depending on whether it is a one- or a dual-purpose variety of plant. The linear density of these fibers is 2.2-3 tex (Kozlowski et al. 2005; Konczewicz et al. 2018). The tensile strength (550-1110 MPa) and specific stiffness (21-50 GPa) of hemp fibers rank them among the stiffest and strongest natural fibers (Manaia et al. 2019).

Hemp fibers consist of elementary fibers that are glued together with pectin substances, forming multi-celled fiber bundles. The elementary hemp fibers are characterized by a multi-layer cylindrical (wall) structure that impacts their physicomechanical properties and their response to different chemical agents. The cell walls consist of microfibrils made from long cellulose chains surrounding the cell lumen. The first or the primary wall is connected to neighboring cells (elementary fibers) through its middle lamella. The thick secondary wall is composed of a few layers of microfibrils deposited at various angles to the cell axis, nearly closing the lumen in mature fibers. These layers are the basic source of cellulose, the most valuable hemp fiber compound for final use. The wall layers are held together by an amorphous matrix that contains hemicellulose, lignin, and pectin. Cellulose provides strength and flexibility, while lignin contributes to the stiffness and strength of the fiber wall. Lignin also impedes degradation (chemical, physical, and microbiological) of the fiber but lowers its absorption capacity. Cellulose and lignin substances are joined together by hemicellulose (Cronier et al. 2005; Manaia et al. 2019; Zimniewska 2022). Other accompanying substances are waxes, ash, proteins, etc. Hemp fiber contains 70–74% cellulose (Ahmed et al. 2022), giving it an advantage over other vegetable fibers, except cotton (which contains about 94%). It also has average levels of hemicellulose (17.9-22.4%), lignin (3.7-5.7%), and 0.9% of pectin (Ahmed et al. 2022). The chemical composition of hemp fiber and fiber dimensions vary depending on the plant variety, growing conditions, and degumming method. Therefore, the main fiber parameters are characterized by value intervals. The diameter of the elementary fibers ranges from 10–50 microns. Their length varies from 5 to 100 mm, but the average fiber length is 35–40 mm (Sponner et al. 2005) or 15–25 mm, according to Kozlowski et al. (2005). The fineness of the elementary fibers ranges from 0.25–0.38 tex, depending on the degree of fiber bundle breakdown during processing (Wiener et al. 2003). The strength of elementary fibers ranges from 27 to 70 cN/tex, with low elongation of 1.5-2% in the dry and 3-4% in the wet state (Sponner et al. 2005; Kozlowski et al. 2005). The cross-section of elementary fibers is mostly polygonal with rounded peaks. The elementary fibers' ends are dull or forked (Wiener et al. 2003). The hygroscopic nature of hemp fiber results in its good moisture regain ranging from 8–13%, which favors fiber processing (Wiener et al. 2003; Manaia et al. 2019). It has been shown that the optimal moisture content of fibers from a further processing viewpoint is between 10 and 12% (Zimniewska 2022). The color of hemp fiber depends on the fiber type and its processing, ranging from yellowishgrey to brown (Kirk et al. 2023).

To extract fibers from harvested hemp stalks, the stem structure must be loosened by weakening interactions among fiber bundles and woody parts. This first step in extracting fibers, which determines the quality of the fibers, can be achieved using various techniques, including biological, chemical, physical, or mechanical methods. The retting method is a biological process that, regardless of which micro-organisms are involved (bacteria, fungi, enzymes), decomposes the natural adhesive (pectin) that binds elementary fibers together and technical fibers (fiber bundles) to woody parts (hurds, shives). There are two retting methods: dew or ground retting and water retting. During dew retting, the hemp stalks are laid on the ground to be exposed to dew and rain. This creates ideal conditions for microorganisms to develop on the stalks, which, along with soil microorganisms, break down the interstitial tissue. This process facilitates the separation of fiber bundles. Dew retting is an affordable process that requires low water and energy consumption. Additionally, dew retting enhances soil fertility due to biodegradable natural residues in the retting process. On the other hand, controlling the dew retting process is challenging since it relies entirely on favorable weather conditions for successful fiber yield and quality. In addition, during the retting of hemp stalks, the field is rendered unavailable for sowing other crops. In water retting, harvested plants are sunk in streams or artificial water tanks, enabling the microbial degradation of non-cellulosic components of hemp stem. Underwater retting of hemp only takes 1-2 weeks, compared to the 5-6 weeks required for dew retting. The influence of the meteorological factors and geographical location is minimized. The retting parameters, such as temperature and pH levels, can be controlled to the optimal level in the artificial water tanks. The water-retted long hemp fibers are characterized by good fineness, mechanical properties, and spinnability. However, due to negative economic and environmental impacts (high cost of drying wet fiber bundles, high consumption of fresh water, and a large amount of wastewater), this method is no longer used in many countries (Kozlowski et al. 2005; Jankauskiene et al. 2017; Manian et al. 2021). In chemical treatments, hemp stalks are exposed to the action of chemicals, and fine fiber yields can be reached if the processing conditions are optimized. It has been shown that pectin can be removed by alkaline boiling treatment, whereas a certain amount of residual lignin remains in hemp fibers. Acid scouring appeared unsatisfactory for pectin and lignin removal (Wang et al. 2003). Although non-cellulosic substances can be effectively removed by chemical processing of hemp fibers, the present trend to protect the natural environment lowers interest in chemical retting methods (Jinqiu and Jianchun 2010). The most used physical methods of fiber degumming (separation from surrounding tissue) are steam explosion and osmotic degumming. In the steam explosion method, hot steam under pressure penetrates between the fibers in the fiber bundle, making the accompanying substances (pectin, lignin, hemicellulose, waxes) water soluble, which further is removed by subsequent washing and rinsing (Nebel 1995). In the osmotic degumming method, hemp stalks are continuously subjected to water flow, which causes swelling of the stalks and an increase in hydrostatic pressure within the stalks. As a result, pectin and other non-cellulosic substances dissolve in the flowing water. The fibers obtained by this method are characterized by light color, thinness, and better thermal stability. Osmotic degumming can deliver highquality long fibers but can also be more effective in removing non-cellulosic substances to deliver elementary fibers (Konczewicz et al. 2018).

The retting or degumming (weakening the bonds between the fiber bundles and accompanying substances) of hemp stems is followed by mechanical processing for fiber separation (decortication). The technical fibers (relatively flexible) should be separated from the brittle and stiff woody parts. This processing involves a breaking operation by which the stems are crushed and split down their length by passing through the pairs of grooved rollers. The wooden parts (shives or hurds) are progressively broken into smaller pieces. In the following process, called scutching, previously broken stems are guided through pairs of rotating blades that beat the stems and remove hurds and short fibers. Scutching produces long fibers, scutched short fibers (tow), hurds (or shives), dust, soil, etc. Hurds are far shorter compared to elementary fibers and significantly more lignified. Their length is 0.5–0.6 mm, and their width is 15–40 microns (Manian et al. 2021). The scutched tow fibers are separated from the hurds and impurities, and then the hurds are pneumatically removed. The removed hurds can be used as natural fertilizer, animal bedding, fuel, or for producing insulation boards.

In the exclusively mechanical fiber extraction method from non-retted hemp stalks, raw stalks undergo much more intensive processing (called decortication) to separate the fibers from hurds. This processing includes a series of breaking, shaking, and scutching operations, which are cyclically repeated until satisfactory fiber separation is achieved. The treated hemp stalks contain more than 60% fibers, which are suitable for applications for composites and paper (Sponner et al. 2005).

After scutching, the decorticated long hemp fibers are exposed to softening by pressing ribbed rollers to increase the fineness of the fiber bundles. Softened scutched fibers are cut from up to 3 m to 60–70 cm on a special cutting machine so that they can be processed on the hackling frames. Long fibers are parallelized during hackling, while short and tangled fibers are combed out (removed). After five consecutive drawing and doubling passages, the slivers transform into roving and then yarn by wet or dry spinning, giving the best quality of hemp yarn compared to other spinning technologies. 70–80 kg of long fiber yarn can be produced from 1 ton of raw hemp stems (Zimniewska 2022). These yarns can be used for technical applications such as industrial fabrics, cordage, and composites.

The short fibers derived from the scutching and hackling processes are known as "tow". The tow still contains tangled fibers, woody particles, and trash. Therefore, the tow processing continues with several cardings by which the remaining hurds and fiber knots are removed while fibers are blended and parallelized. After three passages of drawing and doubling, pre-spinning, and spinning (wet or dry), short fiber yarns of different quality can be obtained for various technical uses (Nebel 1995).

The wet spinning technology of hemp yarns is based on the traditional flax spinning technology. This technology ensures the best quality of long-fiber hemp yarn, but it is labor-intensive, unproductive, and uneconomical. The current situation is outdated equipment, which is unlikely to change for the better considering the niche hemp sector (less than 1% share of hemp and flax in the world fiber production in 2019, Zimniewska 2022). To reduce labor costs, harvest mechanization, cutting automatization, and wet-spinning of long fibers modernization are essential (Vandepitte et al. 2020). Bearing in mind the higher productivity of the cotton spinning technology, a great effort was being made to modify hemp fibers in length and spin-technical characteristics (cottonization) to resemble those of cotton so that the fiber processing could be carried out on the cotton machines. In the cottonization process, pectin and lignin must be removed to gain a more or less complete isolation of elementary fibers. This can be done by various elementarization methods: mechanical, chemical, and enzymatic (Cierpucha et al. 1999; Sedelnik 2004; Kozlowski et al. 2005; Fisher et al. 2006). Cottonized (or woollenized) hemp fibers can be spun into 100 % hemp yarn or blended with cotton or cotton-like fibers (or wool or woollike fibers) on the cotton (or wool) spinning systems. The quality of yarn spun from cottonized hemp fibers (or hemp and cotton mixture) is inferior to that of the long hemp fiber yarn regarding the yarn's evenness and strength. However, the modification of hemp fibers (cottonization) allowed the production of yarns having a linear density in the range of 25–50 tex for the high-quality clothing sector (Czekalski et al. 2000). Hemp spinning has traditionally been based on linen spinning technology, which was considered more suitable for hemp yarn production for certain purposes. However, the cottonization of hemp fibers has opened up new opportunities for expanding the application of hemp fibers for various high-end products.

#### 4.3 HEMP SUSTAINABILITY

Pollution from the clothing and textile industry sectors may not be as visible as that from the mining and oil industries, but it has a significant environmental impact. The negative aspects of the clothing and textile industries are reflected in water consumption, water pollution, microplastic release, carbon emission, etc. A strong argument favoring hemp commercialization is its eco-friendliness. Being renewable and biodegradable, hemp is a crop that has already been acknowledged for its great potential in terms of sustainability. Hemp has been confirmed as an attractive crop well suited to various growing conditions. The extensive root system of the hemp plant makes it very competitive against weeds. According to a report by Bosca and Carus (1998), wheat crop yields were reported to be 10–20% higher after hemp cultivation. When grown for fiber, hemp requires little or no herbicides and minimal pesticide use, which makes it a good rotational crop (Fortenbry and Bennett 2004).

Besides the phytoremediation ability of the hemp plant or the capacity of heavy metals accumulation from the soil with no or small decrease in yield (Cleophas et al. 2023), it has been shown that hemp fits well in the phytoattenuation strategy. This means that hemp, while restoring contaminated land, is safe for fiber production for textiles. This is confirmed by the results of a recent investigation, which promotes the safe textile application of hemp fibers grown on Cd and Pb-contaminated land. After undergoing standard demineralization and bleaching processes in the textile industry, the overall and removable levels of Cadmium (Cd) and Lead (Pb) were lower than the toxicity limits specified by the OEKO-TEX label. This indicates that the fibers are safe to be used for textiles (De Vos et al. 2023a). However, the field retting method should be avoided to prevent the fibers from additional heavy metal uptake (De Vos et al. 2023b).

Hemp is recognized for being a low-maintenance crop compared to cotton, both regarding agrochemicals and water usage. According to Dugue Schumacher et al. (2020), the lowest water requirement for cotton's growing season is 2.5 times higher than that of hemp's lowest water requirement. Wise et al. (2023) collected available data on water consumption (28 published sources), and indicated that hemp requires 38% less water, has a 60% lower water footprint, 84% less crop irrigation requirement, and 91% lower irrigated water footprint than cotton. In addition, Cherrett et al. (2005) stated that hemp is more productive, yielding up to 3 tons of dry fiber per hectare, compared to 1.35 tons of cotton lint per hectare. Dugue Schumacher et al. (2020) showed that the apparel industry can meet its demand with hemp fiber using only 1/3 of the land needed for the same amount of cotton fiber production.

It has been evaluated that the textile industry emits 1 ton of  $CO_2$  for every 19.8 tons released into the environment (Liu et al. 2023). However, plant fiber-based textile products exhibit excellent carbon sequestration ability, implying their capacity to remove  $CO_2$  from the atmosphere. During plant growth,  $CO_2$  is absorbed and stored within plants, remaining trapped in plant-based products throughout their lifespan.  $CO_2$  will be discharged into the atmosphere when the product is decomposed or biomass combusted. Therefore, the longer the lifespan of products, the more effective their carbon storage will be. Liu et al. (2023) investigated the carbon storage and the postponed emission effect of three hemp garments: a T-shirt, slipcover, and handicraft. The results indicated that the emission effects of the stored carbon are 3.83%, for the T-shirt, 19.68% for the slipcover, and 41,12% for the handicraft, at their different lifespans (5, 25, and 50 years, respectively). This carbon storage capacity of the hemp plant (fibers) positively affects climate change.

Overall, the ecological assessment of hemp indicated its excellent ecological credentials in terms of effects on soil, crop rotation, pest management, fertilization, and agro-biodiversity (Piotrowski and Carus 2011). From the economic aspect of sustainability, hemp fiber costs 77.63% less in agricultural activities than cotton fiber. Agricultural activities include field preparation such as fertilization, cultivation and seed, water consumption, and pest control costs (Dugue Schumacher et al. 2020). On the other hand, the current industrial extraction process of hemp fibers is not fully sustainable, and great efforts are being made toward sustainable fiber preparation processes (Nair et al. 2015; Kozlowski and Rozanska 2020). In addition, there is potential for reducing energy use in fiber processing and labor requirements. Considering the next value chain steps, i.e., hemp fiber processing into yarns and fabrics for high-added value products, the maximum technological, economic, and ecological growth has not been fully realized (Stankovic 2023a).

# 4.4 HEMP FIBER FOR VARIOUS APPLICATIONS

Hemp is a highly versatile plant that has immense potential for various applications. The hemp market includes over 25,000 products in various industries, including textiles, paper, agriculture, construction, furniture, food, medicine, personal care, and recycling, with new applications constantly emerging (Crini et al. 2020). All parts of the hemp plant, including the stems, leaves, flowers, and seeds, can be utilized in various applications. Leaves or their biorefinery products are used in medicine, cosmetics, dietary supplements, animal bedding, mulch, etc. Hemp seeds have been used for human consumption, animal feed, cosmetics, personal hygiene, and technical use in the energy sector in nuts, oil, or cake form. All parts of the hemp stalk can be utilized in various sectors, such as textiles (clothing, home, and technical products), paper, construction, energy, innovative composite products, etc. This is further elaborated by schematic representation in Figure 4.1.



FIGURE 4.1 Multidirectional uses of hemp fibers.

#### 4.4.1 HEMP TEXTILES

Hemp is widely recognized as a valuable fiber source for various textile applications. The archaeological and historical records evidenced the ancient significance of hemp fiber as a textile fiber. Hemp fibers have long been known for their high strength-toweight ratio and high modulus due to which they have been widely used for technical applications such as paper, cordage, and sailcloth. Probably the most famous hemp fabric intended for technical applications is a canvas - heavy-duty woven fabric in a plain weave pattern commonly known for its durability and waterproofing properties. Apart from typical technical textile products such as tents, tarps, sails, and more, tightly woven fabrics are also used for other purposes such as painting canvas, rugs, upholstery coverings, footwear parts, clothing accessories like bags, hats, belts, and some garments like work jackets (Crini et al. 2020). The high strength of hemp fibers has led to their wide use for versatile technical textile products. However, hemp fibers are also characterized by excellent hygienic properties (hygroscopic nature, high absorbent), anti-static, anti-allergic, anti-microbial properties, good breathability, ultraviolet radiation blocking properties (depending on lignin content), and thermal and electric properties (Rehman et al. 2021; Ahmed et al. 2022). Because of these properties, hemp fiber has made a breakthrough in the high-quality textile and fashion sectors. Hemp fiber expanded its use for healthy and comfortable clothing, home, and decorative textiles. This affirmation of hemp fibers in the textile field would not have been possible without collaborative efforts to develop new hemp fiber processing technologies (fiber individualization) that allow for the customization of fiber dimensions according to the specific application. By hemp fiber individualization (cottonization), its fineness and length matched those of the cotton fiber, reducing the spinning difficulties of lignified hemp fiber.

Generally, hemp fabrics show good thermophysiological comfort properties, such as air permeability, moisture management, and heat transfer properties, but they also exhibit some shortcomings regarding tactile comfort. In the investigation of the compressibility of plain knitted fabrics made from yarns having the same linear density and twist but different fiber content (hemp, cotton, viscose, and acrylic), the hemp knit exhibited less compressibility compared to cotton and acrylic knits, but similar to that of the viscose knitted fabric (Stankovic 2008a). However, the hemp knit exhibited the least permanent changes (plastic deformation) in its thickness during cyclic compression (Stankovic 2014). Considering that folding (or playing) two or more yarns produces a "new" one with modified and improved properties compared to the single components, an investigation was conducted to compare the comfort properties of plain hemp knitted fabrics produced from single or two-folded hemp yarn. The results indicated a positive effect of hemp yarn folding on air and water vapor permeability, compression, tensile, and shear deformations. In addition, an improvement in the uniformity of these properties of the knitted fabric was shown (Stankovic and Bizjak 2014).

In scientific research, efforts are being made to improve some properties of hemp fabrics, such as softness, flexibility, and crease recovery. Li et al. (2010) proposed the treatment by liquid ammonia (L/A) followed by crosslink finishing of hemp woven fabric by which bending modulus, crease recovery, and water vapor and air permeability

improved. In addition, Hwang and Ji (2012) have shown that L/A treatment can also enhance the wicking speed and drying ratio of the hemp fabric. Ji and Lee (2016) used the Kawabata Evaluation System (KES) to analyze the mechanical and hand properties of hemp fabrics treated with L/A. Ji and Lee (2016) investigated the hand and mechanical properties of L/A-treated hemp fabrics using the Kawabata Evaluation System (KES). After L/A treatment, the total hand value of hemp fabrics significantly increased, indicating an improvement in their flexibility and softness. They also found that L/A treatment improved the moisture management properties: wetting time, absorption rate, spreading speed, maximum wetted radius, and one-way transport capability of hemp fabrics (Lee and Ji 2017).

Spinning in a blend with cotton (or cotton-like) fibers makes it possible to improve the fineness, softness, flexibility, and other properties of hemp-based textiles. In their investigation, Atav et al. (2023) produced single-pique knitted fabrics using hemp, cotton, and a 30% hemp/70% cotton blend, which were compared based on their mechanical and thermal comfort properties. It has been demonstrated that hemp and hemp/cotton knits can achieve the same performance as pure cotton knit regarding water vapor and air permeability, abrasion resistance (except pure hemp knit), and bursting strength. Comparing pure cotton and hemp/cotton blended fabrics, Liu et al. (2011) found that the wicking height and drying rate of the blended fabric were higher than those of the cotton counterpart.

In the systematic investigation conducted by Kim and Kim (2018), hemp/Tencel blended yarns were produced using three different spinning methods: ring, Sirospun, and air vortex system with a 30%/70% blending ratio and linear density of 30 tex. The yarns were ply-twisted and used to produce weft-knitted fabrics tested for hand and tactile comfort. The hairiness and unevenness of the single hemp/Tencel air vortex yarn were reduced compared to other produced yarns. Ply-twisted air vortex yarn was characterized by lower tenacity, breaking strength, and initial modulus compared to the ring and Siro-spun yarns, resulting in higher compressibility and extensibility and lower bending rigidity of the air vortex knitted fabric than those of the ring and Siro-spun counterparts. All these properties of hemp/Tencel air vortex yarn and knitted fabric made the fabric softer and more formable, confirming the feasibility of spinning hemp fibers on the air vortex system.

As a more sustainable alternative to pure cotton yarns in denim fabric production, an effort was made to develop ring, core-spun, and dual-core spun hemp-containing yarns in various blend ratios (20% or 30% of hemp fiber content). The results revealed that hemp fiber usage in blended yarns slightly worsened yarn properties (evenness, tenacity, breaking elongation) except for hairiness. Yet, 20% of hemp-containing yarns were proposed for use in denim fabric production instead of pure cotton yarns (Avci and Demiryurek 2022). The same research group developed hybrid yarns containing about 30 % cottonized hemp fibers and one or a few other components such as organic cotton, viscose, elastane, or polyester using a conventional ring spinning frame or modified ring spinning machine. Their results indicated that using hemp and viscose in the yarn provided optimal yarn properties due to a synergetic effect that compensated for the undesirable properties of both fibers (Okay et al. 2023). It has been shown that hybrid hemp-blended yarns can be produced by folding single hemp yarn and viscose or Tactel (textured polyamide) filaments. These hemp/filament

yarns exhibited higher capillary height and wicking rate than pure hemp yarn. This resulted in higher wicking height of the rib-knitted fabrics made of hemp/filament yarns compared to that of pure hemp knit (Stankovic et al. 2006). In addition, the results indicated higher compressibility of the hemp/Tactel rib knitted fabrics provided by the high bulk structure of the textured filament (Stankovic 2008b).

Textile fabrics of different fibers can be produced by combining yarns made from various fiber types. For example, a fabric can be produced using one type of warp yarn and yarn of a different fiber composition as a weft. Rani et al. (2019) compared the thermal and tactile comfort properties of pure cotton, pure hemp, and union (cotton warp and hemp weft) plain woven fabrics. They indicated higher air permeability, better moisture management, and lower thermal insulation value of the hemp and union fabrics, making them more suitable for summer clothing. The tactile comfort properties of the fabrics were assessed by low-stress mechanical parameters using KES, according to which the parameter - the total hand value (THV) of the hemp and corresponding union fabrics are estimated as comparable or even better than those of the cotton fabric for summer clothing application. Saricam (2022) evaluated the thermal, moisture, and tactile comfort properties of hemp-based denim fabrics (warp was always cotton yarn) compared to pure cotton fabric. The hemp/cotton denim fabrics exhibited better moisture comfort properties (water retention, vertical wicking, drying rate), and warmer feeling than cotton fabric. Regarding tactile comfort, the hemp/ cotton fabrics showed higher compressibility and less shear rigidity but lower extension and enhanced bending rigidity than pure cotton fabric. Measured FAST (Fabric Assurance by Simple Testing) parameters and calculated values of formability indicated that the tactile comfort properties of the hemp/cotton fabrics were within acceptable limits concerning the production of denim garments. In addition, it has been shown in this investigation that common industrial washing treatments can improve the comfort properties. In the investigation conducted by Ahirwar and Behera (2022), the aesthetic and hand properties of plain woven hemp/cotton fabrics were evaluated. The hemp-blended fabric was characterized by a better crease recovery angle compared to its pure cotton counterpart. In addition, the higher pilling propensity, roughness, and surface friction were exhibited by hemp-blended fabric. In the investigation conducted by Petrulyte et al. (2013), hemp or flax yarn was used to form piles in terry woven fabrics (intended for home textiles), with hemp proving to enable higher water vapor absorption of the fabric.

A method was proposed by which hemp-blended knitted fabrics were produced by combining different yarns during knit production when two single yarns were fed through each feeder. Hemp/cotton knitted fabrics were produced by assembling hemp and one of three cotton yarns with the same linear density but different twist intensities. This way, the blending ratio of produced knitted fabrics was 50% hemp/50% cotton. By comparing the comfort properties of these hemp/cotton knitted fabrics with pure hemp knit, a reduction in air permeability and water vapor permeability of the hemp/cotton knits was observed, with the trend of decreasing their thermal resistance. It has also been shown that the basic yarn structural parameter - twist intensity, can be used as a design tool for quality comfort performances (Stankovic et al. 2019). In a more recent investigation, hemp/cotton rib knitted fabrics were produced by combining one of the two-folded cotton (two twist levels) and

hemp yarns. It has been shown that using two-folded hemp yarn enables higher thermal insulation, while single hemp yarn enhances heat transfer through the knit (Stankovic et al. 2022). Investigations also indicated that combining viscose and hemp yarn in the production of plain knitted fabrics contributed to better heat transfer ability in both stationary and dynamic ranges (Stankovic et al. 2008; Pavlovic et al. 2014), as well as improved moisture management properties (Novakovic et al. 2015). By introducing acrylic together with hemp yarn in plain knitted fabric, the compressibility of hemp/acrylic knitted fabric increased by 35% compared to its hemp counterpart (Stankovic 2006). A combination of "warm" acrylic with "cool" hemp yarns in knitted fabric led to the value of the heat transfer coefficient of the hemp/acrylic knit being between the heat transfer coefficients of the pure hemp and pure acrylic counterparts. The acrylic component increased the thermal diffusivity of the hemp/ acrylic knitted fabric, meaning a quicker reached steady state and a decrease in the thermal absorptivity or coolness to touch. Clothing items (undershirts) produced from these knitted fabrics were exposed to the ten-week trial period, which included wash and wear cycles, during which the hemp and hemp/acrylic knitted fabrics underwent greater structural changes compared to acrylic knit. The changes positively influenced the thermal properties of these knits in both stationary and transient conditions (Novakovic et al. 2020a). It has also been shown that adding acrylic yarn together with hemp varn in the knitted fabric can improve the liquid transfer ability of the knit, both before and after undergoing wear and care cycles. Moreover, the ability of the hemp/acrylic knit to transfer liquid is not conditioned by the moisture content of the knit (Novakovic et al. 2020b). Sensory evaluation of these knitted fabrics during the wear trial tests indicated the hemp knit as the most preferable due to the neutral thermal sensation it arouses. Considering thermal comfort, the hemp/ acrylic knit was at an advantage. The hemp knit was perceived preferable regarding thermal comfort and thermal sensation during higher physical efforts (Stankovic 2023b).

The above indicates extensive efforts to expand the range of hemp-based textile products. Besides common textile materials, such as woven and knitted fabrics, the research resulted in the development of nonwoven textiles containing hemp as a component. Yang et al. (2013) developed stitch-bonded nonwoven lining and insole material composed of 50% hemp/40% polyester/10% cotton. Hemp fiber contributed to good moisture absorption and high evaporation rate of the developed material, which resulted in a quickly reaching equilibrium moisture content. In the research conducted by Massaoud et al. (2015), 3D fibrous structures intended for insole products were developed with hemp fabric exhibited the highest thermal conductivity, water absorbency, and the "coolest" touch.

Some of the proposed methods for improving the comfort properties of hemp textiles are promising for increasing the value of hemp textiles by imparting new functionalities, such as the ultraviolet (UV) protection ability. For example, the formation of hybrid hemp/filament yarn can positively affect the UV protection properties of the rib-knitted fabrics made thereof (Stankovic et al. 2017). In addition to an improvement in the thermal and tactile comfort properties of knitted fabrics produced by simply joining hemp yarn with cellulosic one (cotton or viscose) during

knitting, their UV protection ability was also enhanced, classifying them as "very good" and "excellent" UV protection textile (according to EN 13758-2 standard) (Kocic et al. 2019). Research into the functionalization of hemp clothing textiles appears to be at an early stage. A good example is a study conducted by Inprasit et al. (2018) in which the dyeability and antibacterial ability of the hemp fabric were improved by treatment with natural bioactive neem extract. The properties of hemp fibers, such as durability, strength, moisture management, and positive sustainability, make them promising for wearable electronics. Siddika et al. (2023) developed a highly conductive hemp yarn by coating it with reduced graphene oxide and polypyrrole using the conventional deep coating method. This yarn was applied to wearable heaters for wearable thermotherapy and wearable strain sensors to monitor body part movements. These hemp-based electronic textiles exhibited excellent durability and repeatability in different climatic conditions. By taking advantage of the intrinsic flexibility of weft-knitted fabrics, Liu et al. (2021) developed highly permeable pres-

sure sensors from three hemp knits differing in knitting pattern (plain, plain derivative, and rib) via their carbonization into pseudo-graphitic structures using thermal treatment. The best performance was exhibited by graphited plain hemp knitted fabric, which showed good sensitivity, quick response, long cycling life, and excellent air, water, and vapor permeability. The developed sensors are applicable for health monitoring, rehabilitation, athlete training, and sports competitions.

The world hemp clothing market is appraised at \$23.02 B by 2031, whereby the share of America and Europe will be the largest, with 65% of the total consumption (Lawson et al. 2022). The research achievements reviewed here are promising, leading to extended and new utilization of hemp fibers in the high-quality, comfortable, healthy home, clothing, and "smart" textile sectors.

## 4.4.2 NON-TEXTILE USE OF HEMP FIBER

The use of hemp fiber to make paper dates back thousands of years. In ancient China, hemp paper was used as paper scrolls. Hemp paper was used for the Guttenberg Bible (1456) (Crini et al. 2020). In North America, hemp paper was used to print the first draft of the American Declaration of Independence (1776) (Ranalli and Venturi 2004). Decreased hemp cultivation and the simple production of paper from wood reduced the hemp demand for pulp and paper applications. However, agricultural and sustainability policies today recommend the application of non-wood fibers in paper production. Having high strength and length and being divisible into fine fibers with appropriate transparency, hemp fibers are suitable for high-quality paper and specialty paper for cigarettes, banknotes, coffee filters, tea bags, etc. (Yu et al. 2017). Compared to wood, hemp fiber is preferable for paper production as it contains more cellulose. In addition, unlike wooden paper, which can be recycled three times, hemp paper can undergo eight recycling cycles (Temirel et al. 2021). Among hemp stalk pulp, pine kraft pulp, and bleached birch pulp, hemp pulp appeared to be the best for papermaking (Danielewicz and Surma-Slusarska 2017). Jinyong and Jianchun (2015) successfully developed hemp filter paper with better oil/air filtration efficiency than the usual cotton filter paper for automobile engines. The fact that the hemp stalk provides only 20-30% fiber limits the usage of hemp fiber for conventional paper. Kenaf and jute, which can yield more fibers per hectare, have an advantage over hemp for the conventional paper industry (Gonzalez-Garcia et al. 2010). Hemp fibers can be used with other pulp fibers (wheat straw, flax, or recycled wood) for a papermaking mixture (Crini et al. 2020). An interesting medical application was proposed by Temirel et al. (2021). They used hemp paper as the base material for capillary-based microfluidic analytical devices (applications in clinical tests, food safety measurements, point-of-care diagnosis, etc.). Using paper as a substrate for microfluidic devices contributed to their cost-effectiveness, ease of fabrication and use, disposability, etc. This hemp paper application was validated for analyzing potassium levels in artificial urine.

In recent years, the development of natural, low-cost filtration materials has received considerable attention. Hemp-based biosorbents have been proposed as an alternative to commercial synthetic materials for water treatment (metal ion removal). They can be used in a raw, modified, or carbon form. Vukcevic et al. (2023) studied the possibility of reusing hemp fibers wasted in the textile sector for textile industry wastewater purification. Good filtration capacity for methylene blue removal from wastewater was attributed to the fibrillated structure, non-cellulosic components of hemp fiber, and the cavities and cracks on the fiber surface. Akkoz and Coskun (2023) developed a hemp adsorbent with a high capacity for Malachite Green Oxalate dye removal, with stable dye removal efficiency after ten usage cycles. Various investigations indicated that hemp fibers are a successful biosorbent for Cd, Zn, Pb, and Ni removal. In addition, it has been demonstrated the application of hemp-based felt for treating aqueous solutions with a mixture of six metals (Co, Cu, Cd, Mn, Ni, and Zn) (Morin-Crini et al. 2019). The high performance of activated carbon for purification has led to the development of carbon-based adsorbents from waste hemp fibers. After carbonizing and activating waste hemp fibers, a developed adsorbent exhibited high adsorption properties toward 15 different pesticides (Vukcevic et al. 2015). A hempzeolite composite was developed to remove organic pollutants, such as toluene, benzene, and chlorobenzene, from aqueous solutions with an 80% removal capacity (Zou et al. 2012). Jinyong and Jianchun (2013) used a hemp/cotton nonwoven produced by spunlaced technology for developing two different oil filters for automobile engines.

Another promising market for hemp is insulation and building materials. Hemp fibers are usually applied in the building industry as mats or fiberboards for thermoacoustic insulation. Studies have shown that hemp fiber mats provide comparable thermal and acoustic insulation to common materials, such as rock wool and stone wool, and with much greater environmental compatibility than petroleum-based materials such as polyurethane foam or polyester (Ingrao et al. 2015). Stapulioniene et al. (2016) developed thermo-acoustic insulation hemp/polylactide (PLA) composites with low thermal conductivity, high sound absorption ability, low flammability, low water absorption, and good water vapor transport ability. The PLA fibers contributed to compressive strength, inhibited flammability, and reduced water absorption, and did not affect the thermal conductivity and water vapor transmittance of the composites. In the recent research conducted by El Majd et al. (2023), novel thermal insulation materials were developed from hemp, hemp/linen/cotton, and recycled cotton by treating with a binder solution and by adding organic microencapsulated phase change materials (PCM) with thermal energy storage ability. It has been shown that the specific thermal capacity of nonwoven hemp was increased by adding PCM by 55.2% in solid state and 62.4% when PCM was in the liquid state. An increase in thermal conductivity was 9.6% for PCM in the solid and 7.4% in the liquid state. For hemp/linen/cotton nonwoven, an increase in specific thermal capacity was about 40% for PCM in the solid and 37% in the liquid state, with a corresponding increase in thermal conductivity of 8.4% and 7%. In addition to bast fibers, hemp hurds or shivs (the woody core of hemp stalk) are used to produce hemp-based concrete, so-called lime-hemp concrete (LHC), hemp concrete, or hempcrete composed of hurds in aggregate forms and lime-based binder. Hempcrete is lightweight, carbon negative, has low thermal conductivity, and has outstanding hygrothermal and acoustic performance. Being porous with quite highly interconnected voids, hemp shives contribute to an appreciable lowering of the hempcrete density. The high porosity of the hemp shivs leads to their high moisture management ability, which is of great importance for building materials to ensure human comfort inside the buildings. Hemp shives can absorb up to 270% water for a few minutes and 400% water during 48-hour immersion (Ahmed et al. 2022). This resulted in hempcrete being more permeable than other building materials and having a superior moisture buffer capacity of  $2 g/(m^2 \% RH)$ (Latif et al. 2015), due to which hempcrete can successfully regulate the ambient relative humidity. In addition, the buffering feature decreases vapor condensation and limits the development of micro-organisms ensuring indoor comfort. On the other hand, prolonged exposure to rain or very high humidity can cause appreciable deterioration of hemp concrete. Therefore, it is better to use it for indoor wall construction and insulation materials (roof, walls, and underfloor) (Seng et al. 2019). A greatly porous structure of hemp concrete led to reduced thermal conductivity in the 0.06-0.19 W/m K range for hempcrete densities from 200 to 840 kg/m<sup>3</sup>. Therefore, hempcrete can prevent heating walls in hot climates and heat loss in cold climates avoiding additional masonry insulation (Ahmed et al. 2022). In addition, hemp concrete is characterized by a high thermal capacity from 1500 J/kg K in a dray state to 2900 J/kg K at 99% relative humidity compared to conventional cement concrete with a specific thermal capacity from 800 J/kg K to 1200 J/kg K (Jami et al. 2019). Thanks to high heat capacity, water vapor permeability, and moisture transfer ability, hempcrete exhibits phase change material properties such as latent heating effects. By regulating indoor relative humidity, which affects the ambient temperature, the indoor temperature is also managed, avoiding artificial heating and cooling (Shea et al. 2012). Besides these positive aspects, there are some limitations to hempcrete usage. The mechanical performance of hemp concrete is relatively modest at compression and tension. Considering that compressive strength is the material's ability to resist the compressive load without changing its shape or undergoing failure, this property is one of the most significant properties of construction materials. Commercial hempcrete wall systems achieved 0.1–0.2 MPa, representing one-twentieth of the compressive strength of a concrete block. The low compressive strength of hempcrete is attributed to its porous and hydrophilic nature, the imperfect arrangement of shives, and the compaction level (Ahmed et al. 2022). Consequently, hemp concrete cannot be utilized as a load-bearing building material. Still, thanks to its low density and high crack resistance at ground movement, hempcrete is appropriate for application in earthquake-prone areas (Jami et al. 2019). Various research indicated an improvement

in the compression strength of hemp concrete by increasing its density (level of compaction), but this compromises its thermal and acoustic insulation properties. Therefore, a compromise between the mechanical properties and insulation has to be achieved depending on the purpose of hemp-based building products.

Considering that half of the world's energy and raw material consumption is spent on road and building construction (Ingrao et al. 2015), hemp building materials offer a promising opportunity for carbon footprint reduction of constructions. Hemp concrete is perceived as carbon-negative since the net greenhouse gas (GHG) emissions arising from its production and installation have negative values (Pittau et al. 2018). This resulted from a carbon sequestration phenomenon, which refers to the material's capability of carbon-storing within. It has been shown that the biogenic carbon stored in the product can be fully restored in the crop field only a year after harvesting. This absorption completely compensates for the initial GHG emissions arising from production and usage. Even though the lime binder is a non-biogenic component of hemp concrete, it also consumes carbon dioxide by carbonation. It has been reported that 1 m<sup>2</sup> of a hemp concrete wall (thickness of 260 mm) consumes 394 MJ of energy for production while sequestering 14-35 kg of CO<sub>2</sub> over a hundred-year lifespan. On the other hand, the conventional concrete counterpart consumes 560 MJ of energy and releases about 52 kg of CO<sub>2</sub> (Walker and Pavia 2014). Since hemp concrete is considered a universal green building material suitable for various applications, it is currently used in many countries such as France, England, Germany, Switzerland, Ireland, Belgium, Israel, South Africa, Canada, Australia, and New Zealand (Crini et al. 2020).

There is a growing interest in natural fiber-polymer composites in which natural fibers are embedded in a polymer matrix, which can also be from natural sources. As early as 1941, hemp fibers were used in the composite as reinforcement in resin matrix for Henry Ford's car bodywork. The composite withstood ten times more impact than the metal panel but, the car was not commercialized due to the economic constraints of the time (Shahzad 2011). As the demand for sustainable and cost-effective materials has increased in recent decades, hemp fiber composites are gaining popularity as a substitute for synthetic fibers as reinforcements. In addition, hemp is an attractive alternative to conventional technical fibers such as glass, carbon, and aramid fibers, due to the lower density and lower price of hemp fiber. Wambua et al. (2003) compared the mechanical properties of natural fiber- and glass fiber-polypropylene composites, and the hemp fiber-reinforced composite was characterized by the highest tensile strength (52 MPa). Besides, hemp composite exhibited higher specific flexural strength and specific moduli compared to glass fiber composite. In addition, the high aspect ratio (length/diameter) of hemp fibers makes them an excellent choice for composite reinforcement. However, since the mechanical properties of fiber-reinforced composites are strongly dependent on fiber dimensions (diameter, length) and fiber-matrix compatibility, the main drawback of hemp fibers in terms of application in composites is the variability in their diameter and length. These hemp fiber characteristics vary significantly depending on the source (genotype), maturity, retting and separation techniques, geographic origin, growing conditions, etc. (Mussig et al. 2020). Another constraint is related to the hydrophilic nature of hemp fiber, which causes difficulties in adhesion with hydrophobic matrices and consequently affects the effective stress transfer through composite (from matrix to fibers). To improve the fiber-matrix interface, various physical and chemical methods are applied. Common physical methods are alkalization, electric discharge, cold plasma treatment, corona treatment, production of hybrid yarns, etc. In the chemical methods, an improvement in the compatibility of strongly polarized cellulose fibers with hydrophobic polymers (matrix) is being made by fiber impregnation, chemical coupling, and change in surface tension (Shahzad 2011; Chen et al. 2013). In addition, hemp fibers' high moisture absorption ability makes them less attractive as reinforcement in composites for outdoor applications. Cyclic absorption and desorption of moisture weaken the interfacial bonding between fibers and matrix, allowing room for further water penetration. This may lead to composite degradation due to the appearance of cracks or fungal growth (Dhakal et al. 2007). The positive and negative aspects of using hemp fibers as reinforcement for composites are encapsulated in Table 4.1.

The form of reinforcement has a governing effect on the composite properties. Low- and mid-performance composites result from the low efficiency of fiber reinforcement in injection-molded short-fiber composites with randomly oriented fibers or fiber bundles. The low reinforcement efficiency leads to low mechanical properties of composites, which is convenient for non-structural, aesthetic applications (Keller 2003). For structural applications, the composite should be reinforced by long fiber bundles or aligned reinforcement in the form of woven, knitted, braided, and multiaxial fabrics. When producing yarns for aligned reinforcements, their twist negatively influences the composite mechanical properties, owing to helical fiber arrangement along the yarn axis, causing their misalignment. In addition, the compact structure of twisted yarn makes it difficult to impregnate the reinforcement with

#### TABLE 4.1

## Positive and Negative Aspects of Hemp-Reinforced Composites (Shahzad 2011; Song et al. 2012; Vaisanen et al. 2017; Sanjay et al. 2018; Manaia et al. 2019; Ahmed et al. 2022)

#### **Positive Aspects**

- Positive sustainability aspects (renewable nature, eco-<br/>friendly behavior, reduced greenhouse gas emissions)Poor matrix-fitLower density compared to other technical fibersHeterogeneity<br/>diameter, degLower production cost compared to conventional<br/>technical fibersSusceptibility of<br/>oxidative deg
- Excellent mechanical strength, Young's modulus, and aspect ratio
- Good thermal and acoustic properties

Low health risk during the manufacturing processes

#### Negative Aspects

Poor matrix-fiber interfacial adhesion

- Heterogeneity of hemp fibers for their length, diameter, degree of separation, etc.
- Susceptibility of hemp fibers to thermal and oxidative degradation during processing
- Dimensional instability of hemp fibers for their high moisture absorption
- Possibility of colonial fungal growth due to moisture uptake
- UV photodegradation and poor moisture resistance affect the outdoor application of hemp composites

resin. Therefore, the use of aligned carded sliver, roving, or yarn with controlled fiber orientation contributes to the development of aligned composites. However, fiber roving should be slightly twisted to reduce its susceptibility to abrasion and tension during further processing. On the other hand, even with a low twist level, the fiber inclination in the roving reduces the mechanical performance of the composite. If wrapping of roving is applied instead of twisting, a reduction in mechanical properties can be avoided. In the study conducted by Corbin et al. (2020a), a hemp roving was wrapped by thermoplastic polyamide 12 multifilament, thus improving the inter-fiber friction and cohesion of the roving. The roving compression caused an increase in the friction between fibers, improving the tenacity from 8 cN/tex for the single roving to 15 cN/tex for the wrapped roving. This made it possible for the wrapped roving to be used for weaving. Baghaei et al. (2013) developed thermoplastic composites from PLA (polylactic acid)/hemp hybrid yarn by wrapping PLA filament around the core, consisting of low-twisted hemp yarn and PLA filament. Corbin et al. (2020b) succeeded in developing woven hemp fabric/epoxy composites whose tensile properties were competitive to the cross-play flax laminate, the quality product available on the market. The woven hemp reinforcements were produced from the same low-twisted hemp roving in plain, satin 6, and twill 6 weave patterns.

The advantages of using textile fabrics as reinforcement in composites are their high strength, satisfactory fiber distribution and orientation, tailoring ability, and simple manipulation during composite fabrication. Various composite properties can be designed by modifying woven fabric composites' weave types and architecture. In the research conducted by Song et al. (2012), two types of PLA composites, reinforced by twill or plain hemp woven fabrics, were prepared by film stacking technique. Composite reinforced by the twill hemp fabric exhibited better mechanical, viscoelastic, and thermal properties due to the structural characteristics of the twill weave. Both composites manifested their potential for automobile and aerospace applications where materials should experience a wide temperature range. Karaduman (2022) investigated the mechanical properties of epoxy/hemp composites prepared by the compression molding method. The reinforcement was hemp woven fabrics differing in the weave pattern. In addition to plain, basket 2/2, and twill 2/2 woven fabrics, a quasi-unidirectional (UD) woven fabric in a plain weave pattern was produced from much thicker warp yarn (1042 tex) than that of the binder (or weft yarn, 38 tex) yarn. The quasi-UD composite was characterized by superior tensile strength, tensile modulus, flexural strength, and flexural modulus, followed by plain, basket, and twill weave composites. Lagraa et al. (2023) also indicated an advantage of quasi-UD reinforcement over balanced woven fabrics at the fabric and composite level. They produced different woven structures by combining fiber content in the warp and weft of the fabric, which was realized by using different rovings and changing the weft density. Negative aspects of using hemp fibers for composite reinforcement, such as high moisture absorption rate and fiber-matrix incompatibility, can be overcome by hybridization of hemp and synthetic fibers in a composite. Jeyaguru et al. (2023) developed Kevlar and hemp fiber reinforced epoxy composites in different weaving patterns. Hybridization was conducted by inserting hemp and Kevlar

fibers in warp and weft direction when producing plain, 2/2 twill, and 3/3 basket woven fabrics. Three layers of woven fabric were joined with the epoxy matrix by compression molding. The resistance to moisture absorption and change in thickness due to swelling of all hybridized composites was higher than those of the hemp/ epoxy composite counterparts. Compared to Keylar/epoxy composites, hybrid (Kevlar/hemp) epoxy composites were characterized by better erosion resistance properties. The basket fabric reinforced composite exhibited the lowest moisture absorption and thickness swelling and increased erosion resistance among all tested composites. Improvement in the properties of hemp-reinforced composites can also be done by hybridizing them with Lyocell fibers (Baghaei and Skrifvars 2016). The hybrid hemp/Lyocell/PLA yarn was produced by wrapping the core of well-mixed hemp, Lyocell, and PLA staple fibers by PLA filament helically. Hybrid yarns with a core consisting of hemp/PLA and Lyocell/PLA were also produced for comparison. Satin-woven fabrics were produced from PLA filament as warp and one of the hybrid yarns as weft and subsequently used as a prepreg for producing composites by compression molding. The results indicated enhanced impact strength, tensile and flexural strength, and modulus by combining Lyocell with hemp fibers in the hybrid yarn, but without changes in water absorption in the composites.

According to available data, only 15% of the world's production of hemp fibers is used for composites (Lagraa et al. 2023). Currently, hemp nonwovens (fleece and needle felt) and plain woven fabrics are widely available. The assortment of hemp fabrics for structural applications should be widened, including aligned hemp fabrics with low (ca. 50 g/m<sup>2</sup>) and high (above 800 g/m<sup>2</sup>) areal densities (Mussig et al. 2020). Although significant efforts have already been made, further research is needed to optimize hemp's application in composite production. Research into the hybridization approach to improving some properties of hemp fibers is vital for the final product (composite) performance. In addition, the application of 3D knitting, braiding, and other specific processes would enable obtaining more oriented hemp reinforcements. Considering that composite matrix is also responsible for the composite properties, it is essential to work on the choice of matrices. Conventional matrix materials are classified into thermosets and thermoplastics. Natural fiber-reinforced thermosets account for the majority of natural fiber composites. These composites are tough and creep resistant. However, thermoplastics have the advantage of being easy to recycle and design. In addition, the production costs are lower. The processing temperature of thermoplastic polymer is a limiting factor for natural fiber-reinforced composites, due to the possible thermal degradation of natural fibers. Therefore, only thermoplastics with processing temperatures below 230°C, such as polyethylene and polypropylene can be applied for natural fiber-reinforced composites (Shahzad 2011). Primarily considering the recyclability of the thermoplastic polymer matrices at the end of the life cycle, a gradual replacement for thermoset polymers with thermoplastic ones is preferred. Since petrochemical matrices are non-biodegradable, making the natural fiber-reinforced composites only partly biodegradable, it is essential to work on the development of bio-based composites composed of biodegradable natural fibers and bio-derived (from a biogenic carbon feedstock) matrices such as polylactic acid (PLA) and polyglycolic acid (PGA).

#### 4.4.3 INNOVATIVE APPLICATION OF HEMP FIBER

Science and technology development extended the usage of hemp fiber for modern textiles and other products. In addition, recent achievements have suggested new application fields of hemp fibers. Wang et al. (2013) created unique partially graphitic nanosheets from hemp fibers via hydrothermal carbonization and chemical activation. The prepared hemp-derived nanosheets were characterized by high specific surface area, significant mesoporosity, and good electric conductivity. The supercapacitor electrode made from prepared nanosheets exhibited excellent electrochemical performance (remarkable capacitance and extreme current density) for diverse applications, including energy storage. Sun et al. (2016) developed activated carbons from hemp fibers and hurds using identical processing methods. The constructed supercapacitor electrodes from these carbons were characterized by the excellent electrochemical performance of a specific capacity and high energy density. In the investigation conducted by Toprakci and Karahan Toprakci (2021), the plain woven fabric was used to develop a range of binder-free and flexible anode materials for Lithium-Ion batteries by the two-step carbonization method. The results indicated the optimal carbonization temperature regarding carbon yield and cell performance (cycling stability, reversible capacity). Wang et al. (2022) added value to waste hemp fabric by its refabrication into carbon fabric with zinc oxide nanoparticles. The composite anode was synthesized by lithium-plating conductive scaffolds exhibiting high cycling stability (in carbonate electrolytes and ether-based electrolytes). When used in a cell, improved electrochemical performance was achieved without compromising its function and safety. These achievements demonstrated the potential application of hemp fibers in the next generation of lithium batteries.

Nowadays, three-dimensional (3D) printing or additive manufacturing has been implemented in different industries and products. As a result, there is an increasing interest in developing 3D printable polymer composites. Koushki et al. (2020) developed a hemp fiber/silicone composite and investigated its 3D printability. They found that the incorporation of 15 wt% hemp fiber enhances the mechanical properties of the composite. However, it has been shown that after mixing hemp fibers with silicon matrix, the composite becomes unprintable due to its high viscosity. To address the challenge, different solvent concentrations were added to the mixture and the results revealed that the composite with 15 wt% hemp fibers and 20 wt% solvent exhibited the 3D-printable ability. In addition, the tests demonstrated the mechanical properties of these 3D-printed products being better than those of the molded counterparts.

Some of the latest research offers innovative hemp fiber uses for wound dressings. Hydrogels are widely applied for wound dressing thanks to their high fluid absorption and retention capacity. Bio-based hydrogels are preferable for wound dressings because of their biocompatibility and nontoxicity. Ahmad et al. (2023) developed the nonwoven hemp-reinforced alginate hydrogel composites for wound dressings using the sol-gel method – hemp nonwoven was immersed in alginate solution and then, in the solution of calcium chloride, to cross-link the sodium alginate. The anti-inflammatory ability of hemp fiber is combined with the exceptional alginate hydrogel's wound-healing capacity. In addition, nonwovens are believed to be ideal reinforcement for hydrogel-based composites because of their porous structure and

Hemp

good mechanical properties (flexibility and strength). The developed hemp/alginate composites showed improved moisture management, fluid absorption, tensile strength, and wound healing, indicating their high potential use for dressing exudate-releasing wounds.

Hemp has numerous practical uses, but the process of transforming it into various products results in a significant amount of waste from harvesting, processing, conversion, and other hemp-derived coproducts. However, all these byproducts are usable and can be transformed into value-added products. For example, hemp hurds, as a low-value byproduct, are mainly used for hemp concrete production and animal bedding. Agate et al. (2020) proposed a unique low-energy and chemical-free process for nanocellulose production from hemp hurd waste. This chapter has already reviewed various strategies for upcycling waste hemp fibers, yarns, and fabrics into added-value functional materials, such as thermal and acoustic isolation products, biosorbents, composites, etc. In addition, one more potential use of industrial hemp is bio-energy production. The agronomic, experimental, and economic examinations confirmed the outstanding potential of hemp as a bioenergy crop (Das et al. 2017). A range of energetic products can be produced from hemp biomass, such as biodiesel (from seeds), bioethanol (from fermented stalks), briquettes or pallets, baled biomass, etc. When hemp fiber products reach the end of their functional life, or when hemp waste can no longer be reused, they can be used as a solid fuel for thermal power generation, closing the circular economy loop.

## 4.5 CONCLUSION

The growing trend toward sustainable development has led to the increased use of eco-friendly bast fibers, such as hemp. Hemp offers significant sustainable advantages, such as easy adoption to various climatic conditions, short cropping periods, low soil quality and nutrient demands, low pesticide and herbicide requirements, moderate water usage, and high biomass yield. The outstanding hemp fiber properties, such as high strength and modulus, low weight, excellent hygienic and protective (anti-electrostatic and ultraviolet radiation blocking) properties, and good thermal and acoustic insulation abilities, made them among the most versatile fibers used for various applications. The range of hemp products is quite diversified, from traditional products, such as clothing, cordage, paper, and insulations, to novel applications for filters, geotextiles, and various composite materials. Science and technology continue to extend the use of hemp fibers in the exclusive clothing sector and composite and energetic sectors. Recent achievements resulted in new applications of hemp fibers, such as battery components and composites for 3D printing and wound dressings. For the future expansion of hemp fiber's application fields and to increase its additional value, technological advancements must continue to improve harvesting and processing technology, thereby enhancing fiber performance and reducing production costs. In addition, upcycling hemp-waste textiles into high-value-added products, competitive on the market, can be an outstanding contribution to sustainability and the circular economy. The hemp market has a bright future. However, efforts are still needed to develop new products and improve product performances and economic and sustainability issues for further commercial success.
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# 5 Application of Plant Fiber and Biomass in Tissue Paper

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#### 5.1 INTRODUCTION

Tissue paper, commonly known as tissue, is a lightweight and versatile product with many applications. It is made from various raw materials, including virgin pulp and recycled fibers. In daily life, tissue paper serves diverse purposes, each tailored to specific needs. It is used as facial tissues, napkins, baby wipes, and bathroom tissue for personal hygiene, providing gentle and convenient solutions. Products like paper towels utilize tissue paper for its absorbency in cleaning tasks. Additionally, tissue paper is used for aesthetic and protective purposes, such as wrapping gift items, due to its delicate and decorative qualities. This ubiquitous paper product, often resembling light crêpe paper, is integral to everyday cleaning, dusting, wrapping, and personal hygiene [1]. The tissue paper market value in 2022 was USD 80.99 billion, and it is expected to reach approximately \$133.75 billion by 2030, with a 6.55% CAGR. Its impressive absorbency is achieved by the arrangement of small hydrophilic cellulosic fibers (tentatively 1-3 mm long and 10 µm wide) with a highly porous engineered structure with a porosity value of 0.9, offering a substantial surface area per unit volume (typically >  $0.6 \text{ m}^2/\text{cm}^3$ ). Tissue paper products must withstand various stresses during manufacturing and consumer usage. Different approaches, viz., additives supplement, replacement of hydrogen bonds, or creation of a reinforced network of fibers, are practiced to overcome the lower wet strength of such paper, which prevents the swelling and fiber-to-fiber separation [2]. In a broader context, paper products are categorized into three primary segments: packaging, printing and writing, and household/sanitary utilities. Tissue products, specifically, are classified based on a triadic system, considering factors such as fiber characteristics, chemistry, production methods, and technological innovations. These categories include Economyand-Value (EV) products, which provide essential affordability and performance; Premium (PR) products, which offer higher quality and additional features; and Ultra (UL) products, which represent the highest tier with advanced features. Ultra (UL) products are characterized by superior performance and higher costs, produced from a high proportion of virgin and high-quality fibers. These products are typically manufactured using advanced technologies like through-air-drying (TAD) in 2 to 3 plies and specialized chemical additives such as softeners, debonders, and wetting agents are added[3, 4]. The properties of tissue products are shaped more by consumer preferences than manufacturing requirements [5]. Each tissue product is meticulously engineered to cater to a specific consumer sector. For instance, kitchen towels are primarily intended for cleaning and absorption. The consumer expects these towels to exhibit strength, particularly when in wet condition, ensuring effective cleaning without disintegration. Another vital property of kitchen towels is absorbency, with consumers anticipating efficient water absorption and retention when drying surfaces. So, a kitchen towels' first and foremost essential and/or functional properties encompass wet strength and absorbency, as reported in the literature [4, 6–8].

The secondary attributes, like a soft feel, bright color, and suitable appearance, while not central to the core requirement of cleaning and drying, can also influence consumer choices. It is worth noting that the difference between the main and secondary characteristics hinges on the product functionality and may vary across consumer segments and geographic regions. Likewise, softness has gained importance in marketing kitchen towels, appealing to consumers who prioritize a hand-touch feel [4, 6, 8]. It is anticipated that the properties of tissue products in the American market may vary depending on a particular application [4]. Bath tissue emphasizes softness and strength, while facial tissue prioritizes absorbency. Napkins target strength and absorbency, with the bulk being a significant towel consideration. Novotny [5] further underscores that bath and facial tissues value softness, absorbency, and brightness, whereas towel and napkin products emphasize absorbency and strength.

Additionally, certain tissue papers are applied in baby diapers and manufacturing sanitary pads [9, 10]. Tissue products can be categorized into two primary markets: Away-From-Home (AFH) products, fulfilling the requirement in the professional and institutional sector, and At-Home (AH) products, fulfilling the requirement in the domestic consumer market. The Away-From-Home goods are specially formulated mainly for offices, restaurants, and hotels, with institutions being their primary customers. These products play a secondary role in end-user requirements, as consumers prioritize factors like cuisine when dining out. AH products are manufactured for household purposes and are typically available through retailers. Consumer preferences regarding performance attributes, along with the cost of the product, take precedence; for example, consumers may prefer premium-grade bathroom tissue owing to its softness and durability, even if the price is higher.

#### 5.2 TISSUE PAPER

Paper products are classified into three primary functional classifications: (i) packaging, encompassing materials used for packaging purposes; (ii) writing and printing, encompassing graphic papers; (iii) liquid absorbing and wiping, including paper for households and sanitation. The last one, often called tissue paper or hygienic papers, encompasses various end-uses, viz., napkins, toilet tissue, facial tissue, kitchen towels, hand towels, and wipes. Such paper, commonly known as tissue, is a lightweight and versatile paper with a wide range of technical applications. It serves various purposes in our daily lives, offering solutions for personal hygiene with products like facial tissues, napkins, and bathroom tissue and cleaning and absorption tasks with paper towels. Tissue paper is notably prevalent in commercial settings, such as offices, corporate sectors, catering establishments, packaging, and healthcare facilities, where consumers exhibit a discerning preference for hygiene products equipped with additional attributes like antimicrobial properties to combat bacterial and viral infections [2]. Tissue paper also has decorative and protective properties, which improves the presentation of gifts and items. It is made from various raw materials, such as virgin and recycled paper pulp. Tissue and towel products are a cost-effective and convenient solution for cleaning up liquid spills, with the ability to absorb five to ten times their weight without leaking the absorbed liquid. Small hydrophilic cellulosic fibers are arranged in a low-density, highly porous structure to produce an impressive absorbency with a significantly high surface area per unit volume. High capillary pressure for liquid intake and exceptional permeability for quick liquid distribution are necessary for efficient fluid absorption. The main function of tissue paper is to absorb liquids and particles from different surfaces, both soft and hard. Additionally, bulk in the paper industry refers to the volume filled by a particular material weight; this is an important parameter in the production of tissue products because bulk softness, absorbency, and paper thickness are strongly correlated with it. Preventing undesired fiber network compaction is necessary to achieve a voluminous tissue product. This can be done by minimizing paper web wet pressing and utilizing advanced manufacturing techniques, such as the through-the-air (TAD) process [11–13].

Tissue products are expected to withstand various tensions during manufacturing and consumer use, necessitating specific strength properties. Paper strength is broadly influenced by fiber characteristics, the nature of the fiber, molecular bonding, and the use of strength/manufacturing chemical additives. While dry creping enhances strength, it can reduce absorbency and softness. Factors like long fibers, high hemicellulose content, and fibrillated fiber orientation affect tensile strength and improved properties. Tensile failure in the tissue products results from inter-fiber bonding and fiber failure, with poor formation compromising tissue strength. Tissue exhibits viscoelastic behavior, and energy absorption is crucial. Due to their low density and thickness, achieving a balance property encompassing the desirable strength, elongation, and absorbency is essential for tissue products. Paper strength largely relies on molecular bonding (hydrogen bonding) among cellulosic microfibers. It is vulnerable to moisture, which poses a challenge for their application in the kitchen and hand towels, requiring some moisture resistance. To achieve requisite paper wet strength, additives are supplemented, or hydrogen bonding is replaced, creating a reinforced network of fibers to prevent swelling and fiber-to-fiber separation. Paper finish content is also critical, as a higher amount could deteriorate wet strength performance. A paper sample retaining 15% or more of its dry strength is considered a wet-strength grade paper, with efficient additives capable of preserving up to 50% of dry strength. Such additives can also enhance dry strength and increase wet strength [12, 14, 15].

Softness is a vital sensory attribute of tissue products, particularly in sanitary applications. It encompasses tactile, auditory, and visual perceptions and is categorized into surface softness (related to the paper's feel) and bulk softness (related to how easily it crumples). Surface softness depends on fiber type, smoothness, and finishing processes, while bulk softness relates to paper stiffness, bulk, and fiber movement within the web. Both human feel and analytical methods are used to assess softness, with high-bulk paper products often exhibiting superior bulk softness due to a lower degree of fiber bonding. The creping procedure enhances bulk softness, where factors like the number of plies, calendaring, fiber coarseness, and the use of specific fiber types influence softness. In contrast, certain pulp types lead to producing softer tissue. Development in innovative tissue products featuring enhanced germ and virus protection attributes significantly contributes to the market's overall expansion. The quality of such papers is expected to be better if they are produced from virgin pulp. Natural fibers like jute, banana, hemp, and pineapple are attributed to their longer length and flexible structure. Such a fiber bundle is extracted from the plant body, cleaned by retting/similar process, and found suitable for developing quality papers. Not only the fiber part but also the agricultural residues have been explored as an alternative source of softwood fibers in high-performance hygiene tissue applications [16–19].

#### 5.3 DEVELOPMENT OF TISSUE PAPER

The history of the tissue paper industry reveals a fascinating evolution in the use and production of tissue-related products. Paper had been utilized for wrapping and padding in ancient China as far back as the 2nd century BC. The first use of toilet paper is documented, dating back to the 6th century AD in early medieval China. Later on, the inception of toilet paper was associated with an emperor in 1391, who ordered large paper sheets to be divided into small pieces and placed in his outhouse. A significant milestone in the development of modern toilet tissue paper was reported in 1907 when a Philadelphia teacher introduced the concept of individual paper towels as a response to a mild cold epidemic in her classroom. Arthur Scott of Scott Paper Company recognized the potential of application in this area, perforating thick sheets of paper into small towel-sized pieces, subsequently sold as disposable paper towels. Later on, the product, renamed 'Sani-Towel,' found its way into hotels, restaurants, and washrooms. Later, Scott introduced the first kitchen paper towel in 1931, creating a new grocery category. Despite its early innovation, paper towels took some time to gain widespread acceptability and replace cloth towels in kitchens. The tissue industry has continued to expand, with diverse tissue products, viz., moist toilet paper, introduced in various regions. Europe plays a substantial role in tissue production, contributing approximately 6 million tonnes annually, representing approximately 23% of the global market. The European tissue market's value stands at tentatively 10 billion Euros annually, with a growth rate of a tentative 3%. Although Europe has a significant tissue market, North Americans consume a much higher amount of tissue paper than their European counterparts. Tissue paper industries are well-represented by the European Tissue Symposium (ETS), a trade association founded in 1971, which encompasses the maximum number of tissue-products manufacturers in Europe. Moreover, the tissue paper manufacturer, as well as the broader paper products manufacturing sector, has made concerted efforts to reduce environmental impact. In this direction, recycled fibers presently constitute roughly 46.5% of the raw material requirement of the industry. Additionally, the industry significantly depends on biofuels as a primary energy source, accounting for about 50% of its energy requirement. The European-Disposables-and-Nonwovens-Association (EDANA) has monitored the industries' environmental performance since 2005. Despite its extensive production, the tissue paper industry has a relatively modest environmental impact, showcasing a commendable commitment to minimizing the ecological footprint [1, 20–22].

# 5.4 PROPERTIES, CLASSIFICATION, AND APPLICATIONS OF TISSUE PAPER

### 5.4.1 **Types**

- Facial tissue: It finds its roots in Japan, where washi or Japanese tissue was used centuries ago and evolved into the modern form known today as Kleenex, introduced by Kimberly-Clark in 1924. It was originally designed to remove cold cream, and Kimberly-Clark expanded its product line to include various innovations like pop-up, colored, pocket-sized, printed, and 3-layer facial tissues [23]. Such tissues, introduced in the 1920s, are soft and absorbent disposable papers designed for various purposes, primarily expelling nasal mucus. Over time, its usage has evolved with functions, viz., treating minor wounds and cleaning spectacles.
- **Toilet paper**: It has a historical trajectory dating back to the late 19th century, with more than 20 billion rolls consumed annually in Western Europe today [21]. Joseph Gayetty is recognized as the inventor of modern toilet tissue paper in the United States. These developments collectively underscore the evolution of tissue paper products and their diverse applications in our daily lives.
- **Paper towels**: It constitutes the 2nd most significant area of consumption of tissue paper. This tissue typically exhibits a basis weight of 20–24 g/m<sup>2</sup> and are commonly two-ply. Such a kind of towel is manufactured from chemical pulp, fully recycled fiber, or a suitable blend of these two. It often incorporates long chemical pulp fiber for strength enhancement. The development of paper towels can be recorded back at the beginning of the 20th century, when pioneers like Henry Chase, William E. Corbin, and Harold Titus started exploring such products [24]. In the subsequent time, the product was perfected and is known as "Nibroc-Paper-Towels." Scott Paper Company introduced kitchen paper towels in 1931. It is the 2nd largest end-use segment of tissue paper in the customer market, typically produced from chemical pulp, recycled fiber, or a suitable blend.
- Wrapping tissue: The wrapping tissue, on the other hand, serves as a thin, lustrous paper primarily considered for gift packaging and safeguarding delicate products. In table settings, terms such as "table napkin" or "face towel" are utilized interchangeably, with distinctions arising in certain regions. In some locales, "serviette" refers to paper napkins, while "napkin" refers to cloth ones. Custom-printed, as per the need of the manufacturer/end-user, this paper is becoming popular among boutique retail businesses, often produced sustainably with FSC-certified, acid-free composition and inks



FIGURE 5.1 Picture of different tissue papers.

of soy-based formulation. Specialty tissues are considered for various purposes in the packing industry, including wrapping items, cushioning fragile objects, and maintaining garment shape. These tissues are often printed with brand name or logos of the producer for aesthetic appeal.

- **Hygienic tissue**: Hygienic tissue is used in personal hygiene care, including facial tissues, bathroom tissue, napkins, and household towels. It gained prominence in the mid-20th century in the United States and later in Western Europe during the early 1960s.
- **Table napkins**: Tissue paper is also used for table napkins, and is available in various folds, sizes, imparted texture designs, and color shades. Raw materials can vary from deinked to chemical pulp, depending upon the quality requirements.

### 5.4.2 PROPERTIES [25–28]

- **Softness**: Tissue paper is known for its softness and gentleness in terms of touch, feel, and texture, which should be comfortable against the skin. This characteristic is more desirable in the case of facial tissues, napkins, and bathroom tissue used in personal hygiene.
- Absorbency: Tissue paper, a distinct category of paper products, is attributed to its good liquid absorbency, particularly in the case of paper towels. It could quickly soak up liquids, making it most effective for cleaning up spills and ensuring drying hands.
- **Strength**: Despite its light weight in terms of areal density and thinness, tissue paper should have adequate strength to satisfy end-user requirements. For instance, toilet tissue must be strong enough to handle everyday use without tearing easily.
- **Flexibility and pliability**: Tissue paper is expected to be flexible and easily folded or crumpled, which is required for various applications, like wrapping gift items and creating decorative crafts. Tissue paper is pliable to conform to different shapes. This property is valuable for wrapping items and providing an added cushioning effect to the packed item.

- **Thickness**: Tissue paper comes in a wide range of thicknesses, e.g., very thin and delicate to thicker and more robust type. Thickness can be selected as per the end application requirement, e.g., thicker tissue paper fulfills the durability requirement.
- **Texture**: Tissue paper can have different surface textures, including smooth, embossed, or patterned finishes, depending upon its application. For example, some tissue paper is embossed for decorative purposes.
- **Color and design**: Tissue paper is available in many colors and designs, making it suitable for decorative purposes and gift wrapping. Its aesthetic appeal enhances its much-added value.
- **Biodegradability**: Many tissue papers are biodegradable and environmentally friendly, as they are produced from natural resources and quickly break down in the environment.
- **Hypoallergenic**: Tissue paper used for facial and other personal hygiene purposes is often hypoallergenic, ensuring the minimum likelihood of causing allergies or skin irritation
- **Ease of dispensing**: Tissue paper is designed and packed in such a process that it should be convenient and hygienic for dispensing, whether in the form of a roll in case of toilet tissue or as an individual sheet in case of facial tissues

#### 5.5 NATURAL FIBER AND BIOMASS-BASED TISSUE PAPER

Different wood or non-wood sources like straw, bamboo, and fibers from hemp, banana pseudostem, and jute fiber can be used in various grades of paper making. End-user preferences play a significant role in determining the product characteristics, especially for kitchen towels, where emphasis is given to the wet strength and absorbency. In the American market, preference varies with the product types, e.g., bath tissue valuing softness and strength, facial tissue focusing on absorbency, napkin tissue maintaining a healthy balance, and towel tissue prioritizing absorbency and strength. Softness, absorbency, and brightness are vital in bath and facial tissue, while towels and napkins prioritize absorbency and strength. Tissue papers have been prepared from the different natural fibers and/or biomasses, viz., jute, hemp, banana, and pineapple, as mentioned in Table 5.1.

An extensive examination delves into the diverse applications of industrial hemp, emphasizing its eco-friendliness across industrial applications. A study underscores hemp root bast paper's efficacy in filtration, demonstrating superior efficiency, compact dimensions, and reduced weight compared to conventional cotton paper [36]. Another study introduces an innovative hemp-derived tissue paper, showcasing favorable properties like burst resistance, water absorbency, mechanical strength, and softness [29]. Sustainable solution development for disposable paper cups has been reported using Mauritian hemp, pineapple, and orange peels, featuring optimal composition, water-tight beeswax coating, and complete biodegradability. It also explores cationic nanocellulose as a material in the paper industry, achieving a 57% improvement in tensile strength [37]. Nanocellulose from hemp pulp for food packaging applications exhibits notable performance, such as density, tensile strength, transparency,

Raw Material Used	Paper GSM and Properties	Possible Applications	References
Hemp fibers from hurds	<ul> <li>i) 30 GSM</li> <li>ii) Cellulose 42%; lignin 27.4%; hemicelluloses and pectin 27%</li> <li>iii) Fiber length (mm) 0.527 ± 0.005 (Autohydrolysis) 0.714 ± 0.006 (Carbonate) 0.688 ± 0.004 (Kraft pulp process)</li> </ul>	Tissue paper & Towel paper	Naithani et al. [29]
Portuguese industrial hemp plant (whole stem)	<ul> <li>i) 60 GSM</li> <li>ii) Better bleachability and beatability</li> <li>iii) Higher tearing resistance</li> </ul>	Laboratory sheets	Baptista et al. [30]
Jute fiber	<ul><li>i) 60 GSM</li><li>ii) ASAM digestion process leads to high paper strength.</li></ul>	Hand sheets, Packaging materials, Laminated paper bags	Roy and Chattopadhyay [31]
Jute fibers	<ul> <li>i) 9–11 GSM</li> <li>ii) Tensile index 8.44 Nm/gm</li> <li>iii) Tear index 20.61 mN.m<sup>2</sup>/gm</li> <li>iv) Burst index 2 Kpa.m<sup>2</sup>/gm</li> <li>v) Brightness 63.2</li> </ul>	Tissue paper	Chauhan et al. [32]
Banana fiber and pseudostem	<ul> <li>i) 60 GMS</li> <li>ii) Lignin 12.1%</li> <li>iii) Ash 9.5%</li> <li>iv) Tensile index- 29.4 Nm/g</li> <li>v) Bursting index- 2 kPa.m<sup>2</sup>/g</li> </ul>	Wrapping paper	Ramdhonee and Jeetah [33]
Banana pseudostem	<ul> <li>i) 40 ± 1 GSM</li> <li>ii) Maximum tensile index of 65 ± 3.5 Nm/g</li> <li>iii) Burst strength 3.76 ± 0.2 kg/cm<sup>2</sup></li> <li>iv) Contact angle 41°</li> <li>v) Air resistance 29.95 ± 0.4 s/100 ml</li> </ul>	Grease proof paper for packaging of butter	Sakare et al. [34]
Pineapple leaves	<ul> <li>i) 60 GSM</li> <li>ii) Compatibility with bagasse</li> <li>iii) Most absorbent paper</li> <li>iv) Tensile index 6.1 Nm/g</li> <li>v) Burst Index 0.72 kPa m<sup>2</sup></li> </ul>	Laboratory hands sheets	Sibaly and Jeetah [35]

# TABLE 5.1 Application of Different Plant Fibers in Tissue and Other Papers

and enhancing breaking force and length beyond premium paper requirements. These studies underscore hemp's versatility, economic potential, and environmental benefits for its diverse applications. Like hemp, the evolution and intrinsic potential of jute as a credible alternative within the paper and pulp sector has also been elucidated for a long time. Originating in the 1970s, the pursuit of non-wood fibers identified like jute, kenaf, and mesta, as potential alternative resources for pulp and paper making.

The viability of jute/kenaf fiber in pulp and paper making has also been reported. The National Institute of Research on Jute and Allied Fiber Technology (currently known as NINFET ), Kolkata, is dedicated to studying the potential of different natural fiber/ biomass in pulp and paper making for their applications in carry bags, tissue paper, office files & folders, wrapping paper and writing papers [31]. Specific studies on jute's utility reveal its feasibility as a raw material for similar applications in environmentally sustainable domains [38]. A comprehensive work on the banana pseudostem emphasized the development of environmentally friendly wrapping paper. The chemical pulping process involving banana fiber, sugarcane bagasse, and wastepaper could develop papers with diverse properties, as indicated in Table 5.1 Utilization of banana fibers for handmade paper production shows pulp yield, tensile, burst, and tear indices meeting the requirement of handmade paper production. It highlights the environmental impact of non-biodegradable polythene by developing banana stem-based oil-proof paper [39]. In the line of work, grease-proof packaging paper was also developed from banana pseudostem fiber [34]. Exploring the integration of degraded hemicellulose from the ramie fiber degumming liquor in the beating process of jute stalk pulp proves advantageous while reducing the energy requirement in the beating operation and improving the strength of paper [40]. A study on flax shives underscores its potential as a fiber resource for paper making through in-situ chemical modification, improving moisture resistance and sheet formation [41]. Research on pineapple leaf fiber in paper production highlights its potential and versatility when blended with bagasse, improved through bio-pulping and xylanase-enzymatic bleaching [35]. A focused study on cellulose nanofibrils (CNF) from cotton fibers analyzes its influence on specialty paper drainage and strength. The study provides insights into CNF characteristics and their relationship with specialty paper performance, emphasizing the need to balance paper strength and drainage considerations for optimal results [42]. In a similar direction, cotton kapok fiber also has potential in pulping and papermaking, as observed from the favorable effects on tensile and bursting strengths, as developed for packaging applications with improved strength and water repellency [43]. The study on the okra plant, consisting of fiber and stick components for paper making following sodaanthraquinone and Kraft pulping methods, reveals that the Kraft process is superior in pulp yield, delignification, and paper properties [44].

#### 5.6 CONCLUSIONS

Tissue paper is a lightweight, thin sheet renowned for its gentle touch, feathery lightness, and impressive absorbency. It is produced from various raw materials, including virgin pulp, recycled pulp, or a mixture of the two. Tissue paper has a wide range of applications in daily life, with each product tailored to serve specific purposes. For personal hygiene, tissue paper is used in the form of facial tissues, napkins, baby wipes, and bathroom tissue, providing a gentle feel and convenient solutions. Tissue paper, with an areal density below 40 gm/m<sup>2</sup>, is typically available in single to threeply sheets, depending on the intended application. Its versatility extends from cleaning and dusting to decorating packages and personal care needs. Tissue products find their place in two distinct commercial markets: the professional and institutional sector, known as "Away-From-Home" (AFH), and the consumer sector, known as "At-Home" (AH). The properties of tissue paper are influenced by factors such as the type of fiber (virgin or recycled), production process, and raw material (wood or non-wood). Paper strength is mainly determined by fiber properties, molecular bonding, and chemical additives. Hardwood, softwood, and plant fibers can produce lightweight tissue paper. Recently, there have been efforts to prepare tissue paper from jute, hemp, banana, cotton cellulose, okra, and pineapple fibers. This chapter summarizes tissue paper's properties, classification, and applications, highlighting the potential of plant fibers and biomass in this sector.

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# 6 Natural Fiber-Based Composites and Their Applications

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#### 6.1 INTRODUCTION

In recent decades, a growing awareness of the finite nature of non-renewable resources has spurred research into renewable alternatives, such as natural fiber-reinforced polymer composites, as a possible substitute for synthetic fibers. These natural fibers are abundant, cost-effective, sustainable, and biodegradable. Many products, at the end of their useful life, often end up in landfills, posing environmental hazards to both humans and the ecosystem. Advances in modern technology and escalating consumer demands are driving an ever-increasing demand for resources and a heightened focus on environmental sustainability [1]. Natural plant-based fibers like flax, sisal, hemp, and kenaf have attained recognition for their outstanding mechanical strength and are increasingly used as reinforcements in automotive components. When these natural fibers are incorporated in optimal quantities within polymer composites, the resulting materials exhibit superior mechanical properties compared to neat polymers, making them particularly suitable for specific applications [2]. Their wide-ranging use in automotive, marine, and construction industries has piqued the interest of researchers in hybridizing various natural fiber-reinforced polymer composites to enhance overall performance while addressing distinct mechanical requirements under varying environmental conditions [3, 4]. In light of pressing environmental and economic concerns, scientists and researchers have been compelled to explore alternative materials in recent years. As a result, bio-composites are becoming increasingly popular as alternatives to conventional polymer composites made of synthetic fibers like carbon and glass [5, 6]. Natural composites entail the amalgamation of plant-based natural fibers, including hemp, jute, flax, sisal, and kenaf, with either biodegradable or non-biodegradable matrices. These plant fibers bring forth many appealing characteristics, encompassing cost-effectiveness, renewability, abundant availability, low energy requirements derived from fossil fuels, reasonably robust mechanical properties, and cost advantages compared to synthetic fibers [7].

Approximately five decades ago, the extensive exploration into natural fiberreinforced polymer composites commenced. Natural fibers such as sisal, coir, banana, pineapple leaf fiber, hemp and jute have already found applications in composite materials. Particularly, fibers originating from plant stems have better mechanical properties and are more valuable because they contain more cellulose. In recent years, researchers have been actively identifying new varieties of stem fibers and developing composites based on these materials. Furthermore, certain researchers have ventured into employing natural particulates as reinforcements in combination with lignocellulosic fibers. The synergistic combination of these approaches presents vast opportunities for research and development in creating novel natural fibers for polymer composites. This chapter reviews various natural fibers and how they may be used as reinforcement material in a polymer composite, enhancing their mechanical properties [8].

Table 6.1 presents a comprehensive listing of commonly utilized natural fibers. These fibers are divided into four categories: fruit, seed, leaf, and bast fibers, corresponding to the various plant sections from which they are extracted. For example, coir fibers, which are obtained from the fruit of the *Cocus nucifera* plant, are located within the tough inner husk and the outer shell of coconuts. Bast fibers, on the other hand, are obtained from the outer cellular layers of plant stems and include examples like flax, jute, ramie, and sun-hemp fibers. Leaf fibers, such as those from pineapple and banana plants, are sourced from leaf cells. Figure 6.1 shows some natural fiberbearing plants, whereas, Table 6.2 provides insightful information on the mechanical and behavioral properties of different natural cellulose fibers. The predominant bast natural fibers typically comprise a woody core bounded by stem tissue [9]. There are many fiber bundles in the stem, and each one is made up of a single fiber cell or filament. These filaments consist of cellulose and hemicelluloses joined by a matrix that

Types of Natural Fibers	Common Name	Family Name		
Bast fiber	Jute fiber	Corchorus-capsularis		
	Hemp fiber	Cannabis-sativa		
	Kenaf fiber	Hibiscus-cannabinus		
	Ramie fiber	Boehmeria-nivea		
	Flax fiber	Linum-usitatissimum		
	Abelmoschus Manihot fiber	Malvaceae		
	Roselle fiber	Hibiscus-sabdariffa		
	Sun hemp fiber	Crotalaria juncea		
Leaf fiber	Agave angustifolia fiber	Asparagaceae		
	Pineapple fiber	Ananas-comosus		
	Henequen	Agave - fourcroydes		
	Abaca fiber	Musa textilis		
	Pandanus fiber	Pandanus		
	Sisal fiber	Agave sisalana		
Seed fiber	Cotton fiber	Gossypiumarboreum		
	Kapok fiber	Ceibapentandra		
Fruit fibers	Coir fiber	Cocos nucifera		
	Borassus fiber	Borassus flabellifer		

# TABLE 6.1 Different Natural Fibers and Their Scientific Names [14–17]



FIGURE 6.1 Natural fiber-bearing plants.

### TABLE 6.2 Physical and Mechanical Properties of Natural and High-Performance Fibers [14–17]

Fiber Name	Density (g/cm³)	Diameter (µm)	Tensile Strength (MPa)	Tensile Modulus (GPa)	Elongation (%)	
Cotton	1.60	11–22	290-600	5.5-13	7-8.5	
Kenaf	1.45	50-200	600–930	50-100	1.60-1.65	
Sisal	1.34	46-250	545-700	9–25	2-5	
Flax	1.50	25	600-1500	28-84	2.8-3.2	
Coir	1.20	150-257	176-300	4–7	32-35	
Bamboo	1.20	85-125	140-225	12-17	1.42-1.50	
Banana	0.9	100-245	160-250	8-12	2.35-2.40	
Hemp	1.49	25-570	550-800	70–75	2-6	
Jute	1.48	30-250	380-750	12-30	1.15-1.60	
Abaca	1.50	10-30	400-830	31-34	2.80-3.00	
Ramie	1.50	20–94	400–940	60–130	3.5-3.8	

<b>c</b> 'l	Cellulose		Lignin	Pectin	Wax	Moisture	Ash
Fiber		Hemicellulose	Content	Content	Content	Content	Content
Name	( /0)	Content (76)	( /0)	( /0)	( /0)	(70)	(/0)
Cotton	82–91	1.5-5.4	0.5-25	1–5	0.5	7-8.5	0.5 - 2
Kenaf	30-55	18-24	8-22	3-8.8	0.7	6.5-10	2-5.2
Sisal	47–77	10-22	8-15	0.8 - 1.0	0.5 - 1.5	11-12	0.5-4.5
Flax	63-72	15-20	2-2.5	1.5-2.5	1.3-1.8	8-13	1-3.1
Coir	20-35	12-16	34–53	4.5-8	10-11	11	_
Bamboo	25-45	13–18	11-33	0.3-4.0	2–3	9.5	1.4-6
Banana	45-60	10-15	11-33	2.2-4.4	2–5	10-11	3.2-3.5
Hemp	54-79	13–23	2.5-14	0.9-3.3	0.3-0.8	6-1.0	0.4-0.8
Jute	45-70	12-22	12-26	0.5-11.7	0.6	12-14	0.5–5
Abaca	55-60	15-20	7-13	0.2 - 1.0	0.2	1–2	1.1–3
Ramie	67–77	5-15	0.5–0.8	1.9–2.1	0.5	8–9	5.5–6

# TABLE 6.3Chemical Composition of Different Natural Fibers [14–17]

may contain lignin or pectin. Pectin plays a role in encapsulating and securing the bundle to the stem. In the process of composite fabrication, the resin is introduced into these fiber bundles, and it's worth noting that lignin is the least resilient among individual cells. In various composite applications, particularly within the German automotive sector, hemp and flax fibers have become notable substitutes for glass in many components. These extracted fibers are subsequently assembled into single or monofilament tests to assess their tensile behavioral properties [3, 4].

Table 6.3 provides detailed information on the chemical composition of different natural fibers. These fibers exhibit high tensile properties due to their significant cellulose content. Notably, flax, hemp, ramie, and nettle fibers outperform other natural fibers in tensile properties [10]. Cellulose presents a well-structured framework of fibrillar elements within the fibers. Lignin, found within the fiber, fills the interstitial spaces in the cell wall between cellulose, hemicellulose, and pectin components. This is particularly evident in structures like tracheids, sclereids, and xylem. The formation of crosslinks between various plant polysaccharides, which give the cell wall and the plant a whole mechanical strength, largely depends on lignin [11]. Additionally, lignin contributes to the transport of water within plant stems. In contrast, hemicellulose is an extensive group of substances incorporated into the matrix of plant cell walls, where cellulose fibers are embedded. Pectin is found in the intermediate lamella, which connects plant cells and promotes their adherence, in addition to the primary cell walls [12, 13].

### 6.2 WHAT ARE COMPOSITES?

Composites are materials composed of two or more distinct components having dissimilar physical or chemical properties that contribute to noticeable differences in the final structure. It is possible for the composite to display properties that are superior to those of its constituent parts. In daily life, composites of all kinds, both natural and man-made, are everywhere. Natural composites, such as wood and human bones, demonstrate how skilled nature is at creating materials that may be used for various purposes. The idea of composite materials has been used by humans for many purposes throughout history, including the construction of building blocks out of straw and clay, the reinforcement of concrete with steel, and the fortification of polymers with different kinds of fiber.

Since fiber-reinforced plastics or composites outperform the metals they frequently replace in terms of mechanical qualities, they have found widespread use in structural applications. Advanced composites were first created for aircraft construction in the 1950s, but their uses have since grown to encompass circuit boards, building materials, automobile parts, and niche sporting products. The most widely used composite materials in the market are composed of non-biodegradable polymeric resins like epoxies and unsaturated polyesters combined with high-tensile-strength fibers like graphite, glass, and aramids. These composites are intended for most applications requiring high strength, stiffness, and long-term durability. Notably, a sizable amount of the fibers and resins utilized in these composites are derived from petroleum resources [18].

Ecologically friendly composites are now a top priority because of the increase in mass volume applications such as aircraft and civil constructions, which have prompted a focus on green production and life-cycle evaluations. Because composites are made by joining two different materials to form a desired shape, their low recycling or reuse potential is now a problem. Currently, more than 90% of composites wind up in landfills, which is quite concerning for the environment. The decomposition of composites in these dumps might take years or even decades, making the ground unsuitable for other uses. In order to overcome these disposal issues and provide more environmentally friendly processes for the production and life cycle of composites, work is now being done [18].

Concerns about pollution and the rising issue of plastic and composite trash have made consumers, governments, and manufacturers more conscious of environmental issues. The rate at which petroleum is being consumed today is unsustainable. It surpasses the rate at which nature produces its resources by a factor of 100,000. Governmental organizations have passed legislation promoting the worldwide use of recycled bio-based green goods in response to these environmental issues. Today, there is a growing demand for new environmentally friendly products and processes due to the rapid depletion of petroleum resources, changing environmental regulations, the emphasis on sustainability, and an increase in global environmental consciousness [19].

The advanced green materials development is guided by growing ideas such as eco-efficiency, green chemistry, 'cradle-to-cradle' design, sustainability, and industrial ecology. Additionally, the trajectory of composite materials is being impacted by this paradigm change. Prominent international producers are coming around to producing recyclable or environmentally friendly goods. Soon, it is fair to expect the broad integration of sustainable, totally biodegradable, and environmentally friendly green plastics and composites into different elements of our everyday lives, given the continuous research endeavors [19].

# 6.3 MATRICES UTILIZED IN THE FABRICATION OF NATURAL FIBER COMPOSITES

A matrix firmly holds the reinforcing phase of composite materials together. The fiber reinforcement phase bears the principal load-bearing stresses in a composite, which gives the material strength and stiffness. On the other hand, the matrix plays a significant role in providing texture, durability, and environmental resistance. The stress transition from the matrix to the fibers directly affects the mechanical properties of composites. Effective stress transmission in composites requires strong interfacial interaction between the polymer matrix and the reinforcing components and appropriate fiber distribution. With dwindling fossil fuel reserves and concerns over the environmental impact of conventional petrochemical-based matrices, substantial research efforts have been directed toward finding viable alternatives. Partially biodegradable composites result from combining synthetic polymers with natural fibers. In contrast, composites that utilize biodegradable matrices are termed "green composites" [20].

### 6.3.1 PETROLEUM-BASED MATRIX

Petroleum-based resins are chemical compounds derived from natural resources or fossil fuels, such as coal, crude oil, tar, and natural gas. Typically, in fabricating Natural Fiber-Reinforced Polymer Matrix Composites (NFRPMCs), two primary categories of petroleum matrices are used: thermosets and thermoplastics. Thermoplastic polymer matrices make use of materials like polystyrene (PS), polyvinyl chloride (PVC), polyethylene (PE), and polypropylene (PP). In contrast, thermoset polymer matrices encompass materials such as polyester, epoxy, vinyl ester, and phenolic (phenol-formaldehyde) [21].

### 6.3.1.1 Thermosets

Thermoset matrices are materials characterized by their insolubility and inability to be melted or re-molded once they undergo curing, which is facilitated by a catalyst or thermal energy. This irreversible transformation is due to the formation of threedimensional covalent bonds that firmly bind the polymer chains. Thermoset resins excel in terms of thermal stability, tensile modulus, chemical resistance, and resistance to creep. Still, they tend to exhibit low fracture toughness and can be brittle at room temperature. Several advantages are associated with thermoset polymers, including their cost-effectiveness and ease of use. They also offer good thermal and mechanical properties, low curing shrinkage, and resistance to moisture. However, a notable drawback is the limited working time available when dealing with thermosets [22].

#### 6.3.1.2 Thermoplastics

The thermoplastic matrix is made up of polymers that can be reshaped, remolded, or melted upon heating. When in a molten state, thermoplastic resins exhibit significantly higher viscosity, approximately 500 to 1000 times more than uncured thermoset resins. These polymers have the unique property of being re-formable and

reshaped without the need for additional heat or chemical reactions, and they solidify at room temperature. They offer advantages such as the ability to withstand high impact, process at elevated temperatures, and exhibit increased resistance to damage compared to thermoset polymers or resins. Thermoplastics are appreciated for their lightweight nature, high fatigue resistance, capacity to withstand high temperatures, resistance to chemicals, and cost-effectiveness. However, a notable drawback is their high flammability [23, 24].

## 6.3.2 POLYESTER RESIN

Polyester resins, specifically of the 'unsaturated' type, are widely utilized in resin systems, notably within marine industries like dinghies, work boats, and yachts. An unsaturated polyester resin belongs to the category of thermosetting materials, implying its capability to solidify from a liquid state under specific curing conditions. This characteristic makes it a common choice for applications requiring durable and resilient materials in marine construction. It is important to note that unsaturated resins differ from saturated resins like Terylene, which do not undergo this curing process. In the composite industry, unsaturated polyester resins are often called polyesters. Polyesters are formed through the reaction between an organic acid and alcohol, producing an ester and water. When glycol undergoes a reaction with di-basic acids, it results in the formation of polyester and water. In composite applications, two primary types of polyester resin serve as standard laminating systems. Orthophthalic polyester resin, known for its cost-effectiveness, is a frequently employed option. On the other hand, isophthalic polyester resin has gained popularity in the marine industry due to its exceptional water resistance properties. Polyester resins are very economical. They have good, lifelong performance stability [25].

## 6.3.3 VINYL ESTER RESINS

When comparing vinyl ester resin to polyester resins, a striking similarity in their molecular structures becomes apparent. Both of these resins exhibit a reduced number of ester groups, and these ester groups are susceptible to degradation when exposed to water and various chemicals. This characteristic makes vinyl esters more resistant to water and to many other chemicals compared to their polyester counterparts. As a result of their enhanced resistance to degradation from water and various chemicals, vinyl esters are frequently favored for applications related to chemical storage tanks and pipelines. In practical applications, these resins are commonly utilized as a 'skin' coat or protective barrier for a polyester laminate that will come into contact with water, as seen in the case of boat hulls. The molecular structure cured by vinyl ester resin results in a harder material than polyester resin [26].

### 6.3.4 EPOXY RESIN

Epoxies tend to outshine most other types of organic compounds due to their resistance to environmental degradation and impressive mechanical properties. This makes them highly suitable for various marine applications. These resins are often chosen as coating materials due to their exceptional resistance to water degradation and adhesive properties, making them well-suited for boat manufacturing. Epoxy resins are a common choice in constructing high-quality boats or as a substitute for water-degraded polyester resins. These resins, along with compatible hardening agents, are available in liquid form, resulting in low-viscosity systems that are easy to process. They can be rapidly and conveniently cured over a temperature range of 5 to 150°C, offering flexibility in choosing the appropriate hardening agent. Additionally, epoxy finds applications in sealing products, paints, varnishes, and coating products, among others [27].

#### 6.3.5 BIOBASED RESINS

These matrices consist of polymers sourced from sustainable materials, which can be either entirely or partially biodegradable. These bio-based resins can be directly derived from plants, such as starch and cellulose, or synthesized by polymerizing plant-based sugars and oils. They come in three main categories: entirely biodegradable (e.g., starch and PHA), partially biodegradable (e.g., cellulose and Polylactic Acid), and non-biodegradable (e.g., bio-polyethylene, bio-polypropylene, and biopolyethylene terephthalate). Approximately a decade ago, petroleum-based polymers were manufactured at a volume of around 236 million tonnes, while bio-based polymers accounted for roughly 3.5 million tonnes. Projections indicate that, by the year 2025, annual production of bio-based polymers will reach 12 million tonnes. To mitigate the adverse environmental impact of petroleum-based polymers, there is a pressing need to increase the production of biodegradable polymers, as their current output is significantly lower. In fully biodegradable matrices, regenerative carbon is initially drawn from the atmosphere and returned to the environment when the polymer degrades. Bio-based polymers offer several advantages compared to petroleumbased polymers, such as being entirely renewable, requiring less energy during manufacturing, being safe (non-toxic), and having a minimal ecological footprint. However, their production costs are typically 10-12% higher than resins obtained from petroleum sources. The advantages of bio-based resins include their biodegradability, non-toxic nature, and cost-effectiveness, while their disadvantages include lower impact strength, lower decomposition temperature, and brittleness [28, 29].

### 6.4 NATURAL FIBER COMPOSITES

Natural fiber-based composites offer biodegradability, cost-effectiveness, nontoxicity, and non-abrasive characteristics, making them a compelling alternative to synthetic fibers for acoustic absorption applications. Organic fibers with excellent physical properties have proven to be high-quality composites with economic and environmental advantages. Examples of such fibers include coir, corn, paddy, sisal, and banana, as well as synthetic fibers like fiberglass, glass wool, and mineral wool, which can serve as probable materials as acoustic absorbers [3, 19]. Natural fiber-reinforced polymer composites have garnered significant attention due to their affordability, abundance, biodegradability, and environmentally friendly attributes. Moreover, these materials are often more cost-effective and environmentally sustainable than pure glass fiber-reinforced composites [20]. Prior to the fabrication of composites, chemical modifications are made to natural fibers to improve their properties and overcome specific limitations. Mercerization or alkaline treatment, for instance, can reduce fiber diameter and improve the overall properties of the fibers and composites [30].

# 6.4.1 FACTORS INFLUENCING THE PHYSICAL PERFORMANCE OF NATURAL FIBER COMPOSITES

The selection of fibers is a critical factor in producing high-quality composites. This selection involves various criteria, including matrix choice, fiber content, fiber dispersion, interfacial strength, fiber orientation, fiber type, extraction method, harvest time, aspect ratio, treatment, composite manufacturing process, and porosity, among others.

#### • Fiber Selection

Fibers can be categorized based on their origin into three main types: plant, mineral, or animal fibers. Plant-based fibers primarily comprise cellulose as their main structural component, whereas animal fibers predominantly comprise proteins. Once extensively used in composites, mineral-based natural fibers are now steered clear of due to associated health risks, including carcinogenic effects when ingested or inhaled. Consequently, some countries have implemented bans on using these mineral-based fibers. In general, plant-based fibers tend to offer higher strength and stiffness compared to animal fibers. An exception to the general characteristics of animal fibers is silk, which can display high strength. However, silk is relatively expensive, has lower stiffness, and is less commonly used. Given these considerations, plant-based fibers emerge as well-suited alternatives for composites with specific structural requirements, a focal point of the current evaluation. Plant fibers are cultivable in diverse regions and can be harvested after relatively short growth periods. Enhanced productivity is typically achieved with plant fiber varieties characterized by higher cellulose content and well-aligned cellulose microfibrils within the fiber structure. This alignment is often found in bast fibers like flax, hemp, kenaf, jute, and ramie, which are better suited to meet the structural demands of supporting the plant stalk [31].

#### • Matrix Selection

The matrix plays a crucial role in fiber-reinforced composites. It is a protective barrier against harsh conditions, safeguarding the fiber's surface from physical abrasion, and facilitates the transfer of loads to the fibers. Among the most commonly employed matrices in Natural Fiber Composites (NFCs) are polymers, due to their lightweight nature and the ability to be processed at lower temperatures. Both thermoplastic and thermoset polymers are utilized as matrices in conjunction with high-quality fibers [32].

### • Interface Strength

Although natural fibers are sourced from renewable materials and the polymer composites derived from them are considered environmentally friendly, some drawbacks are still associated with their utilization. These drawbacks are often linked to using unmodified or raw fibers in composite preparation. To address these issues, it's essential to mitigate quality variations, enhance moisture resistance, and improve the limited thermal stability of the unmodified fibers [33, 34].

#### • Fiber Orientation

Achieving optimal mechanical properties in composites typically requires aligning the fibers parallel to the direction of the applied load. However, achieving this alignment is more challenging with natural fibers than synthetic yarns. Some alignment can be managed during injection molding, influenced by factors like the viscosity of the matrix and mold design [35].

# 6.5 PRETREATMENT OF NATURAL FIBER FOR COMPOSITE FABRICATION

Natural fibers comprise three primary components: cellulose, lignin, and hemicelluloses. Cellulose stands out as the predominant structural component of natural fibers. It is a semi-crystalline polysaccharide made up of anhydro-D-glucose, which incorporates three hydroxyl groups. Hemicellulose, on the other hand, is a branched, entirely amorphous compound containing numerous acetyl and hydroxyl groups in its molecular structure [36]. Lignin is a highly intricate structure with amorphous polymers composed of phenylpropane groups, primarily aromatic but with lower water absorption capacity compared to cellulose and hemicelluloses. These hydroxyl groups can create hydrogen bonds with the hydroxyl groups in water molecules from the air. Consequently, all-natural fibers exhibit a hydrophilic nature. To enhance fiber structure and quality and promote improved adhesion between the fiber and the matrix within composites, pre-treatment of natural fibers is essential when used alongside synthetic fibers. Various pre-treatment techniques are available to tailor the fibers according to specific research requirements. Examples include mercerization, alkaline treatment, and graft copolymerization. Among these techniques, mercerization and alkaline treatment are particularly effective, as they serve the purpose of fiber refinement by reducing fiber diameter. Mercerization, in particular, is a common method for producing high-quality fibers [37]. It works by smoothening the fiber's surface through the removal of substances such as lignin, pectin, and hemicelluloses. It improves key aspects such as fiber-matrix interface adhesion, fiber strength, durability, resistance to fungal growth, and, most importantly, reduces fiber diameter. Furthermore, natural bioresources can also be employed as natural pre-treatments to enhance the performance of natural fibers in composite materials tailored to specific end applications. Surface wetting and fiber-matrix adhesion characteristics of natural fibers may be impacted by waxy material residues. Cellulosic fibers' main drawback is that they are less compatible with hydrophobic polymer matrices due to their hydrophilic character. The fibers' ability to stick together with most binder resins is diminished when they include free water molecules. The separating agent in the fiber-matrix interface is made up of water molecules. In order to attain good mechanical characteristics in composites, an ideal connection at the fiber-matrix interface is essential [38].

### 6.5.1 Physical Modifications

Physical treatment methods modify the surface characteristics of natural fibers without altering their chemical composition, enhancing the properties of natural fiber composites. Physical treatments include the use of argon treatment, laser treatment, heat, UV treatment and plasma treatment. These treatments improve the thermal, mechanical, and physical characteristics of surface-modified natural fibers over chemical treatment methods. Currently, chemical treatment procedures are preferred by scientists over physical treatment methods, due to the cost difference between the two [39].

### 6.5.2 CHEMICAL MODIFICATIONS

The primary challenges faced by natural fiber composites from the contrasting hydrophilic characteristics of natural fibers and the hydrophobic polymer matrix. The lack of compatibility between these natural fibers and the polymer matrix results in suboptimal interfacial bonding between them. To mitigate this issue, chemical treatments are applied to the reinforcing natural fibers, reducing their hydrophilicity and enhancing their adhesion with the matrix phase. Numerous research efforts have been dedicated to improving the interfacial bonding between the matrix and the reinforcing material through various chemical treatment methods. The following sections outline several chemical treatment techniques utilized to reduce the hydrophilicity of natural fibers and their implications on composite performance [40, 41].

### 6.5.2.1 Alkali Treatment

Alkali treatment removes hemicellulose and waxy materials from fibers, significantly reducing their amorphous content without causing chemical change. In the end, this procedure improves the mechanical characteristics of the composites by increasing the surface abrasion and thermal endurance of the fibers [42]. One commonly utilized chemical for treating plant-based fibers is sodium hydroxide or NaOH. Lignin, hemicellulose, pectin, waxy materials, and surface contaminants are eliminated. As a result, it reveals the fibrils and gives the fibers a surface roughness. Furthermore, it starts the process of alkalization, which transforms cellulose from cellulose I, which is its native state, to cellulose II. Cellulosic fibers swell as a result of alkalization, and the extent of this swelling varies according on the kind of alkali that is employed. Maximum swelling is found when sodium ions (Na+) in NaOH stretch even the tiniest gaps between the cellulose lattice planes, due to their perfect size. A new cellulose I lattice with relatively wide gaps between the cellulose molecules—which fill with water molecules—is created when excess NaOH is removed by washing. The cellulose's -OH groups transition into -ONa groups during this process. But constant water washing eliminates the related Na+ ions and changes the cellulose into cellulose II, a new crystalline structure. The stability of cellulose II is higher than that of cellulose I [43, 44].

## 6.5.2.2 Graft Copolymerization

One of the best ways to change the properties and structure of biopolymers is chemical grafting. Through the process of graft copolymerization, different vinyl monomers are bonded to the cellulose backbone in an attempt to incorporate cellulose with synthetic monomers to produce a material that combines the best qualities of each [45]. Graft copolymerization produces branched polymers by covalently bonding side chain grafts containing functional groups to the primary chain of a cellulose backbone. Many characteristics of cellulose, including its resistance to microbiological harm, elasticity, heat resistance, water permeability, and processing capabilities, can be improved chemically via graft copolymerization with synthetic monomers. Natural fiber composites have been made with the help of these grafted fibers. When compared to unmodified fibers, modified fibers have shown enhanced tensile, flexural, and impact properties in composites [46, 47].

#### 6.5.2.3 Silane Treatment

The surface energy of fibers is directly associated with their hydrophilic character. Silane is a multipurpose chemical substance that is used to modify fiber surfaces by acting as a coupling agent. Commonly utilized silanes can confer hydrophilic properties to the fiber substrate. Examples of such treatment involve the application of vinyl-trimethoxysilane or aminopropyl triethoxysilane. Natural fibers contain minute pores or voids. Therefore, silane coupling agents are applied to coat the surface of natural fibers. These coupling agents can penetrate these micropores or voids, forming interlocked coatings on the fiber surface, thereby reducing the polar component and surface energy of natural fibers. In comparison to natural fiber treated with alkali, those treated with silane display excellent mechanical properties [48, 49].

### 6.5.2.4 Acetylation Modification

Acetylation is another chemical process used to modify natural fibers, involving the conversion of the hydroxyl (OH-) groups within the cellulose structure into acetyl groups, thus increasing their hydrophobicity. The primary objective of this chemical treatment is to encapsulate the OH- groups and make natural fibers more hydrophobic. These hydrophobic fibers greatly improve the composites' overall dimensional stability when they are used to create natural fiber composites. Additionally, improved interfacial bonding with the matrix is also achieved by the acetylation procedure, which also roughens the fiber's surface and decreases voids. During this procedure, acetyl groups are grafted onto the cellulose structure of the fiber, either with or without the presence of a catalyst. It's important to note that fibers do not react favorably with acetic acid or acetic anhydride. Acetylation is employed to reduce the hydrophilic nature of natural cellulose fibers. Moreover, research has shown that acetylation leads to enhanced flexural and tensile properties in natural fiber-reinforced polymeric composites. It also improves the matrix-fiber interphase and fiber adhesion. The resulting composite exhibits improved tensile strength, along with enhanced surface morphology and stress transmission from the matrix to the fibers [50, 51].

### 6.5.2.5 Benzoylation Treatment

The process of benzoylation is employed to reduce moisture absorption within the cell wall of natural fibers. It entails treating the fibers with a solution of benzoyl chloride. This treatment enhances the bond between the fiber and the matrix interface, thereby increasing the composite's strength and improving the natural fiber's thermal stability. Prior to benzoylation, the fibers are subjected to alkali treatment. Hemicellulose, lignin, waxes, and oil compounds are efficiently removed from the structure of cellulosic fibers by this procedure, leaving a large number of exposed OH<sup>-</sup> groups on the fiber's surface. During the benzoylation process, the OH<sup>-</sup> groups on the surface of the natural fiber are substituted by benzoyl groups, which attach to the cellulose structure of the fiber. Hence, benzoylation considerably enhances the interfacial contact with the hydrophobic matrix while decreasing the natural fiber's potential to be hydrophilic. Benzoyl chloride treatment produces fiber composites that are more thermally stable than composites reinforced with untreated fibers [52, 53].

#### 6.6 APPLICATION OF NATURAL FIBERS AS GREEN COMPOSITES

Natural fibers are primarily employed to reinforce non-degradable plastics, creating more environmentally friendly composites. Their widespread use is driven by their cost-effectiveness and global availability. These fibers are effective acoustic and thermal insulators because of their hollowed and cellular structure, which is also soft on processing equipment. Furthermore, it has been shown that processes like mercerization and silane treatment enhance their mechanical properties and lessen their vulnerability to moisture. In order to produce more ecologically friendly composites, non-biodegradable thermoplastic polymers, including polyethylene (PE), polyvinyl chloride (PVC), nylon, and polyesters (PET), are reinforced with a variety of biodegradable plant-based fibers [54]. Additionally, plant-based fibers have been combined with thermosetting resins like polyurethane (PU) and epoxy. Surprisingly, a lot of these composites are made from wood fibers that are extracted by grinding wood products or sawdust, which is a by-product of sawmills. Known as "wood-polymer composites" (WPCs), these composites are mostly used in nonstructural applications and generally have a wood fiber content of 30% to 70%. Throughout the previous ten years, the market for these composites has grown substantially, and future expansion is anticipated. Wood flour and fibers are combined with formaldehyde-based resins to produce medium-density fiberboards (MDF) and particle boards. Plants with longer fibers, including pineapple, jute, flax, kenaf, ramie, linen, henequen, hemp, sisal, and banana, have better mechanical qualities. They can work as affordable substitute reinforcements for composites that need intermediate strength, even if they cannot completely replace high-strength fibers like Kevlar®, graphite, or glass. The fact that these fibers, made from plant stems or leaves, regenerate yearly gives them a significant advantage over wood, which takes 20 to 25 years to achieve full maturity. Some natural fibers, including flax and pineapple, can have up to 1000 MPa strengths based on processing and other factors. On the contrary, fibers such as ramie can have a modulus of more than 120 GPa [55]. These fibers have densities between 1.4-1.5 g/cc, and they can provide some mechanical qualities similar to those of glass fibers. Certain plant-based fibers with hollow structures have better heat and noise insulation characteristics, which makes them useful for insulation and sound absorption applications. These composite materials may be used to build cubicle walls in contact centers and offices because they absorb noise [56].

Regenerated cellulose fibers, including high-tenacity viscose rayon, can also be used as reinforcements in green composites in addition to natural fibers. Tencel fibers, which were created lately, offer a more ecologically friendly alternative to the difficult and damaging conventional viscose rayon production method. The availability in continuous form, as contrasted to plant-based fibers that are usually obtained in shorter lengths, is another noteworthy feature of these fibers [57].

#### 6.6.1 NATURAL FIBER COMPOSITES FROM STARCH-BASED MATERIALS

Starch is a molecule made up of  $\alpha$ -D-glucose units found in two different forms. There are two types: amylopectin, which has a highly branching structure, and amylose, which is linear. Starch granules differ in size and form based on their origin. Starch can be used instead of synthetic polymers when durability over time is not critical. Potatoes, maize, wheat, and tapioca are regular sources of starch. Starch is generally semi-crystalline, and the amount of amylopectin in it dictates how crystalline it is. Through a process of restructuration, starch can be transformed into a thermoplastic material. Acetylating starch can enhance its resistance to moisture. When blended with other polymers, starch-based materials gain increased flexibility. However, due to its hydrophilic nature and relatively poor mechanical properties, starch is limited in its applications. It is often used as a standalone material rather than part of a blend [58].

#### 6.6.2 NATURAL FIBER COMPOSITES FROM SOY PROTEIN RESIN MATERIALS

Soy proteins are complex macromolecules usually made up of 20 amino acids or more. Many of these amino acids have reactive sites that can interact when a plasticizer is present. Therefore, using additives like plasticizers or cross-linking agents, the extrusion process turns soy protein into plastic. Soy protein isolate, which has 90% protein, and soybean protein concentrate (SPC), which has 50-70% protein, are the two main forms of soybean protein sold on the market. Under oxidative circumstances, covalent sulfur bonds at cysteine residues cause soy proteins to become cross-linked. Dehydroalanine (DHA) is produced when alanine's side chain is removed beyond the  $\beta$ -carbon atom. It may also react with cysteine and lysine to generate lanthionine crosslinks and lysinoalanine crosslinks, respectively [59]. Additionally, when asparagine and lysine interact, they can create an amide-type crosslink. The SPI polymer undergoes these reactions throughout the curing process, which produces a resin with moderate strength. The SPI polymer's polar amine, amide, carboxyl, and hydroxyl groups cause it to be extremely hygroscopic and moisture-sensitive. By varying the molding temperature, pressure, as well as initial moisture content, soybean protein plastic's overall mechanical characteristics may be managed. To make soy proteinbased biodegradable plastics, biodegradable polymers are frequently blended with soy protein plastic. A growing number of industries, including the automotive sector and packaging industries, are using these materials. In addition to being economic and ecological, the natural fibers utilized in these composites have superior overall mechanical properties [60, 61].

#### 6.7 APPLICATION OF NATURAL FIBER COMPOSITES

Applications for natural fiber-reinforced composites may be found in a wide range of sectors, including electronics, automotive, aerospace, marine, and sports goods [19]. These materials are quickly becoming a viable substitute for materials based on metal or ceramics. Although these composites exhibit desirable particular qualities, there is a problem with their intrinsic unpredictability. However, these constraints could be circumvented with the development of natural fiber processing methods and their composites. Considering the unique characteristics of individual natural fibers can provide a solid foundation for investigating new uses and prospects in bio-composites or natural fiber composites in the context of the ecologically concerned "green" materials landscape of the 21<sup>st</sup> century. Natural fiber composites' integration into various applications opens new avenues for scholarly inquiry and gives businesses the tools they need to produce sustainable solutions for these materials for potential future uses [2, 6].

#### 6.7.1 NATURAL FIBERS IN STRUCTURAL AUTOMOTIVE APPLICATIONS

Due to global trends and stringent environmental regulations, the automotive industry increasingly turns to lightweight materials and eco-friendly solutions. There are many different types of natural fibers, but only a small number are used in the automobile industry. Over the last ten years, natural fiber composites have gained popularity among European manufacturers as a way to comply with EU laws requiring high levels of vehicle recyclability and reuse. The automotive industry's adoption of natural fibers has spurred research in natural fiber composites. In Germany, natural fiber composites based on polyester and polypropylene (PP) are already used to manufacture various automobile components. Natural fibers are predominantly employed as reinforcements for door panels, rear parcel shelves, seat coverings, and various damping and insulation components. These applications have demonstrated excellent fiber-matrix bonding without signs of debonding or delamination. Furthermore, a hybrid composite consisting of glass fibers and kenaf reinforced with epoxy is employed to improve the mechanical qualities of structural elements in automobile bumper beams. This hybrid composite is appropriate for usage in some automobile bumper beams because its modulus and tensile strength are higher than those of common car bumper beam materials. Jute fibers have replaced glass fibers in the frontal bonnet of vehicles, considering environmental, social, technical, and economic aspects. Jute fibers outperformed glass fibers in this structural application in all aspects except technical performance. Researchers have also explored using pineapple and cassava flour to reinforce polylactic acid and biopolymers. This was done to reduce total volatile organic compound emissions, with comparisons to synthetic polymers like Polypropylene and Polyethylene. Pineapple and cassava-based composites significantly reduced odor emissions, making them highly suitable for automotive interior components. Comparably, glass fibers have been investigated and used with abaca, jute, and their hybrid composites in producing composites. The study found that compared to other composites, the hybrid composite had a noticeably higher tensile strength. In addition, the abaca-based composite outperformed the jute and hybrid composites regarding impact and flexural strength. This suggests that hybrid composites are excellent replacements for various automotive applications, while abaca-based composites suit applications requiring high-impact strength. For this reason, jute and abaca fibers may be utilized successfully as reinforcement in polymeric composites to make engine covers and mudguards for automobiles. Natural fiber composites are also used in the railroad sector for floor and roof panels, berths, dividers, and modular restrooms. Natural fiber use is essential in railroads to achieve high speed, energy efficiency, weight reduction, decreased track wear, and lower inertia. The car industry's transition from conventional fuels to renewable energy sources often supports global sustainability networks. This network sees farmers as the beginning point for a bio-based automotive supply chain system that extends to the automotive sector [62, 63].

### 6.7.2 NATURAL FIBERS IN BUILDING APPLICATIONS

The utilization of natural fiber-based composites is experiencing rapid growth to meet the modern world's demand for cost-effective construction solutions. These composites provide superior acoustic and thermal insulation qualities, particularly in the building sector, because of the lumens/voids present in natural fibers. Structural beams and panels have been developed and evaluated for natural fiber-based composites, especially those utilizing bio-based polymers. Additionally, straw has found widespread application in building materials, where straw bales are excellent candidates for construction materials. Moreover, areca fibers' exceptional acoustic properties for structural applications have been demonstrated by their usage as reinforcement in natural fiber composites. Jute fiber-reinforced polymeric composites are employed in structural applications such as ceilings, window panels, flooring, wall partitions, mobile or prefabricated buildings, and partition panels. Natural fiber-reinforced polymer composites are also employed to manufacture window frames and molded panel components [64, 65]. Research has indicated that composites combining Borassus fruit fiber and High-Density Polyethylene (HDPE) serve well as packaging materials and find various applications in secondary structural roles. In addition, some researchers have explored the use of jute/polypropylene composites for structural and manufacturing feasibility, particularly in producing insulated panels construction materials. These composites are used as outer skins for panels. Furthermore, as loadbearing structural elements, engineers have effectively produced cellular beams and plates utilizing unsaturated polyester reinforced with flax, hemp, and jute fibers [66, 67]. This study revealed that natural fiber-based composites can effectively serve as load-bearing components with enhanced structural efficiency by arranging the cellular material. Composites made of jute and abaca fibers have shown promise in the packaging and housing sectors. Reports on using bamboo fiber to create structural concrete components are also available. Green composites are used in various construction materials, such as fences, doors, and bridges. Additional natural fibers are utilized for reinforcing consist of hemp, maize stalk, coir, and flax fibers [68].

#### 6.7.3 NATURAL FIBERS IN DECKING/FURNITURE APPLICATIONS

With their wood-like appearance and low maintenance requirements, natural fiber composites are an excellent substitute for wood in various applications, including window systems and decking. For instance, banana fiber has the potential to replace wood-based goods like medium-density boards and plywood. Studies on the acoustic qualities of different fiber types have demonstrated that they can perform better than plywood made of wood, making them ideal for a wide range of furniture applications. Paperboards and hardboards can also be made with products based on natural fiber composites. There has also been an investigation into applying jute/polymer composites in furniture manufacturing, including kitchen cabinets, tables, and chairs. Decks and docks made of natural fiber-reinforced composites were influenced by researchers [69, 70].

#### 6.7.4 NATURAL COMPOSITES FOR CIVIL ENGINEERING

Natural fiber composites have the potential to revolutionize civil engineering by combining their inherent advantages with the specific physical constraints of the field. This synergy holds the promise of captivating advancements. Natural composites are crucial in development and form the foundation of various civil engineering projects. Over the past three decades, composite materials, especially plastics and ceramics, have emerged as dominant and innovative materials. The applications of composite materials are on a continual rise, steadily infiltrating and conquering new markets. In engineered resources, modern composite materials now command a significant share. As time progresses, civil engineering has increasingly recognized the benefits of incorporating composite materials into construction practices. There has been a notable surge in the use of composite materials in constructing structures over the years. Moreover, the growing emphasis on energy efficiency, safety, and reliability has made numerous industries need to adopt natural composites within civil engineering. The trend of employing natural composites in the construction industry has endured for quite some time [71].

Moreover, contemporary challenges in civil engineering involve creating reinforced components capable of withstanding natural disasters such as earthquakes and hurricanes. Addressing these challenges necessitates the innovative utilization of composite materials in both new structural systems and existing buildings. Natural fiber composite materials have proven to be highly effective in enhancing the earthquake resilience of concrete structures worldwide. Anticipations suggest that advancements in composite engineering can pave the way for more robust infrastructure in civil engineering, playing a crucial role in shaping the future of construction systems. This integration of natural composite materials is essential for environmental engineering, pushing the boundaries of what can be achieved in building and construction practices [72, 73].

### 6.8 CHALLENGES FACED IN MANUFACTURING NATURAL FIBER COMPOSITES

The biggest challenge in producing natural fiber composites is that they must endure high temperatures. This is especially important when dealing with problems like the smell that comes naturally with natural fibers like jute, hemp, and kenaf. Furthermore, the high cost of natural fiber-based composites makes them
unsuitable for widespread adoption by the massive automobile industry. Incomplete knowledge of the performance of green and sustainable natural fiber composites is another obstacle in this industry. This problem is made worse by the wide range of components that go into making these composites. To overcome these obstacles, coordinated efforts are needed to improve the thermal resilience of green composites, increase their affordability, and compile thorough performance data for wellinformed decision-making. To produce high-quality composites, the main difficulty is to carefully choose the right processing conditions and parameters, make sure the composite elements are chosen precisely, and assess the material's overall properties. One of the main disadvantages of using poor natural fiber properties is how limited their application might be in green composites. In addition, reducing the impact of material origin on the whole life-cycle environmental impact is a major problem in environmentally friendly composites. Important parameters, including product longevity, energy usage, and service efficiency, are frequently used to assess sustainability. A holistic approach to the durability of natural composites across the material life cycle is underscored by the need to address end-of-life treatment for discarded composite materials at the product design phase. The discovery of chemicals and residues escaping from plastic consumer goods has raised public concern about the environmental impact of plastic trash. Bio-based plastic raw materials have been introduced and are currently on the market as a reaction. It is important to remember that although these materials provide an option, carbon dioxide neutrality is not guaranteed. Plastics have almost become essential in many aspects of modern life, such as healthcare, food distribution, construction, automobile and transportation, packaging, and household electronics. Urgent action is required to implement technically sound and environmentally sound material recycling procedures to mitigate the environmental impact. This is especially important in highly populated nations like China, India, and many African countries, where efficient waste management is essential to reducing the adverse environmental consequences of plastic usage in these high-impact industries [74, 75].

#### 6.9 CONCLUSION

Composites made of natural fibers provide a potential new direction in materials research, with a wide range of significant applications. These composites' intrinsic benefits, like their lightweight, renewability, and eco-friendliness, make them a viable substitute in several sectors. Natural fiber-based composites have proven their adaptability in various industries, including construction and automotive, by providing an appealing trade-off between environmental responsibility and performance. These composites have uses beyond simple structural improvements since they also greatly lower carbon emissions and encourage using materials more sustainably. The mechanical characteristics of composites based on natural fibers should continue to advance as long as research and development in this area flourish. Natural fiber-based composites stand out in the rapidly changing field of materials engineering due to their exceptional technical qualities and potential to solve urgent environmental issues. A more sustainable and robust future might be fostered by the widespread adoption of composites based on natural fibers as sectors prioritize sustainability more and more.

#### DECLARATION

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper. Generative AI and AI-assisted technologies have been used in the writing process to improve the readability and language of the work only. After using this tool, the authors reviewed and edited the content as needed and take full responsibility for the publication's content.

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# 7 Natural Fibers in Smart Textiles for Advanced Applications

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#### 7.1 INTRODUCTION

Human beings from the primitive days are surrounded by textiles a full 360°, starting from daily wear to household products to technically sound industrial items to medical textiles. Technological advancement and the growing population demand for goods and services that are more consumer-friendly and generate ease of risk handling are gaining enormous attraction nowadays. The ever-accelerating research and scientific process has led to recent interest in advanced materials. These intelligent textiles, which are basically textiles that are incorporated with electronic components and technology to perform various smart functionalities, are called smart textiles or intelligent textiles. These e-textiles have the ability to respond and react to stimuli from their environment or the wearer [1]. Smart textiles are an important part of wearable technology and can have numerous advanced applications in healthcare, sports, and optical industries. Today, these e-textiles/smart textiles have become a major topic of discussion, leading to their enhanced research. Hence, smart textiles have emerged as a revolutionary product that can perform intelligent functionalities [2].

Textile is a consumer-centric industry. The next-generation performance fibers incorporated into different applications like medicine, sports, electronics, etc., are based on engineered textiles manufactured for complex functionalities. As the time goes on, the use of textiles in various applications has continued to broaden [3]. With the advancements in science and technology, textiles and their applications have also grown manyfold. Traditionally, humans perceived textiles as materials for apparel and home furnishings, whereas now, with diversifications, textiles have gained their place in high-tech end uses for intelligent/smart textiles. Intelligent textiles are now designed for outdoor adventures like camping and hiking tents, medical implants and sutures, sportswear, and much more. Smart, wearable textiles are gaining the lime-light due to their thermal, electrical, and optical properties and their application in various areas. The most exciting part of these smart textiles is their potential to

develop into innovative and intelligent clothing that can generate responses [4]. However, certain factors, like the performance of the material and its sustainability, sophisticated and time-consuming fabrication techniques, generation of toxic wastes from the manufacturing process, and their limited end-of-life portion processability have become barriers to their wide adoption [5].

With an estimated 92 million tonnes of textile waste being produced annually, the textile industry has become the 2<sup>nd</sup> most polluting industry [6]. With advancement and research, it has been established that 95% of these wastes are reclaimable and recyclable. However, still up to 85% is left untreated and dumped into landfills [7]. Manufacturing smart textiles by incorporating electronics will lead to more environmental challenges and waste generation [8]. Reports show that up to 57 components are used for producing e-textiles, and these components have different recycling procedures [9]. Hence, using eco-friendly materials, sustainable techniques, and strategies to produce smart and sustainable textiles that can be recycled or degraded over time is very important.

#### 7.2 NATURAL FIBERS IN SMART TEXTILES

This article focuses on applying sustainable fibers and their manufacturing methods to develop eco-friendly smart textiles. Due to this reason, natural fibers have started gaining popularity. Because they are sustainable, comfortable, and biocompatible, the use of natural fibers has increased. The integration of these fibers with technology can produce eco-friendly functional textiles. The major fiber varieties are natural (cotton, flax, hemp, etc.) and man-made (polyester, nylon) fibers. Cotton and polyester are found to be the most widely used fibers in the world [10]. While cotton is a natural and biodegradable fiber, its processing techniques are not environmentally friendly [11]. Man-made cellulose-based fibers like viscose, modal, and lyocell could potentially play a significant role in manufacturing sustainable wearable electronics. Cotton is comfortable and breathable, which makes it suitable for application in e-textiles. Research shows that cotton fibers have been used with polypyrrole for superior electrochemical performance applications as electrodes for supercapacitors with high capacitance and strong anti-bacterial activity toward S. aureus [12]. Other applications like conductive patterns with ink-jet printing combined with graphene have gained the limelight. The luxurious fiber silk is a strong natural fiber used in various areas for its comfort and other potential characteristics in the health and medical sectors. Silk has numerous applications in areas like silk composite electronic textile sensors, conductive fabrics, wearable pressure sensing devices, etc. [13, 14]. Wool is a well-known fiber for its natural insulation characteristic, making it an admirable fiber in smart textiles. It has been used in thermo-regulation, wearable health monitors, sportswear, safety, outdoor applications, etc. [15]. Other fibers like hemp, bamboo, linen, and ramie are gaining importance in smart textiles [16]. Being robust, soft, breathable, and lightweight, these fibers have been widely used in personal protective clothing [17].

The purposeful generation of application-based products by combining suitable fibers with intelligent technology to enhance the built-in nature of the fiber is a boon. With the aid of natural fibers, it has been possible to take a step forward in this regard [18, 19]. The following are some notable reasons why natural fibers have gained importance in smart textiles.

- *Sustainability*: The natural fibers are biodegradable and renewable in nature, which could make eco-friendly e-textiles.
- *Comfort*: These fibers are well known for their ability to provide comfort and breathability. Some of them are soft, perspiration friendly, and show good moisture management, which makes them preferred to wear close to the skin, for example, in the case of health monitoring.
- *Biocompatible*: Natural fibers are human-friendly and comfortable to the skin, with a lowered risk of skin irritations when used for prolonged periods of time.
- *Aesthetic*: Natural fibers have a soothing/pleasing quality and appearance that attract the fashion and textile industries.
- *Other properties*: Inherited properties like thermal regulation, insulation, and an anti-bacterial nature are advantages for smart textiles.

All these factors make them versatile sources of fibers in smart textile applications. Unfortunately, green, sustainable natural fibers also have certain limitations/ challenges. For example, it is reported that biodegradable cotton is not sustainable, as the processes involved in its processing are mostly toxic and energy-intensive. Also, natural fibers are less conductive, which requires coating/modification with conductive materials to create wearable electronics. These processes sometimes affect the natural feel and behavior of the fiber. Other factors, like durability, moisture absorption, being highly sensitive to the environment and economy, are some of the major challenges. To overcome some of these limitations, researchers and manufacturers are actively working to overcome these challenges by developing modified fibers, coatings, and other treatments for natural fibers to enhance their conductivity, durability, and resistance to environmental factors. Innovations in materials science and manufacturing processes are helping to make natural fibers more viable for a wide range of smart textile applications while maintaining their sustainable and comfortable qualities.

Today, smart textiles show applications in almost all fields, like medicine, transportation, personal protection, outdoor applications, sportswear, etc. The applications of smart textiles can be widely distributed. They are applied to wearable health monitors to integrate sensors to monitor basic health functions such as heart rate and temperature, providing real-time health data to the wearer, and that can be used by sports personnel. Natural fibers can be incorporated into textiles to harness energy from the environment, such as solar or kinetic energy, to power embedded electronics. Smart textiles made from natural fibers like wool can regulate body temperature by changing their properties in response to environmental conditions. This is particularly useful in outdoor clothing. Natural fibers can be used in textiles equipped with sensors to monitor air quality, humidity, or pollution levels, which can be classified under environment monitoring textiles. Fibers like hemp and flax can be used in smart textiles for military and industrial applications, providing durability and resistance to harsh conditions [20].

## 7.3 NATURAL FIBERS AND THEIR TYPES

Fibers obtained from natural sources like plants, animals, or minerals are called natural fibers. They are mainly classified into three different types based on their sources or origin (Figure 7.1). These fibers have been used for thousands of years by human beings for clothing, shelter, and other essential items. They have gained a lot of research interest because they are known for their durability, biological degradability, and renewability [21]. These fibers are considered to be environmentally friendly compared to synthetic fibers. With gradual research and studies of natural fibers, it was found that their inherited characteristics can be useful for their application in many fields like clothing, home decor, industrial textiles, etc.

## 7.3.1 PLANT

In plants, fibers are elongated cells that are much longer than they are wide. These plant-derived fibers can be classified as lignocellulosic, which includes woody species that comprise cellulose, hemicellulose, and lignin. Some examples of lignocellulosic fibers are jute, flax, hemp, ramie, and sisal. Non-lignocellulosic fibers are fibers that do not contain lignin and are found in potatoes, beets, and cotton, among other crops [22]. With the quest for sustainability, the need to use renewable resources and raw materials in various fields of technical textiles, smart textiles, construction, etc., has become necessary [23]. Research has been carried out on the



FIGURE 7.1 Types of natural fibers.

application of these fibers on reinforced polymer composites, cement composites, biomedical applications, etc. [24, 25].

#### 7.3.2 ANIMAL

Naturally available fibers derived from animal sources are called animal fibers (i.e., silk and wool). The basic building blocks of these fibers are proteins. Importantly, extracted animal fibers from different animal sources have unique characteristics that make them useful in textile industries [26]. Also, the fiber types vary with the species they are derived from. For example, there are different types of silk based on the extraction procedure and silkworm properties. The textile industry is a huge consumer-based industry that uses various fibers, polymers, and chemical additives. Traditionally, animal fibers like wool, silk, etc., were used in winter clothes and warm bedding. With the gradual movement of textile and allied industries toward sustainability, these fibers are now used in structural fibrillar architecture, due to their special characteristics like compressive elasticity and chemical resistance [27]. They have also been used in biomedical fields for artificial muscles, actuators, etc. [28]. Although cellulosic fibers are age-old and have vast applications, protein fibers are important due to the demand for high-quality fabric with softness, comfort, and fashion aesthetics. Thus, animal fibers play a significant role in matching the customers' demand. Also, other hair fibers like cashmere, camel, and yak hair are in great demand [29].

#### 7.3.3 MINERALS

The generic term dedicated to all non-metallic, inorganic fibers is mineral fibers. Mineral fibers like glass and asbestos are gaining the limelight due to their good insulation, fireproofing, sound-proofing, and electrical and thermally conductive properties. They are soft, flexible, and a good insulator of electricity, heat, and corrosion. Mineral fibers are used as fillers in fireproofing and thermal insulation materials [30]. Basalt is a mineral fiber that occurs from volcanic rock and ore after melting at high temperatures. This high-performance fiber is used as reinforcements in composites in aerospace, automobiles, civil, defense, and fire protection fields [31].

#### 7.4 NATURAL FIBERS IN SMART TEXTILES

Natural fibers are widely used in all industrial fields (i.e., transportation, wearable electronics, construction, biomedical, personal and healthcare, protective textiles, defense, etc.) These fibers are important components in architecture and construction. It is very important to choose the materials used for structural upgrades, which would aid functional efficiency and improve other properties of structures. With the growing environmental concerns, it is very necessary to use sustainable fiber components for fiber-reinforced polymers. Major fibers like glass, carbon, and aramid fibers were used in functional construction, whereas very little work is seen on structures with natural fibers. Bamboo, coir, jute, and sisal are among those natural fibers of huge interest due to their excellent ballistic properties and physical properties, such

as impact strength and moderate tensile and flexural properties compared to other lignocellulosic fibers [21, 32].

Another highly accelerating sector is wearable electronics. These comprise conductive fibers, fabrics, electrodes, actuators, etc. These conductive fabrics are very useful in creating conductive textiles, which can be used in wearable textiles, medical textiles, and military equipment. Reports show that a copolymer was grafted onto cotton fabrics using citric acid (CA) and sodium hypophosphite (SHP) as crosslinking agents and catalysts, respectively, following a two-dip and two-pad process. The presence of a large number of -OH groups in the copolymer resulted in a successful grafting procedure. It was observed that the grafted cotton fabrics show thermo-sensitive properties [33]. Studies show that textile fibers like hemp, jute, and kenaf were used for manufacturing apparel in military sectors. When combined with electronics and nanotechnology, this material can produce an intelligent response system with outstanding defensive properties. They can help increase combat soldiers' performance by sensing, adapting, and responding to the combat situation. The conductivity was achieved in this work by a dip and dry process using CNT (carbon nanotube) ink. The prepared fabric was analyzed by SEM, UTM, FTIR, TGA, impedance analyzer, piezoelectric effect, and abrasion resistance to investigate its structural, morphological, and mechanical properties, degradation temperature, conductivity, and abrasion [34]. Silk was experimented on by dyeing silk from the silkworm Bombyx mori in conjugation with polymer: polyelectrolyte complex PEDOT: PSS. This resulted in long yarns with high Young's modulus, maintaining bulk electrical conductivity. The high degree of ambient stability made these fibers suitable for dry cleaning [35].

The research was carried out on conductive wires made up of core-sheath structure with cotton yarn of the 20 Ne having a core made up of stainless steel (SS), copper (Cu), and silver treated copper (Cu/Ag) of different diameters (40, 50 and 60 microns). The yarns were produced conventionally by ring spinning and evaluated. The properties of these cotton yarns were improved to a varied extent [36]. Synthesis of conductive hydrogels by a sol-gel method was carried out with non-woven cotton alginate that showed best surface resistivity values below 100 Ohm per square. These hydrogels acted as a 3D networking system, which helped in electron flow; silver ions provided conductivity, and cotton substrate provided strength to the hydrogel [37]. They have the potential for application in smart and technical textiles. Today, electrical resistance in fabrics has become a point of attraction. It can be used in real time monitoring e-textile systems for biomedical applications like sensors that can monitor various physiological parameters, drug delivery, etc. There are reports where cotton fabric was embedded with new functional groups like chloroacetyl chloride, which can act as a bridge between dye and cellulose macromolecules. This concept was developed to observe the sensor property of the newly produced material, and the obtained fabric showed a reduction in the growth of B. cereus, A. johnsonii, and P. aeruginosa [38]. With growing interest in e-textiles and the need for sustainability, the incomparable silk fiber was dip-coated to construct a conductive silk fiber. These etched conductive silk fibers maintained properties of the silk fiber and CNTs, such as high mechanical performance, super-hydrophobicity, resistance to solvent, and thermal sensitivity [39].

Green composites play a vital role in sectors like automotive, aerospace, and marine, which can be manufactured by combining renewable polymer matrix with polymers. Natural, eco-sustainable fibers with comparable properties to synthetic fibers are used in high-end technical applications. Animal fibers comprise complex proteins, including wool, cashmere, mohair, llama, alpaca, vicuna, and rabbit hair. Chicken feathers are used with resins to fabricate fiberboards, i.e., are used in composites. Tussah Silk fibroin and PLA nanofibers were woven to form a 3D cross-sectional fabricated system to form scaffolds. Also, plasma treatment was applied to wool threads for thread-based microfluidic devices. Dielectric barrier discharge (DBD) plasma was evaluated on wool fiber with different gases, viz. oxygen, argon, nitrogen, and air. The used DBD plasma in wool fiber showed an improved wettability. This can be attributed to removing the fatty acid layer from the outermost cuticle surface [29]. Articles were published to show the use of silk in architecture [27].

With the increase in electronic industries, electromagnetic pollution has multiplied rapidly now. The exponential growth of these industries can lead to tremendous increases in the future. An environment-benign solution for fabricating efficient EMI shielding materials with natural fibers experimented with in-situ polymerized pyrrole monomer with jute fabrics, which were noted to have efficient EMI shielding capabilities. Reportedly, jute fabric was found to have the highest value of EMI shielding efficacy for application in robotics and artificial intelligence (AI) [40].

Natural fibers are used for sustainable wearable electronics, such as miniature linear actuators. These actuators, also known as artificial muscles, can work like skeletal muscles, showing contractile action. These have potential applications in soft robotics, prosthetics, exoskeletons, and smart textiles. Researchers studied the anisotropic properties of natural fibers, such as wool, cotton, and flax, that can be utilized to provide muscle-like contractile motion. The fibers were twisted to form modified muscles like cylindrical coils. These actuator-based muscles provide higher output stress, strain, and work capacity than other available alternates [28].

Reportedly, thermally set twisted, coiled, and plied silk fibers were experimented with for water fog and humidity-driven torsional and tensile actuation. A significant value of reversible torsional stroke of  $547^{\circ}$  mm<sup>-1</sup> by torsional silk fiber was found when exposed to water fog. It was found that coiled-and-thermoset silk yarns provide a 70% contraction when the relative humidity is changed from 20% to 80%. This excellent actuation behavior of silk was due to the loss of hydrogen bonds when it absorbed water. The resulting structural transformation was caused by molecular characterizations and dynamic simulation. The silk muscles will lead to enormous industrial applications such as smart textiles and soft robotics, with their abundant, economical, and comfortable characteristics [41, 42].

#### 7.5 NEED FOR NATURAL FIBERS IN SMART TEXTILES

The urge to use sustainable and green resources has gradually increased with the growing eco-pollution concerns, government norms, and depletion of petroleum resources. This has pushed natural fibers into the limelight. Natural fibers have a wide range of textile applications attributed to their exceptional mechanical and physical properties, like specific modulus, low density, toughness, economical, degradable,

and non-toxic nature. Nowadays, smart textiles-based sustainable wearable electronics with the property of responding to environmental stimuli are highly valued in healthcare, protective, defense, conductive, and many other fields of applications. However, with the growing environmental concern, the material performance, sustainability, renewability, and biodegradability of the components used in their manufacturing process are very important [43, 44]. Wearable electronics are worn just like a second skin of the body, combined with actuators, sensors, conductors, etc. Hence, incorporating natural fiber provides comfort and warmth and allows the body to perspire [45].

Fiber-based smart textiles are encouraged due to their unique flexibility and lightweight properties. Other attractive properties, like their brilliant mechanical and physical properties, enabled their wide textile and non-textile applications. These fibers have been reinforced in natural fiber composites, as they are renewable in nature and provide acceptable strength for their use in indoor and outdoor applications. These natural fiber composites have emerged as sustainable alternatives to synthetics, replacing metals, polymers, etc. [18]. The drive for green alternatives has been generated by the declining petroleum reserves worldwide, expensive petroleumbased products, their disposal cost, non-biodegradability, etc., whereas, natural materials are biodegradable, eco-friendly, renewable, and inexpensive [18]. Listed below are suitable properties of natural fibers useful for their high-end application:

- Length and smoothness
- Moisture absorption
- Thermal conductivity
- Strength
- Insulation property
- Therapeutic properties

Natural fibers like jute and hemp are used in conducting yarn with stainless steel yarn in intelligent textiles for antistatic, power and signal transfer, thermal conductivity, or even heat-resistant applications. The hemp fiber induces the best mechanical strength property to the yarn [46]. Natural fibers such as banana, bamboo, and jute fibers are used in various applications, such as automotive interiors as noise-absorbing materials currently containing traditional materials like glass, foam, etc., which are difficult to recycle [47]. These are a few examples of the utilization of natural fibers in smart textiles, which has been a booming trend, too.

## 7.6 ADVANCED APPLICATIONS OF SMART TEXTILES

With the growing demand for consumer-centric appliances and advancements in information science, smart textiles have gained huge attention. The interdisciplinary knowledge has accumulated technology, information, nanotechnology, etc., which has led to the development of response-generating appliances. Smart fibers and textiles, like shape memory textiles, color-changing textiles, thermo-regulated textiles, waterproof and moisture resistant textiles, self-cleaning, and e-information smart textiles, are the major types of smart textiles that have diverse applications in the



FIGURE 7.2 Advanced applications of smart textile.

fields of safety, security, health, etc., as illustrated in Figure 7.2 [20, 48]. The type of fiber/textile that responds to pressure or any external force by regaining its deformed shape is the shape memory fiber smart textiles [49]. Other sensitive fibers or textiles, like photosensitive color-changing textiles, thermo-sensitive textiles, and conductive fibers, which have excellent electrical conductivity and reduce static electricity, act as EMI shields, and detect and transmit electrical signals. pH-sensitive smart textiles are some of the important types of smart textiles mainly applied in various fields. Today, in developed countries, consumers are more concerned about their overall health. The emergence of these textiles has led to the ease of lifestyle management, due to which R&D in this field has surged. Today's age of intelligence has motivated the production of combination fabrics that give a textile feel and intelligent functionality. Wearable technology is a futuristic advancement that brings technology to the consumer's periphery.

TABLE 7.1							
Major Fields of Medical Application of Smart Textile							
Field of Medical Application	Product						
Surgery	Bandages, sutures, soft tissues, implants, etc.						
Hygiene	Uniforms, hospital textiles						
Controlled drug delivery systems	Smart bandages & plasters						
Therapy and wellness	Electrical stimulation, physiotherapy, etc.						

Above are a few advanced application fields of smart textiles, as illustrated in Table 7.1.

#### 7.6.1 HEALTH AND HYGIENE

The field of medical science is gradually advancing every day with the use of new technology and devices. The functional textiles with incorporated sensing and responding technology has led to application products for healthcare with actuating and sensing. Much research dedicated to medicine and health shows in-vitro to in-vivo applications for physiological communication with the environment [50]. Use of geotextiles embedded with fiber optics has also been reported for real-time, online monitoring of tailing disposal facilities [51]. Reports show a geotextile-based sensor for measuring temperature and strain, a woven sensor to assess pulse oximetry [52], etc. Research has been carried out on drug delivery and controlled release of medicines from microcapsules in the medicinal sector [53]. Other application areas show smart textiles in the neuromuscular belts for the back with electrical stimulation for pain by releasing regular electrical impulses [54]. Many studies have been carried out on regenerative medicine with hybrid structures of biopolymers like scaffolds, fiberbased open porous implants, implants for tubular grafts, etc. [55].

#### 7.6.2 E-TEXTILES AND ELECTRONICS

Programmable smart textiles have become crucial to a new emerging technological field. Integrated smart textiles with microelectronic systems (STIMES), a conjugate of microelectronics and technology such as artificial intelligence, have been the need of the hour. Various experimental research projects have shown potential applications in healthcare, intelligent textiles, smart city management, robotics, etc. [5]. Wearable electronics feature mechanical flexibility, integrated knit structure, and comfort to the wearer, along with an integrated intelligent response system. Reportedly, coaxial fiber sensors were produced for piezo-resistive sensing materials that showed a fast response, reversible behavior, mechanical stability, and good repeatability due to their high detection performance and excellent integration [56]. The kind of e-textiles generated show huge applications in health care and monitoring.

#### 7.6.3 SENSORS AND MONITORING

Smart textiles-based sensors vary in the functionality and applications, materials and technology used, and their integration level into textiles. Categorically, sensors can be divided into 4 types:

- Fiber-based: Single yarn sensor
- Textile-based: Textile substrate but an inseparable part of the device
- Textile structured: Each part is a textile material
- Textile integrated: When a textile is a function carrier

The implementation approach of the sensors varies from the area of application. For example, optical fiber technology can evaluate certain assessments like temperature, moisture, blood oxygen level, sweat pH level, respiration rate, and movement. Although sensors vary with various applications, their working principle is mainly based on a substrate and sensing or active materials. Textile sensors have now been developed to be a part of sensors based on electrodes, temperature, pH, conductivity, resistance, humidity, pulse oximetry, etc. [50].

Reports are found where textile sensors are used in obstacle avoidance situations. These sensory shirts are developed for sensing any obstacle for visually challenged persons [57]. Shape memory fibers for compression systems were used to assess chronic venous disorder [58]. Wearable body sensors for medical and health care were researched, which involved biomedical computing, wireless data acquisition, and sensor body networking [59]. Smart textiles are also applied to the structural health monitoring of composite structures, which act as piezoelectric sensors [60]. Sensors and monitors sometimes work hand in hand. Fibrous sensors have been manufactured to monitor the weaving process by observing the kinematics of weaving and the mechanical stress enforced during the process [61]. Textile heat flux meters were introduced to manage and maintain the heat transfer in the weaving process, and flux was generated [62]. These flux meters were used to analyze the effect of moisture on heat transfer during weaving. Also, research in the sectors of electro-conductive smart textiles and electromechanical sensors is gearing up [63]. Integrated smart textiles are implemented to sense the mechanical output by quantities like position, movement, speed, acceleration, elongation, forces, pressure, and vibration [64].

#### 7.6.4 ENERGY GENERATION AND STORAGE

With the constant advancement in the field of science and resources, textiles in integration with energy harvesters have helped to generate green and sustainable, consumer-friendly wearable energy for the on-body electronics in today's e-era. Energy with the aid of smart textiles can be produced from biomedical, body heat, biochemical sources, solar, and other hybrid forms. The surface charging behavior is exploited to recover energy by layer stacking of textiles with different electron affinities to produce energy [65]. Various energy storage devices with textile-based batteries and capacitors have been developed. Electrodes made up of stainless steel

yarn, silver coated yarn, etc., are some of the textile energy storage smart devices [66]. Researchers are also studying light guide fiber-based textiles that collect and propagate the sunlight and produce energy [67].

#### 7.6.5 PROTECTION AND SECURITY

Individual security and protection are of huge significance today. Some of the appliances, like policeman uniforms, military equipment, personal safety equipment, etc., are the sectors of focus. Technologies based on manufacturing vital sign sensors for athletes' communication protocols have been established [68]. Application of smart textiles can be seen in personal protective electronics like end-of-life indicators, phase-changing materials, physiological condition regulators, shear thickening fluids as shock absorbers, etc [69, 70]. Keeping workplace safety in mind, occupational protection of employers can be taken care of with proper usage of PPE like smartwatches [71].

#### 7.6.6 FUTURE PERSPECTIVE

Smart textiles are an integrated conjugation of adaptive textile materials with multi-functionalities. Although a wide arena of advancements has been seen in the field of applications of smart textiles, a noticeable gap still exists between the customer requirement and the state of the art of the devices. A futuristic view of these e-textiles with textile comfort will gradually lead to the adaptation of this intelligent apparel in the daily utilization of consumers. The perspective of addressing this gap can be objectified by giving a holistic approach to smart textile operation, exploring the manufacturing method of the stimuli-responsive systems, discussing the emerging application area, and identifying the market and scope for future development. In the present era of sustainability, natural fibers and textiles have become the greener alternatives to polymers and synthetic fibers for their application in wearable electronics and intelligent textiles such as soft assistive robotics, medical and health, protective, and defense. Incorporating fibers from natural sources has evolved to be the desirable stimuli response system, the second skin to the wearer. To meet the present-day needs and function, it is important for interdisciplinary collaborations that can focus on the major breach between research and relevant products, bringing smart textiles to the future. With the increasing regulatory restrictions, developed countries are focused on devices from biomaterials like chitosan, facing a shift of fibers from animal sources like collagen, given customers' present demand for comfortable, smooth, soft products with fashionable aesthetics and intelligent wear.

The demand for e-textiles and intelligent apparel is growing fast and will take many folds soon with the rapidly growing industry. Futuristic projections on the new technologies and functions for intelligent clothing can be attributed to the advanced functionalities of clothing by the wearer. The establishment of these functionalities mainly requires technical feasibility and manufacturing specialties that can lead to a smart or intelligent sustainable environment.

#### 7.7 CONCLUSION

In this chapter, we have discussed the present status of electronic wearable textiles and intelligent clothing. The article focuses on the utilization and incorporation of natural fibers in the manufacturing of smart textiles. It briefly describes the advantages and possible reasons for using natural fibers. It throws light on the sustainable approach of manufacturing smart textiles nowadays, keeping environmentally friendly apparel in mind. This article aims to bring the relevance of natural fibers in smart textiles to the application in high-end response-demanding sectors in an integral channel for better understanding. Smart textiles are referred to as the future of this generation, being the stimuli-responsive systems. The present chapter is a view of smart materials and integrated textiles. The article describes the importance of the application of smart textiles in different areas like medical, protection, energy, wearable electronics, etc. It explores the wide range of applications in prosthetics, implants, sensors, conductors, stimuli responding devices. Finally, it also presents the futuristic trend of e-textiles considering environmental and commercial aspects. This chapter throws major importance on wide research requirements for adapting and designing such wearer friendly sustainable e-textiles. Thus, the approach addresses the bridge between utilizing eco-friendly raw materials like naturally sourced fibers for the advanced application of stimuli-response systems/ intelligent wearable textiles.

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# 8 The Utilization of Natural Plant Fibers as a Sustainable Substitute Material for Textile and Other Uses

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#### 8.1 INTRODUCTION

In the 21st century, humanity is faced with a unique and significant task. This challenge involves effectively managing and reducing the environmental consequences of industrial activities, all while ensuring the continued fulfillment of the increasing worldwide need for various resources [1, 2]. In light of the urgent issues surrounding climate change, resource depletion, and pollution, the imperative for sustainable alternatives has become increasingly paramount. One crucial element of this shift toward sustainability involves the investigation of natural plant fibers as a flexible and environmentally benign alternative material [3, 4].

In today's environmentally conscious era, plant fibers sourced from jute, flax, hemp, and bamboo have become a sustainable option due to their natural origin [5-11]. These fibers demonstrate a wide range of characteristics, such as exceptional tensile strength, low density, and the capacity to undergo biodegradation [12-14]. The utilization of these materials has the capacity to bring about a significant transformation in various

sectors, such as textiles and construction, by presenting a sustainable and renewable substitute for materials generated from petroleum [15, 16]. Polyethylene terephthalate (PET), a widely used polymer resin belonging to the polyester family, has emerged as a prominent fabric material for combining with natural fibers. Nevertheless, acquiring knowledge regarding the detrimental consequences of plastic and polyester and their permanent implications on the environment was a process that required a significant amount of time [17].

Textiles are very important to us in our everyday lives. Textiles include many things, from the most basic needs to the most high-fashion items. They have a big impact on many parts of our daily lives. Textiles are used in science, industry, and medicine in addition to their usual uses as decorations, home accessories, and packing [18, 19]. The textile business in India has a special place because it is one of the oldest. It accounts for 14% of all industry production in India [20]. The long history of India and textiles goes back hundreds of years. During the late 17th and early 18th centuries, when cotton exports were booming, these textiles gained much attention worldwide. India has been known throughout history as a major producer of many natural products, such as cotton, silk, jute, and wool. Over the years, the textile industry has been a big part of the Indian economy. It has led to creating jobs, a boost in industrial output, and an increase in exports. However, some believe the textile industry poses a significant environmental risk. Creating industrial textiles involves a series of steps, including pre-treatment, dying, printing, and finishing [21, 22]. Not only do these manufacturing processes use a lot of electricity and water, but they also make a lot of trash. Many dyes, chemicals, and other things are used in the textile business to give fabrics the qualities they need. There are a lot of effluents from these activities [21, 23].

The growth of natural fibers is influenced by the continent and climate zone, resulting in year-round availability in certain regions. The quality and quantity of fibers during harvest, as well as during and after cleaning and processing, are subject to variations due to the influence of local and worldwide weather conditions [24, 25]. The supply of natural fibers is influenced by geopolitical or regional events. Technological advancements strongly correlate with the progress of industrialization [26]. The fundamental attributes of the textile industry underwent a transformation over time due to advancements in the production of synthetic materials. Mass-produced, blended fabrics held a dominant position in the textile industry. The concept of productivity emerged as a central focus in the operations of major textile plants in India and other regions. Consumers experienced advantages due to the comparatively reduced cost of synthetic fabrics compared to their natural counterparts [27, 28]. Textile enterprises leveraged advancements in materials, design techniques, and printing technologies as a means to attain a competitive edge and expand their market presence.

The current global industrial environment is undergoing a significant shift due to an increasing recognition of the environmental impacts associated with traditional manufacturing methods. Industries are seeing heightened examination and demands from consumers, authorities, and shareholders to embrace sustainable methods and mitigate carbon emissions. The issue of climate change, along with its consequential global warming, has prompted widespread attention and efforts to mitigate plastic consumption. Suddenly, there was a resurgence in discussions surrounding natural plant fibers, characterized by a heightened emphasis and enthusiasm.

The utilization of plant fibers derived from natural sources has become increasingly prominent due to their environmentally favorable attributes, thereby positioning them as a sustainable alternative. These natural fibers offer a feasible substitute for synthetic materials, originating from the fundamental elements of the natural world [29–32]. The distinctive amalgamation of characteristics they possess situates them as a versatile primary substance with the capacity to alleviate some of the most urgent problems confronting society in the present era. The chapter will comprehensively analyze the characteristics, practical uses, and ecological ramifications of plant fibers derived from natural sources. This chapter will examine the potential of these fibers to contribute toward a more sustainable and environmentally conscious future. In the pursuit of reconciling economic advancement with environmental preservation, the utilization of these fibers serves as a symbol of optimism, illustrating the harmonious collaboration between innovation and the natural world in addressing the challenges posed by a dynamic global landscape.

#### 8.2 NATURAL FIBER IDENTIFICATION

Natural fibers offer numerous benefits, including impressive tensile strength, low density, and biodegradability, making them an attractive option for environmentally conscious businesses [33]. Compared to synthetic fibers like polyester and nylon, natural fibers not only match their synthetic counterparts in strength and performance but also have a significantly lower environmental impact [34]. Their versatility extends beyond textiles, finding applications in composites, paper, packaging, and even as reinforcements in construction materials. Their adaptability and sustainability make them promising candidates for future use in the construction industry [35]. The next sections provide an in-depth discussion of the characteristics that are shared by all types of fibers.

#### 8.2.1 LENGTH/DIAMETER

The measurement of length is a significant attribute that determines the use of a textile fiber. In a broad context, the reference to fiber length in the range of a few centimeters implies a corresponding fiber diameter of a few microns. In the context of filament fiber, the aforementioned ratio would exhibit a significant magnitude and may lack relevance [11, 36].

## 8.2.2 ELASTICITY

The elasticity performance depends upon the garments' ability to exhibit a reinforcing nature regarding their material and spatial characteristics after deformation induced by external forces [37]. The stretch level, the material's duration in a stretched state, and the time required for recovery often influence elasticity or elastic recovery.

## 8.2.3 FINENESS

Fiber composition heavily relies on the level of fineness. Measuring fiber thickness involves determining its width, diameter, and cross-sectional area [38]. It's interesting to note that only a few fibers have a fully circular cross-sectional shape.

## 8.2.4 STRENGTH

It is crucial to prioritize the durability of the cloth. To guarantee longevity, the fabric needs to have adequate strength. The fabric's durability depends largely on the strength of the fibers that make up the cloth [39]. Factors like bending, tensile strength, and bursting strength are evaluated according to the direction and usage of force.

## 8.2.5 SPINNABILITY

Spinnability is the term used to describe how well a material can be spun into a thread or yarn. The findings suggest that the individual fibers should possess the ability to be transformed into a yarn, which may subsequently be woven into a fabric that exhibits adequate strength [40]. The concept of spinnability is commonly employed in the process of developing man-made fibers.

## 8.2.6 UNIFORMITY

The concept of uniformity is a significant aspect of having minimal variances in length and diameter among the fibers. Achieving a more consistent fiber quality ensures uniformity in the yarn and the resulting fabric [41].

## 8.2.7 THERMAL CONDUCTIVITY

The thermal conductivity of a material indicates how well it can conduct heat. Clothing primarily serves to enhance aesthetics. Nevertheless, the primary objective is to reduce the effects of extreme temperatures, particularly to safeguard against low temperatures. The heat conductivity of fibers is a significant property [42].

## 8.2.8 CHEMICAL AGENT RESISTANCE

The response of fibers to chemical substances exhibits significant variability based on their respective classifications. In general, fibers derived from plants exhibit a tendency to be less resistant to acidic environments while demonstrating more resilience in alkaline conditions. Artificial cellulose fibers are sensitive to acid and alkaline conditions. In contrast, synthetic fibers are stable in acidic and alkaline environments [4, 43, 44].

## 8.2.9 Hygroscopic Property

The hygroscopic quality of a substance refers to its ability to attract and retain moisture from the surrounding environment. When a fiber is in the atmosphere, it has the inherent ability to absorb moisture automatically [45, 46]. Nevertheless, the rate of rise in humidity does not always exhibit a straight correlation with the increment ratio.

## 8.3 CLASSIFICATION OF TEXTILE FIBERS

Natural and synthetic fibers used in the textile industry are easily distinguished categories. Natural fibers are defined as fibers in their fibrous state in nature. Manufactured fibers, alternatively known as man-made fibers, are synthetic materials produced by industrial processes.

#### 8.3.1 NATURAL FIBERS

The term "natural fiber" refers to plant-based fibrous material that is generated via the process of photosynthesis. The sources are divided into three groups: animal, plant, or mineral. Plant or vegetable fibers are more precisely categorized as cellulose-based fibers, extracted by isolating them from different plant parts like the stalk, stem, leaf, or seed. Cotton, jute, and linen, which are derived from cellulose or plant-based sources, represent natural fibers [47, 48]. Animal-derived fibers are more precisely known as protein-based fibers. Wool, silk, and mohair are natural fibers obtained from animals known for their protein composition. Mineral fibers are derived from natural resources extracted from the Earth's crust [49]. Throughout history, humans have employed natural fibers to make essential items such as clothing, shelter, and other vital commodities. With the exception of silk, staple fibers refer to all natural fibers derived from cellulose and protein that are obtained in relatively short lengths [39]. Silk is classified as a filamentous fiber with a continuous structure [50]. Natural fibers have environmentally friendly properties since they are renewable and completely biodegradable. Natural fibers are utilized in various applications, such as clothing, home, and industrial textiles [51].

Figure 8.1 illustrates an extensive classification of natural fibers. The subject matter can be categorized according to its origins and properties. The information on the mechanical properties of the various types of fibers derived from various sources is presented in an easy format in Table 8.1. There exist several prominent categories of natural fibers, which are as follows.

#### 8.3.1.1 Cellulosic Fibers

Cellulosic fibers are a type of natural fiber that is predominantly formed from cellulose. Cellulose, a complex carbohydrate, can be found in plant cell walls. Cellulose consists of linear chains of glucose units linked together to create cellulose. Carbon, hydrogen, and oxygen comprise cellulose's three fundamental components. These fibers are highly regarded for their versatility, comfort, and durability, making them popular in various sectors, especially fashion and textiles. It is also known as plant fibers because it may be derived from various plants' stems, leaves, and bark. The cellulose fibers share many of the same fundamental features, yet they all have their own unique drawbacks. Other examples of such materials include hemp, flax, jute, ramie, sisal, and bamboo [51–53]. Cellulosic fibers are considered more sustainable



FIGURE 8.1 Types of natural fibers.

## TABLE 8.1Physical and Mechanical Properties of Natural Fibers [52]

		Density	Diameter	Micro- Fibrilar	Tensile Strength	E-Modulus	Elongation at Break
Fiber Types		(g/cm³)	(µm)	Angle (°)	(MPa)	(GPa)	(%)
Bast	Flax	1.5	12-30	5-10	345-1100	27.6	2.7-3.2
	Hemp	1.48-1.49	16-50	2-6.2	690	30-60	1.6
	Jute	1.3-1.4	5-25	8	393-773	13.0-26.5	1.2-1.5
	Kenaf	_	12-36	_	930	53.0	1.6
	Ramie	1.5	16-125	7.5	400–938	61.4–128.0	1.2-3.8
Leaf	Abaca	1.5	_	_	400	12.0	3.0-10.0
	Curaua	1.4	_	_	500-1150	11.8	3.7-4.3
	Pineapple	_		_	413-1627	34.5-82.5	1.6
	Sisal	1.4	50-200	10-22	468-640	9.4-22.0	3.0-7.0
	Banana	_	_	12			
Seed/ Fruit	Coir	1.1	100-460	30-49	131-175	4.0-6.0	15.0-40
	Cotton	1.5-1.6	12-38	_	287-800	5.5-12.6	7.0-8.0
	Oil Palm	0.7-1.55	_	42	248	3.2	25.0
	Kapok	_		_			_
Grass	Bagasse	1.25	_	_	290	17	_
	Bamboo	0.6–1.1	—	—	140-230	11–17	—

than synthetic fibers like polyester and nylon because they come from renewable plant sources. When sustainable agricultural techniques are employed, cellulosic fiber production can reduce a negative impact on the surrounding environment [54]. These practices include organic cultivation and responsible harvesting. Cellulosic fibers are put through various processing procedures to transform the basic plant components they are made from into textile fibers that can be used [55–57]. Pulping, spinning, and finishing are all possible outcomes of these operations [24]. Cellulosic fibers are renowned for their natural characteristics, such as breathability, moisture absorption, and biodegradability. To improve certain properties, such as tensile strength or elasticity, they can be combined with other types of fibers, such as polyester or wool. Unlike synthetic fibers, cellulosic fibers may be more prone to developing creases and shrinking in size. To keep their eco-friendly reputation, it is necessary to use environmentally responsible dyeing and finishing techniques [58, 59]. Cellulosic fibers are commonly used in manufacturing a wide range of products, including clothing, bedding, towels, and nonwoven items like wipes and sanitary products. Applications in technical fabrics, such as those used in the automotive and medical industries, are also being investigated as possible uses for these materials [33, 60]. Researchers are always looking for new ways to process, dye, and polish cellulosic fibers that will allow them to function better and be more environmentally friendly. Encouraging cellulosic fibers' usage and sustainability is possible by educating consumers about the benefits of selecting items manufactured from cellulosic fibers. Overall, cellulosic fibers offer a sustainable and adaptable alternative for textile and other applications. Furthermore, current research and development work aims to make them even more eco-friendly and functional. Stem, leaf, and seed comprise the three primary categories defining cellulosic fibers.

#### 8.3.1.1.1 Stem Fibers

The fibrous tissue that is present in the stems or stalks of certain plants is where stem fibers are obtained. Stem fibers can be spun into yarn. Various plants can be used to extract stem fibers, with popular options including flax, hemp, jute, kenaf, and ramie. In most cases, the extraction process begins with the harvesting of the plants. It continues with the separation of the fibrous tissue from the non-fibrous portions, such as the leaves or the material composed of wood [4, 61, 62]. This can be accomplished by various methods, including soaking, mechanical decortication, or chemical treatments. Stem fibers are known for their exceptional tensile strength, making them suitable for applications that demand durability and resistance to wear.

#### 8.3.1.1.2 Leaf Fibers

The leaves of certain plants, typically those with fibrous leaf structures, are used to extract the natural fibers known as leaf fibers. Because these fibers are known for their strength, flexibility, and low weight, they are excellent materials for various applications, both conventional and modern. Agave, sisal, and abaca are all common plants that can be used to harvest leaf fibers. The process of extracting fiber often entails harvesting the leaves, separating the fibrous material from the non-fibrous components, and occasionally recycling or other treatments to break down the

cellular structure and make fiber extraction easier. Leaf fibers are highly sought after for applications requiring a durable, strong material that can withstand stretching and breaking. In applications where weight is an important consideration, such as in the production of ropes and twines, the fact that leaf fibers are relatively lightweight makes them an attractive material choice [63–66].

#### 8.3.1.1.3 Seed Fibers

Seed fibers are natural fibers obtained from the seeds or seedpods of certain plant species. The aforementioned fibers are highly esteemed because of their exceptional strength, durability, and remarkable adaptability across a wide range of industrial contexts. Cotton, kapok, and coir are frequently encountered as primary sources of seed fibers. The extraction procedure commonly entails the separation of the fibers from the seeds. The aforementioned objective can be accomplished using many procedures, including ginning for cotton, decorticating for kapok, and retting for coir. Numerous seed fiber plants are widely acknowledged as sustainable due to their yearly cultivation and ability to yield fibers without resorting to deforestation or excessive pesticide application. Nevertheless, cotton cultivation in conventional farming practices is often linked to substantial water consumption and the application of chemical inputs [64–67].

#### 8.3.1.2 Protein Fibers

Protein fibers, alternatively referred to as animal fibers, are naturally occurring fibers that are predominantly sourced from the proteins present in the hair or fur of animals. These fibers are renowned for their thermal properties, smooth texture, and luxurious nature, rendering them highly sought after within the textile and fashion sectors. Protein fibers are derived from a variety of animal sources. Typical sources of natural fibers encompass several animal species, such as sheep (providing wool), silkworms (yielding silk), goats (supplying cashmere and mohair), rabbits (producing angora), and camels (contributing camel hair) [68, 69]. Following the harvesting process, animal fibers undergo a series of distinct processing stages, encompassing washing, carding, spinning, and weaving, with the ultimate objective of converting them into practical fabrics or materials. Protein fibers are renowned for their inherent characteristics, including thermal insulation, air permeability, and hygroscopicity. These entities frequently possess inherent hypoallergenic properties and exhibit resistance to olfactory stimuli. Protein fibers have extensive application across various consumer goods, encompassing articles such as sweaters, scarves, socks, blankets, and luxury fashion apparel. Furthermore, they are employed in the manufacturing process of high-end textiles, accessories, and household items.

#### 8.3.1.3 Mineral Fibers

Mineral fibers encompass a classification of inorganic fibers originating from natural minerals. These fibers possess notable heat, fire, and chemical resistance properties, rendering them highly beneficial in diverse industrial applications. Mineral fibers are commonly sourced from naturally existing minerals, such as rock or slag. Typical sources encompass asbestos, basalt, glass, and slag wool [70, 71].

#### 8.3.1.4 Microorganism-Based Fibers

Biofibers, also known as microorganism-based fibers, are generated via the culture of various microorganisms, including bacteria, fungi, algae, and other similar microorganisms [72]. These fibers present a sustainable and pioneering approach to manufacturing textiles and materials.

## 8.3.1.5 Natural Composite Fibers

Composite fibers originate from different sources, like wood fibers mixed with resin or coconut fibers mixed with rubber.

## 8.3.2 MAN-MADE FIBERS

Man-made fibers (Figure 8.2), often known as synthetic fibers, are textile fibers that are manufactured using chemical methods instead of being obtained from organic sources such as plants or animals. These fibers possess diverse characteristics and are useful in many applications across several industries. Artificial fibers can be intentionally designed to possess distinct characteristics, such as robustness, flexibility, moisture absorption, and flame retardancy, contingent upon their intended usage. Synthetic fibers frequently have several advantages, including enhanced durability, colorfastness, resistance to pests and dampness, as well as convenient maintenance.



FIGURE 8.2 Types of manmade fibers.

Moreover, synthetic fibers have the potential to be more economically efficient compared to natural fibers. Nevertheless, there are ongoing endeavors to mitigate the environmental consequences by implementing recycling initiatives and adopting sustainable production methods. Synthetic fibers find extensive utilization across various domains, encompassing the garment industry (such as sportswear, swimwear, and outdoor attire), household textiles (including bedding, upholstery, and carpets), industrial goods (such as ropes, filters, and geotextiles), as well as technical textiles (including airbags, medical textiles, and bulletproof vests). The current focus of research involves enhancing synthetic fibers to have better properties, including creating conductive fibers for use in wearable electronics, smart textiles, and ecofriendly synthetic options. Blending synthetic fibers with natural fibers like cotton or wool is a popular technique to create textiles with improved functionality and comfort by leveraging the strengths of each material. Synthetic fibers encompass a variety of materials, such as polyester, nylon, acrylic, rayon, spandex, polypropylene, and aramid [73-75]. Synthetic fibers can be categorized according to their chemical composition and the method by which they are produced. Several often-used classifications include:

- **Polyester** fibers are renowned for their exceptional durability, remarkable resistance to wrinkles and fading, as well as their remarkable ability to wick away moisture. These materials are frequently utilized in the manufacturing of garments, household textiles, and several industrial sectors [75, 76].
- **Nylon** fibers possess notable strength, elasticity, and resistance to abrasion. These materials find extensive application in various consumer goods, including but not limited to garments, hosiery, cords, and outdoor equipment [77].
- Acrylic fibers are characterized by their softness, lightweight nature, and thermal properties. Synthetically produced fibers are frequently employed as a viable alternative to wool in the manufacturing of various textile products, such as knitwear, blankets, and faux fur [78].
- **Rayon**, also referred to as artificial silk, is a textile material derived from natural cellulose sources like wood pulp [79]. Silk is highly esteemed for its lustrous aesthetic and luxurious feel, rendering it a sought-after material in various textile applications, such as apparel and household decoration.
- **Spandex**, often known as elastane, is a type of synthetic fiber that exhibits exceptional elasticity and imparts stretch and recovery characteristics to textiles [80]. These items are frequently utilized in the manufacturing of sportswear, swimwear, and form-fitting apparel.
- **Polypropylene**, a thermoplastic polymer, has notable characteristics such as being lightweight, displaying resistance to moisture, and possessing exceptional insulating properties [81]. They find application in various contexts such as thermal undergarments, floor coverings, and outdoor fabrics.
- Aramid fibers, such as Kevlar, provide exceptional strength and exhibit high resistance to heat. They are employed in various applications where the utmost importance is placed on great strength and flame resistance, such as manufacturing body armor and industrial safety equipment [82].

## 8.4 COMMON NATURAL FIBERS

## 8.4.1 COTTON

Cotton is one of the cellulosic fibers that is used in most applications. The fibers of the cotton plant are responsible for this material, known for its flexibility, breathability, and ability to absorb and keep moisture. Cotton is one of the most widely known and used seed fibers. It is obtained from the cotton plant's seed hairs [47, 83]. Cotton fibers are prized for their softness, breathability, and absorbent qualities. Cotton plays a significant role in the textile industry, used in various products such as clothing, bed linens, and medical supplies.

## 8.4.2 FLAX

Flax plant stems are harvested to obtain fibers for making flax yarn. Because it is long-lasting and allows air to pass through it, linen is frequently used to produce house textiles and summer clothes [53, 84].

## 8.4.3 Немр

The hemp plant is the source of the fibers known as hemp [85]. They are sturdy and long-lasting and may be used in various applications, from industrial equipment to clothes.

## 8.4.4 Вамвоо

The pulp of bamboo plants is used in manufacturing bamboo fibers. They are wellknown for their suppleness, the ability to wick away moisture, and long-lasting nature [86].

## 8.4.5 Карок

The fibers of the kapok tree are derived from its seedpods. These items are very light and float easily, which makes them ideal for use in life jackets, pillows, and mattresses [87]. Kapok is often used as a natural alternative to synthetic filling materials.

## 8.4.6 COIR

Extraction of coir fibers occurs from the outer husk of coconuts. They are known for their durability, resistance to saltwater, and natural resistance to pests and fungi. Coir fibers make products like ropes, mats, brushes, and horticultural products such as potting mixes and erosion control materials [88].

## 8.4.7 WOOL

Wool, derived from sheep's fleece, is widely recognized as a prominent example of protein fibers. The material is highly regarded for providing warmth, effectively managing moisture, and exhibiting suppleness [89]. Various varieties of wool exist, each possessing distinct attributes. For instance, Merino wool is renowned for its exceptional softness and delicate fibers.

## 8.4.8 SILK

Silk is a naturally occurring protein fiber generated by the larvae of specific insects known as silkworms. The material is renowned for its glossy aesthetic, velvety consistency, and its ability to be both lightweight and breathable. The silk production process entails the collection of silkworm cocoons, which are subsequently subjected to processing methods aimed at extracting the silk fibers [90].

## 8.4.9 CASHMERE AND MOHAIR

Cashmere is sourced from the fleece of cashmere goats, while mohair is obtained from the hair of angora goats [91]. Both fibers are widely recognized for their remarkable softness, warmth, and lightweight texture. Cashmere is highly valued because of its exceptionally fine fibers and opulent texture.

## 8.4.10 ANGORA

Angora fiber is derived from Angora rabbits and possesses exceptional softness and warmth. Frequently, it is combined with various fibers to produce opulent textiles [91].

## 8.4.11 CAMEL HAIR

The collection of camel hair fibers is derived from the Bactrian camel [91]. Camel hair textiles are renowned for their exceptional insulating capabilities and ability to provide warmth and lightweight comfort.

## 8.5 USES OF NATURAL FIBERS

Natural fibers' many properties and characteristics contribute to their extensive range of applications. Several examples are examined and depicted in Figure 8.3.

- **Rope**: Hemp, jute, and sisal are materials that offer great tensile strength and endurance, rendering them well-suited for use in the creation of rope.
- **Sports**: Sports equipment, such as skateboards, snowboards, and surfboards, can be manufactured using materials derived from hemp and bamboo.
- **Textiles**: Natural fibers like cotton, wool, silk, and linen are frequently used in making clothes, bedding, and other textile goods. Moreover, they are used to produce nonwoven fabrics and industrial textiles.
- **Paper**: The production of paper involves the utilization of several materials, including wood, bamboo, and hemp. The manufacturing process utilizes these materials to produce an extensive array of paper products, including books, newspapers, and packaging materials.



FIGURE 8.3 Uses of natural fibers.

- **Personal Care**: Bamboo, hemp, and cotton are commonly employed in the manufacturing process of sanitary products, wipes, and various personal care articles.
- **Biocomposites**: It covers a variety of materials that use natural fibers for reinforcement. Materials such as wood-plastic composites, natural fiber-reinforced polymers, and natural fiber-reinforced concrete are good examples.
- **Building materials**: Various building components, such as insulation, flooring, and roofing materials, can be produced using bamboo, hemp, and flax fiber.
- Agriculture: The utilization of coir and sisal fibers in the production of twine and rope finds application in agricultural settings, specifically for tasks such as securing plants to stakes or trellises and creating biodegradable mulch mats.
- **Industrial Products**: Industrial products cover a variety of items made from materials like jute, sisal, and coir. These natural fibers are commonly used in creating various industrial products, such as mats, geotextiles, and abrasive items.
- Automotive: Within the automotive industry, natural fibers like flax and hemp are used as reinforcement agents in biocomposite materials, which find applications in structural components and interior elements of vehicles.

## 8.6 TEXTILES

In today's world, textiles go beyond just meeting basic clothing needs. They also allow people to express their unique sense of style. Fabric is what is seen and is the most maneuverable of props; it is the most easily changed, replaced, or added to. It dictates the mood, determines the style, and reveals the wearer's taste. The sun and light can be controlled by the fabric. It prevents the colors of the interior from fading and keeps the furniture from deteriorating due to the sun's rays. Fabrics have
the capacity to make music and even speech richer and more resonant, as well as minimize noise and enhance the amount of space that can be used for work or living. The textile sector in India continues to enjoy India's unwavering support and close cooperation. Fabric plays a significant part in the home textiles industry by performing functions such as protecting individuals from drafts or heat and reducing heat loss during the colder months. It shields against the sun's glare and protects against the darkness of night and the excessive brightness of the sun in the morning and evening [27, 92]. The fabric prevents the colors of an interior from fading and shields the furnishings from the damaging effects of sunshine. The fabric improves the livability and workability of a small space, lowers noise levels, and makes music and even speech richer.

In its most basic form, the processing of textiles begins with the collection of fiber. It is then followed by the conversion of fiber into yarn in a spinning mill, the fabrication of fabric, wet processing, and the production of garments in industries that manufacture garments. The cloth goes through desizing, scouring, bleaching, mercerizing, dying, printing, and finishing procedures while it is being wet-processed. In this context, the processes of desizing, scouring, bleaching, and mercerizing are referred to as pre-treatment operations of fiber [93].

#### 8.6.1 FABRIC PRODUCTION

Woven fabrics tend to be more durable. In Figure 8.4, woven fabrics are formed by intersecting two sets of yarns at right angles. These yarn sets are known as the warp and the weft. Before being turned into fabric, warp yarn goes through various preparatory operations, unlike weft yarn. Preparing the warp involves winding, warping, sizing, and drawing in. Preparation for warping involves the use of spun yarns. Preparing yarn for packaging involves winding it to achieve a specific shape and size. Yarn is supplied to the loom for the weft, and the warp yarns are processed to form a layer on the warp beam using warping. The yarn is treated with size material to enhance its strength and smoothness for the subsequent process. The warp sheet and weft are intertwined at the loom to create woven fabric [94].

Dolez et al. conducted a study wherein they categorized textile fibers into distinct classifications according to their makeup. Exploring the cellulose-based fibers category involves examining natural and regenerated cellulose fibers. The commodity synthetic fibers category includes the examination of polyolefin fibers, polyester fibers, polyamide fibers, modacrylic fibers, and nanofibers. The high-strength inorganic materials



FIGURE 8.4 Weaving patterns.

category involves the investigation of basalt fibers, carbon fibers, carbon nanofibers, carbonaceous nanomaterials, metal fibers, and boron fibers. Furthermore, the study also found high-performance polymer fibers, including aramid fibers, polyethylene, and other polymer fibers [95].

# 8.6.2 APPLICATION OF WOVEN FABRICS

Woven fabrics have a wide range of applications, such as sportswear, geotextiles, medical uses, composites, and textiles for electronics, home textiles, seatbelts, airbag construction, automobile engineering, industrial products, protection, and other similar uses [96–98]. Below are some key applications of woven fabrics.

# 8.6.2.1 Sports Applications

Woven materials are popular in sportswear. These are sportswear and gear. As shown in Figure 8.5, the woven fabrics are mostly used in athletic apparel and shoes. Generally, sportspeople use shoes, pants, and shirts for most sports [96–99]. Some athletes wear helmets or body armor. Athletic wear includes shorts, tracksuits, wet suits, salopettes, and underwear.

# 8.6.2.2 Automotive Applications

Figure 8.6 depicts interior trims, safety equipment such as seatbelts and airbags, carpets, filters, battery separators, hood liners, and hoses manufactured from fabrics in vehicle applications. Belt reinforcing is a popular use for fabrics [100].



FIGURE 8.5 Woven fabrics in sports textiles.



FIGURE 8.6 Woven fabrics in automotive applications.

# 8.6.2.3 Electronics Applications

E-textile technology enables developing inexpensive, large-scale computing devices and wearable technologies. The development of rechargeable, mechanically active, and conductive fibers for e-textiles is currently underway [101].

# 8.6.2.4 Medical Applications

Woven textiles are employed for medical devices that require exceptional stability, resistance to multiple loading cycles, or precise porosity to facilitate air or fluid circulation. Medical textiles have applications in various fields, including permanent body implant prostheses, scaffolds for tissue culture, and clinical and sanitary applications [102, 103]. Figure 8.7 depicts the use of textiles in many significant medicinal applications.

# 8.6.2.5 Filter Applications

The quality of woven fabrics now impacts filter properties in various ways [104]. Surface treatments alter the performance of woven fabric to address particular requirements, enhancing the filter's effectiveness in a particular environment.



FIGURE 8.7 Textiles in medical applications.

# 8.6.3 FASHION BRANDS

Many well-known fashion labels and retailers have expanded their product selections to include items made from natural fibers. The following section will look over a few of them.

# 8.6.3.1 Bamboo

Using bamboo in clothes production signifies a notable transition within the fashion sector toward adopting environmentally sensitive methodologies. The combination of its ecological credentials, comfort, and utility renders it a favorable option for individuals seeking to enact positive change through their fashion selections. Some of the famous brands are Mary Young, YALA, Boody, Weare Thought, Tasc Performance, Free Fly, BAM, Asquith, Etsy, Encircled, Diane Kennedy, Posh Peanut, Terrera, Cariloha, Mr. Davis, and Nancy Dee [105].

# 8.6.3.2 Cotton

Organic cotton fibers are used in a wide range of personal hygiene products, such as sanitary products, cotton puffs, ear swabs, makeup remover pads, textiles, home furnishings, children's products, and various fashion items. Organic cotton farming involves growing cotton without pesticides, fertilizers, or chemicals, guaranteeing a chemical-free process. India is the foremost global producer of organic cotton fibers, accounting for approximately 47 % of the total production. Following India, China contributes 21 %, while Kyrgyzstan accounts for 12 % of the global production. According to a 2014 study by Textile Exchange, Indian fashion retailers anticipate an increase in demand by 2025 for various product categories like denim, T-shirts, slacks, shirtings, bedsheets, towels, and other home furnishing materials. Many well-known global apparel brands have integrated this type of cotton into their products.

### 8.6.3.3 Flax

The fibrous material used to make linen fabric is obtained from the stems of the flax plant. Archaeologists and paleobiologists found flax fibers during excavation attempts, indicating their potential use in making ropes or strings due to their twisted structure. The maturation period for flax, spanning from the first planting of seeds to the final harvest, typically encompasses a duration of approximately 90 days. Cultivating flax plants poses considerable challenges, requiring substantial time and labor investments. The primary factors contributing to the higher cost of linen fabric are the challenges associated with its harvesting process. Some of the well-known brands that offer linen clothes include Whimsy & Row, Velvety, tentree, Linen Fox, Not Perfect Linen, MATE, AmourLinen, OhSevenDays, Baltic Linen Art, Reformation, Magic Linen, Beaumont Organic, Nomi Designs, LoveAndConfuse, Linen Handmade Studio, Neu Nomads, and EILEEN FISHER [106].

# 8.6.3.4 Hemp

Hemp, originating from the cannabis sativa plant, is a versatile bast fiber with numerous industrial uses [107]. With over 25,000 potential uses, various components of the hemp plant, such as its fibers, can be employed in the production of diverse products, including clothing, biofuel, bricks, paper, and textiles. Given the plant's capacity for sustainable development and the breathable, anti-microbial, UV-protective, and durable properties of hemp fabric, it can be argued that garments crafted from hemp exhibit many highly favorable attributes. However, the market for hemp fashion remains rather specialized, making it challenging to identify reliable sources for purchasing sustainable hemp apparel. Several popular manufacturers that specialize in hemp apparel include VALANI, Rawganique, BackBeat Co, Toad&Co, Patagonia, Tentree, Thought, Eileen Fisher, WAMA, Vege Threads, and LANIUS.

# 8.6.3.5 Silk

Silk is a form of protein fiber that occurs naturally and can come in numerous variants, some of which are appropriate for manufacturing textiles through weaving. Silk is derived from the cocoons of larvae from the mulberry silkworm species Bombyx mori. These larvae are purposefully cultivated and raised in controlled circumstances to produce silk, which is a material that is well-known around the world. Labera Silk, Emsilk Design, The Serenity Wear, Chaandri, Kew Pure Silk, Aphea Fashion, Felice Donna, Secret Garden By Tina, Fayna Lingerie, Eleganza Home, Brooklyn Silk Lodge, GY Studios, Oliva Oceana, LelaSilk, Oceana Linen House, and More Sunday are among the well-known silk garment companies [108].

### 8.6.3.6 Wool

Wool clothing is an environmentally friendly option because it is both biodegradable and made from fibers that can be replenished. It is resilient in addition to being versatile. It is manufactured by firms like Ibex, Duckworth, Smartwool, Icebreaker, Smitten Merino, Minus33, Woolly, Unbound Merino, Wool &, and WoolX, and it is sold in a wide range of different types of clothing, including everything from socks to hats [109].

### 8.6.3.7 Jute

The jute plant fibers are made up of cellulose and lignin. This material is frequently used in the manufacturing of various items, such as bags, ropes, upholstery, carpet, canvas, sweaters, cardigans, rugs, linoleum backing, curtains, ghillie suits, agricultural erosion prevention, and sapling bags.

# 8.7 WORLD NATURAL FIBER PRODUCTION 2023

The Discover Natural Fibers Initiative (DNFI) and the Food and Agriculture Organization (FAO)of the United Nations collected 2023 world natural fiber production data, as shown in Figure 8.8. DNFI promotes the common interests of all natural fibers in competition with oil-based and wood-based artificial fibers by sharing information and experiences. Global natural fiber production is expected to reach 32.4 million tonnes in 2023. Due to an unexpectedly large fall in the cotton production estimate, the projection is 600,000 tonnes lower than the July forecast. Cotton and wool production forecasts for 2023 are provided by the United States Department of Agriculture (USDA), Cotton Outlook, and the International Wool Textile Organisation (IWTO). Philippine monthly data for the first seven months is used to anticipate abaca production for 2023. Jute production is estimated from Bangladesh and India crop reports. USDA predicts a 3.5% drop in cotton production to 24.8 million tonnes in 2023-24. Cotton Outlook predicts 25.4 million tonnes of cotton production in 2023–24, 600,000 more than the USDA. During this season, forecasting agencies often disagree. USDA estimated the 2023-24 U.S. cotton crop at 3.05 million tonnes in its August crop report, the first of the season based on an objective yield survey involving trained enumerators visiting randomly selected fields to measure crop development. The August estimate is 550,000 tonnes lower than the July analysts' trend-based area and yield predictions. USDA's revised forecast is 3% lower than last season's 3.15 million tonnes and the weakest crop in 8 years. The 2023–24 U.S. cotton yield is expected to be 873 kgs per hectare, 100 kgs below the 3-year average. In August, the Australian Wool Production Forecasting Committee confirmed its April forecast of 216,000 tonnes of shorn wool production for the 2022–23 season (July-June), 2.9% higher than 2021/22. An unprecedented 3.02 kgs of pure wool per head was produced by abundant pasture. Shaving sheep is



**FIGURE 8.8** World natural fiber production (Estimated up to 2023) (Values in Metric Tonnes).

#### Source: World Natural Fiber Production, Sep 2023 [110].

anticipated to climb 0.8% to 72.1 million in 2023–24. If the yield of shorn fiber per head returns to average, 2023–24 Australian wool production could drop 1% [110].

Australian Wool International (AWI) sponsors hands-on in-shed training for shearers and wool handlers to attract new workers and improve productivity, skills, and professionalism. Australian sheep are predicted to reach over 72 million in 2023, and a good shearer can handle 150 heads daily. As of August 13, the Bangladesh Department of Agriculture and Extension reported 73% jute harvest completion. Jute was planted on 745,000 hectares in 2023, up from 729,000 in 2022. Bangladesh is building the world's largest jute mill for US\$360 million. World mohair production fell from 22,000 tonnes in 1990 to 4,000 tonnes in 2022. Prices have not been high enough to support mohair production, and angora goat rearing is more laborious than sheep farming. Coir fiber, derived from coconut husks' fibrous outer layer, is multifunctional. Coir fiber's free-onboard-ship (fob) price in Indonesia fell from US\$250/MT in February 2022 to US\$90/MT in February 2023. The Chinese market is crucial to the coir fiber sector, but Covid shutdowns in 2022 reduced demand. India, Indonesia, and Sri Lanka exported the most coir goods in 2022 [110].

#### 8.8 ENVIRONMENTAL SUSTAINABILITY

Numerous types of plant fibers, such as cotton or jute, possess the characteristic of being biodegradable. This implies that upon reaching the end of their functional lifespan, these items have the ability to undergo natural decomposition, hence reducing the strain on landfills and mitigating long-term environmental contamination. Plant fibers are environmentally friendly and derived from sustainable sources like cotton plants, flax, hemp, or bamboo. These agricultural commodities can be renewed on a seasonal basis, rendering them viable options for the sustainable manufacture of textiles and materials. Sustainable agricultural methods employed in the development of fiber-producing plants, such as organic farming and rain-fed techniques, have the potential to mitigate water consumption in contrast to water-intensive crops like cotton. The use of water-efficient agricultural practices plays a vital role in the preservation of freshwater supplies. Sustainable agricultural practices frequently encompass implementing strategies that entail diminished utilization of pesticides and fertilizers, resulting in a lower flow of chemical substances into ecosystems and water reservoirs. Organic gardening emphasizes employing natural methods for pest control and promoting soil health. Opting for plant-based fibers instead of synthetic alternatives can contribute to preserving biodiversity by mitigating the need for petrochemicalderived materials. The manufacture of these minerals is frequently linked to habitat damage and pollution [54].

Certain plant species, such as hemp and bamboo, possess the capacity to sequester carbon dioxide from the environment during their growth stages, mostly due to their fiber-producing nature. This can help reduce climate change. The use of closed-loop technologies within the textile production sector, which involves the recycling of water and chemicals while minimizing waste generation, has the potential to significantly augment environmental sustainability within the industry. The investigation of biotechnology applications has the potential to result in the creation of genetically modified plant varieties that exhibit enhanced sustainability and produce higher-quality fibers while requiring fewer resources. Thorough life cycle analyses of plant-based fiber products are essential for identifying areas for enhancement and reducing their environmental impact. Promoting the adoption of eco-labels and certifications for products derived from natural-based plant fibers can facilitate informed decision-making among customers and foster the adoption of environmentally conscious practices by producers.

Implementing plant-based fibers in long-lasting products and encouraging textile waste recycling and repurposing could significantly reduce the environmental impacts linked to the textile sector. To summarise, integrating plant fibers obtained from natural sources into textiles and diverse other applications is closely linked to ecological sustainability. Through the deliberate selection of these resources, the implementation of sustainable agricultural and manufacturing techniques, and the comprehensive consideration of the full life cycle of products, it becomes feasible to diminish the ecological impact of the industry and actively contribute to advancing a more sustainable future.

ISO 14001 and the Ecomanagement and Audit Scheme are widely recognized environmental management systems (EMS) that incorporate environmental considerations into company operations. EMS can be formal, standardized, and publicly accepted, or it can be an internal business environmental policy less advanced than ISO 14001. Resource-constrained small and medium enterprises face particular challenges in this regard. Discussing EMS and corporate environmental policy can assist companies in conveying their environmental and social responsibilities to stakeholders [111–113].

#### 8.9 CHALLENGES AND FUTURE DIRECTIONS

Despite their many benefits, natural fibers are subject to the difficulties listed below. Because of these challenges, continuous research and innovative thinking are required.

- Wrinkling: These fibers are prone to wrinkling and may need to be ironed or steamed to regain their original form.
- Shrinkage: Changes in the fit and shape of clothing and other products created from natural fibers can be caused by a phenomenon known as shrinkage. Natural fibers can experience shrinkage when they are subjected to heat or moisture.
- **Cost**: Cultivating and collecting natural fibers makes them significantly more expensive than their synthetic counterparts.
- **Pilling**: After continuous use and washing, a fabric made from natural fibers may pill, forming little balls on the fabric's surface.

Look into and create more eco-friendly and effective ways to extract natural plant fibers. This can involve developing new enzymatic or mechanical techniques to use less energy and produce less waste. Investigate the potential for merging various plant fibers with other eco-friendly materials (such as bio-plastics) to produce unique composite materials with improved features like greater durability or fire resistance. Explore the potential of nanotechnology in enhancing and modifying natural plant fibers to increase their versatility and suitability for various applications. Explore methods for extracting fibers from agricultural and industrial waste, like crop wastes and byproducts, as discussed in various studies [114–117]. This can potentially reduce waste and enhance the sustainability of fiber manufacturing.

To handle the disposal of plant-based textiles at the end of their useful lives, look at biodegradable treatments. Look into the breakdown of these materials and their interactions with the environment. Investigate new markets and uses for plant-based fibers, such as in the construction industry (for sustainable building materials), the automotive industry (for interior components), and the medical textile sector (for biodegradable wound dressings).

Conduct thorough life cycle analyses of items made from plant fibers to determine how much of an environmental impact they have, taking into account things like water use, energy use, and carbon footprint. Improvements should be guided by LCA data [118, 119]. To assure product quality and environmental responsibility, work toward creating and implementing international standards and certifications for sustainable plant-based fibers.

Spend money on educational efforts to inform consumers about the advantages of purchasing goods made from plant-based fibers. Stress the benefits of using environmentally and socially responsible textiles and materials. To promote innovation and develop a more comprehensive strategy for sustainable fiber consumption, encourage cooperation between researchers, companies, and policymakers. Explore circular economy models for products made of plant-based fibers, considering recycling and upcycling opportunities to extend their lifespan and reduce waste [114–118]. Using plant fibers for various applications should align with environmental, economic, and social sustainability goals.

#### 8.10 CONCLUSION

Natural-based plant fibers' eco-friendliness, adaptability, and environmental benefits make them an attractive and sustainable alternative to petroleum-based materials. This is because natural-based plant fibers are derived from plants. Plant fibers derived from natural sources offer a potentially fruitful route toward a more sustainable and environmentally conscientious future. Concerns about the environment and the depletion of resources are continuing to grow on a worldwide scale. The entire potential of these materials can only be realized via the coordinated efforts of scientists, business leaders, and government officials. Working together, we can make the planet a healthier, more sustainable place to live.

#### DECLARATION

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper. Generative AI and AI-assisted technologies have been used in the writing process to improve the readability and language of the work only. After using this tool, the authors reviewed and edited the content as needed and take full responsibility for the publication's content.

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# 9 Nanocellulose Fibers and Their Potential in Textile Applications

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#### 9.1 INTRODUCTION

The growing environmental issues caused by the overuse of non-green materials have fueled studies into degradable, feasible, and continuous innovations. Human practices such as extensive use of natural resources, fossil fuels, and infinite production waste for industries have significant environmental consequences. The demand for products for human consumption is increasing every year. The global market for textile fiber was valued at 75.5 million tonnes globally in 2010, and it is projected to grow to 133.5 million tonnes over the next 20 years [1]. The products from textiles were also discarded into the surroundings. The majority of waste generated from fibers disintegrates slowly, up to 200 years for the decomposition process to occur. Commonly, waste from cotton is the main contributor to ecological pollution and textile waste [2]. Because of their diffusion properties, non-biodegradable polymers persist longer in the environment, boosting solid garbage. Thus, hazardous compounds are discharged into the surroundings. Besides, due to the high request for plastics, the growing interest in fossil fuels enhances carbon release. Therefore, Initiatives to generate biodegradable polymers, also called biopolymers, from various natural resources are expanding to reduce the ecological harm of traditional polymer disposal [3].

Naturally occurring cellulose polymer has desirable properties, including sustainability, relatively cost-effectiveness, biocompatibility, and biodegradability. It is abundant in lignocellulosic biomass, including forest wastes, agriculture residues, and energy crops [4]. Many studies are being conducted on cellulose in combination with applications in various sustainable goods. Conventional study practices and numerous industrial uses are being changed because of the progress of the development of nanotechnology. It is possible to tune their characteristics by reducing the size of the polymer chain. The difference in size changes from micro- to nanometre, from cellulose to nanocellulose fibers. Various characterization methods are available to obtain the numerous properties of these materials. Besides its non-toxic, biodegradable, and low-density properties, nanocellulose also possesses advanced characteristics, including a reinforcement agent, good thermal and mechanical stability, and durability [5].

#### 9.2 NATURAL FIBER AND NANOCELLULOSE

Natural fibers, primarily composed of hemicellulose, cellulose, and lignin, have a highly complex chemical composition and structure. Cellulose is a continuous homopolymer comprising (1-4)-glycosidic linkages connecting d-glucose units, as depicted in Figure 9.1 [6, 7]. The lignin and hemicellulose are embedded in the matrix. Cellulose is the crystalline part of natural fiber, while hemicellulose and lignin are amorphous parts. Crystalline content is present at about 35–50 wt% of the dry weight of biomass, but hemicellulose and lignin are in lower amounts [8]. Nanocellulose, obtained from cellulosic fiber as fibers and crystals with diameters of less than 100 nm, has gained significant attention [9].

Several factors are driving the demand for nanocellulose in multiple fields [10]:

- Nanocellulose is composed of multiple levels of three-dimensional (3D) arrangements.
- There is growing interest in its chemical and physical characteristics, which provide hydrophilic properties and allow for modification and functionalization through diverse techniques.
- Nanocellulose exhibits superior properties of nanomaterials, such as good mechanical strength, a large surface area, and a low thermal expansion coefficient.

Conventionally, cotton and wood are the primary sources of cellulose, used in the textile sector for manufacturing products and as sources of heat and building materials [11]. However, given the declining demand for traditional paper products, developing innovative uses and marketplaces for cellulose is critical to rekindling interest in this material. Concurrently, a global trend toward sustainable development and the bioeconomy promotes the application of natural materials, like cellulose nano-structures, to replace conventional oil-based materials to create innovative goods



**FIGURE 9.1** The repeating unit of (1-4)-glycosidic linkages connecting d-glucose. (Permission from Sato et al. [7].)



**FIGURE 9.2** Morphologies of nanocellulose: CNF, CNC, and BNC. (Permission from Norrrahim et al. [14].)

while minimizing their negative environmental effects. There has been a spike in interest presently in nanocellulose. Nowadays, hundreds of tonnes of nanocellulose are manufactured industrially each year. Nanocellulose is cellulose with at least one dimension less than 100 nm. Nanocellulose is classified into three types [12]:

- Cellulose nanocrystals (CNC), also known as nanocrystalline cellulose, cellulose (nano) whiskers, or rod-like cellulose nanocrystals.
- Cellulose nanofibrils (CNF), which include nanofibrillated cellulose (NFC), microfibrillated cellulose (MFC), or cellulose nanofibers.
- Bacterial nanocellulose (BNC), also known as microbial nanocellulose.

Figure 9.2 shows the morphology of all three nanocelluloses. Nanocellulose fibers are long, flexible fibers with a micrometer-long length and a nanometer-wide width that are often utilized to manufacture cellulose membranes used as filler materials or in applications that combine additional fibers [13].

# 9.2.1 CELLULOSE NANOCRYSTALS (CNC)

Acid hydrolysis is a common method for forming cellulose nanocrystals (CNC). CNC is less flexible, cylindrical, elongated, and rod-shaped, having 100-6,000 nm and 4-70 nm of length and width, respectively, with 54-88% of crystallinity index. Nowadays, sulfuric acid is still considered an excellent technique for preparing CNC [15]. However, recently, CNC has been produced using a variety of types of acids like hydrobromic acid, hydrochloric, phosphoric, and hydrochloric acid. Moreover, combinations of organic and inorganic acids have also been utilized in CNC manufacturing [16–19]. Figure 9.3 illustrates the amorphous cellulose chain area reduced by acid hydrolysis from cells of a rod structure [20]. Utilizing hydrochloric acid yields CNC exhibiting higher thermal stability compared with sulphuric acid. However, because there was insufficient electrostatic repulsion between the crystals, agglomeration and a less stable aqueous solution occurred in the case of using hydrochloric acid [21]. Thus, the sources of cellulose and reaction parameters contributed to determining the final properties of CNC. For example, these conditions affect crystallinity, density, mechanical characteristics, shape, and thermal stability. Several methods for acquiring CNC have been recorded, including mechanical, chemical, biological, and



**FIGURE 9.3** Schematic of CNC from acid hydrolysis. (Reprinted with permission from Aravind et al. [20].)

combination methods [22, 23]. Surprisingly, combination techniques tend to represent an appealing alternative, since these innovations include various strategies to address the shortcomings of separate procedures by improving CNC features, increasing outputs, and reducing expenses. CNC is not as flexible as CNF because of the synthesis technique; they are formed through acid hydrolysis (or enzymatic), which eliminates amorphous sections of cellulose; hence, they are less flexible than CNF [24].

#### 9.2.2 CELLULOSE NANOFIBRILS (CNF)

Cellulose nanofibril (CNF) contains amorphous and crystalline regions. The length and diameter are in micron scale and 3mm, respectively [25]. CNF has several distinguishing characteristics, including excellent mechanical characteristics, ecological sustainability, a relatively small coefficient of thermal expansion, and an affordable price. While CNF obtained from plants typically contains impurities like lignin, hemicellulose, pectin, and other things, CNF recovered from bacteria is quite clean and exhibits longer chains [26].

CNF is typically produced through mechanical disintegration and chemical, biological, and chemical-biological processing. However, their considerable energy consumption constrains the use of mechanical technologies. Thus, to save energy costs, biological or chemical pretreatments are used to facilitate subsequent mechanical processes [27]. The enormous energy consumption imposed by the mechanical disintegration process in earlier decades is a barrier to isolating CNF. CNF, however, progressively began to attract much attention for manufacturing purposes after it was discovered that pretreatment techniques, such as 2,2,6,6-tetramethylpiperidine-N-ox yl (TEMPO)-mediated oxidation or enzymatic hydrolysis, had a significant impact on the mechanical disintegration process [11].

#### 9.2.3 BACTERIAL NANOCELLULOSE

The structure of bacterial nanocellulose (BNC) differs from that of plant cellulose, given that it is a three-dimensional (3D) porous network. BNC possesses excellent

levels of purity, high crystallinity (up to 80%), great thermal stability, strong levels of polymerization, and high levels of flexibility and 118 GPa for Young's modulus. Furthermore, BNC nanofibers have a surface area greater than plant cellulose and an outstanding aspect ratio [28].

Bacterium, including *Sarcina ventriculi* and *Acetobacter*, as well as *agrobacterium*, *alcaligenes*, *rhizobium*, *pseudomonas*, *salmonella*, and other species, are fermented to generate BNC. *Acetobacters*, including *A. pasteurianus*, *K europaeus*, *A. hansenii*, and *A. xylinum*, are the most significantly effective producers of bacterial cellulose [29]. Three cultivation techniques are often used in the fermentation process to manufacture BNC, such as agitation, static, and shaking cultivations. BNC is first synthesized by cultivating a bacterial strain in a medium rich in nutrients that comprises carbon, nitrogen, and other components [30]. Moreover, BNC is very suitable for cellular adhesion and binding due to its structural similarity to the collagenous cell protein. Further, its safety in practical usage contributed to extensive applications [31].

#### 9.3 NANOCELLULOSE FIBERS IN TEXTILE APPLICATIONS

Natural fiber-based textiles have been essential to humanity since the beginning of time, and they continue to be employed extensively in the current textile industries due to their distinctive features as premium fibers for fabric. Natural fibers with shorter strands cannot be used to make yarn because of the diversity in staple length. Due to this, natural fibers like cotton, wool, hemp, or silk are wasted during manufacturing and end applications. These fibers have good inherent qualities, which make new findings for them highly marketable [32]. Tailored nanoparticles have enabled the production of a new wave of environmentally friendly textile products. Nanotechnology has significantly enhanced the fibers used in the textile industry, such as nanocellulose, which combines cheap cost, lightweight, electric conductivity, eco-friendly materials, and high resistance, allowing an incredibly broad range of potential uses [33]. Textile products have the potential to enhance professional and consumer uses with the incorporation of nanotechnologies. Nanomaterials included within the polymer matrix or deposited onto the fiber's outermost layer can improve textiles' quality for both specialized and general applications. The high concentration of hydroxyl groups in nanocellulose particle structure makes it possible to reconstruct textile surfaces, and nanocellulose fibers are also resistant to climate variations [34].

#### 9.3.1 TEXTILE COATING

In industry, coatings are applied to various materials, including textile, glass, metal, concrete, paper, and furniture. Global demand for inexpensive coating materials has increased since they provide the finish producers with greater value and enhance their profit margins. The need to replace plastic coatings with environmentally friendly options is growing along with the demands that coatings must meet in terms of quality and performance criteria. Globally, there is an increasing need for textile coating materials. To maintain hygiene and public health, it is vital to specify qualities such

as fire retardant properties, breathability, and water resistance to combat the germs and pathogenic organisms on the surfaces of fabrics [35].

The natural or synthetic fiber required to be coated depends on the varying requirements for the chemical functionalization of nanocellulose. For this reason, nanocellulose's surface functionalization must offer the required adherence to the substrate. Numerous textile materials, including nylon, cotton, cotton, polyester, polyamide, polyethylene, cotton, and silk, have been used in textile coatings containing cellulose. Saremi et al. claim enhanced surface coating is achieved for textiles made of cellulose, polyester, and nylon, as well as polymer films when nanocellulose is crosslinked with polycarboxylic acids. Compared to CNF, the CNC coating's nanocellulose showed greater adherence due to fabric structure. High porosity and minimal density were also essential components for a coating's strong adherence [36]. Chattopadhyay and Patel introduced CNC into the polyester fabric in a project. They came to the conclusion that CNC increased absorbency, decreased water and air permeability of polyester fabric, strengthened the staining endurance, and enhanced the rapidity against soaping [32]. Furthermore, according to Nourbakhsh's research, laser-treated polyester and nylon fabrics with nanocellulose coatings demonstrated greater dye absorption [37]. According to Rai et al., adding CNF to natural indigo dye has numerous benefits, including reducing prolonged dipping and conserving water and other resources by avoiding the consumption of harmful chemicals. On the fabrics, they applied hydrogel fabricated from CNF that was combined with chitosan and natural indigo particles. The observations revealed that the weight growth, bending stiffness, and air permeability remained unchanged. Through physical crosslinking, chitosan improved the coating's adherence and attachment to fabrics [38].

#### 9.3.2 CANVAS CONSOLIDANTS

Given the potential risks connected to the poor reversibility and degradation of adhesives that are currently in use, the investigation and analysis of novel approaches to structural painting consolidation are crucial. A consolidant replacement is currently required, and it must exhibit the features of reversibility, stability, compatibility, userfriendliness, and non-hazardousness. These attempts include using a highly volatile or small quantities of solvents, achieving a high stabilization of the canvas, and stabilization film elasticity and response to humidity changes that need to be similar to or even less than those from the canvas in order to avoid any tension [39]. Cellulosic artworks that have been subjected to acid and have been severely damaged typically need to be deacidified to stop future cellulose depolymerization, as well as consolidate to increase their mechanical strength. Older artifacts are frequently highly delicate, so it is best to avoid using two independent conservation techniques for them in order to minimize the dangers and strains on the original materials [40]. In contrast to the frequently used vinyl and acrylic-based resins, nanocellulose's use in preservation has the specific advantage of allowing for the use of a substance closely related to the canvas and, thus, more compatible with it. Clusters of cellulose chains in the nanometer range make up nanocellulose. This nanomaterial is able to form a stronger bond with treated canvas substrate, resulting in that the treatment and treated material share similarities in chemical nature [41]. Furthermore, consolidants made of nanocellulose act as shielding for artistic substrate by avoiding contact with external substances that can harm the original artworks. The ability to reverse these restoration tactics is an additional great advantage [42].

Recent research has focused on using nanocellulose as consolidants for canvases because it is completely compatible with cellulose-based works of art. Known as a suitable material that can reinforce delicate canvases without needing a liner, this material can strengthen delicate canvases when used as a consolidant. Bridarolli et al. enhanced the binding between CNF and canvas by adding an intermediate layer, such as a cationic polymer. Even though the amount of CNF applied is minimal, the strong mechanical characteristics of CNF and the substantial adhesion that enhances the effect of PAAE make this multilavered treatment an effective reinforcement technique [43]. In another study, nanocellulose was used by Nechyporchuk et al. as canvas consolidants. They used CNC and CNF carboxymethylated (CCNF) as bio-based substitutes for other conventional materials. Comparing the three, CNF outperformed CNC and CCNF. However, in the region of interest for elongation, three nanocelluloses showed better consolidation than lining with synthetic adhesive (Beva 371) and linen canvas [44]. Due to the damaged painting canvas's lack of mechanical integrity, Kolman and friends combined CNF and carboxymethyl cellulose silica nanoparticles. In contrast to the silica particles, which entered the canvas's insides, the CNF produced a film on the canvas's surface. Adjusting the ratio of silica particles to CNF makes it possible to adjust the formulations' reinforcing characteristics. A high CNF level in the formulation produced ductile behavior, but a high silica content produced stiffer canvas as an alternate method of canvas restoration [45].

#### 9.3.3 ANTIBACTERIAL TEXTILE

Microbial contamination of surfaces, especially textiles, is common in hospitals and other healthcare facilities because it can result in infections and subsequent crossinfections. Therefore, it is crucial to stop the development of secondary infections and dangerous bacteria inside a therapeutic setting. Infections acquired in hospitals not only delay patient recovery and increase the chance of developing serious illnesses, but they also add to the overall expenses of providing healthcare [46]. Fabrics produce an environment that encourages the growth of microorganisms. In addition to eradicating undesired bacteria and preventing the spread of diseases, textile antimicrobial finishing must also fulfill three more fundamental needs, which could be listed as follows: (1) compatibility: the antimicrobial finishing agent should not adversely affect the fabric's characteristics or look must be appropriate for industrial textile production, (2) safety: the product cannot be hazardous to people, and its use on textiles cannot irritate or create skin allergies and (3) durability: the reagent must withstand washing, drying, and leaching [47]. Compared to synthetic fibers, cellulose is more prone to bacterial colonization because the structure is more porous and hydrophilic, maintaining a quantity of water, and quick food and gas transmission. This offers the ideal medium for bacterial growth. The many advantageous features of cellulose fibers, such as breathability, comfort, hydrophilicity, biodegradability, softness, mildness, ecologically friendly, and others, make them popular in healthcare and hygiene products. As a result, it is vital to modify textile materials, particularly cellulose fibers, with antimicrobial chemicals [48]. As an antibacterial substance for germs, nanocellulose can be an excellent contender because of its well-known adaptability and great properties [14].

The fabric for antimicrobial purposes was created by first pre-treating surface polyester fabric (PE) with a suspension of nanocellulose and cetyl-trimethyl ammonium bromide (NCs@CTAB-NCC), followed by dipping PE in a suspension of graphene oxide-based silver nanoparticles (AG) to produce PE decorated with graphene oxide-based silver nanoparticles (AG/NCC-PE). After that, PE was further manufactured using a chemical reduction with vitamin C (LAA) to create silver nanoparticles based on reduced graphene oxide (ARG/NCC-PE). Regarding two strains of *S.aureus* and *E.coli*, the textiles showed no appreciable modifications in their antibacterial efficacy. The product offers promise for producing durable, antibacterial hydrophobic fabrics that are mechanically stable and long-lastingly safe for human use in medicine, disease prevention, and personal protective equipment [49].

#### 9.3.4 MILITARY TEXTILES

One of the industries that uses the most textiles and has the most unique and important needs is the military. The overall criteria for military protective textiles are stringent and varied because they are frequently exposed to unanticipated situations on short notice [50]. It might be difficult to choose and design textiles for use in making military equipment. Some critical considerations need to be considered for such an ecosystem, such as heat endurance and excellent longevity, water repellence of moisture and durability against tears and breaks, ballistic injury protection, and durability against chemical and biological dangers. The kinds of textiles employed by the armed forces include combat outfits, shelters, parachutes, sails, safety harnesses, body armor, explosive ordnance disposal bomb suits, sandbags, boots, camouflage patterns, and other products [14]. Additionally, nanocellulose-embroidered clothes can shield military personnel from harmful chemicals, noxious fumes, and viruses. Additionally, technology might make it possible to continuously monitor body functions. Research of exceptionally tough nanocomposite materials presents excellent opportunities for textile and military aerospace materials. Wet-drawing and ionic cross-linking were used in a study by Xi et al. to create strong and durable waterborne polyurethanes-TEMPO oxide CNF (WPU-TOCNF) nanocomposites. The resultant fiber and film have strength, extension at the break, and toughness that are precisely controlled to achieve 715 MPa (323 MPa), 41% (52%), and 186 MJ m3 (110 MJ m3), respectively. Ionic cross-linking between soft (WPU) and rigid building blocks (TOCNF) also increased the strength, extension at the break, and toughness of the obtained fiber and film. Out of all the materials studied, this one has the highest toughness made of cellulose [51].

#### 9.4 CONCLUSION

Cellulose is currently of considerable interest in producing renewable and sustainable products. Nanocellulose, a green material with outstanding features, can substitute for non-biobased materials like plastics. Extracted through various techniques, nanocellulose is a promising area of current and future research, particularly for the textile sector. Although numerous studies have demonstrated nanocellulose as a successful new green biobased material for textiles, further advancements, and research are needed for future applications.

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# 10 Nanocellulose and Its Emerging Applications in Textile Processing

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#### **10.1 INTRODUCTION**

Textile-based materials, such as fibers, yarns, and fabrics, are intricate manufactured products with a long historical record. As far as the Industrial Revolution is concerned, the textile industry has become one of the most polluting organizations in all industrial sectors. The Earth's population rose fourfold throughout the twentieth century, but the consumption of textile fibers increased twentyfold (Rai et al., 2021). The exponential increase in the human population has increased the need for clothes, which has increased the demand for textile substrates (Saremi et al., 2020). The wet processing of textiles produces a lot of wastewater throughout the dyeing, printing, washing, and finishing operations (Kim et al., 2017).

The existing textile wet processing industry uses loads of water and discharges harmful untreatable effluents into the environment, including salts, hydroxides, and unreacted colors. Hence, wastewater containing harmful pollutants must be carefully treated before discharge; reducing the number of contaminants before release is preferable. Increased use of these chemicals in the textile industries harms the environment and people directly and indirectly involved with the processing sector (Kabir et al., 2019; Panda et al., 2021). Reactive dyes are among the most widely used synthetic colorants for cotton fibers, among numerous other dyes. Reactive dyes have diverse color palettes, stability, and excellent dry/wet fastness properties. However, significant amounts of salt, greater than 20 gpL, are required in reactive dyeing to reduce the zeta potential of fiber. As a result, textile effluent has a significant environmental impact due to the high quantities of salt used. This problem worsens as the total dissolved solids (TDS) regulation in effluents tightens (Lewis, 2014).

Recent attempts for environmentally friendly textile wet processes include effluent treatment with activated carbon membranes,  $H_2O_2$  removal, enzymatic scouring, and ultrasonic wet processing, resulting in less energy and water consumption (Lara et al., 2022; Madhu and Chakraborty, 2017). Several drawbacks, such as the requirement for significant capital expenditures, scalability, reproducibility, and limited availability,

still constrain the widespread utilization of novel technologies in an industrial environment. To adopt a sustainable textile process, the critical areas of textile wet processing, like pretreatments, dyeing, printing, finishing, etc., should create a minimum pollution load with simultaneous minimum consumption of resources like water, energy, etc., without hazardous chemicals. Therefore, there is still a need to develop more sustainable processes that significantly decrease the expenses associated with the economy and the environment (Azanaw et al., 2022; Parisi et al., 2015).

Cellulose biomass is one of the most abundant and renewable natural biomasses, non-toxic, sustainable, and eco-friendly. Nanocellulose (NC) is a designed nanostructured cellulose that is classified on the manufacturing process or particle size as nanofibrillated cellulose (NFC), nanocrystalline cellulose (NCC), or bacterial nanocellulose (BNC), as discussed in Table 10.1. As shown in Table 10.2, NCs have applications in water purification, thermal insulators, membranes, biocomposites, mechanical reinforcements, flexible electronics, and food packaging because of their nontoxicity, large surface area, porous surface, and functionality caused by abundant surface hydroxyls (Li et al., 2021). NFC can be used as a coating and finishing material for textiles because of its great force of attraction for cellulosic substrates, achieved primarily by van der Waals force, mechanical interlocking, and strong hydrogen bonding (Reshmy et al., 2020). Herein, we present sustainable textile wet processing approaches based on nanocellulose that would significantly minimize chemicals and water usage in textile dyeing.



FIGURE 10.1 Applications of nanocellulose in developing sustainable textiles.

# TABLE 10.1Techniques of Extraction for Nanocellulose Production (Reshmy et al., 2020)

Sources	Treatments	Technique of Extraction	Nanocellulose Types
Wood pulp, agro biomass,	Surface modification	TEMPO-mediated oxidation,	CNFs
grasses		Deep eutectic solvents, Ammonium persulfate	CNCs
Wood biomass and other lignocellulosic fiber sources	Mechanical process	Cryocrushing, High intensity ultrasonication Twin screw extrusion, Ball milling	CNFs
Bagasse, wood pulp, agro biomass, and Cotton linters	Enzyme catalyzed	Static culture	CNCs CNFs
Wood pulps (Softwood and hardwood), agro biomass	Green strategies	Using phosphotungstic acid Organolic strategy Ionic liquid and solvents	CNCs
Wood pulp, agro biomass, paper industry	Pressure induces methods	Steam explosion High pressure homogenization Micro fluidization	CNCs

# TABLE 10.2Applications of Nanocellulose

Application

areas	Properties	End Uses	References
Food Packaging	Improved barrier, rigid, resilient	Films in Packaging	Jiang et al. (2021), Kroyer (1995)
Biomedical	Biodegradability, excellent biocompatibility and non-hazardous	Scaffolds, absorbing pads, antimicrobial Films, Sanitary napkins, bandages, wound dressing	Biranje et al. (2023), Xue et al. (2017)
Cosmetics	Durability, functionality, biocompatibility, good elasticity	Cosmetic coating agents for nails, hair, or eyelashes	Ferreira et al. (2020), Meftahi et al. (2022)
Electronics	Compatible, durable, excellent mechanical, and high dielectric	Electronic displays, sensors, and windows	Johar et al. (2012)
Optical materials	Resilience, strength, compatibility, crystalline	Sensors, Electronic transistors	Sezali et al. (2021)
Automobile	Good electrical, magnetic, thermal, physicochemical properties	Lightweight and light strength component	Sezali et al. (2021)
Construction	Increase fracture toughness, strength, durability, low cost, low density	Block sensor to monitor the stress level	Wang et al. (2020)
Textile	Easy care, low impurity, good robustness, ecofriendly	Antimicrobial, paste printing, dyeing	Lohtander et al. (2021), Rai et al. (2021)

(Continued)

# TABLE 10.2 (CONTINUED)Applications of Nanocellulose

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areas	Properties	End Uses	References
Aerospace	High strength, lightweight	Windows, sensor	Sezali et al. (2021)
Water purification	Biosorable, cost effective, ecofriendly	Filtration	Karim et al. (2016) Khalid et al. (2021)
Paper industry	Easily available, ecofriendly	Greaseproof paper	Dhali et al. (2021), Nechyporchuk et al. (2016)

#### **10.2 NANOCELLULOSE IN TEXTILE COATING**

Nanocellulose and its modified derivatives have potential applications to replace petrochemical-based fractions for textile, paper coating, biomedical, package filler, and replacement plastic (Emin and Koyuncu, 2021). The study has been performed to impart functional properties to textile material by functionalizing nanocrystalline cellulose (NCC) using silver nanoparticles. This study extracted NCC using acid hydrolysis methodology from Sugarcane bagasse (SCB). Extracted NCC was applied onto the textile substrate via a spin coater. The application of silver nanoparticles (SNP) onto a textile substrate was previously functionalized with a known caustic solution (10% by weight). SNP was synthesized using silver nitrate (0.1 M) and aqua ammonia (28%) to form an ammonia complex of silver and then reduction followed by a known concentration of glucose (0.1 M). The efficiency of cotton fabric without treatment with NCC was also functionalized and served as a control sample. During the evaluation, it was observed that the effective loading on a textile substrate treated with NCC was greater than that of the control sample (Hasan et al., 2020). Nanofibrilated and nanocrystalline cellulose can be used as coating material on natural (cellulosic) and synthetic polymeric (Polyester and Nylon) textile substrates. In this study, the treatment with a reactive random copolymer of glycidyl methacrylate (GMA) and oligo (ethylene glycol) methacrylate (OEGMA), P (GMA OEGMA) copolymer and a polycation polymer polyethyleneimine (PEI) that can form a cross-linked network increases the coating's stability. The highest coating adhesion and stability were seen in the case of P (GMA OEGMA). Alternately, the coating is strengthened by cross-linking polycarboxylic acids with nanocellulose. NCC coatings outperform NFC coatings regarding adhesion to all substrates (Saremi et al., 2020). Cellulose nanofibrils (CNF) derived from wood as a precursor were used to make sustainable and disposable e-textiles using textile printing technology. First, the textile substrate was printed using CNF with a specific concentration (1% On weight of fabric (OWF), followed by inkjet printing using CYAN ink and a further inkjet printing technique with silver nanoparticle ink. Analytical techniques such as optical microscopy, atomic force microscopy, calorimetry, electrical signal analysis, tensile strength, and contact angle were used to analyze untreated and treated samples. The CNF-pretreated samples showed better performance as sustainable electronic textiles (Nechyporchuk et al., 2017b).

Cellulose was treated with 2,2,6,6-tetramethylpiperidine 1-oxyl radical (TEMPO) to get a derivative TEMPO-oxidized nanocellulose (TOCN). Derivative TOCN was treated with hexa decylamine (HAD) in the presence of catalysts to make a hydrophobic amide derivative. Ligation of this derivative was done with a polyisocyanate derivative in the presence of Tetrahydrofuran (a solvent exchanger) to get stable dispersion to have water-repellent and wash-sustainable coatings on fabrics (Ke et al., 2020).

#### **10.3 NANOCELLULOSE IN FUNCTIONAL FINISHING OF TEXTILES**

In pursuit of an eco-friendly UV protective finishing agent, nanocellulose was modified with chitosan (bio-resourced polysaccharide). Nanocellulose of particle sizes ranging from 100 nm, 200 nm, and 800 nm was taken for study. 5% of each nanocellulose was suspended in deionized water stirred at high-speed stirring (15,000 RPM). 0.1 gm chitosan and 2 mL acetic acid were added to this solution and heated at 50–60°C for two hours. Then, 0.1 mL tannic acid solution was added and stirred at 90°C to get a functional finishing agent, followed by application of pad-dry-cure. Investigations showed that Nanocellulose significantly impacts morphology, mechanical properties, hydrophilicity, UV protection, and launderability (Yang et al., 2020).

To impart conductive properties to textile substrates, cellulosic were functionalized by nanocellulose/polypyrrole coating. Nano-cellulose nano-whiskers (CNW) are derivatized through graft polymerization with nano carbamoyl ethylated cellulose (NCEC) copolymers and monomers of CNW-Polyacrylamide (CNW-PAAm). CNW, CNW-PAAm copolymer, and NCEC are used to mediate oxidative polymerization of polypyrrole (Ppy). Optimization regarding the concentration of pyrrole and ferric chloride was done (Hebeish et al., 2016). Under the influence of chemical oxidative polymerization of pyrrole solution employing iron chloride (III) as an oxidizing agent in the presence of cotton fibers, a set of Ppy/nanocellulose conductive composite films was created. Results showed excellent conductivity and mechanical and thermal characteristics of cotton fabrics treated individually with various nanocellulose/polypyrrole materials (Hebeish et al., 2016). Cellulose combined with lignin can be wet spun from aqueous suspensions of CaCl<sub>2</sub> to create composite filaments, followed by carbonization at 900°C to produce the corresponding carbon microfibers (CMFs), as shown in Figure 10.2a. These composite fibers can be a scalable and



**FIGURE 10.2** Conversion of TOCNF/lignin hydrogel filaments into electroconductive carbon microfibers (a), wet-spun cellulose nanofibrils with silica nanoparticles to create flame-resistant cellulosic fibers (b) (Nechyporchuk et al., 2017a) (Reprinted with permission from (Wang et al., 2019) Copyright© 2017 American Chemical Society.)

energy-efficient alternative to conductive carbon microfibers and are promising composites for fiber-shaped microelectrodes, capacitors, and textile-based wearable electronics (Wang et al., 2019).

Allicin [S-(2-propenyl)2-propene-1-sulfinothioate] obtained from garlic can be a natural antibacterial agent for functionalizing textile material. It has antibacterial activity against both gram-positive and gram-negative bacteria and also has antioxidant, anti-carcinogenic, anti-inflammatory, antigenotoxic, immunomodulatory, and anti-thrombotic properties. Allicin was conjugated with nanocellulose and applied to cotton fabric through solid covalent bonding, using 3-aminopropyl triethoxysilane (APTES). On evaluating the antimicrobial test, the fabric was found to withstand its imparted antimicrobial activity after many laundering cycles (Jafary et al., 2015).

To overcome the fouling defect of the membrane in wastewater treatment, a nanocellulose-based nanoporous thin film is fabricated by coating nanocellulose fibers on poly-acrylonitrile (ePAN). Cellulose nanofibers were produced from bleached jute fiber using TEMPO-mediated oxidation. Membranes were fabricated with various levels of oxidation (DO) and area density from cellulose nanofiber and thin-film nanofibrous composite (CNF-TFNC). The CNF-TFNC membranes' negatively charged surface allowed for self-cleaning capabilities utilizing a straightforward hydraulic flushing procedure, which might significantly increase the CNF-TFNC membrane's life span and filtering performance in real-world wastewater treatments (Yang et al., 2021).

With a carbodiimide cross-linker to conjugate cellulose nanoparticles with lysozyme and allicin independently, the particles were made and then examined for their antibacterial activity, chemical crosslinking, and surface properties. They were then compared with allicin, lysozyme, and nanocellulose alone, and their antibacterial qualities were assessed using the microdilution method. *Aspergillus niger, Staphylococcus aureus, Escherichia coli*, and standard strains of *Candida albicans* were the targets of the good antifungal and antibacterial activities of allicin- and lysozyme-conjugated nanocellulose (ACNC and LCNC, respectively). Nanocellulose itself demonstrated few antimicrobial activities. MIC50 and MIC90 values were the same for ACNC and LCNC, even though allicin and lysozyme had distinct MICs against each strain. According to the authors, textile products, food packaging, and food items can all employ ACNC and LCNC as antimicrobial agents in trade (Jebali et al., 2013).

Hexamethylenediamine (HMDA) modified cellulose nanofibrils (CNFs) are added to a ring-spun viscose yarn as an antibacterial enhancer. CNF-HMDA maximizes the spraying ability of the viscose fiber with the lowest sticking, without flowing and tearing issues during spinning. The antimicrobial properties of the yarn increased with the amount of CNF-HMDA, as evidenced by a 99 % reduction for *S. aureus* and *C. albicans* (log 1.6–2.1) in up to 3 hours of exposure at a minimum of 33 mg/g, and for *E. coli* (log 0.69–2.95) at 100 mg/g of its addition, yielding 45–21% of bactericidal efficacy. This result is linked to uniformly distributed CNF-HMDA when applied via a bi-polar nozzle with tiny (0.4 mm) holes that evaporate rapidly. It also enhances the yarn's wettability but does not lessen its tenacity or fineness. Instead, it produces a strong positive charge (0.663 mmol/g). The functionalized yarn is hydrophobically bound by the HMDA's methylene chain, which obstructs the amino groups and lessens the antibacterial activity of the yarn (Kokol et al., 2021).

Two distinct surface modification processes-plasma and laser treatment-were used on fabrics made of polyester and nylon. In both treated and untreated fabrics, polyethylene glycol was used to immobilize cellulose nanocrystals. Fabrics were examined for the surface morphology, wettability, and dyeability of samples coated and uncoated with nanocellulose. Using plasma and laser treatment, nanocellulose was applied to polyester and nylon fabrics, resulting in a coating layer on the treated fibers' surface. The surface of the fibers was coated with a cellulosic layer, erasing the grooves in the ripple-like structure of the laser-treated fibers. The results also indicated a decrease in the intensity of functional groups associated with nylon and polyester on the surface. The dyeing results of coated and uncoated fabrics are consistent with these findings. The amount of dye absorption was higher in the case of lasertreated polyester and nylon 66 fabrics coated with nanocellulose (Nourbakhsh, 2014). By mixing carbon nanotubes (CNTs) into nanocellulose, wearable sensing fibers with good mechanical qualities and sensing capabilities are fabricated. Tunicate nanocellulose (TCNF) is uniformly composited with single-walled carbon nanotubes (CNTs), enabling liquid-crystal characteristics. This process continually produces TCNF/ CNT composite fibers in aligned orientations using wet spinning. The TCNF/CNT fibers with exceptional gas (NO<sub>2</sub>) sensing capabilities demonstrate high selectivity and sensitivity. Furthermore, by employing a direct weaving technique, the TCNF/ CNT fibers can be flawlessly combined with traditional fabrics while enduring severe and complicated distortions without losing their inherent sensing capabilities. (Cho et al., 2019). Monolithic integration of a photodetector and a perovskite-based optical waveguide amplifier on a nanocellulose substrate demonstrates the feasibility of stretchable signal manipulation and a biodegradable receptor system. An integrated optical amplifier-photodetector is created using the photocurrent produced in the organic-inorganic lead halide perovskite under applied bias. By monitoring the light signal as it travels through the waveguide, this type of photocurrent has applications, such as controlling the operating temperature without significantly affecting the amplifier's performance (Suárez et al., 2018). Silver nanowires and polypyrrole conductive polymer were used as conductive doping materials in two of the samples, whereas nanofibrillated cellulose was the only material used in one sample. Fabrics that have been coated were dried and electrically characterized. The Peltier effect was illustrated, and temperature gradients and electrical currents were recorded across conductive sample pairs. The fabric's Joule heat saturation limited the Peltier effect's temperature range (Morgan and Sharma, 2018).

A solvent-free acid hydrolysis process was used to prepare phosphorylated nanocellulose (P-NC) from date palm sheath fibers. The composite treatment of jute cloth involved the application of eighteen distinct treatment methods. Various fractions of phosphorylated nanocellulose (1-4%) and chitosan (0.5-2%) were utilized to investigate the impact of treatment on the resultant jute fabric composites. The treated jute cloth showed good weight uptake, phosphorus content, and tensile qualities essential for antibacterial and thermal stability qualities on jute cloth (El-Shafei et al., 2019).

Nanocellulosic reinforcing liquors were applied in varying quantities to cotton and polyester/cotton blend fabrics. Results showed that adding nanocellulosic to cotton and blend fabric samples significantly improved the performance and tensile parameters of reinforced fabrics irrespective of the type of nanocellulose utilized; these qualities rose with increasing concentration of nanocellulose. Even at low concentrations of nanocellulose, the treated textile material showed good shielding properties against high-energy electron beams because of nanocellulosic reinforcement (Hebeish et al., 2018).

A versatile and recyclable composite material was created using cotton textiles and cuprous oxide-nanocellulose (Cu<sub>2</sub>O-NC), which possesses both antibacterial and extremely effective photocatalytic color degradation capabilities. *In-situ* synthesis of Cu<sub>2</sub>O was carried out using flexible cotton fabrics as substrates. This resulted in Cu<sub>2</sub>O growing uniformly on cotton fibers and enveloped in NC. When tested with methylene blue (MB), the breakdown rate of composite material reached 98.32%, indicating its photocatalytic degradation capability (Su et al., 2021).

Hydrophobication of polystyrene matrixes containing cellulose nanofibril (CNF) at fiber contents of 0.5, 1, 5, and 10% was achieved using silvlation. The material was then extruded into single filaments using single and dual heat extrusion techniques. Si-O-C bond formation points to enhanced fiber-matrix adhesion. A break in hydrogen bonds was seen, suggesting that adjacent fibrils were being attached to parallel nanocellulose fibers. The functionalized CNF exhibits higher thermal stability than the untreated sample, as indicated by the steady creation of Si bonds. Elastic modulus and ultimate tensile strength are increased throughout the extrusion process when the CNF is positioned linearly in the Polystyrene (PS) matrix. The CNFs are compatible with the thermoplastic matrix and have a large capacity for reinforcing once they are functionalized. (Ufodike et al., 2018). The nanocellulose-based flame-retardant and conductive fibers were developed comprising CNF core and silver nanoparticles using wet spinning as shown in (Figure 10.2b). The conductive nanofibers of silver nanowires (AgNWs) and cellulose nanofibrils (CNFs) showed a high electrical conductivity, flexible and mechanical characteristics. Additionally, the fibers performed well in creating a soft circuit as a mechanical elastic conductor (Nechyporchuk et al., 2017a; Wang et al., 2017).

#### **10.4 NANOCELLULOSE IN DYEING**

Researchers have created an environmentally friendly method of dying cotton using nanofibrillated cellulose (NFC) fibers that would cut the amount of water, salt, and alkali used by a factor of ten while still achieving the same level of dyeing performance as an exhaust dyeing method. (Kim et al., 2017). Showcase the mapping of materials and cutting-edge technology for ecological coloring during the pre-treatment and dyeing phases. It includes biochemical (enzymes) and physical (plasma, UV radiation, and ozone technologies) pre-treatment techniques. About the dyeing processes, it addresses ecological materials (natural dyes) and novel technologies (such as plasma, supercritical CO<sub>2</sub>, AirDye®, ultrasonic, microwave, Nano-DyeTM, and electrochemical). Researchers have emphasized the significance of ecological coloration and the need for educational initiatives to train professionals in the fashion and textile industries. They have also highlighted the need to address obstacles and financial feasibility in implementing the suggested alternatives (Lara et al., 2022).

An environmentally friendly indigo-dying technology that can reduce water use by up to 25 times and eliminates the need for any reducing agent or alkali has been
developed by researchers using nanocellulosic materials. This process reduces dye fixing to less than 90%, compared to 70–80% in conventional dyeing. Lighter or deeper tones are achieved in a single process as opposed to traditional vat dyeing, which requires several (up to 8) dips in a reducing vat, followed by oxidation. This dyeing process treats cotton denim fabric or yarn with nanocellulose hydrogel loaded with chitosan and fine natural blue particles. The resulting nanofibrillated cellulose mesh-like conformal coating encloses indigo particles, while chitosan improves the coating's attachment and adherence to fabrics by physical crosslinking (Rai et al., 2021; Spagnuolo et al., 2022).

Mercerized-cationized cotton textiles dyed without salt exhibited much-improved colorfastness qualities compared to untreated cotton dyed using a standard dyeing procedure, except colorfastness against wet crocking. Fabrics that had been mercerized and cationized, as opposed to untreated cotton textiles, were dyed by lowering the  $Na_2CO_3$  concentration from 20 to 5 g/L while preserving the colorfastness and dye fixation of the cloth. Because mercerized-cationized cotton fibers don't require an electrolyte in the dye bath effluent and use less dye and  $Na_2CO_3$  during the dyeing process, it may be less environmentally hazardous than typical reactive dye dyeing (Fu et al., 2013).

Reactive colors can interact and conjugate with soluble polysugars in cotton and nano-fibrillated cellulose (NFC) hydrogels. These dye-conjugated soluble polysugars are dumped into wastewater, contaminating the water and reducing the efficacy of the dyeing process. Researchers have demonstrated that applying polycarboxylic acid as a post-treatment to cotton textiles dyed with NFC causes the poly sugars labeled with soluble dyes to permanently chemically graft. The esterification reaction forms chemical cross-links with the NFC fibers on the cotton fabric. In the washing stage, this combination leads to a 60% reduction in dye discharge and a 30% improvement in dye fixation. The method used to achieve this enhancement keeps the stiffness and breathability of the textiles. The sophisticated textile method is used to evaluate a variety of reactive dyes that cover the entire visual spectrum (Liyanapathiranage et al., 2020).

### **10.5 MISCELLANEOUS APPLICATIONS**

The different approaches used for nanocellulose reinforcement are shown in Figure 10.3a. Cellulose nanofibrils (CNF) and silica nanoparticles (SNP) were treated with polyelectrolyte as a combination for canvas consolidation. The formulas resulted in less than 5% weight gains when applied to model-degraded canvases. Tensile testing demonstrated the mechanical success of the consolidation while scanning electron microscopy and micro-X-ray fluorescence measurements were used to investigate component distribution across the canvas depth. CNF improved flexibility by creating a coating on the canvas surface. SNP's deeper penetration and fiber-scale strengthening resulted in higher stiffness. The SNP/CNF ratio could be adjusted to produce sufficient reinforcement by balancing the two effects (Kolman et al., 2018). Using nanocellulose reinforcement, sustainable regenerated protein fibers were developed by using wet spinning, as shown in Figure 10.3b. Casein/Nanocellulose-based biobased fibers can displace their fossilbased counterparts, e.g., in textile, nonwoven, or composite applications with improved mechanical and morphological properties (Nechyporchuk and Köhnke, 2019).



**FIGURE 10.3** Various methods for nanocellulose reinforcing have been employed. Wet spinning (left), dry spinning (center), and melt spinning (right) are common methods of spinning fibers (a), adapted with permission from (Rosén et al., 2021) Copyright© 2020 The Authors Published by Wiley-VCH GmbH, Adapted schematic illustration of the wet spinning method for creating regenerated casein-nanocellulose composite fibers (b), Adapted with permission from (Nechyporchuk and Köhnke, 2019) Copyright© 2018 American Chemical Society.

To investigate the feasibility of recycling denim waste for nanocellulose manufacture and utilization, nanocellulose was isolated from indigo-dyed denim fabric, and its properties were compared to those of bleached cotton fabric and wood pulp. Sulfuric acid hydrolysis and 2,2,6,6-tetramethylpiperidin-1-yl) oxyl (TEMPO) oxidation were used to create cellulose nanocrystals (CNC) and cellulose nanofibers (TOCN), respectively. Denim-derived nanocellulose was more thermally stable and had higher crystallinity than wood pulp-derived nanocellulose, but it had the same yield, shape, size, and crystallinity as bleached cotton nanocellulose. When applied to a polyvinyl alcohol film, blue indigo-CNC enhanced its mechanical properties and provided remarkable UV blocking (Zhong et al., 2020).

An eco-friendly substitute for synthetic resins and other traditional canvas consolidants includes cellulose nanofibrils (CNF), cellulose nanocrystals (CNC), and carboxymethylated cellulose nanofibrils (CCNF) have been developed. Essentially, all the tested nanocellulose is reinforced in the appropriate elongation regime, in contrast to some conventional consolidants. The best consolidation per additional weight was demonstrated by CCNF; however, in comparison to other nanocelluloses, it required handling at extremely low solids content, exposing canvases to higher water volumes. CNC provided the strongest consolidation after the same number of coatings, but it reinforced the least per added weight and could be used in higher concentrated suspensions. CNF was situated between CCNF and CNC. All nanocelluloses showed greater consolidation in the elongation region of interest as compared to linen canvas and lining with synthetic adhesive (Beva 371) (Nechyporchuk et al., 2018).

From bleached eucalyptus kraft pulp, various degrees of substitution (DS) were created, and their applications in textile warp size were carefully investigated. Using the response surface method, the high-count cotton warp sizing recipe with a negligible amount of CCNFs was optimized. It was found that the optimized sizing formula outperformed the original sizing, even when the temperature and sizing percentages were reduced. The results demonstrated the potential of CCNFs with a DS of 0.38 as an industry auxiliary for sizing pure cotton warps, and the advantages of the recently developed sizing formula included good size performance, energy efficiency, environmental friendliness, and lower emissions (Zhou et al., 2022).

The sluggish rate of oxygen transfer into the viscous media is probably why leuco indigo was successfully stabilized on a nanocellulose suspension. It was evident from visual studies that natural indigo kept the leuco-form intact for a far longer time than synthetic indigo. The increased stability was attributed to radical scavenging species in natural indigo, as synthetic indigo did not demonstrate any noticeable antioxidant properties. The promising results indicated that the paste formulation could be suitable for screen-printing images onto cotton. Leuco indigo remained stable on the nanocellulose carrier, negating the requirement for re-reduction before dyeing and using less potentially harmful reducing agents (Lohtander et al., 2021). Wood pulp and glycidyl trimethylammonium chloride were reacted to create surface-functionalized quaternized cellulose nanofibrils or Q-NFC for anionic dye adsorption. This reaction was accomplished by mechanical disintegration. By mechanically homogenizing the chemically processed cellulose/water slurries with trimethylammonium chloride, very viscous and clear aqueous dispersions of cellulose nanofibrils were produced (Pei et al., 2013).

### **10.6 CONCLUSION**

One especially fascinating application area is using nanocellulose in paper and textile coatings, both as nano- or microfibers and nanocrystals. These materials are translucent, edible, biocompatible, biodegradable, and readily functionalized. However, before their widespread application in industrial manufacturing can be realized, a few problems must be resolved. The availability of nanocellulose, which is currently restricted by the energy needed for its large-scale manufacture, is the primary cause of these constraints. Nanocellulose has been suggested as a viable material for nanocomposites because of its versatility, which includes its abundance, exceptional mechanical capabilities, low weight, biocompatibility, and biodegradability. These days, cellulose and polymer nanocomposite composites are a hot topic. Due to its eco-friendliness and cost-effectiveness, nanocellulose is now being used extensively in textile processing, particularly in the dyeing of cellulosic materials. Nowadays, nanocellulose is being used in several fields of material research.

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## **11** Chitosan A Biobased Sustainable Textile Auxiliary

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## 11.1 BIOBASED POLYMERS AND THEIR GREEN BENEFIT

According to the principles of Green Chemistry, industrial chemical activities should minimize or eliminate the generation of hazardous substances, prioritize energy efficiency, and give preference to the use of renewable material feedstocks and energy sources, such as agricultural products or waste materials, to reduce the reliance on non-renewable (fossil) resources, and promote sustainability [1, 2]. Cellulose, xylan, pectin, lignin, chitin, chitosan, proteins, and cyclodextrins are examples of biopolymers or bioderived materials that can be easily obtained from readily available renewable sources or agro-industrial waste.

These materials are attractive due to their low added value and abundant availability. The transformation of agro-industrial waste into raw materials for industry makes the production chain more sustainable and greener, reducing the dependence on fossil resources. Agro-industrial activities lead to the recycling of atmospheric  $CO_2$  (one of the greenhouse gases), which does not occur when fossil resources are used, as they are immobilized in underground deposits and are mined at high cost. Furthermore, these biomaterials do not compete with the food production chain and have other merits, such as biodegradability and biocompatibility.

In the following section, the obtention or production processes and the structural and physical-chemical characteristics of chitosan, and a general overview of the most recent applications in the textile area, such as in fiber manufacturing, dyeing, and finishing, and in the treatment of wastewaters, highlighting the challenges and perspectives to create more environmentally friendly alternatives to conventional processes, are presented. In particular, the design of high-performance textiles with relevant functionalities such as high durability, UV and antiradical protection, hydrophobicity, and antimicrobial properties is made possible by incredible advances in nanotechnology [3]. Considerations regarding smart textiles and highly technological materials are also highlighted. Scientific articles and patents cited throughout the manuscript were searched in databases such as the Web of Science and the Scopus platform, preferably in the last 5 years.

### 11.2 INTRODUCTION ON CHITOSAN

Chitosan is an outstanding biopolymer obtained from the deacetylation reaction of chitin, the second most abundant polysaccharide on Earth, which is found in the cell walls of mushrooms and the exoskeleton of insects and crustaceans and generated as a byproduct in fishing and aquaculture activities. Crustacean shell, for example, has a chitin content between 13–42% [4]. Chitosan has become the focus of many studies because of its bioactive and antibacterial properties, gelling capacity, and the ability to form micro- and nanocapsules [5, 6]. An important structural feature is the presence of primary amino groups (Figure 11.1) that confer solubility in a slightly acidic environment and the possibility of chemical modifications or even Lewis acid-base interactions. While chitin is a linear biopolymer composed of *N*-acetyl-*D*-glucosamine connected by  $\beta$ –(1→4) glycosidic bonds, chitosan is composed of  $\beta$ –(1→4)-D-glucosamine (or: 2-amino-3-deoxy- $\beta$ -*D*-glycopyranose) units (deacety-lated unit) (see Figure 11.1) intercalated by randomly distributed acetylated units of *N*-acetyl-*D*-glucosamine, because deacetylation reactions are incomplete [4].

Chitosan is biodegradable and biocompatible and displays antimicrobial/biocidal properties, enabling several practical applications in areas such as medicine, food, environment, and materials. The biocompatibility of chitosan is related to the structural and functional similarity with glycosaminoglycans, molecules commonly found in mammalian tissues. Since glycosidic bonds can be cleaved by chitinases, lysozyme, and colon resident bacteria, chitosan has a biodegradable character that justifies the enormous application potential in healthcare, biomedicine, pharmacology (drug-delivery systems) and regenerative medicine (wound healing) [7].



FIGURE 11.1 Chemical structures of chitin and chitosan.

### 11.2.1.1 Preparation of Chitosan from Chitin

Waste from fishing and the cultivation of crustaceans such as shrimps and lobsters contain high amounts of chitin. Chitin represents over 35% of the shrimp exoskeleton and remains as solid waste. It is insoluble in water and most organic solvents, nontoxic, and has properties such as high biodegradability and biocompatibility that allow industrial applications even in the medical and biomedical areas [8, 9]. Recent studies report an important potential of green solvents, such as ionic liquids and deep eutectic solvents, for the better valuation of this residue in the biorefinery concept [10–15]. Chitin processing involves washing, crushing, and generally acid and alkaline treatments to remove minerals, proteins, lipids, and pigments (Figure 11.2). Harsh pH and high-temperature conditions generally decompose the polymeric structure so that milder and more eco-friendly treatments become interesting [16].

In the conventional deacetylation method, chitin is treated with 40% NaOH at 100°C for 1–3 h and produces chitosan with a deacetylation degree of around 85–90% and low molecular weight. This method consumes relatively large amounts of chemical reagents [4]. Some examples of alternative treatments involve deep eutectic



**FIGURE 11.2** Chemical processes involved in the chitin extraction and deacetylation, generating chitosan [4, 16].

solvents [11, 17], microwave heating [18] and processes employing biotechnology [19]. In the last case, demineralization, deproteination, and deacetylation are carried out with enzymes and metabolites, such as lactic acid, obtained from the fermentation of glucose [20]. The process is relatively milder, with less use of chemical reagents, less waste generation, and produces higher quality chitosan structure, although it has higher costs and is time-consuming, which unfortunately makes application on a large scale difficult [4].

### 11.2.1.2 Chitosan properties

While chitin is poorly soluble and, therefore, difficult to manipulate, making its application often unfeasible, chitosan has a basic character and is readily soluble in slightly acidic media, such as 1% (v/v) acetic acid [21]. Examples of acids used to solubilize the biopolymer are citric, lactic, and hydrochloric acid [4]. The most relevant properties of chitosan are its degree of deacetylation and molecular weight, which determine important properties such as tensile strength, flexibility, surface area, porosity, and conductivity. Samples with high degrees of deacetylation have higher solubility and antibacterial activity [20]. To modulate certain properties, such as solubility in different media or binding to a certain molecule, chitosan can be subjected to alkylation, acetylation, generation of imines, and crosslinking reactions using epoxide; it can be oxidatively cleaved, among other reactions. Chitosan polymer chains are also subject to non-covalent interactions such as hydrogen bonds and ionic and hydrophobic interactions [22]. In the presence of water, chitosan forms hydrogels, which are very useful in wound-healing textiles [23]. The primary amine group in the chitosan structure with pKa between 6.3 and 7.2 (varying according to the degree of deacetylation and molecular weight) [24] is protonated in acid media [25]. It displays a high affinity for anionic dyes, such as reactive, acid, and direct dyes, which explains the high interest in this biopolymer for wastewater treatment or cationization of cellulosic fibers [26].

## 11.3 CHITOSAN IN THE TEXTILE INDUSTRY

In the textile sector, chitosan may be used in several ways: 1) In the manufacture of textile fibers, it may be either used for the generation of pure chitosan fibers or blended fiber composites, together with natural or synthetic polymers or applied as a covering on cotton or wool fibers. 2) In textile finishing, it provides antimicrobial properties, and incorporating antibacterial properties into textile materials is of special interest for their application in the medical field [27]. Its cationic properties can also be exploited for the cationization of cellulosic fibers and for treating effluents and wastewater.

The increasing interest in this biopolymer can be seen from recent review articles compiling different chitosan applications in textile processes [5, 20, 28–32].

## 11.3.1 CHITOSAN IN THE FIBER-FORMING PROCESS

The semi-crystalline structure of chitosan is maintained through specific interactions (i.e., inter- and intramolecular hydrogen bonds between the -NH<sub>2</sub>, -OH groups); and

non-specific (i.e., Van der Waals interactions). The strong affinity between polymer chains limits their thermal stability, which prevents their extrusion into fibers via a melt-spinning [33].

Fibers can be obtained through biopolymer dissolution and regeneration as an alternative to melt extrusion, such as in wet spinning. Under certain conditions, such as in polar solvents or ionic liquids containing organic or inorganic electrolytes, the intermolecular interactions are disrupted, and the polymeric chains dissolve. Then, the biopolymer can be regenerated by injecting the polymer solution in an antisolvent bath (where the polymer is not soluble), assuming a specified form according to the spinneret (nanoparticles, fibers, films) [33]. In the case of chitosan, the biopolymer can be dissolved in an acidic medium and then extruded into an alkaline coagulation bath [32].

Another used technique is electrospinning, which uses a high voltage applied to a polymer solution or melt. The electrostatic repulsion induced by the electric field generates a very thin film on the surface, which is collected in the form of fibers [34], However, both described methods are unsuitable for producing pure chitosan fibers, so it must be mixed with other components [35, 36]. The limitations of pure chitosan fiber are related to its mechanical strength. The polymeric structure of chitosan is made up of rigid chains, with little movement due to intense hydrogen bonding, with insufficient entanglement and flexibility, which makes electrospinning difficult. These limitations can be overcome by producing blends with other polymers [35, 36], or using coagulants and/or crosslinkers [37], such as glyoxal, glutaraldehyde and citric acid, which bind to the fibers through covalent bonds. Crosslinking increases its stability and mechanical strength, by forming a more stable structure, resistant to physical deformation and chemical degradation, therefore increasing its durability [38].

It is possible to impart several properties of great functional value through the appropriate functionalization of fabrics with chitosan, such as antimicrobial/biocidal activity, UV protection, water repellence, flame retardance, and antistatic and shrink resistance. Some of these properties are due to the nature of chitosan itself; others are modulated through the attachment of species to the biomolecule scaffold.

### 11.3.2 CHITOSAN AS AUXILIARY IN THE PREPARATION STAGE OF TEXTILES – PURIFICATION, AND REMOVAL OF IMPURITIES

Natural textile fibers contain unwanted impurities like waxes, fats, pectins, and proteins. In addition to these impurities, sizing agents, spinning gums, and dirt from the previous textile processing stages are contaminants that can interfere with the subsequent processing of textile fibers; therefore, their removal is necessary, which can be carried out in an alkaline environment, with or without the addition of hydrogen peroxide, depending on the type and degree of impurities and the processing procedure [39].

Another method involves using biotechnology through enzymes, such as  $\alpha$ -amylase. When adding soluble chitosan as an additive to cotton desizing reactions with  $\alpha$ -amylase, a positive effect on catalysis was observed, in addition to stabilizing the enzyme at high temperatures. Chitosan is allowed to reduce  $\alpha$ -amylase dosage to one-third to obtain desizing efficiency greater than 95% in treatment at 100°C for 10 min. In other words, chitosan acts as a stabilizer and enhancer of  $\alpha$ -amylase action in enzymatic cotton desizing at high temperatures. The mechanism apparently involves the formation of hydrogen bonds between chitosan, amylase, and starch on the cotton surface [40].

The biopolymer was also studied as a support to immobilize amylases during enzymatic desizing of cotton. The immobilized enzymes maintained its efficiency during starch desizing, could be easily separated from the medium, and recovered and reused for up to four desizing cycles, making the process more economically viable [41].

### 11.3.3 CHITOSAN AS AUXILIARY IN FUNCTIONAL TEXTILES

### 11.3.3.1 Antibacterial Properties

Chitosan has been widely investigated to produce antimicrobial textiles, targeting environments that require sterile conditions [5, 42, 43]. The antimicrobial activity of chitosan is related to the presence of protonated amino groups in its polymeric chain [32]. These cationic groups interact electrostatically with anionic compounds in the bacterial cell wall, such as lipopolysaccharides and teichoic acids, causing structural changes and disrupting the microbial membrane. The molecular weight and deacetylation degree of chitosan define the amount of free amino groups in the polymer and determine its antimicrobial effectiveness [32, 44, 45]. A higher deacetylation degree and lower molecular weight of chitosan and its derivatives demonstrated higher antimicrobial, antioxidant, and anticancer capacities. However, its anti-inflammatory effect was found to be not dependent on the molecular weight and more structuredependent [46]. Low molecular weight chitosan can penetrate the cell wall and cause intracellular antimicrobial activity, such as binding to DNA, inhibiting RNA and protein synthesis, and mitochondrial functions [44, 45]. Chitosan, with a higher molecular weight, acts mainly as a chelator, capturing nutrients and preventing them from being absorbed by bacteria, altering the permeability of membranes and causing leakage of cellular components [44, 45].

The effectiveness of chitosan differs in relation to the classification of bacteria. According to their cellular structure, bacteria are differentiated into Gram-positive and Gram-negative, depending on the color they acquire when stained with specific dyes (stain: crystal violet; counterstain: safranin or fuchsin). Gram-positive bacteria have thicker peptidoglycans, while Gram-negative bacteria have more lipopolysac-charides (LPS), which causes different staining in the Gram test. Additionally, these two types of bacteria respond differently to certain types of antibiotics and chitosan [45]. Besides differences in cell surface structure, differences in electrostatic surface charges were supposed to be responsible for different sensitivity to chitosan. However, results have shown that the mode of action of chitosan is more complex than simple electrostatic interactions [45]. Chitosan's innate antimicrobial activity against bacteria, yeast, and some fungi enables its use as a safe and environmentally friendly bioelimination agent in the pretreatment of textile fibers. Research demonstrates the efficiency of chitosan solutions in inhibiting microbial growth on cotton, wool, silk, and other fibers [32].

Chitosan displays a good affinity for cellulosic fibers in an acidic environment [20]. Chitosan is positively charged at acidic pH, while cellulose is negatively charged across the entire pH range. These polymers are then associated by hydrogen

bonding and Van der Waals forces. However, the coating does not withstand repeated and severe washings, as the established interactions are comparatively weak. For finishing applications, it is necessary to immobilize chitosan on cellulose via covalent bonds and crosslinking [47, 48]. The necessary chemical reactions and procedures are well known from durable press finishing of cotton textiles and include crosslinking with polyfunctional reagents such as dialdehydes (glutaraldehyde), dimethylol dihydroxy ethylene urea (DMDHEU), low formaldehyde resins and polycarboxylic acids, generally applied in the presence of a catalyst and through a pad-dry-cure procedure [48, 49].

A combined treatment of cotton fabrics with chitosan and RTV (room-temperaturevulcanizing) silicone was used to impart antimicrobial and water-repellent properties [50]. Cotton treated with 10% chitosan acquired antimicrobial activity against *Staphylococcus aureus* (Gram-positive bacteria) and *Candida albicans* (yeast), but not against *Escherichia coli* (Gram-negative bacteria) and *Aspergillus niger* (fungus). The highest static water contact angle (162.2°), and thus the most waterrepellent fabric, was achieved with 3% (owf) of chitosan.

Flinčec Grgac, Tarbuk [26] investigated the application of chitosan to plain woven fabrics composed of 100% cotton or polyester/cotton (65%/35%) blends to impart durable antimicrobial properties. First, fabrics were subjected to a cold (25°C) alkaline (20% NaOH) pretreatment (jigger, 10 passages) to open the structure of cotton fibers and partially hydrolyze polyester fibers. Chitosan was then applied as a slurry (0.5–1  $\mu$ m; 10 g L<sup>-1</sup>) at 25°C with citric acid (70 g L<sup>-1</sup>) as crosslinking agent and sodium hypophosphite monohydrate (65 g L<sup>-1</sup>) as a catalyst in a jigger (3 passages) - dry (2 min, 110°C)-cure (4 min, 150°C) process. The mercerization, like alkaline pretreatment, improved the mechanical properties. Although the amount of chitosan was reduced after five washing procedures, the antimicrobial effect persisted. It was concluded that alkaline pretreatment is essential to allow effective fixation of chitosan and persistent antimicrobial activity. It can be observed from the procedure that catalyst and crosslinking agents are used in much higher concentrations than chitosan, and it should be questioned if the process is effective.

The same group investigated other polycarboxylic acids, such as maleic acid and 1,2,3,4-butane tetracarboxylic acid (BTCA), as crosslinking and chitosan-activating agents and sodium hypophosphite monohydrate as a catalyst, where BTCA proved to be the better solution to provide increased wash fastness [48].

Another two interesting approaches to achieving washing durable antibacterial properties on viscose fabrics with chitosan nanoparticles (without and with embedded Zn) functionalized fabrics used oxidation with 2,2,6,6-tetramethylpiperidine-1-o xy radical (TEMPO) and coating with TEMPO-oxidized cellulose nanofibrils (TOCN), to introduce functional groups (COOH and CHO) suitable for irreversible binding of chitosan nanoparticles. Both pretreatments resulted in more efficient and wash durable antibacterial activity of viscose fabrics [51].

As pointed out by Luo, Yao and Li [29] the antimicrobial test methods used for antibiotics are not suitable for chitosan that is present on textile fabrics in the solid state, and the existing antimicrobial textile testing systems are nonsatisfying and not adequate for mild antimicrobials such as chitosan. Therefore, the reported results are mostly hard to compare.

Another strategy to obtain self-disinfecting cotton fabric with bactericidal activity used polyacrylate to bind chitosan nanoparticles to the textile surface [52]. Although promising, the final product was only water washed instead of using soap or an alkaline detergent, which is usually used in washing cotton garments.

## 11.3.3.2 Flame Retardancy

Chitosan is a biomacromolecule studied as a component of flame retardant systems due to its ability to form a protective carbonaceous residue when exposed to heat [53]. Chitosan and its chemically modified derivatives, often in combination with other additives such as phosphorus compounds, were recently used to improve the fire resistance of various polymeric materials, including conventional polymers, textile fabrics, foams, and wood [53]. Carboxymethyl chitosan nanoparticles were fixed on graphene sheets and decorated with polypyrrole-silver nanocomposite, and the resulting material was applied to cotton/polyester fabric [54]. While the graphene sheets are a binder to coat the cotton surface, the pyrrole polymerizes, attaching the polypyrrole-silver nanocomposite to the graphene-modified textile. The carboxymethyl chitosan nanoparticles exfoliate the graphene sheets and form a dispersed and stable complex so that it can coat the cotton surface uniformly. The coating presents, in addition to low electrical resistance (i.e., high electrical conductivity), antimicrobial activity against E. coli and S. aureus and flame-retardant behavior. The burning rate of the coated fabric was 30.9 mm/min, which is 80% lower than that of the uncoated fabric (149.3 mm/min). Chitosan-based treatments have been shown to reduce flammability and improve fire performance of these materials. Challenges remain mainly concerning the durability of treatments and the development of systems entirely from renewable resources [53].

## 11.4 APPLICATION OF CHITOSAN AS A TEXTILE AUXILIARY

Several auxiliary chemical products are used during fiber processing to optimize this operation. As demonstrated below, chitosan can efficiently replace many chemicals as a more sustainable alternative.

## 11.4.1 OPTICAL WHITENING

Chitosan microspheres were explored as a controlled release system for an optical whitening agent (optical brightener) and were loaded with 4,4'-bis[(4-anilino-6-morpholino-1,3,5-triazin-2-yl)amino]-stilbene-2,2'-disodium disulfonate (CBUS), with an encapsulation efficiency of about 90% [55]. The microspheres exhibited pH-dependent controlled release of CBUS, with nearly 100% being released at pH 2 within the first 90 minutes. At pH between 4.5 and 7, no release was observed.

Release of the optical brightener occurred due to the protonation of chitosan amino groups and the quick disassembly of the microspheres at low pH. The encapsulation increases the stability of fluorescent brighteners to light. It enables subsequent controlled release and optical bleaching of the cotton fabrics, mediated by adding acid as a chemical stimulus. The results demonstrate the potential of chitosan microspheres as smart, pH-responsive optical brightening agents for textile applications [55]. However, such low pH values are uncommon in the processing of cellulosic textiles and might lead to fabric damage.

### 11.4.2 APPLICATIONS OF CHITOSAN IN TEXTILE DYEING

Using chitosan in cellulosic dyeing is an interesting alternative because coatings with the polycationic biopolymer improve the affinity of cellulosic fibers for anionic dyes [56, 57]. Therewith the need for the use of salt (electrolyte) is reduced [58]. Depending on the dye class, dyes and chitosan are associated through hydrogen bonds or ionic interactions [59]. Verma, Singh and Rose [56] applied chitosan (2,5% w/w) in a bath (liquor ratio 1:30) together with citric acid (4%) and sodium hypophosphite (5%) to cotton by impregnation during 60 minutes at 90°C and pH 5, squeezing off the excessive liquor in a pad-mangle, subsequent drying at 100°C for 7 minutes and curing at 150°C for 3 minutes. The fabric was then dyed with C.I. Reactive Red 120 and evaluated. A similar strategy was described before to fix cyclodextrins onto cotton fibers, where hypophosphite is the catalyst and citric acid, a polycarboxylic acid, functions as crosslinker [60]. The optimal dyeing conditions of pH 5.0, 80°C, and 60 minutes, resulted in a dye absorption of 78.9%, and a color strength of 18.72 (K/S value), with very good fastness against washing and rubbing (4-5). The authors argue that results are significantly improved with respect to the standard alkaline dyed fabric where a dye bath exhaustion of 68.36% and a K/S of 13.03 was reported. However, it is not quite clear from the manuscript if the comparison refers to dyeing with the same amount of dye, as alkaline dyeing was carried out with 1% owf, while optimum dyeing of chitosan cationized fabric was carried out with 2.5% owf of the dye, although other dye concentrations were tested for the fabrics prepared with chitosan.

Polyester (PET) was also modified with chitosan and its lactate derivative to achieve antimicrobial properties. In this case, a green single-step dyeing and finishing procedure in supercritical  $CO_2$  with a disperse dye, chitosan, and the anchoring and crosslinking agents dodecylamine and hexamethylene diisocyanate was used [61]. Besides the advantages of the supercritical process, such as the absence of residues, low viscosity, excellent diffusion, and high solvating power, polyester fabrics with improved antimicrobial properties (bacteria reduction rate of 75–93%), good color intensity and fastness properties were obtained [61].

The functionalization of cotton fabrics was also conducted with chitosan in the form of nanostructures, which were obtained by the ionic gelation method, in the presence of sodium tripolyphosphate, and additionally a reactive (Yellow Everzol) or a disperse dye (Navy blue Itosperse) were encapsulated. The dyes were encapsulated with high efficiency, around 90%, and had improved stability. Chitosan nanoparticles were applied to textiles in a pad-dry-cure procedure to dye cotton [59, 62], enabling more environmentally friendly processes [59, 62]. Dye uptake was maximum on viscose/wool fabric with 17.6% dye uptake, while on cotton, viscose/lycra, cotton/lyocell, cotton/polyamide, and polyester, dye uptake was less than 5% [59].

The encapsulated dye attaches to the cotton fiber through the coating of the fibers, which involves hydrogen bonding. Additionally, antimicrobial properties are provided without risk to the skin [62]. However, the authors did not provide any data on washing or rubbing fastness of the dyed fabrics or any comparison to traditional dyeing

procedures, which are fundamental for proofing the concept of any alternative dyeing process. Furthermore, neither the chemical structure of the dyes nor their Color Index were provided, which makes it difficult to better understand the mechanisms involved in the process.

As an alternative to ordinary, commercial dyes, some pigments derived from natural sources have been studied for textile dyeing. Considering their more sustainable nature compared to synthetic dyes, in addition to the ability to generate a wide chromatic range from the same natural source, natural dyes present themselves as a possible alternative to reduce impacts generated by this stage of textile processing [63].

Cotton fabrics were treated with different concentrations of chitosan (0%, 0.25%, 0.5%, and 1% w/v) and dyed with *Mangifera indica L*. (mango) leaf extract. The results showed that pre-treatment with chitosan, regardless of concentration, promoted increased K/S values compared to the untreated control, indicating greater absorption of the natural dye. Furthermore, the pre-treated samples exhibited greater color fastness. It was concluded that chitosan enhances the fixation of natural dyes in cotton fabrics, allowing more solid and chromatically intense colors [63].

It was shown that treating cotton with chitosan improves the fabric's affinity to extracts from *Aronia melanocarpa*'s berries [64]. The natural dye, consisting of anthocyanins and phenolic compounds, has antioxidant activity, and the combination of chitosan and the dye provides antibacterial and antioxidant properties to the fabric.

A similar strategy was employed to adhere Peanut Skin Extracts (PSE) to Flax Fabrics [65]. The treatment of flax with chitosan increased the absorption of PSE, and the resulting fabric showed better color fastness, UV resistance, and scavenging free radicals.

### 11.4.3 ANTI-FOULING PROPERTIES

Anti-fouling/self-cleaning and hydrophobic and flame-retardant properties on cotton were obtained from a composite coating of graphite carbon nitride (GCN) and phosphorylated chitosan (PCS) [66]. The modified fabric displayed photocatalytic behavior upon exposure to sunlight, leading to degradation (self-cleaning effect) of Rhodamine B dye, achieving a decoloration yield of nearly 90% in 15 min. The photodegradation mechanism appears to be related to the electronic excitation of the GCN, which produces photogenerated electrons and hole pairs; these electrons reduce  $O_2$  to  $O_2^-$ , which in turn degrades the organic dye. Regarding flame retardant behavior, the modified fabric exhibited a 55.8% reduction of heat release compared to a cotton control fabric (151.7 and 343.5 W/g, respectively) [66].

### 11.4.4 UV PROTECTION

Cotton was functionalized with a nanocellulose and chitosan nanocomposite (CS/NCs), providing UV-blocking, enhanced mechanical properties, and hydrophobicity [67]. In this study, commercial nanocellulose with an average diameter of 20 nm, and different average lengths (100 nm, 200 nm, and 800 nm) were functionalized with chitosan, generating nanocomposites, hereinafter referred to as "CS/NCs". The CS/NCs nanocomposites were initially produced by dispersing nanocellulose (NC) and adding chitosan

(CS) and tannic acid. Chitosan is a dispersant and binding agent, while tannic acid was applied as a plasticizer and cross-linker. The CS/NCs were then applied to the cotton surface using the pad-dry-cure method.

It was found that the UV protection factor (UPF) increased as a function of the decrease in particle size of NC. Specifically, achieving UPF values of 35.29 (CS/NCs-800 nm), 47.17 (CS/NCs-200 nm), and 62.89 (CS/NCs-100 nm), which were significantly higher than that of untreated cotton (14.31), and fabric treated only with chitosan (20.44). The UV protection effect was long-lasting even after 30 laundering cycles [67].

Chitosan also proved efficient for UV protection of cotton fabrics when applied with onion skin extract, which has a reddish color. A cotton fabric dyed with the extract and treated with chitosan presented a brown color and had a UPF of 84.80, significantly higher than the fabric dyed and treated with alum (conventional mordant composed probably of aluminum potassium sulfate; not additionally specified by the authors), which had a UPF of 66.70. This significant UV protection is attributed to the high interaction capacity of chitosan with natural dye molecules, increasing their fixation on the fabric [68].

### 11.4.5 SMART TEXTILES

Highly technological materials based on chitosan were recently presented [69–71]. The function of chitosan was to facilitate the incorporation of another species into the fabric by reducing the electrostatic repulsion between them [71]. An electronic textile with a strain sensor containing graphene oxide for movement monitoring was presented [70]. A polyamide/spandex knitted fabric was modified with chitosan to increase graphene oxide absorption. Finally, the material was finished with silica dioxide and poly(dimethylsiloxane) (PDMS). The sensor had a response time of 22 ms and stable cycling durability, with over 4000 cycles; it was mounted on a pressure sensor, allowing it to monitor human physiological signals in real time. Additionally, the prepared sensor presented superhydrophobicity, photothermal effects, and UV protection, with the potential to be used in the smart clothing sector, such as in winter sportswear for athletes [70]. A fabric for health monitoring and medical diagnosis was generated by applying colorimetric pH sweat and lactate sensors [69]. Therefore, layers of chitosan, sodium carboxymethylcellulose, and a dye indicator or a lactate assay were applied to cotton substrates. Methyl orange and bromocresol green were used as pH indicators and lactate enzymatic assay to detect lactate. These parameters indicate possible dehydration and pressure ischemia and should be monitored in athletes. The combination of different stains provides an easily identifiable visual signal of the athlete's health status.

### 11.4.6 CHITOSAN IN EFFLUENT AND WASTEWATER TREATMENT

In addition to its use as an active ingredient in the formulation of textile articles, as mentioned previously, chitosan was used extensively in designing systems for the remediation of wastewater from the production process. The most obvious application is its use as an adsorbent, mainly for anionic dyes, taking advantage of its polycationic character and low cost [72]. An elegant strategy for removing textile dyes is immobilizing a cationizing agent on a solid surface, allowing reuse and easy liquid phase separation. This was achieved by fixing chitosan onto magnetic nanoparticles. A biocomposite based on nanoparticles of MgO, Fe<sub>3</sub>O<sub>4</sub>, and chitosan, crosslinked by glutaraldehyde, was prepared and used for capturing Reactive Blue 19 dye (C.I. 61200) from an aqueous solution [73]. The maximum adsorption capacity was 193.2 mg/g, at pH 4, 45°C, and 20 minutes of contact. In another work, FeO nanoparticles, prepared by biosynthesis with Aspergillus tamarii, were incorporated into chitosan beads using the sol-gel method without crosslinkers. The resulting composite was used to remediate real wastewater (containing, for example, reactive red 198 (C.I. 18221), reactive red 195, and reactive red 141) and removed 94.7% of the color after 90 minutes of contact. The composite adsorbent could be easily separated from the medium using a magnet, and after dye desorption, it could be reused for at least seven cycles [74]. A nanofiltration membrane was designed by coating nanoporous polyacrylonitrile (PAN) with a chitosan film and crosslinking with an anionic dye, direct red 80 - DR80, in a simple and eco-friendly way, without the use of complex synthetic methods or toxic chemicals. The crosslinking agent binds to chitosan through electrostatic interactions and makes the membrane surface negatively charged [75]. The composite presented high durability and almost quantitative rejection (separation efficiency) for anionic dyes such as direct red 80 (DR80), C.I. 35780; congo red (CR) C.I. 22120; amaranth (AM) C.I. 16185; a cationic dye, alcian blue 8GX (AB8GX), C.I. 74240; and a zwitterionic dye, acid blue 90 (AB90), C.I. 42655. Another wastewater treatment method that has gained increasing interest is based on advanced oxidation processes (AOPs), such as heterogeneous photocatalysis. Silver nanoparticles (AgNPs) were prepared using chitosan, both as a reducing agent and a stabilizer [76], and explored in the photochemical decomposition of acid red 37 dye. The dye photodegradation efficiency was compared with other systems, such as UV/TiO<sub>2</sub>, UV/ZnO and UV/(TiO<sub>2</sub>-ZnO). Combining TiO<sub>2</sub>-ZnO with 0.18 g L<sup>-1</sup> of AgNPs, the decomposition of the dye was achieved very quickly ( $t^{1/2} = 3 \text{ min}$ ) under UV irradiation (254 nm, total output at ultraviolet, 13.4 W).

## 11.5 CHALLENGES AND LIMITATIONS OF THE USE OF CHITOSAN IN TEXTILE PROCESSES

Chitosan has proven to be a promising, sustainable auxiliary for various textileprocessing applications, such as dyeing, functional finishes, and antibacterial treatments. This versatility arises from its excellent properties, such as biodegradability, non-toxicity, antimicrobial properties, and the ability to form films on fibers. However, some limitations need to be overcome for using chitosan to make its use viable on a larger scale in the textile industry.

One of these limitations is its low solubility in water, especially at neutral and alkaline pH, making its application in textile processes generally conducted in aqueous media difficult. Furthermore, chemical modification methods to increase its solubility are still complex and expensive. However, low solubility opens the possibility to form self-assembled micro- and nanostructures. This characteristic is an important perspective, as it can constitute a vehicle for several active ingredients to confer new functions to textiles. New textile strategies need to be developed to improve the fixation processes of these nano- or microcarriers. Another challenge is the difficulty of forming thin, uniform chitosan films on fabrics, limiting their fixation and durability. Chitosan interacts with cotton fibers through hydrogen bonding; however, these bonds are weak, fixation is not durable, and the coating leaches with repeated washes. Therefore, fixation through crosslinking agents is necessary, which increases costs, and sometimes, another process step is necessary.

There are also questions regarding the availability and costs of obtaining chitosan from chitin, requiring the removal of proteins and minerals. Ultimately, optimal processing conditions must be determined on a case-by-case basis for each application.

Thus, despite the immense potential of chitosan, many studies are still needed to overcome these technical limitations and enable the widespread use of this biopolymer in the textile industry. Furthermore, the environmental impact of chitin extraction and its deacetylation needs to be better evaluated. Optimized processing techniques and reduced costs are needed, and there are still challenges to establishing chitosan as an environmentally friendly alternative to conventional textile processes and auxiliaries.

Another point emphasized throughout this review was the critical analyses of all research papers cited herein. We could show that only by the meticulous reading of the sometimes too rapidly published articles can the flaws and shortcomings that have passed the review process be detected. For example, molar ratios of chemicals in some processes studied in research papers do not make much sense for real industrial applications, and the final properties of developed products should be evaluated with sufficient rigor before publication; negative results should not be omitted. Otherwise, promises cannot be held, and outcomes described in the articles are useless.

### **ABBREVIATIONS**

**owf** on weight fabric (% mass product per mass textile material) **UPF** UV protection factor

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## **DECLARATIONS CONFLICT OF INTEREST**

The authors declare no conflict of interest.

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# 12 Cyclodextrins as Biobased Sustainable Textile Auxiliaries

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## 12.1 INTRODUCTION TO CYCLODEXTRINS

Cyclodextrins (CDs) are cyclic oligosaccharides composed of glucose (D-(+)glucopyranose) subunits linked together by  $\alpha$ -1,4 glycosidic bonds. The most relevant examples are  $\alpha$ -CD,  $\beta$ -CD, and  $\gamma$ -CD, respectively composed of 6, 7, and 8 units of glucose monomers (Figure 12.1). Their structure is cone-shaped and relatively rigid, and the saccharidic hydroxyl groups are on the outer part of the macrocycle. This causes cyclodextrins to have a hydrophilic outer shell and a lipophilic cavity, allowing the inclusion of small and hydrophobic molecules in a host-guest interaction [1–3]. In this way, it is possible to solubilize hydrophobic substances in an aqueous medium. The main natural cyclodextrins have different dimensions and physical properties according to the number of glycosidic units (Table 12.1).

This 'receptor' unit characteristic enables numerous important academic and industrial applications in catalysis [4-8], analytical chemistry, including naked-eye determination of analytes [9, 10], chromatography and purification [11–14], cosmetics [15], pharmaceutical applications [16, 17], food formulations [18], and textiles [19]. Cyclodextrins are biocompatible, approved by the Food and Drug Administration (FDA), and appropriate for human and environmental-friendly uses and applications [20]. It is believed that  $\alpha$ - and  $\beta$ -CDs were first isolated in 1891 [21] and synthesized from starch by enzymatic reaction (fermentation). Nevertheless, they were properly characterized only a few decades later [22–24]. This enzymatic degradation of starch is still today's process used in industrial production [25]. In addition, semi-synthetic CDs are synthesized for specific needs by modifying the natural CDs [26]. The main interest in these macrocycles relies on their particular complex forming capacity with various substances through non-covalent bonds and by accommodating them completely or partially in the interior of their cavity. This type of association is known as a host-guest complex, in which the "host" component is usually a larger molecule that encloses/surrounds the smaller "guest" molecule (or ion, or even a radical), according



**FIGURE 12.1** Structure and properties of  $\alpha$ -,  $\beta$ - and  $\gamma$ -CD [2].

### TABLE 12.1 Characteristics of $\alpha$ -, $\beta$ - and $\gamma$ -Cyclodextrins [2]

	α-	β-	γ-
glucose units	6	7	8
molar mass (g mol <sup>-1</sup> )	972	1135	1297
solubility in water at room temperature (g·100 mL <sup>-1</sup> )	14.5	1.85	23.2
$[\alpha]_{\rm D} 25^{\circ}{\rm C}$	$150 \pm 0.5$	$162.5 \pm 0.5$	$177.4\pm0.5$
cavity diameter (Å)	4.7 - 5.3	6.0 - 6.5	7.5 - 8.3
height (Å)	$7.9 \pm 0.1$	$7.9 \pm 0.1$	$7.9 \pm 0.1$
diameter of outer periphery (Å)	$14.6 \pm 0.4$	$15.4 \pm 0.4$	$17.5 \pm 0.4$
volume of the cavity $(Å^3)$	174	262	427

to the concepts of supramolecular chemistry [27, 28]. The formation of the host-guest complex with CDs is reversible, so the system can be exploited for controlled release. Complexation changes some intrinsic properties of the substrate, such as solubility, reaction behavior, and resistance to light and oxygen. In general, the reactivity of the guest species decreases (i.e., is stabilized), but in some cases, the CD cavity displays catalytic activity as an enzyme, accelerating chemical reactions. In the food industry, for example, cyclodextrins serve to encapsulate certain components and protect them

from the action of oxygen and light, preventing changes in taste and odor. In addition, CDs can be used to remove substances with unpleasant taste and odor, improving the organoleptic qualities of food [18]. The purpose of the inclusion of drugs in cyclodex-trins is to increase their solubility and promote transport in biological systems (drug delivery) [17], in addition to significantly increasing their stability [16]. Another interesting feature of CDs is the possibility of including guest molecules with certain selectivity. Besides differences in the size of the cavity of the three main CDs, the microenvironment of cyclodextrins is chiral [29, 30]. Therefore, they are exploited in the molecular recognition and/or separation of enantiomers [11–14].

As evident from Figure 12.1 and Table 12.1, the size of the macrocycle's cavity depends on the number of glucopyranoside units, and therefore, the interaction of some guest species can be modulated through proper selection of the type of cyclo-dextrin or even through a specific modification of the CD. However, a second guest molecule, like a surfactant or a fatty acid, may interfere in forming the association constant of the guest-host complex with a desired guest compound, and the formation of ternary complexes has been reported [31, 32].

CDs are produced from starch and linear  $\alpha(1-4)$ glucans by an  $\alpha$ -amylase type enzyme, the cyclodextrin D-glucanotransferase or glycosyltransferase (CGTase; EC 2.4.1.19), through an intramolecular transglycosidation reaction. In this reaction, CGTase hydrolyses an  $\alpha$ -1,4 bond of a linear glucan (G<sub>n</sub>) and transfers the newly formed reducing terminal to the non-reducing terminal of the same molecule, forming a cyclic molecule, cyclodextrin, with x glycosidic units (G<sub>x-cycle</sub>) and a linear fragment (G<sub>n-x</sub>). Generally, a mixture of the main  $\alpha$  (6 units),  $\beta$  (7 units), and  $\gamma$  CD (8 units) is produced together with smaller amounts of larger CDs. Proportions depend on the origin of the CGTase and reaction conditions. The production profile of CDs can also be tailored by genetic modifications of the enzyme. Details on the different CGTase, the producing microorganisms, and the production process can be found in the specific literature [33–36]. Separation and purification of inclusion complexes with appropriate guest-molecules [25, 37, 38].

Natural cyclodextrins and CD derivatives are produced and marketed commercially, for example, by the company Wacker Chemie AG (Germany) as CAVAMAX and CAVASOL, respectively [39]. Other producers are Ensuiko Sugar Refining, Nihon Shokuhin Kako, Roquette, Ashland, Shandong Xinda, Yunan Yongguang, Qufu Tianli, Zibo Qianhui, Jiangsu Fengyuan, Mengzhou Huaxing, Mengzhou Hongji, etc., where the top three producers had a 57% market share. The global cyclodextrin market was estimated to grow to the size of US\$ 328.2 million by 2028, with a CAGR (Compound Annual Growth Rate) of 6.1% during the forecast period 2022–2028 [40].

### 12.2 APPLICATIONS IN TEXTILES

In the textile industry, the unique properties of cyclodextrins enable their application in dyeing, finishing, and wastewater treatment processes [19, 41]. Most of the protocols presented use  $\beta$ -CD, probably due to the combination of price and cavity size, despite its solubility being lower than that of  $\alpha$ -CD and  $\gamma$ -CD.

A recent search in the Web of Science database (2023/12/19) with the terms textile\* and cyclodextrin\* encountered 533 (core collection)/788 (all databases) results, 437 research articles, and 279 patents, being 61 review articles, but only 9 reviews are more specific on the topic textile\* containing both terms in the title. Publication numbers reveal increasing interest in the topic (Figure 12.2). Our group [41] reviewed the literature on the subject up to 2010, and Bezerra [19] reviewed the literature up to 2020. Other review articles did not focus on cyclodextrin applications in textiles alone. The protocols discussed here were selected from the last 5 years in the Web of Science database.

Cyclodextrins may be used in textile processing as an auxiliary that aids in the process without remaining on the textile substrate. Alternatively, they may be immobilized on the textile as a receptor molecule to change fiber properties. In the second case, two conditions must be met for practical and economic reasons: the cyclodextrin must be anchored to the fiber, and the host macrocycle must form a stable complex with the desired guest molecule(s).

In cases where definitive CD fixation is required, CDs must be chemically modified to establish a covalent bond or strong interaction with the fiber polymer, since CDs are highly soluble in water and would be lost during washing [30, 42–44]. An example of a covalent bond establishing CD-derivative is monochlorotriazinyl- $\beta$ cyclodextrin (MCT- $\beta$ -CD), with a reactive group, the same that is used in reactive dyes, capable of fixing CDs on surfaces through reaction with nucleophilic groups, such as -NH, -SH, -OH (present in cellulosic fibers and wool) [30].

Another strategy involves the use of crosslinking agents, such as epichlorohydrin, glyoxal, glutaraldehyde, and polycarboxylic acids, that are applied together with the cyclodextrin by the pad-dry-cure technique, usually in the presence of a catalyst. Polycarboxylic acids are applied in the presence of a catalyst such as sodium



FIGURE 12.2 Number of publications, including patents since 1975.

hypophosphite monohydrate as a catalyst. Citric acid and 1,2,3,4-butane tetracarboxylic acid (BTCA) [45, 46] are good crosslinkers due to the numerous carboxylic groups that bind to cellulose through ester bonds; both demonstrated better results than polyacrylic acid. Citric acid is more interesting than BTCA as it is more available, cheaper, and less toxic. However, some yellowing of textiles has been reported with citric acid [47], so BTCA has become the preferred solution. Noteworthy, cyclodextrins undergo self-polymerization in the presence of an acid such as methanesulfonic acid [48, 49].

### 12.2.1 Dyeing Process

The use of CDs as dyeing auxiliaries has been studied, and an improvement in dye leveling, exhaustion, and color intensity has been claimed. CDs can accommodate dyes in their hydrophobic pocket, forming inclusion complexes and thus withdrawing dye molecules from the equilibrium solubilized dye–*fiber adsorbed dye* and acting as a retardant (leveling agents). The association of CDs and dyes leads to retardance of the formation of dye-fiber bonds. It makes dyeing more uniform, as the CD-Dye complex has a greater molar mass and consequently displays reduced diffusion through the dyeing bath and the fiber [19, 50]. To our knowledge, there is not any substantial proof that the complex CD-dye diffuses into the fiber, which, to our understanding, in the case of more hydrophobic fibers such as polyamides (PA6 and PA66) or polyester (PET) must be difficult as CD molecules are more hydrophilic than disperse or acid dyes. When CDs are used as dyeing auxiliaries, they are not fixed to the fiber or retained in the fiber because of their relatively high solubility and low affinity to the textile, and they are finally discarded with the effluent.

The dyeing of polyamide fibers with disperse dyes in the presence of  $\beta$ -cyclodextrin as an auxiliary agent has been investigated by various groups [51–53]. Ferreira, Espinoza-Quinones [52] used (2-Hydroxypropyl)- $\beta$ -cyclodextrin (2-HP- $\beta$ -CD) as a cyclodextrin-based alternative to commercial leveling agents in the dyeing of knitted fabrics composed of PA6 microfibers with the dye C.I. Disperse Red 60 and compared dyeing results without any additives with 2-HP- $\beta$ -CD and with the commercial leveling agents. Although authors claim that dyeing with the CD derivative presented the best uniformity and positively affected color intensity, this cannot be clearly seen from the complex description of dyeing data by different adsorption models. It is also unclear why the authors, used  $\Delta E$ , instead of color strength K/S, which in standard annotation is the color difference to a standard dyeing procedure and usually should not be larger than 1. While for the CD auxiliary, a  $\Delta E$  of 63.29 (color) and a  $\sigma\Delta E$ (deviation representing uniformity of dyeing) of 0.02047 was determined for the commercial leveling agent, and in the absence of any leveling agent  $\Delta E$  were 62.47 and 62.0 and  $\sigma_{AE}$  were 0.05136 and 0.1532, respectively [52].

The same group investigated the dyeing of a PA fabric (it was not identified as to whether it was PA6 or PA66) with a previously prepared 1:1 complex of  $\beta$ -CD and C.I. Disperse yellow 211 and reported that higher coloristic intensity in comparison with the conventional process was achieved. The fabrics dyed with the  $\beta$ -CD:DY211 complex were darker ( $\Delta L < 0$ ; L is luminosity), more saturated, and more yellow ( $\Delta b > 0$ ) [51].

Hydroxypropyl- $\beta$ -cyclodextrin (HP- $\beta$ -CD) has also been investigated as an auxiliary in trichromic dyeing of polyamide 6.6 fabrics with acid dyes. The dye that was complexed by the CD (C.I. Acid Blue 62) was more affected in its dyeing behavior, but also both other dyes (C.I. Acid Red 42 and C.I. Acid Yellow 42) suffered alterations in their overall dyeing kinetics. As the main outcome of this research, better uniformity in dyeing was attributed to slower dyeing kinetics, i.e., a more controlled exhaustion rate in the critical dyeing phase, when compared to the commercial leveling agent Albegal B, that retarded C.I. Acid Blue 62 dyeing, accelerated C.I. Acid Yellow 42 exhaustion, but did not affect exhaustion of the third dye [54].

Another approach to improve dyeing efficiency was investigated by modifying fabrics covalently with cyclodextrins to retain dye molecules through host-guest interaction. For example, polyester, cotton, and polyester/cotton blended fabrics have been modified with  $\beta$ -CD, using citric acid or 1,2,3,4-butane tetracarboxylic acid as crosslinker [55]. CD interacts with polyester via physical adhesion on polyester fibers or entanglement in polyester fabrics. Consequently, hydrophilicity increases from 0.41% to 2.22% in polyester fabric, but a decrease in the mechanical strength of polyester fabrics was observed. The higher hydrophilicity is expected because of the introduction of additional hydroxylic groups, and the loss in resistance is typical for crosslinking used in wrinkle-resistant cotton treatments, but the authors attribute it to possible damages during the curing process. Different from the original polyester fabric, it was possible to dye CD-modified polyester with cationic dyes at 70°C because of the host-guest effects of the CD on the modified fiber, as well as the interactions between the carboxylic groups of citric acid and the positive charge of the dye.

In addition to dyeing with ordinary, synthetic dyes, some interesting examples address natural dyes. They have some advantages, as they are obtained from natural sources, are biodegradable and environmentally safe. The limitations in its application include, mainly, the low stability against high temperatures, oxygen, and UV light, but also lower molar absorbance, difficulty in reproducing color homogeneously, in addition to the need to use mordants (sometimes toxic metals), to fix the dye to the fiber. In this sense, considering their receptor site, cyclodextrins can stabilize the dye.

In this way, silk was dyed with grape seed proanthocyanidin extract [56] and extracts of sumac (*Rhus coriaria*), onion (*Allium cepa*), red cabbage (*Brassica oleracea*), madder (*Rubia tinctorum*), turmeric (*Curcuma longa*) [57], provided that the fiber was previously functionalized with  $\beta$ -CD, through a crosslinker such as succinic acid, and sodium hypophosphite catalyst [57]; or 4-carboxylphenylboronic acid functionalized 2-hydroxypropyl- $\beta$ -cyclodextrin (HP- $\beta$ -CD) covered magnetic Fe<sub>3</sub>O<sub>4</sub> particles [56]. These procedures were reported to provide UV resistance, higher color strength [56], tensile strength, color stability, and softness (soft handle) [57].

### 12.2.2 ENZYMATIC PROCESSING

Cyclodextrins have also been applied in the enzymatic processing of textiles. When the enzyme is immobilized on some surface, it can be reused, significantly reducing operation costs and increasing its stability. Srivastava, Singh [58] immobilized keratinase (fibrous proteins present in the structural part of wool, nails, hair, and feathers) on chitosan (90% yield) and chitosan grafted- $\beta$ -cyclodextrin beads (93% yield). The chitosan was linked to  $\beta$ -cyclodextrin through a glutaraldehyde spacer, and then the enzyme was mixed with the chitosan- $\beta$ -CD composite. The resulting material was used in the processing of wool fibers. The immobilization of the enzyme increased the temperature optimum for maximum relative enzyme activity from 70°C (free enzyme) to 75°C. The enzymatic activity was also higher in relation to the free enzyme in all pH ranges. Enzyme immobilization also increases the possibility of reuse. After 20 activity cycles, 40% and 50% activity were retained for chitosan and chitosan- $\beta$ -CD, respectively.

## 12.2.3 FINISHING

### 12.2.3.1 Encapsulation and Release of Molecules of Interest

Cyclodextrins are also useful for producing functional textiles that need to be immobilized to the fabric. An interesting application is the functionalization of fabrics with cyclodextrins and encapsulation of substances such as fragrances, drugs, and insect repellents [59], aiming at the controlled release of such substances. With the depletion of the charge, the cyclodextrins could again be recharged with the substance. Encapsulation of active compounds in cyclodextrins stabilizes them during manufacturing, storage, and use processes. In addition to releasing the aforementioned substances, the macrocycles can also adsorb molecules responsible for undesirable odors [60]. Cotton was positively charged through quaternary ammonium cationization. Subsequently, aromatic nanocapsules linked to cyclodextrin through epichlorohydrin and loaded with lavender essence were adsorbed onto this fabric [61]. An interesting feature is that the modification did not involve adhesives but an "in situ" immobilization mechanism, in which the nanoparticles diffused into the fibers and then were "grown" by reaction with an alkaline solution. The modified fabric presented a long-lasting fragrance release, continuously controlled for up to 120 days. In addition, it also presented wash resistance, so after 5 washes, up to 91.19% of the essential oil remained in the fabric, almost 5 times more than in the non-modified fabric.

### 12.2.3.2 Antimicrobial Properties

The same encapsulation strategy can be explored in the design of fabrics with antimicrobial behavior by fixing cyclodextrins on fabric and loading them with antimicrobial substances [62–64].  $\beta$ -cyclodextrin was fixed on cotton fabrics by a commercial, acrylic-based binder and loaded with thymol and carvacrol [64]. The fabric presented antibacterial activity against *Klebsiella pneumoniae* (Gram-negative) and *Staphylococcus aureus* (Gram-positive bacteria), even after 20 washing cycles. Monochlorotriazinyl- $\beta$ -cyclodextrin was fixed on indigo-dyed cotton denim and then loaded with Clove oil, Lavender oil, Tulsi oil, and Vanillin AR [62]. The modification and incorporation of essential oils conferred antimicrobial properties against the Gram-positive *S. aureus* and the Gram-negative *E. coli* bacteria, as well as UV protection. These properties persisted even after 15 washes. Although wettability was reduced by incorporating the essential oils, this was partially recovered after several

washes. More innovative cyclodextrins-based systems employ stimulus-responsive system strategies ("smart materials") [65, 66], such as thermosensitive cotton textiles. Systems of this type based on host-guest interactions are more interesting than those based on covalent bonds, as they avoid complex bonds with the substrate (drug), and require less energy to break the bonds [67]. Sun et al., in a recent protocol, prepared a thermosensitive hydrogel containing cyclodextrins derivatives (2-h ydroxypropyl- $\beta$ -cyclodextrin, HP- $\beta$ -CD; dimethyl- $\beta$ -cyclodextrin; DM- $\beta$ -CD), and water-soluble cyclodextrin polymer ( $\beta$ -CDP)) and curcumin, a natural drug with anti-inflammatory, antimicrobial, and antioxidant effects. The hydrogel can maintain an adequate humidity level and releases curcumin (or other complexed drugs) at temperatures close to 40°C; therefore, it provides a promising wound dressing [68].

### 12.2.3.3 Comfort Properties

Incorporating CD into polyacrylonitrile by electrospinning imparts breathability, moisture-wicking, and anti-static properties [69]. The resulting composite is a fibrous membrane, in which factors such as hydrophilicity and pore sizes were affected by the proportion of cyclodextrin in the matrix. Hydrogen bonds between the hydroxyl of CD and the cyano group of polyacrylonitrile cause an increase in the viscosity of the spinning solution and a decrease in conductivity, facilitating the formation of pores between fibers. These pores significantly increase the breathability of the fabric. Moisture-wicking of the modified material was more than four times that of cotton.

## 12.2.3.4 Easy-Care Finishing

Easy-care finishing, common on cotton fabrics, is difficult on wool, particularly the durable crease generation and resistance to washing and shrinking. These limitations were circumvented by applying hydroxypropyl- $\beta$ -cyclodextrin (HP- $\beta$ -CD) with pad-dry technique and shrink-proofing polymers, extending its lifetime [70]. The application of cyclodextrin has minimal changes in the color of the fabric, while making the fabric with durable effects such as crease definition. In another protocol, cotton was modified with several different substances (glyoxal, ethylenediamine, and monochlorotriazinyl-cyclodextrin), by pad dry-cure method, generating a material with multiple and simultaneous properties (and some of them even barely compatible): improved wrinkle recovery angle, hydrophilicity, antibacterial effect, and breaking resistance, increasing comfort and resistance of the fabric [71].

## 12.2.3.5 Hydrophilic Finishing

Cyclodextrins can be used to make synthetic polymer surfaces hydrophilic [69, 72]. In a recent example, poly(ethylene terephthalate) (PET) was modified with  $\beta$ -cyclodextrin, previously oxidized with sodium periodate, together with chitosan, by the immersion-padding method [72]. In this case, the oxidized- $\beta$ -cyclodextrin acts as a crosslinker, binding to chitosan by imine bonds. Applying only chitosan or oxidized cyclodextrin in PET did not confer advantageous properties to the fabric, nor did they prove to be durable to washing, as they are not properly immobilized. However, when both reagents are applied to the PET fiber, a crosslinked structure is formed, highly resistant to washing and with hydrophilic properties.

### 12.2.3.6 UV Protection

Textiles can also acquire protection against UV radiation, for example, by applying  $\beta$ -CDs and a certain guest molecule, which is encapsulated and absorbs this range of radiation. In this sense, fabrics were functionalized with cyclodextrins and loaded with benzophenones (UPF: up to 51) [73], Michler's Ketone (UPF: 46) [74] ZnO nanoparticles (UPF: 50±10) [75], and Coumarin (UPF: 39,6) [76], for example. In these cases, the presence of CD generally increases the UPF both in relation to untreated fabric and the fabric treated only with the guest molecule. For example, the UPF of untreated cotton is  $6 \pm 1$ ; fabric impregnated only with ZnO nanoparticles is  $13 \pm 1$ ; while with ZnO:  $\beta$ -CD complex, the UPF rises to  $50 \pm 10$  [75]. The presence of  $\beta$ -CD is suggested to make the distribution of ZnO in the fibers more homogeneous (possibly due to a complexation between the metal oxide and the hydroxyl groups of CD), which significantly increases UV protection and wash durability (at least 5 washing cycles). With benzophenones as guest molecules, it is suspected that the CD cavity protects it from photodecomposition and photooxidation, in addition to reducing the energy required for keto-enol tautomerization and making energy dissipation more favorable [73].

### 12.2.3.7 Fire Retardancy

In some important cases, cyclodextrin functions as a compatibilization agent: 9,10dihydro-9-oxa-10-phosphaphenanthrene-10-oxide, a fire retardant, is not properly fixed alone in cellulose acetate butyrate by continuous melt spinning. However, when it is included in the form of an  $\gamma$ -CD complex, compatibilization of the fire retardant and the fiber is achieved [77]. Approximately 0.8 wt% of the fire retardant is enough to extinguish the flame; additionally, the retardant is not released, reducing organophosphorate toxicity. Phytic acid, a dihydrogenphosphate ester of inositol, was grafted on β-cyclodextrins and intercalated in Ni/Al layered double hydroxide, generating a composite, which was applied in polypropylene by melt blending technique, forming a flameproof fabric. The inclusion complex ensures dispersibility and compatibility with the PP matrix [78]. In another interesting example, a complex of ammonium phytate and cyclodextrin was applied to polyethylene terephthalate (PET) via the pad-dry-cure method, generating an intumescent flame-retardant material [79]. The addition of the complex significantly increased flame-retardant characteristics and char yield. The fire suppression mechanism is based on the decomposition of the material, generating incombustible gases such as NH<sub>3</sub> and H<sub>2</sub>O, which swelled the polymeric matrix and diluted atmospheric oxygen. Polyphosphoric acid is also generated on the PET fabric's surface, dehydrating and carbonizing PET into char residue. Additionally, the treatment improved the mechanical properties, such as the tensile strength, and proved non-irritating to the skin.

## 12.3 WASTEWATER TREATMENT

The complexing nature of cyclodextrins can be advantageous for treating textile wastewater. As cyclodextrins are water soluble, it is necessary to immobilize them on the accessible surface of a solid support material so that the adsorption system can be separated from the liquid phase at the end of the adsorption process.

### 12.3.1 Dye Adsorption

The mechanisms of dye removal from textile wastewater employing  $\beta$ -cyclodextrins are largely governed by host-guest interactions. In some cases, synergistic effects, such as host-guest interactions with CD and electrostatic interactions of the charged dye with polar groups of another material (such as chitosan), may occur [80]. Certain parameters must be considered in the adsorption process, particularly the pH of the medium, since it alters the charge of the dye and, therefore, its affinity with the CD cavity. Numerous cyclodextrin-based materials have been presented for the adsorption of anionic [80–85] and cationic dyes [48, 49, 85–97]. In an illustrative example, Oiu, Li [81] developed a dye- adsorption system based on an aerogel composed of cellulose ester and  $\beta$ -cyclodextrin ester, crosslinked with polyethyleneimine, an amino dendrimer. The obtained material was efficient in the capture of anionic dyes with an adsorption mechanism based on host-guest inclusions and the presence of abundant amine groups. An adsorption capacity toward methyl orange (MO) up to 1013.11 mg g<sup>-1</sup> at 25°C was achieved, and the material displayed antibacterial activity against E. coli and S. aureus. In addition, the material could be regenerated by elution with ethanol and NaOH, although the capture capacity in the second cycle decreased by about 16%, which was explained by the very high affinity of the sites for the dye, preventing complete desorption. Jia, Wang [86] presented  $\gamma$ -cyclodextrin based nanocubes crosslinked with phosphonitrilic chloride trimer, with outstanding adsorption capacities for methylene blue, bisphenol A, and Pb(II) of 2173.21, 998.54, and 966.06 mg g<sup>-1</sup>, respectively. The adsorption equilibrium was claimed to be reached only after 10-30 s, and the high adsorption characteristics of the material were attributed to the synergetic effects of host-guest interaction, electrostatic adsorption, hydrogen bond interaction, and chelation. The material could be regenerated up to 10 times by soaking it in HCl/methanol. In another memorable example, Rodríguez-López, Pellicer [82] used a combination of adsorption on β-CDs-functionalized magnetic polymer and subsequent photochemical degradation to remediate Direct Red 83:1 dye. The polymer could be magnetically separated, allowing easy separation of the adsorbed dye, which could then be desorbed, and the adsorbing material could be reused for at least six cycles without significant losses. The remaining dye in the desorption solution was eliminated through an advanced oxidation process using pulsed light and hydrogen peroxide.

### 12.3.2 Dye/Salt Nanofiltration

Nanofiltration membranes containing  $\beta$ -cyclodextrin were prepared and used to separate inorganic salts and dyes [98–100]. For example, a nanofiltration membrane, based on  $\beta$ -cyclodextrin and polydopamine crosslinked with glutaraldehyde, separated very efficiently a mixture of salts (NaCl and Na<sub>2</sub>SO<sub>4</sub>) from dyes (C.I. Direct red 23 and C.I. Reactive black 5). The CD cavity works as a transport channel, which allows transport across the membrane of salts, which have low hydration radius. The negatively charged dyes, thanks to the presence of sulfonic groups, have a larger size in aqueous solution and, therefore, almost do not permeate the membrane. Moreover, the membrane presented antimicrobial activity against *E. coli* and *B. subtilis*, which was explained by the hydrophobicity of the material, making it difficult for the colony to contact the surface of the membrane [98].

### 12.3.3 PHOTOCHEMICAL DEGRADATION (Advanced Oxidation Process)

Another method of remediating textile waste is the decomposition using electromagnetic radiation [101–103]. Tian, 2022 [101] anchored β-CD on ZnO/ZnSe heterojunction, by ester bonds and employed the resulting material in the degradation of two azo dyes, methyl orange (MO) and Congo red (CR). The addition of  $\beta$ -CD not only increased the surface area of the nanomaterial (thanks to its cavity), but also increased the energy absorption region, and allowed more efficient charge separation, which is important for the electron transfer process, and consequent photobleaching of the dyes. Dye degradation rates were 4.3 (MO) and 2.4 (CR) times higher than those of ZnO/ZnSe, without CD. The authors suggested that the higher photodegradation rate may be related to a host-guest mechanism involving the dyes and the radical species in the cavities of the CDs. In a comparable study, Yadav, 2022 [102] prepared a ternary nanocomposite Copper Oxide/Zinc Oxide with  $\beta$ -CD (CuO/ZnO/ $\beta$ -CD) and degradated malachite green and methylene blue. β-CD addition improved the recombination process of electron-hole, enabling photodegradation, with better efficiency than the composite CuO/ZnO. The higher performance of the CuO/ZnO/β-CD composite was also attributed to the formation of host-guest interactions involving the cavities of CDs and the dyes.

## 12.4 FINAL CONSIDERATIONS AND PERSPECTIVES

Cyclodextrins do not have any intrinsic properties that would benefit textile materials, but they serve as containers permitting the complexation of guest species such as perfumes, odors (bad smells) and with UV protection, antimicrobial, flame retardant, or insect repellent properties. Furthermore, the complex with dye molecules allows for improvement and optimizes the dyeing of textiles. Fixation of cyclodextrins to textiles for finishing purposes and smart textiles requires the introduction of reactive groups in cyclodextrins or combinations of CDs and crosslinkers with catalysts. It is still limited to pad-dry-cure application, while exhaustion application is not possible due to the lack of substantivity of CDs to most textiles. Although more articles on cyclodextrins with textiles have been published in the last few years, demonstrating rising interest, the principles of the functioning of CDs for textile applications have already been described long before, as detailed in our previous review on this topic in 2010 [41]. In fact, no relevant innovations have been reported since then, but new research has further explored the complex forming ability of CDs in more detail and upcoming newer materials such as nanomaterials and smart materials. The cost of CDs, although today, in general, widely available and much cheaper than at the beginning of their research, is still a relevant factor and must be weighed with respect to the achievable advantages and competitiveness. For example, using CDs in textile effluent treatment seems only justified if the material can be recycled a thousand times. Considering the relatively high cost of pure commercial CDs, it is astounding that almost no reports were made on non-purified CD mixtures or the less investigated CDs with more than 8 glucose units. Mixtures of CDs could even be advantageous in the case of trichromic dyeing, when three dye molecules with different structures and sizes must be retarded to achieve uniform dyeing. The same is valid for the functionalization of textiles when more than one target compound needs to be immobilized on a textile through complexation. Although using these macrocycles is promising, further advancements are still needed to improve the process.

## **ABBREVIATIONS**

CD Cyclodextrin

- owf on weight fabric (% mass product per mass textile material)
- NPs Nanoparticles
- UPF UV protection factor

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## DECLARATIONS CONFLICT OF INTEREST

The authors declare no conflict of interest.

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# 13 Enzymes for Sustainable Textile Processing Recent Advances

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## **13.1 INTRODUCTION**

Enzymes are proteins (with some rare exceptions, such as catalytic RNA) that act as catalysts to speed up chemical reactions. In nature, almost all metabolic processes are conducted through enzymes, making life possible [1]. Therefore, they can be obtained from animal organs, plant material, and microorganisms [2]. An iconic example is the case of orotidine phosphate decarboxylase, an enzyme involved in the biosynthesis of pyrimidine nucleotides, generating an enhancement of an impressive 10<sup>17</sup> in reaction rate. To put it into perspective, the uncatalyzed reaction in a neutral medium has a half-life  $(t_{i,j})$  of 78 million years; in the presence of the enzyme, the reaction occurs in just 18 milliseconds [3]. Enzyme-substrate interactions have first been described by the simple lock-and-key principle, proposed by Emil Fischer in 1894. According to this concept, the active site of a given enzyme has a threedimensional structure that is complementary to the shape of its substrate - similar to the way a key fits into a lock. The active site of the enzyme and the substrate have precise and complementary geometries (Figure 13.1a) that permit the enzyme to recognize and bind to the substrate in a highly selective manner. Once the substrate binds to the active site of the enzyme, conformational changes occur that cause a certain chemical reaction [4, 5]. A more modern model, the "induced-fit model," suggests that the binding of a certain substrate in the active site generates a change in the shape of the enzyme, resulting in a proper alignment of the catalytic groups on its surface (Figure 13.1b) [4, 5].

Enzymes are classified according to the type of reaction they catalyze. In summary, oxidoreductases catalyze oxidation/reduction reactions; transferases cause functional group transfer; hydrolases catalyze hydrolysis reactions; lyases cleave chemical bonds by reactions other than hydrolysis or oxidation; isomerases alter some bonds, generating an isomeric form; ligases generate covalent bonds and translocases catalyze the movement of ions or molecules across membranes, or their



FIGURE 13.1 Lock and key model (A) and induced fit model (B) adapted from reference [5].

separation within membranes [1, 2]. These designations can be subdivided according to the specific substrates of the catalysts. For example, amylase is a class of enzymes that catalyzes the hydrolysis (therefore, it is a hydrolase) of the polysaccharide starch *(amylum)* into oligomeric or monomeric sugars; while enzymes that cleave peptide bonds into amino acids are called proteases (also hydrolases). Lipases are hydrolases that catalyze the hydrolysis of fats, and cellulases hydrolyze the cellulose structure into mono- or oligosaccharides [2]. Enzymes have become important in developing modern and greener textile processing in the textile industry. Different enzymes are potentially used at different stages of textile processing in substitution to conventional chemical methods to make processes more sustainable and with lower environmental impact [6–8].

The manufacture of textile articles made from natural fibers begins on the farm, through the cultivation and harvesting of raw materials (cotton, wool, silk, among other fibers); undergoing fiber preparation and yarn manufacturing (spinning), fabric manufacturing (e. g. weaving and knitting), and then wet processing (Figure 13.2). Wet preparation refers to the series of chemical and mechanical treatments that are



FIGURE 13.2 Textile manufacturing steps [10, 11].

applied to fibers, yarns and fabrics to improve their appearance, performance, and functionality. It includes processes like desizing, scouring, bleaching, dyeing/printing, and finishing. Wet textile processing ensures that the textiles meet desired properties such as color, texture, durability, and other physical or chemical properties (hydrophilicity, strength, etc.) and can be divided into three main stages: pretreatment, dyeing, and finishing. Pre-treatment consists mainly of desizing (only for plain woven fabrics), scouring, and bleaching, but depending on the fiber, other specific procedures are included, such as carbonizing (for wool), degumming (silk), or mercerization (cotton) [9–11].

Some enzymes stand out in applications in the textile industry and are already wellestablished, while others have significant potential but are still under investigation. Amylase is industrially used for desizing purposes [11–18]; scouring can be done with pectinase [19–23], catalase decomposes excess hydrogen peroxide, after chemical bleaching [24] and cellulase are used for biopolishing and bio stoning purposes [25– 27]. Still on the scientific level, glucose oxidase is exploited in bleaching [28–30], laccase [31–34] and peroxidase [35, 36] are useful in the dyeing stage, cellulases generate improvements in hydrophilicity [37, 38] and xylanase is used for biopolishing [39]. Additionally, textile effluents can be remediated indirectly with the participation of enzymes using microorganisms [40]. For practical applications, enzymes can be obtained from microorganisms, which present several advantages, such as rapid production and the use of cheap substrates like agro-industrial or industrial wastes [7, 16, 19]. The present chapter will focus on the most recent publications on enzyme applications for textiles, which means the developments of the last five years, as previous investigations have been reviewed in detail on various occasions [41–46].

# 13.2 SUSTAINABLE PREPARATION OF TEXTILE FIBERS WITH ENZYMES

## 13.2.1 ENZYMATIC DESIZING

To prevent yarn breakage during weaving, the threads are coated with a substance called 'size', which reduces friction and strengthens the yarn. Starch is generally used as the film forming polymer thanks to its low cost and biodegradability. An aqueous solution of the biopolymer together with other ingredients is heated, and applied to form a film on the warp yarn, increasing its strength. This layer needs to be removed later, as it makes the surface hydrophobic, interfering with processes such as bleaching and dyeing. Amylase is ideal for this purpose, as it degrades starch (build-up by  $\alpha$ -1,4 glycosidic bonds between glucose units) selectively without damaging the cellulosic (contains  $\beta$ -1,4 glycosidic bonds) cloth structure [9]. Since starch is soluble at high temperatures, it is convenient to use extremophilic enzymes (extremozymes, thermophiles), allowing the reaction at high temperatures and reducing processing time [47, 48]. Extremozymes can be obtained from extremophiles or through genetic engineering. For example, a truncated α-amylase was found to have an activity 2.87-fold greater than full-length  $\alpha$ -amylase and greater thermostability, with an optimum temperature at 55°C, while full-length amylase has optimal activity at 45°C. It also performed better in removing starch from polystyrene, silk, and cotton fabrics [11]. Zafar, Aftab [12], working with an  $\alpha$ -amylase enzyme from *Thermotoga petrophila*, cloned into *E. coli*, observed optimal desizing conditions at 85°C, 6.5 pH, enzyme concentration of 150 U ml<sup>-1</sup>, for 1 hour incubation. In this case, in a neutral environment, almost 80% desizing was obtained, while this percentage, unfortunately, decreased significantly in acidic (pH 5) or basic pH (pH 8) by 50%. The activity and thermostability of an  $\alpha$ -amylase BLA from Bacillus licheniformis was improved by rational design, generating mutations, one of which improved nearly 3-fold in the specific activity at pH 8.5 compared to the wild-type. The mutant enzyme retained 75% residue activity after pre-incubation at 70°C for 30 min, indicating high thermostability [13].

Xylanase also has potential in the starch desizing of cotton, linen, and indigo-dyed cotton, as well as for bioscouring and biopolishing cellulosic fabrics. The enzyme was produced from solid-state fermentation of agricultural waste, emphasizing rice straw waste, by marine fungal isolate Trichoderma longibrachiatum. It showed optimal activity at 60°C [49]. Worthy of note, the starch-containing effluent itself, from the desizing stage, was used as feedstock to produce amylase enzyme using the fungus Trichoderma reesei. The amylase was then used in the desizing process, operating under optimal conditions at 80°C, pH 5, CaCl<sub>2</sub> 3 g L<sup>-1</sup>, for 45 minutes. Ideally, there is a waste recycling process in a circular model [15]. However, the recovered enzyme from the effluent caused stains on the fabric, reducing the interest in this technology for practical applications. In another example, textile effluent was used to extract Aspergillus species to produce extracellular amylase, which was produced by fermentation technique and later used in cotton desizing, with optimal conditions at pH 4.5 and 60–70°C [16]. The enzyme showed good activity at high temperatures (>80%) between 50 and 90°C); however, in a higher pH range, the relative activity of the enzyme decreased significantly: 60% at pH 5, and only 20% at pH > 7.  $\alpha$ -Amylase was immobilized on Chitosan (AC) and Eudragit® S-100 (AE) (a copolymer of methacrylic acid and methyl methacrylate), enabling reuse. The enzyme immobilization was carried out by covalent bonds; in chitosan, glutaraldehyde was used as a crosslinker, and in Eudragit<sup>®</sup> S-100, carbodiimide was used as a coupling agent [17]. The enzyme immobilized on chitosan (AC), as well as the native enzyme, had an optimum pH of 5.5–6, while for the enzyme immobilized on Eudragit (AE), the optimum was shifted to pH 7. The optimum temperature of the native enzyme and AE was  $55-60^{\circ}$ C. while for AC, it increased to 70°C. In immobilized enzyme-desized cotton, however, a decrease in enzyme activity when immobilized on Eudragit (62.7% retained for AE) or chitosan (41.5% retained for AC) was observed, requiring a higher concentration of the composite to be added to compensate for the decrease in activity. Due to the acidbase characteristics of the support materials, the enzyme immobilized on chitosan is insoluble in alkaline media, whereas the enzyme immobilized on Eudragit is insoluble at pH < 4. These characteristics allow for easy separation and reuse. After 5 use cycles, AE and AC maintained 52% and 45% of the original activity, respectively [17].

#### **13.2.2 ENZYMATIC SCOURING**

Raw cotton is mainly composed of cellulose, hemicellulose, pectin, wax, and moisture [50, 51]. While cellulose is in the fiber interior, non-cellulosic impurities are concentrated in the external cuticle and primary cell wall and make the fiber surface hydrophobic, interfering with dyeing, and must be removed. Traditionally, non-cellulosic impurities are removed chemically, e.g., with a hot sodium hydroxide solution. These harsh conditions, however, damage cotton structure more than necessary and generate large volumes of effluents. In the search for environmentally friendly conditions, with lower consumption of energy, temperature, and chemicals, the use of pectin degrading enzymes and ester cleaving enzymes like lipases, esterases, cutinases, or proteases have been successfully explored. Additionally, enzymatic processes were found to damage cotton fibers to a lesser extent than chemical processes, as they are more specific and are conducted under milder temperatures and pH conditions [41].

In a more recent research work, a knitted cotton fabric was bioscoured with a commercial pectinase (pectate lyase), to increase its wettability [20-22]. It was found that 1 g L<sup>-1</sup> of the enzyme is sufficient to remove pectin at pH 8.5 in 30 minutes at 55°C to achieve a hydrophilic textile. Due to the specificity of the enzymatic treatment, other impurities, such as waxy and fatty substances, are not removed, requiring subsequent washing with surfactant (0.2 g/L) at 70°C to guarantee melting and emulsifying these hydrophobic impurities. The incomplete removal of impurities leads to lower weight loss than the alkaline treatment, but the degree of whiteness of the fabric is lower than that of the chemical treatment, requiring additional bleaching for lighter colors or dyeing with a dark color. However, unlike alkaline treatment, which is aggressive and generates non-specific degradation of cellulose, enzymatic treatment produces fabric with less damaged fibers [21]. The same group [20] also investigated the reuse of the scouring bath in up to 10 bioscouring cycles. It showed that it was possible to maintain the level of pectin removal without significant interference in subsequent dyeing and quality loss in the dyeing results. However, the hydrophilicity (water absorption and wicking height) of the scoured fabric decreased after 2 use cycles, and nonuniform water drop absorption was observed, which was attributed to the redeposition of hydrophobic matter from the reused scouring bath. It was suggested that increased surfactant concentration after the second reuse could mitigate this problem.

Advanced techniques like ultrasound have also been explored in textile processing. Ultrasound is a sound wave with a frequency greater than 20 kHz, involving a cavitation phenomenon. This phenomenon generates microbubbles, microjets, and shock waves, facilitating micromixing, blending, and mass transfer [52, 53]. Ultrasound lowers the diffusion barrier and enhances the enzyme interaction with the substrate [23]. Patil and collaborators pretreated jute with ultrasound-assisted enzymatic scouring and obtained results comparable to the conventional, relatively timeconsuming method. The optimized conditions were 2.8% (owf) enzymes (a mixture of cellulase, pectinase, and xylanase), 1 g/L wetting agent, 55°C, and 10 minutes of operation at 40 kHz. As a result, they managed to remove hydrophobic impurities and improved water absorption in a shorter time than conventional scouring. However, parameters such as whiteness (52.4–54.1) and brightness (22.4–23.8) were inferior to enzymatic scouring without ultrasound (59.8 and 29.2, respectively), carried out for 45 minutes at 55°C [23]. Aggarwal, Dutta and Sheikh [19] used a similar strategy as described above for amylase production for the isolation of *Candida* species from textile mill effluent to produce a thermostable pectinase (maximum activity in the range of 40–0 C and more than 70% activity retained at 90°C), that was further used for enzymatic scouring of cotton (pH 5, 55°C) to obtain hydrophilic cotton fabrics (absorbency < 3s).

### **13.2.3 BLEACHING OF FIBERS**

A sustainable way of bleaching cotton fabrics is based on glucose oxidation with glucose oxidase (GOX), generating gluconic acid and  $H_2O_2$ , an active, *in situ* bleaching agent [28]. The enzyme was immobilized on chitosan, using glutaraldehyde as a crosslinker, enabling reuse. Whitening efficiency with the *in-situ* generated hydrogen peroxide was lower than conventional bleaching, possibly due to the interaction of hydrogen peroxide with glucose and, in the case of free enzyme, with the protein, leading to overstabilization. Only a 10% higher concentration of enzymatically generated peroxide was required for the immobilized GOX, instead of the 30% higher  $H_2O_2$  concentration (0.8–0.85 g L<sup>-1</sup>) needed for the free GOX, when compared to the conventional process (which uses 0.5 g/L), to achieve an equivalent degree of whiteness (CIE 60). With GOX enzyme immobilization, the  $H_2O_2$  generation improved the whiteness value so that 0.55-0.6 g L<sup>-1</sup> H<sub>2</sub>O<sub>2</sub> generated results equivalent to the conventional process. However, this efficiency was not durable, and in the 5th bleaching cycle, the enzymatic activity decreased to only 32% of the original activity, severely affecting the respective degree of whiteness [28]. Glucose oxidase was also used in the bleaching of merino wool fibers, with an improvement in the whiteness index and dye uptake compared to untreated wool [30].

Laccase from *Madurella mycetomatis* was heterologously expressed by *Pichia pastoris*, and subsequently fixed on a nanocomposite based on a metal-organic structure (MOF) coated with silica, and functionalized with glutaraldehyde, increasing its stability. The immobilized enzyme was tested in the bleaching of woven cotton fabric, demonstrating better performance than the free enzyme and the conventional bleaching procedure with NaOH/H<sub>2</sub>O<sub>2</sub>. The Berger whiteness index (WI) of fabric treated with immobilized enzyme is up to 1.37 times greater than that of the fabric treated with free enzyme and up to 1.63 times higher compared to the fabric bleached with NaOH/H<sub>2</sub>O<sub>2</sub> alone [29].

Another possibility to bleach cotton textiles enzymatically at a lower temperature than alkaline bleaching and with pH proximate to neutral is by *in-situ* formation of peracetic acid by arylesterase, as described in detail elsewhere [41]. In a recent publication, enzymatic bleaching with the commercial arylesterase Prima-Green EcoWhite and propylene glycol diacetate as peracetic acid precursor was combined with the natural surfactant sodium surfactin to obtain simultaneously a bleached and hydrophilic textile [54]. The enzymatic process was carried out at 60°C for 45 minutes, followed by rinsing at 85°C and an afterwash with catalase at 60°C to remove pectin and then excess peroxide. Very small amounts of 0.005-0.1 g L<sup>-1</sup> of sodium surfactin added to a nonionic commercial surfactant (1 g L<sup>-1</sup>) during the enzymatic bleaching were sufficient to lower water drop absorption down to 8.86 s to less than 1 s, respectively. Surfactin (Figure 13.3) is a lipopeptide with an extremely low critical micelle concentration of 0.0003%, and it was proposed that its efficiency in



**FIGURE 13.3** Structure of Surfactin A, a natural surfactant produced by *Bacillus subtilis*, a cyclic lipopeptide with surface active properties, that can be used in its sodium salt form in enzyme bleaching or in enzymatic anti-felting treatments of wool.

the process was due to slowing down the activity of the arylesterase, allowing the pH to remain elevated for a long enough time to allow alkaline scouring as in a traditional bleach. Indeed, 2 g  $L^{-1}$  sodium carbonate was added to elevate the initial pH, but not higher than pH 10; otherwise, the ester bond in surfactin might have suffered hydrolysis.

## 13.2.4 BLEACH CLEAN-UP

After the bleaching process, the remaining  $H_2O_2$  must be decomposed to avoid interference with the dyeing process. Catalase (hydrogen peroxide oxidoreductase) is commercially used for this purpose to substitute sulfur containing reducing agents. However, these enzymes generally exhibit limited stability in alkaline pH ranges. Recently, it was demonstrated that the immobilization of catalase derived from a psychrotolerant bacteria belonging to the *Serratia* genus increased this stability. Covalent enzyme immobilization was performed on a regenerated cellulose membrane, using a divinyl sulfone reagent. The immobilized enzyme exhibited relatively good stability at pH 7–10, and it was possible to reuse it for up to 4 cycles, while the native enzyme is unstable at pH higher than 9 [24].

# 13.2.5 ENZYMATIC PRETREATMENT AND ANTI-SHRINKAGE TREATMENT OF WOOL

Proteolytic enzymes combined with pre-treatment with a biosurfactant resulted in an anti-felting effect in the wool top. Specifically, PATO5<sup>T</sup> protease from the extremophile Bacillus patagoniensis, combined with surfactin (Bacillus subtilis O9 biosurfactant), reduced top felting tendency by 41% without significant impairment in wool tensile strength [55]. It is important to mention that synthetic surfactants such as CTAB remove lipids from the outer layer of wool, but on the other hand, they inhibit the action of the protease [56, 57]. Differently, treatment with surfactin improved (i.e., had a synergistic effect) the action of the protease. The costs of this procedure were minimized, using surfactin and proteases as crude extracts [55]. Wool was treated separately with two enzymes, laccase, and protease, followed by coating with biopolymers (chitosan, starch, and gum arabic), using pad-dry-cure [58]. As a result, the fabric presents lower area shrinkage (3.57%, in the case of protease and chitosan treatment) than untreated wool (16.37%). Furthermore, the treatment also reduces yellowness. It was suggested by the authors that the treatment of wool with enzymes results in the hydrolysis of cysteine disulfide bonds, and therefore there is selective cleavage of the cuticle surface, which subsequently binds to the biopolymer. In the case of starch and gum arabic, the interaction only occurs through hydrogen interactions, but chitosan has -NH2 and -OH groups, which react covalently with the -COOH groups, generating stable CONH bonds [58]. The results are in line with those obtained by Zhang, Huang [59] who treated wool with cutinase from Thermobifida fusca, followed by keratinolytic enzyme from Bacillus subtilis. In this case, the area shrinkage of the resultant fabrics decreased from 12.4% to 5.86%. In addition, the enzymatic treatment also removes the lipid layer from the fiber, improving wettability and dyeability with C.I. Acid Blue 80 dye. The K/S increases from 3.284 (untreated wool) to 6.492 (Cutinase-Keratinolytic treated).

# 13.3 ENZYMES IN THE DYEING OF TEXTILE FIBERS

Enzymes can modify the fiber's surface, making it more hydrophilic, or increase its affinity for the dye (chemical modification), or react with the dye. Furthermore, enzymatic processes for producing dyes are also relevant [60–65].

# 13.3.1 WETTABILITY AND IMPROVEMENT IN DYEING

Using pigments from natural sources in dyeing fabrics is interesting for several environmental reasons; however, generally, mordants are needed to fix them to the fibers, which mostly contain toxic metals, putting into question the favorable ecological argument. However, an inspiring method used laccase enzyme mediation to fix flavonoid dyes from *Hibiscus sabdariffa* flowers [31], St. John's wort and white onion peel extracts [32], green tea (*Camellia sinensis*) extracts [33] and hydroquinone [34] on cotton [31, 32, 34] or on wool [33]. In general, the phenolic residues present in the extract are enzymatically oxidized, generating radicals that undergo polymerization, generating a film that adheres to the textile surface [31, 32], or that react chemically with the fiber [33] or with a template [34]. However, with the chemical transformations of the

pigment, electronic changes occur, which lead to changes in color, and must be taken into consideration [32]. With the fixation of natural, phenolic dyes, UV-protection, antibacterial and antioxidant properties are usually achieved [33, 34].

Similarly, peroxidase was used to dye wool with aromatic substances [35], producing different colors such as blue, red, yellow, pink, and brown, depending on the precursor and pH, and silk with natural phenolic compounds [36], which obtained colors varying between yellow and shades of brown. Notably, the process is energysaving in some cases, enabling dyeing at temperatures as low as 30°C [35].

Enzymes can also be exploited to modify the surface of fibers, improving interaction with dyes. Lotus fabric, a cellulosic fiber originating from the roots and stem of *Nelumbo nucifera*, a perennial aquatic plant, was treated with cellulase, improving surface characteristics, including increased wettability, morphological changes, and roughness. The optimum conditions for the enzymatic treatment were 2% (owf) enzyme, pH 5.5, material-to-liquor ratio LR of 1:20, and 55°C, for 60 minutes. This treatment improved the absorption of natural dyes such as Azadirachta indica extract (Neem Tree), Jaipuri pink, and Apsara yellow. In the first case, the dyeing imparted antimicrobial properties to the fabric. Specifically, the treated fabric resulted in a growth reduction of 97% for *S. aureus* and 94% for *E. coli*, and the enzymatic pretreatment increased wash fastness [37].

Samant and collaborators treated cotton separately with three enzymes: acid cellulase, neutral cellulase, and xylanase; they later dyed the fabric with catechin from *Acacia catechu*. Enzymatic treatments improved the dyeability of cotton (with emphasis on xylanase), compared to chemically pretreated fabric, with good fastness properties. The treated and dyed fabrics also presented good UV protection and antimicrobial properties against *Staphylococcus aureus* (Gram-positive bacteria) and *Escherichia coli* (Gram-negative bacteria) [38].

Wool was dyed with green tea extracts (*Camellia sinensis*), with the inclusion of laccase and MgSO<sub>4</sub> [33]. In this case, polyphenolic components present in the extract, such as epicatechin, are polymerized by the action of the enzyme in the amorphous structure of wool, being immobilized thanks to covalent bonds and hydrogen interactions with the polymer, fixing the dye in the wool. Good to excellent rubbing fastness and washing fastness were obtained through dyeing with 1% (owf) enzyme and 1% (owf) MgSO4 for 60 minutes at 85°C. Under these conditions, a color strength (K/S) value of 3.392 was obtained. Increasing the amount of enzyme to 2% owf, the K/S increased to 3.571, but the textile presented less friction and washing fastness due to unfixed dye molecules on the fabric. The fabric dyed with tea extract also showed antibacterial activity (although the type of bacteria is not detailed), UV protection (UPF > 50, while undyed wool has UPF = 5.6), and antioxidant activity [33].

In addition to natural fabrics, enzymes have also been used to increase the hydrophilicity of synthetic fabrics. The surface modification of polyethylene terephthalate (PET) fibers using a commercial lipase (Lipolase 100L-EX, 100,000 U/g, Novozymes; LR of 1:80) at 5–15% (owf) for hydrolysis improved water vapor permeability, compared to the untreated fabric, as well as to conventional, alkaline-treated fabric. The enzymatic treatment also improved dyeability; as ester bonds were hydrolyzed, more functional groups were created, and the reactivity of the fiber was increased. As a finishing procedure, this process makes the fabric softer and causes its original shape to be quickly recovered after low-stress load. Unfortunately, the authors did not adequately describe the pH (Tris-buffer), temperature, concentration, and treatment time conditions of the enzymatic stage [66]. Cutinase-producing *Fusarium falciforme* was isolated from papaya peel and applied to modify polyethylene terephthalate, significantly improving its hydrophilicity [67]. The PET fabric was treated with 100 U of crude cutinase per gram of dry weight of fabric in 0.1 M Tris–HCl buffer at the optimum pH of the enzyme and for 24 h at a LR of 1:50 and with constant shaking at 125 rpm, which enhanced wetting time, water adsorption and moisture regain, to a degree comparable or better to conventional treatment with NaOH; consequently improving its dyeability and softness. More detailed experimental conditions for the enzymatic treatment are not provided, but it is understood that they are 35°C and pH 9.0, at which the enzyme activity is optimal. Given the high material-to-liquor ratio and the long treatment time, this approach is still far from industrially and economically viable.

# 13.4 ENZYMATIC FINISHING OF CELLULOSIC FIBERS

# 13.4.1 BIOPOLISHING

Pilling is the formation of small, unwanted balls or clumps of fibers on the surface of a fabric and arises from the entanglement of loose fibers, thanks to friction in processes such as rubbing, using, or washing. This process not only affects the appearance but also reduces the lifetime of the fabric. Biopolishing decreases the formation of pilling in fabric but also generates tensile strength loss [68]. Recombinant Xylanase II (XynA2) from *Caulobacter crescentus*, an aquatic bacterium, was applied to clean and degum denim jeans. When treated with 1 U mg<sup>-1</sup> XynA2 at pH 8 and 60°C for 12 h, the denim fabric presented a smooth and polished surface, with a dry loss of 4.9%. However, excessively long treatments (24 h) with the enzyme cause fabric damage (tensile strength loss) [39]. For a commercial application, certainly, a higher enzyme concentration will be necessary to reduce treatment time. Cellulase has also been successfully used to reduce fibrils in cotton, modal, and denim fabrics [25]. Improved mechanical properties were found with the treatment, such as increased tensile elongation and resilience and better comfort, such as smoother and more polished textiles.

# 13.4.2 TEXTILE FINISHING WITH IMMOBILIZED ENZYMES

Despite the broad consensus regarding their advantages of operating in milder conditions, consuming less energy and chemicals, producing textiles with better finishing quality, and generating less environmental impact compared to conventional processes, enzymatic processes have limited applicability, largely due to the high operating cost. Enzyme immobilization, on the other hand, has the potential to stabilize and allow separation and reuse for a greater number of operation cycles. Madhu and Chakraborty [69] performed complete cotton fabric pre-treatment, using enzymes immobilized on reversibly soluble-insoluble polymer supports. Commercial amylase, pectinase, and glucose-oxidase enzymes were covalently fixed to chitosan and Eudragit S-100<sup>®</sup>; and were used for desizing, scouring, and bleaching, respectively. Through the evaluation of parameters such as absorbency and whiteness, in addition to uniformity, color yield, and fastness ratings after reactive dyeing, it was demonstrated that the enzymes immobilized on these supports could be used for up to 3 cycles of operation without significant difference in the quality of the fabric. Side effects of treatments with fiber-degrading enzymes can be minimized by immobilizing the enzyme. When the enzyme is free, it can penetrate the fabric, degrade its structure, and reduce its mechanical resistance. This effect is reduced with immobilization, as it minimizes the enzymes mobility [27]. In an inspiring example, cellulase was immobilized in kaolin, reducing the loss of tensile strength of the knitted fabric and enabling the reuse of the enzyme without compromising the efficiency of biopolishing [26]. The enzyme was immobilized to kaolin by covalent bonds and adsorption, and the enzymatic treatment of cotton was carried out with a 1:30 fabric-to-liquor ratio, pH 5, 50°C, and 90 min of operation. Compared to the free enzyme, treatment with immobilized enzyme generated a similar or superior reduction in pilling but with less damage to mechanical properties, since the enzyme is restricted to the most superficial layers of the fabric [26]. These results align with those obtained by [27], who compared the treatment of cotton with free cellulase and with the enzyme immobilized in calcium alginate. Treatment with immobilized cellulase produces a better whiteness index and reduces the loss of tensile strength [27]. However, one should bear in mind that immobilization is generally costly and timeconsuming, and more than 10 reuse cycles will certainly be necessary to pay off the investment for immobilization. Furthermore, reuse raises additional organizational and logistic challenges.

## 13.5 PROCESS INTEGRATION

Conventional textile processing employs several steps, which consume chemical reagents and generate effluents that are generally unused, meaning economic losses and environmental impact. Process integration, in another way, can optimize the global production process, combining stages to reduce resource consumption, minimize waste generation, and increase overall efficiency and sustainability [70]. In the context of the textile industry, ideally, several steps would be performed in a single bath, reducing water consumption, minimizing waste generation, and making the process economically viable [6]. The combination of several processes mediated by enzymes has a certain limitation related to the conciliation of optimal operating conditions. Bleaching with H<sub>2</sub>O<sub>2</sub> occurs in alkaline pH [9], while enzymatic treatments are generally carried out in an acid-neutral environment. This limitation can be partly resolved using extremozymes, which function in wider temperature (thermophilic) and pH (acidophilic and basophilic) ranges [48]. A thermophilic lipase enzyme, used in the pre-treatment of wool to remove the lipid layer from wool, has maximum activity at pH 6. However, dyeing wool with anionic dye must be carried out at a more acidic pH (4.5), because the surface amines are presumably protonated, having greater affinity for the anionic dye. Consequently, as there is no coincidence in the optimal treatment conditions, the enzyme is not used to its maximum potential, which requires a longer operating time [71]. An interesting proposal is to couple desizing and scouring together with the bleaching step since the enzymatic treatment of starch with amylase generates monoglycols, such as glucose, as a by-product, which can be used to generate hydrogen peroxide, through treatment with glucose oxidase (GOX). In principle, there are savings in reagents, water, energy, and time, and less wastewater is generated compared to the multiple steps processes. Mojsov treated cotton with  $\alpha$ -amylase, amyloglucosidase, pectinase and glucose oxidase (GOx), in an "all-enzymatic" one-bath process [72]. The peroxide generated was also converted to peracetic acid by reaction with tetraacetylethylenediamine, TAED (an activator). Bleaching at pH 5 and 55°C ("non-activated" peroxide) produced a whiteness index (WI) of only 7.1. Increasing the temperature to 90°C and the pH to 10.5, the WI rose to 8.3. In the presence of TAED activator, pH 7.5 and 55°C, WI increases to 46.2; while conventional bleaching, using H<sub>2</sub>O<sub>2</sub>/NaOH, generated WI of 74.6 [72]. As can be seen, the degree of whiteness using enzymatic treatment unfortunately struggles to reach the degree obtained using conventional chemical methods. On the other hand, Udhayamarthandan and Srinivasan [18] obtained interesting results using mixed methods (enzymatic + chemical);  $\alpha$ -amylase desizing, alkaline scouring, and H<sub>2</sub>O<sub>2</sub> bleaching processes of cotton were combined into a single-step pretreatment. Not only operating time was saved, but also up to 80% of water and steam, in addition to reducing wastewater volume and parameters such as chemical oxygen demand, biological oxygen demand, total dissolved solids, pH, and turbidity. The treatment resulted in good desizing, scouring, and bleaching (whiteness index above 78), in addition to tensile and tear strength levels comparable to that of the two-step process. Wool was subjected to simultaneous scouring and dyeing with an anionic dye (C.I. Acid Blue 203), using a thermophilic lipase (TPL) from Bacillus aerius strain 24 K [71]. Using a dyeing bath with 1% (owf) of the dye, 2% (w/v) TPL enzyme, an LR of 1:50 and 70°C, at pH 4.5 a dye exhaustion of 76.9% was obtained after 3 h. This result is comparable with the conventional scouring process (77.4%) dye exhaustion, dyeing for 1h) and bioscouring in two stages (73.8% dye exhaustion, also dyeing for 1 hour). As can be seen, the integrated process requires a longer time to achieve similar results as the other processes mentioned.

## 13.6 SMART TEXTILES

The integration of enzymes with fabrics can be explored through a smart textiles approach to design clothes with functions beyond the usual ones, such as monitoring vital signs. A wearable sensor of medical interest was designed to detect glucose in sweat [73]. To this end, electrodes based on gold composite fibers were produced and coated with a thin layer of Ag/AgCl or Prussian blue (PB). The resulting material is highly elastic and suitable for use as a wearable fabric and is not influenced by strain. The PB electrode was then modified with glucose oxidase (GOx) to serve as the working electrode, while the gold electrode with Ag/AgCl served as the reference electrode. A glucose limit of detection (LOD) of 6  $\mu$ M was achieved, and a working range between 0 to 500  $\mu$ M. Non-invasive, real-time monitoring of glucose levels is possible, being of great use for diabetic patients. Nierstrasz and coworkers have been investigating inkjet printing of enzymes upon different surfaces, such as films, paper, and textile materials, to apply controlled small quantities of active

proteins to these surfaces for purposes such as antibacterial activity [74, 75]. The main challenge, according to the authors, is to retain enzyme activity, as in the ink paste, enzymes are exposed to diverse mechanical (shear) forces (relative high viscosity) and physio-chemical interactions. Printing of lysozyme was evaluated by the retained activity varying different print parameters such as printer system and printing temperature, print paste composition and enzyme concentration, where glycerol was found to be an adequate viscosity modifier [75]. In another approach, they fixed lysozyme to a plasma-treated PA 6.6 fabric via sequential printing of tyrosinase and lysozyme, where tyrosinase permitted crosslinking of the lysozyme via the tyrosine residue to the plasma-activated textile surface. The modified textile surface exhibited antibacterial activity, and repeated use of the printed fabric retained satisfactory activity up to four reuses and a month of cold storage, however, with decaying residual activity [74].

## 13.7 ENZYMES IN TEXTILE WASTE TREATMENT

Although this topic is beyond the scope of this chapter and the interested reader is referred to the more specific literature on biological effluent treatment and reuse of textile waste, we decided to include a more general overview. After the complex processing of textile raw materials through several stages (Figure 13.2), large quantities of water contaminated with organic (carbohydrates, dyes, fats and waxes, sulfur, phosphor, and chlorine-containing organic chemicals, etc.) and inorganic (salts, heavy metals, etc.) impurities responsible for high COD (Chemical Oxygen Demand), and BOD (Biological Oxygen Demand) and elevated pH and conductivity are generated [76]. These effluents must be adequately neutralized, treated, or separated. Methods commonly used by the industry involve physical-chemical procedures such as ozonization, oxidation, membrane filtration, ion exchange, adsorption, and coagulation. Biological methods are also well established and are interesting because they are cost-effective and environmentally friendly [40]. These treatments may involve (immobilized) enzymes directly, or microorganisms, which act through enzymatic processes. According to the type of targeted pollutants, enzymes such as proteases, cellulases, amylases, or lipases are selected (in practice, mixtures of them can be used), which break down these complex organic compounds into simpler, environmentally friendly substances. In the microbiological approach, mixed cultures of specific fungi or bacteria are selected. These metabolize pollutants by producing enzymes, which degrade them to generate simpler molecules. Through appropriate enzymatic treatment of textile waste, it was even possible to reuse water in agricultural cultivation [77].

Besides liquid textile effluents, the textile industry generates considerable amounts of solid industrial and post-consumer textile waste. Industrial fiber residues are mostly shorter, mixed with trash and non-fibrous materials, and of low value, while post-consumer waste is composed of dyed and finished fiber blends. Besides physical and mechanical separation and recycling, this solid waste may be separated or converted by enzymatic and chemical methods into monomeric compounds and residual undegraded polymers. For example, a blend of polyester and cellulose fibers may be separated by degrading the polyester through chemical alkaline treatment with the cellulosic fibers remaining intact or by hydrolyzing the cellulose fibers and recovering the PES fibers. Similarly, wool/cotton or wool/viscose blends can be separated chemo-enzymatically through the use of protease, recovering the cellulosic fiber fraction or sulfuric acid or cellulase, recovering the protein fiber [78–83]. Cellulose fibers can also be recycled through enzymatic depolymerization and valuing waste for use in pulp mills [84]. It must be considered that cellulose cannot be directly depolymerized by enzymatic action due to the high crystallinity and high degree of polymerization [85], requiring pre-treatments to reduce the crystallinity of this polymer.

## 13.8 GENERAL CONSIDERATIONS AND PERSPECTIVES

Enzymes are ubiquitous catalysts in nature, in various metabolic processes, and have found diverse potential in academic and industrial applications. However, sometimes, disadvantages are related to their limitations to narrow ranges of pH and temperature. These limitations can be, in some cases, solved by exploiting extremozymes that enable operation at high temperatures, leading to accelerated reactions and reduced operating time. Improved, thermoresistant enzymes can also be obtained through genetic engineering. Currently, enzymes in the desizing and scouring stages are well recognized and widely used industrially, as they do not damage the fabric structure, unlike chemical methods, and are economically feasible. Enzymatic bleaching, on the other hand, does not yet meet the whiteness requirements for light-colored or white fabrics as obtained by chemical bleaching. Combined enzymatic-chemical methods, e.g., supplementation of hydrogen peroxide from an external source to the enzymatic process, may be interesting. More advanced green technologies such as ultrasound present promising results, reducing reaction time through increased process efficiency, but to the best of our knowledge, application is still limited to a small scale. Some strategies stand out in the search for making enzymatic methods economically competitive with conventional methods. Some studies have demonstrated the feasibility of reusing the substrates and even the enzyme solution, reducing operating costs [20]. Obviously, this reuse must not compromise the quality of the final product, such as causing stains on the fabric. Immobilization of enzymes, on the other hand, has the advantage of allowing reuse for more than one cycle, in addition to generally increasing stability over wider pH and temperature ranges and less fiber damage, especially in the case of fiber degrading enzymes such as cellulases for cellulosic fibers or proteases for wool. However, immobilization is costly and time-consuming, and repeated use without significant activity loss is not always granted. Using enzymes generated indirectly through the cultivation of microorganisms, preferably consuming cheap substrates, such as agro-industrial or industrial waste, instead of commercial enzymes, reduces production costs. Some processes presented are still on a laboratory scale, considering the objectives of this review. To be applied on an industrial scale, they must be more economically and operationally competitive than conventional methods. It can be observed that many scientific investigations still use high liquor ratios (> 1:10), long treatment times (> 30 minutes), or high enzyme dosages that are impractical for industrial applications. The environmentally sustainable character, however, is undeniable. Effluents

from enzymatic processes are less contaminated with inorganic salts, and the global consumption of water, energy, and chemicals is considerably lower than conventional methods.

# **ABBREVIATIONS**

- LR material-to-liquor ratio (g fabric/mL liquor)
- owf on weight fabric (% mass product per mass textile material)
- PA 6.6 polyamide 6.6
- **PET** polyethylene terephthalate
- **UPF** UV protection factor The icons used in Figure 13.2 were designed by Freepik (https://www.freepik.com/icons).

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# DECLARATIONS CONFLICT OF INTEREST

The authors declare that they have no conflict of interest.

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# 14 Textile Dyeing Effluents Challenges, Characterization, and Sustainable Treatment

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# 14.1 INTRODUCTION

In today's era of rapid industrial expansion to meet human needs and desires, industries ranging from heavy to niche sectors are engaged in fierce competition to increase production capacity, capture market share, and boost revenue, all while striving to maintain profit margins [1]. The textile industry, for instance, comprises approximately 3000 fabric manufacturing and more than 5000 finishing facilities [2]. Textiles are among the biggest industrial domains, alongside chemical processing industries, food, refineries, and metals, regarding water consumption. The global growth of the textile sector has led to significant water consumption, particularly in the dyeing and finishing stages. For instance, a textile plant with a daily manufacturing capability of 8000 kg consumes around 1.6 million liters of water, with dyeing and finishing procedures accounting for nearly 25% of the total water consumption [3]. The cosmetics, textiles, rubber, paper, and printing industries daily produce substantial volumes of polluted water. To manage the harmful and intricate polluted water generated by these factories presents a challenge. Water contamination due to textile effluents is a great danger to economic progress. The released water contains highly dissolved and harmful materials, such as organic dyes and microbial pathogens, polluting the surroundings and diminishing the accessibility of potable water [4]. The intricate composition of dyes hinders their degradation, complicating wastewater treatment. The existence of organic substances and the harmful nature of the effluents discharged by textile sectors further harm the natural ecosystem [5]. Hence, acquiring insights and devising strategies for efficiently treating textile effluents to preserve the ecosystem is crucial. In recent times, substantial research has been developed into wastewater treatment. Physical, chemical, and biological techniques have been explored to eliminate pollutants and dyes from textile effluents [6]. Conventional techniques, including electrochemical, ozonation, and bleaching, struggle to remove color due to the aromatic structures of dyes used in textiles. These dyes resist degradation in the presence of radiation and under aerobic conditions, necessitating the development of sustainable and eco-friendly wastewater treatment methods.

## 14.2 CHARACTERIZATION OF TEXTILE EFFLUENTS

Characterizing textile effluents is crucial for devising efficient remediation techniques and process optimization [7]. The textile industry employs different precursors, like artificial fabrics, woolen, and cotton, resulting in effluent generation during preliminary treatment, dye application, pattern printing, and finishing processes, as shown Figure 14.1. Variables for characterization of textile effluent encompass COD, color, acidity levels, particulate suspension, BOD, ammonium nitrogen, cloudiness, organic nitrogen content, carbonaceous content, halogenated organic compounds, organic matter content, suspended solids, total phosphate content, sulfur content, nitrogen oxides, volatile particulate matter, water hardness, surface-active agents, basicity, lipids, dissolved solids, chloride ions, heavy metals, electrical conductivity, and organic nitrogen content [8, 9]. Notably, COD levels in composite effluents are very high, with primary pollutants arising from textile dyeing and end-stage processes. Heterocyclic dyes and aromatic compounds are prevalent in current textile practices [5]. Dyes consist of a dye carrier and dye group, wherein light exposure causes structural oscillation for light absorption, resulting in observable hues. Even low dye amounts can produce intense hues. Intricate and stable formations are found not solely in textile effluents but also in all types of intricate compounds. Dyes are structured into ionic and non-ionic categories. Ionic dyes encompass reactive and acidic varieties, while non-ionic dyes maintain dispersion as they do not break in aqueous media [10]. Examples of ionized dyes include acid red-B, methyl orange, and rhodamine-B. Additionally, dyes are categorized as basic, acid, disperse, direct, reducing, or reactive. Acidic dyes carry a negative charge, while basic dyes hold a



FIGURE 14.1 Characteristics of effluents released at each stage of the industrial procedure [7].

positive charge. Dyes are considered active when acidic and involve metals; reduced dyes if sourced from natural indigo, and disperse dyes if lacking ionic properties [11]. Direct dyes are highly favored due to their ease of utilization, versatility in color options, and cost-effectiveness. Most direct dyes feature di-azo and tri-azo structures, with azo dyes exhibiting the broadest color spectrum (comprising 60 to 70% of this dye class) [12]. Pigments, heavy metals, oils, sulfates, and surfactants are also found in textile effluents, posing risks to water purity and aquatic ecosystems. Heavy metals are employed in textile dyeing. Reports suggest that the metallic components within dyes contribute to coloration, enabling dyes to serve as fiber colorings. Heavy metals, like copper, chromium, arsenic, and zinc, present in small amounts, can attach to fibers and dyes, with detrimental environmental consequences [13]. The primary pollutants in textile effluents include elevated levels of suspended solids, COD, pH levels, thermal energy, coloration, and other dissolved compounds. Typically, textile effluents exhibit a vibrant hue and are characterized by large ratios of BOD to COD and significant salinity levels [14]. The ratio of BOD to COD in combined textile effluent is nearly 0.25, indicating a substantial presence of nondegradable substances. According to Paździor et al. (2019), the treatment of textile effluent involves the analysis of different effluents produced throughout the dyeing processes [15]. These pollutants are gathered under equilibrium conditions. Lower amounts of pollutants can be present in these wastewaters.

# 14.3 METHODS OF TREATING TEXTILE EFFLUENTS

## 14.3.1 PRIMARY TREATMENT

The initial phase of textile effluent focuses on eliminating coarse particulate matter, suspended solids, and surplus grease and oil [16]. This process begins with the screening of the wastewater for larger debris like plastics, rags, and small fibers, using both fine and bar screens. Coarse screening, with a diameter of 6 mm or greater, is the initial step to safeguard from the negative effects caused by waste materials, such as plastics, rags, etc. This is followed by two secondary filtrations with a mesh size of 1.5-6 mm and then 0.2-1.5 mm, further reducing particulate matter in the wastewater. Following screening, the wastewater undergoes settling to remove solid particles, with suspended particles being removed by a mechanical method. Neutralization is then employed to decrease the pH of the wastewater, typically utilizing chemicals like sulphuric acid to adjust the pH to the ideal range of 5–9 for treatment [17]. Sedimentation is employed in which suspended particles in a wastewater effluent settle down under the influence of gravity [18]. While efficient for some particles, simple sedimentation may not sufficiently remove colloids. Hence, settling may also involve coagulation, where chemicals like lime, ferrous sulfate, alum, and ferric sulfate are added to alter the functional properties of colloidal particles, causing agglomeration and settling [19]. The particles that have settled are subsequently gathered as sludge. Disposing of them presents one of the primary hurdles in the treatment process. In mechanical flocculation, the effluent is gently mixed over time to encourage the formation of denser particles that can then settle as sludge. This type of flocculation is often employed [20]. However, this method has drawbacks, including the danger of short-circuiting and difficulty controlling floc generation. Careful management is essential to ensure that sludge disposal does not reintroduce solids into the process.

### 14.3.2 SECONDARY TREATMENT

The secondary method primarily aims to lower the levels of BOD, oil, and phenol in effluents and regulate its color. This is predominantly achieved biologically through the action of microbes operating in the presence or absence of air. Aerobic microorganisms utilize carbon-based substances providing nutrients and energy, oxidizing organic solutes into water and carbon dioxide, and breaking nitrogen-rich material into NH<sub>3</sub> [21]. Various aerobic systems, such as biofilters, aerobic ponds, and biological treatment plants, are employed for secondary processes. Aerobic lagoons, a commonly utilized natural method, comprise large containers with polythene or rubber lining. Wastewater from the primary method undergoes aeration for approximately 3-6 days, after which the sludge generated is eliminated [14]. This process achieves an organic pollutant removal rate of 99% and phosphorus elimination rates of 15 to 25%. Additionally, ammonia oxidation is observed in aerobic ponds, and aquatic plants inhabiting the lagoon aid in further eliminating total suspended solids [22]. However, drawbacks include the substantial space requirements and the threat of microbial pollution. Biofiltration systems, another prevalent secondary method, predominantly occur in the presence of oxygen. Primary method effluents are sprayed over a filter bed, typically made of synthetic resin, coal, or polyvinyl chloride. A gellike membrane of microbes forms on the filtration bed, facilitating the conversion of organic substances into water CO<sub>2</sub>. Despite their space-efficient nature, the trickling filter method is expensive and may emit foul smells. Aerated sludge methods are widely employed, involving a continuous air supply of textile effluents within a tank to enable bacteria to break down dissolved and particulate organic material [23]. Some organic material is transformed into carbon dioxide, while the remainder is changed into newly grown bacteria. Sedimentation helps remove the sludge formed from the effluent, with some sludge recycling for microbial replenishment. Although this process achieves a BOD elimination rate of 90 to 95%, it is a lengthy procedure. Sludges generated from primary and secondary procedures pose significant dumping challenges, containing microorganisms and organic materials [24]. Treatment methods include aerobic and anaerobic processes. The aerobic method, which involves aerobic microorganisms and air, transforms sludge into organic matter, carbon dioxide, and water. Anaerobic treatment, without air and with anaerobic microbes, breaks down sludge into CH<sub>4</sub>, CO<sub>2</sub>, and organic matter.

## 14.3.3 TERTIARY TREATMENT

The tertiary method utilizes various techniques, such as reverse osmosis, ion exchange, and electrodialysis [25]. Electrolytic precipitation involves applying current to the effluent, leading to electrochemical reactions that cause metallic ions to stick to suspended solids, generating heavier ions that settle and can be eliminated further. However, a drawback is the need for prolonged contact time between the

effluent and the electrode. Reverse osmosis employs membranes to eliminate total dispersed solids, bigger particles, and ions from wastewater, achieving high efficacy (>90%) [26]. It's effective for treating textile effluents containing large amounts of salts from processes like cotton dyeing. Electrodialysis separates soluble minerals using electrostatically anion-specific membranes and electricity to transport ions across membrane filters [27]. Distributing multiple membranes across the system impedes the effluent flow, causing a build-up of ions captured or precipitated, leaving behind neutralized ions. Membrane fouling must be avoided by eliminating colloidal particles and suspended solids before electrodialysis [28]. Ion exchange involves passing effluents via layers of resin beads, either positively or negatively charged, to remove cations or anions. Photocatalytic degradation and adsorption methods are effective for decolorizing dyes. At the same time, thermal evaporation with sodium persulfate is an environment-friendly method, as it doesn't form sludge or release harmful gases in the process of vaporization [29, 30].

# 14.3.4 Advanced Oxidation Treatment Methods

Advanced oxidation processes (AOPs) have garnered attention for their efficacy in treating various suspended solids in effluents. These techniques offer efficient means to eliminate dyes and pollutants. Generally, AOPs encompass various processes like photocatalysis, ozonation, Fenton-like processes, and Fenton reactions [31]. Various kinds of AOPs, which generate hydroxyl radicals, are being closely explored for decolorizing textile wastewater. The high reactivity of hydroxyl radicals is due to their unpaired electrons, aiding in the oxidation of recalcitrant organic substances. The reaction rates observed for reactions comprising OH radicals in organic substance elimination typically vary from 109 to 1,010 M<sup>-1</sup> S<sup>-1</sup> [32]. Other oxidizing agents, like permanganate, sulfate radicals, chlorine dioxide, and hypochlorite, are employed for treating textile effluents. The electro-Fenton method combines the Fenton reaction with electrochemical processes. Pollutant breakdown occurs through the Fenton reaction in solution and oxidative reaction at the anode. In the electro-Fenton method, hydrogen peroxide is consistently formed within the solution via cathodic oxygen reduction in acidic electrolysis [33]. Moreover, this method reduces iron residue production through regenerating ferrous ions by reducing ferric ions at the cathode. A study comparing effluents treated with AOPs against those treated with the biological process using Pleurotus ostreatus and Fusarium oxysporum found that AOPs achieved COD removal efficacy of 79% COD. In contrast, biological treatments achieved 39% and 42% elimination rates of COD, respectively [14]. Photo-oxidation, a key mechanism in AOPs, operates at any pressure and temperature conditions without producing by-products. Essentially, AOPs generate OH radicals, which effectively degrade difficult-to-oxidize components [34]. These radicals are typically produced through compound formulations containing hydrogen peroxide, ultraviolet irradiation, ozone gas, titanium dioxide particles, ferrous compounds, electron beam exposure, and ultrasonic agitation. OH radicals are highly reactive, exhibiting a rapid reaction rate with electron-dense substances due to their redox potential of 2.33 V, surpassing that of traditional oxidizing agents [35]. The oxidation process involves an initial attack by OH radicals, followed by hydrogen removal and electron exchange.

The rate of the oxidation process hinges on three primary parameters: pollutant concentration, radical concentration, and oxygen availability. Maintaining adequate concentration of the radical species relies on factors such as acidic levels, temperature, ion presence, radical scavengers, and pollutant type [36]. The oxidization process follows first-order kinetics with a reaction rate coefficient of  $10^8-10^{10}$  M<sup>-1</sup>s<sup>-1</sup> and OH amount ranging from  $10^{-12}$  to  $10^{-10}$  M. The appropriate method varies based on effluent COD levels. Optimal hydroxyl radical generation necessitates selecting two or more oxidizing agents yielding the highest radical concentration.

# 14.3.5 BIOLOGICAL TREATMENT

The method of biodegrading dyes presents a sustainable solution for eliminating dye contaminants from textile effluents. This method is cost-effective and can operate efficiently within optimal timeframes. Biomaterials like microalgae, bacterial strains, fungal species, and yeast cultures can degrade and assimilate different artificial dyes for biodegradation purposes as shown in Figure 14.2. Many microbes have shown promise in decoloring and breaking down toxic dyes [37]. Biological methods are appealing, eco-friendly, and economically viable. Figure 14.3 shows the biological treatment for dye removal. Multiple bacterial strains have demonstrated effectiveness in dye removal via both anaerobic and aerobic processes. Studies dating back to the 1970s identified bacteria strains like *Aeromonas hydrophila*, *Bacillus subtilis*, and *Bacillus cereus* capable of degrading azo dyes [39]. The effectiveness of microbial decoloration is affected by the flexibility and effectiveness of selected microbes. Bacteria in this process have the ability to decolor which is due to the enzyme production. For instance, *Aeromonas veronii GRI* was separated from textile wastewater and showed



FIGURE 14.2 Different biomaterials used for dye removal [38].



FIGURE 14.3 The biological method for treatment of textile effluents [2].

efficacy in degrading methyl orange, particularly when supplemented with sucrose, yeast, and *Bacillus* biosurfactant. Researchers noted that dye removal was likely due to enzyme functions within cells and suggested that the presence of surfactants can enhance dye decoloration. Azo dyes, characterized by their electron-deficient nature and -N = N- chromophore moiety, pose challenges to degradation due to the presence of electron-deficient groups. However, bacteria equipped with diverse enzymes, including laccase, azo reductase, peroxidases, tyrosinase, NADH-DCIP reductase, and MG reductase, can decompose textile dyes [40]. Among these enzymes, laccases, azoreductases, and peroxidases play crucial roles in azo dye decolorization.

# 14.3.6 Hybrid Treatment Systems

Nearly all physical, chemical, and biological methods have been explored for treating textile dyes and their wastewater. Therefore, it is crucial to compare and combine these methods to create a robust system that is both effective and economically viable [41]. Hybrid treatment approaches demonstrate the potential to evolve existing methods into more innovative and cutting-edge methods, leveraging old methods while incorporating new technologies. These hybrid methods often involve cross-disciplinary approaches that optimize reaction variables and reaction rates uniquely [42]. For example, in a combined approach of a physicochemical and biological method, the utilization of a coagulant enhances biological color removal to some extent [43]. The choice between the biological-coagulation or coagulation-biological approaches relies on factors like coagulant dosage or amount of sludge [20]. Likewise, the latter method is effective for treating basic wastewater and toxic metals in textile wastewater. Researchers have developed intriguing hybrid approaches such as fuel cells with photocatalytic peroxide-coagulation, focusing on organic contaminant degradation and electricity production [44]. They utilized materials like titanium oxide and zinc oxide on carbon cloth, along with photocatalytic fuel cell photoanodes, achieving significant dye degradation. Additionally, various other techniques can be combined with biological methods.

# 14.4 CHALLENGES AND FUTURE PROSPECTS

While physical treatment methods are commonly employed for textile effluents, they often lack adaptability and are expensive and less efficient. The presence of different effluent substances can further hinder these methods, restricting their practical applicability. In contrast, the bacterial treatment method emerges as a low-cost and eco-friendly alternative, increasingly adopted for treating effluents generated from textile dyeing. However, removing color from textiles containing artificial and intricate dyes remains challenging. Each technique has its benefits and limitations as shown in Table 14.1. Further research efforts should focus on developing innovative

# TABLE 14.1 Advantages and Disadvantages of Different Methods Used for Textile Effluents [7]

Methods		Advantages	Disadvantages
Chemical	Chemical Coagulation	Rapid retention time and high dye removal rate.	<ul> <li>Significant expenses associated with chemical substances</li> <li>Necessity for pH regulation</li> <li>Managing the substantial volume of produced waste</li> <li>Encountering difficulties in the waste disposal phases</li> </ul>
	Fenton reaction	Effective elimination of different dye varieties in a brief timeframe.	<ul> <li>Significant expenses associated with chemical substances</li> <li>Necessity for pH regulation</li> <li>Managing the substantial volume of produced sludge</li> <li>Encountering difficulties in the waste disposal phases</li> </ul>
	Photocatalysis	Effective elimination of various dyes.	<ul><li>New Technique</li><li>Secondary products generation</li></ul>
	Ozonation	<ul><li>Effective elimination of various dyes.</li><li>Sludge is not generated</li></ul>	<ul><li>Reduced efficacies in the removal of dispersed dyes.</li><li>Small retention time</li></ul>
Physical	Adsorption	Effective elimination of various dyes.	Necessitates the utilization of reusable adsorbent materials
	Membrane filtration	Effective elimination of various dyes.	Generating dense sludgewith substantial quantities of untreatable waste.
Biological	Aerobic	<ul> <li>Effective elimination of azo dyes.</li> <li>Cost-friendly</li> <li>Sludge can be reused in the process</li> </ul>	Short retention period
	Anaerobic	<ul> <li>Economically viable</li> <li>Produced biogas can be utilized as a source of fuel</li> </ul>	<ul><li>Cell replication persists for an extended duration.</li><li>Generation of aromatic compounds</li></ul>

approaches for treating textile effluents, considering the combination of two or more methods, like AOPs and microbial treatments [45].

# 14.5 CONCLUSION

The worldwide ecological issues related to the textile manufacturing sector primarily revolve around water pollution from releasing harmful chemicals like dyes, solvents, bleaching agents, and other substances in various textile processing stages. Toxic chemicals can originate from raw materials and a diverse array of modifiers used in producing the final product. Textile industry effluents are typically characterized by their color and high BOD, TDS, TSS, and COD levels. Additionally, textile wastewater often contains a variety of dye mixtures, further complicating treatment efforts. Consequently, effectively treating textile effluents presents significant challenges. The release of untreated wastewater into aquatic bodies can impede the permeation of oxygen and light, resulting in adverse impacts on aquatic life. Various methods for dye removal are used, and each has its benefits and limitations. It was observed that hybrid systems offer the potential for achieving high levels of dye removal rate and are economically viable.

# DECLARATION

The authors declare that they have no known financial interests or personal relationships that could have influenced the work reported in this paper. Generative AI and AI-assisted technologies were used solely to enhance the readability and language of the manuscript. After using these tools, the authors reviewed and edited the content and take full responsibility for the final content of the manuscript.

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# 15 Natural Materials in Functional Finishing of Textiles

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#### **15.1 INTRODUCTION**

Textiles are an indispensable element in our daily lives. Three major textile processing steps are preparatory or pre-finishing, dyeing and printing, and finishing [1]. The initial stage, often referred to as the pre-treatment process (inclusive of steps like singeing, gassing, sizing and de-sizing, washing, bleaching, and mercerization), serves the purpose of purifying textiles, yarns, or fabrics, ensuring their readiness for dye absorption and further chemical treatments. in the following procedures. Dyeing and printing aim to impart colors or patterns to the textile substrate for decoration, distinguishing, and warning purposes. Thus, in most cases, the preparatory or prefinishing, dyeing, and printing are two necessary steps for textile production. Due to the diversifying demands and the growing health awareness from customers, additional functionalities such as antibacterial, anti-UV, and deodorant have been incorporated into textiles. For example, inorganic antimicrobials (e.g., Ag, Cu) and organic antimicrobials (e.g., quaternary ammonium salts, biguanides, haloamines, phenols) are effective against microbial attack on textiles, especially those in close contact with skin or under extreme usage condition. However, the resistance after long-term usage and the potential environmental hazards of some of them are questionable. Another representative example is the increasingly avoided or banned usage of halogenated flame retardants (FRs) on textiles. The quest for sustainability within the textile industry is intensifying, focusing on utilizing eco-friendly functional agents and polymer bases derived from renewable sources, driven by growing health concerns. The prevalent utilization of hazardous chemicals in textile manufacturing has been identified as a major contributor to global unsustainability. Moreover, the rapid depletion of fossil fuel reserves, predicted to occur within four to five decades due to excessive consumption, underscores the exigency to explore alternative resources. This shift toward renewable resources is crucial and immediate, as the bio-based product sector from such sources gains prominence amidst dwindling fossil fuel supplies, which serve as the primary raw material source for numerous industries. Agricultural processing industries, meanwhile, produce substantial waste in the form of residues and byproducts. Traditionally, these are either incinerated or buried, yet many residues hold untapped potential as feedstock for creating high-value goods. Regarding textile substrate, natural finishes are most commonly integrated with natural fiber over synthetic fiber, which is incompatible during application. To date, many studies and reviews have targeted the topic of natural dyeing of textiles. However, this review summarises the research progress on textile finishing (e.g., antimicrobial, UV protection, antioxidant) using the extracts, especially those obtained from plants, animals, and microorganisms.

# 15.2 CONVENTIONAL FINISHING OF TEXTILES AND THEIR CHALLENGES

As customers' interest in functional textiles is growing, the negative impacts of textile finishing have received great attention ever since. In terms of conventional finishers, there are two major challenges, i.e. the potential hazards to the environment and to the customers. The water effluent generated from the finishing step usually contains unfixed functional agents such as antimicrobials, FRs, as well as auxiliaries (e.g., surfactants, salts), and those would break the ecological balance if not properly handled. Owing to the robust governmental regulations concerning the ecological impact of textile wet processing, numerous conventional chemicals have been prohibited due to their detrimental effects on the surroundings. In addition, one significant difference between finishers against dyes in the effluent is the colorless nature, which makes them harder to be detected without advanced equipment such as High-Performance Liquid Chromatography (HPLC), GC-MS (Gas chromatographymass spectrometry) or additional detection methods. Apart from upgrading the utility of finishers through the optimization of the textile finishing process, searching for environmentally friendly substitutes becomes important. In the recent decade, active phytochemicals like saponins, tannins, flavonoids, glycosides, anthocyanins, etc., impart textiles with various specialty properties like antimicrobial, antifungal, UV protection, insect repellent, and aroma, which have gained popularity in both academia and industry.

# 15.3 FUNCTIONAL FINISHING OF TEXTILES USING BIO-BASED MATERIALS

#### 15.3.1 ANTIMICROBIAL FINISH

Textile items, particularly those with direct and prolonged contact with human skin, are prone to microbial invasion, as they can absorb nutrient-laden substances from bodily fluids. The attached microbial leads to the baleful propagation of opportunistic pathogens and adversely affects the surface appearance, decreases mechanical strength, and generates foul odor due to bacterial deterioration and pigmentation. Antimicrobial finish is of particular significance in terms of medical textiles such as surgical gowns, gloves, hospital furnishing fabrics, patient uniforms, etc. AATCC Test Method (TM) 100, JIS L 1902 are commonly used quantitative test protocols for antimicrobial-treated textiles. Despite the diversity of organisms employed in quantitative assessments, the standard practice involves comparing results against Gram-negative and Gram-positive bacteria. A broad range of substances are utilized for antimicrobial treatment in textiles, encompassing metal ions and their salts, nanosilver, organometallic compounds, phenolic compounds, triclosan, as well as quaternary ammonium compounds, all of which serve as synthetic antimicrobial agents commonly used in the textile processing industry. Bio-based extracts have gained a significant position in the antimicrobial finishing of textiles in the past few decades to eliminate the potential hazards of synthetic functional reagents to humans and the environment. Many bio-based antimicrobials from plants, animals, and microorganisms have been explored on textiles based on their specificity against microbes, i.e., antibacterial, antifungal, or/and antiviral. The following sections include the recent research progress in the bio-based antimicrobial finishing of textiles.

#### 15.3.1.1 Plant-Based Antimicrobials

The natural plant is a rich source of effective antibacterial phytochemicals, such as phenols, tannins, anthocyanidins, flavonoids, saponins, glycosides, alkaloids, carotenoids, and terpenoids [2]. Most of these compositions inhibit low molecular mass (<1000 Da), which is similar to that of synthetic dyes, thus making them available for direct application to textiles through the conventional exhaustion method. Zhou Y. et al. [3, 4] furnished silk with three flavonoids (Baicalin, quercetin and rutin) through a conventional adsorption. The adsorbing capability of flavonoids on silk, dependable on their chemical structures and treatment conditions, is significant in determining antimicrobial activity. They also found that the gram-negative strain (e.g., Escherichia coli, short for E. coli) is more sensitive to flavonoids-finished silk than the gram-positive strain (e.g., Staphylococcus aureus, short for S. aureus). Li Q. et al [5] applied grape seed proanthocyanidins to cashmere driven by ionic interaction confirmed by Sips and Langmuir models in this research. The treated cashmere shows 95% and 92% bacterial reductions against E. coli and S. aureus, respectively. Zhou Q. et al. [6] furnished wool fabric using tannin-rich waste/fallen leaves extract of Chinese tallow/Sapium sebiferum L. through simple adsorption technique total phenolics (gallic acid) and flavonoid (catechin), and found high antibacterial activities against E. coli and S. aureus. Fazlıhan YILMAZ [7] transferred the antibacterial feature of licorice (rich in phenolic compounds) to wool. The treated wool has a good antibacterial effect against gram-positive and gram-negative. Joshi et al. [8] used High Pressure Liquid Chromatography (HPLC) to extract antibacterial compounds from neem trees and further applied them to polyester/cotton blend fabric for antibacterial properties. The results showed that the antibacterial rate of polyester/cotton blend fabric treated by antibacterial compounds from neem trees was over 90% against gram-positive bacteria. The antibacterial activity is resistant to 5 times machine washings. Mohamed et al. extracted antibacterial ingredients from guava leaf powder. The results showed that the ethanol extract, water extract, and ethanol/water extract of guava leaves contained strong chemical substances, such as carbohydrates, tannins, flavonoids, alkaloids, anthocyanins, and glycosides. At the same time, the microcapsules made of guava leaf extract as the core were coated on cotton fabric, which had good antibacterial activity against many pathogens [9]. To date, new plant resources for the extraction of antimicrobials have been continuously explored. However, in most cases, the effective antimicrobial compositions are within the scope mentioned above. The difference lies in the dominant antimicrobial composition, which differs from plant species. It also needs to emphasize that polyphenol is commonly mentioned in the antimicrobial finishing of textiles, which includes flavonoids (flavonols, flavones, flavanones, anthocyanidins, flavanols, and also isoflavones) and non-flavonoids. Nonflavonoid compounds primarily encompass phenolic acids, classified into benzoic acid derivatives like gallic and protocatechuic, and cinnamic acid derivatives, prominently comprising coumaric, caffeic, and ferulic acids. Another significant group includes stilbenes, with resveratrol being the key member, available in both cis and trans configurations. Lastly, lignans are formed through oxidative dimerization of two phenylpropane units [10, 11]. Thus, the polyphenol finishing of textiles is not further mentioned in this text.

Recently, the exploration of substantial plant-based antimicrobial compounds, including lignin and tannin, integrated into textile materials has garnered attention. Lignin, an aromatic polymer predominantly found in plants' secondary cell wall thickening, exhibits molecular weights typically ranging from 1000 to 20000 g/mol. which are influenced by the extraction method employed. Despite its potential, the use of lignin as an antibacterial agent for fabrics remains an underexplored area of research. This naturally dark-colored phenolic substance is often eliminated during the cellulose fiber production process. However, lignin's presence of phenolic units and methoxy groups enables it to disrupt bacterial cell walls, thereby exhibiting antibacterial properties [12]. The concentration of lignin coating was determined by its Minimal Inhibitory Concentration (MIC), demonstrating its ability to hinder bacterial growth and curb proliferation within six hours of contact [13]. Nevertheless, the widespread adoption of lignin as an antimicrobial agent is hindered by its visually unappealing dark color on coated fabrics, which might deter consumers. Tannins, natural polyphenolic antimicrobial agents derived from plant polyphenols, interact with proteins, amino acids, alkaloids, and other polymers. Over the last few decades, there's been significant interest in utilizing vegetable-origin tannin-rich dyes for textile coloring. These compounds, with molecular weights ranging from 500 to 3000 Daltons, can be sourced from various plant parts like roots, barks, leaves, flowers, fruits, and shells. The high tannin content in certain plant extracts has sparked interest in their dyeing potential for textiles, conferring odor control, antimicrobial, antifeedant, and UV protection properties. Natural polyphenols' antimicrobial efficacy against bacteria, fungi, and yeast makes them appealing for use across multiple sectors, including pharmaceuticals, food, feed, dyes, cosmetics, and chemicals. Several mechanisms for tannins' antimicrobial effect have been proposed, including inhibiting extracellular microbial enzymes, depriving the substrates such as electrolytes, UV-absorbing material, proteins, etc., required for microbial growth or direct action on microbial metabolism through inhibition of oxidative phosphorylation. Ikigai and colleagues discovered that green tea extract's catechins caused 5,6-carboxyfluorescein release from phosphatidylcholine liposomes in bacteria, leading to cell death due to membrane disruption. They found that Gram-positive bacteria were more vulnerable to catechins than Gram-negative ones, possibly because the latter's outer membrane, rich in lipopolysaccharides and negative charges, provides greater resistance. Another proposed mechanism attributes the antimicrobial activity to hydrogen peroxide production [2].

Natural dye extracts generally harbor diverse phytochemical compounds that give rise to their antimicrobial properties. The effectiveness against bacterial species can be attributed to these bioactive elements. They disrupt the integrity of the cytoplasmic membrane, leading to cellular content leakage and damage to membrane proteins. Additionally, they contribute to the disintegration of the cell wall, decrease the intracellular ATP levels, curb cell division, obstruct ATP and DNA synthesis, coagulate the cytoplasm, and diminish the proton motive force in both gram-positive and gram-negative bacteria [14].

Beyond the direct application to textiles, combining plant-derived antimicrobials with inorganic chemicals can further enhance the antimicrobial functions. Zhou Y. et al. fabricated silver nanoparticles using flavonoids, ferulic acid, and chlorogenic acid and further applied them to silk [15–18]. They discovered that silk's antimicrobial activity and durability are greatly enhanced by combining them with silver. These plant extracts also acted as stabilizers and reducing agents during the generation of silver nanoparticles, which enables their green synthesis to be available for the fabrication of eco-textiles.

#### 15.3.1.2 Animal-Based Antimicrobials

Animal-based antimicrobials widely exist in nature including amino acids, natural peptides, macromolecular sugars, etc., among which, chitosan is the most commonly applied as/onto textile materials. Chitosan, initially identified by Rouget, ranks as the second most copious amino polysaccharide globally, following closely behind cellulose. Its inherent antimicrobial properties stem primarily from the dynamic interplay between its cationic amino groups and the anionic membranes of microorganisms. A widely endorsed theory postulates that the positively charged NH3+ units within chitosan's structure engage in electrostatic attractions with the negatively charged bacterial cell membrane. This interaction disrupts cellular integrity, leading to the seepage of intracellular components like amino acids, vitamins, and the like, which in turn disturbs regular metabolic functions, ultimately causing bacterial cell demise. However, chitosan is a non-soluble antibacterial agent, which can only inhibit the growth of bacteria, but it is difficult to kill bacteria adsorbed on the surface of the fabric. The primary challenges hindering the extensive use of chitosan stem from its limited solubility under neutral or alkaline environments, coupled with its inadequate adhesion to textile fibers, leading to poor longevity. To address these issues, a surge of international research endeavors has recently emerged, focusing on chitosan and the development of its derivatives, aimed at enhancing both its solubility characteristics and its capacity to effectively bond with diverse textile materials. Jiang et al. modified chitosan with n-butyraldehyde by the Schiff alkali method to obtain N-alkyl chitosan (N-CTS), and then N-CTS was etherified with CHPTAC-ethyl to prepare n-alkyl quaternary ammonium chitosan (N-CCTS). Through comparative experiments, it can be found that the antibacterial activity of N-CCTS finished rabbit hair fabric against E. coli and S. aureus was 99.9%, which is higher than N-CTS and CTS. In addition After 25 times' washing, the bacteriostatic rate still remains about 90% [19]. Lim SH et al. prepared water-soluble chitosan derivatives (HTCC) by quaternizing glycidyl trimethyl ammonium chloride with the amino group on chitosan. Then, under alkaline conditions, the fiber-reactive chitosan derivative (NMA-HTCC) was prepared by introducing functional (acrylamidomethyl) groups, which form covalent bonds with cellulose under alkaline conditions, on the primary alcohol groups (C-6) of the chitosan backbone. NMA-HTCC overcomes the defects of chitosan itself and the weakness of poor antibacterial activity, leaving effective growth inhibition of E. coli and S. aureus up to 20 min [20]. Moreover, chitosan has poor antibacterial effect on gram-negative bacteria. Therefore, antibacterial agents are usually prepared by chitosan modification or the use of metal ions or metal oxides to improve antibacterial activity. Malini et al. combined porous chitosan with green synthesized nano-zinc oxide to prepare chitosan/ZnO nano-composite film in order to enhance antibacterial activity. By the method of controlling variables, the antibacterial rate of the cotton fabric after antibacterial finishing against the gram-negative strain was higher than that of the gram-positive strain. At the same time, cotton fabrics also have a certain anti-fouling property [21]. Kiakhani M S. et al. prepared chitosan derivatives (Ch-NPs) containing various nanoparticles comprised of silver, copper, zinc, etc. Scanning electron microscope analysis showed that Ch-NPs successfully modified the wool surface. The modified wool has a high bacteriostatic rate against E. coli and S. aureus [22]. In the latest study, Zhang et al. synthesized a highly watersoluble chitosan-based polymer antibacterial agent CS-g-DMC. Then, CS-g-DMCI was cross-linked with cellulose via 1, 2, 3, 4-butane tetracarboxylic acid BTCA, and thus attached to the cotton fabric. The results of the antibacterial experiment showed that the antibacterial rate of cotton fabric after antibacterial finishing against E. coli and S. aureus reached 99.9%. Even after 10 times of washing, the antibacterial rate maintained about 95%. In addition, the hydrophilicity and breaking strength of the cotton fabric after antibacterial finishing have also been improved [23].

#### 15.3.1.3 Microbial-Generated Antimicrobials

In the recent decade, greater emphasis has been placed on the application of microbialbased antimicrobials obtained from the fermentation of microorganisms such as fungi and bacteria. A vast array of robust pigments, encompassing carotenoids, flavonoids, quinones, and rubramines, are synthesized by microorganisms, with fermentation offering superior pigment production and reduced waste compared to plant and animal sources. Consequently, the biofabrication of dyes and pigments through fermentation methodologies has garnered significant interest lately. The development of environmentally friendly antimicrobial treatments for textiles can be realized by employing natural substances like cyclodextrins. These ring-shaped oligosaccharides possess a water-attracting exterior and a lipid-compatible inner hollow. The usage of cyclodextrin and its derivatives, specifically  $\alpha$ -cyclodextrin,  $\beta$ -cyclodextrin, and  $\gamma$ -cyclodextrin, is on the rise within the textile sector [24]. Cyclodextrins (CD) were discovered in the late 19th century; beautiful crystals were observed from a starch digest of Bacillus amylobacter having the chemical composition represented  $(C_6H_{10}O_3)_3H_2O$ . Three major structures of CD are  $\alpha$ -CD  $\beta$ -CD and  $\gamma$ -CD with MWs are 972, 1135, and 1297 g/mol, respectively.

An abundant production of vivid crimson pigments was observed in a bacterial isolate obtained from marine sediment samples. Through 16S rDNA sequencing, the

strain was classified as a member of the Vibrio genus, showing the highest sequence similarity to Vibrio gazogenes. The structures of colorants were characterized by (Nuclear Magnetic Resonance) NMR Spectroscopy and (Liquid Chromatography-Mass Spectroscopy) Through LC-MS analysis, it was confirmed to be a blend consisting of prodigiosin, cycloprodigiosin, and heptaprodigiosin. These pigments demonstrated effectiveness in dyeing wool, silk, nylon, and acrylic textiles, displaying attributes akin to both ionic and disperse dyes, a correlation that aligns with their identified chemical structures. Upon heat exposure, the dye's stability exhibited a 15% decline in concentration. Notably, the colored fabrics exhibited antibacterial properties, effectively inhibiting the growth of E. coli and S. aureus bacteria within a 16-hour contact period [25]. Findings revealed that the color-treated wool textiles demonstrated a bactericidal effect, eliminating approximately 50% of S. aureus and E. coli after a 16-hour contact period. Conversely, the silk fabrics displayed a diminished effectiveness, while cotton fabrics demonstrated no antibacterial properties whatsoever. These observations confirm that the colorants impart antibacterial properties onto the fabrics. The reduced potency on silk and cotton can be attributed to the lower concentration of colorants on these materials, a consequence of their lesser dye absorption capacity. Notably, the cotton fabric dyed showed the least intensity in color [25]. Microbial-generated antimicrobials are a novel and promising category over the plant-based and animal-based. groups, thus showing great potential. Table 15.1 summarizes the common evaluation methods for antimicrobial properties.

#### **TABLE 15.1**

Classification	Feature	Standard Number	<b>Criterion of Acceptability</b>
Halo circle method	Simple and fast, but the results can't be quantified	GB/T 20944.1 AATCC 90 JISL 1902 8.4 ISO 20645	The zone of inhibition ≥ 0 mm, no or only a few colonies,
Parallel scribe method		AATCC 147	No colony growth in the area where the sample and AGAR were in contact
Absorptive method	Accurate, time- consuming and	GB/T 20944.2	Inhibition value $\ge 1$ or inhibition rate $\ge 90\%$
	high fare	AATCC 100	No clear rules
		JISL 1902 8.1 ISO 20743:8.1	3.0 > Antibacterial activity value ≥2.0 effective; Activity value ≥ 3.0, more effective
Shaking method	Accurate, widely used, time-	GB/T 20944.3	Staphylococcus and Escherichia coli ≥70%, Candida ≥60%.
	consuming and high fare	ASTME 2149	No clear rules
Transferring method	Precise, containing little water	JISL 1902 8.2 ISO 20645:8.2	3.0>Antibacterial activity value ≥2.0, effective; Activity value ≥ 3.0, more
Decalcomania	Precise, dry	JISL 1902 8.3	effective
method	condition	ISO 20743:8.3	

# Test Methods for Antimicrobial Property

#### 15.3.2 UV PROTECTIVE FINISH

The ozone layer, water vapor and atmospheric CO<sub>2</sub>, effectively shields the Earth from high-energy UV-C radiation and around 90% of UV-B, allowing only about 94% of UVA and 6% of UVB to reach the surface. These wavelengths play an essential part in vitamin D synthesis, photosynthesis, biological processes, and maintaining ecological balance. However, human activities have led to ozone depletion and climate change, resulting in increased penetration of UV-B, often referred to as 'leisure' rays, into the troposphere. This heightened exposure poses significant health hazards, including sunburn, cataracts, growth abnormalities, photokeratitis, premature aging, and cancer, affecting individuals of all skin tones. Occupational and recreational exposure to UV radiation, sought-after for its tanning effects, can become hazardous with prolonged exposure. Over time, tans can transform into carcinogenic agents, promoting tumor development. UV absorbers are compounds that selectively absorb ultraviolet light between 200-400 nm while remaining transparent to visible light, functioning as optical filters. Organic UV absorbers typically consist of aromatic structures with conjugated systems and functional groups like hydroxyl, carbonyl, and amino groups, serving as chromophores for their sunscreen properties. Notable examples include o-hydroxy benzophenones, o-hydroxyphenyl benzotriazoles, o-hydroxyphenyl hydrazine, and salicylic acid derivatives. Upon absorbing UV energy, these sunscreen agents undergo a photo-induced excited state intramolecular proton transfer (ESIPT), transforming into enol tautomers. They then stabilize by releasing excess energy as heat, reverting to their original form. Other mechanisms for regaining reactivity involve direct radiationless decay or intersystem crossing-induced deactivation, both preventing the formation of harmful radical species. Natural compounds abound with properties that effectively shield against ultraviolet radiation. A wealth of studies underscores the environmentally sustainable method of utilizing these natural substances to impart UV-protective qualities to textile materials. The efficacy of these natural sources in guarding against UV rays is largely attributed to the absorption properties of their integral functional groups. These include polyphenols found in tannins, flavonoids, anthraquinone, terpenoids, and the aromatic conjugated systems, predominantly derived from plant sources. Sarkar's research revealed that vegetable and animal dyes derived from madder, indigo, and cochineal significantly augmented the ultraviolet protection of cotton textiles [26].

It was observed that deeper hues and greater dye concentrations augmented the Ultraviolet Protection Factor (UPF), indicating a direct correlation between UV absorption and dye content. Researchers have also explored the dyeing potential and UPF of natural dyes obtained from agricultural waste, such as peanut skins and chickpea husks, which resulted in cotton, wool, and silk fabrics exhibiting high UPF ratings [27–29]. Another investigation disclosed that incorporating 5–20% lemon-grass oil into wool fabrics significantly boosted their UPF while simultaneously providing color, moth resistance, and a pleasant fragrance. Untreated and treated fabrics were assessed for their UPF values to highlight the difference. Following treatment with nano lignin, the UPF of untreated cotton (initially UPF = 6.18) and linen (UPF = 5.20) rose to 29.88 and 25.42, respectively. However, the UPF values of the

	United States Standard AATCC 183-2014	China Standards GB/T 18830-2009
Range of application	Fabrics in dry, wet, and stretched states	All fabrics
Sample size	Two (one dry, one wet)	Four
Sample placement	Each rotation was 45°, and a total of 3 tests were performed	Random placement
Experiment environment	Temperature (21±1)°C	Temperature (21±2)°C
	Relative humidity (65±2)%	Relative humidity (65±4)%
Wavelength of test/nm	280–400	290-400
Minimum wavelength interval/nm	2	5
Modified standard deviation	Yes	No
UV protection requirement	UPF ≥ 15	UPF > 40
	15–24 good	Average transmittance of
	25–39 better	UVA < 5%
	≥40 excellent	

## TABLE 15.2 Test Standard for UV Protection Performance

treated fabrics diminished after washing cycles due to the loss of nanolignin through leaching. The UV shielding attribute was linked to the presence of phenolic compounds, with nanolignin demonstrating superior effectiveness, due to its enlarged surface area [30, 31]. The ultraviolet protection factor (UPF) was determined for untreated and nano lignin-treated textiles to evaluate the difference. Untreated cotton displayed a UPF of 6.18, while linen had a UPF of 5.20. Treatment with nano lignin elevated these figures to 29.88 for cotton and 25.42 for linen. However, subsequent washing cycles led to a decline in the UPF of the treated fabrics, presumably from the loss of nanolignin. The obstruction of ultraviolet radiation was linked to the phenolic compounds present, with nano lignin demonstrating superior effectiveness, due to its expanded surface area [32]. Table 15.2 compares the United States Standard (AATCC 183–2014) and China standards (GB/T 18830-2009) regarding the measurement conditions and evaluation.

#### 15.3.3 FLAME RETARDED FINISH

The enhancement of textile flame resistance is crucial for technological applications, considering their prevalent use and associated fire hazards in daily life. To address these safety concerns, numerous flame retardant (FR) compounds have been developed, employing both conventional and unconventional methods, including inorganic FRs, nitrogen-containing FRs, halogenated FRs, and nanofillers. Nevertheless, most conventional FRs are non-biodegradable, posing an environmental challenge as they persist in landfills, resistant to degradation by soil or water microorganisms. Consequently, these substances can linger in the environment, potentially infiltrating the food chain, adhering to aerial particles, traveling over distances, and finding their way into freshwater

sources, food products, ecosystems, or even becoming breathable if suspended in the air. Over the last decade, substantial research has focused on applying bio-based FRs to textile finishes, such as small molecules like phytic acid and large molecules like chitosan, lignin, proteins, and deoxyribonucleic acid. These natural compounds have garnered attention due to their promising eco-friendly properties as innovative flame retardants, particularly for cotton, polyester, and their blend fabrics [33].

*Phytic acid*: Phytic acid (PA) is a prominent phosphorus storage form in industrial crops and is being extensively studied for its potential in developing flame-retardant (FR) textile materials. In particular, PA has shown promising results in preparing FR silk and wool fabrics, due to its ability to be adsorbed onto protein fibers through ionic attraction [34, 35]. Various PA-based FR approaches, like PA-doped hybrid silica sol [36, 37], PA/nano-TiO<sub>2</sub> organic-inorganic systems [38, 39], as well as PA/ chitosan and PA/PEI polyelectrolyte complexes [40, 41], have been designed to enhance the functionality of silk and wool textiles. Additionally, reactive phytate FR compounds with carboxyl [42, 43], epoxy [44], and ammonium phosphate groups [45] have been synthesized to improve the washing resistance of finished silk and wool fabrics through covalent bonding.

Chitosan: The research conducted by Pan and colleagues employed the Layer-by-Layer technique for constructing dual-layer assemblies on cotton fabrics, which comprised chitosan and phosphorated chitin [46]. These layers were applied in quantities of 5, 10, and 20. Their vertical flame propagation tests exhibited that 20-layer assemblies, synthesized with a high concentration of phosphorated chitin (2 wt. %), endowed the cellulose substrate with self-extinguishing capabilities, preserving nearly 90% of the fabric from combustion. Moreover, heat release rate and total heat release measurements from cone calorimetry tests (conducted under a 35 kW/m2 radiant heat flux) indicated reduced peaks for all treated fabrics, compared to untreated ones. Zhou et al. integrated chitosan and phytic acid into PLA, using two eco-friendly complexes - tannic acid/ferric salt and another compound [47]. They assessed the thermal and flammability characteristics of the PLA composites through thermogravimetric analysis (TGA), limited oxygen index (LOI), UL-94 test, and cone calorimetry. Additionally, they examined ultraviolet protection and tensile strength. Both PLA/TAFe and PLA/CTSPA displayed earlier weight loss and higher residual char than pure PLA. A 3 wt% addition of CTSPA increased the LOI value of PLA from 19.6% to 30.5%. In the UL-94 test, PLA/CTSPA and PLA/TAFe/CTSPA attained V-0 classifications with minimal dripping. Cone calorimetry results showed that incorporating 2.5 wt% each of TAFe and CTSPA significantly decreased the peak heat release rate and total heat release. Notably, the combination of TAFe and CTSPA was efficacious in curbing carbon monoxide production

*Lignin*: Bio-based lignin has emerged as a prominent flame-retardant additive, drawing significant interest due to its inherent capacity to produce a high char yield upon decomposition – a fundamental characteristic of effective intumescent flame-retardant systems. It is documented that lignin undergoes thermal degradation at temperatures exceeding 15°C, culminating in the formation of a thermally stable char at approximately 700°C. This char layer functions as a barrier, inhibiting heat transfer and oxygen diffusion, thus constraining fire spread and further ignition. The synthesis

of lignin-based flame retardants typically involves either physical blending, where lignin is directly mixed with polymers, or chemical modification, which entails altering lignin through chemical reactions.

Alginates: Naturally derived polysaccharides known as alginates have been extensively applied to creating functional materials due to their adaptability, eco-friendliness, non-toxic nature, and cost-effectiveness. A standout attribute of alginates is their inherent flame-resistant property, significantly contributing to their utilization in producing high-value, environmentally friendly fire-retardant materials. A novel, eco-friendly, and phosphate-free coating technique was employed by Pan Y. et al. [48] to create a flameresistant cotton fabric via a layer-by-layer assembly process. In a pioneering approach, cotton fabrics were enhanced by combining cationic polyethyleneimine with anionic alginate, subsequently interconnected with barium, nickel, and cobalt ions. This treatment resulted in a 28% decrease in flame propagation rate for the treated cotton, and this resistance was relatively preserved even after washing, mainly due to the exceptional fire-retardant properties of the metal-ion-crosslinked alginate. Thermogravimetricinfrared (TG-IR) and Pyrolysis-Gas Chromatography/Mass Spectrometry (Py-GC/MS) analyses revealed that alginate's pyrolysis follows a dual pathway of decarboxylation and esterification, significantly contributing to the material's flame retardancy. Although an initial investigation into the flame-retardant mechanisms of alginate, including the catalytic impact and synergy of diverse metal ions, has been conducted, more extensive research is suggested.

Proteins: Zhang et al. [49] developed a novel cotton fiber with antibacterial, antioxidant, and drug delivery capabilities by grafting gallic acid functionalized polylysine (GA-PL) onto cotton using a sustainable and cleaner method, addressing the need for versatile medical textiles with bioactive properties. Zhang et al. [50] employed a combined treatment of casein phosphopeptide (CPP) and metal ions to prepare environmentally sound and wash-resistant flame-retardant silk fabric, demonstrating enhanced flame-retardant properties with a LOI index of up to 28% and a damaged length of 11.0 cm. Zhang et al. [51] employed a dip coating technique with polylysine (PLL) biomolecule to enhance the hydrophilicity and antibacterial properties of polyamide (PA) fabric, utilizing a Schiff base reaction for improved washing durability, providing a novel approach to improve the comfort and functionality of PA textiles. Zhang et al. [52] synthesize the Maillard reaction products between polyglutamic acid and glucose to treat silk, demonstrating successful coloration, durable antibacterial activity, and good antioxidant activity. while also highlighting the potential for improving light fastness through UV-absorbing modifications.

*Deoxyribonucleic acid*: Alongi J. et al. [53] Deoxyribonucleic acid (DNA) from herring sperm has been employed as a novel flame retardant system for enhancing the thermal stability and flame retardant properties of cotton fabrics. Indeed, DNA could be considered an intrinsically intumescent flame retardant as it contains the three main components that are usually present in an intumescent formulation, namely: the phosphate groups, able to produce phosphoric acid, the deoxyribose units acting as a carbon source and blowing agents (upon heating a (poly)saccharide dehydrates forming char and releasing water) and the nitrogen-containing bases (guanine, adenine,

## TABLE 15.3 Advantages and Disadvantages of Various Flame-Retardant Finishing Processes

#### Flame Retardant Finishing Process

11000035		raranages	Disudvantages
Traditional craft	Dip baking method Dip drying method Coating method	Simple operation, flexible, universal, low cost	Genera durability, affecting the fabric feel, poor environmental protection
	Spraying method Grafting method	Various initiation methods, good durability	Complex reaction manipulation, damaging the fabric
New craft	Sol-Gel method	Low temperature reaction environment, fine flame retardant layer, good uniformity	Long reaction time, high cost
	Layer assembly method	Less affected by the substrate, allowing for precise customization	Multiple process steps
	Microencapsulation	Improving compatibility, maintaining flame retardant stability	Difficult to control shell thickness
	Plasma method	Energy saving and water saving, simple operation, high interface bonding strength, little fabric damage	Limited activation area, high equipment requirements

Advantages

thymine, and cytosine) that may release ammonia. The flammability tests in horizontal configuration have shown that after two applications of a methane flame for 3 s, the DNA-treated cotton fabrics do not burn at all. Furthermore, no ignition has been observed when exposed to an irradiative heat flux of 35 kW·m<sup>-2</sup>. Finally, an LOI value of 28% has been achieved for the treated fabrics as opposed to 18% for the untreated fabric. Table 15.3 displays the advantages and disadvantages of different flame-retardant treatment processes.

#### **15.3.4 OTHER FUNCTIONALITIES**

Aside from the prevalent scholarly investigations, there exist less-discussed instances of textile's natural treatment. This segment highlights several applications, such as odor control, fragrance, anti-pilling, anti-static, insect-repellent, and water-repellency, which may stimulate further exploration in this field.

*Odor neutralization finishing*: Amid rising living standards, health and sanitation have garnered significant attention. Scent sensitivity has reached new heights, creating a persistent societal demand for deodorization. This process seeks to maintain health and living conditions by controlling the release of malodorous substances from industrial and commercial operations. Despite this, the study of deodorizing

Disadvantages

properties using natural dyes remains limited. Typically, the concentration of ammonia gas - a typical odorant - is measured through the gas-detecting tube method.

Textile odor can stem from adhered bacteria. After use, these microorganisms flourish due to body sweat and warmth, leading to fabric odor. Rather than eliminating bacteria, most odor-resistant treatments hinder their growth. Natural dyes, derived from renewable sources, often carry a distinctive, agreeable scent. The plant-based chemicals within these dye extracts impart a pleasing aroma and confer bacterial resistance to the textile. Another way is to directly absorb odor from the external environment. Tang R.-C. et al. applied natural lac dye to chitosan fiber to prepare healthy and hygienic textile materials. They found that the dyed chitosan fiber exhibited markedly enhanced antioxidant activities and deodorizing performance toward ammonia, which increased, obviously, with an increase in application concentration of lac dye [54]. Kim H.-D. et al. [55] extracted four kinds of natural colorant solutions from amur corktree, Dryopteris crassirhizoma, Chrysanthemum boreale, and Artemisia, and applied them to cotton, silk, and wool. They discovered the deodorizing performance of fabrics dyed with various natural colorant extracts was 34-99% and increased in the order of cotton < silk < wool. The highest value of *Chrysanthemum* boreale might be due to the main component flavonol of Chrysanthemum boreale, which could react with ammonia-based foul odors through the sensory (neutralizing) reaction mechanism. The deodorizing properties of Sappan wood, Black tea, Peony, Clove, Gardenia, Coffee sludge, Cassia tora L., Pomegranate) on cotton, silk, and wool fabrics have also been investigated [56, 57].

*Aroma finishing*: Attaining textiles with a sustained fragrance release capability is both an appealing commercial objective and a substantial challenge in textile chemistry and engineering. This endeavor encompasses dual hurdles. First, the precise quantity of fragrance compound applied to the textile must exhibit exceptional fastness. Secondly, the scent discharge should occur steadily and systematically. Historically, incorporating the scent during conventional dyeing, finishing, or printing techniques produced fragranced fabrics, albeit with limited fragrance retention, particularly after washing.

In alignment with ascending global trends and evolving individual consciousness regarding health and aesthetics, specialized value-added textiles, including those imbued with fragrance, are witnessing substantial growth. Essential oils, prevalent across diverse sectors, often necessitate encapsulation due to their high volatility and chemical instability. Despite the proposition of multiple substances, like silica, melamine formaldehyde, and urethanes, as encapsulating agents, these solutions often entail ecological concerns, complex procedures, and elevated costs. Consequently, cyclodextrins (CDs) have emerged as a promising, eco-friendly, secure, straightforward, and relatively cost-effective alternative as molecular containers. They are extensively utilized for the efficient encapsulation of aromatic and volatile substances. In recent times, extensive research has been devoted to aromatic functional textiles integrated with  $\beta$ -CDs [58]. However, the thermal treatment can give contradictory effects on washing durability of textile fragrant finishing. Higher temperature and longer curing time usually increase fastness of capsules on textiles, with decreased aroma inside the capsules, because the higher the temperature in curing, the more vitalization of fragrance.

Antistatic finishing: The electrostatic occurrences in cashmere and wool items are frequent, primarily due to the charging that results from friction on their hydrophobic scale layers, an issue exacerbated in arid and cold conditions. Researchers have explored numerous ionic and non-ionic compounds to enhance the humidity of textiles, thereby mitigating static buildup. Among these, quaternary ammonium compounds serve as prevalent antistatic agents in the textile sector. Nonetheless, their lack of durability and potential ecological and health hazards associated with bioaccumulation pose concerns. Recently, there has been growing interest in developing eco-friendly and biodegradable functional agents derived from natural plants for textile processing. Our research team endeavors to impart anti-pilling attributes to cashmere fabric using grape seed proanthocyanidins (GSPs). We observed that the half-life periods diminished from 3.2 seconds to less than 0.5 seconds under 65% relative humidity, attributed to the enhanced hydrophilicity of the treated cashmere. Furthermore, the pilling grade of the treated cashmere escalated to above level 4, a result of reduced fiber entanglement stemming from the increased fiber surface smoothness [5].

*Insect repellent*: Bites from insects like mosquitoes can inflict discomfort, inflammation, and even sickness, with mild symptoms typically involving itchiness, inflammation, and pain. Annually, a significant number of fatalities occur due to mosquitoborne diseases, among which malaria is prominent. To combat this issue, researchers worldwide have been exploring and developing mosquito-repellent textiles. While potent synthetic repellents exist, there's a growing need for environmentally sustainable alternatives, due to ecological concerns. Numerous natural substances, extracted from plant parts, such as roots, stems, leaves, flowers, fruits, and seeds, exhibit mosquitorepelling properties. However, the efficacy of these repellents depends on the quality of their essential oils. A key challenge for researchers lies in devising a controlled release system for these natural oils to prolong their effectiveness while maintaining eco-friendliness.

Several natural mosquito repellents have emerged, including citronella oil, castor oil, clove oil, eucalyptus oil, cedar oil, rosemary oil, peppermint oil, lemongrass oil, geranium oil, and Chrysanthemum extracts. In recent years, extensive research has focused on incorporating insecticides into textiles intended for outdoor activities. Typically, insect repellent textiles are created by applying repellent compounds onto the finished fabric, necessitating the use of binders that can adhere to the material and sustain the mosquito-repelling agents. Alternatively, repellents can be integrated into fibers or yarns during their production, especially in synthetic fibers, to create insectresistant textiles before the manufacturing process [59].

#### 15.4 CONCLUSION AND FUTURE PERSPECTIVES

Although great achievements have made on the natural finishing of textile, there are several research expectations placed on this topic. First, the adsorption mechanism of natural finishes to textile has not yet been comprehensively investigated. A full understanding of the adsorption of natural finishes with various structures on textiles substrates is helpful for the parameter determination in practical application. Second, one key challenge is the low affinity of these compounds to the textile substrate, which not only affects their loadings on textiles but also leads to poor fastness during laundering. Mordant is one common measure, however, the heavy color change (distinctive from natural dyeing) and the potential hazards makes it not a good option in the presence of natural finishes. Thus to develop fixer or crosslinker suitable for natural finishes is one research direction. Third, unlike natural dyes, the resource for natural finishes is limited and expected to be explored. Further, how to reutilize the agro-waste for extraction to fulfil the requirement for a circular economy is needed. Fourth, the combination of sustainable technology (e.g. ScCO<sub>2</sub> technology) and green chemicals (e.g. enzymes) are anticipated. Last but not least, the small amount of textile natural finishing is insufficient in standard evaluating methodology, making it hard to compare the effectiveness between different strategies.

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# 16 Natural Plant Extracts for UV Protective Textiles

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#### 16.1 ULTRAVIOLET RADIATION AND THEIR EFFECTS ON SKIN

The recent population explosion and rise in environmental pollution have significantly increased temperatures in tropical regions. Consequently, consumers have become more conscious of the importance of sun protection because it is directly linked to damage and heightened exposure to UV light. Solar radiation is the primary energy source and necessary component for the sustenance of the human species. Ultraviolet (UV) radiation is a type of electromagnetic radiation and constitutes about 5% of the total sunlight that reaches Earth. In contrast, visible light makes up approximately 50%, while infrared radiation accounts for around 45% [1]. Despite its relatively small fraction, it possesses the highest quantum energy compared to other forms of radiation. Depending on the biological activities, wavelength, and physical properties, the UVR spectrum comprises UVA (320-400 nm), UVB (290-320 nm), and UVC radiation (100–290 nm) [2]. The energy of ultraviolet light is comparable to the bond energy of organic molecules, resulting in significant harm to human skin. UVA rays, with a wavelength of 320-400 nm, induce a conversion of melanin precursors in the skin, resulting in fast pigmentation. This process occurs within a few hours, but the effects are limited and short-lived. However, it infiltrates the dermis, also known as the actual skin, resulting in accelerated aging characterized by decreased suppleness and the appearance of lines and wrinkles. UVB rays, with a wavelength range of 290-320 nm, have a shorter wavelength but higher intensity. These rays can penetrate the skin to a depth of a few millimeters. As a result, they cause the cells in the outer layer of the skin to create a stable pigment, leading to reddening (erythema) or sunburn. UVB is widely believed to be a primary factor in developing skin cancer, as it hinders the production of DNA, RNA, and proteins. Ultraviolet radiation with the shortest wavelength (100-290 nm), known as UVC, is extremely harmful to human skin. However, these rays are absorbed by the ozone layer, resulting in reduced damage [3].

#### **16.2 ULTRAVIOLET PROTECTION MATERIALS AND PRODUCTS**

Medical professionals recommend many methods to guard against UV radiation, including applying sunscreens, avoiding direct sunlight during peak hours, and wearing protective gear like wide-brimmed hats to shield the eyes, face, and neck [4]. Clothing is also considered the most effective method for shielding the skin from UV radiation, as stated by the World Health Organization (WHO) and the U.S. Environmental Protection Agency (EPA) [5]. Hussain and Jahan [6] elucidates that clothing can shield the skin from solar radiation by the fabric's ability to reflect, absorb, and disperse solar wavelengths. The effectiveness of appropriate clothing in providing protection is determined by the composition of the fibers (whether they are natural or synthetic), the way the fabric is constructed (including its porosity, weight, and thickness), and the treatment given to the fabric during wet processing (such as the use of dyes, UV absorbers, and other finishing chemicals) [7, 8, 9]. Research indicates that cotton, rayon, and flax are less effective in absorbing UV radiation than polyester, the most effective UV absorber. Wool, silk, and nylon also have better UV absorption properties [10]. The UPFs are influenced by the characteristics of the fibers, which differ in their UV transparency [11]. Untreated cotton fabric has a greater Ultraviolet Protection Factor (UPF) due to natural colors, pectin, and waxes that absorb UV radiation. In contrast, bleached cotton fibers are more transparent to UV radiation. Linen and hemp, in their raw state, have a UPF (Ultraviolet Protection Factor) of 20 and 10 to 15, respectively. However, even with lignin content, they are not completely effective in protecting against UV radiation. According to many studies, cotton fabrics, when dyed, exhibit increased Ultraviolet Protection Factor (UPF), while undyed, bleached cotton shows significantly lower UPF values. Protein fibers have varying effects in facilitating UV radiation. Wool has significant absorption in the wavelength range of 280-400 nm and extends beyond 400 nm. Mulberry silk experiences more significant deterioration compared to muga silk. Aliphatic polyamide fibers have lower absorption in the UVA and UVB areas than polyester fiber [12, 13]. The textile industry benefits from natural fibers as they are renewable resources, and their export can positively impact many economies. Promoting the utilization of natural fibers is advocated because end-use items derived from them are ultimately obtained from organic sources. Summer attire typically consists of fabrics such as cotton, viscose, rayon, linen, polyester, or a blend of these materials. Consumers typically prefer lightweight, non-synthetic textiles such as cotton and linen for summer clothing [14]. These fabrics have desirable qualities such as having good absorbency, being eco-friendly, and being nonallergic to skin, as Crews et al. [10] mentioned. Cotton is plentiful, and its mechanical characteristics are highly suitable for the manufacturing of clothing [13]. As these natural fibers/textiles exhibit less UV protection, UV protective agents like titanium dioxide and ceramic materials can be used for textile finishing, which helps increase their protection value [15, 16]. In recent times, natural products such as natural plant extracts in textile finishing have increased in popularity, which is attributed to their environmental friendliness and absence of drawbacks associated with their synthetic counterparts, such as high cost and negative ecological impact if produced ethically [17]. According to reports, many natural dyes not only impart distinctive and refined colors when dyeing but also offer UV-protecting properties to fabrics [18]. Consequently, a growing interest has been in incorporating UV protection qualities into textiles by utilizing natural substances. Further, as there are different varieties of plants, it is also essential to understand the potential of plants, their extracts, and their bioactive components in providing medical properties, especially UV protection.

#### **ROLE OF PLANT EXTRACTS IN PROTECTION FROM** 16.3 UITRAVIOLET RADIATION

Life on Earth depends on sunlight, and one of the important components of sunlight is ultraviolet radiation, which helps maintain ecological balance. With the increase in health issues due to adverse effects of UV radiation, synthetics, and chemicals, there is a growing demand for products developed through natural and safe materials. Understanding the available product categories for consumers to protect themselves from UV radiation, it is evident that materials like natural plant extract are a potential option for developing sustainable and suitable products for different skin types [19–21] [Figure 16.1]

#### NATURAL PLANT EXTRACTS: AN ALTERNATIVE APPROACH 16.4

#### 16.4.1 MEANING AND SIGNIFICANCE OF NATURAL PLANT EXTRACTS

Plant extracts are often regarded as the foremost sources of biomolecules, which can be extracted from different portions of plants. Plants with medicinal properties are utilized to extract biomolecules through various solvents and extraction procedures [22–24]. Natural extracts serve as colorants, functional food items, nutraceuticals, perfumes, preservation agents, edible/non-edible oils, and fats in the cosmetic, pharmaceutical, and food sectors. Natural goods are inherently intricate. Hence, it is imperative to consider many factors for different applications, such as significance, chemical properties of the primary chemicals involved, and potential modes of operation [25]. Natural plant extracts have emerged as a promising alternative for UV

Increase in health problems			
	Adverse effect of synthetic	;	
1. UV rays	drugs and chemicals	Society looking for	
2. Microbes (Svobodova et al., 2003)	1. Human being	natural products	
	2. Environment	Natural fibers vs. synthetic	
	(Kumar et al., 2011)	Drugs and other products prepared from natural sources	
		( <i>Khattak et al., 2014</i> )	

#### . . .

#### FIGURE 16.1 Need for natural and safe products.

protection. Historically, various cultures have utilized plant-based remedies for skin protection against the sun. The appeal of natural plant extracts lies in their diverse bioactive compounds, often referred to as phytochemicals, which exhibit antioxidant, anti-inflammatory, and photoprotective properties. These compounds act as a natural defense mechanism in plants, and when applied to the skin, offer similar protective benefits.

# 16.4.2 HISTORICAL USE OF PLANTS FOR MEDICINAL PROPERTIES AND SKIN PROTECTION

The utilization of plant extracts for safeguarding the skin has a long history, spanning numerous millennia. Due to their possible medicinal and defensive attributes, diverse societies have integrated botanical components into their skincare regimens. [26]. Various cultures across the globe have utilized botanical extracts for the purpose of skincare, drawing upon traditional wisdom and empirical observations. Here are some examples.

- *Ancient Egypt*: Egyptians were known for using plant-based oils and extracts for skincare. Red ochre ground in water was used on lips and cheeks. Henna, Olive oil, and aloe vera were used as colors on palms and hair, moisturizers, perfumes, and materials to protect from harsh environmental conditions [27].
- *Ancient China*: Traditional Chinese medicine has a long history of utilizing herbal extracts for various health and beauty purposes. Ginseng, green tea, and licorice root are historically used plants for their skin protective properties [28, 29].
- Ancient Greece and Rome: Olive oil was a popular skincare ingredient in ancient Greece and Rome. It was used to moisturize and protect the skin from the sun and wind-drying effects. It was also used as soap, perfume, and anointment for the dead [27, 30].
- *Ayurveda in India*: Ayurveda, the traditional system of medicine in India, has a rich history of using plant extracts for skincare. Ayurvedic practice is around 3000 years old, with a long history of managing disease. The 3 basic principles, called *doshas* (*vata*, *pitta*, and *kapha*), are derived from 5 elements of Indian philosophy [31]. Neem, turmeric, and sandalwood are some of the plants traditionally employed, along with saffron, which is also known and used for its antioxidant properties [32].
- *Native American Traditions*: Native Americans used aloe vera to soothe and heal the skin and hydrate and protect it from extreme climates in areas like deserts. It was also used to treat sunburn and for making soap. Other plant and plant extracts include cranberry and ginseng, to name a few [33].
- *Traditional African medicine*: Different African regions have a rich tradition of using plant extracts for skincare. Shea butter, for example, is derived from the nuts of the shea tree and has been historically used for its moisturizing and protective properties [34]. Another plant named Achyranthes aspera is used as an antioxidant and anti-inflammatory agent [35].

#### **16.4.3 PLANTS WITH UV PROTECTIVE PROPERTIES**

Natural products serve as a reservoir of novel chemical variations and are highly favored in contemporary times. Various plant products, including extracts derived from different parts of plants such as roots, stems, leaves, flowers, fruits, and seeds, possess features such as UV protection. Phenolic acids, flavonoids, and anthraquinones derived from plants have been recognized as effective sunscreen agents, due to their ability to absorb ultraviolet rays in the UV region [36]. In vitro analysis conducted on animal models and human subjects proves that administering certain flavonoids can mitigate the deleterious effects of ultraviolet radiation (UVR) on the skin. A study conducted by Gupta in 2005 shared the positive potential of an extract derived from the Q. infectoria plant that provided effective protection against both UV radiation and common bacteria. Another plant that is well known for its medicinal value is Aegle marmelos (Linn.) Correa, also known as Bael, is reported to exhibit several medicinal properties, such as antioxidant, antimalarial, radioprotective, anti-inflammatory, and antiviral. Phytochemicals such as flavonoids, alkaloids, sterols, tannins, phlorotannins, and flavonoid glycosides are extracted from the leaf and exhibit free radical scavenging activity [37]. Psidium guajava tree, also known as guava, possesses medicinal properties as the leaves and bark of the plant are extensively used in ancient therapeutic medicine and are popular in today's time as well because of the presence of rich polyphenolic compounds, which are responsible for antioxidant properties [38]. As an example from marine life, red algae are reported to show better UV resistance than terrestrial plants. Mycosporine is a fungal metabolite with a 310 to 320 nm UV absorption range and is substituted with amino acid residues. They are biosynthesized by the shikimic acid pathway for synthesizing aromatic amino acids, protecting aquatic organisms against solar radiation. Their strong UV-UV absorption maxima, photostability, and antioxidant nature make them one of the strongest compounds for sunscreens and therapeutic effectiveness [39]. Singh et al. [2] suggested that using chitosan to join MAAs together or with MAA-nanoparticles can provide stability to the product [40]. In a study by Gupta [41], various plants were examined to determine their efficacy in protecting against solar radiation. Methanol was used to make extracts from several plants, including their leaves and fruits. These extracts were then analyzed using a spectrophotometer to get absorption spectra within the 200-400 nm wavelength range. The ultraviolet radiation protective properties of the leaves of Mentha piperita, A. indica, Oscimum sanctum, aloe vera, and the fruits of Lycopersicon esculentum and Carica papaya were examined. Out of the materials examined, L. esculentum (tomato pulp) exhibited the highest absorption level in both the UVA and UVB regions, although all other extracts exhibited the highest level of absorption in the UVC range, followed by UVB, and UVA. Furthermore, there has been a discussion regarding using naturally derived plant compounds as sunscreen resources. This is due to their ability to absorb ultraviolet rays in the UVA and UVB range and their antioxidant properties. There is compelling evidence that ultraviolet (UV) light, which damages DNA, causes the dermal tissue of plants to accumulate flavonoids and other phenolics that absorb UV light. These compounds possess exceptional antioxidant and photoprotective capabilities. The results demonstrate that active constituents accountable for

UV absorption can be extracted from plant sources and utilized to provide enhanced sun protection. *Using* plant extracts not only offers defense against environmental risks but also preserves the environment, prevents pollution, and promotes the production of eco-friendly textiles, ensuring that they are accessible to individuals and the general public, thereby fostering genuine awareness. These textiles mitigate pollution by avoiding toxic chemicals commonly employed in the finishing process. Additionally, eco-friendly finishing promotes tree plantation, as herbs and plant parts serve as the primary raw material source. Furthermore, the trash generated by ecofriendly finishing acts as a natural fertilizer for soil and plants, serving the same function [42].

#### 16.4.4 PHYTOCHEMICALS IN PLANT MATERIAL

Natural plant extracts are rich in compounds with many therapeutic properties, including UV protective properties. These non-nutrient bioactive plant compounds are referred to as phytochemicals (phyto meaning "plant"), and they are responsible for plant protection against environmental hazards, which include pollution, drought, microbial attacks, and UV exposure [43, 44]. On the other hand, the study of natural products is called phytochemistry, and the science of applying indigenous remedies, including plant-based treatments, is called ethnopharmacology [45]. Phytochemicals are considered an important part of the medicinal world. They are not essential nutrients and are not important for sustaining life in the human body, but they help in fighting diseases. The phytochemicals exhibit many protective effects by undergoing different mechanisms such as antioxidant properties (antioxidants neutralize reactive oxygen species generated due to UV radiation, thus reducing DNA damage and oxidative stress); anti-inflammatory effects (protect skin by reducing the inflammation induced in the skin resulting in swelling and redness due UV exposure); DNA repair as well as protection (reduce skin cancer and repair DNA lesions); collagen synthesis (to maintain skin structure and elasticity, phytochemicals promote collagen synthesis); melanin production (phytochemicals contribute in the skin's defense mechanism against UV damage, as they influence melanin production, and hence play an important role in protecting the skin by absorbing and dissipating ultraviolet radiations); UV absorption (inherent property of phytochemicals to absorb UV rays, prevents it from penetrating in the skin). Numerous works have been done to explore the use of these materials to protect the human body [46], and a concise overview of the results from prior research on the ability of plant phenolics to protect the skin against damage caused by UV radiation also suggests their potential in this area. Skin exposure to UV radiation produces many reactive oxygen species (ROS). These substances can interact with DNA, proteins, fatty acids, and saccharides, resulting in oxidative harm. These injuries cause several detrimental effects, including disrupted cell metabolism, changes in the structure and function of cells, disruption of regulatory pathways, and modifications in the growth, division, and death of skin cells. These mechanisms can result in photo-aging and the formation of skin cancer. Using antioxidants as photo-protectors is one method to shield human skin from the detrimental effects of UV irradiation. Naturally occurring herbal chemicals, including phenolic acids, flavonoids, and high molecular weight polyphenols, have garnered significant interest as advantageous protective agents in recent years. [47]. Here are some key phytochemicals present in plants with UV protective properties and their roles:

- Tannin: From a chemical point of view, it is difficult to define tannins since the term encompasses some very diverse oligomers and polymers. It might be said that the tannins are a heterogeneous group of high molecular weight polyphenolic compounds with the capacity to form reversible and irreversible complexes with proteins (mainly), polysaccharides (cellulose hemicelluloses), pectin, alkaloids, nucleic acids, and minerals. Based on their structural characteristics, it is possible to divide the tannins into four major groups: gallotannins, ellagitannins, complex tannins, and condensed tannins. Tannins are reportedly present in grapes, blueberries, chocolate, legumes, grasses, etc. The tannins are used as anti-inflammatory, antioxidant, diuretics as well as used as caustics in the dye industry for cationic dyes, especially for textiles [48]. Previous studies revealed the potential antioxidant and UV protective properties of natural active ingredients of Chinese bayberry tannin [49]. To detect the presence of tannin in the plant material, a ferric chloride test is employed, in which a few drops of ferric chloride are added to the plant extract, and the presence of a blackish precipitate confirms the presence of tannins [50, 51].
- Flavonoids are polyphenolic compounds present in many beverages, fruits, and vegetables and provide color and aroma in fruits; they attract pollinators in flowers [52]. They play an important role in protecting plant tissues from damage through UV radiation and act as UV filters. These compounds are reported to exhibit multiple biological properties, including antimicrobial, cytotoxicity, anti-inflammatory, and antitumor activity. Among the above-mentioned properties, almost every group of flavonoids (especially luteolin and catechins) can act as powerful antioxidants, protecting the human body from reactive oxygen species and free radicals [48]. Current studies also reveal the application of flavonoids in chemoprevention [53]. To analyze the presence of flavonoids, a lead acetate test is employed, and the occurrence of yellow color precipitates after adding lead acetate solution confirms the presence of flavonoids [50, 51].
- Tocopherols (Vitamin E): Tocopherols are antioxidants that protect cell • membranes from oxidative damage caused by UV radiation. They help in maintaining membrane integrity and reduce the risk of lipid peroxidation. Vitamin E is a lipid-soluble vitamin found in various foods, particularly soy, nuts, whole-wheat flour, and oils. Numerous cutaneous benefits have been demonstrated when vitamin E is applied topically. The most important property of vitamin E is its strong antioxidant capacity. The term "protector" has been used to describe the actions of vitamin E and its derivatives because of their ability to quench free radicals, particularly lipid peroxyl radicals. Several studies have indicated that they can reduce UV-induced erythema and edema. Clinical improvement in the visible signs of skin aging has been linked to reductions in both skin wrinkling and skin tumor formation. Tocopherol and its acetyl ester derivative, tocopherol acetate, have been studied extensively. While tocopherol is the most active form of vitamin E, topically applied vitamin E esters have also been shown to penetrate the epidermis [54].

#### 16.5 SOLVENT SELECTION FOR EXTRACTION

The extraction of plants involves the use of solvents, and the selection of solvent determines the quality of the extract obtained. The solvent should be selected by considering the plant type and part, the phytochemical required, and the availability and sustainability of the solvent. The solvents are categorized as polar and nonpolar solvents. These solvents are used for extraction in regard to the end product as methanol, and ethanol is polar solvents and are used to extract polar compounds. In contrast, hexane and dichloromethane are non-polar solvents and are used to extract non-polar compounds [55, 56, 57]. The solvents, along with their polarity, are listed in Figure 16.2. Bubalo et al. [58] shared the application and utilization of green solvents such as supercritical and subcritical fluids (e.g.,  $CO_2$  and water) as well as natural deep eutectic solvents, which have the potential of extracting the bioactive compounds from plants and can contribute in sustainable development.

#### **16.6 EXTRACTION METHODS**

Extraction is a process that involves the penetration of a solvent into the plant material (grounded, powdered, and coarse) to solubilize and dissolve the secondary metabolites in the solvent used for extraction. This method helps enrich the solvent with the extracted components for further use [60]. With innovation in science and technology, numerous extraction methods are developed for using the potential of plants. A few of the methods are discussed below:



FIGURE 16.2 Polar and nonpolar solvents [59].

#### 16.6.1 MACERATION

Maceration is an extraction method in which plant material (powder or coarse form) is soaked in a selected solvent and kept at room temperature with intermittent agitation for at least three days [61]. After extraction, the micelle is separated through filtration, which is then followed by evaporation of the solvent [62]. This method is convenient and appropriate for plant materials that are thermolabile [63].

# 16.6.2 DECOCTION

This technique is useful to extract the phytochemicals that do not depose with the rise in temperature. The powdered plant material is added to a suitable container, and water is poured. The heat is applied, and the solution is boiled for at least 15 minutes to expedite the extraction. The heat exposure duration depends on the plant material type used and the photochemical to be isolated [61, 59].

# 16.6.3 PERCOLATION

Percolation is one of the well-known processes employed to obtain fluid extracts. It basically means "passing the solvent or liquid through any solid in small quantity; to be more specific, drop by drop." This process involves using an apparatus known as a percolator, a narrow cone-shaped glass vessel with openings on both ends. The plant material required in this extraction technique is coarse and should not be completely powdered. This point needs to be taken care of during the extraction process because the fine material will later hinder the separation of extract and plant material [61]. The percolation process is reported to be used as a common technique to prepare Chinese medicine, which follows three simple steps of extraction: adding the plant material to a percolator, slow addition of solvent, and collection of extracted solution. The advantage of the percolation process includes simple equipment, easy operation, and the possibility to extract thermally unstable components [64].

## 16.6.4 SOXHLET EXTRACTION METHOD

The Soxhlet extraction method employs the use of a thimble, water cooling system, reservoir, bypass tube, siphon tube, round bottom flask, as well as heating mantle. Before starting the extraction process, the plant material needs to be placed in the thimble (prepared with Whatmann No. 1 filter paper), which is then loaded in the Soxhlet chamber, and the solvent is added to the round bottom flask. As the solvent is heated, its vapor moves to the column and reaches the chamber; it enriches the solvent with extractive substances. This solvent filled with extractive components moves through the siphon tube in a round bottom flask, thus emptying the extractor. The soluble, active components of the plant remain in the flask, while the solvent volatilizes repeatedly [65, 66, 67]. The process continues till the plant material is completely extracted, which can be analyzed by a change in the color of the solvent in the chamber, as shown in Figure 16.3. This extraction method is useful in obtaining large quantities of extract with a smaller amount of solvent, and no filtration is required. It is suitable for plant materials that are partially soluble or contain insoluble contaminants [61].



**FIGURE 16.3** Soxhlet extraction process exhibiting a change in color of the solvent in the chamber during the extraction process (a). Light color shows the maximum extraction of extract from plant material (b).

#### 16.6.5 MICROWAVE-ASSISTED EXTRACTION

Apart from other conventional methods of extraction, microwave-assisted extraction is an advanced process that is used solely or in combination with other methods to prepare extracts from plant material. The technique uses the mechanism of dipole rotation and ionic transfer by displacement of charged ions present in the solvent and drug material. This method involves electromagnetic radiations with frequencies between 300 MHz and 300 GHz, with wavelengths of 1 cm to 1 m. Microwave radiations are applied on an object which absorbs electromagnetic energy and converts it into heat. Due to this heat, the solvent moves into the plant material used for extraction. It is important to note that this method is suitable for polar solvents; also, the dipole rotation favors the migration of ions and solvent penetration and assists the extraction process. However, for nonpolar solvents, a small amount of heat is produced by radiation. Apart from many advantages, this method is suitable only for compounds falling under phenolic and flavonoid categories [59].

#### 16.6.6 Ultrasound-Assisted Extraction

Studies reveal certain limitations of conventional extraction methods, such as time, energy, and solvent requirements. Therefore, advanced methods are employed to overcome these limitations. One such method is ultrasound-assisted extraction (UAE), which is efficient in extracting bioactive plant-based compounds in less time, energy, and solvent. It also requires less temperature due to its non-thermal characteristics. The other parameters, such as frequency, power, duty cycle, temperature, time, and solvent type, need to be considered to develop the required end product.

The basic principle of extraction through UAE lies in the utilization of sound energy at an ultrasonic frequency exceeding 20 KHz, which ruptures the cell wall of the plant material and provides more surface area for the solvent to penetrate, thus allowing the extraction of secondary metabolites (polyphenols, carotenoids, polysaccharides) in the extract [59, 68].

## 16.7 PILOT STUDY – ANALYSIS OF UV PROTECTIVE POTENTIAL OF SELECTED PLANT EXTRACTS

To understand the role of solvents and techniques involved in plant extraction and to evaluate the potential of UV protective properties of plants, a pilot study was conducted by comparing the sun-protective properties of plant extracts. While several natural substances have UV-protecting properties, there is still a scope to identify the UV protective potential of many new plants and their parts, as well as analyze their effectiveness in comparison to each other. Hence, it is imperative to do further research on the advancement of environmentally friendly UV protective compounds derived from plants that can be used on textiles to enhance their functionality. This study aims to identify the potential of UV-protective natural extract by analyzing it through different scientific methods. This study aims to identify the therapeutic



• UV absorption and sun protection factor

FIGURE 16.4 Research design.

(UV protection) potential of the natural extract by employing scientific methods. The methodological procedure adopted to fulfill the objectives of the present study, "Analysis of UV Protective Potential of Plant Extract," was divided into three phases, which are discussed under the following heads:

Phase I: It dealt with the selection and procurement of raw materials.

Phase II: It dealt with the preparation of plant extracts

**Phase III**: It dealt with the analysis of plant extracts for UV protective properties.

# 16.7.1 PHASE I: SELECTION AND PROCUREMENT OF RAW MATERIAL (SOLVENT AND PLANTS)

On a review basis, an exhaustive list of plants with UV properties was prepared. Three plants (*Portulaca oleracea, Syzygium cumini* (L.), and *Psidium guajava* (L.) listed in Table 16.1, were selected as per their availability in and around Hisar City, Haryana, India, and only the regenerative parts of the plants were collected and used for the present study. Methanol was selected as the solvent for extraction, based on a literature review and sourced from the local market of Hisar, Haryana, India.

# 16.7.2 Phase II: PREPARATION AND EXTRACTION OF PLANT EXTRACTS

The process of extraction of plants was carried out in the steps which include drying of plants (required parts collected, washed, and dried in the shade); grinding of dried plant material (ground to powder form, sieved and weighed); extraction through the Soxhlet process (hexane was used to remove chlorophyll, and then the plant materials were subjected to methanol extraction). The output of each stage is presented in Table 16.2.

# TABLE 16.1Plants Selected for the Study

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S. No.	Plants	Scientific Name	Local Name	English Name	Family	Parts Used
1.		Portulaca oleracea	Bichu-butti	Purslane	Portulacaceae	Stem and Leaves
2.		Syzygium cumini (L.)	Jamun	Black plum	Myrtaceae	Leaves
3.	PCK	Psidium guajava (L.)	Amrood	Guava	Myrtaceae	Leaves



#### 16.7.3 PHASE III: ANALYSIS OF PLANT EXTRACTS

Plant extracts were analyzed on the parameters mentioned below:

a. The yield percentage of extracts was calculated by using the following formulae [69], and the results obtained are given below:

Yield percentage = 
$$\frac{\text{Yield obtained (g)}}{\text{Weight of dry plant material (g)}} \times 100$$

The results revealed the maximum amount of yield of methanolic extracts was obtained (19.5%) in the case of *S. cumini* (L.), whereas *P. oleracea* and *P. guajava* (L.) exhibited 18% & 17.6%, respectively (Figure 16.5).



FIGURE 16.5 Yield percentage of plants under study.



FIGURE 16.6 Qualitative phytochemical analysis of plant extracts.

b. Qualitative phytochemical screening:

Fresh methanolic extract of plants was screened for secondary metabolites by following the process described by Vermani et al. [50] and Saidulu et al. [51]. The qualitative phytochemical analysis of extracts indicated the presence of alkaloids in *P. oleracea*; tannin, saponin, and phenols in *S. cumini* (L.) and *P. guajan* (L.); flavonoids were present in all three extracts, as shown in Figure 16.6. The results also suggested that the maximum number of photochemicals were present in *S. cumini* (L.) and *P. guajava* (L.), and these phytochemicals, as per literature, also exhibit UV protective properties. Therefore, to understand the UV protective potential of these extracts in comparison to each other, further analysis was performed.

c. Analysis of UV absorption and sun protection factor of plant extracts: UV-Vis spectrophotometer was used to analyze the UV absorption property of extracts (10 mg/ml solution). Control (solvent) and extract in separate cuvettes were added, and reading was noted at the wavelength range (290–320 nm). 

IABLE 16.3   Values of EE × I Used in the Calculation of SPF			
Wavelength ( $\lambda$ nm)	EE × I (Normalized)		
290	0.0150		
295	0.817		
300	0.2874		
305	0.3278		
310	0.1864		
315	0.0839		
320	0.0180		
Total	1		

Notes: EE-erythemal effect spectrum and I-Solar intensity.

Sun protection factor (SPF) value was calculated by using formulae given by Mansur et al. [70] and by utilizing values given by Sayre et al. [71].

$$SPF = CF \times \sum_{290}^{320} EE \times I \times Abs$$

Where EE ( $\lambda$ ) – Erythemal effect spectrum, I ( $\lambda$ )–Solar intensity spectrum, Abs–Absorbance of the sunscreen product, and CF–Correction factor (=10). The values of EE × I were constant and predetermined, as shown in Table 16.3.



FIGURE 16.7 UV absorption and SPF value of plant extracts.

TABLE 16.4 Comparative Analysis of Plant Species					
Parameters	P. oleracea	S. cumini (L.)	P. guajava (L.)		
Yield (%)	18.0	19.5	17.6		
Phytochemicals (numbers)	2.0	4.0	4.0		
Sun Protection Factor	22.92	25.01	22.4		

Results revealed that *S. cumini* (L.) showed the highest absorbency and SPF values (25.1) compared to the other two plant extracts.

Thus, two inferences were revealed through this case study: plant extracts can be used as UV protective material, and different plant extracts exhibit different levels of protection. This study concluded that all three extracts have UV/sun protective properties, but *S. cumini* (L.) exhibited the highest yield and UV protection compared to other plant extracts.

#### 16.8 CONCLUSION

Nature is filled with all kinds of materials which can sustain and protect life. Keeping the importance of natural resources in mind, industries should ethically exploit the plant's potential for developing consumer-friendly products. The human race is looking forward to a healthy lifestyle, which will be complemented with chemical-free and sustainable products. The potential of natural plant extracts revealed in the pilot study paves the way for large-scale production of durable UV protective finishes on cotton and other textile fibers, yarns, and fabrics.

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# 17 Insect Repellent Role of Natural Dyes Recent Advances and Future Prospects

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# 17.1 INTRODUCTION

Which organism is responsible for the highest number of deaths on Earth? Dispite its small size, the mosquito is regarded as one of the most lethal creatures on Earth, lacking aggression or enormous size. Remarkably, this minuscule mosquito is accountable for almost one million fatalities annually [1]. Mosquitoes are the primary carriers of parasites, diseases, and arboviruses, including malaria, filariasis, dengue, and chikungunya, making them the most important vectors in public health impact [2, 3]. Of these vectors, dengue is regarded as one of the most harmful viral infections efficiently transferred to people by a bite. The majority of cases and fatalities were documented in the sub-Saharan Africa region. Additionally, mosquito-borne diseases impact Asia, Latin America, certain regions of Europe, the Middle East, and two Pacific nations [4]. In 2019, the American region recorded 3.10 million cases of

dengue, whereas Bangladesh reported 1.01 million cases, the Philippines reported 0.42 million cases, Vietnam reported 0.320 million cases, and Malaysia reported 0.131 million cases [5].

Workers working in rural areas, such as the countryside, parks, forests, etc., could prefer to wear insecticide-treated garments as a means of defending themselves from hematophagous arthropods, including mosquitoes [6]. Hence, it is imperative to devise a solution to address this issue, such as employing a chemical that can be applied to the skin, clothing, or other surfaces to deter insects (and arthropods in general) from landing or climbing on those surfaces [7]. A highly efficient method to manage many diseases transmitted by mosquitoes is to disrupt disease transmission by eliminating or preventing mosquitoes from biting humans [8]. Mosquito repellent fabric, an emerging discipline, offers an optimal solution for combating mosquito bites by effectively repelling and incapacitating mosquitoes upon contact [9].

Textiles are fibrous materials, either natural or synthetic, generally utilized for clothing purposes. They play a crucial role in protecting and providing comfort to the human body [10]. Textile natural fibers consist of plant-derived fibers, such as cotton and flax, as well as animal-derived fibers like wool and silk. Chemical finishing processes can enhance the dependability of natural textiles for use in apparel [11]. Synthetic fibers encompass plant cellulose fibers (e.g., rayon) as well as petroleum-derived fibers, including polyesters, polyacrylic acid, and polyolefin. Textile has been systematically considered and effectively utilized as a protective outer layer, often referred to as a human "second skin." The primary function of human skin is to protect against many natural hazards [12]. The resulting product is conceived as a supplementary skin layer by incorporating a unique protective feature onto textile material. Based on the overall outlook, these clothing materials should possess various functional characteristics, including comfort, ease of maintenance, moisture regulation, heat adjustment, resistance to biological and chemical toxins, and self-cleaning properties, such as sunscreen or anti-ultraviolet capabilities [13]. Using bio-based natural insect repellant is a modern environmentally friendly option in the current environment. These repellents have already been used traditionally, are highly effective, and are safer than synthetic ones [9]. Nevertheless, there exist numerous natural repellents that should be employed at concentrations below a crucial limit, above which they may also function as hazardous solutions [14]. There are two methods to learn about natural insect repellents: bioprospecting and ethnobotanical investigations. Bioprospecting is the predominant method for discovering potential natural repellents. In addition to the safety limitations of their use, another issue linked with natural repellents is their durability when applied to textiles. To tackle the problem, multiple methodologies have been showcased [15, 16].

The performance can be enhanced through the application of microencapsulation techniques and the utilization of nanotechnology, which effectively slows the release rate of active chemicals from the extracts, resulting in an extended duration of insect-repellent efficacy [17–19]. This chapter provides a brief description of various types of plant-based repellents, including their mechanisms of action against insects, their application on textiles, their integration into textile substrates, evaluation methodologies, and the application areas in which they are relevant to textiles. The different natural dyes used for achieving these properties are also summarized with examples.

# **17.2 TEXTILE FINISHING**

The process of finishing textile means modifying textile quality after it has been produced. This modification may entail applying a mechanical or chemical procedure to a textile. It is common practice to give the fiber or fabric for necessary functional qualities [20]. Finishing is a term that typically describes a process applied to textile material to improve quality after coloring, however, before cutting and sewing them into clothing, and a variety of products. Finishing, as used in the textile industry, refers to any process carried out after the fibers have been dyed to enhance the appearance and performance of the fabric. This includes both woven and knitted fabrics that have been finished into usable materials [21].

Today, most fabrics are manufactured in factories. After its production, it is very dirty and harsh. No one wants to wear or use it for other things. In this state, this is called greige goods or gray products. These dirty, gray goods must go through finishing processes before they can be used for clothing, bed linens, interior furnishings, and other uses [22]. Detailed research and development (R&D) in fabric finishing has been stimulated by the high demand for high-value-added fabrics that provide consumers with desirable material characteristics and functional properties, such as easy care, antibacterial, and anti-UV performance [23].

# 17.2.1 OBJECTIVES OF TEXTILE FINISHING

- To make the fabric more aesthetically pleasing.
- Finishes are used to extend the fabric's life or durability.
- To complete the task and fulfill the fabric's specific requirements.
- To bring out or suppress certain inherent textile material qualities, such as softening, de-clustering, brightening, etc.
- To modify a textile material's surface property.
- To give textile fabrics new qualities or features, such as flame-retardant, water-repellent, or water-proof finishes [24].

# 17.2.2 CLASSIFICATION OF TEXTILE FINISHING

Textile finishing has been classified in various ways based on quality, durability, and techniques [25]. The different classifications of textile finishing is schematically represented in Figure 17.1.



FIGURE 17.1 A schematic representation of various classifications of textile finishing.

# 17.3 TYPES OF FINISHING

There are two main types of finishing.

- Chemical finishing.
- Bio-finishing [26].

# 17.3.1 CHEMICAL FINISHING

Chemical finishing is using chemicals to give fabric with the desired characteristics. It is commonly known as "wet" finishing and refers to procedures that modify the chemical constituents of the materials to which they are applied. In other words, a cloth treated with a chemical finish will have a different elemental analysis than a fabric that was not finished [27]. Padding is the most common method of applying chemical finishes, followed by curing and drying. Depending on the type of chemical finish, type of cloth, and equipment accessibility, the main methods are padding, exhaustion, coating, spraying, and foam application [28].

# 17.3.2 BIO-FINISHING

Different chemicals are employed in the textile wet processing sector for various finishing factors, negatively impacting the environment. The need of the hour is for a creative, environmentally friendly method that makes it easier to process fabrics



FIGURE 17.2 Chemical and bio-finishing of textile.

without harming the environment and produces outcomes comparable to those of chemically treated fabrics [29]. Therefore, to avoid this negative impact that is harmful to the environment, the researchers are moving toward bio-finishing, which is more eco-friendly. Bio-finishing is a biological method that includes wet treatments of fabrics (Figure 17.2). It involves enzymatic desizing, biological scouring, biological bleaching, biological washing, biological polishing, finishing with biopolymers, aromatherapy, and specialty finishes like wrinkle-free effect, antimicrobial finish, insect repellent, etc. [30].

# 17.4 INSECT-REPELLENT FINISHES OF TEXTILE

According to the World Health Organization (WHO), interior residual pesticide spraying, long-term insect repellents, insecticide-treated clothing or remedies when people are away from homes or if not under nets, and places where malaria vectors prefer to bite are all effective ways to prevent diseases brought on by insect bites. Insect repellents have been incorporated into textile materials as an alternate and creative method to ensure long-lasting safety [31]. Insecticides are a group of personal care chemicals that are used to prevent the reproduction of arthropods and, as a result, to prevent the transfer of disease to the skin, clothing, and other surfaces. These substances can be produced using natural ingredients. As a result, researching insect-repellent materials is highly recommended. The two primary categories of insect-repellent compounds are synthetic insect repellent and bio-based insect repellent [32, 33].

The natural oils, as well as essential oils and their extracts from diverse natural and herbal sources, are used to make bio-insect repellent, which is an alternative to synthetic repellents like diethyl meta-toluamide (DEET), dimethyl phthalate (DMP), alletrin, permethrin, malathion, etc [34]. The chemical structure of different bio-based molecules used in the functional finishing is shown in Figure 17.3. The need for environmentally friendly insect repellents is therefore significant. Natural colors used for functional finishes are currently popular and encourage living sustainably [35].



**FIGURE 17.3** Chemical structure of bio-based molecules used in finishing (a) chitosan, (b) imidazolium, (c) triclosan, (d) N-N-diethyl-m-toluamide (DEET), (e) permethrin, (f) cypermethrin, (g) pyrethrum, and (h) bioallethrin. (Reproduced with permission from ref. [34], Copyright 2019 Elsevier.)

The following qualities should be present in a repellent for it to be an ideal insectrepellent textile:

- It should not be detachable by abrasion
- It should not be lost due to skin absorption
- It should not be removed by sweating
- It should have a slower evaporation rate
- It should have better adhesion to the substrate surface [36].

## 17.5 PLANT-BASED INSECT REPELLENTS

Plant parts like the root, bark, leaf, fruit, wood, seed, and flower are employed with excellent functional properties to extract bio-based dyes from plants. That plays an important role in protecting against insects, mosquitoes, bugs, and flies [37].

This chapter describes botanical plants like neem, lemon eucalyptus, citronella oil, holy basil, and lemongrass with excellent insect-repellent properties (Figure 17.4).

## 17.5.1 NEEM (AZADIRACHTA INDICA)

*Azadirachta indica*, a member of the Meliaceae family, works well to keep insects and mosquitoes away. Neem oil is a well-known natural alternative to N.N-diethyl-meta-toulamide (DEET) as an insect repellant. Among numerous insecticidal products, neem oil is the major active ingredient. Azadirachtin, which is the primary active ingredient in neem, as well as nimbolinin, nimbin, nimbidin, nimbidol, gedunin,



FIGURE 17.4 Insect-repellent plant species.



**FIGURE 17.5** Main chemical components in neem leaves. (Reproduced from ref. [38], Copyright CC-BY terms, 2023.)

salannin, sodium nimbinate, and quercetin are among its constituents [38]. The chemical structures of different constituents in neem leaves is shown in Figure 17.5. The performance of several textile qualities was evaluated to assess the impact of a solution extracted from green and dry neem tree leaves on the performance of textile properties and insect repellency [39].

Azadirachta indica, commonly known as neem is one of the versatile insectrepellent plants. Accordingly, Hooda et al. [40] applied the extract of neem on cotton fabric. The finishing of the fabric was applied using the exhaust process. It concluded that insect repellency value increased when neem extract was applied to cotton fabric that had been enzymatically scoured and was noted at 96.66% and 96.80% with 3 g/L and 5 g/L, respectively. Insect-repellent textile finishing using *Azadirachta indica* was reported by Rastogi *et al.* [41] as an excellent insect, mosquito, bug, and fly repellent. It evaluated how long the formulations would last. The study performed



**FIGURE 17.6** Analysis of cage methods (a) prepared cage, (b) collected mosquitos. (Reproduced from ref. [42], Copyright CC-BY terms, 2022.)

cage tests on the treated fabrics to assess the formulation's effectiveness. It concluded that this potential action is due to the presence of azadiradirachtin in the neem plant, which is powerful against house flies and mosquitoes.

The extract obtained from neem is an excellent, useful insect repellent [42]. reported the evaluation of the insect-repellent qualities of dyed silk fabric by using neem leaves as a natural dyeing source. To investigate mosquito-repellent properties, the study performed the modified cage method (Figure 17.6). It concluded that the FTIR study revealed that samples with bioactive dyes and silk fabric treated with neem exhibit excellent insect repellency properties. The highest levels of insect repellency were achieved by dyeing fabric with neem extract and mango bark mordant solution (75% and 66.67%, respectively), as mango bark is a natural insecticide. Sajib et al. [43] reported that neem (*Azadirachta indica*), a medicinal plant, is utilized to induce natural mosquito repellent action, which is environmentally safe. They applied the neem extract on knit and woven fabric using a pad-dry-cure and exhaust method, and their effectiveness was tested by repelling mosquitoes. It concluded that fabric treated with neem extract showed 90% insect repellency due to bio-functional moieties.

Neem has also been found by Mishra *et al.* [44] as a versatile insect repellant for textiles. It reported that the repellency effectiveness of neem extract on the female anopheles is better than that of *Culex* mosquito species. In their article, they emphasize the bioactive functionality of neem to prevent the action of mosquitoes on textile finishing fabric. The reviewed data and information about the neem tree used for textile finishing as an insect repellent have been evaluated by Mujallid et al. [45]. Neem trees contain many bioactive compounds; the Salannin compound is one of the most important and has a significant effect as an insect-repellent, especially against mosquitoes. They reported the fact that 60% of mosquitoes can be repelled by fabric finished with green neem leaf.

#### **17.5.2** EUCALYPTUS (CORYMBIA CITRIODORA)

The scientific name of eucalyptus is *Corymbia citriodora*, a natural insect repellant made from the leaves of eucalyptus trees. It belongs to the Myrtacace family. Eucalyptus essential oil is mostly used by cosmetic industries because of its refreshing smell. The main active ingredient of eucalyptus is citronellal. Another component identified in eucalyptus oil, para-menthane-3,8-diol (PMD), when isolated, has been demonstrated to be a powerful insect repellant [46]. Only PMD has received approval from the Centers for Disease Control as a plant-based insect repellent. Traditionally, eucalyptus leaves have been used to treat bronchitis and asthma. Recent research has shown that leaf extracts and essential oils contain antibacterial, antifungal, anthelmintic, and anti-diabetic activities [47].

Eucalyptus oil as insect repellent was applied to cotton and trevira knit fabrics using a padding technique with an acrylic-based binder has been reported by Turan et al. [48]. The chemically active component found in eucalyptus may restrict mosquitoes' biological function, which would also lessen the problem and transmission of disease caused by mosquitoes. The researcher has concluded that eucalyptus oil extract with 15% and 30% showed better results in the field test conducted to evaluate the effectiveness of mosquito repellency. The insects are kept away by the eucalyptus oil extract scent. The extract obtained from eucalyptus has been found an excellent insect repellant for textiles. Therefore, Anwar et al. [49] developed a complicated coacervation technique for producing microcapsules from eucalyptus oil. They performed the pad-dry-cure procedure to coat the polyester/cotton (PC) fabric with a military camouflage print with the as-prepared microcapsules of eucalyptus (Figure 17.7). The coated fabric showed a good potential to repel the mosquitoes



**FIGURE 17.7** Mosquito repellent fabric produced from eucalyptus oil. (Reproduced from ref. [49], Copyright CC-BY terms, 2023.)

utilized in the cage test, according to their conclusion. The fabric showed the highest repellency rate, about 97%, using eucalyptus oil and moringa gum.

The importance of essential oil from Eucalyptus globulus leaves has a 90% success rate in keeping insects away, as reported by Endris et al. [50]. The study applied the active essential oil to the fabric using a pre-soaking and padding technique. It evaluated that this study aims to develop a viable method for environmentally friendly eucalyptus leaf extract dyeing and multifunctional finishing on 100% cotton. The bioactive components of citronellal exhibit excellent properties to prevent the attack of insects. Essential oils, particularly eucalyptus, which exhibit excellent insect repellency, provide a safe, non-toxic alternative finish for fabrics to protect people from vector-borne infections. Dhillon et al. [51] applied the insect repellency textile finish on cotton fabric. The effectiveness of the created microencapsulated coatings against the *Aedes, Anopheles*, and *Culex* mosquito species was examined in a cage test. It concludes that even after 15 washings, the eucalyptus oil microencapsulated finish offered 100% protection from *Aedes* mosquitoes.

*Eucalyptus* is a versatile insect-repellent plant. Another study by Kamboj et al. [52] attempted to apply a dyeing finish to cotton cloth using an extract of *Eucalyptus citriodora* leaves. This affordable natural insect-repellent finishing ingredient is highly effective, environmentally friendly, and biodegradable. It reported that this botanical plant source shows good insect repellency on cotton fabric. The following concentrations of eucalyptus leaf extract were used: 35%, 45%, and 55%. According to data, insect repellency increases by 70–80% when concentration increases from 40–55%. Hence, a 55% extract was standardized for use in dying cotton fabric.

## 17.5.3 CITRONELLA (CYMBOPOGON NARDUS)

Citronella belongs to the Cymbopogon family and Poaceae genus. In 1948, the U.S. Environmental Protection Agency first authorized citronella *Cymbopogon nardus* oil for use as an insect repellent. Today, natural insect repellents frequently contain this oil. The main bioactive components of citronella are citronellal, geraniol, and citronellol. Citronella oil has been discovered to have an insecticidal effect because it blocks neurological pathways, which interferes with an insect's metabolism and prevents it from feeding [53]. Citronella essential oil is registered by the US EPA (Environmental Protection Agency) as an insect repellent, due to its high efficacy, low toxicity, and customer satisfaction. Utilizing gelatin-arabic gum microcapsules, microencapsulation extends the effectiveness of natural repellents citronella. On treated fabric stored at room temperature (22°C), citronella could repel for up to 30 days [54].

Preparation of microcapsules from natural citronella oil and then finishing a polyester and cotton fabric with it is studied by Tariq et al. [55]. They performed a sophisticated method to create microcapsules with citronella oil as its core material, which were then applied to fabric using the pad-dry-cure technique. It evaluated that fabric dyed with citronella oil showed great insect repellency and acted as a good insecticide. The optical microscopy images of the microcapsules with citronella oil are shown in Figure 17.8. It concluded that due to active components of citronella oil, the finished fabric showed 90% mosquito repellent action. After 30 washings, the fabric still had 80% of its ability to keep mosquitoes away, proving that it endures as freshly



**FIGURE 17.8** Optical microscopy images of the microcapsules with citronella oil in different magnifications (a) = $\times$ 40, (b) =  $\times$ 100, (c) SEM images. (Reproduced with permission from ref. [55], Copyright 2022 Elsevier.)

finished fabric. Citronella oil is used as a versatile insect repellent textile finish and bio-based source due to its environment-friendly nature in contrast to synthetic finishes, which has been found by Sariisik et al. [56]. The study evaluated that Citronella oil, a sustainable insect repellant, was coacervated with an ethyl cellulose shell and then applied on fabric. Mosquito mortality rates were reported to be 72%, 65%, and 55% for printing, coating, and impregnation, respectively, and the treated materials continued to exhibit repellency after five washing cycles.

The extract obtained from citronella has excellent insect-repellent properties, as reported by Elsayed et al. [57]. The study applied the extract to cotton fabric with a larger and more lasting insect shield. It reported that the action of this bio-based insect repellent performance was over 90% over 3 weeks. They performed standard techniques to measure and test an insect-repellent treated fabric's ability to offer protection. It concluded that this bio-based natural source has excellent insect-repellent action. Evaluation of the working of microcapsules using citronella oil and then applied on cotton fabric is made by Wijayapala et al. [58]. The research reported the ability of the fabric to repel mosquitoes was compared to finished cotton fabric with microencapsulated extract. It performed the traditional pad-dry process to pad cotton fabrics with microencapsulated citronella oil to fabrics sprayed with an ethanol solution of the essential oil, the microencapsulated oil showed stronger and longer-lasting protection from mosquitoes, ensuring a repellent effect of greater than 90% for three weeks.

#### 17.5.4 ROSEMARY OIL (ROSMARINUS OFFICINALIS)

The fresh flowering plant tops of the rosemary (*Rosmarinus officinalis*) are primarily steam-distilled to produce the essential oil. Rosemary belongs to the Lamiaceae (Labiatae) family. 1,8-cineole (eucalyptol, 24.6%),  $\alpha$ -*Pinene* (17.7%), camphor (12.4%), and camphene (11.3%) are the main active components of this essential oil (Figure 17.9). This essential oil offers a wide range of biological properties (antifungal, antibacterial, antiviral, anti-moulds, antioxidant, insect repellant, and pest management), which are fascinating physicochemical characteristics, due to their bioactive composition. It has been demonstrated that rosemary oil completely repels adult *Culex quinquefasciatus* and *Anopheles stephensi* mosquitoes 100% for up to 8 h [59].



**FIGURE 17.9** Major chemical components of rosemary oil. (Reproduced from ref. [59], Copyright CC-BY terms, 2022.)

Among essential oils, rosemary oil is a versatile insect repellent in textile finishing. Singh et al. [60] made the chitosan-gelatin combination with rosemary essential oil to prepare microcapsules, which were used to create a multipurpose linen fabric. That linen fabric showed insect-repellent function due to active moieties such as camphor and camphene. It evaluated that microcapsules were more effective in repelling Anopheles mosquitoes for an extended period by more than 90%. The extract obtained from rosemary oil has excellent insect-repellent potential, as found by Soroh et al. [61]. They applied the natural dye extract of rosemary oil microemulsions on cotton polyester fabrics. They exhibit excellent insect-repellent action against *Aedes aegypti* due to the presence of 1,8-cineole,  $\alpha$ -*Pinene*, camphor, and camphene. After 15 days in storage, a 46.6% mortality rate was noted. Evaluation of rosemary (Rosmarinus officinalis) essential oil to create microencapsulated textile finishes has been served by Dhillon et al. [51]. They reported that the extract has a specific bioactive composition to repel mosquitoes on cotton fabric. The effectiveness of the created microencapsulated finishes against the Aedes, Anopheles, and *Culex mosquito* species was evaluated in a cage test. It reported that rosemary showed a 40–49% insect-repellent rate due to its bioactive chemical constituents. However, fabric treated with rosemary exhibited 100% protection for up to five washings.

#### 17.5.5 CINNAMON OIL (CINNAMOMUM VERUM)

Cinnamon belongs to the Lauraceae family of plants, which can have insect-repellent action using essential oils from their bark and leaves. The excellent insect repellency rate is due to the presence of cinnamaldehyde, cinnamyl acetate, caryophyllene oxide, and eugenol bioactive constituents (Figure 17.10). Different studies showed that the best method for getting rid of *Aedes aegypti* larvae was the use of cinnamon essential oil [62].

Cinnamon bark oil can be utilized to finish linen fabrics with repellent qualities against mosquitoes as reported by Mohamed et al. [63]. The mesoporous silica-based microcapsules containing the cinnamon bark oil infused into linen fabric. Due to its composition, cinnamon bark showed an excellent rate of insect and mosquito-repellent action. The study concluded that the bark extract showed a maximum repellency of



**FIGURE 17.10** Major chemical components of cinnamon essential oil. (Reproduced from ref. [63], Copyright CC-BY terms, 2023.)

85% against the *Aedes aegypti* mosquito. Cinnamon bark is used as a versatile insect repellent in textiles is served by Singh et al. [64]. The study reported the application of chitosan-gelatin microcapsules coated with cinnamon bark oil on linen fabric. The bioactive composition of cinnamon extract makes it insect-repellent and controls the action of insects. Eugenol, cinnamyl acetate, anethole, and cinnamaldehyde are all constituents of cinnamon bark oil known to have mosquito larvacidal action. It concluded that cinnamon bark finish linen exhibits excellent 100% mosquito repellent action against the *Anopheles* mosquito. The extract obtained from cinnamon oil is applied on cotton fabric due to its remarkable insect-repellent properties, which has been reported by Affincy et al. [65]. The pad dry cure method is used to apply an extracted chemical to fabric to create repellent action. The components of cinnamon are poisonous to insects and have an overpowering aroma. The pad dry-cure process increases the fly repellent's effectiveness to 100%. It concluded that finishing with this natural source was harmless, friendly to the environment, affordable, simple to use, and most effective for repelling flies.

## 17.5.6 MARIGOLD (TAGETES ERECTA)

Marigold is also known by its scientific name, Tagetes erecta, which is used as a versatile insect repellent in textiles. It belongs to the Asteraceae family. The main active constituents, such as pyrethrin, flavonoids, and carotenoids, are responsible for insect repellency behavior [66]. Marigolds repel Mexican bean beetles, squash bugs, and other insects because of their natural anti-inflammatory, antispasmodic, and antifungal characteristics. It has been demonstrated that the marigold flower has a strong odor that repels mosquitoes, such as adult *Anopheles stephensi* [67].

Marigold petals were used as a versatile insect repellent in textiles, especially to repel mosquitoes and flies. They applied bio-based and non-hazardous extract to cotton fabric. The study was conducted by Kantheti et al. [67]. They performed a cage test method to check the efficiency of insect repellent action. They concluded that the test findings exhibit *Tagetes erecta* has excellent above 90% repellent action. Another study by Punia et al. [68] evaluated that 60% of mosquitoes can be repelled by fabric

treated with this extract. The study concluded that this affordable natural insectrepellent finishing ingredient is highly effective, environmentally friendly, and biodegradable. The extract obtained from marigold flowers is applied on textiles as insect-repellent finishes. Gupta et al. [69] observed that this natural plant extract was applied to cotton fabric. In a mosquito cage box, prepared pad-dry-cure samples were tested for mosquito repulsion. The study reported that marigold flower extract has promising insect-repellency properties that showed 95% insect-repellent action.

## 17.5.7 PEPPERMINT (MENTHA PIPERITA)

Peppermint is a hybrid species of mint that belongs to the taxonomic family Lamiaceae of plants and is widely distributed throughout temperate regions of the world. Peppermint contains non-essential components including phenolic acids, triterpenoids, flavonoids, steroids, and Peppermint essential oils (PEO) like menthone, menthol, neomenthol, and isomenthone, which are biologically active metabolites [70]. The most active component of peppermint is menthol, along with menthone and pulegone, and the insect-repellent property of peppermint is due to these moieties [71].

Parvaz *et al.* [4] conducted research in which they explored the mosquito repellence activity of peppermint essential oil and garlic cloves over the knitted fabric. The study used different concentrations of peppermint garlic extract solution (alcoholic) and applied over the knitted fabric by using the exhaust dyeing method to check mosquito repellence. They mixed powdered peppermint leaves with garlic at a weight ratio of 3:1 to check whether a small amount of garlic affects the repellence activity of peppermint. By adding 5 g of powdered peppermint and garlic with 80 ml methanol and the solution left for 24 h, the peppermint garlic solution was filtered and increased concentration by evaporating methanol up to 30 mL. The overview of the fabric preparation is shown in Figure 17.11. Knitted fabric dyed with peppermint



**FIGURE 17.11** Overview of mosquito repellent fabric finishing using Peppermint. (Reproduced from ref. [4], Copyright CC-BY terms, 2023.)

garlic extract shows dry rubbing fastness (4/5–4), wash fastness (4/5–3/4), light fastness (8–6/7), and exhibit mosquito repellence ranging from (50–85.6%) with excellent antifungal property. It is reported that three memories, menthol, menthone, and neo menthone, are present in peppermint oil, responsible for controlling mosquito larvae and mites and exhibiting biocidal properties [72]. They dyed linen fabric with essential oils of different plants (peppermint, cinnamon, and lavender) via silica encapsulation technique. They use solvent evaporation methods for the microencapsulation of plant oils. Fabric dyed with peppermint oil 15% encapsulated in silica with biopolymer (pectin), and crosslinker gives excellent mosquito repellency compared to other cinnamon and lavender oils.

An experiment conducted by Uckun et al. [73] to show the recent trend to impart insect repellent properties by using various species of the Lamiaceae family, peppermint oil belonging to this family with remarkable repellence against mosquitoes (Aedes aegypti, Culex quinquefasciatus) and used as insect repellent in textile finishing. It reported peppermint mint essential oil has moderate repellent activity against mealybug insects and 100% repellence against common houseflies (Musa domestica). Different plant species reviewed by Coetzee et al. [59] that have insectrepellent activity against mosquitoes, flies, and ticks are used in textiles as insectrepellent finishing. They reported encapsulated chitosan nanoparticles of peppermint essential oil formed by the emulsification of oil in water by ionic gelation, and then solidified it by freezing dry at 35°C for 72 h. Nanoparticles show insect repellent efficiency of 65–70% against red floor beetle (Tribolium Castaneum). Mehta et al. [74] stated the importance of essential oils aromatherapy and their role in textiles in repelling insects. They reported peppermint as a sustainable mosquito repellent and antimicrobial for cellulosic fabric (cotton) and effectual for decreasing Staphylococcus aureus (S. aureus) and Escherichia coli (E. coli). Nanocapsules of peppermint were formed via microemulsion and applied to the cotton fabric to check washing lastingness and different techniques likes FT-IR spectroscopy, scanning electron microscope, and thermogravimetric analysis to check the distribution of nanocapsules on the fabric surface. After 25 washings, 16% of the peppermint oil was left on cotton fabric, which had 100% antimicrobial activity against S. aureus and E. coli.

To avoid mosquito bites, a study was conducted by Elsayed et al. [57] in which they used different insect repellents directly on the skin and the textile material by nanoencapsulation or microencapsulation. They reported a protective barrier against insects offered by insect-repellent textiles to prevent disease transference through a bug bite. Microencapsulated nanoparticles of peppermint essential oil show 100% resistance against common houseflies (*Musca Domestica*). Kumar et al. [75] reported mosquito repellent agents are classified into two types based on their mode of action: one is the olfactory mode, in which humidity-sensin g holes of mosquitoes are blocked, and the other is the tactile mode, in which mosquito repellent agents directly attack the CNS and kill them before their contact with the fabric. They reported a 100% remarkable repellence effect of peppermint extract for 150 min against the larval and adult mosquitoes. They reported two methods of applying plant extract over the fabric surface. One is the direct method, in which plant essential oil extract is directly applied on the fabric by using the pad-dry-cure method. The second one is microencapsulation, in which plant essential oil extract is closed in microcapsule and then fabrics is finished with the exhaustion method.

### 17.5.8 LEMONGRASS (CYMBOPOGON FLEXUOSUS)

Lemongrass is a member of the family Poaceae and genus Cymbopogan. Various species of lemongrass exist in nature. *Cymbopogon flexuosus* is a species of lemongrass that is commercially cultivated, and their essential oil has greater solubility in alcohol [76]. Lemongrass is a perennial grass with stem and leaves that emerge directly from the soil and grow up to 250 cm in height, with green leaves, cultivated mostly in the wasteland and infertile soil. Lemongrass is native to tropical and subtropical regions of Australia, Europe, Africa, and the Indian subcontinent [77]. Leaves of lemongrass are used in food and beverages as flavoring agents but are mainly used to extract essential oil. Citral-generating beta-ionone, formed from the mixture of neral and geranial isomers, is the most important component of lemongrass, and it is 75–85% present in lemongrass essential oil. Citral has insecticidal and antimicrobial properties and is used as a natural insect repellent; due to citral, lemongrass established itself as an insect repellent [78].

Parul et al. [79] reviewed essential oils of different plant species for their mosquito repellence activity on fabric. Lemongrass essential oil is one of them, and it has mosquito repellence activity on cotton and single jersey fabric using the pad and dry cure imparting technique. The repellence activity was evaluated by an excito chamber test. They also reported different methods to impart essential oil on fabric surfaces: dipping, spraying, and pad-dry cure. Different binders are used to increase wash durability during the finishing of textile fabric with essential oils. Gogoi et al. [78] reported a work in which lemongrass oil was coated to wool fabric with and without chitosan. Chitosan is used to bind lemongrass oil to wool fabric for a longer duration. FTIR and SEM is used to check lemongrass oil and chitosan on the surface of wool fabric. Lemongrass oil and chitosan both performed well with anti-moth activity, UV protection, and long-lasting aroma on woolen fabric. The coated fabric shows damage due to moth attack less than 0.32% while the prescribed limit is 2%. Natarajan et al. [80] reported microencapsulation of lemongrass oil to cotton fabric using padding and exhaust method to check wash durability. In the exhaust method, microcapsulation of lemongrass oil to cotton retained aroma only for 10 washes, while microcapsulation by padding method retained aroma for 30 washes. They also reported microencapsulation of aqueous lemongrass extract to polyester fabric showed 92% mosquito-repellent activity, while encapsulation of lemon grass menthol showed only 80% mosquito-repellent activity.

Bhatt et al. [81] conducted research in which nylon net fabric was treated with lemongrass essential oils nanoemulsion, depositing polyelectrolyte multilayer on the textile substrate by using layer by layer (LBL) technique. SEM and GCMS are used to check active ingredients on the fabric surface. The mosquito repellence activity increased by increasing the number of layers or by increasing the concentration from 50 g/L to 100 g/L, leading to increased repellence levels of 40–70%. Achieving mosquito mortality over repellence is very tough, even at 50 g/L and 75 g/L. Treated fabric shows effective mosquito repellence and antimicrobial activity even after



**FIGURE 17.12** Washing effect of mosquito repellent fabric treated by (a) 100 g/L and 10 layer, (b) 100 g/L and 20 layer. (Reproduced from ref. [81], Copyright CC-BY terms, 2019.)

25 washes, which is shown in Figure 17.12. Halbkat et al. [82] reported lemongrass, a botanical repellent against mosquitoes, shows 92% repellence when infused with polyester fabric. Lemongrass oil is the main ingredient in many insect-repellent brands such as Badger Anti Bug Spray, Alba Botanica Anti-Bug Spray, Just Neem Adios Herbal Spray, and many more. They use this commercially available insect repellent to check their insect repellence over jersey fabric made of 80% nylon and 20% elastane. Then, they apply standard four pump spray over the fabric surface and place it in an arm-in-cage assay. The fabric showed excellent mosquito bite repellence against *Aedes aegypti*.

#### 17.5.9 HOLY BASIL (OCIMUM SANCTUM L.)

Holy basil (*Ocimum Sanctum L.*), locally known as tulsi in the Indian sub-continent, belongs to the Lamiaceae family. *Ocimum sanctum* is a synonym for *Ocimum tenui-florum* cultivated in different regions of the world due to their medicinal, food, insect repellent, and perfumery importance [83]. Holy basil is an aromatic, sweet-scented perennial shrub with hairy stems and leaves up to a height of 30–60 cm [84]. Hydrodistillation or steam distillation is used to extract the essential oil of holy basil to get its valuable metabolites. Essential oils of holy basil are mainly methyl eugenol, eugenol, 1,8-cineole, b-caryophyllene, germacrene D, b-elemene, a-cubebene, Carvacrol, b-pinene, b-Selinene, a-Humulene, a-Pinene, p-Cymene, Sabinene, c-Terpinene, Limonene, Thymol, a-Terpinolene (Figure 17.13). In addition to essential oil, holy basil is rich in phenolic compounds, terpenoids, alkaloids, flavonoids, sterols, glycosides, and polysaccharides [85].

Chukwuma et al. [85] reported holy basil is used locally to mitigate or treat stress, colds, coughs, insect bites, and stings. Essential oils possess ovicidal activity against *Aedes aegypti* eggs for 24–72 hours when exposed to the oil within a concentration range (100–1000 ppm). They also reported 100% repellence of the adult *Aedes. aegypti* mosquito when exposed to a solution having 20% essential oil of holy basil in ethanol. Chandrasekaran et al. [86] conducted a study to evaluate the combined effect of holy basil and wild turmeric extract on single jersey knitted cotton, microdenier polyester fabric for antimicrobial properties. Micro-denier polyester fabric



**FIGURE 17.13** Major chemical components of holy basil. (Reproduced from ref. [83], Copyright CC-BY terms, 2018.)

and lyocell show good antimicrobial activity against *Staphylococcus aureus* in 12 hours and 24 hours, respectively. Cotton fabric also shows good antimicrobial activity against *Staphylococcus aureus* and *Escherichia coli*. They also reported that the insecticidal properties of holy basil are due to eugenol. Chaiphongpachara et al. [87] evaluated the larvicidal activity against larvae of *Aedes aegypti* of seven herbal oils; holy basil is one of them. They extract the essential oil of holy basil using steam distillation and store it in amber glass bottles. To check larvicidal activity, 1 ml of ethanol was mixed with deionized water to form a diluted solution of essential oils. Deionized water is used to form a diluted solution of essential oils in a 250 ml beaker of varying concentrations of 0.025, 0.050, 0.075, 0.100, 0.125, 0.150, and 0.175 ppm. *Aedes aegypti* mortality increased with an increase in the concentration of essential oils in solution. They reported larvicidal activity against *Aedes aegypti* at the concentration range of 0.025 to 1.75 ppm. Holy basil has the 2<sup>nd</sup> highest larvicidal activity among seven herbal extracts (LC50 = 0.07 ppm and LC90 = 0.12 ppm), with (81–97%) mortality of *Aedes aegypti* larvae.

Natarajan et al. [80] discuss eco-friendly, UV protective, insect repellent, and fragrance finishing of textiles using various methods such as pad-dry-cure, exhaust method, and microencapsulation of natural products. They reported essential oil of holy basil is used in refreshing, stress relieving, and mosquito repellent finishing of textile fabric up to a maximum of 15–30 washes. Choudhary et al. [88] reported that the Ocimum sanctum is very useful for healing stings and bites of insects; for this purpose, the infected area is covered with a paste made from fresh holy basil leaves. Holy basil leaves also possess antimicrobial, antifungal, and highly antibacterial properties, so any product made from holy basil exhibits all of these properties.

## 17.5.10 LAVENDER (LAVENDER ANGUSTIFOLIA)

Lavender (Lavender angustifolia) is a member of the Lamiaceae family and genus Lavandula cultivated worldwide due to its prime importance in perfumery, medicine, food processing, and cosmetic and insect repellence [89]. Lavender is an herbaceous biennial aromatic plant having an unbranched, single-spike stem, 30–50 cm in height with a small, narrow, fragrant purple-blue flower at the end of the stem. It naturally originated from the North Atlantic to the Middle East and is grown on dry, sunny, and calcareous soil. Lavender has been commercially grown since the beginning of the twentieth to extract essential oils in different regions of the world, mainly in France, Bulgaria, China, Spain, Poland, Morocco, and Ukraine. Essential oils in plants are mainly classified into two categories: hydrocarbons and oxygenated compounds derived from hydrocarbons, like esters, phenols, ketones, and esters. Recent studies reveal that lavender essential oils are mainly composed of Linalool, Linalyl acetate, lavandulyl acetate, myrcene, camphor, limonene, 1,8-cineole, terpinene-4-ol, and 3-octanone (Figure 17.14) [90]. The insect repellence of Lavender (Lavender angustifolia) is due to mainly linalool and a little bit of limonene compound [91–93].



**FIGURE 17.14** Major chemical components of lavender. (Reproduced from ref. [90], Copyright CC-BY terms, 2021.)

Mohamed et al. [72] reported the formation of cellulosic fiber with mosquito repellence and fragrance finish by microencapsulation of essential oil (cinnamon, peppermint, and lavender) into silica mesoporous microcapsules via the solvent evaporation method. 3 grams of Hexadecyl-trimethylammonium bromide was mixed in a 100 ml mixture of ethanol and water (80:20 v/v) for 30 min at 65°C. Tetraethyl orthosilicate is added to the above-prepared solution drop by drop with stirring for 30 min. The solution was kept stirring at 80°C for 2 hours until cloudiness appeared. The prepared silica was washed with distilled water, followed by drying at 60°C in an air-drying oven, and then calcinated prepared silica at 800°C for 6 hours. 50 ml ethanol was used to prepare mesoporous silica of each essential oil and the mixture was stirred for 3-4 hours at room temperature. The solvent was evaporated by using rotary evaporation, then microencapsulated oil in mesoporous silica was collected and applied on linen fabric. Peppermint oil has the highest mosquito repellence, followed by lavender oil. They also reported that the use of pectin as a binder increases mosquito repellence and antimicrobial activity. The purpose of the Mishra et al. [94] studies was to examine mosquito repellence of linalool and limonene. They reported linalool is extracted from lavender essential oils, due to the higher content of linalool in it, by using column chromatography and column distillation. They concluded that linalool extracted from the lavender plants shows mosquito repellence up to 80.90% and is also effective against Staphylococcus epidermides, Rhodococcus equi (0.60 mg/mL) and exhibited 85% mortality against the rice weevil after 24 hours at the concentration of (0.1 µl/720 mL).

Šimůnková et al. [95] conducted a study in which they used lavender oil to protect Norway spruce wood (Picea abies) against attacks by termites and brown-rot fungus (*Rhodonia placenta*). An average density of 412 kg m<sup>-3</sup> and dimensions of  $50 \times 25 \times$ 15 mm mature Norway spruce wood were used for testing. Ethanol 70%, water 25%, and lavender oil 5% are used to form the solution. Hydrophobic substances consist of 6% aqueous dispersion containing 2% pure wax emulsion and 2% silane-siloxane emulsion. Both solutions were applied on wood samples by 8-hour dipping and then examined for insect repellence. Results show that lavender oil protects wood from insects and this insecticidal activity is due to the presence of linalool, linalyl acetate, and lavandulyl acetate. Yeguerman et al. [96] reported the formation of essential oil polymeric nanoparticles extracted from lavender, peppermint, rosemary, germanium, and palmarosa against German cockroaches (Blattella germanica) to examine insect repellence. The melt dispersion method was used to prepare essential oil polymeric nanoparticles (EOPN). Continuous stirring on a hotplate was used to melt 20 g of polyethylene glycol 6000 Mw. at 65°C, and then 2 g of each essential oil was added. The essential oil mixture was stirred at 15,000 rpm for 10 min, then cooled at -4°C for 45 min, and the product was sieved using 230 mesh stainless steel. To check insect repellence, the product was applied to the filter paper, and then the filter was introduced into glass vials where insects were added. EOPN of lavender shows less insecticidal repellence than peppermint and palmarosa (P < 0.05).

## 17.5.11 CLOVE OIL (SYZYGIUM AROMATICUM L.)

Clove (Syzygium aromaticum L.) belongs to the Myrtaceae family and is an aromatic evergreen plant native to the island of Maluka and Indonesia. The clove is dark brown

with an aromatic smell and pungent taste. The Zanzibar clove variety is widely cultivated due to more flowering bud production among three other varieties of clove: Ambo, Siputih, and Sikotok [97]. Due to the prime importance of their essential oils in the medicinal, food processing, nutritional, cosmetic, and insect repellent industries, cloves are cultivated worldwide, including Indonesia, China, India, Pakistan, Madagascar, and Sri Lanka. Recent studies reveal that cloves contain approximately 15–20% weight of essential oils [98]. Dried floral buds of clove are used to extract essential, eugenol (80%–90%) is the main active component of clove essential oil along, alpha-humulene (0.55%), eugenyl acetate (15%–17%), alpha-terpenyl acetate (0.1%), beta-caryophyllene, (5%–12%), and methyl eugenol (0.2%) (Figure 17.15) [99–101].

Deng et al. [102] evaluated complete protection time against the Aedes species by using seven commercially available plant essential oils *in vivo* human-hand in cage test. They used Fennel oil, Cinnamon, Peppermint, Ginger oil, Black pepper, Patchouli oil, and Clove bud oil extraction for this experiment. They reported that when the concentration of essential oils in solution is 0.20%, only three extracts of cinnamon, clove, and DEET got 45 min complete protection time, while when a high dose of EOs was used, clove got second highest CPT 120 min among six other essential oils. They evaluated clove essential oil as the most effective in protection against mosquitos. Atiya et al. [103] conducted research by introducing the use of clove and eucalyptus oil on historical textiles to protect against insects. They used plan silk fabric dyed with natural dyes extracted from turmeric and madder in the presence of Alum, Copper sulfate, Iron III chloride FeCl<sub>3</sub> mordents. Clove and eucalyptus oil



**FIGURE 17.15** Major chemical components of clove oil. (Reproduced from ref. [101], Copyright CC-BY terms, 2021.)

were applied over the fabric surface, and their impact was studied by using FTIR, UV imaging, SEM, and CIE lab. They concluded that the morphology of silk fibers was not affected by clove and eucalyptus oil, and the aroma of these oils was retained for up to 3 months; in fact, these oils also impart excellent insect repellence against black carpet beetle and *Aedes fasciatus* larvae. Obtained results showed that a 3% solution of clove oil induces 92% mortality of insects, while eucalyptus oil exerts low toxicity.

Atkovska et al. [104] reviewed different essential oils used as eco-friendly insect repellent, their insect repellence efficiency, and recent developments. They reported that clove essential oil insect repellence is due to eugenol, eugenol acetate,  $\beta$ -caryophyllene, cinnamaldehyde, thymol, and carvacrol. They concluded that 100% clove oil provides excellent mosquito repellence for up to 120-240 min against the mosquito species (Aedes. aegypti, Culex. quinquefasciatus, and Anopheles Dirus) without causing any harm to the human skin. El Gohary et al. [105] examined clove essential oil's insecticidal and repellent activity on third-instar larvae and female *Culex pipiens.* They use ethanol, water, petroleum ether, and ethyl acetate to extract the essential oil of 30 g clove (for each solvent) by soaking in these solvents. They mixed 0.5 ml of each extract except water extract with 0.5 ml of tween 80, and then added 10 ml distilled water. Results revealed that third-instar larvae were much more susceptible to water extract than three other solutions, which are  $LC_{50} = 39$  ppm, while LC<sub>25</sub> and LC<sub>95</sub> are (17 and 303.8 ppm). Among these four extracts, water extract was the most active, followed by ether, petroleum, and ethyl acetate extract. Salazar-Bryam et al. [106] reported using clove oil with rhamnolipids in an emulsion with excellent protection against Aedes aegypti and Culex quinquefasciatus. The study used purified glycerol as a source for the production of rhamnolipid, using K<sub>2</sub>HPO<sub>4</sub>, MgSO<sub>4</sub>, H<sub>2</sub>O, KCl, NaNO<sub>3</sub> and the trace element solution contains sodium citrate dehydrate, FeCl<sub>3</sub> 6H<sub>2</sub>O, ZnSO<sub>4</sub> 7H<sub>2</sub>O, CoCl<sub>2</sub> 6H<sub>2</sub>O, CuSO<sub>4</sub> 5H<sub>2</sub>O and MnSO<sub>4</sub> H<sub>2</sub>O, at 37°C for 72 hours. They evaluated the insecticidal activity of rhamnolipid and clove oil solution both separately and together in the form of emulsion. Results revealed that when both were evaluated alone for their repellence, they were not very effective, having  $LC_{50} = 140 \text{ mg/L}$  for rhamnolipid and  $LC_{50} = 154 \text{ mg/L}$  for clove when used against Aedes aegypti larvae, while against Culex quinquefasciatus  $LC_{50}$ was 130 mg/L and for clove is 19 mg/L. when both were mixed, their mortality of mosquito larvae increased from 50-100%.

## 17.6 CONCLUSION AND FUTURE PERSPECTIVE

The usage of synthetic insect-repellent chemicals is currently being challenged by communities worldwide due to their carcinogenic, non-biodegradable, and potentially harmful impacts on the environment and human health. More research should be done on novel technologies that can use naturally bio-based insect repellent sources as a suitable and environmentally safe replacement for synthetic chemicals in textile finishes to create a greener future. This comprehensive study identified highly potent plant extracts and associated chemical markers with potential for use in mosquito control operations. Research on insect repellency has advanced significantly, and their effectiveness has been established. To extend the longevity of treated fabrics' repellent components, more study is necessary. The next major challenge is to close this gap by utilizing an efficient natural finishing agent to increase the durability of the plant-based final products after repeated washing. Future research may be beneficial in examining the insect-repellent action of fabrics with these bioactive natural constituents in textiles. Future research in this area has the potential to lead to more improvements, such as the development of smart textiles that can inhibit the action of insects and protect human beings from contagious diseases.

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