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Cellulose-Reinforced Nanofibre Composites

Production, Properties and Applications

Edited by Mohammad Jawaid, Sami Boufi, and Abdul Khalil H.P.S.

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Bionanomaterial from agricultural waste and its application

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3.1 Introduction

Agricultural biomass refers to all the organic materials that are produced as by-products from agriculture activities, such as leaves, straws, husks, hulls, shells, and animal waste. Huge amounts of biomass are generated as by-products of agricultural (and wood) harvesting and processing activities every year, particularly in developing countries.

Agricultural biomass is the most abundant biomass found in nature. Its content in primary waste is about 30% [1], in forest biomass 40%–50% [2], secondary waste 65%–70% [3], and in industrial crops more than 90% [4]. Globally, biomass waste is produced and unutilized in million metric tonnes annually [5]. Of this enormous production, biomass waste is only a minor fraction of total biomass use for various applications in large-scale industries and community-level enterprises. Approximately 10% of agricultural biomass waste is converted to raw biomaterial to provide future materials to obtain products with higher performance [6]. Sumanthi et al. [7] investigated the sustainability and advancement in utilization of oil palm biomass for value-added products. They showed that only 10% of oil palm biomass is used as an alternative raw material for various applications such as reinforcing agents in composite materials, animal feed, fertilizer, chemical derivatives, and other applications.

In addition to the harvested crop itself, large quantities of wastes are generated in agricultural production systems. Industrial crops of kenaf, jute, abaca, ramie and other crops, and fruits such as coconut and oil palm generate considerable amounts of waste. These wastes constitute a major part of total annual production of biomass agricultural wastes and are an important source of biomaterial for industrial purposes.

Agricultural biomass is the most abundant resource and one that is also renewable. However, utilization of agricultural biomass has been based on the paradigm of a fossil resource-based society, and thus, it is critically important to establish a sustainable production and utilization system for agriculture resources, especially those in tropical regions where resources and biodiversity are plentiful. Therefore, it is becoming more and more important to establish sustainable and recycling-based societies dependent on renewable resources; otherwise, humankind cannot survive.

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In addition, establishment of basic science and technology dealing with lignocellulosic materials, including agricultural waste, is indispensable to promotion of the composite industries of Southeastern Asian countries.

Plenty of waste is produced due to the increased activity in the modern agricultural sector, representing a tremendous threat to the environment. Meanwhile, a declining supply of raw material is cause for concern and in this context the natural fiber can be seen as a good alternative material for local timber industries to produce value-added products, such as biocomposites. Utilization of natural fiber, especially agricultural waste fiber, needs further development as a long-term strategy to develop the tremendous wealth of natural plant fiber that is currently underutilized. Agriculture waste can be obtained from plants such as oil palm, bagasse, corn stalks, coir, bamboo, pineapple, banana, as well as rice husk, and is extracted from different parts of the plant (stem, leaf, seed, fruit, stalk, and grass/reed) [8].

The renewable and biodegradable properties of cellulose found in agricultural waste offer biorefinery functions as cellulose fiber. Cellulose fiber itself is made from biomass-derived fiber that has been defibrated to the size level of several hundredths of a micron and smaller until nanometer-sized. Cellulose fibers exhibit a unique structural hierarchy derived from their biological origin. They are composed of nanofiber assemblies with a diameter that ranges from 2 to 20 nm, and a length of more than a few micrometers. Cellulose nanofibers (CNFs) have been proven to be a promising material for many fields, including high gas barrier packaging material, filter material, electronic devices, food, cosmetics, medicine, biocomposites, and health care, due to their morphology and physical properties.

Recent research on nanocellulose production uses cereal by-products such as wheat straw, soy hulls, soybean straw, sorghum fibers, and rice straw, as well as other crop residues such as cassava bagasse, banana stem, pineapple leaves, sugarcane bagasse, corn stalks, cornhusk, oil palm biomass, grape hulls, and orange bagasse. This biomass, the abundance of which is residue produced by agricultural industry, serves as the best biomaterial to obtain CNF. Study by Changsarn et al. [9] reported that CNF resulting from biomass presents a larger crystalline region and a higher specific surface area. These results suggested that this bionanomaterial is important for the development of nanocomposites for their applications. Furthermore, Lavoine et al. [10] and Durán et al. [11] considered that nanocomposites with CNF as filler enhance the barrier properties used for food packing.

On the other hand, the extremely rapid development of nanomaterials from biomass and the use of nanoparticles have received much attention as a viable alternative for the development of metal nanoparticles. Many attempts have been made to manufacture bionanoparticles, such as Adam et al. [12], Hata et al. [13], Dungani et al. [14], and Rosamah et al. [15]. They suggested that the potential of nanoparticles for filler/reinforcement in polymer composites is seen as highly promising, because bionanoparticles have marvelous and complex structures that are important in understanding their chemical applications.

In this chapter, first we will discuss the fundamental properties of different agriculture wastes as future materials. We will also concentrate our discussion on technologies to produce bionanomaterials, and their use in polymer nanocomposites. After that, types of bionanomaterials such as nanocellulose and nanoparticles will be highlighted. At the end, some points regarding production of nanocomposites and their applications for various purposes will be discussed.

3.2 Overview of waste as green potential from biomass

Biomass is an essential part of the renewable portfolio; unlike other sources of renewables, it can be used as biomaterial for various biocomposite products [16–20]. The biomass itself is derived from three principal sources: agricultural products, forestry waste, and biogenic waste. In general agricultural products consist of oil- and sugar-containing plants; forestry products consist of wood, bark, branches, and stumps; while biogenic waste is derived from the agricultural, commercial, and household sectors. Furthermore, these sources of biomass exist in three forms: gaseous, solid, and liquid.

Recent advances in biomass waste development, conversion process technologies, and their products offer significant opportunities for an exploration and development of improved materials from these renewable resources. The major conversion technologies, such as twining, decortication, and tuxying, convert biomass waste to materials and their products such as biocomposites, pulp and paper, automotive, medical, packaging, construction, aerospace, marine, electronics, pharmaceutical, and biomass energy production [21].

Rapid increase in volume and types of agricultural biomass waste, as a result of intensive agriculture in the wake of population growth and improved living standards, is becoming a burgeoning problem. Furthermore, this waste is of high value with respect to material and energy recovery. Billions of tons of agro products are produced each year, of which the waste has potential for biomaterial resources (e.g., fibers). Assuming that 40% of the production is available as waste and at least 10% of the waste by weight can be obtained as fiber, millions of metric tons of fibers are available every year and the amount will increase annually. Table 3.2 shows the annual biomass-based natural fiber production from various sources.

Since the biocomposites market is growing rapidly, it becomes urgent to design superior strength biocomposites to exploit in particular applications. These wastes could be potential resources for reinforcing materials in biocomposite applications. The utilization of such resources will not only provide sustainable and less expensive material but will also contribute to waste disposal management as well as overcoming environmental problems. However, the agricultural waste fiber is classified as non-wood fiber moderate quality [4]. In addition, its lack of good interfacial adhesion and hydrophilic nature have made its usage difficult [26]. Therefore, good understanding of the fundamental properties of agricultural waste fiber including its modification technologies are indispensable. Several treatment and modification processes can be applied to change its hydrophilic nature to hydrophobic in order to overcome the above-mentioned problems [27].

Table 3.1 **Cellulosic biomass waste conversion to materials and their products**

Biomass waste	Products
Pineapple leaves, sugarcane residues	Animal feed, industrial absorbents, additives for beverages and biocomposites
Wheat straw	Wheat straw PP pelletized feedstock, fertilizer, biocomposites
Rice husk	Silica, metal finishing, water soluble oil and synthetic lubricant
Sugarcane bagasse	Lumber materials, biocomposites, paper and packaging materials, paper wares
Abaca leaves	Fiber craft, cordage, textile and fabrics, pulp, and specialty papers
Coconut husk	Coconut fiber rope and twine, brooms and brushes, doormats, rugs, mattresses and upholstery, often in rubberized coir pads
Sugar mill boiler ash from bagasse	Filtration materials and absorbent products
Oil palm fruit residues	Biodegradable packaging materials, construction, pulp and paper, automotive components
Kenaf fibers, jute fiber	Soundproofing systems, thermal insulators, automotive components, electronics, pharmaceutical
Abaca leaves	Abaca leaf sheath, aerospace, marine, and electronics
Coconut coir	Coconut twines
Banana stem	Banana fiber, biocomposites, pulp and paper
Flax	Biodegradable bags and covers, energy sports equipment

3.3 Fundamental properties of various agriculture waste

Most basic of all in selecting agricultural waste is the characteristics of the fibers, i.e., the various properties by which a fiber may be evaluated. Physical, mechanical, and chemical properties of various agricultural waste-based fiber were examined to assess their suitability for various future applications. These fundamental properties will not only help in opening up a new avenue for these fibers, but also emphasize the importance of natural fibers from agricultural waste as future biomaterial. The following summarizes the properties of agricultural waste-based fiber and gives the specifics of these properties for each fiber source.

3.3.1 Types of agriculture waste

When managed on a sustainable basis, biomaterials available in primary products from industrial crop and agriculture wastes form an abundant, local, and environmentally

Table 3.2 **Annual production of biomass-based natural fibers and sources**

Fiber source	World production (10^3 Tons)	Origin
Bamboo	10,000	Stem
Oil palm fruit	23,500	Fruit
Sugarcane bagasse	75,000	Stem
Banana	200	Fruit
Coir	100	Stem
Wood	1,750,000	Stem
Pineapple	1200	Leaf
Rice straw	28,900	Stem
Rice husk	26,750	Fruit/grain
Jute	2500	Stem
Kenaf	770	Stem
Flax	810	Stem
Sisal	380	Stem
Abaca	70	Stem
Kapok	100	Stem

Sources: From Taj SM, Munawar A, Khan SU. Natural fiber-reinforced polymer composites. Proc Pak Acad Sci 2007;44(2):129–44 [22]; John MJ, Thomas S. Biofibres and biocomposites. Carbohydr Polym 2008;71(3):343–64 [23]; Hambali E, Thahar E, Komarudin A. The potential of oil palm and rice biomass as bioenergy feedstock. In: I7th Biomass Asia Workshop; 2010 [24]; Faruk O, Bledzki AK, Fink HP, Sain M. Biocomposites reinforced with natural fibers: 2000–2010. Prog Polym Sci 2012;37(11):1552–96 [25].

friendly source of raw materials. In addition, the use of agricultural waste biomaterials for component material of composites contributes to income and employment in developing countries [28]. The ambitious policy developments combined with other associated benefits has led to a large increase in the use of agricultural biomaterials for biocomposite products in many countries (European, Australia, and American) [23].

There are many different types of biomaterials based on agricultural biomass. Industrial crops are crops that have the potential to yield a wide range of products. Crops produce filamentous matter from the bast tissue or other parts of plants, and are processed to be used for industrial purposes. There are a number of different industries and products including bioenergy [29], industrial oil and starch [30], fiber [31], and rubber and related compounds [32]. Primary wastes are obtained directly from the agriculture operations, whereas secondary wastes are obtained as by-products of the industrial processes associated with agriculture products. Other sources of agricultural-based biomaterials include kenaf, jute, and tertiary wastes, which consist mainly of the palm oil industry. Forest biomass consists of woody materials generated by industrial processes (timber industries in particular) such as wood chips, bark,

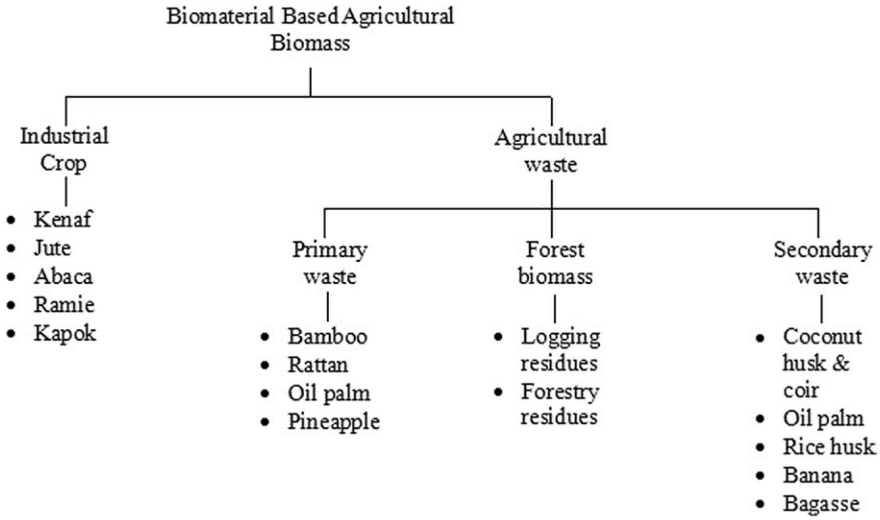

Figure 3.1 Classification of biomaterial-based agricultural biomass.

sawdust, shavings, chips, etc., or directly by logging residue (tree tops, branches, small stems, and deadwood). The use of forest biomass from forestry residues such as wood chips, bark, sawdust, timber slash, and mill scrap for biomaterial contributes to raw material of biocomposites such as particleboard, oriented strand board, and fiberboard [33,34].

At present, the largest share of biomaterial from agriculture comes from secondary wastes from the agricultural industries, namely, the coconut, coir, and oil palm industries. In this context, the use of industrial crop production is not enough for biomaterials; in secondary wastes, it represents a great potential source of raw material to increase the use of agricultural biomass for various applications, independent of the large industrial processes [18,19,35]. Finally, biomass, which includes plant species and agricultural waste, is another source of biomaterial, and its contribution to the sustainability of raw materials is expected to increase in the future [36]. The classification of agricultural biomass as biomaterial for biocomposite components according to their origin is presented in Fig. 3.1.

Type of agriculture waste includes residues from fruit, leaf, seed shells, grass, stalks or trunk, bast and straw, and waste wood. Fig. 3.2 shows the classification of agricultural waste forms based on conversion of cellulosic biomass waste to biomaterials and their products. The figure illustrates the great potential of agricultural waste for conversion to a variety of biomaterials.

3.3.2 Structure and chemical composition

This part deals with the structure and chemical composition of biomass-based natural fibers. Natural fibers' cell structures are basically the same, consisting of a primary

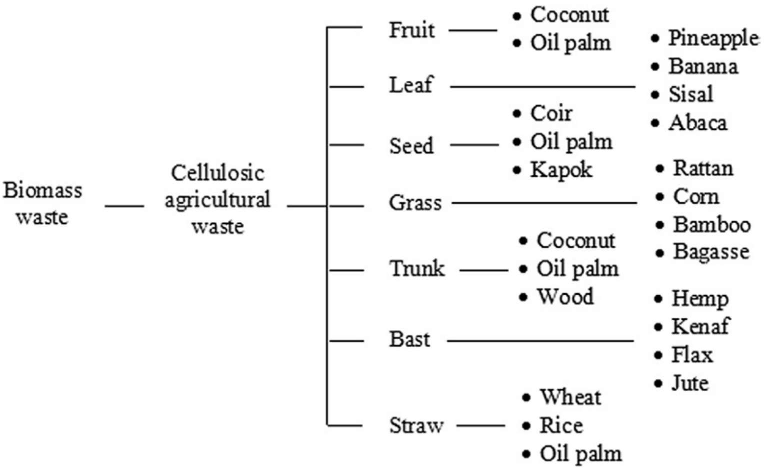

Figure 3.2 Classification of agricultural waste as a biomaterial.

and secondary cell wall, lumen, and middle lamella [37]. The secondary wall consists of three layers (S1, S2, and S3) and determines the mechanical properties of the fibers [38]. Each fiber bundle contains individual fiber cells or filaments, and is made of cellulose and hemicellulose, bonded together by lignin or pectin. Cellulose is a skeletal component in all plants and these polymers are organized in a cellular hierarchical structure. The primary structural component of cellulose cell walls contains many polar hydroxyl groups, which allow them to interact with adjacent molecules to form fibers. These fibers are structurally strong and resistant to chemical attack, so biomass products are widely used in various applications. Hemicellulose is similar in structure to cellulose, and is believed to be a compatibilizer between cellulose and lignin [39]. Lignin polymers are often found in most cell structures in association with cellulose; it is primarily hydrocarbon in nature and makes up a major portion of insoluble fiber.

The importance of biomaterials' fiber dimensions (length, width, thickness, and lumen width) on the physical and mechanical properties of products is well documented. Basiji et al. [40] have shown that, under certain conditions, impact strength and modulus of rupture in wood–plastic (polypropylene, PP) composites depends strongly on fiber length, whereas Singh and Samanta [41] report that increase in raw material fiber length enhances the mechanical properties of the natural fiber-reinforced composites. Using image analysis, Fidelis et al. [42] also found that the highest mechanical performance with tensile strength and Young's modulus in sisal and jute fibers could be accounted for by size of the cell walls and the real area of the fibers. Ghasemi et al. [43] have also suggested that the fiber cell wall thickness is an important parameter for biomass fibers to have excellent mechanical properties.

The revealing of cell structure and chemical composition of cellulosic fibers is important for assessing the suitability of various biomass fiber raw materials and ultimately can be useful in various applications of new biomaterials. In an extensive

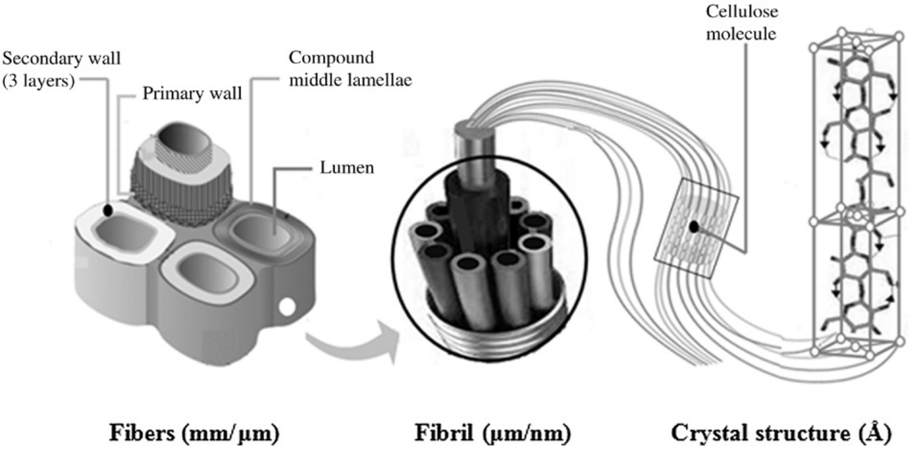

Figure 3.3 Model of cell structure of biomass-based natural fibers.

review of the literature, Abdul Khalil et al. [44] stressed that fibers have a wide range of variations in their properties, depending on various sources such as soil, genotype, climate, and agronomic practice. Moreover, Dittenber and Gangarao [45] investigated various factors affecting the fiber quality, such as plant growth, harvesting stage, and fiber extraction process. Norton et al. [46] suggested that chemical composition and cell structure of biomass fibers could be caused by soil component.

According to Rowell et al. [47], differences in cell structure in the biomass fiber will result in differences in physical properties. They reported that the physical properties of biomass fiber will be different because of differences in its morphology. A detailed overview of the morphological structure of biomass fiber by scanning electron microscopy (SEM) observation in this chapter will be useful to others in investigating fiber types.

Extraction is the process that separates the major components of biomass and converts it into fiber, lignin, and sugars for others to process into value-added products. This process separates the primary constituents of cellulosic biomass into three components (cellulose, hemicellulose, and lignin). This continuous process employs a cellulose extraction technique that removes lignin, resulting in a solid fraction containing a relatively pure cellulose or fiber.

Due to their main chemical composition, biomass fibers are also called cellulosic or lignocellulosic fibers. Those constituents are scattered throughout the cell wall, which consists of a primary and secondary wall. The portion of these chemicals in the cell wall layer is affected by the fiber origin, climate condition during cultivation, and the extraction method [46,48], and influences the fiber properties chemically and physically [49,50].

The bagasse fiber bundles shown in Fig. 3.4 were mechanically separated from sugarcane stem residue. Fig. 3.4 shows the surface roughness from the raw fiber state. According to Hemmasi et al. [51], Satyanarayana et al. [52], and Driemeier et al. [53], the diameter of bagasse fibers ranges from 10.10 to 34.21 μm. The cell wall thickness

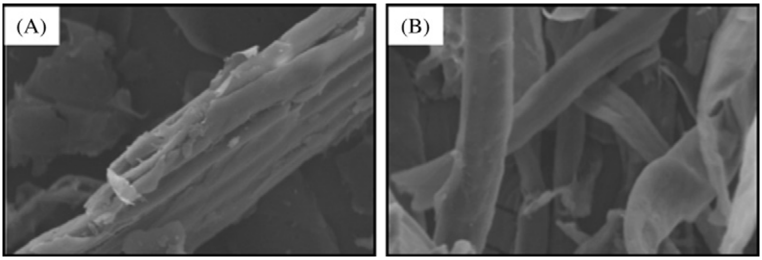

Figure 3.4 Scanning electron micrograph of morphological bagasse fiber: (A) raw material; (B) fibers after combined hydrothermal and alkaline pretreatment.
Source: From Guilherme A, Dantas PVF, Santos ES, Fernandes FAN, Macedo GR. Evaluation of composition, characterization and enzymatic hydrolysis of pretreated sugar cane bagasse. Braz J Chem Eng 2015;32(01):23–33 [54].

is reported to range from 4.85 to 5.64 µm. The cross-sections show fiber forms, with length of fibers ranging from 1.54 to 5.84 mm.

The percentage of chemical composition in bagasse fibers indicated a cellulose content of 32.33%–48.49%, which was higher than hemicellulose content of 19.25%–24.44%, while the whole bagasse contained lignin and ash at 17.3%–26.5% and 1.54%–5.35%, respectively [54,55].

In the longitudinal sections, the fiber bundles of rice straw have clean surface after the alkali and enzyme treatments [56]. There is still a great variety in shape and size of the bundles, as shown in the cross-sectional views. The fiber length ranges from 0.55 to 0.57 mm [57]. Fiber width and cell wall thickness range from 6.75 to 9.45 µm and 4.55 to 5.64 µm, respectively [58].

Rice straw ranges from 38.72% to 40.74% cellulose, 25.34% to 26.20% hemicellulose, 12.62% to 14.24% lignin, and about 16.33% to 16.99% ash [59,60]. Reddy and Yang [58] investigated fiber potential of rice straw, and reported that the cellulosic fiber formed by using alkali and enzyme treatment produced about 50% high-quality fibers.

Fig. 3.5 shows that pineapple fiber cross-section has a rougher structure, compact surface, and many fiber matrices [61]. Mishra et al. [62] investigated the microstructure of pineapple fiber through surface modifications by alkali treatment. They showed that these form fibers irregular cross section. Moya et al. [63] reported fiber diameter of 4.38–7.56 µm and a fiber length of 3.34–4.64 mm. Fig. 3.6 shows cell wall thickness in a range of ca. 1.46–2.30 µm [61,64].

Chemical constituents of pineapple fiber are composed of three main categories: cellulose (66.2%–74.3%), hemicellulose (19.5%–21.22%), and lignin (4.2%–10.5%), with some other small quantities (ash) at 2.0%–4.73% [61,65,66].

Fig. 3.6 shows coir fibers extracted from a coconut's outermost husk. These fibers have length ranging from 0.30 to 1.00 mm. The SEM image cross-sections reveal that the diameters and the wall thickness of the coir fiber cells exhibited variations from 8.90 to 19.33 µm and from 4.24 to 12.63 µm, respectively [35,52,67]. Based on Fig. 3.7,

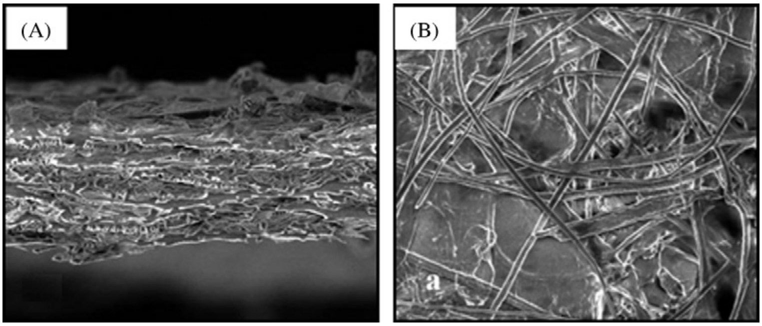

Figure 3.5 Scanning electron micrograph of morphological pineapple fiber: (A) cross-section image; (b) surface morphology.
Source: From Daud Z, Mohd Hatta MZ, Mohd Kassim AS, Awang H, Mohd Aripin A. Exploring of agro waste (pineapple leaf, corn stalk, and napier grass) by chemical composition and morphological study. BioResources 2014;9(1):872–80 [61].

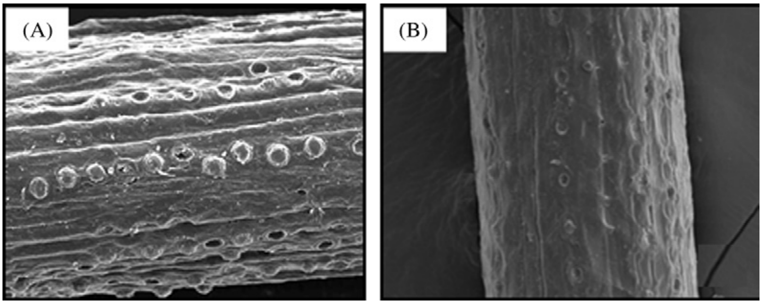

Figure 3.6 Scanning electron micrograph of morphological coir fiber: (A) raw material; (B) coir fiber–$NaClO_2$ treated fiber.
Source: From Muensri P, Kunanopparat T, Menut P, Siriwattanayotin S. Effect of lignin removal on the properties of coconut coir fiber/wheat gluten biocomposite. Composites Part A 2011;42:173–9 [72].

Arsène et al. [68] suggested that coir has a high lignin content, which helps make it resistant to burning [69], microbial attack [70], and moisture uptake/swelling [71].

Amount of cellulose in the coir was about 20.02%–22.90%, while hemicelluloses, lignin, and ash content were about 10.02%–14.70%, 44.75%–48.21%, and 1.00%–1.103%, respectively [68,73].

Banana fiber is a multiple-celled structure (Fig. 3.7). These fibers has large lumens, rare and fiber tips pointed and flat, ribbon like individual. In Fig. 3.8, banana fibers appeared to be quite parallel, with cell wall thickness whose size ranges from 1.12 to 1.57 μm [74]. The banana fibers were embedded in each bundle (Fig. 3.7); the diameter of those fibers was approximately 20.70–23.70 μm and fiber length ranged from 1.26 to 2.54 mm [52,58,75].

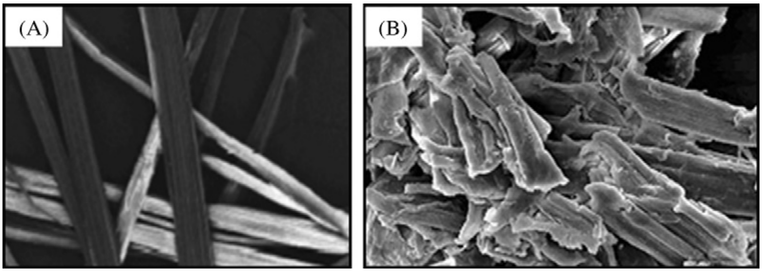

Figure 3.7 Scanning electron micrograph of morphological banana fiber: (A) raw material; (B) bleached banana fiber.
Source: From Deepa B, Abraham E, Cherian BM, Bismarck A, Blaker JJ, Pothan LA, et al. Structure, morphology and thermal characteristics of banana nano fibers obtained by steam explosion. Bioresour Technol 2011;102:1988–97 [77].

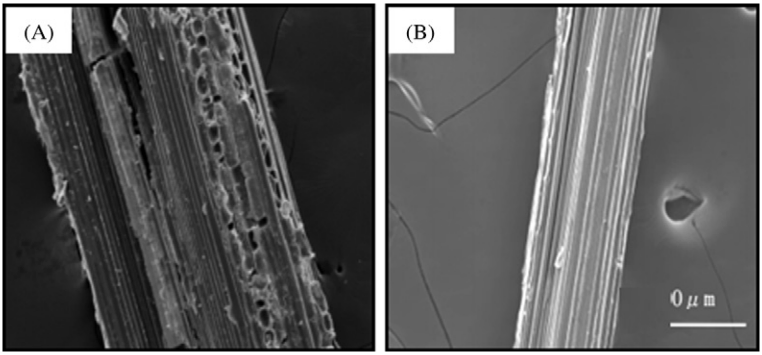

Figure 3.8 Scanning electron micrograph of morphological bamboo fiber: (A) raw material; (B) alkaline-treated fiber.
Source: From Phong NT, Fujii T, Chuong B, Okubo K. Study on how to effectively extract bamboo fibers from raw bamboo and wastewater treatment. J Mater Sci Res 2012;1(1): 144–55 [83].

Bilba et al. [76] characterized the chemical composition of banana. They reported that cellulose showed the highest value (27.87%–35.09%), followed by hemicellulose (12.95%–17.01%) and lignin (14.41%–15.73%). Ash content showed that the lowest value was about 8.62%–8.68% [68].

Fig. 3.8 shows a cross-sectional micrograph of a phloem fiber cap in a vascular bundle of a bamboo culm. It can be seen that the outer culm wall has high bending stiffness and strength [78]. Fig. 3.8 shows that these fibers are approximately 12.91–42.32 μm in diameter [79]. The length value of the fibers is 2.98–5.63 mm and cell wall thickness was obtained from 2.41 to 13.32 μm [80–82].

The investigations by Li et al. [84], Wang et al. [85], and Shibata [79] showed that the chemical composition of vascular bundles of a bamboo culm with cellulose,

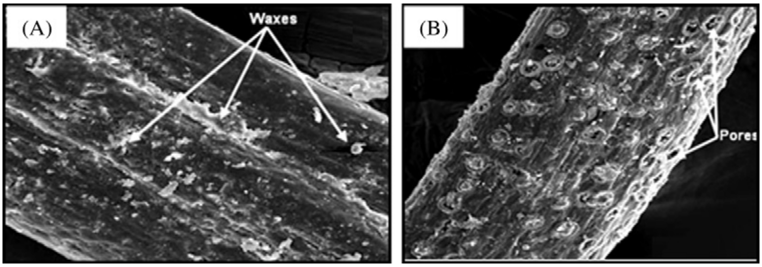

Figure 3.9 Scanning electron micrograph of morphological oil palm fiber: (A) raw material; (B) NaOH-treated.
Source: From Then YY, Ibrahim NA, Zainuddin N, Ariffin H, Md. Zin Wan Yunus W, Chieng BW. Static mechanical, interfacial, and water absorption behaviors of alkali treated oil palm mesocarp fiber reinforced poly(butylene succinate) biocomposite. BioResources 2015;10(1):123–36 [89].

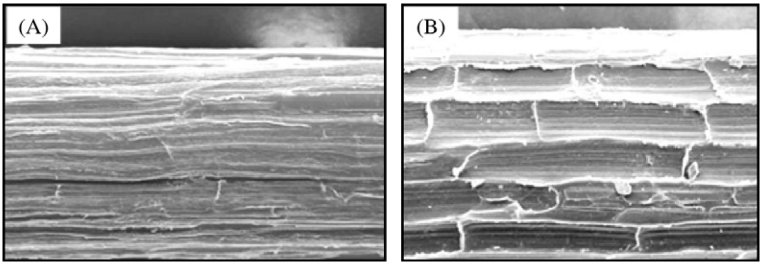

Figure 3.10 Scanning electron micrograph of morphological sisal fiber: (A) fiber surface images; (B) acetylated sisal fibers.
Source: From Fávaro SL, Ganzerli TA, de Carvalho Neto AGV, da Silva ORRF, Radovanovic E. Chemical, morphological and mechanical analysis of sisal fiber-reinforced recycled high-density polyethylene composites. eXPRESS Polym Lett 2010;4(8):465–73 [92].

hemicellulose, and lignin as the major components comprising about 20.3%–61.5%, 19.3%–21.4%, and 11.1%–32.2%, respectively. They also mentioned that ash content was 1.7%–5.1%.

As can be seen in Fig. 3.9, the cross-section of oil palm fiber is oval and fairly uniform in dimension. It contains various sizes of dimension such as length fiber of range from 0.33 to 50.31 mm, with fiber diameter and wall thickness of fiber cells varying between 8.30 and 20.50 μm and 2.83 and 4.35 μm, respectively [63,86,87]. The surface of oil palm fiber, shown in Fig. 3.10B, was relatively porous and rough; these fibers have silica-like bodies with rounded shape [88]. Abdul Khalil et al. [75] and Law et al. [88] suggested that the chemical composition of the oil palm varied. Cellulose, hemicellulose, and lignin contents were 14.3%–65.2%, 12.5%–38.7%, and 17.3%–26.5%, respectively.

The length of sisal fiber is between 0.85 and 1.00 mm and the diameter is about 100–300 μm, with wall thickness of fiber cells between 11.25 and 12.50 μm [90,91].

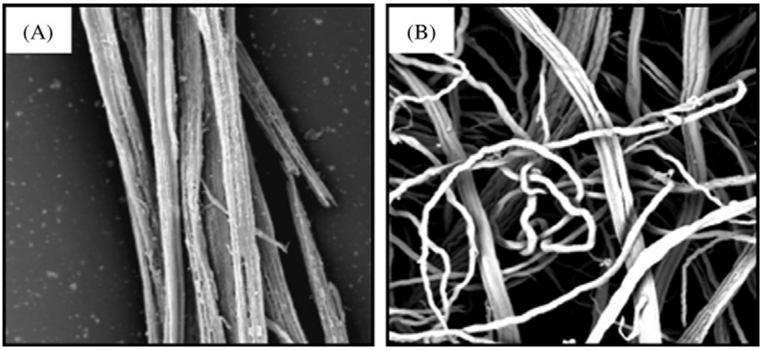

Figure 3.11 Scanning electron micrograph of morphological jute fiber: (A) raw material; (B) alkali-treated.
Source: From Baheti VK, Abbasi R, Militky J. Ball milling of jute fibre wastes to prepare nanocellulose. World J Eng 2012;9(1):45–50 [94].

In principle, sisal fibers have considerable surface roughness (Fig. 3.10) and are microstructurally heterogeneous. The sisal showed the presence of cellulose, hemicellulose, and lignin with 43.85%–56.63%, 21.12%–24.53%, and 7.21%–9.20%, respectively. This, to a great extent, is in agreement with investigation reported by Fávora et al. [92].

The surface morphology of jute fibers depended on their thickness, which was due to variation in fiber maturity [93]. The pores and voids could be decreased in number on the top portion of the fiber surface as compared to the surfaces of the middle and cutting portions. Jute fibers presented few fiber cells; cell wall thickness was 2.5 μm [42], diameter of lumens 60.00–110.00 μm; and fiber length 3.00–3.50 mm [90] (Fig. 3.11).

According Mwaikambo [95], the presence of cellulose and hemicellulose in the raw jute was 69.21%–72.35% and 12.55%–13.65%, respectively. Lignin, the major element present, was quite higher, at 12.67%–13.21% [94].

Fig. 3.12 shows abaca fibers. In the cross-section of the fiber bundle shown in Fig. 3.12A, there is a large lumen in the center of every cell. Fig. 3.12B shows that the surface of abaca fiber bundles is composed of polygonal cells of 18.56–21.69 μm in diameter with a cell wall thickness of 4.07–5.11 μm [96]. The fiber length as shown in Fig. 3.14B is 4.14–5.05 mm [97].

Cellulose and hemicellulose are the most essential chemical components found in lignocellulosic materials such as abaca. This was demonstrated by Del Rio and Gutiearrez [99] using energy-dispersive X-ray spectroscopy, which showed cellulose and hemicellulose contents as 69.23%–70.64% and 21.22%–21.97%, respectively. Other researchers reported that abaca contains lignin up to 5.87% [99,100].

The morphology of kapok fibers shows a hollow structure with a thin fiber wall and large lumen (Fig. 3.13A). These fibers have width, lumen diameter, cell wall thickness, and fiber length of 8.14–10.90, 12.10–16.90, 0.80–1.00, and 20–30 μm, respectively [101,102]. In their application, these fibers are good for stuffing beds,

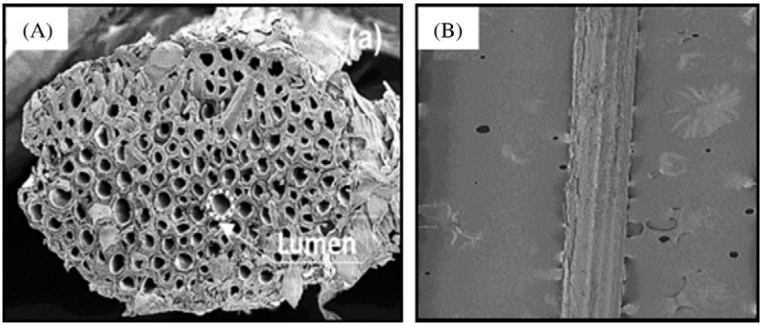

Figure 3.12 Scanning electron micrograph of morphological abaca fiber: (A) surfaces of abaca fiber bundles; (B) longitudinal sections.
Source: From Cai M, Takagi H, Nakagaito AN, Katoh M, Ueki T, Waterhouse GIN, et al. Influence of alkali treatment on internal microstructure and tensile properties of abaca fibers. Ind Crops Prod 2015;65:27–35 [98].

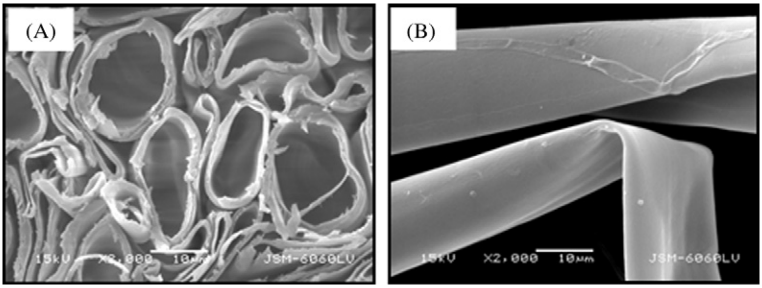

Figure 3.13 Scanning electron micrograph of morphological kapok fiber: (A) cross-section; (B) longitudinal view.
Source: From Rijavec T. Kapok in technical textiles. Tekstilec 2008;51(10–12):319–31 [105].

due to their light weight and the fact that they are too inelastic to be spun (Fig. 3.13B). Kapok normally consists of about 65.63%–69.87% cellulose and 5.46%–5.63% lignin [103]. According to Anigo et al. [104], kapok also contains 6.66%–10.49% hemicellulose, and 2% ash.

Fig. 3.14B shows the structure of kenaf fibers is coarse morphology, where fibers are not uniform, and are round polygonal in shape. Meanwhile, the presence of lignin and impurities (silica nodules) is shown in Fig. 3.14B [106]. Single fibers of bast kenaf were bound in a bundle of approximately 5.74–26.59 µm diameter and 2.27–2.51 mm length fiber. The mean vessel diameter and cell wall thickness are 284 and 6.39 µm, respectively [17]. The cellulose content (37.50%–63.00%) is higher in the bast part than in the core of kenaf. The lignin varies on average from 18.00% to 24.30%, while hemicellulose content is 15.10%–21.40% [75,107]. Most wax is deposited in the epidermis, where wax content affects the fiber properties of kenaf [108].

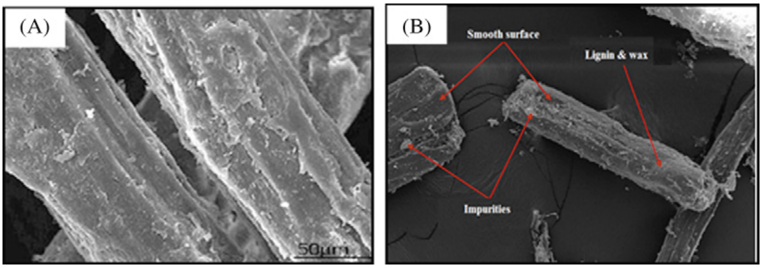

Figure 3.14 Scanning electron micrograph of morphological kenaf fiber: (A) transverse section of bast fibers; (B) the raw kenaf fiber bundles.
Source of A: From Jonoobi M, Harun J, Shakeri A, Misra M, Oksman K. Chemical composition, crystallinity, and thermal degradation of bleached and unbleached kenaf bast (*Hibiscus cannabinus*) pulp and nanofibers. BioResources 2009;4(2):626–39 [109]. *Source of B*: From Safinas A, Saad Md, Bakar AA, Ismail H. Properties of kenal bast powder-filled high density polyethylene/ethylene propylene diene monomer composites. BioResources 2013;8(2):2386–97 [108].

3.3.3 Physical and mechanical properties

The physical and mechanical properties of biomass fibers are important and closely related to the structure of the biomass fibers themselves. The biomass fibers are basically natural organic fibers, and show high variability in their various properties. This poses different problems in characterizing the quality of fibers' physical and mechanical properties. The most important physical property is density, while the mechanical properties of single fibers are measured using value of modulus and tensile strength. It is important to mention biomass fibers as biomaterials for development and fabrication of polymer composites [110,111].

Finally, it is seen from literature that biomass fibers have the best potential for filler/reinforcement in polymer composites. A comparison of physical and mechanical properties of selected biomass fiber is given in Table 3.3.

3.4 Bionanomaterial from agricultural waste

Bionanomaterial from lignocellulosic biomass is rapidly growing with production of cellulose nanofibrils or nanoparticles. Both types of nanocellulose materials are used in various applications due to their low density, optical transparency, high mechanical properties, large surface area (aspect ratio), flexibility, specific barrier properties, low thermal expansion, biodegradability, and environmentally friendly nature [123–125]. The technique for the production of nanocellulose can be through mechanical, chemical, and chemomechanical treatment processes [126–132]. With the appropriate modification and characterization, nanocellulose could broaden the applications of biobased polymers to the great benefit of many industries, such as transparent films [133], strength enhancers in paper [134], reinforcements for polymer composite [135],

Table 3.3 Physical and mechanical properties of selected agricultural waste fibers

Fiber source	Density (g/m^3)	Tensile strength (MPa)	Young's modulus (GPa)	References
Oil palm	0.7–1.55	227.5–278.4	2.7–3.2	[86,51]
Bagasse	0.31–1.25	257.3–290.5	15–18	[53]
Banana	0.65–1.36	51.6–55.2	3.00–3.78	[64,112,113]
Coconut (coir)	0.67–1.15	173.5–175.0	4.0–6.0	[64,113]
Pineapple	1.25–1.60	166–175	5.51–6.76	[64]
Rice straw	0.86–0.87	435–450	24.67–26.33	[114,58]
Jute	1.3–1.45	300–700	20–50	[115,116,91]
Kenaf	0.15–0.55	295–955	23.1–27.1	[117,118,119]
Bamboo	0.6–1.1	360.5–590.3	22.2–54.2	[120]
Sisal	1.45–1.5	300–500	10–30	[115,116,91]
Abaca	1.42–1.65	879–980	38–45	[91]
Kapok	0.68–1.47	80.3–111.5	4.56–5.12	[103,121,122]

Source: From Hemmasi AH, Khademi-Eslam H, Pourabbasi S, Ghasemi I, Talaiepour M. Cell morphology and physico-mechanical properties of HDPE/EVA/Rice hull hybrid foamed composites. BioResources 2011; 6(3): 2291–308 [51]; Driemeier C, Santos WD, Buckeridge MS. Cellulose crystals in fibrovascular bundles of sugarcane culms: Orientation, size, distortion and variability. Cellulose 2012; 19: 1507–15 [53]; Reddy N, Yang Y. Properties of High-Quality Long Natural Cellulose Fibers from Rice Straw. J Agric Food Chem 2006; 54(21): 8077–81 [58]; Alwani MS, Abdul Khalil HPS, Islam MN, Sulaiman O, Zaidon A, Dungani R. Microstructural study, tensile properties, and scanning electron microscopy fractography failure analysis of various agricultural residue fibers. J Nat Fibers 2015; 12: 154–68 [64]; Vijayalakshmi K, Neeraja CYK, Kavitha A, Hayavadana J. Abaca fibre. Trans Eng Sci 2014; 2: 16–9 [91]; Chaiarrekij S, Apirakchaiskul A, Suvarnakich K, Kiatkamjornwong S. Kapok I: Characteristcs of kapok fiber as a potential pulp source for papermaking. BioResources 2011; 7: 475–88 [103]; Sumaila M, Amber I, Bawa M. Effect of fiber length on the physical and mechanical properties of random oreinted, nonwoven short banana (Musa balbisiana) fibre/epoxy composite. Asian J Nat Applied Sci 2013; 2: 39–49 [112]; Sakthivel M, Ramesh S. Mechanical properties of natural fiber (banana, coir, sisal) polymer composites. Sci Park 2013; 1: 1–6 [113]; Bouasker M, Belayachi N, Hoxha D, Al-Mukhtar M. Physical characterization of natural straw fibers as aggregates for construction materials applications. Materials 2014; 7: 3034–48 [114]; Alves C, Ferrao PMC, Freitas M, Silva AJ, Luz SM, Alves DE. Sustainable design procedure: The role of composite materials to combine mechanical and environmental features for agricultural machines. Mater. Design 2009; 30: 4060–8 [115]; Bongarde US, Shinde VD. Review on natural fiber reinforcement polymer composites. Int J Eng Sci Innovat Technol 2014; 3: 431–6 [116]; Wambua P, Ivens J, Verpoest I. Natural fibres: can they replace glass in fibre reinforced plastics? Compos Sci Technol 2003; 63(9): 1259–64 [117]; Munawar SS, Umemura K, Kawai S. Characterization of the morphological, physical, and mechanical properties of seven non-wood plant fibre bundles. J Wood Sci 2007; 53(2): 108–13 [118]; Paridah M, Khalina A. 2009. Effects of soda retting on the tensile strength of kenaf (Hibiscus cannobnius L.) bast fibres. Project Report Kenaf EPU 14 (Suppl. 1), 2009: 21–28 [119]; Rathod A, Kolhatkar A. Analysis of physical characteristics of bamboo fabrics. Int J Res Eng Technol 2014; 03(08): 21–25 [120]; Mwaikambo LY, Ansell MP. The determination of porosity and cellulose content of plant fibers by density methods. J Mater Sci Lett 2001; 20: 2095–6 [121]; Mojica ERE, Merca FE, Micor JRL. Fiber of kapok (Ceiba pentandra) as component of a metal sensor for lead in water samples. Philippine J Crop Sci 2002; 27: 37–42 [122].

emulsions and oxygen barrier films for plastics packaging [136,137], and many others [138].

There are various studies on lignocellulosic biomass as filler or reinforcement in polymer composites, such as coir fiber in PP composite [139], bagasse fiber in thermoplastic composites [140], nanocellulose sisal fiber-reinforced polyolefin composites [141], carbonized jatropha seed shell as filler in vinyl ester biocomposites [142], nanocellulose fiber char in poly(vinyl acetate) formed composites [143], oil palm shell (OPS) nanoparticles as filler in polyester hybrid composites [15], etc. When incorporated in polymer matrices, nanosized cellulose could impart higher stiffness to the nanocomposites. It is an ideal reinforcement agent in polymer composites due to its large aspect ratio resulting from interconnected network structures through hydrogen bonding.

The morphology, dimension, crystallinity, and surface chemistry are key properties of nanocellulose for end use. A variety of cellulosic biomass other than wood fiber, such as oil palm, kenaf, rice straw, bamboo, bagasse, and pineapple have been utilized for the extraction of nanocellulose [77,144]. Selecting the cellulosic biomass depends on the availability of the fiber in a country, the chemical components for its application, and economic considerations [37]. In spite of being the most abundant cellulose biomaterial on earth, the processing of cellulose into types of nanostructures has only recently received considerable attention. Over the past few decades, lignocellulosic biomass has attracted a great deal of scientists and researchers using biomaterials to isolate nanocellulose to fabricate diverse functional materials.

3.4.1 Properties and characterization of CNFs from agricultural waste

Many studies have been done on isolation from biomass and allowing different kinds of nanoscaled cellulosic fillers to be obtained. However, the biomass structure of agricultural waste consists of inherent properties, such as strong lignin layers, low cellulose accessibility to chemicals, and high cellulose crystallinity, which inhibit the digestibility of the biomass for cellulose extraction. Some biorefinery processes are necessary to deconstruct noncellulosic content in lignocellulosic biomass, while maintaining cellulose product for further hydrolysis into nanocellulose material [145,146]. Lignocellulosic plants first require the breakdown of the supramolecular cell wall structure, thus increasing accessibility to the polysaccharide components of the raw lignocellulose. The next step is to break down the cross-linked elements in the raw material (lignin, cellulose, pectin, and hemicellulose) to increase the accessibility to the cellulose microfibrils [147]. Nanocellulose extracted by processing conditions and different cellulosic source can be classified into nanofibrillated cellulose (NFC) and nanocrystalline cellulose (Fig. 3.15).

Generally there are two main families of CNFs, differing in size and crystallinity, which are cellulose nanocrystal (CNC) and NFC [127,148]. CNC is also known as nanowhiskers [150,151], nanorods [152], and rod-like cellulose crystals [152]. CNC is usually isolated from cellulose fibers through acid hydrolysis, using sulfuric acid, hydrochloric acid, etc. [153,154].

The nanoscale structure of nanocellulose was revealed by transmission electron microscopy (TEM). TEM images of the nanocellulose (CNC and NFC) suggest transverse cleavage of microfibril fibers into free and individualized nanocellulose (Fig. 3.16). Furthermore, diameter of nanocellulose had a gradual decrease due to the progressive removal of the amorphous portion of cellulose fiber with increase in process time [155]. Surface morphology from SEM analysis shows the form of the

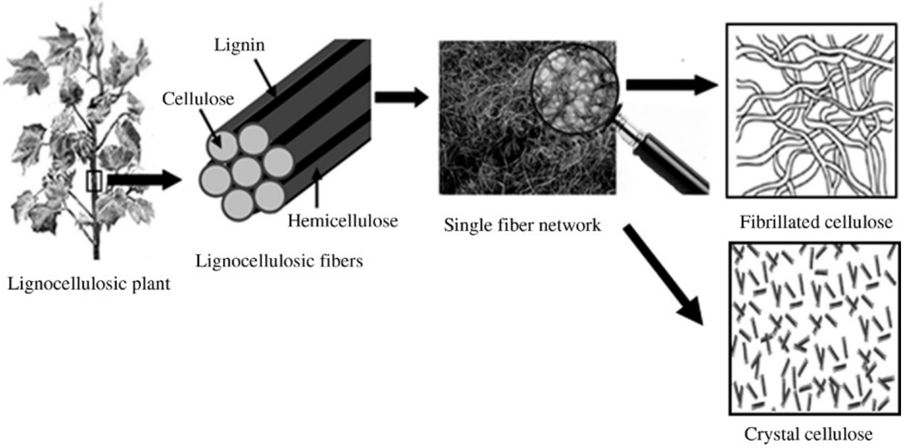

Figure 3.15 The schematic principle of isolating noncellulosic content in lignocellulosic biomass into CNFs.

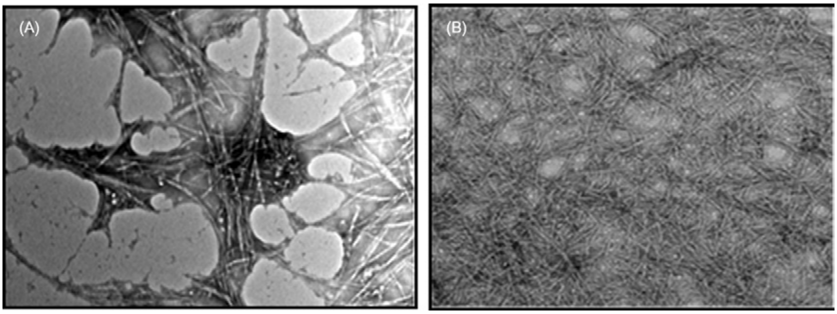

Figure 3.16 TEM image of nanocellulose from agricultural waste: (A) TEM image of nanocrystal/nanowhiskers; (B) TEM image of NFC.
Source of A: From Saurabh CK, Dungani R, Owolabi AF, Atiqah NS, Zaidon A, Aprilia NAS, et al. Effect of hydrolysis treatment on cellulose nanowhiskers from oil palm (*Elaeis guineesis*) fronds: morphology, chemical, crystallinity, and thermal characteristics. BioResources 2016;11(3):6742–55 [158]. *Source of B*: From Fatah IYA, Abdul Khalil HPS, Hossain MdS, Aziz AA, Davoudpour Y, Dungani R, et al. Exploration of a chemo-mechanical technique for the isolation of nanofibrillated cellulosic fiber from oil palm empty fruit bunch as a reinforcing agent in composites materials. Polymers 2014;6:2611–24 [159].

smooth surface of the fiber and the individual in a bundle [156,157]. TEM images of the nanowhiskers suggest that amorphous regions from fiber are removed through transverse cleavage of microfibril fibers (Fig. 3.16A). On the other hand, NFC is flexible and has an entangled network structure, matted and not individualized, therefore the total length cannot be determined, as seen in Fig. 3.16B.

A NFC is a fiber resulting from isolation process of lignocellulosic biomass with a diameter of 100 nm or less and length of several micrometers. Reported by Chao et al.

[160], the treated cellulose nanofibril has diameter distribution in the range 15–35 nm, which was similar to that of the untreated CNF (about 15–40 nm). Processing this biomass presents a large crystalline region with 150 m^2/g on specific surface area, also called microfibrillated CNFs. CNC has a perfect crystalline structure and high modulus, close to the theoretical modulus of cellulose [148].

There are various reports on isolation of nanocellulose from nonconventional agricultural waste, including pineapple leaf fibers [161], grass [162], rice husk [163,164], rice straw [165], empty fruit bunches [129,166], etc. In these types of nanocellulose, after applying various pretreatments, tailor-made nanofibrils with specific morphology and surface chemistry are produced. Different properties of these two types of nanocellulose will result in varying reinforcement of nanocomposites to the thermoplastic and thermoset of polymer matrices. As well as being completely renewable, safer to handle, and cheaper to produce, nanocellulose materials also possess exceptional physical and chemical properties.

Applications being developed for nanofibers include stimulating the production of carbon nanofibers, which can improve the performance of flame retardant in furniture [167]. Many methods have been made to isolate the cellulose fibers. Alemdar and Sain [168] imply that cellulose chains are packed in an ordered manner to form compact microfibrils, which are stabilized by both inter- and intramolecular hydrogen bonding. CNC are very polar and attract each other by H bonding, so that treatment by acid hydrolysis formed separating individual crystals. Isolating the CNFs can be done by steam explosion method [169], high-intensity ultrasonication combined with chemical pretreatments [170], 2,2,6,6-tetramethylpiperidine-1-oxyl radical (TEMPO) method [10], acid treatment, ultrasonication method [171], and enzymatic hydrolysis [172].

In recent years, biological pretreatment using microorganisms has been strongly beneficial. The formation of CNF with microorganisms as tools gives various advantages, such as nontoxicity, biodegradability, and low inhibitory factors. Some fungi, bacteria, and yeast have the ability to degrade cellulosic contents in plants using their enzymatic mechanism, and the resulting products can be used for nanofiber materials [173–177]. In the near future, the optimized use of microorganisms as pretreatment agents is expected to be an efficient method for cellulose degradation and will contribute to the production of CNF.

The mechanical properties of CNC contain a small number of defects [178]. The axial elastic modulus is close to that derived from theoretical chemistry and is potentially stronger than steel and similar to Kevlar [179]. The first report showed that Young's modulus of CNC is 130 GPa [180], and then Zimmerman et al. [181] reported this value as 250 GPa. However, Eichhorn [182] showed that the modulus of tunicate cellulose nanowhiskers was 143 GPa. Menezes et al. [179] imply that experimentally the elastic modulus is around 137–167.5 GPa. CNC has higher strength than steel and higher stiffness than aluminum. It has elastic modulus and bending strength of 138–167 and 10 GPa [183,184]. CNC has high availability, light weight, and high mechanical properties. It consists of slender parallelepiped rods, depending on its origin, and the lateral dimensions range from about 2 to 50 nm in diameter for length than can reach several tens of micrometers [185].

Iwamoto et al. [186] evaluated the elastic modulus of single NFC using TEMPO oxidation and acid hydrolysis treatment. They showed elastic modulus values of

145.2 ± 31.3 and 150.7 ± 28.8 GPa, respectively. They considered that NFC has large surface area that increases its interaction with secondary materials at the nanolevel. Thus it can be concluded that crystalline domains of nanocellulose resulted in an increase in mechanical properties of nanocellulose-incorporated composites.

The thermal characterization of NFC was conducted using thermogravimetric and derivative thermogravimetric curves analysis. These were used to determine its potential use in high temperature applications. Thermal analysis showed that the weight loss of NFC was observed from 50 to 150°C; at this range of temperature, the moisture content evaporates [187]. The degradation behavior of agriculture waste started at 290°C [168] and 300°C [187]. The degradation temperature of CNFs of agriculture waste started at 300°C and continued up to 400°C [188]. Nuruddin et al. [187] showed that the degradation temperature for the microfibrils extracted from rice straw started at 332°C and continued up to 370°C, where all cellulose was pyrolyzed, the solid residues being of about 20%. In general, the sulfate groups, smaller fiber dimensions, and crystal structure of CNC prepared by sulfuric acid hydrolysis on the surface could promote the thermal degradation [189,190].

The physical characterization of NFC based on the morphology and dimension has been conducted with TEM and AFM analysis by different methods. Many studies have found a difference in characterization of NFC, from 1 to 50 nm in thickness and by several μm in length [146]. The results of TEM show that the NFC from agricultural waste obtained after the chemomechanical treatment typically are 100–300 nm in length and 5–20 nm in width [191]. The dimensions of CNC depend on the sources; they depend strongly on the processing techniques and the prepared samples exhibited distinct features. With acid hydrolysis method, stronger acidity, higher temperature, and longer reaction time might yield shorter CNC [192].

The structure of CNC was studied using X-ray diffraction (XRD). XRD is used to investigate the effect of chemical and mechanical treatments on the crystallinity and crystal type of the NFC. In all cases, the cellulose crystal structure of nanocellulose fiber indicated that the native cellulose crystal structure was preserved [77,188]. With sulfuric acid hydrolysis method, strong acid hydrolysis usually resulted in removal of the amorphous areas and a higher crystallinity during hydrolysis [78,193]. However, TEMPO-mediated oxidation did not influence the crystalline structure of isolated cellulose [194–196]. Furthermore, several researchers have reported the diffraction peak of CNFs at the 110, 200, 004 crystallographic plane [138,197] and the crystallinity index of CNFs between 70% and 80% [198].

3.4.2 Properties and characterization of nanoparticles from agricultural waste

Nanoparticles have one dimension that measures 100 nm or less. In other words, we can say that they are the collection of atoms bonded together with a structural radius of less than 100 nm. The properties of many conventional materials change when formed from nanoparticles. This is typically because nanoparticles have a greater surface area per weight than larger particles, which causes them to be more reactive to some other molecules. Nanoparticles are very ordinary in nature, for instance,

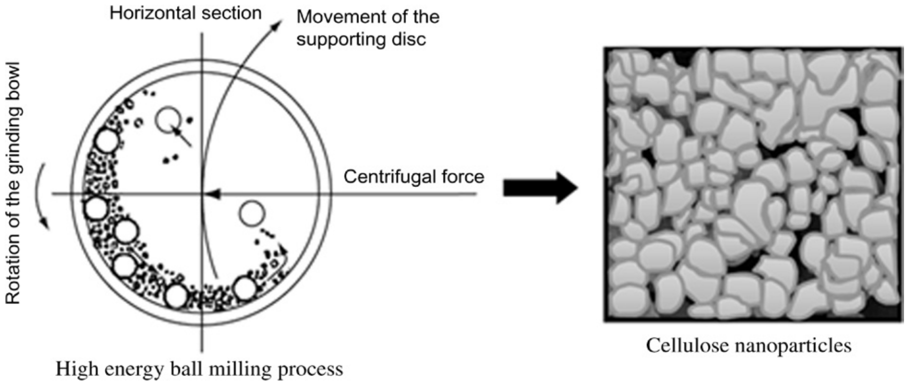

Figure 3.17 The schematic principle of pulverization of lignocellulosic biomass by high grinding energy to produce cellulose nanoparticles.

proteins exist in almost all biological systems. These can include, e.g., fullerenes, metal clusters, large molecules such as proteins, and even hydrogen-bonded assemblies of water molecules, which exist in water at ambient temperatures.

Until recently, metallic nanoparticles, especially silver nanoparticles, were considered as the most promising due to their large surface-area-to-volume ratio. The use of renewable plant material on synthetics of silver nanoparticles offers enormous benefits as a viable alternative for the development of metal nanoparticles because of their wide range of applications [199]. Agricultural wastes such as sesame husk [200], OPS [14], oil palm ash [201], and coconut shell (CS) [202] have been used to generate cellulose nanoparticles.

Many attempts have been made to use agricultural wastes to produce nanoparticles by a variety of chemical and physical methods. The combined action of chemical treatment and high-energy planetary ball-milling process is another effective method of nanoparticles synthesis [94]. The principle of this combined method is that chemical treatment removes lignin and hemicelluloses from cellulosic materials and then the ball-milling process further grinds the material into powder form, which is nothing but cellulose nanoparticles. The principal properties of nanoparticles include size, shape, and surface structure, and processing tends to introduce surface imperfections (Fig. 3.17). These surface imperfections can significantly impact on the overall nanoparticle surface physicochemical properties [203,204].

Lignocellulosic biomass consists of polymeric materials that contain different amounts of oil. The presence of remaining oil within the lignocellulosic biomass such as oil palm, coconut, and jatropha is one challenge. The second challenge that was discovered was irreversible adsorption and aggregation of nanoparticles when solvents were removed during purification, which led to significant loss of material and created a problem afterward [205]. Any impurity and contamination on the particle will lower the effect of this biomass for advanced applications such as composites [206], pulp and paper production, etc. The oil removal process is crucial to eliminate this problem, and could also benefit further applications. Several methods can be

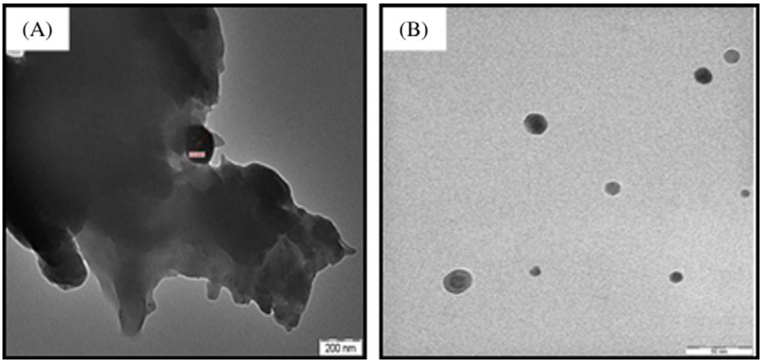

Figure 3.18 TEM images of nanoparticle CS. (A) Before extraction; (B) after extraction.

used to separate oil from the lignocellulosic biomass; these include mechanical pressing, supercritical fluid extraction, and solvent extraction [207,208]. The successful extraction of oil removal of lignocellulosic biomass nanoparticles was reported by Sulaeman et al. [202]. Using an soxhlet extraction unit (Soxtec 2043, Foss, Hillerød, Denmark), which connected to a reaction flask containing 250 mL of *n*-hexane, the resulting nanoparticles showed a clear increase in size and the elimination of the remaining oil within the nanoparticle samples (Fig. 3.18).

As a filler, nanoparticles from agriculture waste could be made in the form of flour, carbon, fiber, etc. When the three dimensions of particulates are in the order of nanometers, they are called isodimensional nanoparticles. They include spherical nanoparticles, nanogranules, and nanocrystals [209]. The filler may be selected such as flours from CS [202], OPS [14], olive stone and pecan shell [210], wood bark [211], wood flour such as soft wood, hard wood, and free bark flours [212], and other cellulosic fillers.

The particle size and size distribution play a crucial role in property characterization of nanoparticles. These properties are chemical, physical, electronic, thermal, magnetic, and mechanical. For example, a study on thermal properties of CS showed that CS nanoparticles had more thermal stability when the size reached to the nanometer scale compared with raw CS [202]. Hence, the novel properties of nanoparticles do not prevail until the size has been reduced to the nanometer scale [213]. In other words, the functional properties of nanoparticles are significantly different from the properties of the bulk material having the same chemical composition. The particle size and size distribution of nanoparticles can be determined with microscopic techniques and utilizing the relationship between particle behavior and size. There are numerous commercially available instruments that can be used for determining particle size and size distribution of nanoparticles, such as TEM, SEM, dynamic light scattering (DLS), X-ray diffraction (XRD), photon correlation spectroscopy (PCS), AFM, Brunauer–Emmett–Teller (BET), etc. Many studies have been made on particle sizing of nanoparticles using different instruments. For example, Dungani et al. [214] investigated the particle size of OPS by TEM. They showed that OPS particle

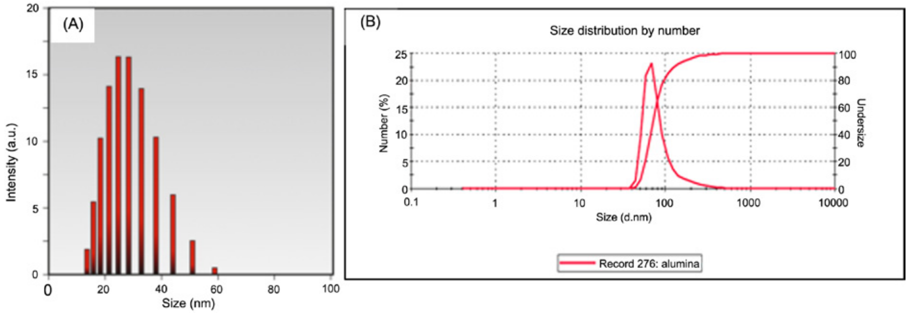

Figure 3.19 Particle size distribution of the nanoparticles. (A) DLS image; (B) PCS image. *Source of A*: From Mollick MdMR, Rana D, Dash SK, Chattopadhyay S, Bhowmick B, Maity D, et al. Studies on green synthesized silver nanoparticles using *Abelmoschus esculentus* (L.) pulp extract having anticancer (in vitro) and antimicrobial applications. Arabian J Chem 2015;doi: 10.1016/j.arabjc.2015.04.033 [215]. *Source of B*: From Akbari B, Tavandashti MP, Zandrahimi M. Particle size characterization of nanoparticles—a practical approach. Iran J Mater Sci Eng 2011;8(2):48–56 [213].

size ranged from 10 to 50 nm with average of particle size close to 50 nm. They also reported that the average particle size was determined from X-ray diffraction peaks using XRD images, with the average size of OPS nanoparticles calculated to be 31.75 nm. By comparing the results from different instruments, one can obtain extra information about the system. Fig. 3.19 shows the DLS and PCS techniques used to determine the average size and particle size distribution of nanoparticles.

Nanoparticles possess a variety of morphologies and their names are characterized by their different shapes. These morphologies sometimes arise spontaneously as an effect of a templating or directing agent during synthesis. Ghaedi et al. [216] investigated the surface morphology of the activated carbon-derived nanoparticles from medlar wood. They showed that a surface morphology could be achieved that is homogeneous and relatively smooth and dense with a large number of pores and cavities in different sizes and shapes. They also observed that the adsorbent exhibited nearly narrow pore size distribution in the mesoporous domain with average pore diameter lower than 10 nm. The functional properties of nanoparticles highly depend on the surface morphology of the particles, so precise measurements of a particle's morphology enable reliable characterization of the nanoparticle's properties. Controlling the morphology of nanoparticles is of key importance for exploiting their properties. For example, surface functionalization of silica nanoparticles with coating polymer by brushes [217] and a thin polymer film [218] is very important as the polymer coating alters the interfacial properties, and thus the mechanical and thermal properties of the matrix polymers can be altered by the compatibility of the nanoparticles within the matrix.

SEM analysis was employed to visualize the size and shape of nanoparticles. From the SEM analysis, it was found that silica nanoparticles from agricultural waste have their own shape and size arrangement [219]. These results suggested that the waste

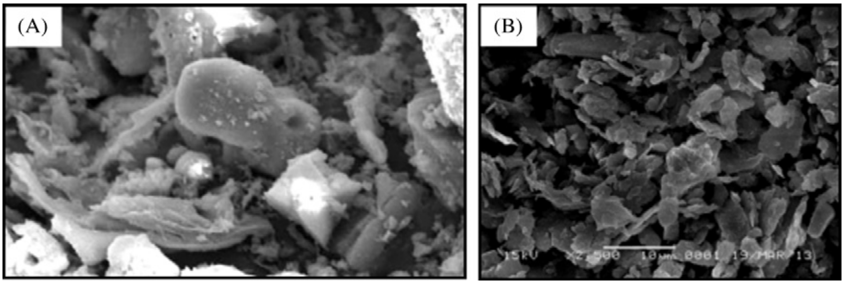

Figure 3.20 SEM micrographs of ground silica powder (A) bagasse ash; (B) CS.
Source of A: From Hariharan V, Sivakumar G. Studies on synthesized nanosilica obtained from bagasse ash. Int J ChemTech Res 2013;5(3):1263–6 [220]. *Source of B*: From Sulaeman A, Dungani R, Islam MN, Abdul Khalil HPS, Sumardi I, Hermawan D, et al. Preliminary study of characterization of nanoparticles from coconut shell as filler agent in composites materials. MAYFEB J Mater Sci 2016;1(2016):1–9 [202].

materials are converted to ash by sintering at 900°C and silica powders characterized by SEM showed bamboo leaf-like particles. However, the groundnut shell ash shows uniform spherical particles. They also observed that rice husk ash has a fiber-like appearance, whereas, sugarcane bagasse ash was highly porous on its structure. Hariharan and Sivakumar [220] studied the waste material bagasse ash as a material from which to obtain nanosilica. They reported nanosilica with various sizes and prismatic and spherical geometry (Fig. 3.20A). SEM imaging on CS nanoparticles found a structure with angular, irregular, and crushed shapes [202]; the authors also reported that these structures broke down after high-energy ball milling (Fig. 3.20B).

TEM is employed to determine the morphology, shape, and size of nanoparticles. Fig. 3.21 shows the TEM images of silica nanoparticles of rice husk ash. Fig. 3.21A shows that the particles are dispersed (heterogeneity). Fig. 3.21B shows that the majority of particles are in the 60–70 nm size range and there are some larger particles in the 105–112 nm range [221].

Fourier transform infrared spectroscopy (FTIR) is used to examine the surface chemistry as the organic functional groups that are attached to the surface of nanoparticles. Ghorbani et al. [222] and Chen et al. [223] investigated the organic functional group of silica nanoparticles of extracted rice husk at combustion temperatures of 600°C and 700°C, respectively. FTIR analysis has detected that the vibration signals at 1075, 780, and 665 cm^{-1} are typical of Si–O–Si bands, which confirms the presence of silica nanoparticles [224]. These three peaks are the main indices of silica materials, which represent successful production [222]. Synthesis of nanoparticles has been developed from banana peel [225], which show characterization of FTIR was a shift in the 2930–2924, 2353–2344, 1732–1726, 1640–1630, 1532–1533 and 1445–1451 cm^{-1}. They also reported that the main surface function groups present as amide group; amino group; and methyl, methylene, and methoxy groups. The main surface functional groups in CS nanoparticles obtained by ball milling process present as a combination of hydroxyl (OH), methylene groups (C–H), carbonyl groups (C=O), and ethers (C–O–C) [202].

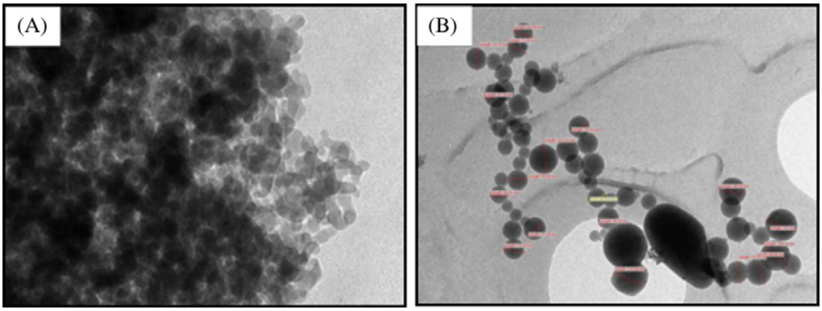

Figure 3.21 TEM image of silica nanoparticles of rice husk ash. (A) The particles are dispersed; (B) a heterogeneity in size.
Source: From Djangang CN, Mlowe S, Njopwouo D, Revaprasadu N. One-step synthesis of silica nanoparticles by thermolysis of rice husk ash using non toxic chemicals ethanol and polyethylene glycol. J Appl Chem 2015;4(4):1218–26 [221].

3.5 Various applications of bionanomaterial

Nanotechnology represents a major opportunity for wood and plant-based materials to improve their performance and functionality, develop new generations of products, and open new market segments in the coming decades. Research on nanocellulose and commercial development for sustainable utilization of biomass is ongoing, mainly in Japan, North America, and Europe. The current market situation continues to grow as the worldwide demand for new products on the market. As the worldwide demand for fiber grows, so does the demand for sustainable resource management and efficient industrial utilization. This means that nanocellulose is a prime candidate for use as sustainable and recycling-based material in industries such as packaging, automotive components, biocomposites, etc.

With improvement techniques of the selected biomass fibers and extraction technologies, as well as modification and characterization, nanocellulose can be applied to composite-based products. Applications of surface-modified nanofibrillar cellulose are for advanced materials such as high-performance nanocomposite materials and films, medical, pharmaceutical, cosmetics, automotive, electronic industries, aircraft manufacturers, and paper and printing industries. Numerous works are underway regarding nanocellulose-based products from various cellulosic sources and their applications [226].

The market for nanomaterials in various products such as structural components continues to grow, mainly driven by the demand for materials that have a high strength-to-weight ratio. Researchers have found that adding natural cellulosic to polymer composites may result in stronger/stiffer components than polymer composites using a similar weight of carbon nanocellulose [182,227–231]. This property could result in the manufacture of components with higher strength-to-weight ratios for such uses as aircraft components.

In the last decade, considerable efforts have been made to develop bionanomaterials. In addition bionanomaterials play an important role in many recent applications. Novel bionanomaterials are leading to new, multifunctionalities for packaging, fillers to improve mechanical and barrier properties of biocomposites, automotive components, medical devices, and other applications. The next section shows that bionanomaterials from agriculture waste have applications in several sectors.

3.5.1 As a reinforcing agent in composites materials

Over the past two decades, there has been a growing interest in the use of bionanomaterials as a reinforcement for polymer composite materials [25,232]. These materials include cellulose fibers and particles that possess desirable specific properties. Cellulose fibers for use as reinforcing elements in composite materials are an interesting alternative to synthetic fibers, such as glass fibers, because of their competitiveness in terms of weight and mechanical properties [117].

However, certain drawbacks such as incompatibility with the hydrophobic polymer matrix, a tendency to form aggregates during processing, and poor resistance to moisture absorption reduce the potential of these fibers for use as a reinforcement of thermoplastic matrices. Several strategies have been reported based on physical treatments in order to improve fiber/polymer compatibility and interfacial adhesion [233,234] and chemical modification of fibers to reduce their polarity and hydrophilicity [235,236].

There is a wide variety of different biomaterials that can be applied as reinforcers or fillers. A nanocomposite is a matrix to which nanoparticles have been added to improve a particular property of the material. Reinforcement is a simple method to reduce defects. The reinforcement of polymer composite materials depends on reinforcing agents such as fibers and nanoparticles [237]. Nanoscale additives, such as carbon black and silica nanoparticles, have been commonly used as polymer reinforcing agents [238]. The properties of nanocomposites have caused researchers and companies to consider using this material in several fields [25,127,159,239].

There has been wide application of bionanomaterial from agriculture waste in industrial applications. Bionanomaterials in reinforcement in polymers, such as in thermoset and thermoplastic polymers, have been used in many applications such as electronics, thermal insulators, aerospace, automotive, building materials, construction, and sports [168,240,241]. In terms of electronic devices, CNFs have low density, high specific modulus, high electrical conductivity, and large surface area, and are highly valuable in the field of super capacitors, nanooptoelectronic components, etc. [242].

Silica-reinforced polymer composite prepared via various processes is promising and has been widely used. Masoodi et al. [241] has studied how CNFs are used as reinforcing agents in the form of layered films in a bioderived resin. Silica nanoparticles have been developed for several applications such as electronic substrates, thin film substrates, electrical insulators, thermal insulators, and humidity sensors [243]. The quality of some of these products is highly dependent on the size and size distribution of the silica particles.

3.5.2 Packaging applications

In line with development of research in the nanotechnology field and concern for reducing environmental impact, cellulose-based materials have gained much more consideration for the development of alternative packaging materials. Studies by several researchers, such as Singh et al. [244], De Moura et al. [245], Youssef et al. [246], and Kalia et al. [247], have reported the use of natural cellulose-based nanocomposites for packaging applications.

Lignocellulose packaging has been used for a wide range of food categories, such as dry food products, frozen or liquid foods, beverages, and even fresh foods [246]; furthermore, it can be used in packaging of nonfood materials, such as medical and pharmaceutical packaging [248,249].

Packaging is being designed as a health system to provide positive impact on consumer health by integrating functional ingredients in the structure of the packaging [250]. Applications being developed for packaging material are durable, can be continuously recycled and reused, and do not contaminate [251]. Researchers have found that the adding nanocellulose to polymeric composites may result enhancement in gas barrier properties and heat stability on polymer composites nanocellulosic-based materials [125,127,247,252–254]. Efforts have been made to reduce poor mechanical and barrier properties of food packaging biobased materials with incorporation of reinforcing structures and matrices such as nanocellulose [255–257].

Studies have reported that the use of cellulosic materials may maximize the mechanical and barrier properties of product packaging materials [37]. Active food packaging systems include the concept of sustainable packaging, which must contain several properties such as protecting food products, enhancing food quality (stability), and releasing active compounds onto food surface [256,258]. According to Kumar and Münstedt [259], antimicrobial could used for a variety of applications, which include fabrication of food packaging materials. The use of antimicrobial agents is its have broad antimicrobial spectrum, good processability and high temperature stability.

3.5.3 Medical application

Recent advances in biocomposites have been supported by producing biofibers, microfibrillated, or nanosize fibers. Reinforcing cellulosic nanofibers offer potential advantages such as high performance of biofiber-based biocomposites [260]. John and Thomas [23] reported that CNFs combined with biodegradable polymers as biofiber-based nanocomposites proved to be very versatile in wide range of medical applications such as cardiac devices, scaffolds for tissue engineering, repair of articular cartilage, vascular grafts, urethral catheters, mammary prostheses, blood bags, penile prostheses, adhesion barriers, and artificial skin. The development of composites from these biofibers has increased commercial prospects for medical applications.

Many studies have been made on the development of biomaterials from agricultural waste to fabricate various versatile medical implants, such as pineapple leaf fibers and polyurethane [169]. In these studies, addition of 5 wt% of cellulose nanofibrils to polyurethane increased the strength and stiffness. Millon and Wan [261] report that the polyvinyl alcohol reinforced bacterial cellulose fibers will form biocompatible nanocomposites similar to that of cardiovascular tissues. Development of nanocellulose polyurethane vascular grafts [262], and micro- and nanofiller in polyurethane composites [263] have been reported. There have been various uses of bionanomaterials reported for the cosmetics and medical product industries, including pharmaceutical [187,264,265], medical [266], veterinary medicine [267], and dental [268] applications; furthermore, they can be used in drug delivery [175], medical implants [269–271], wound healing dressings [242,272,273], tissue engineering, and cellular culture [171,274,275].

3.5.4 Automotive industry application

Improvement in the performance of automobiles is of great importance for meeting both consumer needs and regulatory requirements in the automotive industry. Nanotechnology and nanomaterials have received great attention in the automobile industry to meet their performance needs [276]. Nanocellulose based materials are increasingly being used in thermoplastics matrix in the automotive industry over the last several years [277]. These plastic composites reinforced with cellulose fiber being used in automotive applications for front-ends, bumper beams, dashboards, and under body shields.

Cellulose-based materials such as natural fiber are emerging as a realistic alternative to glass-reinforced composites for application in automotive components. Natural fiber composites can deliver high-performance, nonbrittle fracture. Moreover, they are considerably cheaper to produce. The use of nanofiber-reinforced plastic composites in the automotive industry has grown significantly in recent years because of their low weight, design flexibility, corrosion resistance, and cost effectiveness [277]. The most common composites in automotive application are in the exterior body panel and are also an important requirement in the passenger compartment [139,278].

Natural fiber like hemp, jute, abaca, banana, kenaf, flax, and sisal have had success as reinforcing fibers in polymer composite from PP, polyethylene, nylon, or thermoplastic polymers for other automotive applications [278–282]. Furthermore, automakers now have been using natural fiber composite thermoset matrices for automotive components (seat backs, package trays, door panels, dashboards, headliners, and interior parts). Many auto companies have been utilizing cellulose fibers composites in their automobile products. Shinoj et al. [283] suggested that Mercedes Benz utilize coconut fiber-based rubber latex composites for seat parts. They also reported that flax/sisal fiber mat-reinforced epoxy have been utilized for door panels. Suddell and Evans [284] reported that Audi uses flax/sisal mat-reinforced polyurethane composite as door trim panels. Ford has been utilizing kenaf-reinforced PP composites for producing door panels [285].

3.5.5 Other applications

Several studies reported other applications of nanobiocomposites such as sporting goods, industrial rubber applications, aerospace components, etc. Most materials used for the sporting goods market are still glass and wooden materials. Efforts have been made to produce biocomposites in their manufacture, including development of biobased materials from agricultural waste. Some cellulose fiber materials like hemp, flax, coir, jute, etc., have been used effectively in sporting goods application. The carbon-reinforced composites have been used successfully to replace wood, glass, and metal in various applications for sporting goods such as fishing rods, ski equipment, tennis racquets, golf clubs, spars/shafts for kayak paddles, windsurfing masts, and bicycle handlebars [286,287].

Many researchers are concerned with the production of carbon black, activated carbon, and silica from biomass and agriculture-based residues, as they can be used for many applications. Nanoscale additives have been studied for the use in both natural and synthetic polymer reinforcing [288]. Silica-reinforced natural rubber prepared via a sol–gel process is promising and has been widely used [289]. Other researchers have focused on carbon black-reinforced elastomer composites to manufacture automotive components such as tires [290], as well as gloves [291] and nanobioceramic composites [292,293].

Polymer composites are widely used in the aerospace industry. Plants and crops from agricultural waste-reinforced composites have been used in polymer composites for making aerospace components. Wu and Radovic [294] reported that reinforced nanoparticles of carbon in epoxy matrix have found use in applications such as nose cones of the space shuttle, rocket nozzles, and aircraft brake discs. They also considered that additional these fillers have been improve the thermal, mechanical, chemical and physical properties.

3.6 Conclusion

Agriculture-based residues are of notable economic and cultural significance all over the world, especially in South Asia, Southeast Asia, and East Asia, being used for various applications such as building materials, as a decorative product, and as a versatile raw product. Agriculture-based residues also have significant potential in composite making due to their high strength, environmentally friendly nature, low cost, availability, and sustainability. Development of basic science and novel technologies for effective utilization of agriculture-based residues is the most significant aim of collaboration between research, development, and commercialization. Bionanocomposites are a fairly new idea in high-strength composite production, with extensive applications utilizing biomaterial in nanometer dimensions as reinforcement. Typical examples of CNF-reinforced nanocomposites can be seen in packaging, automotive components, medical, aircraft components, and other applications.

The properties of natural fiber as reinforcement in polymer composites influence their composites' performance, hence, it is essential to understand the properties of natural fiber, including their physical, mechanical, and morphological properties, as well as their chemical composition. These biomaterials offer many of the advantages associated with nanosized materials, such as larger interface, flexibility in surface, and reduction in flammability. Furthermore, bionanocomposite production has superior mechanical performance. Overall, it can be concluded that isolated CNF from pineapple leaf, bagasse, kenaf, oil palm, jute, bamboo, banana, etc can be a suitable alternative reinforcing agent or filler in functional composite for various engineering applications.

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