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Lignocellulose for Future Bioeconomy

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About the book

Description

Lignocellulose for Future Bioeconomy discusses the conversion and utilization of lignocellulosic biomass. This book focuses on the utilization of lignocelluloses for various products, including biopolymers, bionanomaterials and bioproducts. Recent findings in scientific investigation, engineering, product development, economic and lifecycle analysis are discussed, as are current synthesis

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Key Features

Presents information on lignocellulosic biomass management and its utilization for the production of bioproducts, biopolymers and bionanomaterials

Highlights the applications of advanced materials developed from lignocellulosic biomass and their contribution towards

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Details

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4

Lignocellulose-Based Nanoparticles and Nanocomposites: Preparation, Properties, and Applications

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INTRODUCTION

Natural fiber reinforced nanocomposites are of interest to both scientists and researchers because of their wide spread availability, sustainability, biodegradability, and renewability (Gallos, Paës, Allais, & Beaugrand, 2017; Malladi, Nagalakshmaiah, Robert, & Elkoun, 2018; Mohammadinejad, Karimi, Irvani, & Varma, 2016; Xiong, Grant, Ma, Zhang, & Tsukruk, 2018; Xue, Mou, & Xiao, 2017). Natural fibers can be derived from plant, animal, and mineral sources. Among these, plant-based cellulose fibers show promise as a reinforcement unit for the preparation of green nanocomposites (Fernandes, Pires, Mano, & Reis, 2013; Kalia, Thakur, Celli, Kiechel, & Schauer, 2013; Mohammadinejad et al., 2016; Namvar et al., 2014; Oksman et al., 2016; Sun et al., 2018). Cellulose is the most abundant, natural, and renewable biopolymer,

representing about 50% of natural biomass having an annual production estimated at more than 7.5×10^{10} tons (Habibi, Lucia, & Rojas, 2010; Trache, Hussin, Haafiz, & Thakur, 2017; Xue et al., 2017). This biomass serves as a virtually inexhaustible source of feedstock, capable of meeting the increasing demand for green and biocompatible products (Klemm et al., 2011; Tang, Sisler, Grishkewich, & Tam, 2017; Trache et al., 2016). Nanoscale cellulose materials, such as cellulose nanofibrils (CNFs) and cellulose nanocrystals (CNCs) can be isolated from various naturally occurring sources of cellulose (Chen & Lee, 2018; Kargazadeh, Ioelovich, Ahmad, Thomas, & Dufresne, 2017; Sun et al., 2018). These materials are carbon neutral, sustainable, renewable, recyclable, nontoxic, and environmentally friendly; capable of replacing any petroleum based products which are potentially toxic (Mariano, El Kissi, & Dufresne, 2014). The unique nanoscale structural features of nanocellulose, such as high aspect ratio, large specific surface area, high stiffness, high tensile strength, low density, and low thermal expansion coefficient, makes nanocellulose a fascinating bionanomaterial (Brinchi, Cotana, Fortunati, & Kenny, 2013; Golmohammadi, Morales-Narvaez, Naghdi, & Merkoci, 2017; Mautner, Mayer, Hervy, Lee, & Bismarck, 2018; Morits et al., 2018). This bionanomaterial can be applied in various fields, such as foods, cosmetics, pharmaceuticals, the pulp and paper industry, electronics, optical materials, barrier films, nanocomposites, membranes, supercapacitors, etc. (Azeredo, Rosa, & Mattoso, 2017; Brinchi et al., 2013; Golmohammadi et al., 2017; Mautner et al., 2018; Morits et al., 2018; Zu et al., 2016). Furthermore, owing to its biological utility, nanocellulose shows potential in biomedical fields, including medical packaging, hemodialysis membranes, vascular grafts, wound dressings, tissue engineering, and drug delivery (Abitbol et al., 2016; Ferraz et al., 2013; Lu, Li, Chen, & Yu, 2014; Plackett, Letchford, Jackson, & Burt, 2014; Serpa et al., 2016). This chapter reviews the extraction and processing methods of nanocellulose from various sources. Additionally, their properties, surface modifications, and applications in the preparation of bionanocomposites are also discussed.

Cellulose is a linear homopolysaccharide composed of β -1,4-linked anhydro-D-glucopyranose units having a flat ribbon-like conformation (Moon, Martini, Nairn, Simonsen, & Youngblood, 2011). The basic chemical structure of cellulose is shown in Fig. 4.1. It consists of cellobiose dimer as the repeating unit with a degree of polymerization (DP) of approximately 10,000 to 15,000 (Siqueira, Bras, & Dufresne, 2010). The DP varies according to the source of cellulose or even to the part of the plant from where it was extracted. An important characteristic of the structure of cellulose is the availability of three hydroxyl groups per anhydroglucose unit. These hydroxyl groups participate in extensive hydrogen bonding (Fig. 4.2), which plays a major role in directing

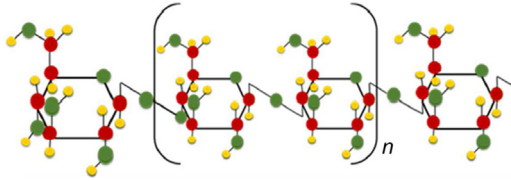


FIGURE 4.1 Schematic representation of the basic chemical structure of cellulose showing the cellobiose repeating unit (Malladi et al., 2018).

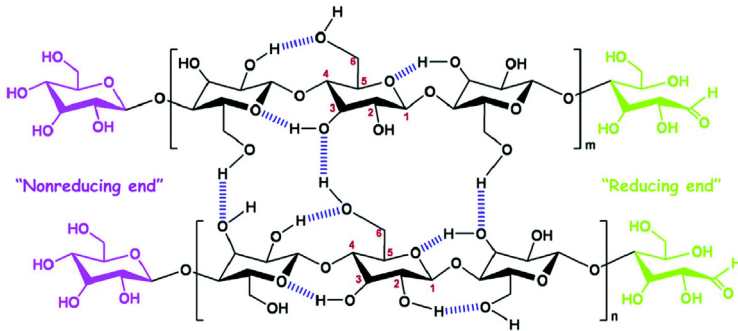


FIGURE 4.2 Schematic representation of inter- and intramolecular hydrogen bonds in cellulose (Lin & Dufresne, 2014).

crystalline packing as well as in governing the physical properties of cellulose (John & Thomas, 2008). Its inter- and intramolecular hydrogen bonding network makes cellulose a relatively stable polymer with high axial stiffness (Moon et al., 2011).

Cellulose is the main structural constituent of plant cell walls. These cell walls are composed of several layers, including the primary wall (P) and the several secondary wall layers (S1, S2, and S3) (Chinga-Carrasco, 2011; Lin & Dufresne, 2014). Each of these layers is characterized by a specific arrangement of cellulose fibrils in the pattern of helical thread and orientated at characteristic angles, depending on the cell wall layer and plant type (Xue et al., 2017). Most of the cellulose mass in the fiber is concentrated in the secondary walls with thicknesses ranging from 100 nm (cotton) to 300 nm (spruce wood). Meier (1962) stated that the cellulosic components of the cell wall consist of the cellulose molecule, the elementary fibril (nanofibril), the microfibril, and the lamellar membrane. Fig. 4.3 illustrates the diverse hierarchical structure of cellulose from cellulose fibers to cellulose molecules. The elementary fibrils are considered to be the universal structural units of natural cellulose having a diameter of 3–5 nm (Heyn, 1969). Cellulose microfibrils are agglomerates of elementary fibrils with a cross-section diameter of

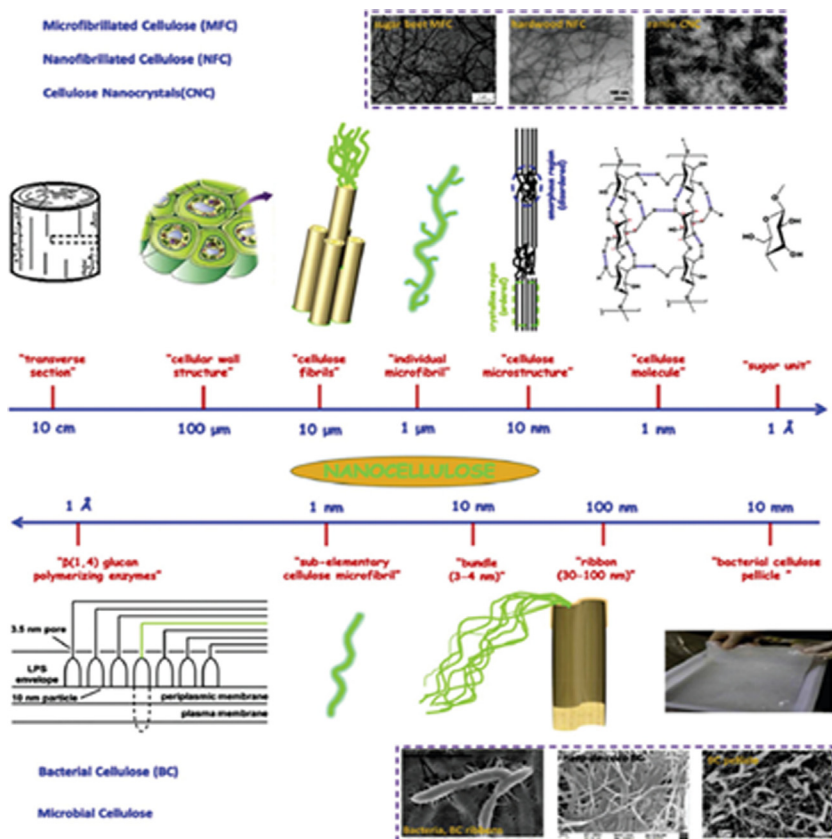


FIGURE 4.3 Schematic diagram of hierarchical structure of cellulose. Top image (from large unit to small unit): CNCs, micro/NFC; bottom image (from tiny unit to small unit): BC. Transmission electron micrographs of sugar beet MFC, hardwood MFC, ramie CNC, and scanning electron micrographs of BC ribbons, nata-de-coco BC, and BC pellicle (Lin & Dufresne, 2014).

5–50 nm and a length of several micrometers, depending on their source (Meier, 1962; Moon et al., 2011). Each microfibril can be considered as a bundle of cellulosic crystals linked along the microfibril axis by disordered amorphous domains (Azizi Samir, Alloin, & Dufresne, 2005). These cellulose fibrils are the dominant reinforcement unit for wood, plants, some marine creatures (tunicates), algae, and bacteria (Moon et al., 2011). Elementary fibrils are formed during biosynthesis processes, involving the parallel stacking of multiple cellulose chains through Van der Waals forces and intermolecular hydrogen bonds between hydroxyl groups and oxygen atoms of adjacent molecules (Moon et al., 2011; Ng et al., 2015). Each such fibril contains regions

where the cellulose chains are arranged in a perfectly ordered (crystalline) manner and regions that are arranged in a disordered (amorphous) manner (Ioelovich, 2008). The highly ordered crystalline regions are stabilized through the formation of a strong hydrogen bonded network between the hydroxyl groups of cellulose, whereas the disordered amorphous regions are not stabilized laterally through H-bonding (Trache et al., 2017). Depending upon the origin of the cellulosic materials, the lengths of the crystalline and amorphous domains vary in the range of 50–150 and 25–50 nm, respectively (Ioelovich, 2008). The highly crystalline components of the cellulose microfibrils can be easily extracted from the plant cell walls using an appropriate combination of chemical, mechanical, and enzymatic treatments. The resulting crystalline components of cellulose have a particle size in the nanometer range, known as “CNCs” (Trache et al., 2017; Xue et al., 2017).

NANOCELLULOSE

The term “nanocellulose” generally refers to products or extracts from native cellulose (found in plants, animals, and bacteria) having at least one dimension in the nanometer range (1–100 nm) (Khalil et al., 2014; Lin & Dufresne, 2014). This nanosized natural cellulose presents remarkable physical properties, large specific surface area, adaptable surface chemistry, and excellent biological properties (Mohammadinejad et al., 2016). It also possesses some interesting features, such as high tensile strength, high stiffness, high flexibility, and good thermal and viscoelastic properties as compared to other synthetic fibers (Thomas, Paul, Pothan, & Deepa, 2011). These excellent features make it ideal to be used as an independent functional material or reinforcement unit in composite materials (Xue et al., 2017). The incorporation of nanocellulose into various biopolymeric matrices offers great possibilities for the development of novel green nanocomposite materials. These bionanocomposite materials can be used for applications in drinking water filtration, in the catalytic degradation of organic pollutants, as absorbents to remove trickled oil from water, for censoring organic contaminants and water borne pathogens, and in energy conversion devices (Mohammadinejad et al., 2016).

Classification of Nanocellulose

Nanocellulose can be classified into three main subcategories based on its dimensions, morphology, functions, and extraction methods, which in turn depend mainly on the cellulosic source and on the

processing conditions (Khalil et al., 2014). The three novel forms of nanocellulose are nanofibrillated cellulose (NFC) or CNFs, nanocrystalline cellulose (NCC) or CNCs, and bacterial cellulose (BC) also referred to as microbial cellulose (Fig. 4.3). Nanotechnology provides the tool for the extraction of these novel forms of nanocellulose from their natural sources using a chemically or mechanically induced top-down approach (Mohammadinejad et al., 2016). The resulting nanocellulose particles show different morphologies, such as entangled network-like structures (CNFs) or rod-like structures (CNCs).

Cellulose Nanofibrils

CNFs, also known as NFC, microfibrillated cellulose (MFC), or cellulose nanofibers were first prepared by Turbak, Snyder, and Sandberg (1983) and Herrick, Casebier, Hamilton, and Sandberg (1983) from softwood through a high pressure homogenization (HPH) process. CNF consists of aggregates of long thread-like bundles of cellulose chain molecules with long, flexible, and entangled CNFs (Khalil et al., 2014). CNF have lengths in the micrometer and widths in the nanometer range. They possess high aspect ratios and exhibit gel-like properties in water with shear-thinning and thixotropic behavior (Nechyporchuk, Belgacem, & Bras, 2016). These fibrils constitute alternate crystalline and amorphous cellulosic zones and exhibit a network-like structure.

CNFs can be isolated from different sources, such as wood, agricultural crop residues, water plants, grasses, and their byproducts (Kalia, Boufi, Celli, & Kango, 2014). They are generally extracted from the mentioned sources via mechanical treatments that overcome interfibrillar hydrogen bonds and release CNF. HPH is the most widely used technique for both industrial and laboratory-scale production of CNF. In addition, other methods, such as microfluidization, microgrinding, cryo-crushing, steam explosion, and ultrasonication, are also applied according to the fiber sources, production conditions, and specific quality requirements (Khalil, Bhat, & Yusra, 2012; Missoum, Belgacem, & Bras, 2013).

Cellulose Nanocrystals

CNCs, also known as NCC or cellulose nanowhiskers, were first produced by Rånby using acid hydrolysis of cellulose fibers dispersed in water (Rånby, 1949). CNC consists of rod-like or needle-like particles with high purity, high crystallinity, and high specific surface area (Golmohammadi et al., 2017; Trache et al., 2017). The size of these particles can vary from 100 nm to several micrometers in length and from 4 to 70 nm in width (Jonoobi et al., 2015). CNC particles isolated from terrestrial woody biomass have relatively low aspect ratios (between 10 and 30), whereas those produced from bacteria and tunicates possess

higher aspect ratios (around 70) (Trache et al., 2017; Valentini, Cardinali, Fortunati, Torre, & Kenny, 2013). These particles are highly crystalline with crystallinity varying between 54% and 88% (Moon et al., 2011). CNC has very limited flexibility compared to CNF due to the lack of amorphous regions (Khalil et al., 2014). The morphology, geometrical dimensions, DP, surface charge, surface area, porosity, mechanical properties, crystallinity, and thermal stability of CNCs vary with the origin of the cellulose fibers, processing conditions, as well as the experimental technique employed to obtain them (Habibi et al., 2010; Trache et al., 2017).

CNCs can be produced from a wide variety of cellulosic materials, including wood, plants (agricultural biomass), marine animals (tunicates), bacteria, algae, and fungi (García, Gandini, Labidi, Belgacem, & Bras, 2016; Hua, Strømme, Mihranyan, & Ferraz, 2015; Sacui et al., 2014; Yarbrough et al., 2017) via an acid hydrolysis process (Brinchi et al., 2013). During acid hydrolysis, the amorphous regions within the cellulosic fibers are removed, which are preferentially removed while the crystalline regions with a higher resistance to acid remain intact (Peng, Dhar, Liu, & Tam, 2011; Trache et al., 2017). Sulfuric and hydrochloric acids are extensively used for this purpose (Beck-Candanedo, Roman, & Gray, 2005; Chen et al., 2015; Tang, Yang, Zhang, & Zhang, 2014). Alternatively, hydrobromic (Sadeghifar, Filpponen, Clarke, Brougham, & Argyropoulos, 2011) and phosphoric acids (Camarero Espinosa, Kuhnt, Foster, & Weder, 2013) have also been used.

Bacterial Nanocellulose

Bacterial nanocellulose (BNC), also known as BC or microbial cellulose, is a highly pure form of cellulose generated by certain bacteria (e.g., *Gluconacetobacter xylinus*) in aqueous culture media containing a sugar source (Nechyporchuk et al., 2016). BNC is generally produced as hydrogels or nanofilms via a “bottom-up” method (Golmohammadi et al., 2017). It is composed of a continuous three dimensional network of nanofibrils assembled in the form of twisted ribbons having a width of 20–50 nm and a length of several micrometers (Golmohammadi et al., 2017; Nechyporchuk et al., 2016). BNC has the same chemical composition as plant-derived nanocellulose, while dimensionally, BNC is normally larger compared to plant-derived nanocellulose. BNC is free of other polymers, such as lignin, hemicelluloses, or pectin. Therefore, BNC is highly crystalline (up to 84% to 89%), generating larger bacterial nanocrystals (Barud et al., 2011; Zhang et al., 2010). BNC exhibits a high Young’s modulus (up to 150 GPa), that is, good mechanical stability and a high DP of between 3000 and 9000 (Nechyporchuk et al., 2016). Furthermore, it has high chemical purity and high water holding

capacity when compared to CNF and CNC extracted from plants (Charreau, Foresti, & Vázquez, 2013; Trache et al., 2017).

BC is secreted extracellularly by certain types of bacteria, such as *Acetobacter xylinus*, *Agrobacterium*, *Pseudomonas*, *Rhizobium*, and *Sarcina* (Campano, Balea, Blanco, & Negro, 2016). BNC is commercially produced with Gram-negative acetic acid bacteria *Acetobacter xylinum* (genus *Gluconacetobacter*) (Trache et al., 2017). BNC has been widely used in various biomedical materials (Wang, Zhu, & Du, 2011; Yasuda et al., 2005) and other engineering materials as a reinforcing agent (Hu, Chen, Yang, Li, & Wang, 2014; Long et al., 2014).

EXTRACTION METHODS OF VARIOUS TYPES OF NANOCELLULOSE

Nanocellulose can be extracted from a wide variety of plant materials, including wood and agricultural biomass. Cellulose is present in wood or plants in combination with other chemical constituents, such as hemicellulose and lignin as shown in Fig. 4.4. Therefore, wood or plants can be considered as cellular hierarchical biocomposites created by nature in which hemicellulose/lignin, waxes/extractives, and trace elements serve as matrix materials, whereas semicrystalline cellulose microfibrils act as reinforcing material (Dufresne, 2013; Trache et al., 2017).

Pure cellulose is obtained after the effective removal of hemicellulose, lignin, and other impurities from the plant fibers. The extraction of nanocellulose from wood/plants requires a multistage process involving a combination of chemical, mechanical, or enzymatic treatments (Malladi et al., 2018; Rashid, Suryani, Muhdjulka, & Yehye, 2018). Fig. 4.5 shows an overview of the different techniques used in the production of nanocellulose. The main purification steps involved in the extraction of nanocellulose are an alkali treatment—using potassium hydroxide (KOH) or sodium hydroxide (NaOH)—and bleaching

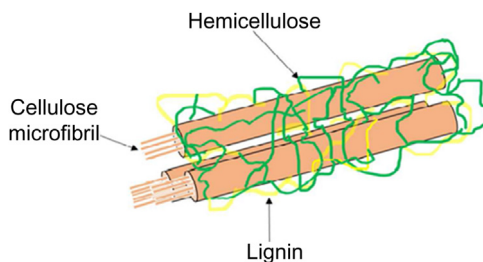


FIGURE 4.4 Cellulose microfibril bundles embedded in a matrix of hemicellulose and lignin (Malladi et al., 2018).

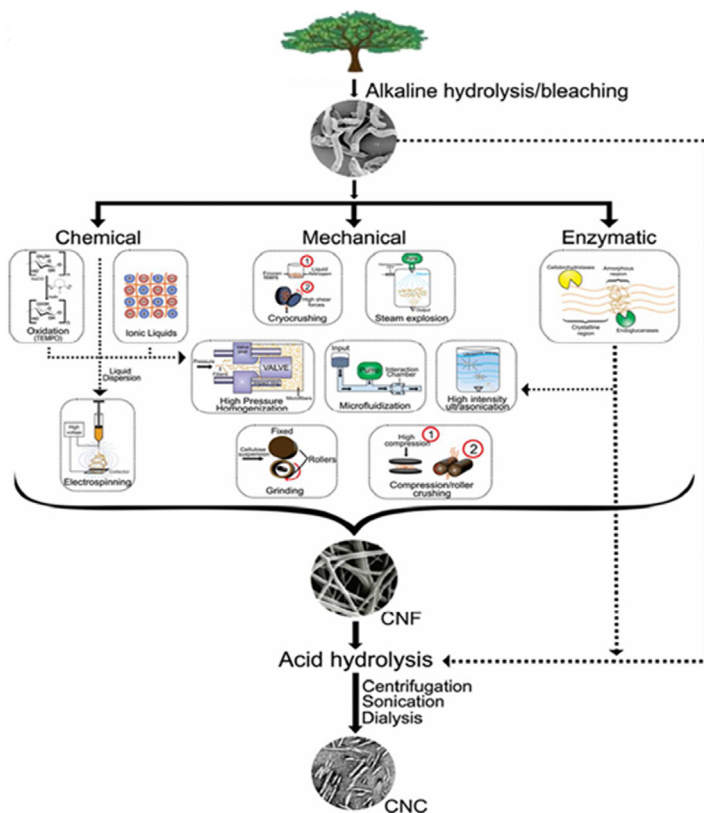


FIGURE 4.5 Different methods used for the production of nanocellulose in the form of nanofibrils and nanocrystals (Rojas, Bedoya, & Ciro, 2015).

(delignification using sodium chlorite (NaClO_2) or hydrogen peroxide [H_2O_2]) processes. CNCs can be obtained through the direct acid hydrolysis of purified cellulose, whereas CNFs are produced by several mechanical treatments, like homogenization, microfluidization, steam explosion, grinding, cryocrushing, ultrasonication, etc. (Khalil et al., 2012, 2014; Nechyporchuk et al., 2016). Details of these methods are summarized here.

Extraction of Cellulose Nanofibrils

High Pressure Homogenization

HPH is a widely used method for large-scale production of CNF, as well as for laboratory-scale preparation of CNFs. In this method, the cellulose suspension is passed into a vessel through a small nozzle under high pressure (50–2000 MPa) (Fig. 4.5) (Khalil et al., 2014;

Nechyporchuk et al., 2016). The resulting shear and impact forces cause the fibrillation of cellulose to nanoscale dimension. The production of CNF gels using homogenization (Gaulin homogenizer) was first reported by Turbak et al. (1983). Dufresne, Cavaillé, and Vignon (1997) also produced cellulose microfibrils from sugar beet pulp using homogenization. The extent of fibrillation depends on the applied pressure and the number of homogenization cycles. Various cellulosic materials have been subjected to HPH, such as wood pulp (Siddiqui, Mills, Gardner, & Bousfield, 2011), sugar beet pulp (Dufresne et al., 1997), cotton (Wang et al., 2013), soybean hulls (Wang, Sain, & Oksman, 2007), sunflower (Chaker, Alila, Mutjé, Vilar, & Boufi, 2013), mangosteen rind (Winuprasith & Suphantharika, 2013), tomato residues (Hua et al., 2017), banana peel (Tibolla, Pelissari, Martins, Vicente, & Menegalli, 2018), sugarcane bagasse (Saelee, Yingkamhaeng, Nimchua, & Sukyai, 2016), and bacteria (Kawee, Lam, & Sukyai, 2018). The main drawbacks of this method are the insufficient disintegration of the pulp fibers and the clogging of the homogenizer when the pulp is pumped through the small nozzle (Kargarzadeh et al., 2017). Other limitations include its high energy consumption (70 MWh ton^{-1}) and the excessive mechanical damage to the crystalline structure of CNF caused during the homogenization process (Kargarzadeh et al., 2017; Nechyporchuk et al., 2016). High energy consumption during the homogenization process can be reduced through pretreatment of the pulp using oxidation, acid hydrolysis, enzymatic hydrolysis, etc. (Nechyporchuk et al., 2016).

Microfluidization

Microfluidization is another method used to produce CNF that was introduced by Zimmermann, Pöhler, and Geiger (2004). Unlike the homogenizer, which operates at constant pressure, the microfluidizer operates at a constant shear rate. In this method, the cellulose suspension is passed through a Z- or Y-shaped channel of 200–400 μm diameter at high pressure ($\sim 2000 \text{ bar}$) (Fig. 4.5) (Kargarzadeh et al., 2017; Khalil et al., 2014). The resulting shear forces against the colliding suspensions and the channel walls leads to the defibrillation of the fibers to nanoscale dimension. Ferrer, Filpponen, Rodríguez, Laine, and Rojas (2012) produced CNF from empty palm fruit bunch fibers using a microfluidizer. In addition to overcoming the clogging in the homogenization process this method also helps to obtain fibrils of uniform dimensions. Unfortunately, this process requires 10 or even as many as 30 passes of the raw fibers through the microfluidizer with excessively high energy consumption ($\sim 30,000 \text{ kWh ton}^{-1}$) (Plackett & Iotti, 2013). Considerable efforts have been made to reduce the energy consumption of this method by applying cellulose pretreatments, which significantly reduced the energy required for CNF production.

Cryocrushing

Cryocrushing is another mechanical fibrillation method for frozen cellulose. In this process, water-swollen cellulose fibers are frozen in liquid nitrogen and then crushed by high shearing forces (Chakraborty, Sain, & Kortschot, 2006; Plackett & Iotti, 2013). The application of high shearing forces to frozen cellulosic fibers causes the rupturing of the cell walls due to the pressure exerted by the ice crystals. This leads to the liberation of nanofibrils with relatively large diameters, ranging between 0.1 and 1 μm (Chakraborty et al., 2006). Using this method, Alemdar and Sain (2008) extracted CNF from wheat straw and soy hulls. The obtained fibrils had diameters of 30–40 nm and lengths of several micrometers (Alemdar & Sain, 2008). Dufresne et al. (1997) also used cryocrushing to obtain CNF from sugar beet pulp. CNFs with diameters of 5–80 nm were isolated from other sources, such as flax, hemp, and rutabaga fibers via the cryocrushing method (Bhatnagar & Sain, 2005).

Grinding

Ultrafine friction grinding is another technique used for the production of CNF. Masuko Sangyo Co., Ltd., Japan was the first company to build grinding equipment (Supermasscolloider grinders) mainly for the production of CNF (Plackett & Iotti, 2013). In this process, the cellulose pulp is passed between static and rotating grinding stones (disks) revolving at ~ 1500 rpm (Kargarzadeh et al., 2017). The shearing forces generated between the disks degrade the cell wall structure and thus the nanofibrils are individualized. Wang et al. (2012) produced CNF for the first time from bleached eucalyptus pulp using the Supermasscolloider grinding equipment. The extent of fibrillation depends on the distance between the disks, the morphology of the disk channels, and the number of passes through the grinder (Kargarzadeh et al., 2017). Iwamoto, Nakagaito, and Yano (2007) extracted CNF using the pulp from radiata pine (*Pinus radiata*) fibers and noted changes in morphology as a function of the number of passes. Furthermore, CNF have also been extracted from carrot residue (Siqueira, Oksman, Tadokoro, & Mathew, 2016), bagasse pulp (Hassan, Mathew, Hassan, El-Wakil, & Oksman, 2012), and kenaf (Karimi, Tahir, Karimi, Dufresne, & Abdulkhani, 2014) using a grinding technique.

High Intensity Ultrasonication

High intensity ultrasonication (HIUS) is a widely used mechanical treatment in which oscillating power is used to produce cellulose fibrils by hydrodynamic forces of ultrasound (Kargarzadeh et al., 2017). This treatment produces high intensive waves due to efficient cavitation that includes the formation, expansion, and implosion of microscopic gas

bubbles when water molecules absorb ultrasonic energy (Khalil et al., 2014). The action of the hydrodynamic forces of the ultrasound on the cellulose pulp leads to the liberation of nanosized fibrils. Many researchers have reported the application of the HIUS process for the extraction of nanofibrils from various cellulosic sources, including wood (Chen et al., 2011), corn husk (Xiao, Gao, Gao, & Li, 2016), banana peel (Khawas & Deka, 2016), rice waste (Rezanezhad, Nazanezhad, & Asadpur, 2013), citrus processing waste from oranges (Tsukamoto, Durán, & Tasic, 2013), etc.

Steam Explosion

The steam explosion process is another promising technique for the extraction of CNF from various cellulosic sources (Abraham et al., 2011; Cherian et al., 2010; Chirayil et al., 2014; Deepa et al., 2011; Kaushik & Singh, 2011; Thomas et al., 2015). Lignocellulosic materials can be fractionated into biopolymer constituents using steam explosion technology. It is a thermomechanical process in which the pulp is exposed to pressurized steam for short periods of time, followed by a rapid release of pressure, which causes the rupture of the fiber cell wall, leading to the formation of CNFs (Abraham et al., 2011; Sun & Cheng, 2002). The steam explosion process is mainly used for the extraction of cellulose fibers from lignocellulosic materials (Overend & Chornet, 1987). The extraction process mainly involves three processes, namely alkaline steam, bleaching, and acidic steam treatments (Abraham et al., 2011; Cherian et al., 2008; Deepa et al., 2011). The immersion of lignocellulosic fibers in diluted alkaline medium facilitates the adhesive nature of the fiber surface by removing natural and artificial impurities and causing the separation of the structural linkages between lignin and carbohydrate and the disruption of the lignin structure (Wang et al., 2007). The aim behind the bleaching process is the removal of lignin left after the alkaline steam treatment. The bleaching treatment using sodium chlorite solution is popularly used in laboratories to remove the lignin from vegetable fibers (Johar, Ahmad, & Dufresne, 2012; Maheswari, Reddy, Muzenda, Guduri, & Rajulu, 2012; Nayak & Mohanty, 2018; Sheltami, Abdullah, Ahmad, Dufresne, & Kargarzadeh, 2012; Trifol et al., 2017; Zuluaga et al., 2009). The mechanism of bleaching involves the oxidation of lignin, which leads to lignin dissolution and its degradation. Further treatment (acidic steam treatment), which helps in the production of CNFs, is the main phase of the extraction process. Acid treatment hydrolyzes the traces of hemicellulose and lignin left after the bleaching process by breaking down the polysaccharides to simple sugars, thus, facilitating the release of cellulose nanofibers (Cherian et al., 2010). The combination of acid and steam treatments, with the maximum explosion of pressurized steam into the interfibrillar

regions, effectively reduces the long microfibril chains to nanoscale dimensions (Abraham et al., 2011). Many researchers have used this method to extract CNF from various lignocellulosic sources, such as banana stem (Abraham et al., 2011; Cherian et al., 2008; Deepa et al., 2011), jute (Thomas et al., 2015), pineapple leaf (Cherian et al., 2010), coir (Abraham et al., 2013), *Helicteres isora* (Chirayil et al., 2014), wheat straw (Kaushik & Singh, 2011), etc. The advantages of steam explosion include a significantly lower environmental impact, low energy consumption, relatively low capital investment, and less hazardous process chemicals. (Abraham et al., 2011; Deepa et al., 2011).

Extraction of Cellulose Nanocrystals by Acid Hydrolysis

The most widely reported method for the extraction of CNC is through the acid hydrolysis process (Brinchi et al., 2013). This process can disrupt the hydrogen bonds and cleave the amorphous domains of the fiber to yield well-defined crystalline particles (Fig. 4.6) (Grishkewich, Mohammed, Tang, & Tam, 2017). Classical extraction of CNCs is usually

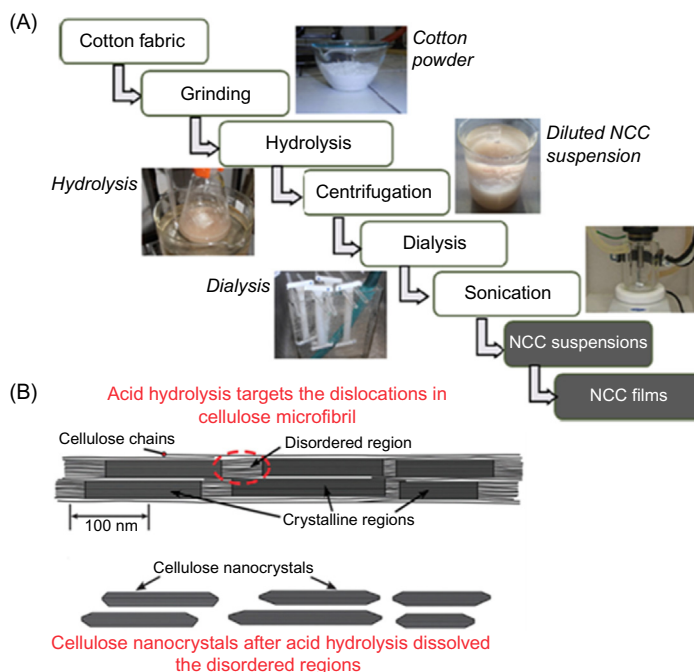


FIGURE 4.6 (A) Schematic representation of various steps used to produce CNC from bleached cotton fabric; and (B) simplified structure of a cellulose microfibril with alternate crystalline and amorphous regions. Amorphous domains can be selectively targeted with controlled acid hydrolysis, leading to the isolation of CNCs (Trache et al., 2017).

carried out using sulfuric acid (60–65 wt.%) hydrolysis at temperatures below 50°C followed by the purification of biomass (Malladi et al., 2018). At the end of the hydrolysis process, the mixture undergoes a series of separation and washing/rinsing steps that are followed by repeated cycles of sonication and dialysis against deionized water to eliminate residual acid and neutralized salts, thus, yielding pure CNC (Fig. 4.6) (Trache et al., 2017). CNCs have also been produced using other acids, such as hydrochloric, sulfuric, and phosphoric acids (Camarero Espinosa et al., 2013; Chen et al., 2015; Sadeghifar et al., 2011; Tang et al., 2014). Each acid treatment introduces specific functional groups onto the nanoparticle surface that impacts on their colloidal stability (Grishkewich et al., 2017).

The temperature and duration of the hydrolysis reaction, and the nature and concentration of the acid used as well as the acid to cellulose fiber ratio are important parameters that influence the properties of the resulting CNC (Rashid et al., 2018; Trache et al., 2017). Many researchers have analyzed the effect of processing conditions on the physicochemical, thermal, and mechanical properties of CNC. For instance, CNC extracted using hydrochloric acid displays low-density surface charges with poor colloidal stability in aqueous suspensions, whereas those produced with sulfuric acid possess negatively charged sulfate ester groups on their surface that induce electrostatic repulsion to yield a highly stable colloidal dispersion (Grishkewich et al., 2017; Ng et al., 2015). Unfortunately, one limitation of this method is the poor thermal stability associated with the sulfate groups in CNC. The sulfate groups can catalyze and initiate the degradation of cellulose, particularly at high temperatures, which restricts the processing of CNC based nanocomposites at high temperatures (Trache et al., 2017). The dimensions of CNC are mainly dependent on the source from which they are extracted (Grishkewich et al., 2017). For instance, CNC isolated from hard wood possess a length and width of 100–300 and 3–5 nm, respectively, whereas CNC produced from tunicate possess a length and width of 1000–1500 and 15–30 nm, respectively (Elazzouzi-Hafraoui et al., 2007; Peng et al., 2011).

PROPERTIES AND APPLICATIONS OF NANOCELLULOSE

Nanocellulose, isolated from the most abundant biomass material cellulose, offers outstanding properties, including biodegradability, biocompatibility, renewability, recyclability, abundant availability, environmental sustainability, low toxicity, special surface chemistry, versatile fiber morphology (nanofibrils, nanocrystals, nanofilms, nanopapers,

aerogels, hydrogels, spherical particles, etc.), good thermal characteristics (high thermal stability, low thermal expansion coefficient), excellent mechanical performance—high tensile strength 750; 0 MPa), high stiffness (Young's modulus of up to 140 GPa)—rheological features (high storage and loss modulus with pseudoplastic and shear-thinning behavior), good optical properties (high optical transparency), high gas permeability, and other physicochemical characteristics (high surface area ($\sim 250 \text{ m}^2 \text{ g}^{-1}$), high crystallinity, high aspect ratio, high flexibility, high porosity, low density, hydrophilicity), etc. (Eichhorn, 2011; Grishkewich et al., 2017; Lin, Bruzzese, & Dufresne, 2012; Lin, Huang, & Dufresne, 2012; Mondal, 2017). As a natural nanoscale material, nanocellulose has received tremendous attention in numerous applications in fields such as the paper and pulp industry, food, textiles and clothing, automotive, construction to aerospace, packaging, paints, oil and gas, cosmetic and pharmaceutical products, polymer nanocomposites, biomaterial engineering, biomedicine, separator in energy devices (batteries and solar cells), membranes, nanofilters, optical and electronic devices, polymer nanocomposites, adsorbent for waste water treatment, security papers, solar panels, bioimaging, biosensors, etc. (Golmohammadi et al., 2017; Grishkewich et al., 2017; Julkapli & Bagheri, 2017; Malladi et al., 2018; Xue et al., 2017). Fig. 4.7 shows the applications of nanocellulose in various sectors. Their excellent mechanical properties (high stiffness and

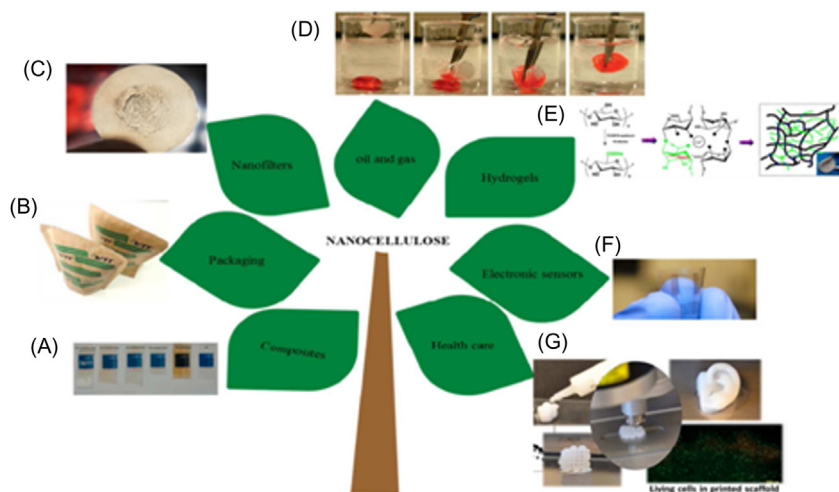


FIGURE 4.7 Applications of nanocellulose in various fields: (A) extruded polypropylene/CNC and modified CNC nanocomposite films; (B) biobased pouches from VTT technical research; (C) nanocellulose filter; (D) CNF aerogels from rice straw absorbing dyed chloroform from water; (E) alginate-oxidized nanocellulose sponge; (F) fabricated transparent and flexible nanopaper transistor; and (G) 3D printed small grids and human ear from CNF and alginate (Malladi et al., 2018).

high tensile strength), high aspect ratio, facile chemical modification, and ultra-low density of nanocellulose favor them as a potential reinforcing agent in the fabrication of high-performance and lightweight materials (Grishkewich et al., 2017; Julkapli & Bagheri, 2017; Malladi et al., 2018; Xue et al., 2017).

Nanocellulose is also an attractive candidate for use in various water treatment systems, such as adsorption, absorption, flocculation, membrane filtration, catalytic degradation, and disinfection because of its high specific surface area, high specific strength, hydrophilicity, biodegradability, and surface functionalization capabilities. Nanocellulose, with its enormous surface area, high aspect ratio, flexibility, optical properties, and ability to strengthen matrices through surface functionalization have found applications in supercapacitors, conductive films, sensors, substrates in electrical devices, and separators in energy storage devices (Grishkewich et al., 2017; Julkapli & Bagheri, 2017). The large surface area and high water holding capacity of nanocellulose facilitate its use as a rheology modifier mainly in paints and personal care products (Malladi et al., 2018). Its exceptional biocompatibility and biodegradability make nanocellulose a promising material for several applications in biomedical fields, such as antibacterial and antiviral agents, tissue engineering and cell culture scaffolds, protein scaffolds/biocatalysts, DNA extraction membranes, hemodialysis materials, biomarkers or sensors, drug delivery, wound dressings and skin tissue repair materials, artificial blood vessels, gene vectors, bone regeneration and cartilage replacement, medical packaging, etc. (Golmohammadi et al., 2017; Grishkewich et al., 2017; Lin, Bruzzese, et al., 2012; Lin, Huang, et al., 2012; Malladi et al., 2018; Rashid et al., 2018; Xue et al., 2017).

BIONANOCOMPOSITES

Bionanocomposites are generally defined as a combination of two or more materials or phases derived from natural or synthetic biopolymers in which one of the phases has at least one of its dimensions in nanoscale (1–100 nm) (Fernandes et al., 2013). The matrix phases of these composites may include biodegradable polymers derived from renewable sources and reinforcing phases either of nanoparticles based on natural polymers (cellulose, starch, chitin, etc.) or synthetic inorganic materials (carbon nanotubes, graphene, nanoclays, etc.) (Sorrentino, Gorrasi, & Vittoria, 2007). Biopolymer matrices provide structural strength and new functionalities when combined with complementary nanoscale components with proper dimensions and functionalities (Xiong et al., 2018). Fig. 4.8 shows a list of the most relevant biopolymers used in the preparation of bionanocomposites. Based on

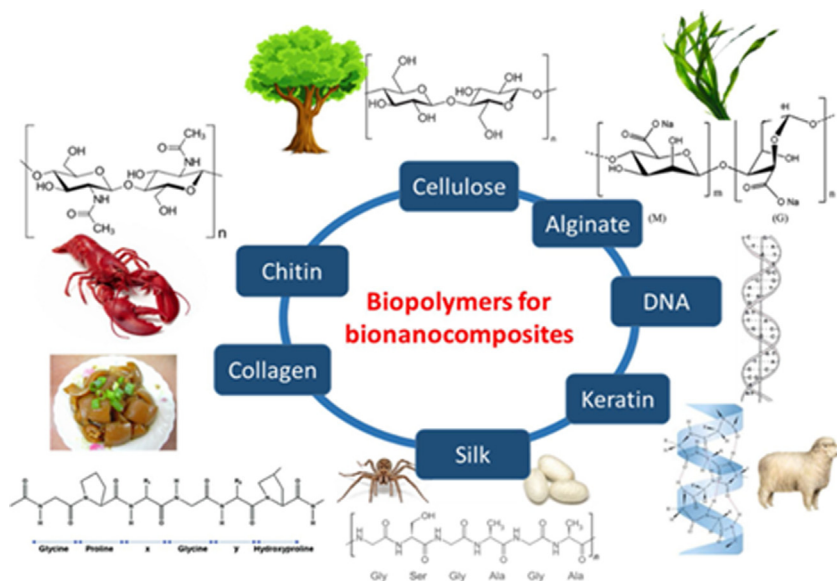


FIGURE 4.8 The sources and molecular structures of naturally derived biopolymer components for bionanocomposites (Xiong et al., 2018).

monomeric unit composition, biopolymers can be divided into three classes, namely polysaccharides, proteins/polypeptides, and polynucleotides (Xiong et al., 2018). Among these polymers, cellulose, chitin/chitosan, and alginate are common polysaccharides bonded by glycosidic linkages; whereas silk, collagen, gelatin, and keratin are typical proteins consisting of long chains of amino acid residues (Xiong et al., 2018). These biopolymers are renewable and can be derived from natural sources, such as plants, exoskeletons of arthropods, skin, silkworm cocoons, spider webbing, and hair (Khalil et al., 2012; Koepfel & Holland, 2017; Kuttappan, Mathew, & Nair, 2016; Rouse & Van Dyke, 2010). Lastly, DNA is a polynucleotide which carries the genetic instructions used in the growth, development, function, and reproduction of all known living organisms and many viruses (Xiong et al., 2018).

All these biopolymers are attractive candidates for the fabrication of high-performance and functional bionanocomposites due to their biocompatibility, biodegradability, renewability, sustainability, flexibility, and the availability of multiple reactive sites for introducing novel functionalities. In this study, we discuss the current progress on the development of bionanocomposites based on two biopolymers, viz. nanocellulose and sodium alginate. The processing strategies, properties and emerging applications of these bionanocomposites are analyzed and discussed briefly.

Processing of Bionanocomposites

Bionanocomposites are generally prepared using solution processing techniques since their thermal degradation temperatures are close to or lower than their glass-transition temperature (T_g) (Williams & Hillmyer, 2008). The most popular solution casting techniques include vacuum assisted filtration and cast-drying, freeze-drying (lyophilization), wet spinning, electro-spinning, cryogelation, etc. (Cheng et al., 2017; Kumar, Rao, & Han, 2017; Lin, Bruzzese et al., 2012; Lin, Huang et al., 2012; Huan, Liu, Cheng, Han, & Bai, 2018). This section mainly focuses on the cast-drying and freeze-drying techniques.

Cast-drying

In the cast-drying process, the colloidal mixture is placed into clean containers, such as petri dishes, or deposited onto clean substrates by various coating techniques, such as bar coating and spraying, to obtain solid films after the complete removal of the solvent (Xiong et al., 2018). For instance, Cheng et al. (2017) has successfully prepared films and sponges based on (TEMPO)-mediated oxidized CNCs and alginate using cast-drying and freeze-drying techniques (Fig. 4.9).

Freeze-Drying

Freeze-drying (lyophilization), is another processing technique that works by freezing the material and then reducing the surrounding pressure to allow the frozen water in the material to sublime directly from the solid phase to the gas phase (Rey, 2016). This process preserves the morphology of nanocomposites in the wet state to prepare highly porous, ultra-lightweight bionanocomposite sponges (Fig. 4.9). Freeze-drying is widely adopted to develop novel porous

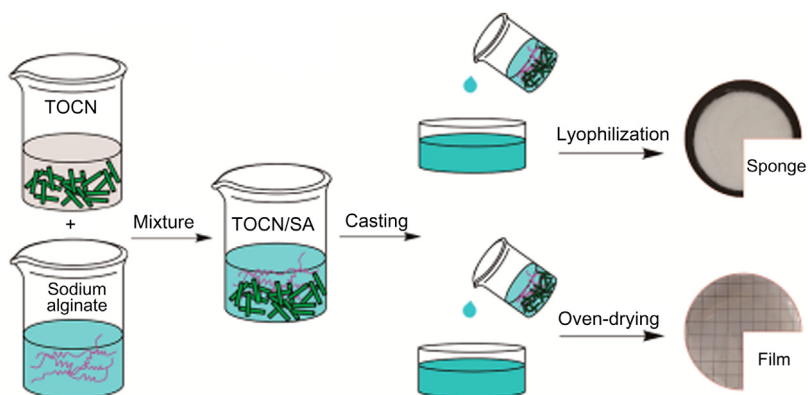


FIGURE 4.9 Scheme of cast-drying technique for preparing oxidized CNC/alginate composite films and sponges (Cheng et al., 2017).

bionanomaterials based on various natural sources, including cellulose (Cheng et al., 2017), starch (Abhari, Madadlou, & Dini, 2017), chitin (Tsutsumi, Koga, Qi, Saito, & Isogai, 2014), chitosan (Yu et al., 2013), alginate (Lin, Bruzzese, et al., 2012; Lin, Huang, et al., 2012), and gelatin (Zhou, Gong, & Gao, 2005).

Nanocellulose/Alginate Bionanocomposites

Alginate is a natural polysaccharide extracted mainly from brown seaweed by alkali treatment. It is a linear copolymer composed of (1–4)-linked β -D-mannuronate (M) and α -L-guluronate (G) residues, covalently linked together in different sequences which determine its physicochemical properties (Kumar et al., 2017; Xiong et al., 2018). Alginate can be crosslinked with divalent cations, such as Ca^{2+} , for the preparation of hydrogels because of the presence of abundant carboxyl groups in the molecular chains (Lee & Mooney, 2012). The gelling property of alginate in aqueous media is due to ionic interaction between the carboxylic group and divalent cations (Lin, Bruzzese, et al., 2012; Lin, Huang, et al., 2012; Šimkovic, 2013). It has been widely used in biomedical applications, especially in tissue engineering and drug delivery applications because of its good biocompatibility, inherent hydrophilicity, low cost, low toxicity, ease of processing, and gelling properties under mild conditions (Cheng et al., 2017; Kumar et al., 2017; Lin, Bruzzese, et al., 2012; Lin, Huang, et al., 2012). Furthermore, nanocellulose, the most abundant natural biopolymer, is also gaining considerable attention in biomedical applications. The synergistic combination of these two biological components can open a promising route toward the development of high-performance bionanocomposites with unique performance and novel functionalities (Xiong et al., 2018). Several studies have been reported on the preparation and properties of bionanocomposite materials derived from these natural biopolymers (Abdollahi, Alboofetileh, Behrooz, Rezaei, & Miraki, 2013; Abdollahi, Alboofetileh, Rezaei, & Behrooz, 2013; Huq et al., 2012; Lin, Bruzzese, et al., 2012; Lin, Huang, et al., 2012; Sirviö, Kolehmainen, Liimatainen, Niinimäki, & Hormi, 2014; Taokaew, Seetabhwang, Siripong, & Phisalaphong, 2013). Here, a few reports have been highlighted that deal with the fabrication, properties, and applications of bionanocomposites based on nanocellulose and alginate.

Kumar et al. (2017) reported the fabrication of bionanocomposite scaffolds based on sodium alginate (SA) and CNCs. Their studies showed that the incorporation of CNC enhances the rheological, mechanical, thermal, and cytocompatibility properties of the nanocomposite scaffolds. These scaffolds may have potential applications in bone tissue engineering fields. In another study, Cheng et al. (2017) investigated the hemostasis applications of bionanocomposite films and

sponges made from oxidized CNCs and alginate. The test results indicated that the prepared bionanocomposites possessed ultrahigh porosity (sponge), surface homogeneity (film), water absorption ability, and chemical stability as well as excellent hemostatic efficiency compared to neat alginate films/sponges.

The mechanical properties of composite hydrogels based on alginate and CNFs have been investigated by [Aarstad et al. \(2017\)](#). Assessments of the composite gels revealed that attractive properties, like high rupture strength, high compressibility, high gel rigidity at small deformations (Young's modulus), and low syneresis, were obtained compared to pure gels. These properties varied with relative amounts of CNF and alginate, alginate type, and CNF quality.

Cartilage tissue engineering and 3D bioprinting applications of nanocellulose/alginate bionanocomposites have also been reported. For example, [Leppiniemi et al. \(2017\)](#) reported the preparation of bioactivated 3D-printable nanocellulose–alginate hydrogels. The developed 3D-printable hydrogels indicated good tissue compatibility and absorbed water in moist conditions, which make them suitable for wound dressing applications. These hydrogels can also offer a platform for the development of biomedical devices, wearable sensors, and drug-releasing materials.

3D printing technology has been applied for the fabrication of fully biobased porous scaffolds of a double crosslinked interpenetrating polymer network from a hydrogel ink of SA and gelatin reinforced with CNCs ([Sultan & Mathew, 2018](#)). CNC provided favorable rheological properties required for 3D printing. Their study revealed the potential of 3D printing in the development of biobased scaffolds with controlled pore sizes, gradient pore structures, and alignment of nanocrystals for optimal tissue regeneration.

[Nguyen et al. \(2017\)](#) investigated the 3D bioprinting property of CNFs/SA for applications in cartilage tissue engineering. Similar results were achieved using alginate sulfate/nanocellulose ink ([Müller, Öztürk, Arlov, Gatenholm, & Zenobi-Wong, 2017](#)). [Poonguzhali, Basha, and Kumari \(2018\)](#) conducted in vitro drug release studies on alginate/nanocellulose bionanocomposites prepared via a solution casting method. The in vitro studies showed that the formulated ampicillin loaded alginate/nanocellulose films gave acceptable physicochemical properties compared to alginate–ampicillin films and were able to deliver drugs in a prolonged release pattern.

In addition to the aforementioned applications, bionanocomposites have also been used for a variety of applications in different areas, such as food, cosmetics and pharmaceuticals, textiles, pulp and paper

industries, paints and coatings, insulation and packaging, bioactive membranes and filters, bioplastics and biosensors, optical, electronic, and mechanical engineering, waste water treatments, and other applications due to their excellent physicochemical, mechanical, electrical, and optical properties along with their potential sustainability, biocompatibility, biodegradability, and renewability (Fernandes et al., 2013; Rashid et al., 2018; Simkovic, 2013; Siqueira et al., 2010; Xiong et al., 2018).

CONCLUSION

The potential applicability of lignocellulose-based biocomposites and nanocomposites has been widely extended due to dwindling petroleum resources, their low cost, and due to increasing ecological considerations. Nanocellulose has received a great deal of attention as a promising green nanomaterial for the development of nanostructured biomaterials. Nanocellulose has attractive structural and physicochemical properties, such as biocompatibility, biodegradability, renewability, low density, optical transparency as well as improved thermal and mechanical properties. All these outstanding properties make nanocellulose an ideal candidate for applications in diverse fields. The development of commercially viable “green products” based on natural resources for both matrices and reinforcements for a wide range of applications is on the rise. These efforts include new ways of producing natural polymers with improved mechanical properties and thermal stability using nanotechnology and natural polymers to make biodegradable plastics and their composites with lignocellulosic fibers. In general, the use of lignocellulosic fibers in biodegradable composites can help generate jobs in both rural and urban areas, in addition to helping reduce waste, thus, contributing to a healthier environment. However, more studies are required on product development and for performance evaluation. The practical applications of such fillers and for their transition into industrial technology, a favorable ratio between the expected performances of these composite materials and their cost is required. There are still significant scientific and technological challenges to overcome.

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