Journal of Polymer Science and Engineering 2024, 7(1), 4496.
https://doi.org/10.24294/jpse.v7i1.4496

Review

Recent advancements in nanocellulose synthesis, characterization, and application: A review

Shamim Ahsan, M. S. Rabbi*

Department of Mechanical Engineering, Chittagong University of Engineering and Technology, Chattogram 4349, Bangladesh
*** Corresponding author:** M. S. Rabbi, rabbi@cuet.ac.bd

CITATION

Ahsan S, Rabbi MS. Recent advancements in nanocellulose synthesis, characterization, and application: A review. Journal of Polymer Science and Engineering. 2024; 7(1): 4496. https://doi.org/10.24294/jpse.v7i1.4496

ARTICLE INFO

Received: 31 January 2024
Accepted: 26 March 2024
Available online: 28 April 2024

Abstract: Cellulose nanocrystal, known as CNCs, is a form of material that can be produced by synthesizing carbon from naturally occurring substances, such as plants. Due to the unique properties it possesses, including a large surface area, impressive mechanical strength, and the ability to biodegrade, it draws significant attention from researchers nowadays. Several methods are available to prepare CNC, such as acid hydrolysis, enzymatic hydrolysis, and mechanical procedures. The characteristics of CNC include X-ray diffraction, transmission electron microscopy, dynamic light scattering, etc. In this article, the recent development of CNC preparation and its characterizations are thoroughly discussed. Significant breakthroughs are listed accordingly. Furthermore, a variety of CNC applications, such as paper and packaging, biological applications, energy storage, etc., are illustrated. This study demonstrates the insights gained from using CNC as a potential environmentally friendly material with remarkable properties.

Keywords: Cellulose Nanocrystal (CNC); manufacturing; characterization; application

1. Introduction

Nowadays, each and every living thing is deeply concerned about the condition of the environment. Throughout history, several artificial products and substances have been created in order to fulfill the needs and desires of individuals. These synthetic materials have the potential to adversely affect both the environment and our way of life. Given these concerns and the growing scarcity of non-renewable resources, there is a growing focus on renewable resources and bio-based materials as viable alternatives for producing valuable commodities. Nanocellulose, derived from lignocellulosic sources, is a plentiful and eco-friendly substance. The material has garnered significant attention from both the scientific and industrial sectors due to its exceptional mechanical capabilities, extensive surface area, ample hydroxyl groups for modification, and inherent environmentally friendly properties [1–3]. Cellulose, an abundant and renewable constituent obtained from biodiversity, is commonly acknowledged as the major component among its equivalents. It is well acknowledged as a plentiful and abundant source of raw materials. The presence of this phenomenon is widespread among a diverse array of organisms, including plants, tunicates, algae plants, and certain bacteria [4,5]. The intrinsic adaptability of the material enables several changes and functionalizations, rendering it extremely suitable for various industrial applications. Current study endeavors persist in revealing novel understandings and progressions in the domain of cellulose, hence necessitating further investigation and recognition [6–8]. The benefits of cellulose can be augmented by the aggregation of cellulose chains, leading to the creation of highly structured domains that can be synthesized as nanoparticles from cellulose. Cellulose nanocrystaline (CNC), referred to as nanocelluloses, has received considerable interest in the realm of advanced materials due to their distinctive physicochemical characteristics [9]. In addition, it is important to underscore that these compounds demonstrate chemical inertness, low mass density, notable rigidity, exceptional resilience, a minimal thermal expansion coefficient, and the capacity to modify their surface chemical properties [10–13]. Nanocellulose exhibits a range of favorable properties in comparison to its bulk equivalent, owing to its nanoscale dimensions. These include the nanoscale effect, surface chemical reactivity, biocompatibility, crystallinity, purity, biodegradability, and high mechanical strength, as well as being environmentally friendly and non-toxic [14–16]. Nevertheless, the characteristics of nanocellulose, including yield, surface chemistry, physicochemical, dimensions and morphology, crystallinity, physicochemical, as well as thermal properties, can be customized for specific applications through variations in the sources, isolation procedure, conditions, and various treatments. This customization enables the development of novel materials and devices, offering a wide array of possibilities [17,18].

Examples of nanostructured materials include microcrystals and microfibrils of cellulose, whereas examples of nanofibers include bacterial cellulose, nanofibrils of cellulose, and nanocrystals of cellulose [19–21]. There are numerous diverse varieties of nanocellulose that can be generated through the use of many different processes. These nanocelluloses are derived from many different kinds of cellulose [11,22,23]. The exploration of producing nanocellulose with a diverse assortment of physical attributes is considered a captivating area of research, as it offers the opportunity to explore underutilized biomass resources. Significant benefits may be gained by using nanocellulose due to its 3-dimensional hierarchical nanostructure and its nanoscale physicochemical features, as investigated in distinctive studies [13,22,24,25], which suggest intriguing potential for diverse applications. Nanocellulose, a material currently undergoing mass production at an industrial level of multiple metric tons per day, exhibits a diverse array of uses across multiple disciplines. The range of applications for nanocomposite materials is extensive and encompasses various fields such as biomedical products, wood adhesives, supercapacitors, templates for electronic components, batteries, electroactive polymers, continuous fibers and textiles, food coatings, barrier/separation membranes, antimicrobial films, paper products, cosmetics, cement, and other emerging uses [26,27]. There has been a noticeable increase in the publication of literature review articles in recent years, with a predominant focus on the production, modification, and applications of nanocellulose [8,24,28–35]. This study is intended to provide a concise summary of the scholarly investigations carried out on nanocelluloses, with a specific focus on CNCs, and to emphasize their recent progress in many fields of application. The text begins by presenting a succinct introduction to cellulose, referred to as nanocellulose, and discusses the techniques used to extract it from different sources. In the subsequent sections, an overview will be presented of contemporary trends and recent scholarly investigations concerning the application of nanocellulose. The scope of this study encompasses a targeted examination of the various uses of this material, with particular emphasis on its utilization in various sectors. This review is expected to make a valuable contribution to the progress of preparation techniques for CNC and the development and assembly of innovative products derived from CNC. The overarching objective is to facilitate a greater profusion of CNC-fabricated substances in cutting-edge applications.

2. Nanocullose details

Cellulose structure and source

One of the most common polymeric resources on Earth is cellulose, a renewable feedstock with a large supply. It is reported that the projected yearly production varies between 10^{10} and 10^{11} metric tons [7]. Surprisingly, a relatively small quantity of approximately 6×10^9 tons is being utilized throughout many industrial sectors, including minerals, chemicals, paper, and textiles. The phenomenon of underutilization continues to exist, even after the molecular structure of this white biomacromolecule was clarified by Herman Staudinger a few years following its original isolation by Anselme Payen in 1838 [36]. Cellulose mostly consists of anhydroglucose units (AGUs), which are comprised of repeated β (1,4)-linked D-glucopyranosyl units. The units are arranged in a conformation referred to as a ${}^{4}_{1}C$-chain. According to the findings of Gopi et al., the polymer’s helical structure is a result of the 180° twist that each monomer unit undergoes in relation to the neighboring units [37]. The aforementioned anhydroglucose units (AGUs) undergo a process of chemical bonding to produce cellobiose units. These cellobiose units then aggregate to form the crystalline structure of cellulose, known as primary fibrils. The phenomenon of fibril bundling initiates the generation of micro-fibrils, which subsequently assemble to become cellulose fibers, often known as macro-fibrils. Cellulose possesses distinct properties like hydrophilicity, chirality, simple chemical modification, and infusibility. The existence of intramolecular and intermolecular chemical groups within the cellulose structure is responsible for these characteristics [38]. The length of cellulose chains is mostly determined by the degree of polymerization. Cellulose derived from wood generally comprises approximately 10,000 AGUs, whereas cellulose derived from cotton consists of approximately 15,000 units. The features of cellulose are determined by the degree to which it has been polymerized as well as the length of the chain it contains. The degree of crystallinity of natural cellulose can range anywhere from around 40% to about 70%, depending on where the cellulose comes from and how it was extracted. In the literature, it has been noted that amorphous regions, which lack a distinct crystalline structure, demonstrate greater reactivity with other molecular groups and possess a lower density when compared to regions characterized by a crystalline structure [39–41].

Crystalline domains demonstrate enhanced resistance to mechanical, chemical, and enzymatic interventions in comparison to amorphous regions. Cellulose has a diverse array of polymorphic forms, including cellulose I, II, III_I, III_{II}, IV_I, and IV_{II}. The alteration of these structures can be achieved using either thermal or chemical methodologies. The alterations witnessed in this specific setting are impacted by a variety of factors, including the orientations of molecules, the existence of van der Waals forces, and both intra- and intermolecular interactions. Moreover, the methodologies employed for the segregation and administration of these modifications play a pivotal role in their development [42–44]. The hydrolytic activity is facilitated by the lowered steric barrier and kinetic considerations, allowing for the dispersion of areas of disorder as chain dislocations among segments of the elementary fibril. On the other hand, the domains that are arranged in an orderly manner demonstrate a higher level of resistance to the hydrolysis process and do not experience any changes. Subsequently, the cellulose fibrils undergo transverse cleavage, leading to the generation of CNC characterized by a substantially elevated level of crystallinity. However, after this subsequent phase, further steps such as washing, solvent removal, neutralization, purification, filtration, centrifugation, sonication, dialysis, fractionation, surface modification, and drying (specifically spray-drying and freeze-drying) are necessary to recover the CNC product following the hydrolysis process.

A low-cost method for removing lignin and other impurities from the surfaces of natural fibers is alkali treatment. Surface treatment of thermosets and thermoplastics with alkaline fibers is common. The process of this treatment causes an increase in surface roughness by breaking hydrogen bonds. The rough texture of natural fibers makes them ideal matrix materials for attachment. Alkaline treatments have the potential to improve mechanical properties while having a negative effect on tensile strength. Surface roughness and cellulose content of fibers are both enhanced by alkaline treatment [45]. The interlocking of polymer resins is improved by an alkaline treatment, which raises the crystallinity index, lowers the spiral angle, and removes cementing components from the fibers. The index and crystallinity of natural fibers are proportional to the percentage of solution [46]. To improve the wettability of epoxy resin, alkali was used to decrease the intercrystalline and intercrystalline lignin in sisal fibers [47]. To determine the ideal concentration of the treatment fluid to eliminate impurities while diminishing surface quality, researchers employed scanning electron microscopy [5,48].

Several theories describe the mechanisms by which the interfacial bonding of silane coupling agents enhances the mechanical properties of biocomposites. Adding salt to fiber-polymer composites makes them stickier. The matrix system determines the salinity level. Composites benefit more from a salt treatment than an alkaline one in terms of Young’s modulus [49]. Saline coupling agents significantly enhanced the interlocking of fibers and polymers. In 80 min, coupling agents are dissolved by airdrying sisal fibers immersed in a 2% aminosaline solution. Also examined are saline solutions in water and ethanol at concentrations of 0.033% and 1% [5,50].

The hydrophobicity of fibers is enhanced through acetylation and other surface changes. Through this modification, acyl groups are substituted for lignocellulosic hydroxyl groups. Acetylation is a surface modification process that uses acetates such as acidic anhydride and acetyl chloride. For 1 h at 30 °C, researchers submerged sisal fibers in 5% and 10% NaOH solutions; for 1 h, alkaline-treated fibers were immersed in glacial acetic acid; and for 1 h, pretreatment fibers were immersed in acetic anhydride with one drop of concentrated H_2SO_4 [51]. Cellulose gains additional adhesive bonding sites for polymer resins when lignin and hemicellulose are removed from fibers through acetylation. The amount of time a fiber is soaked is also crucial when changing its surface. Polymer resins bind with fibers less effectively after prolonged soaking. The bending strength and modulus of sugar palm fibers treated with 4% acetyl acid were maximal before they began to decrease [52].

The hydrophobicity of natural fibers is decreased and matrix interaction is increased by benzoyl chloride. When compared to untreated fibers, those treated with benzoyl showed a 25% increase in thermal diffusivity and a 16% increase in conductivity [53]. Benzoyl treatment altered the surface properties and visual appearance of sisal fibers. The surfaces of the fibers are chemically roughened by reacting with benzoyl chloride [54]. Benzoylation improves thermal stability, decreases water absorption, and reinforces fiber-matrix bonding. Benzoyl chloride and sodium hydroxide are used to treat the surface of Sansevieria ehrenbergii fibers. Composites produced from treated fibers demonstrated superior thermal stability compared to those made from untreated fibers [55].

The process of copolymerization between cellulose hydroxyl groups and permanganate ions is initiated by potassium permanganate in acetone to improve the surface features of natural fibers. The mechanical and thermal properties of biocomposites are enhanced when lignin and permanganate ions are used to remove lignin from fiber cells. Natural fiber $KMnO_4$ oxidation was employed in the treatment. When the concentration of $KMnO_4$ is increased, the hydrophobicity of natural fibers is reduced [56]. The fiber surfaces are degraded due to the long soaking time and high $KMnO_4$ concentration. Although $KMnO_4$-treated sisal fibers showed an initial increase in crystallinity, this effect was attenuated as concentration increased [57]. The surface properties are affected by the chemical reactions that take place between the cellulose fibers. Compared to NaOH, the tensile, flexural, and impact strengths of sugar palm fibers treated with $KMnO_4$ were lower. Sugar palm fibers are significantly oxidized at higher $KMnO_4$ concentrations, which in turn reduces the flexural and impact strengths of the biocomposites [58].

3. CNCs preparation

After undergoing pretreatment, the fibers are put through a controlled procedure that involves the hydrolysis of strong acids. During the CNC fabrication process of strong acid hydrolysis, hydronium ions are able to break through the cellulose chain in both the aromatic and amorphous regions. Infiltration leads to the creation of cellulose crystallites, which in turn are caused by the hydrolysis of glycosidic linkages. When the cellulose nanoparticles created by this process are suspended in water, they are referred to as cellulose nanocrystals (CNCs) and cellulose nanowhiskers (CNWs), which are also common names for nanoparticles [59]. The following subsections illustrate the various types of acid hydrolysis along with their corresponding advantages and disadvantages.

3.1. Sulfuric acid hydrolysis

The successful application of sulfuric acid hydrolysis for the synthesis of sulfonated cellulose nanocrystals (SCNCs) from cellulosic biomass sources was not achieved until 1947. The weight percentage (wt%) of sulfuric acid is a crucial metric that must be taken into account while treating the SCNCs. According to Nagarajan et al., insufficient acid hydrolysis may result in the formation of a high-amorphous zone containing low-crystalline particles when the acid concentration is below 63–64 wt% [60]. This process leads to a lower dissolution of amorphous and aromatic polymers. The production of SCNCs exhibits higher yields when the acid content falls within the range of 63 to 64 wt% and the temperature is maintained between 45 °C and 60 °C for a duration of 30 to 120 min. It can be inferred that when the concentration of acid exceeds 64 wt%, there is a likelihood of swelling and partial breakdown occurring in the crystalline areas [38,61]. The disordered components inside cellulose fibrils were effectively dissolved using the process of sulfuric acid hydrolysis [62]. Nevertheless, the α-cellulosic crystalline regions inside the cellulosic fibrils exhibit no chemical reaction with sulfuric acid and remain intact. Consequently, the cellulose fibrils undergo transverse cleavage, resulting in the formation of rod-shaped CNC with dimensions ranging from 2.3 to 60 nm in width and 100 to 500 nm in length. These CNCs exhibit a relatively high level of crystallinity and possess exceptional mechanical characteristics [60,30]. A random replacement process with sulfate ester is carried out on the hydroxyl groups that are located on the surface of the CNC. Ultimately, it is observed that the hydrolysis event results in the formation of negatively charged semiconductor nanocrystals (SCNCs), hence contributing to the establishment of a colloidal suspension with enhanced stability. Nevertheless, the thermal stability of SCNCs is quite low as a result of the sulfate group present on their surface [63]. Moreover, the process of neutralizing sulfated cellulose nanocrystals (SCNCs) using sodium hydroxide (NaOH) contributes to the restoration and enhancement of the thermal properties of CNCs produced by hydrolysis with sulfuric acid [64,65].

3.2. Hydrochloric acid hydrolysis

One well-known and often-utilized method for producing cellulose nanocrystals (CNCs) is the hydrolysis of hydrochloric acid. The HCL hydrolysis method involves much longer optimal reaction times (2–4 h) and temperatures (60–105 °C) compared to H_2SO_4 hydrolysis. Keeping the acid concentration between 2.5 and 6.0 N is optimal for CNC yield efficiency [66]. A high degree of crystallinity is produced as α-cellulose during the HCl hydrolysis. This α-cellulose exhibits rod-like, structured

CNCs with diameters ranging from 10 to 12 nm and lengths ranging from 285 to 304 nm [67]. Isolated CNCs tend to clump together when they come into contact with solvents or polymers because they do not possess adequate negative charges. As a direct consequence of this, there is an extensive network of hydrogen bonding connections both inside the molecule itself and between the molecules that make up the complex. This is a direct result of the extremely high concentration of hydroxyl groups that the surface possesses. It has been discovered that CNCs obtained through HCL hydrolysis are more thermally stable than those acquired through H_2SO_4 hydrolysis [68]. Furthermore, the CNCs produced by this hydrolysis exhibit the ability to create stable interfaces between oil and water, known as Pickering emulsions. On the other hand, nanocrystals that have a carboxyl group on their surface don't respond in this way when they come into contact with an interface [69,70].

3.3. Phosphoric acid hydrolysis

Phosphoric acid is a type of mineral acid that is often used to prepare CNCs from biomass. Using a concentration of phosphoric acid that is between 70 and 75 weight percent in this acid hydrolysis process results in an increase in the efficiency with which CNCs are produced from fibers. Notably, the temperature is kept between 100–120 °C, and the duration of the hydrolysis process is typically between 80 and 120 min [71]. One of these benefits is the potential use of the material in applications involving bone scaffolding. In these settings, the material can encourage the formation of new bone while simultaneously delivering greater thermal stability and flame resistance. The isolated CNCs exhibit a rod-like shape, distinguished by a diameter that ranges from 15 to 32 nm and a length that ranges from 238 to 475 nm [72]. During the process, the surfaces of α-cellulosic nanocrystals acquire a charge on their phosphate groups. The extracted CNCs possess a tiny zeta potential, which indicates their low colloidal stability characteristic [73,74].

3.4. Organic acid hydrolysis

Recent research has revealed that CNCs can be isolated from organic acids such as, formic acid, maleic acid, citric acid, and oxalic acid as alternatives to mineral acid. CNCs are obtained using a process of $FeCl_3$-catalyzed hydrolysis of organic-based formic acid. It is shown that the utilization of $FeCl_3$ as a catalyst leads to improved production of CNCs due to better hydrolysis efficiency [75]. The entire process taken 6 h with 88 wt% of formic acid and the temperature is maintained 95 °C. The post-processing procedure resulted in a notable enhancement in the crystalline indices of the CNCs. Additionally, despite their diminutive size, the CNCs exhibited remarkable thermal stability.

Chen et al. used a toluenesulfonic acid solution at 50 wt% as a strong organic acid to speed up the separation of CNCs [76]. By maintaining ambient pressure and heating the eucalyptus pulp to 100 °C for 45 min, the acid easily passes through the cellulosic microfibril bundles and into the amorphous zone. Subsequently, CNCs are separated in a longitudinal orientation along the fibrils.

Researchers have used a 70% oxalic acid solution to isolate CNCs. CNCs can be isolated from eucalyptus pulp. This method exhibits a quicker approach with only half an hour at a temperature of 100 °C. The isolated CNCs are found to be more stable at high temperatures than their non-isolated counterparts. This is because CNCs and water don't come into as much physical contact.

Seta et al. applied a maleic acid solution with a high concentration to perform hydrolysis on ball-milled bamboo pulp [77]. The pulp is treated with a liquid solution whose weight-to-pulp's weight ratio was 100:1, yielding a maleic acid concentration of 75 wt%. The hydrolysis process was completed at a temperature of 110 °C for 3 h while ensuring steady stirring. The CNCs obtained exhibit favorable thermal stability at a temperature of 291 °C with a rod-shaped morphology of lengths ranging from 105.6 to 223.8 nm. In contrast, the yield of cellulose nanocrystals (CNCs) obtained through this particular procedure is somewhat lower (24%) in comparison to the hydrolysis method with formic acid.

The utilization of organic acid hydrolysis technologies demonstrates a higher degree of environmental sustainability in comparison to traditional mineral acid hydrolysis techniques. The utilization of organic acids in recycling processes has several advantages, including reduced corrosion of equipment utilized in the operation. Hence, the current trends greatly endorse and facilitate the achievement of CNCs' industrialization through the utilization of organic acid hydrolysis. **Table 1** presents a comprehensive overview of the acids employed in the hydrolysis process, including their ideal working conditions and notable properties.

Table 1. Comparative study of several key characteristics of different acid hydrolyzed CNCs.

Sl No	Name of the acid hydrolysis	Concentration of acid (wt%)	Reaction temperature (°C)	Reaction time (min.)	Post-processing method	Nature of CNCs	Size of CNCs (nm)	Ref.
01	Hydrochloric acid hydrolysis	37	50	60	Centrifugation and sonication	Rod like structure	10 ± 2 (Width) 285 ± 58 (Length)	[78]
02	Sulfuric acid hydrolysis	64	45	60	Centrifugation, dialysis tube and sonication	Rod like structure	12 ± 1.2 (Width) 280 ± 20 (Length)	[38]
03	Phosphoric acid hydrolysis	80	50	420	Centrifugation, dialysis tube and sonication	Rod like structure	2–6 (Width) 100–330 (Length)	[79]
04	Formic acid hydrolysis (organic)	88	92	720	Centrifugation	Rod like structure	5–20 (Width) 50–200 (Length)	[75]

4. Surface modification of CNC

Surface chemistry can be altered through chemical manipulation, physical interactions, and biological means [80]. The presence of OH groups on the surface of nanocellulose has the effect of making it very hydrophilic. The surface functionalization of nanocellulose can be accomplished either during the manufacturing process or after it has been completed [81–84]. Their performance in a particular application is improved as a result of these modifications, which achieve desirable attributes. According to George and Sabapathi, incorporating any chemical functionality into a nanocellulosic material allows for the surface of the material to be modified such that it reacts differently with outer molecules [85]. The change of the surface of polysaccharide nanocrystals can lead to the formation of polymeric matrices that have greater reinforcing, which means that they have improved thermal and mechanical properties, as stated by Lin et al. [81].

As stated in earlier works, the most common methods for chemically altering the surface of cellulose nanocrystals are covalent surface modifications such as sulfonation, polymer grafting, oxidation, esterification, nucleophilic substitution, and etherification. These modifications modify the surface of the nanocrystals in a variety of different ways [80,82–86]. Polyacrylamide was grafted onto cellulose nanocrystals (CNC) and subsequently integrated into polyvinyl alcohol (PVA) in a recent study that used a solution-casting process to reinforce nanocomposite films. Polyacrylamide was then included in the PVA. With cinnamoyl chloride and butyryl chloride grafted onto them, hydrophobically modified cellulose nanofibrils displayed good surface qualities and were able to stabilize oil-water emulsions, as demonstrated by research conducted by Tong et al. [87]. Due to the fact that they are unable to stabilize Pickering emulsions, nanocelluloses that have a high surface charge density are not suitable for use as interfacial stabilizers. The surfaces of nanocelluloses can be changed by grafting hydrophobic polymers onto them in order to reduce the amount of interfacial tension that exists between them. Because of this, they become more wettable when exposed to the oil phase. According to Kaboorani and Riedl, polymer coatings and adsorbing surfactants have also been employed in this research [88].

Irradiation, surface fibrillation, ultrasonic treatment, and electric discharge (plasma treatment) are some of the available therapies for physical surface functionalization [80]. All of these treatments are possible. Afrin and Karim, for instance, used enzymes to modify the surface of nanocellulose in two different ways: (a) direct modification, in which the enzyme comes into direct contact with the nanocellulose; and (b) indirect enzyme-mediated modification [82]. Both of these enzyme-based approaches were utilized. It was determined by them that the enzymatic technique for creating nanocellulose and functionalizing it further is a more sustainable alteration method than the chemical modification strategy. This was the result that they arrived at. In order to maintain the integrity of the crystal structure, the approach must only alter the surface of the cellulose nanofibrils (CNF) without altering the morphology. This is one of the most significant obstacles that must be overcome when attempting to alter the CNF. According to Robles et al., the creation of hydrophobic surfaces of CNC and CNF through surface modification with silanes results in an increase in the stability of these materials [89]. **Figure 1** provides a schematic representation of the most widely used techniques for modifying the surface of nanocellulose. **Table 2** demonstrates the impact of surface treatments on natural fibers.

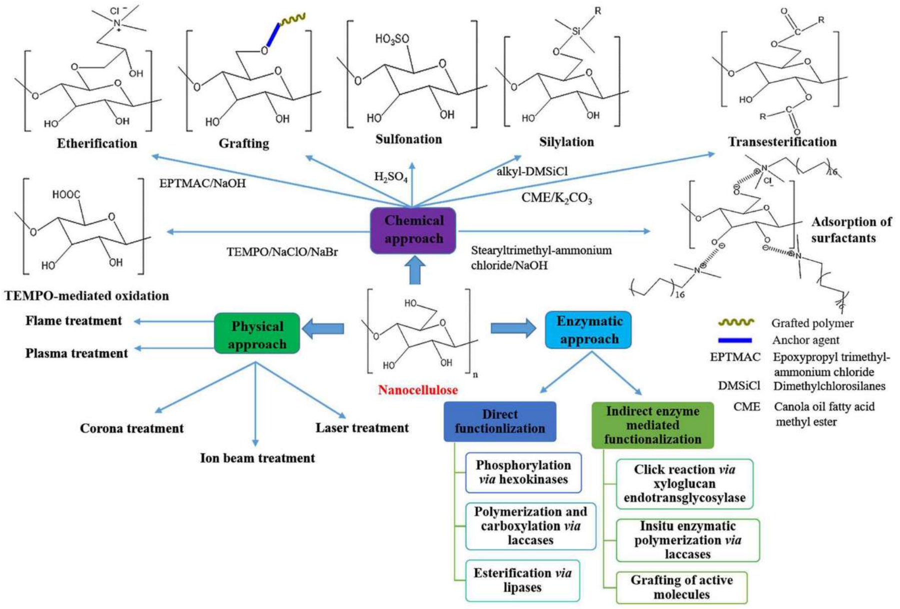


Figure 1. Schematic representation of the most commonly used surface modification routes for nanocellulose [7].

Table 2. The consequences of surface treatments on natural fibers.

Surface Treatment	Treatment effect	References
Plasma treatment	The surface roughness of plant fibers is increased by etching, leading to a more mechanically interlocking interaction with the matrix.	[90], [91], [92], [93], [94]
Vacuum Ultraviolet Irradiation treatment	Enhances the surface characteristics encompassing adhesion, wettability, tribological properties, fouling resistance, barrier performance, insulating capacity, dye absorption, and biocompatibility.	[95]
Ozone treatment	Contributes to the preservation of its physical attributes.	[96]
Corona treatment	Increase the basicity and acidity of the fiber surface	[97], [98], [99]
γ -Ray treatment	The structural integrity of natural fiber is shown to increase as a result of exposure to gamma radiation, which induces intercross-linking between adjacent cellulose molecules.	[100], [101], [102], [103]
Laser treatment	Reducing the concentration of lignin while simultaneously enhancing the structural characteristics of the fibers.	[104], [105], [106]

5. Characterization methods

5.1. Fourier-transform infrared spectroscopy

Fourier transform infrared spectroscopy (FTIR) is widely recognized as a non-destructive analytical technique that offers both quantitative and qualitative information about natural fibers. The chemical contents of natural fibers are analyzed to obtain an infrared absorption spectrum. The major range of frequency for observing infrared spectra derived from natural fibers is typically between 400 and 4000 cm^{-1} [107,108]. The FTIR peak positions on the natural fiber, along with the accompanying chemical stretching mode vibrations, are shown in **Table 3**.

Table 3. FTIR peak locations and the vibrations of the associated chemical stretching mode [109–115].

Wave number (cm^{-1})	Allocations
3600–3500	O–H stretching oα-cellulose
3500–3300	N–H stretching (Amine)
3500	N–H stretching (Amine)
3100–3000	C=C–H stretching
2960–2850	C–H stretching
2590–2540	S–H stretching
1740–1720	C=O stretching of hemicelluloses
1685–1655 & 1600	α, β unsaturated stretching
1680–1650	N=O stretching
1650–1630	OH (Absorbed water)
1600	C=O stretching (Amide)
1600,1580,1500,1450	C=C stretching
1450–1400	CH_2 Symmetric bending
1567,1380	NO_2 stretching
Above 1500	C=O, NH, C=C, C=N functional group stretching
Below 1500	Single bonds and bending vibrations
1395–1385	t-butyl stretching
1365	CH bending (deformation)
1350–1260	C–O stretching
1150–1070	C–O–C stretching
1050–1020	Symmetric C–OH stretching of lignin
815–750	N–O stretching
800–600	C–S stretching
770–735	C–C deformation
520–420	S–S stretching

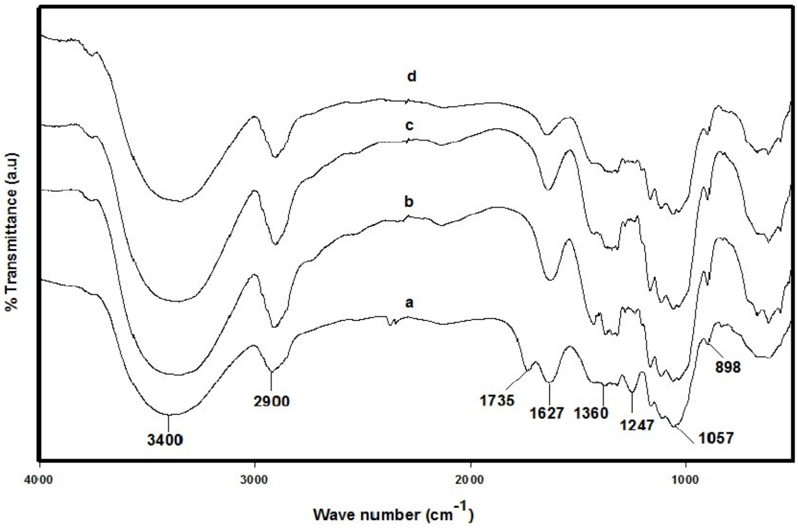


Figure 2. FTIR spectra of **(a)** raw fibers, **(b)** alkali-treated fibers, **(c)** bleached fibers, and **(d)** CNC [123].

C=O bond stretching vibrational modes in carboxylic acids and hemicellulose esters, C=C bonds in lignin's benzene rings, and the C=O bonds of lignin's acetyl group are all effectively blocked, as was the infrared absorption spectrum of natural fibers (A), pre-treated fibers (B), and bleached fibers (C), as well as cellulose nanocrystal (D). This discovery demonstrates that the processing of CNC resulted in the removal of hemicellulose and lignin from the CNC [116]. The spectral range from 1635 to 1640 cm^{-1} has been identified to include vibrational modes of OH-group stretching and bending in absorbed water [117,118]. Vibrations of the OH-group, the most abundant organic group in the stem, are attributed to the spectral region between 3300 and 3400 cm^{-1}. The observed peaks within the spectral range of 2800–2900 cm^{-1} can be attributed to the stretching vibrations of C–H bonds [119,120]. Additional peaks are seen in the raw fibers' spectra at 1730 and 1250 cm^{-1}. The C=O bond stretching vibrations in the acetyl and C-O bond stretching vibrations in the aryl groups account for the observed peaks. Lignin and hemicellulose both include these functional groups [120–122]. The aforementioned compounds are found to be completely absent in fibers that underwent alkali treatment and bleaching. Chemical treatments have a clear effect on the chemical composition of fibers, as evidenced by the differences in peak intensities between raw fibers and CNC in the spectrum areas of 2700 to 3600 cm^{-1} and 850 to 1500 cm^{-1}. In addition, the 850 to 1500 cm^{-1} transmittance peaks, which are characteristic of the cellulose material's crystal structure, were not observed in the CNC. With each successive treatment, the height of these peaks diminished. The absence of lignin, hemicellulose, and wax in the fibers suggests that they have been processed. **Figure 2** presents a visual representation of the Fourier Transform.

5.2. X-ray powder diffraction (XRD) analysis

X-ray diffraction, often known as XRD, is a non-destructive and expeditious type of analytical technique that is frequently utilized for the determination of the chemical properties of natural fibers [107,124,125]. The spectrum obtained from the experiments, which corresponds to a specific fiber, exhibits the diffraction peaks associated with both the amorphous and crystalline areas. The crystallinity index (CI) is determined using the formula,

$$CI = \left(1 - {}^{I_{AM}}/_{I_{00}}\right) \times 100\%$$

where I_{AM} represents the intensity of the amorphous phase and I_{00} represents the intensity of the fully crystalline phase [126]. **Figure 3** displays a comparison of the diffractograms of cellulose following each chemical treatment. The peaks is identified at a 2θ angle of 14.7°, 22.6°, and 34.4° are indicative of the normal cellulose I structure. The CNC exhibited a distinct and pronounced peak at an angle of 22.6°. Wax, hemicellulose, and lignin are found to be present in a unique zone that was indicated by the wide pattern of the diffractogram that was acquired from the raw fibers.

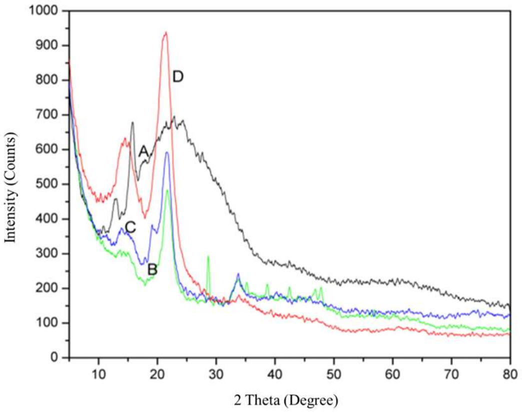


Figure 3. The X-ray diffraction (XRD) pattern of natural fibers **(A)**, pre-treated fibers **(B)**, bleached fibers **(C)**, and cellulose nanocrystals (CNC) was analyzed [127].

It was found that the crystallinity improved by using the alkali treatment due to the fact that the lignin and hemicellulose components were removed by the treatment. After going through the acid hydrolysis procedure, the nanocrystals had a crystallinity of around 64.2%, which is just marginally greater than the crystallinity of the bleached fibers. Because alkali treatment results in the formation of cellulose nanofibrils rather than cellulose crystals, the change in crystallinity index that occurs between acid hydrolysis and alkali treatment is almost insignificant [116]. The amorphous component included within the amorphous region was successfully extracted using the chemical treatment [128]. The degree of crystallinity showed a significant increase of 252% when comparing the raw fibers (18.2%) to the fully treated fibers (64.2%) [116]. The study reported an observed crystallinity of 69% for derived nanofibers made from poplar wood [129]. The increase in crystallinity seen in the fiber after treatment is documented in the literature, with values ranging from 78% for banana rachis [116] to as high as 450% for banana peel bran [128].

5.3. Scanning electron Microscope (SEM) analysis

A scanning electron microscope (SEM), which is one of the most flexible pieces of equipment that is now accessible, can be used to facilitate the surface topography assessment of the micro and nanostructure morphologies of cellulose-based nanocomposites. This assessment and differentiation of objects on a nanoscale can be accomplished. The use of SEM is the most effective method for illustrating the properties of nanocomposite materials as a function of nanofiber dimensions, shapes, orientations, and interactions between the matrix and nanofibers [130]. In order to map out the topography of the material as it travels across its surface, it makes use of electron beams that have been carefully focused during the process.

It is possible for the electron beam to penetrate the sample to a depth of a few microns when it hits the surface; this is contingent upon the accelerating voltage as well as the density of the sample. A multitude of signals, such as X-rays and secondary electrons, are produced as a result of this interaction contained within the sample. Several studies have found that the resolution of SEM images is typically associated with the size of the electron beam spot, which is smaller at higher acceleration voltages (AV). This is the case whenever the acceleration voltage is increased [131–134].

In order to prepare nanocomposite samples for electron irradiation without causing electrostatic charges to form on the surface, the samples must have surfaces that are electrically conductive and must be grounded to a sample holder. SEM imaging of cellulose nanocomposites can result in a number of undesirable consequences, including scanning errors, picture distortions, and nanocomposite charging. This is due to the fact that CNCs are not conductive. Sample preparation is an essential step in the process of creating nanocomposites, which are frequently sputtered in a vacuum with an extremely thin layer of conductive materials such as carbon, gold, or platinum [135]. When working with cellulose nanocrystals, scanning electron micrographs are typically utilized for the purpose of evaluating the surface morphology of nanocomposites as well as the interaction between polymers and CNCs [132,134,136]. For instance, SEM is frequently utilized for the purpose of analyzing the fracture surface of nanocomposites that have been manufactured, as well as for determining the dispersion of CNCs inside a host polymer matrix when subjected to tensile or impact stresses [131,133]. As shown in **Figure 4**, SEMs of CNCs that were manufactured with various acid concentrations are exhibited.

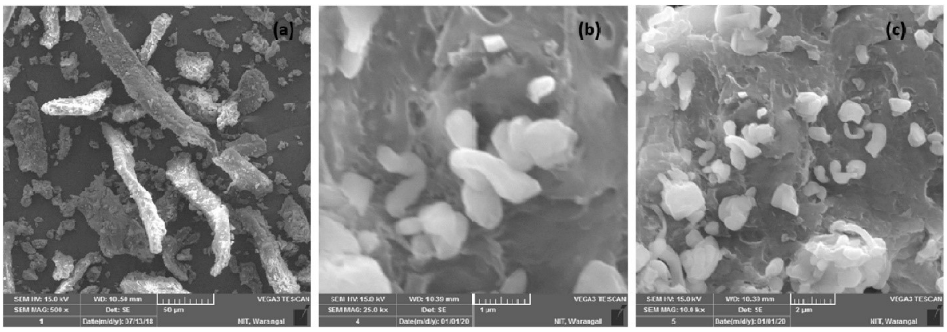


Figure 4. SEM images of as-synthesized CNCs at **(a)** 50 μm; **(b)** 1 μm; and **(c)** 2 μm scale [137].

5.4. Atomic force microscopy (AFM) analysis

The technique known as atomic force microscopy (AFM) makes it possible to capture surface profile details with a granularity that is frequently greater than one micron. AFM has the capability to directly quantify the predominant forces involved in adhesion processes, as demonstrated by studies conducted by Balnois et al. [138] and Ben Sghaier et al. [139]. This approach necessitates the scanning of a sharp-tipped cantilever over the fiber. The deflection of the cantilever is facilitated by the repulsive forces generated upon contact between the cantilever tip and the fiber. The AFM has the capability to analyze and describe the surface properties of fibers in three dimensions [140]. The AFM topography, as illustrated in **Figure 5**, exhibits the presence of fibers at the macro, micro, and nanoscales [141].

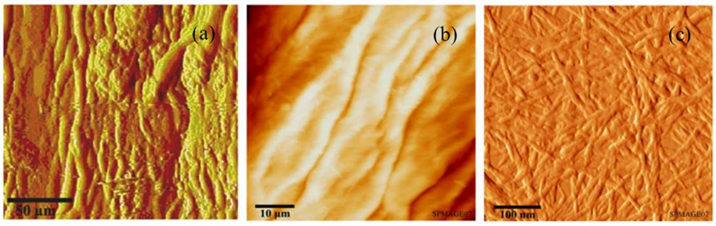
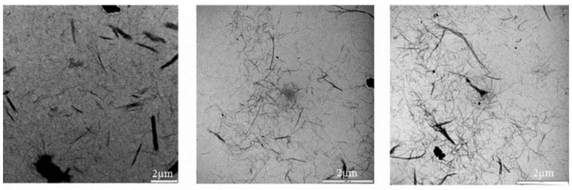


Figure 5. The atomic force microscopy micro-, nano-, and macro-fiber topographies of a banana pseudo stem [141].

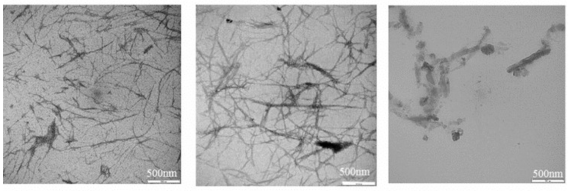
The mean diameter of the nanofibers was calculated to be around 30 nm. Micro- and nanofibers are formed more effectively by removing the intermediate lamella and main cell walls. Consequently, the surface has been enriched with a higher concentration of cellulose, and the dimensions of the fibers have been effectively reduced to the nanoscale. According to the findings of Chokshi et al. [142], the microfibrillar angle is identified as the key determinant of the intrinsic fiber strength properties. It is reported that achieving high fiber strength requires an increased proportion of cellulosic content and a decreased value of MFA. The key factors contributing to the poor elongation of banana fibers are the lower microfibrillar angle (11°) and the relatively high percentage of crystallinity [143].

5.5. Transmission electron microscope (TEM) analysis

Transmission electron microscopy (TEM) micrographs provide accurate measurements of the fiber diameters and are useful for studying the axial thickness of a cell wall and its component sublayers [144]. Nevertheless, the use of these techniques necessitates a rigorous procedure for the processing of samples. The fibers are subjected to a double boiling process using an excessive 1% NaOH solution for a duration of 3 h. Subsequently, a solution of HCL with a concentration of 0.05 M is applied to the fibers, and then they are thoroughly rinsed with water. The fibers are immersed in distilled water for a duration of 4 h, facilitating the creation of a sample that enables meticulous study of the structural characteristics of the fibers using TEM [145]. Images captured using an electron microscope The TEM images present in **Figure 6** illustrate the structural characteristics of distinct nanocellulose samples derived through varying acid hydrolysis conditions.



35% (v/v) acid, 3h, at 45°C　35% (v/v) acid, 6h, at 45°C　35% (v/v) acid, 3h, at 50°C



40% (v/v) acid, 3h, at 45°C　45% (v/v) acid, 1h, at 45°C　50% (v/v) acid, 1h, at 45°C

Figure 6. TEM images of nanocellulose samples using different acid hydrolysis conditions [146].

6. Application of CNCs

CNCs are considered a promising and environmentally friendly alternative to inorganic materials. The principal utilization of CNC is in the reinforcement of thermoplastic nanocomposites, owing to their advantageous mechanical characteristics and convenient modification. A diverse assortment of polymers has been widely studied as the host matrix for many applications, including a wide spectrum of functions.

6.1. Packaging

Food packaging materials have a twofold function: firstly, to protect food from external factors that may interfere with or contaminate it, and secondly, to improve the nutritional quality of the food by preventing the migration of small molecules. Although polymer materials are widely used in the packaging industry, they are not completely impervious to small molecules, including gases, organic compounds, and water vapor. This deficiency in their barrier properties might have adverse effects on their appropriateness for particular applications. Numerous methodologies have been explored in order to mitigate the limitations associated with polymer matrices. The utilization of CNC as reinforcing agents and permeability enhancers is a commonly employed strategy for enhancing the barrier characteristics of polymer matrices. In recent decades, numerous scientists have integrated CNC into various polymers to create nanocomposite films that exhibit exceptional performance. These films have found applications in the food packaging industry. Extensive literature has documented several significant avenues for enhancing packaging materials that incorporate CNC. Yadav and Chiu conducted a study whereby they investigated the impact of incorporating different weight percentages of CNC into glycerol films containing κ-carrageenan [147]. The study revealed a significant reduction of 52% in water vapor permeability (WVP) due to the physical impediment generated by CNC, which restricts the flow of water across the packing film. Furthermore, a significant rise in permeability was noted in the nanocomposite films. Salmieri et al. developed oregano essential oil that incorporated PLA-CNC nanocomposite films with antibacterial properties. It was found that there was a noticeable increase in the tensile strength of nanocomposite films after including CNC in packaged food goods, which increased their water absorption capacity [148].

de Oliveira et al. derived CNC from oats and eucalyptus to fabricate aerogels for use as absorbers in the food packaging industry. Hey reported a decrease in water absorption characteristics, which can be attributed to the presence of larger pore sizes. Hence, the utilization of aerogels with reduced crystallinity has been proposed as a means to mitigate moisture accumulation in food packaging applications, particularly in the context of meat packing, with the objective of minimizing water condensation [149]. **Table 4** provides an overview of how CNC is used in the food packaging business in conjunction with various polymer matrices.

Table 4. CNC nanocomposite examples in the food packaging industry.

CNC content	Polymer matrix	Thickness	RH (%)	Oxygen barrier property	Property	Ref.
30	Edible coating on alginate film	-	0 50 70	92 $(\mathrm{mL}.\frac{\mu m}{m^2 d^{-1}})$ 250 1427	• The lipid oxidation of chicken has a low level during the initial three-day period of storage.	[150]
4	PET film	1 µm.	0 20 40 60 70 80	0.38 $(\mathrm{mL}.\,m^{-2}d^{-1}bar^{-1})$ 1.03 2.05 23.07 56.41 83.97	• Reduced CO_2 barrier when relative humidity is high	[151]
0.20	Hybrid PVA cellulose bioactive aerogels	-	-	0.38 $(\mathrm{mL}.\,m^{-2}d^{-1}bar^{-1})$	• Bioactive chemicals are released in a controlled manner. • Enhanced water absorption; maintained material integrity while submerged	[152]
1.2 2.4 3.6	Chitosan-nano clay film	40 µm	50	8.1 $(\mathrm{mL}.\,m^{-2}d^{-1}atm^{-1})$ 5.4 7.2	• Enhanced tensile modulus and strength • Increased resistance to heat	[153]
5 7 11	Starch-glycerol-montmorillonite film	-	-	35.26 $(\mathrm{mL}.\,m^{-1}d^{-1}Pa^{-1})$ 37 38.5	• Improvement in tensile strength and tensile Modules • Improvement in barrier properties • Incrementing disintegrability	[154]
1 5	PLA-Ag	200–220 µm	-	16.5 $(\mathrm{mL}.\,min.\,m^{-2}day^{1}$	• Raised the degree of crystallinity in general • The barrier effect is effective • Lessening of vapor transmission through the material	[155]

6.2. Drug delivery

Over the course of the last decade, researchers have focused their attention on several methodologies within the pharmaceutical industry. These methodologies include compression, spray and dip coating, and encapsulation, all of which aim to incorporate particular drugs into polymeric structures for the purpose of targeted distribution and/or controlled release. The utilization of coating materials to encapsulate the medicine is strongly advised in order to get a sustainable, controlled release within a specified timeframe [156,157].

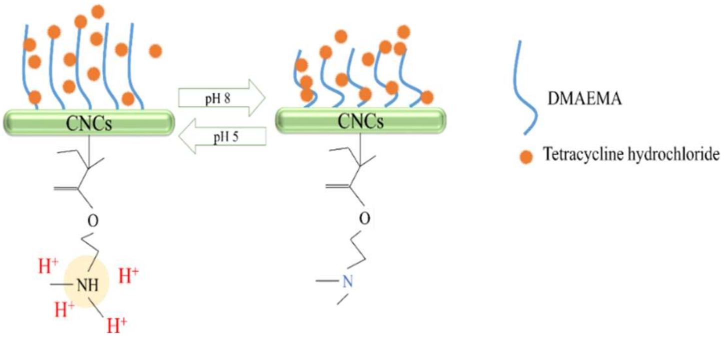


Figure 7. Visual representation of in vitro drug release profiles of PHBV/CNC-g-PDMAEMA composite membranes at varying pH levels [160].

According to the most recent research, CNC has a significant amount of potential both as a medicinal substance and as a carrier for drug delivery systems. Because of its one-of-a-kind characteristics, CNC can be loaded with both charged and neutral medications, released in a controlled manner, and transported to specific cells, where they can have the greatest impact [158,159]. Chen and colleagues investigated the potential uses of poly (3-hydroxybutyrate-co-3-hydroxyvalerate) (PHBV) electrospun composite membranes that were reinforced with poly (dimethylaminoethyl methacrylate) (PDMAEMA)-grafted CNC. A visual representation of such a structure is depicted in **Figure 7**. The goal of the project was to develop membranes that, when subjected to PDMAEMA in a gamma configuration, would enable the controlled and prolonged release of a model drug [160].

Because of their hydrophilic properties and porous structures, which are both made possible by their chiral nematic structures, CNC are ideally suited for use as active components in medicinal products [161], especially in terms of hydrogels. The key factors contributing to the enhancement of drug absorption and dissolution through the integration of CNC into porous hydrogels are CNC's notable hydrophilicity and its tendency to undergo swelling in the presence of water. CNC hydrogels have been widely utilized in many applications due to their high permeability, particularly in converting chemicals [162] as well as thermal [163], hence facilitating drug release in CNC hydrogels. Among these stimuli, pH has been shown to be the most prevalent trigger.

Ooi et al. effectively synthesized semi-interpenetrating network (IPN) hydrogels composed of gelatin and CNC that exhibit stimulus-responsive behavior in response to variations in pH. The use of unadulterated CNC with a strong affinity for water played a crucial role in regulating the porous microarchitecture of the hydrogels. This, in turn, influenced the swelling properties and the rate at which drugs were released under varying pH conditions [164].

However, the hydrophilic characteristics and strongly negative surface properties of virgin CNC limit its potential use for hydrophobic medicines, including anticancer treatments. Consequently, further surface modification techniques can be employed to control the characteristics of CNC in order to facilitate the binding of non-ionized or hydrophobic medicines that would typically not adhere to untreated CNC surfaces. Castro et al. endeavored to functionalize CNC using β-cyclodextrin (β-CD) in order to introduce novel active substances onto the CNC surface and enable the sustained release of antibacterial agents [165]. A schematic representation of propargyl group functionalization in CNC for use in nanomedicine is shown in **Figure 8**. In an alternative study, the surface of CNC was subjected to functionalization by the incorporation of propargyl groups. This modification was carried out with the purpose of producing nanomedicine containing radionuclides, which may be utilized in the context of radio-immune treatment. In the study, the researchers introduced oligoethylene glycol methacrylate and aminopropyl methacrylamide into CNC to create a limited quantity of primary amine pendant groups that had the ability to transport pharmaceutical substances. The investigation into the cellular uptake behavior of CNC conjugates in the context of nanomedicines revealed that ovarian cancer cell lines exhibited uptake of these conjugates. The human breast cancer cell line exhibited a diminished capacity for internalizing CNC conjugates [165].

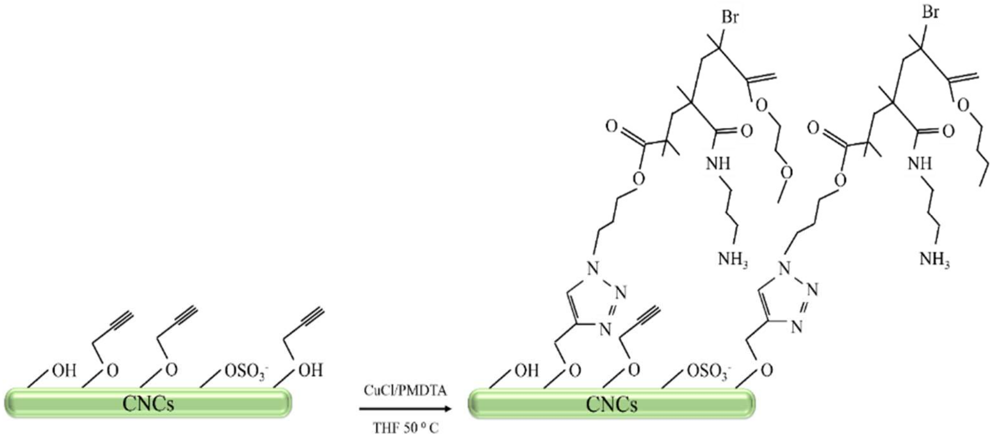


Figure 8. Propargyl group functionalization in CNC for nanomedicine applications [165].

6.3. Wound dressing

Wound dressing materials are specifically engineered to fulfill many crucial functions, including the provision of a protective barrier against bacterial infiltration, facilitation of gaseous exchange, management of wound infections, and facilitation of the wound healing process. There has been a shift in recent years towards using polymers derived from natural sources and hybrids thereof in the field of medical dressings. This trend may be attributed to their inherent biocompatibility, biodegradability, and comparatively less cytotoxic effects. The utilization of CNC in wound dressing materials has been seen, wherein they serve as either a reinforcing agent or a major element. This is mostly attributed to their widespread availability and remarkable mechanical properties [166,167].

In their research, Zhang et al. developed a nanocomposite hydrogel that repairs itself by combining modified CNC carboxymethyl chitosan (CMC) [168]. The goal of developing this hydrogel is to create a more comfortable environment for severe burn wound beds by absorbing and retaining exudate. Analysis of the healing process revealed that, compared to chitosan-based hydrogels, CMC-DACNC hydrogels had better self-healing characteristics. As depicted in **Figure 9**, this may be a result of DACNC’s reinforcing effects.

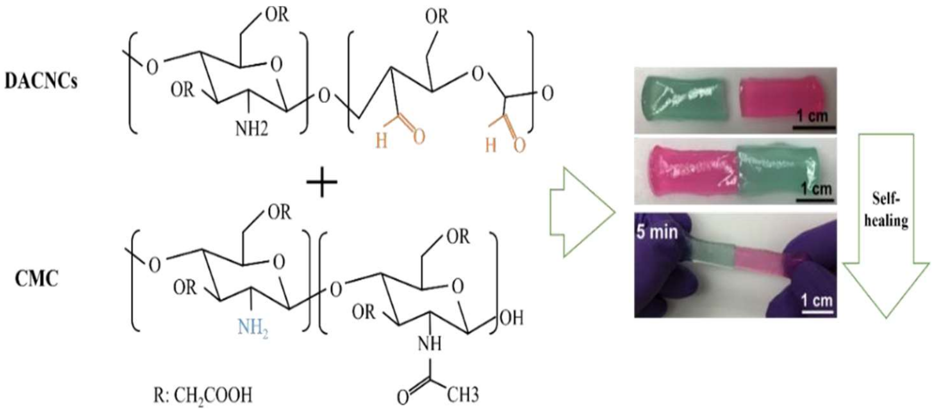


Figure 9. Gel formation and the self-healing process of carboxymethyl cellulose (CMC) and dialdehyde cellulose nanocrystals (DACNC) [168].

Huang et al. described a simple method for synthesizing cellulose nanocrystal (CNC) films, which serve as a delivery mechanism for antimicrobial medications in the setting of diabetic wound dressings [168]. CNC's presence in the PVA-CNC film facilitates a robust connection between curcumin and the film, which in turn protects the curcumin from being washed away.

Cheng et al. conducted a comprehensive investigation to learn more about the effects of TEMPO treatment on modifying CNC by adding carboxyl groups [169]. Calcium ions (Ca^{2+}) are employed as a cross-linking agent in this process. The findings of their study demonstrate that the composite components did not have any impact on the physiological activity of the blood cells.

6.4. Tissue engineering

Biomedical engineering has become more popular over the past ten years, and the use of naturally occurring plastics in tissue engineering has become an important area of study [170]. Within the field of tissue engineering, a pivotal consideration in the identification of suitable synthetic tissues for transplantation into human or animal organs is the advancement of a permeable membrane characterized by meticulous geometric properties and a linked network of pores. The hydrophilic hydroxyl groups found in CNC have been demonstrated in multiple studies published in the literature to have the ability to form a permeable, flexible membrane with a multi- and ultrafine open-pore structure. This unique characteristic of CNC enables enhanced cell adhesion and promotes cell survival, hence facilitating the regeneration of damaged tissues or organs [171]. Lam et al. utilized CNC derived from sugarcane bagasse as a raw material to fabricate porous nanocomposite scaffolds composed of polyvinyl alcohol (PVA) and CNC. These scaffolds are specifically designed for the cultivation of human skin cells. The studies documented the development of porous scaffolds that exhibited no cytotoxic effects and had a cell survival rate of 82%. The pore size of these scaffolds was shown to be significantly influenced by the concentration of CNC. The elevated levels of CNC content led to an enhancement in pore size as a consequence of a decrease in the proportion of free PVA volume. The presence of a larger concentration of CNC led to an observed increase in the degree of swelling, as the CNC particles restricted the mobility of the polymer chains [172].

Gao et al. conducted a study to examine the effects of integrating the in-situ composite process with freeze-drying methodology [173]. This modification, the inclusion of CNC in the nanocomposite scaffolds, led to an enhancement in the surface hydrophilicity of the scaffolds. The enhanced hydrophilicity facilitated favorable interactions between cells and the scaffold, promoting cell development and proliferation.

6.5. Organic photovoltaic (OPV) cells

Organic photovoltaic (OPV) cells are types of solar cells that utilize organic absorbing layers composed of polymers or tiny molecules. These absorbing layers have the ability to capture incident light and convert it into electrical energy through the process of fluorescence absorption and subsequent conversion. CNC nanosheets exhibit inherent traits related to transparency and dispersion of light, making them highly appealing as substrates for efficient organic photovoltaic cells that possess improved light management capabilities. Conversely, the attainment of robust hydrophobic properties and superior mechanical properties is vital for OPV cells [153,174].

6.6. CNC nanocomposites for supercapacitors

Materials possessing a substantial capacity and a swift charging cycle comprise supercapacitors. CNC does not possess inherent electrical conductivity. Nevertheless, they may be utilized as environmentally sustainable and recyclable materials in the fabrication of composite supercapacitors. Chen et al. conducted a study wherein they achieved a uniform distribution of metal nanoparticles (MNPs) on CNC. The application of melamine-formaldehyde (MF) resin to CNC has the dual purpose of protecting CNC from oxidation and facilitating the development of a porous structure with nitrogen functions, which in turn promote the deposition of more metal [175]. Another study synthesizes core-shell PPy/PVP/CNC nanorods by physically adsorbing PVP onto CNC. This technique modified CNC hydrophilicity and promoted pyrrole formation. The core-shell system’s specific capacitance increased by 35%, according to a study [176].

6.7. Smart materials

In recent times, there has been a significant allocation of research efforts towards the advancement of renewable smart materials utilizing biobased components such as CNC [177]. CNC’s large surface area, controlled shape, and structure make it a promising bio-sensing component stabilizer. CNCs are ideal for bioactive chemical immobilization due to their unique properties [178,179]. Furthermore, a wide range of noncovalent surface modification techniques can be employed to modify the structure of CNC by introducing specific functional groups.

The sensitivity of nanocomposites to variations in temperature is primarily determined by either the lower critical solution temperature (LCST) or the upper critical solution temperature (UCST), which is referred to as the critical temperature range. Two critical temperatures, known as the lower critical solution temperature (LCST) and the upper critical solution temperature (UCST), have a substantial impact on the phase behavior and miscibility of polymer solutions. These critical temperatures demarcate distinct regions whereby the aforementioned properties exhibit divergent characteristics, depending on whether they are above or below these thresholds. In this regard, Kato et al. developed a new and efficient solution. A mechanically durable and thermoresponsive nanocomposite film is fabricated via a solvent casting technique, employing poly(2-phenylethylmethacrylate)-grafted CNC as the primary material. It is suggested to use nanocomposite films in thermal cutoff safety devices, such as thermal fuses, that need to reduce conductivity below the lower critical solution temperature (LCST) [180]. Specifically, it was discovered that transparency or semi-transparency occurred below the lower critical solution temperature (LCST), but light scattering and consequent opacity were detected above the LCST [181].

The literature has documented that prior to utilizing CNC as a pH-sensitive system, some preparatory procedures must be undertaken to include pH-sensitive connections onto the surface of CNC [182,183]. The use of CNC in intrinsically pH-sensitive polymers is a method commonly applied for the synthesis of pH-sensitive CNC nanocomposites [184]. The primary reaction of hydrogels to diverse external stimuli is swelling and deswelling, which is caused by the creation of hydrogen bonds between water molecules and the hydrophilic components of the hydrogel. Targeted drug release can be facilitated by adding diverse functional groups to the surface of CNC to cause specific gelation characteristics in response to the pH gradient within the human body [185]. For example, the pore size of pH-responsive CNC hydrogels decreases in acidic settings and increases in alkaline conditions as the hydrogel’s pH changes.

7. Challenges of using CNC and recommendations

This review provides an in-depth analysis of recent advancements in the synthesis, modification, and utilization of nanocellulose, and cellulose nanocrystals in particular, as active components in a wide variety of novel and fascinating applications. These findings have the potential to act as a driving force behind additional research in this field. In particular, it is necessary to address certain challenges, particularly in the areas of surface and end-reducing modifications, the scaling up of production, and the methods and techniques that are used to describe cellulose nanocrystalline (CNC). Over the last decade, a multitude of research and review articles have been written on this subject, and an abundance of methods and tactics for the characterization of CNC have been developed and published. For cellulose nanocrystalline (CNC), these characterization approaches yield information on various aspects, including their size (length, diameter, and aspect ratio), shape, surface charge, surface chemistry, crystallinity, mechanical qualities, and rheological features. NC properties and, by extension, characterization methodologies are significantly influenced by several factors, including the production method (mechanical, chemical, or biological), the cellulose source material (wood, pulp, recycled paper, bamboo, agricultural wastes, cotton, or other biomass sources), and the final use.

The realistic transfer of lab-scale manufacturing to industrial or commercial scale, the achievement of material feasibility, and the introduction of these products to the market are all areas that require additional research to address the existing knowledge gaps. These include, but are not limited to, the optimization of the entire process, the development of new techniques to make new materials based on NC, and the reduction of the amount of energy and time that is consumed by NC-based products.

In industrial contexts, there are currently no technologies that can provide fast and accurate characterization, making it impossible to guarantee CNC quality and reliability. Additionally, the methods of nano-characterization that are now in use have some restrictions and downsides that must be considered. These approaches are dependent on off-line measurements that are carried out in laboratories. These measurements require a significant amount of post-processing and analysis, in addition to a substantial financial commitment and personnel that have received intensive training. On top of that, monitoring and control routines are unable to be applied in CNC production processes due to the significant delay in information collection that is brought about by the absence of real-time characterization methods. This section provides an overview of the most significant limitations and flaws associated with the CNC category in question.

8. Conclusion

Today’s scientists, researchers, and academics are mostly focused on creating eco-friendly and bio-based nanomaterials to shield the planet from negative impacts. Derived from several lignocellulosic fiber types, nanocellulose is regarded as a sustainable and adaptable resource. It can be applied as a reinforcing material in a variety of industries, including food packaging, printed electronics, paper manufacture, and structural applications. This review article’s objective is to give readers a full grasp of nanocellulose, in particular cellulose nanocrystals (CNCs), as well as the major properties of CNCs and prospective applications for these qualities. The following are the main findings from this review:

Because they contain large levels of noncellulosic components, including hemicellulose, lignin, and waxes, raw lignocellulosic fibers are not appropriate for use as reinforcement in a variety of applications. To effectively remove these noncellulosic elements from biomass and improve the physical structure of nanocellulose, chemical pretreatment is essential.

The conventional method of separating CNCs, mineral acid hydrolysis, has a number of drawbacks, including poor thermal stability of the CNCs, excessive water use for neutralization, and environmental problems. For the time being, researchers are focusing their efforts on organic acid hydrolysis and mixed acid hydrolysis, both of which involve the utilization of only a minute quantity of mineral acids. Organic acid hydrolysis is one of these techniques; it yields CNCs with better colloidal stability, less equipment corrosion, excellent thermal stability, and acid reusability.

The need for environmentally sustainable materials has led researchers worldwide to investigate nanocellulose materials and their potential uses in a range of industries. Studying the surface and morphological behavior of these materials is crucial to understanding them completely and making appropriate use of them. This article serves as a useful resource for aspiring CNC researchers by highlighting significant characterization approaches used to analyze the microstructure of CNCs.

There is only a limited application of bio-based products for sustainable purposes. Researchers, scientists, and academics must have a broad perspective in order to create environmentally acceptable nanocellulose composites that do not harm ecosystems or the environment. The usage of CNCs in a variety of bio-based goods is examined in this article, along with their potential application in the creation of ecologically friendly products in the future.

In conclusion, this review paper offers a thorough overview of the methods used to process nanocellulose and is an invaluable resource for long-term expansion in the manufacturing of high-grade nanocomposite materials. Presently, governments and enterprises across the globe are allocating substantial financial resources to scientists and researchers working on nanotechnology applications in order to boost productivity and encourage environmental sustainability. The study concludes that research on nanotechnology is growing rapidly and has a lot of potential to support commercial and sustainable uses in the future.

Conflict of interest: The authors declare no conflict of interest.

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