

Cellulose Nanocrystals and Nanofibers: A Review on Synthesis, Characterization, and Military Applications

Waleed M. Gebreel¹, Ahmed M. Maraden¹ and Hosam E. Mostafa¹

¹ Department of Chemical Engineering, Military Technical College (MTC), Cairo, Egypt.

Ahmedmaraden@yahoo.com

Abstract. Cellulose nanocrystals and cellulose nanofibers are emerging nanomaterials derived from renewable sources, exhibiting remarkable mechanical strength, high crystallinity, and biocompatibility. This review provides comprehensive insights into their synthesis, characterization, and potential applications, with an emphasis on military and advanced technological uses. Cellulose nanocrystals are primarily obtained through acid hydrolysis, whereas cellulose nanofibers are produced via mechanical disintegration techniques. Various pretreatment strategies, including alkaline and bleaching treatments, enhance extraction efficiency and improve the overall properties of these nanomaterials. Significant insights into surface chemistry, crystallinity, and shape can be obtained through techniques such as X-ray diffraction, transmission electron microscopy, scanning electron microscopy and Fourier transform infrared spectroscopy. Cellulose nanocrystals and cellulose nanofibers exhibit excellent reinforcing properties in polymer composites, making them suitable for defense applications such as light weight armor, ballistic resistant materials, and flame-retardant coatings. Despite their promising attributes, challenges remain in large-scale production and surface functionalization. This review highlights the significance of cellulose nanocrystals and cellulose nanofibers in advancing sustainable, high performance materials.

Keywords: Cellulose nanocrystals, Cellulose nanofibers, Acid hydrolysis, Mechanical disintegration and Military applications.

1. Introduction

The growing emphasis on renewable and sustainable materials, such as cellulose, starch, and gelatin, arises from their potential to address critical global challenges, including natural resource depletion, energy crises, and environmental pollution. Figure 1 illustrates that cellulose is the most abundant of these biopolymers, consisting of β -D-glucose monomers linked by β -(1, 4)-glycosidic bonds. Its molecular structure features alternating crystalline and amorphous regions, which play a crucial role in its fibrous morphology and broad applicability across multiple fields. [1]. Cellulose, primarily sourced from lignocellulosic biomass including forest residues, agricultural waste, and energy crops is a renewable, biodegradable, and biocompatible material. These properties make it indispensable across various industries, such as textiles, paper

manufacturing, cosmetics, and pharmaceuticals, with an estimated global production of 1.3×10^{10} metric tons annually.[2] .

A major breakthrough in cellulose-based research is the development of nanocellulose (NC), a nanoscale derivative with outstanding mechanical properties, a high surface area, abundant hydroxyl functional groups, and enhanced environmental sustainability. NC is classified into two distinct forms: cellulose nanocrystals (CNCs) and cellulose nanofibers (CNFs), which differ in structural characteristics, physicochemical properties, and extraction techniques. Due to its exceptional attributes, NC has attracted considerable interest in both scientific research and industrial applications.[3] .

This review examines recent advancements in the development of CNFs and CNCs, focusing on pretreatment strategies, synthesis methods, processing techniques, and characterization approaches. Additionally, it explores their emerging military applications in advanced technologies.

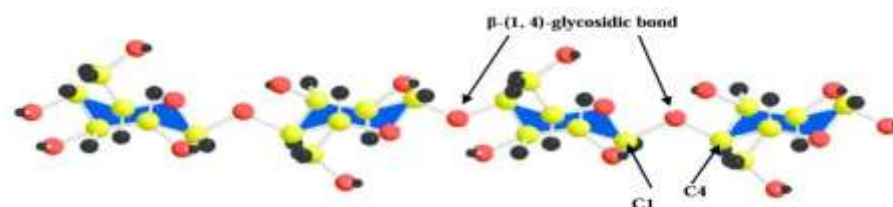


Figure 1. The structure of a cellulose molecule[1] .

2. Cellulose Nanocrystals: from plant cell walls to nanomaterials

Cellulose naturally exists as slender, thread-like microfibrils, which are structural components of plant cell walls. These microfibrils are composed of cellulose crystals linked by amorphous regions. The highly ordered crystalline domains can be extracted through acid hydrolysis, resulting in nanoscale particles known as cellulose nanocrystals (CNCs), as shown in Figure 2. Over time, various terms have been used to describe this material, such as cellulose nanowhiskers and nanocrystalline cellulose. However, in 2011, TAPPI's International Nanotechnology Division standardized the terminology, designating CNCs as the most appropriate term for this nanomaterial.[4] .

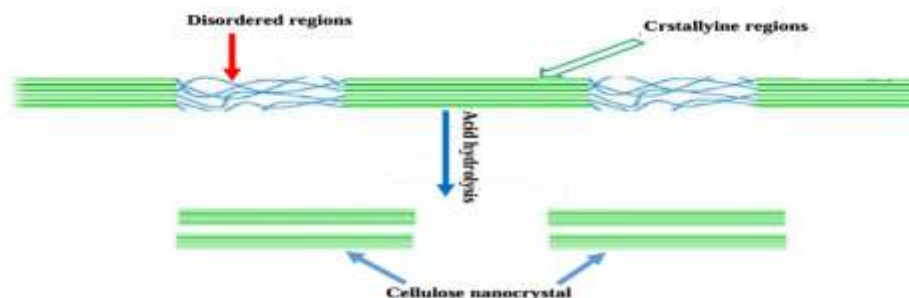


Figure 2. Schematic of a cellulose microfibril's Crystalline and amorphous regions [5] .

2.1 Synthesis of CNCs

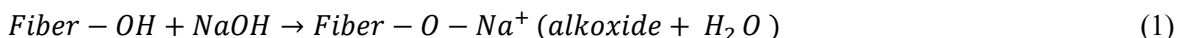
Typically, cellulose nanocrystals (CNCs) are extracted using top-down approaches, including chemical, physical, or enzymatic techniques. Both the source material and the extraction method significantly influence the properties of CNCs. The extraction process generally comprises two main stages. The first stage, pretreatment, involves purification, homogenization, or other preparatory treatments such as chemical or enzymatic methods to enhance yield. The second stage focuses on isolating crystalline nanocellulose from

the pretreated material. While the pretreatment process is determined by the nature of the source material, the isolation process is critical in defining the morphology, and characteristics of the resulting CNCs. Figure 3 illustrates the procedures for CNC synthesis [6].



Figure 3. Process flow for synthesizing CNCs from various sources [6].

2.1.1 Alkaline treatment. Alkaline treatment, or mercerization, is a widely used chemical method in which natural fibers are immersed in sodium hydroxide (NaOH) solutions. This treatment removes amorphous components such as hemicellulose and lignin, increases fiber density, and modifies the fiber surface to improve resin wetting. However, improper optimization of treatment conditions can lead to fiber damage, including defibrillation or pore formation [7]. The alkaline treatment enhances the crystalline cellulose content and improves the mechanical properties of composites by facilitating charge transfer between the fiber and the matrix. The process consists of three key steps: (1) Reagent Selection, with NaOH being the most effective hydroxide for increasing cellulose accessibility; (2) Fiber Immersion, where fibers are soaked in a 12–17.5% NaOH solution at 60–80°C for 2–4 hours to enhance porosity and hydrolysis; and (3) Washing, where residual NaOH and dissolved impurities are removed with boiling water. The overall mechanism disrupts hydrogen bonds in the fiber network, resulting in enhanced structural and functional properties. The mechanism is as follows [8]:



2.1.2 Bleaching treatment. Bleaching is a vital cleaning process that removes chromophores, the color producing structures of stains, which washing alone cannot eliminate. The process involves physical, chemical, or mechanical methods, often combined. Chemical bleaching utilizes oxidizing or reducing agents to break down colored substances, leaving colorless residues that can be rinsed away. Historically, early bleaching agents included wood ash and urine, evolving to modern chemical bleaches like hypochlorite's, peroxides, and photo bleaching agents. Chlorinated bleaches, such as sodium hypochlorite, are effective but have stability and environmental concerns. Oxygenated bleaches like hydrogen peroxide are safer alternatives, decomposing into water and oxygen. Recent advancements include photo bleaching agents (PBAs), which use light to activate oxygen and achieve effective low-temperature bleaching [9].

2.2 Acid Hydrolysis as a Technique for Extracting CNCs

After pretreatment, the fibers undergo a controlled strong acid hydrolysis process. During this procedure, hydronium ions penetrate the cellulose matrix, cleaving glycosidic bonds in both crystalline and amorphous regions, leading to the formation of cellulose crystallites. When the resulting nanoparticles are dispersed in water, they are commonly referred to as CNCs or CNWs, both widely recognized terms for these nanostructures. The following sections provide a detailed discussion of various acid hydrolysis techniques, highlighting their respective advantages and limitation

2.2.1 Sulfuric acid hydrolysis. Rånby (1951) pioneered the use of sulfuric acid hydrolysis to create colloidal suspensions of cellulose nanocrystals (CNCs), a method still favored for its efficiency and stability in laboratory settings. This process selectively hydrolyzes the amorphous regions of cellulose, which contain

structural irregularities, while preserving the crystalline domains. Hydronium ions (H_3O^+) penetrate these amorphous regions, cleaving glycosidic bonds and releasing nanocrystals. The reaction progresses more rapidly in amorphous areas than in crystalline ones.[10]. Figure 4 illustrates the acid hydrolysis mechanism. Optimal CNC production occurs when sulfuric acid concentrations range from 63 to 64 wt%, with temperatures maintained between 45°C and 60°C for 30 to 120 minutes. Higher acid concentrations risk degrading crystalline structures. The process involves controlled hydrolysis, quenching, centrifugation, dialysis, and mechanical processing, such as sonication. Additional treatments, including filtration and ultracentrifugation, help standardize particle size and improve dispersion uniformity[4].

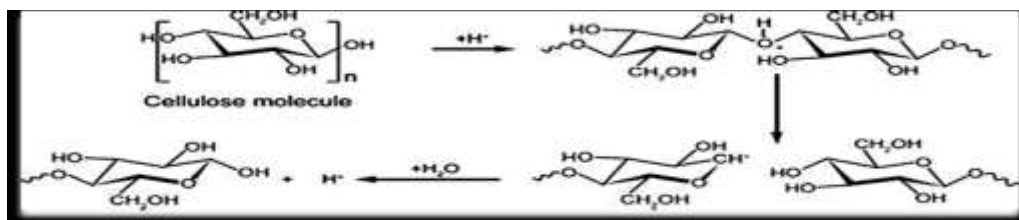


Figure 4. Diagram depicting the mechanism of the acid hydrolysis reaction [10].

2.2.2 Alternative Acid Hydrolysis Techniques for CNC Extraction. Beyond sulfuric acid hydrolysis, hydrochloric (HCl) and phosphoric acid hydrolysis are widely employed techniques for cellulose nanocrystal (CNC) extraction. HCl hydrolysis necessitates extended reaction times (2–4 hours) and elevated temperatures (60°C–105°C), with an optimal acid concentration of 2.5–6.0 N to enhance CNC yield. This process produces highly crystalline α -cellulose, yielding rod-like CNCs (10–12 nm in diameter, 285–304 nm in length) with improved thermal stability but a tendency to aggregate due to insufficient surface charge. [11]. In contrast, phosphoric acid hydrolysis, conducted at 100°C–120°C with 70%–75% acid concentrations for 80–120 minutes, efficiently isolates CNCs with diameters of 15–32 nm and lengths of 238–475 nm. Notably, phosphoric acid-derived CNCs exhibit enhanced thermal stability and flame resistance, making them particularly suitable for biomedical applications, such as bone scaffolding. However, the low zeta potential of these CNCs limits their colloidal stability. Despite these challenges, both methods provide CNCs with distinct physicochemical properties, broadening their applicability in industrial and biomedical fields. [12].

2.3 Structure and Morphology of CNCs

Cellulose nanocrystals derived from cotton cellulose through acid hydrolysis followed by freeze-drying display distinct morphological types, including rod-like, spherical, and porous lattice structures. Acid hydrolysis at 45°C produced crystalline cellulose products exhibiting these three predominant morphological forms: rods (Figure 5a), spheres (Figure 5b), and a porous network (Figure 5c). Among these morphologies, the spherical form was the most prevalent, while the porous network appeared in the least quantity [13].

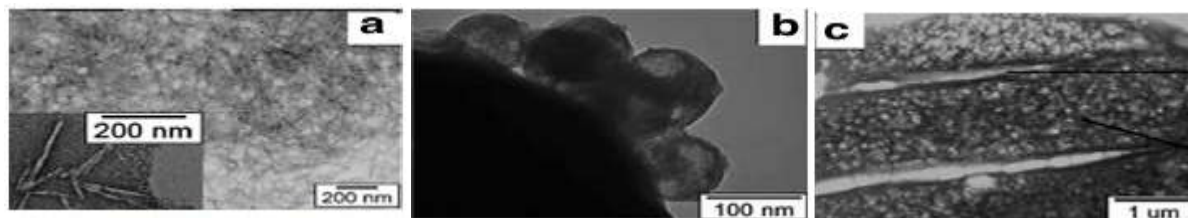


Figure 5. TEM micrographs illustrating the morphological diversity of CNCs, depicted as (a) rod-shaped structures, (b) spherical particles, and (c) a porous network [13].

2.4 Dimension of CNCs

The geometric characteristics of CNCs, including their length and diameter, are influenced by various factors, such as the origin of the cellulose microfibrils and the conditions employed during acid hydrolysis (e.g., duration, temperature, and purity). Due to the diffusion-controlled nature of the hydrolysis process, CNCs exhibit a broad distribution in both length and diameter. A summary of CNC dimensions from different sources and preparation methods is provided in Table 1. [14].

Table 1. A summary of the dimensions of CNCs, categorized by source and preparation method [14].

Cellulose source	Hydrolysis method	Length (nm)	Width (nm)	Aspect ratio (L/D)
Wood	H ₂ SO ₄ hydrolysis	100-300	3-5	20-100
Cotton	H ₂ SO ₄ hydrolysis	100-150	5-10	10-30
Ramie	H ₂ SO ₄ hydrolysis	70-200	5-15	~12
Sisal	H ₂ SO ₄ hydrolysis	100-300	3-5	~60

3. Cellulose Nanofibers (CNFs): production and properties

Cellulose nanofibers, a novel cellulosic material, were first introduced by Turbak and Herrick [15]. They successfully produced cellulose with nanoscale lateral dimensions by repeatedly passing a softwood pulp aqueous suspension through a high-pressure homogenizer. Due to intense shearing forces, this mechanical treatment generates highly entangled nanofibril networks containing crystalline and amorphous regions. CNFs exhibit a high aspect ratio and form hydrogels in water, displaying shear-thinning and thixotropic behavior. The degree of fiber disintegration depends on processing conditions, yielding flexible CNFs with lateral dimensions ranging from approximately 5 nm, corresponding to elementary fibrils, to tens of nanometers, representing individual microfibrils and their aggregates. Typically, CNFs have diameters between 5 and 50 nm and lengths in the micrometer range. A comprehensive review of CNF production methods and their properties is provided in Section 3.2.

3.1 Production of CNF

Plants primarily consist of cellulose, lignin, pectin, hemicellulose, pigments, and other secondary components. Cellulose chains are organized into microfibrils, which are aggregates of polysaccharides further assembled into fibrils, ultimately forming the plant cell wall. This hierarchical structure not only enhances the mechanical stability of plant structures but also highlights the exceptional strength and advantageous mechanical properties of cellulose as a biomaterial.

Wood-derived microfibrils contain both crystalline and amorphous regions. Under strong acidic treatment, the amorphous regions undergo dissolution or degradation, resulting in the release of CNFs. The extraction of CNF nanocellulose generally follows a three-stage process: pretreatment, extraction, and purification. A crucial step in achieving efficient fiber separation and cellulose nanofibril production, while simultaneously reducing energy consumption, is the removal of lignin, extractives, and hemicellulose from the cell wall of lignocellulosic materials derived from both wood and non-wood sources. Various techniques, including alkali treatment, oxidation, and bleaching, have been extensively documented as effective methods for this purpose.

3.1.1 Pretreatment strategies for cellulose nanofiber extraction. Pretreatment is a crucial step in the extraction of CNFs, as it facilitates subsequent processing stages. Since nanocellulose extraction is a multistep process, the chosen method influences the characteristics of the resulting CNFs. To obtain high-quality CNFs, non-cellulosic components such as lignin and hemicellulose must be removed from lignocellulosic biomass, typically through alkali or bleaching treatments.

Alkaline pretreatment with NaOH or KOH is widely used to eliminate lignin and other impurities. This process disrupts acetyl groups in hemicellulose and lignin-carbohydrate ester linkages, thereby solubilizing lignin while preserving its aromatic structure. The cellulose purification process involves neutralization, filtration, and rinsing. Caustic soda treatments under controlled conditions (8–16% concentration, 90–160°C temperature, 1–2 h duration) effectively prepare the raw material for cellulose separation.

Bleaching, an alternative purification method, isolates pure cellulose through acid-chlorite treatment. Lignocellulosic biomass is treated with a mixture of glacial acetic acid, hot distilled water, and sodium chlorite under continuous stirring for 4 hours. Once the pH is adjusted to 7 and lignin is removed, the material is thoroughly rinsed with distilled water. For "holocellulose" production, the purified residues are dried at ambient or elevated temperatures[16] .

3.2 Mechanical disintegration

Mechanical methods can be employed to disintegrate dry cellulose pulp into smaller fragments. However, these approaches primarily result in fiber shredding rather than the delamination of elementary fibrils. Consequently, the obtained CNF exhibit a low degree of polymerization, crystallinity, and aspect ratio, which adversely affects the mechanical properties of the resulting nanomaterials. Among the various mechanical techniques for CNF production, homogenization (using homogenizers and micro fluidizers), grinding, and ball milling are the most widely utilized. These methods are particularly effective in facilitating fiber cell wall delamination and CNF isolation while also being scalable for industrial applications. As a result, they are currently the preferred approaches for large-scale CNF production [17].

3.2.1 Homogenization. High-pressure homogenization is an efficient, solvent-free technique for producing CNFs. In this process, pretreated cellulose pulp, diluted in distilled water, is injected through a micron-sized gap under high pressure (69–207 MPa), generating shear forces that promote fibrillation. This method yields CNFs with uniform crystalline size and strength, making it suitable for industrial applications. However, its high energy consumption and the requirement for multiple passes estimated at 7.5 kWh/g for 30 passes pose challenges for large-scale production [18].

3.2.2 Grinding. Is a straightforward, energy-efficient, and environmentally sustainable mechanical method for producing CNFs, making it well-suited for large-scale industrial applications. In this process, pretreated cellulose fibers (1 wt %) are dispersed in water to form a slurry, which is then passed between the grinding stones of a shear grinder operating at 1500 rpm. Defibrillation is conducted through multiple passes under consistent conditions until a gel-like nanocellulose suspension is obtained. This wet grinding technique yields CNFs with a uniform width of approximately 15 nm. However, the intense mechanical forces applied during grinding disrupt the cellulose crystalline regions, resulting in CNFs with reduced crystallinity and mechanical strength [18, 19].

3.2.3 Ball milling. Ball milling has been a widely used technique for material grinding and particle refinement since the 1990s. It involves milling balls of varying sizes within a rotating jar, facilitating material breakdown through collisions and friction, as shown in Fig 6. Several types of ball mills, including planetary, mixer, and vibration mills, are commonly employed in both industrial and laboratory settings. Among these, the planetary ball mill is the most effective for cellulose and biomass defibrillation. The grinding efficiency depends on factors such as ball size and quantity, milling speed, dry or wet conditions, the ball-to-material weight ratio, and milling duration. These variables significantly influence the final particle size and surface characteristics of the milled material[20].

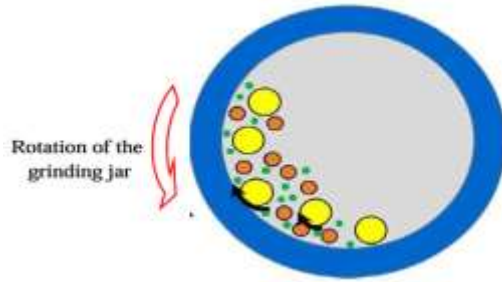


Figure 6. Diagram of the Planetary Ball Milling Process[20].

3.3 Characterization methods of CNFs and CNCs

3.3.1 X-ray powder diffraction (XRD) analysis. Is a non-destructive analytical technique widely used to evaluate the crystalline and amorphous regions of cellulose fibers. The diffraction peaks obtained from the XRD spectrum are characteristic of the fiber being analyzed, providing a crystallinity index (CI), which is calculated using Segal's method (Equation 2)[21].

$$I_C [\%] = \frac{I_{(crys+am)} - I_{(am)}}{I_{(crys+amm)}} \quad (2)$$

Where the term $I_{(crys+am)}$ denotes the peak intensity (in counts per second) observed around $2\theta = 22.80^\circ$, corresponding to both the crystalline and amorphous regions of the cellulose. In contrast, $I_{(am)}$ refers to the peak intensity around $2\theta = 18.0^\circ$, representing the amorphous fraction of the cellulose.

The study by Narsimha Pandi et al [22]. Investigates the effect of ultrasound-assisted acid hydrolysis on the structural properties of cellulose during the synthesis of cellulose nanocrystals (CNCs) from cotton. XRD analysis confirms the transformation of cellulose into CNCs, as evidenced by the emergence of distinct diffraction peaks at $2\theta = 14.65^\circ$, 16.74° , 22.58° , and 34.55° , which are characteristic of CNCs. These peaks correspond to the $(1 \bar{1} 0)$, (110) , (200) , and (004) crystallographic planes, indicating a highly ordered crystalline structure. The increase in crystallinity is attributed to the removal of amorphous regions via acid hydrolysis and ultrasound treatment. In contrast, untreated cellulose exhibits a broad and diffuse XRD pattern, indicating lower crystallinity and a predominantly amorphous structure. This pronounced difference in diffraction patterns highlights the effectiveness of the treatment process in selectively removing disordered regions while preserving the crystalline domains of CNCs (Figure 7).

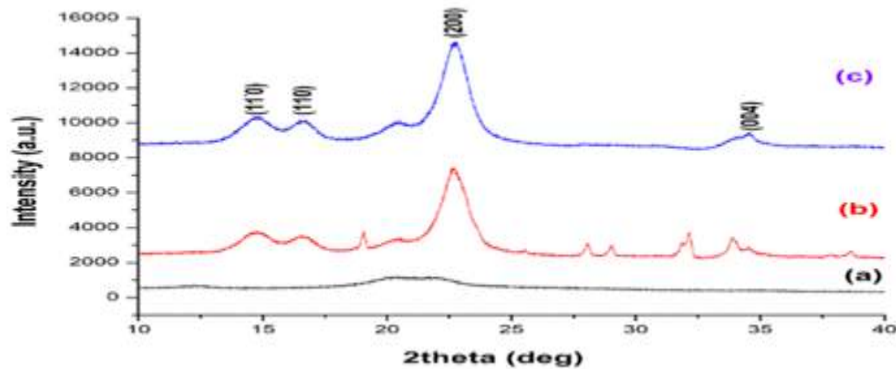


Figure 7. XRD patterns of (a) raw cotton, (b) cellulose nanocrystals (CNCs) prepared using a 30% acid concentration, and (c) CNCs prepared using a 50% acid concentration[22].

3.3.2 Scanning electron Microscope (SEM) analysis. Is an effective tool for analyzing the surface topography of cellulose-based nanocomposites at the micro- and nanoscale. This technique allows for a detailed assessment of nanofiber dimensions, shapes, orientations, and their interactions with the matrix. SEM is particularly valuable for characterizing the structural features of nanocomposite materials [21].

3.3.3 Transmission electron microscope (TEM) analysis. Is extensively utilized for morphological characterization, encompassing the analysis of size, shape, and structural properties. It provides exceptional throughput, making it highly advantageous for the rapid analysis of a large volume of nanoparticles. TEM is particularly proficient in characterizing nanoparticles within the size range of 10 nm to 1 μm [3].

3.3.4 Fourier Transform-Infrared Spectroscopy (FTIR). Is a well-established non-destructive analytical technique widely used to obtain both quantitative and qualitative information on natural fibers. It is also extensively applied in the characterization of CNCs and CNFs, providing crucial insights into their chemical structure and functional groups. The infrared spectra of natural fibers are typically observed within the frequency range of 400 to 4000 cm^{-1} [3].

Narsimha Pandi et al [22]. Synthesized CNCs from cotton using ultrasound-assisted acid hydrolysis and characterized the functional groups of CNCs via FTIR. The FTIR spectra of CNCs showed characteristic peaks at 3417 cm^{-1} (O–H stretching), 2902 cm^{-1} (C–H stretching), 1644 cm^{-1} (C–O stretching), and others, indicating the presence of key cellulose structural features. Compared to raw cotton, CNCs exhibited reduced peaks associated with non-cellulosic components, confirming the successful removal of amorphous regions and an increase in crystallinity. FTIR analysis thus confirms that ultrasound-assisted acid hydrolysis preserved essential functional groups while enhancing the crystallinity of CNCs. (Figure 8).

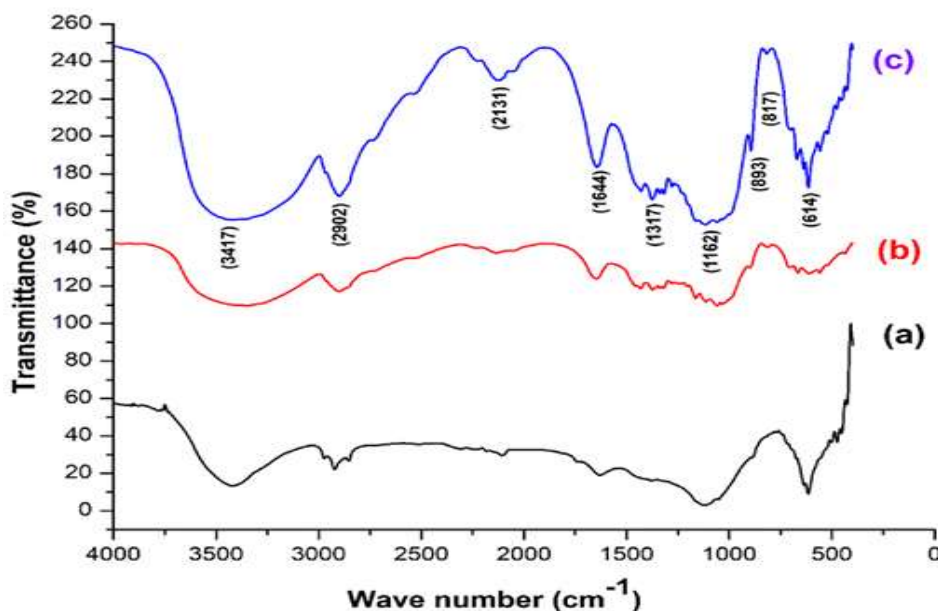


Figure 8. FT-IR Characterization of (a) Raw Cotton, (b) Cellulose Nanocrystals (CNCs) Extracted Using a 30% Acid Concentration, and (c) CNCs Extracted Using a 50% Acid Concentration.[22].

4. Military Applications of CNFs and CNCs

4.1 Military packaging.

Military packaging requires materials with superior mechanical strength, water resistance, and barrier properties to ensure the long-term preservation and protection of critical equipment. Unlike commercial packaging, military packaging must withstand extreme transportation and storage conditions. Nanocellulose has emerged as a promising material for such applications due to its exceptional mechanical properties and functional versatility.

Nanocellulose has potential applications across various military packaging categories, including bags, chipboard cartons, food packaging, cushioning, fast packs, tapes, and wraps. However, its full potential remains underexplored. The development of standardized military transport packaging arose from historical logistical inefficiencies, ensuring optimal protection, combat readiness, and sustained equipment performance. Consequently, ongoing research and development efforts are focused on advancing military packaging materials, with nanocellulose playing a pivotal role.

Industries have already integrated nanocellulose into packaging, as exemplified by Unisource's bamboo-nanocellulose packaging for electronic devices [23]. These materials align with military standards by offering enhanced protection, multi functionality, and environmental sustainability. However, the inherent brittleness and high moisture absorption of nanocellulose paper films limit their direct application in military settings. To address these challenges, multi layering strategies incorporating waxes, high-aspect-ratio nanoclays, and surface-active compounds have been explored. Additionally, functionalization with titanium dioxide and stearyl chloride has rendered nanocellulose super hydrophobic, significantly reducing its moisture absorption.

Biopolymer-based packaging materials such as poly lactic acid (PLA), starch, and poly hydroxyal kanoates offer biodegradability and environmental benefits but exhibit limitations in mechanical strength and thermal stability. The incorporation of nanocellulose into these biopolymers has significantly improved their structural integrity. Studies have demonstrated that nanocellulose enhances the thermal stability of PLA, while CNCs improve the rigidity, thermal stability, and moisture resistance of starch-based films, making them more viable for military packaging applications [24].

4.2 Military textiles.

Textiles are integral to military equipment, serving a wide range of functions across various applications. In military operations, textiles are employed in essential gear such as combat uniforms, tents, shelters, parachutes, body armor, and bomb suits for explosive ordnance disposal. These materials play a crucial role in enhancing operational efficiency, protection, and overall mission effectiveness.

The textile industry continues to advance by integrating innovative technologies to develop high-performance fabrics with specialized properties, including antimicrobial activity, fire resistance, self-cleaning capabilities, and enhanced water and air permeability. While these advancements benefit both commercial and military applications, military textiles are specifically engineered to prioritize hydrophobicity, high mechanical strength, and fire-retardant properties, ensuring enhanced durability and protection in extreme conditions.

Nanocellulose, recognized for its versatile and tunable characteristics, has emerged as a promising material for military textile applications, particularly in uniform development. According to the National Research Council, materials intended for military use must meet rigorous performance standards, including cost efficiency, high strength, durability, operational effectiveness, environmental sustainability, precision, lightweight properties, and seamless integration with existing military systems. Nanocellulose has been identified as a promising alternative to conventional materials such as Kevlar, offering significant advantages for body armor fabrication [24].

4.3 Nano cellulose-modified propellants

Energetic compounds serve as the primary active chemical components of explosives and propellants, playing a crucial role in both commercial applications (e.g., demolition and mining) and military operations. Propellants are materials capable of generating a substantial volume of gaseous molecules at high temperatures during combustion while sustaining the reaction independently, without requiring an external oxidizer. Significant research and development efforts have been directed toward exploring nanocellulose as a potential propellant. For instance, Jamal et al. [25]. Synthesized nitrocellulose from bacterial nanocellulose (BNC) through a nitration process for application as a gun propellant. Their study demonstrated that a reaction time of 60 minutes was optimal for achieving a high nitrogen content, as confirmed through material characterization. Furthermore, K. Okada et al.[26] . Developed nitrocellulose nanofiber (NCNF) via the esterification of CNF in a water suspension derived from disk-milled cotton powder. Their findings indicated that NCNF exhibited a specific surface area ten times greater than that of conventional nitrocellulose (NC) and a burning rate 3.5 times higher, highlighting its potential for enhanced propellant performance.

4.4 Fire-Resistant Materials.

Nanocellulose-based composites contribute to improved fire resistance in various military applications, including protective gear such as uniforms, helmets, and vehicle components. These materials effectively minimize smoke generation and toxic gas emissions during combustion, enhancing safety in hazardous environments [24].

4.5 Protective Masks.

The rapid advancement of filtration materials has significantly influenced water and air treatment technologies, particularly in commercial and military sectors. In commercial applications, these materials are widely used in respiratory protection devices, such as N95, N99, and N100 masks, surgical masks, household air filters, and water purification systems. Military applications, on the other hand, integrate filtration materials into protective masks, filter cartridges, portable water filtration units, and vehicle air filtration systems to protect personnel from hazardous biological and chemical agents.

Beyond these applications, ensuring access to clean water remains a global challenge, as contaminants such as heavy metals, organic and inorganic compounds, and microbial agents threaten environmental and operational stability. To address this, researchers are exploring advanced filtration strategies that integrate biosorption and nanotechnology. In particular, nanocellulose has emerged as a highly effective material due to its high surface area and hydroxyl-rich structure, which enhance contaminant removal. Its adaptability and superior adsorption capacity make it a promising solution for both military and commercial filtration needs.[23]

4.6. Energy Storage Devices.

The increasing global demand for energy, driven by rapid economic growth and technological advancements, necessitates continuous innovations in energy storage and generation. The military sector, in particular, requires advanced energy solutions for various applications, including surveillance, communication, and power management in warfighting systems. Conventional energy devices, typically composed of plastics, inorganic semiconductors, and petrochemical-based materials, pose significant environmental concerns due to their non-biodegradability. Consequently, researchers are actively exploring sustainable alternatives.

Nanocellulose has emerged as a promising material for electrochemical energy storage (EES) applications, including military technologies. Its exceptional mechanical properties allow integration into polymer electrolytes, enhancing ion transport and electrode cycling performance. However, challenges related to the

dispersion and conductivity of nanocellulose necessitate functionalization with conductive polymers, metallic particles, or carbon-based materials such as graphene and carbon nanotubes. Functionalization can be achieved through either coating nanocellulose with conductive materials or direct mixing via in situ polymerization and blending. The development of nanocellulose-based energy materials represents a crucial step toward environmentally friendly and high-performance energy solutions for modern technological and military applications [24].

5. Conclusion

This review article provides a comprehensive understanding of the fundamental principles and properties of nanocellulose materials, with a particular focus on CNCs and CNFs. Lignocellulosic fibers inherently contain significant amounts of non-cellulosic substances, including hemicellulose, lignin, and wax, which hinder their direct utilization as reinforcement materials in various applications. To address this limitation, biomass chemical pretreatment is essential for the effective removal of these non-cellulosic components, thereby improving the structural integrity and yield of nanocellulose. CNFs are predominantly obtained through mechanical processing methods, among which ball milling stands out as an efficient, economical, and scalable approach for producing substantial quantities of CNFs from pretreated fibers. Meanwhile, acid hydrolysis remains one of the most widely adopted techniques for extracting CNCs from cellulose-rich sources, as it selectively degrades the amorphous regions of cellulose while preserving the crystalline domains, resulting in CNCs with high crystallinity. This review offers an in-depth analysis of nanocellulose processing methods, serving as a crucial resource for advancing sustainable and high-performance nano-based composite materials. Additionally, the promising role of nanocellulose in military applications continues to be an area of growing interest, with recent studies highlighting its potential integration into defense-related technologies.

References

- [1] J Huang , A Dufresne and Lin N 2019 Nanocellulose: from fundamentals to advanced materials: J. Wiley & Sons
- [2] P. Kaur, N. Sharma ,M. Munagala, R. Rajkhowa, B Aallardyce, Y Shastri and R Agrawal 2021 Nanocellulose: Resources, physio-chemical properties, current uses and future applications *Frontiers in Nanotechnology* 3 747329
- [3] E Pinto, W N Aggrey, P Boakye, G Amenuvor, Sokama-Neuyam Y A, M K Fokuo, H Karimaie, K Sarkodie, Adenutsi C D and S Erzuah 2022 Cellulose processing from biomass and its derivatization into carboxymethylcellulose: A review *Sci. Afr.* 15 e01078
- [4] W P F Neto 2017 Morphological investigation of cellulose nanocrystals and nanocomposite applications. Université Grenoble Alpes; Universidade Federal de Uberlândia)
- [5] T Zhong, G Jian, Z Chen, M Wolcott, S and C A Fernandez 2022 Interfacial interactions and reinforcing mechanisms of cellulose and chitin nanomaterials and starch derivatives for cement and concrete strength and durability enhancement: A review *Nanotechnol. Rev* 11 2673-713
- [6] T Appidi, M V Sushma and A K Rengan 2022 *Handbook of Nanocelluloses: Classification, Properties, Fabrication, and Emerging Applications*: Springer) pp 1-31
- [7] W Ouarhim, N Zari and R Bouhfid 2019 Mechanical and physical testing of biocomposites, fibre-reinforced composites and hybrid composites: Elsevier) pp 43-60
- [8] D A Gopakumar, D Pasquini, H Abdul Khalil and S Thomas 2021 Functional Nanocellulose Aerogels for Varying Technological Applications *Nanocellulose: Synthesis, Structure, Properties and Applications* 449 80
- [9] S Dixit 2002 Bleaching Agents: Chemicals that Ensure Stain-Free Clothes *Chem. Weekly-Bombay-* 47 183 202
- [10] A Dufresne 2017 Nanocellulose: from nature to high performance tailored materials: De Gruyter
- [11] N Hastuti, K Kanomata and T Kitaoka 2018 Hydrochloric acid hydrolysis of pulps from oil palm empty fruit bunches to produce cellulose nanocrystals *J. Polym. Environ* 26 3698-709

- [12] S Ahsan and M Rabbi 2024 Recent advancements in nanocellulose synthesis, characterization, and application: A review JPSE 7 4496
- [13] P Lu and Y-L Hsieh 2010 Preparation and properties of cellulose nanocrystals: rods, spheres, and network Carbohydr. Polym. 82 329-36
- [14] J George and S Sabapathi 2015 Cellulose nanocrystals: synthesis, functional properties, and applications Nanotechnol. Sci. Appl 45-54
- [15] A F Turbak, F W Snyder and K R Sandberg 1983 Microfibrillated cellulose, a new cellulose product: properties, uses, and commercial potential. In: J. Appl. Polym. Sci, pp 815-27
- [16] A Meftahi, M E Momeni Heravi, A Barhoum, P Samyn, H Najarzadeh and S Alibakhshi 2022 Cellulose nanofibers: synthesis, unique properties, and emerging applications Handbook of Nanocelluloses: Classification, Properties, Fabrication, and Emerging Applications 233-62
- [17] O Nechyporchuk, M N Belgacem and J Bras 2016 Production of cellulose nanofibrils: A review of recent advances Ind. Crops Prod 93 2-25
- [18] K Nagarajan, N Ramanujam, M Sanjay, S Siengchin, B Surya Rajan, K Sathick Basha, P Madhu and G Raghav 2021 A comprehensive review on cellulose nanocrystals and cellulose nanofibers: Pretreatment, preparation, and characterization Polym. Compos 42 1588-630
- [19] W Chen, K Abe, K Uetani, H Yu, Y Liu and H Yano 2014 Individual cotton cellulose nanofibers: pretreatment and fibrillation technique Cellulose 21 1517-28
- [20] P Phanthong, P Reubroycharoen, X Hao, G Xu, A Abudula and G Guan 2018 Nanocellulose: Extraction and application Carbon Resources Conversion 1 32-43
- [21] E J Foster, R J Moon, U P Agarwal, M J Bortner, J Bras, S Camarero-Espinosa, K J Chan, M J Clift, E D Cranston and S J Eichhorn 2018 Current characterization methods for cellulose nanomaterials Chem. Soc. Rev 47 2609-79
- [22] Pandi N, Sonawane S H and Kishore K A 2021 Synthesis of cellulose nanocrystals (CNCs) from cotton using ultrasound-assisted acid hydrolysis Ultrasonics sonochemistry 70 105353
- [23] H A Khalil, Y Davoudpour, C K Saurabh, M S, Adnan A, Dungani R, Paridah M, Sarker M Z I, Fazita M N and Syakir M 2016 A review on nanocellulosic fibres as new material for sustainable packaging: Process and applications Renewable and Sustainable Energy Reviews 64 823-36
- [24] M N F Norrahim, N A M Kasim, V F Knight, F A Ujang, N Janudin, M A I A Razak, N A A Shah, S A M Noor, S H Jamal and K K Ong 2021 Nanocellulose: The next super versatile material for the military Materials Advances 2 1485-506
- [25] Y A Gismatulina 2023 Promising Energetic Polymers from Nanostructured Bacterial Cellulose Polymers 15 2213
- [26] K Okada, Y Saito, M Akiyoshi, T Endo and T Matsunaga 2021 Preparation and characterization of nitrocellulose nanofiber Propellants, Explosives, Pyrotechnics 46 962-8