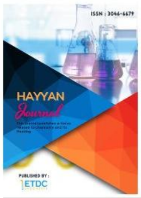

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Synthesis Methods of Nanocellulose from Various Biomass Sources: A Review

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ABSTRACT

Nanocellulose has become a strategic renewable nanomaterial because it combines high specific surface area, tunable surface chemistry, biodegradability, and strong reinforcement ability. This review analyzes synthesis methods of nanocellulose from various biomass sources, particularly agricultural residues, non-wood plants, forestry biomass, and agro-industrial waste. The review focuses on chemical, mechanical, enzymatic, and hybrid extraction strategies, including acid hydrolysis, alkaline and bleaching pretreatment, high-pressure homogenization, ultrasonication, TEMPO-mediated oxidation, deep eutectic solvent pretreatment, ionic liquid-assisted extraction, and bacterial biosynthesis. The synthesis shows that acid hydrolysis remains effective for producing highly crystalline cellulose nanocrystals, while mechanical and enzymatic routes are more relevant for cellulose nanofibrils. Green solvent-assisted and mechano-enzymatic approaches provide better prospects for reducing chemical waste and energy demand, although industrial scalability, product consistency, and techno-economic validation remain major barriers. Biomass composition strongly influences yield, crystallinity, morphology, thermal stability, and dispersibility. Therefore, future nanocellulose production should be designed through feedstock-specific, application-oriented, and environmentally responsible process integration.

Keywords: Nanocellulose, Biomass, Cellulose nanocrystals, Cellulose nanofibrils, Green synthesis

I. INTRODUCTION

The growing demand for sustainable materials has accelerated research on nanocellulose derived from renewable biomass. Nanocellulose is generally obtained from cellulose-rich resources and is valued for its nanoscale dimensions, high aspect ratio, large surface area, mechanical strength, biodegradability, and abundant hydroxyl groups that enable chemical functionalization. These characteristics make nanocellulose attractive for biocomposites, packaging, membranes, biomedical materials, adsorbents, and energy-related devices. Compared with petroleum-derived fillers, nanocellulose offers a pathway for converting agricultural and forestry residues into value-added materials within a circular bioeconomy framework (Jonoobi et al., 2015; Kaur et al., 2021; Norizan et al., 2022).

Nanocellulose is commonly classified into cellulose nanocrystals (CNC), cellulose nanofibrils (CNF), and bacterial nanocellulose (BNC). CNC is typically produced by removing amorphous cellulose domains through acid hydrolysis, producing rigid rod-like particles with relatively high crystallinity. CNF is commonly obtained through mechanical fibrillation, often supported by chemical or enzymatic pretreatment, and forms entangled fibrillar networks with high water retention and film-forming ability. BNC is produced by microbial fermentation and differs from plant-derived nanocellulose because it is naturally secreted in a highly pure nanofibrillar network without lignin and hemicellulose impurities (Rostamabadi et al., 2024; Taokaew, 2024; Vanderfleet & Cranston, 2021).

The choice of biomass source is a critical determinant of synthesis efficiency and final material performance. Agricultural residues such as sugarcane bagasse, rice husk, corn cob, banana pseudostem, pineapple leaves, date palm waste, hemp, barley straw, palm empty fruit bunches, durian husk, coffee parchment, and cashew apple bagasse have been explored as alternatives to wood pulp. These sources differ in cellulose, hemicellulose, lignin, ash, extractives, and silica contents, causing variation in pretreatment severity, fibrillation efficiency, particle size, crystallinity, thermal stability, surface charge, and colloidal stability (Das et al., 2023; Deepa et al., 2015; Gabriel et al., 2021).

Traditional isolation of nanocellulose often involves a sequence of alkali treatment, bleaching, acid hydrolysis, washing, dialysis, and mechanical dispersion. This route is effective but can generate large amounts of acidic and alkaline effluents. Mechanical fibrillation is less chemically intensive but may require high energy input, especially when applied to insufficiently pretreated fibers. Enzymatic methods can reduce fiber recalcitrance under milder conditions, but enzyme cost, reaction time, and process control must be carefully considered. Recent studies therefore increasingly combine chemical, enzymatic, and mechanical routes to improve yield, reduce energy consumption, and tailor the final nanocellulose structure (Bharimalla et al., 2015; Cebreiros et al., 2021; Fang et al., 2024).

Green chemistry approaches have become central in the development of new nanocellulose synthesis routes. Deep eutectic solvents (DES), natural deep eutectic solvents, ionic liquids, recyclable solid acids, organosolv systems, microwave-assisted pretreatment, and one-pot extraction have been proposed to reduce the environmental burden of conventional processes. These routes can promote delignification, cellulose swelling, hydrogen-bond disruption, and improved nanofibrillation while allowing partial solvent recovery. However, green routes still need stronger evidence regarding solvent recyclability, life cycle performance, cost, product safety, and industrial-scale robustness (Liu et al., 2017; Ma et al., 2019; Shamsuri et al., 2022; Wang et al., 2020; Wu et al., 2021).

Despite the large number of studies, there is still no universal synthesis route suitable for all biomass types and applications. Variability in raw material composition, pretreatment conditions, acid concentration, reaction temperature, enzyme dosage, mechanical energy, and washing protocols often causes inconsistent product properties. For this reason, review-based synthesis is necessary to compare routes, identify methodological strengths and weaknesses, and formulate process selection principles. This article reviews synthesis methods of nanocellulose from various biomass sources and discusses their implications for efficiency, physicochemical properties, environmental performance, applications, and future research directions.

II. METHOD

This article was prepared using a literature review approach. The analysis was developed from a SciSpace-assisted review dataset entitled “Synthesis methods of nanocellulose from various biomass sources”. The review question was transformed into several targeted search statements covering chemical, mechanical, enzymatic, bacterial, and hybrid synthesis routes; different biomass sources; CNC, CNF, and BNC products; and comparative indicators such as yield, crystallinity, morphology, dispersibility, energy demand, environmental footprint, and application performance.

The literature selection process used transformed search queries and citation chaining. The initial search retrieved 551 candidate papers, and backward and forward citation chaining added 76 papers, resulting in 627 candidate papers. Relevance assessment identified 611 papers relevant to the topic, including 405 papers categorized as highly relevant. The synthesis in this manuscript prioritizes studies that provide clear information on biomass source, pretreatment strategy, extraction method, product type, physicochemical properties, environmental consideration, and application relevance.

The selected literature was analyzed using thematic synthesis. The first analytical layer grouped studies according to the major synthesis route: acid hydrolysis, chemical pretreatment followed by mechanical fibrillation, enzymatic or mechano-enzymatic treatment, TEMPO-mediated oxidation, DES and ionic liquid-assisted extraction, and bacterial biosynthesis. The second layer compared biomass sources and their influence on nanocellulose yield, crystallinity, morphology, and thermal stability. The third layer identified research gaps related to standardization, scalability, energy consumption, solvent recovery, techno-economic assessment, life cycle assessment, and product safety.

III. RESULTS AND DISCUSSION

3.1. Relationship between biomass composition and nanocellulose properties

The reviewed literature shows that biomass composition determines the intensity of pretreatment and the quality of nanocellulose obtained. Biomass with relatively high cellulose content and lower lignin content is generally easier to delignify and fibrillate. Non-wood and agricultural residues are therefore important candidates because many of them contain accessible cellulose microfibrils and can be obtained as abundant low-cost waste. However, the presence of lignin, hemicellulose, waxes, pectin, ash, silica, and extractives may require stronger alkali treatment, bleaching, oxidation, or solvent pretreatment before nanoscale isolation can occur effectively (Das et al., 2023; Deepa et al., 2015; Jayanthi et al., 2024).

Comparative studies indicate that cellulose crystallinity, particle dimension, and thermal stability are not only determined by the extraction reagent but also by the anatomical structure of the biomass. For instance, residues with high lignin can hinder cellulose accessibility but may also generate lignin-containing nanocellulose with improved hydrophobicity and UV-blocking ability. This creates a trade-off between obtaining purified white nanocellulose and retaining certain lignin fractions for functional applications such as packaging, coating, and composite films (Lamm et al., 2024; Trifol et al., 2021).

3.2. Acid hydrolysis and conventional chemical extraction

Acid hydrolysis remains one of the most widely used approaches for producing CNC. Sulfuric acid hydrolysis is particularly effective because it selectively removes amorphous cellulose domains and introduces sulfate ester groups that improve aqueous colloidal stability. The route can produce CNC with high crystallinity and rod-like morphology, but excessive acid concentration, high temperature, or prolonged reaction time can degrade cellulose and reduce yield. Hydrochloric acid and organic acids are also used, although each acid type affects surface charge, dispersibility, thermal stability, and aggregation behavior differently (Gabriel et al., 2021; Prasannakumar et al., 2022; Vanderfleet & Cranston, 2021).

The conventional chemical route usually begins with alkali treatment to remove hemicellulose, followed by bleaching to remove lignin and residual chromophores. Acid hydrolysis is then applied to obtain CNC, followed by repeated washing, dialysis, centrifugation, or ultrasonication to stabilize the suspension. This approach is experimentally accessible and produces well-defined CNC, but it is associated with corrosive chemicals, wastewater treatment burdens, and potential loss of cellulose during purification. Consequently, recent studies have attempted to reduce acid severity, combine hydrolysis with recyclable catalysts, or integrate chemical treatment with mechanical or enzymatic assistance (An et al., 2016; Cidreira et al., 2024; Ma et al., 2023).

3.3. Mechanical, chemo-mechanical, and enzymatic routes

Mechanical fibrillation methods such as high-pressure homogenization, grinding, microfluidization, and ultrasonication are central to CNF production. These methods break down purified or partially purified fibers into nanoscale fibrils through shear, impact, and cavitation forces. CNF produced through mechanical routes has strong network-forming ability and is suitable for films, hydrogels, coatings, membranes, and reinforcement systems. However, purely mechanical processing can be energy-intensive when fibers have not been sufficiently swollen or delignified (Balea et al., 2021; Kassim et al., 2021; Malucelli et al., 2019).

Chemo-mechanical routes reduce the energy barrier by modifying the fiber before fibrillation. Alkali pretreatment, bleaching, carboxymethylation, TEMPO-mediated oxidation, and mild acid treatment can increase fiber swelling, weaken interfibrillar hydrogen bonding, and improve individualization of fibrils. TEMPO-mediated oxidation is especially important because it introduces carboxylate groups on cellulose surfaces, improving dispersion and enabling high-quality CNF at lower mechanical energy. Nevertheless, oxidation systems require careful control of reagent dosage, pH, residual oxidant removal, and cost-effectiveness (Habibi et al., 2006; Isogai, 2013; Yousefi et al., 2024).

Enzymatic and mechano-enzymatic strategies use cellulases, hemicellulases, or pectinases to weaken fiber walls under milder conditions before mechanical disintegration. These routes can reduce energy demand and avoid harsh chemical degradation. Cebreiros et al. (2021) showed that enzymatic and mechanical pretreatments can enhance eucalyptus kraft pulp nanofibrillation, while Spagnuolo et al. (2024) reported high-yield CNC synthesis through a mechano-enzymatic approach. However, enzyme specificity, enzyme loading, pretreatment time, and process sterilization remain important operational factors.

3.4. Deep eutectic solvent, ionic liquid, and other green-assisted processes

Deep eutectic solvents and ionic liquids are increasingly explored as alternatives to conventional alkaline bleaching and strong acid hydrolysis. DES systems can dissolve or disrupt lignin and hemicellulose, swell cellulose fibers, and facilitate subsequent ultrasonication or homogenization. For example, DES-assisted extraction has been applied to barley straw, durian husk, hemp, coffee parchment, bamboo, and palm bunches, showing potential for improved delignification, fiber accessibility, and nanofibrillation (Baraka et al., 2024; Haque et al., 2025; Lim et al., 2024; Pradhan et al., 2024a; Pradhan et al., 2024b; Sonyeam et al., 2024).

Ionic liquids can disrupt cellulose hydrogen-bond networks and support nanocellulose extraction under milder conditions compared with traditional acid systems. They can be combined with microwave assistance, ultrasonication, or solid acid catalysis to improve process efficiency. Nevertheless, ionic liquid cost, viscosity, purification demand, toxicity profile, and solvent recovery strongly influence industrial feasibility. For this reason, recent research emphasizes low-cost ionic liquid systems, recyclable solid acids, and integrated pretreatment-extraction schemes (Peng et al., 2023; Pradeep & Patel, 2024; Shamsuri et al., 2022).

Green solvent-assisted routes are promising but should not be evaluated only from laboratory yield. Their practical value depends on solvent preparation, recyclability, wastewater load, energy required for solvent removal, equipment compatibility, and product quality after multiple recycling cycles. Studies on DES and ionic liquid processes should therefore include mass balance, solvent recovery efficiency, life cycle assessment, and techno-economic analysis to demonstrate whether the route is genuinely more sustainable than optimized conventional methods (Forssell et al., 2025; Wang et al., 2020; Wu et al., 2021).

3.5. Comparative synthesis routes

Table 1. Comparative synthesis routes for nanocellulose from biomass.

Synthesis route	Main product and feedstock suitability	Key advantages	Main limitations
Acid hydrolysis	Mostly CNC from purified cellulose, agricultural residues, forestry biomass, and non-wood fibers.	Produces high crystallinity and well-defined rod-like particles; widely reported and experimentally simple.	Uses corrosive acids, creates wastewater, may reduce thermal stability or yield if reaction is uncontrolled.
Mechanical fibrillation	Mostly CNF from bleached pulp, non-wood fibers, and chemically pretreated residues.	Avoids strong acid hydrolysis and produces network-forming fibrils for films, hydrogels, and membranes.	High energy demand without sufficient pretreatment; product dimension can be heterogeneous.
Enzymatic or mechano-enzymatic	CNF or CNC from pulp and lignocellulosic fibers after selective enzyme treatment.	Milder conditions, lower chemical burden, and potential energy reduction before fibrillation.	Enzyme cost, reaction time, substrate specificity, and process control can limit scale-up.
TEMPO-mediated oxidation	Highly charged CNF or modified CNC from cellulose-rich feedstocks.	Improves dispersibility, fibrillation efficiency, and compatibility for aqueous systems.	Requires oxidants and careful purification; reagent recovery and cost remain important.
DES and ionic liquid-assisted	CNF or CNC from agricultural residues, non-wood biomass, bamboo, palm waste, and agro-industrial residues.	Improves swelling/delignification, supports solvent recovery, and aligns with green chemistry goals.	Industrial feasibility depends on solvent cost, toxicity, recyclability, viscosity, and life-cycle performance.
Bacterial biosynthesis	BNC from sugars, hydrolysates, or biomass-derived fermentation media.	Produces highly pure nanofibrillar cellulose without lignin or hemicellulose.	Fermentation time, medium cost, sterility, and productivity are critical barriers.

Table 1 indicates that each synthesis route has a specific domain of suitability. Acid hydrolysis is still appropriate when the target is highly crystalline CNC, while mechanical and chemo-mechanical routes are more suitable when the target is CNF for film, hydrogel, or membrane applications. Enzymatic pretreatment and TEMPO oxidation function as enabling steps because they improve fibrillation while lowering process severity. DES and ionic liquid systems represent a new generation of extraction routes, but their green claims should be validated through quantitative energy, solvent recovery, and waste analysis rather than only through descriptive statements.

3.6. Application relevance and surface functionalization

The application of nanocellulose depends strongly on the synthesis route. CNC with high crystallinity and controlled aspect ratio is suitable as a reinforcing phase in polymer composites and barrier films. CNF is suitable for freestanding films, porous networks, membranes, aerogels, hydrogels, and rheology modifiers because of its long fibrillar morphology and entangled network structure. BNC is often directed to biomedical and food-related applications because of its purity, water retention, and biocompatibility (Alzubi & Fan, 2025; Rashid et al., 2023; Reshmy et al., 2021).

Surface functionalization expands the compatibility of nanocellulose with hydrophobic matrices and enhances its function as adsorbent, antimicrobial carrier, flame-resistant sheet, or active packaging component. Carboxylation, acetylation, esterification, phosphorylation, and hydrophobization can tune charge, dispersibility, thermal behavior, and interfacial bonding. However, functionalization also adds processing steps, reagents, and purification requirements.

Therefore, modification should be designed based on target application rather than performed as a routine step (Khumalo et al., 2024; Lam & Hemraz, 2021; Tahir et al., 2022; Zhang et al., 2024).

For packaging and composite applications, nanocellulose improves mechanical strength, oxygen barrier performance, and matrix reinforcement, but water sensitivity and interfacial compatibility must be managed. For water treatment and membrane applications, hydroxyl and modified functional groups can support adsorption or antifouling behavior. For biomedical applications, purity, endotoxin level, long-term safety, and regulatory compliance are essential. These application-specific requirements reinforce the need to connect synthesis route, surface chemistry, and end-use performance in a single design framework (Choudhury et al., 2020; Ghosh et al., 2024; Norizan et al., 2022; Yusuf et al., 2023).

3.7. Research gaps and future direction

Several gaps remain despite the rapid growth of nanocellulose research. First, many studies report successful extraction but do not provide complete mass balance, chemical consumption, energy input, or wastewater characterization. Without these parameters, it is difficult to compare the true sustainability of different routes. Second, differences in terminology, reporting units, and characterization protocols limit cross-study comparison. Standard reporting should include biomass composition, pretreatment yield, nanocellulose yield, particle dimension, crystallinity index, zeta potential, thermal stability, surface chemistry, and dispersion stability (Kaur et al., 2021; Wang & Zhang, 2024).

Third, most studies remain at laboratory scale. Industrial translation requires continuous or semi-continuous processing, feedstock supply chain assessment, solvent recycling, equipment durability, quality control, and economic benchmarking. Processes that perform well for purified pulp may not be robust when applied to heterogeneous agricultural residues. Therefore, scale-up studies must evaluate seasonal biomass variability, ash and silica content, storage conditions, and pretreatment reproducibility (Bharimalla et al., 2015; Illa et al., 2022; Sonyeam et al., 2024).

Fourth, future research should move from extraction-centered studies to application-oriented process design. For example, membranes, hydrogels, active packaging, and composite fillers may require different morphologies, surface charges, and degrees of purity. A route that maximizes crystallinity may not be optimal for network-forming films, while a route that retains lignin may be advantageous for hydrophobic packaging or UV-blocking functions. Thus, feedstock selection, pretreatment strategy, and post-functionalization should be selected according to the desired performance indicators.

Overall, the future development of nanocellulose synthesis should integrate green chemistry, biorefinery concepts, and product-driven engineering. Promising directions include one-pot extraction, recyclable DES systems, low-energy mechano-enzymatic fibrillation, electrochemical oxidation, bacterial nanocellulose from biomass hydrolysates, and hybrid lignin-containing nanocellulose. The most competitive routes will be those that produce consistent nanocellulose quality while reducing chemical burden, energy consumption, production cost, and environmental impact.

IV. CONCLUSION

Nanocellulose can be synthesized from a wide range of biomass sources through chemical, mechanical, enzymatic, bacterial, and hybrid routes. The review shows that no single method is universally superior. Acid hydrolysis is effective for producing high-crystallinity CNC but has environmental and waste-management limitations. Mechanical fibrillation is essential for CNF production but requires pretreatment to reduce energy demand. Enzymatic, TEMPO-mediated, DES-assisted, and ionic liquid-assisted routes provide important opportunities to improve efficiency and sustainability, although their scale-up and economic feasibility still require stronger validation.

The properties of nanocellulose are governed by the interaction between biomass composition and processing parameters. Feedstocks with lower lignin and hemicellulose contents generally facilitate extraction, while lignin-containing nanocellulose may provide useful additional properties for specific applications. Future research should focus on standardized reporting, mass balance, energy and life cycle assessment, techno-economic analysis, solvent recovery, and application-oriented synthesis. A feedstock-specific and product-driven strategy will be essential to advance nanocellulose from laboratory-scale extraction toward sustainable industrial utilization.

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