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Advances in Nanocellulose-Enhanced Polymers and Composites: Structure, Performance, and Applications

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Abstract

The most common biopolymer is cellulose, which can be converted into nanocellulose (NC) the sustainable nanomaterial possessing the outstanding characteristic of biodegradability, renewability, low density, high aspect ratio, and excellent mechanical performance. Such distinctive features make NC a promising filler in polymer and composite systems. Recent developments in the preparation of nanocellulose using various natural and artificial sources have facilitated scalable production processes that have less energy requirements and are cost effective. The focus in this review is given on practical preparation methods of nanocellulose and how they affect structure property relationships when incorporated into polymer matrices. Several characterization methods, such as rheological behaviour, powder flow properties, and thermal analyses are presented in order to demonstrate their use in the evaluation of NC-based composites. Special attention is paid to the multifunctional use of polymer nanocellulose composites in the field of structural materials, barrier films, biomedical scaffolds, drug delivery systems, aerogels, wound healing, biosensing, and bioimaging. Comparing nanocellulose to neat polymers, representative studies demonstrate that it can improve biodegradation rates, decrease oxygen permeability by up to 80 times, and increase tensile strength by 20–300%. The extended applicability of nanocellulose shows the possibility of using the material as a sustainable reinforcement of the next generation, high-performance, and environmentally friendly polymer composites.

Keywords: Nanocellulose, Preparation methods, Characterization of nanocellulose, Applications Chemical treatment of cellulose, Acid hydrolysis

INTRODUCTION

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The most common carbohydrate on Earth is cellulose, which makes up almost 40 percent of all of the organic matter on Earth and is replenished via photosynthesis [1]. The cellulose has a structure made of repeating glucose groups ($C_6H_{10}O_5$) that form crystalline and amorphous structures, the ratio of which depends on the source of the plant and has direct proportionality to its mechanical characteristics [2,3].In addition to the fact that cellulose is naturally found and is renewable, it is a very viable material in development of composites due to its low density, low cost, biodegradability, biocompatibility, and low cytotoxicity. On the nanoscale, cellulose can be converted to nanocellulose, with high surface area, adjustable crystallinity and capable of forming very strong intermolecular interactions with polymer

matrices. It can also be modified functionally, e.g., the addition of acidic, chloride, or oxide groups, which increases its interfacial compatibility and broadens its possible uses [4]. These properties have had the benefit of making cellulose and cellulose-derived materials useful reinforcements in the production of lightweight, high-performance, and eco-friendly polymer composites especially in biomedical, packaging, and structural applications.

Recent developments in the manufacture and functionalization of nanocellulose using a range of natural and synthetic sources have expanded its usage in a wide range of polymer systems by enabling scalable, cost-effective, and energy-efficient production methods [5–8].

Types of Nanocellulose

Cellulose Nanocrystals (CNCs)

CNC separates by acid hydrolysis using enzymatic methods and mineral acids [9–13]. CNCs can generally be processed through acid hydrolysis of cellulose either with mineral acids or enzymatic treatments that preferentially degrade amorphous regions and produce highly crystalline, rod-shaped CNCs with a high aspect ratio. The resulting nanocrystals are highly stiff and reinforcement of strength and modulus and barrier performance is appealing to polymer composite. When the cellulose fiber disintegrates, a stiff, nanoscale fiber will be formed [14]. Pulp passes between two stones and generates shear pressure and heat. The last NFC defines according to the number of grinding and HPH strokes. Though 10 cycles are suggested in grinding, 14 of 30 processing cycles give useful results [15]. CNC processing parameters including acid type and hydrolysis time have a strong impact on the CNC morphology and surface chemistry and thus, their dispersion and interfacial adhesion in polymer matrices. The schematic representation of the preparation methods of cellulose nanocrystal fibers is shown in Figure 1.

Cellulose Nanofibers (CNFs)

Although mechanical treatment is usually used to separate cellulose nanofiber (CNF), a short and flexible fibril, other techniques such as pharmacological and enzymatic treatments [16–17] are also employed. The CNFs are mostly obtained by the mechanical destruction of cellulose fibres into long, flexible fibrils, but enzymatic or chemical pretreatments can be used to save more energy. High-pressure homogenization, ball milling, and ultrasonication are common methods that are aimed at the production of CNFs with customized size and surface properties. CNFs offer a superior reinforcement and rheological modification in polymer composites because of entangled network structure and large aspect ratio, which are needed in toughness- and barrier-enhancing applications.

Bacterial Nanocellulose (BNC)

Bacteria such as Sarcina ventriculi, Acetobacter, Azotobacter and Pseudomonas are capable of synthesizing bacterial nanocellulose (BNC) via the digestion of glycerol and glucose. [18,19]. It is a promising reinforcement material in polymer composites due to its high crystallinity, purity and distinctive three-dimensional nanofibrillar network. Its morphology and mechanical properties are affected by growth conditions such as pH, oxygen availability and nutrient sources. BNC has attracted attention in more sophisticated composite applications where high strengths, flexing and biocompatibility are needed.

Sources of Nanocellulose

This paper proposes various types of nanocellulose sources that can be sourced out of a broad variety of renewable resources, such as wood, plants, algae and microbial cultures [20]. Cellulosic materials play a vital part in medical therapy and disease prevention, and they can be delivered in three big ways, oral, transdermal, and topically. The source and processing technique used has a great influence on the morphology, crystallinity, and surface chemistry which also dictate the performance of the composites. These green nanomaterials are also finding their way into polymer matrices in an effort to produce light-weight, high-strength and eco-friendly composites to be used in packaging, biomedical and structural applications.

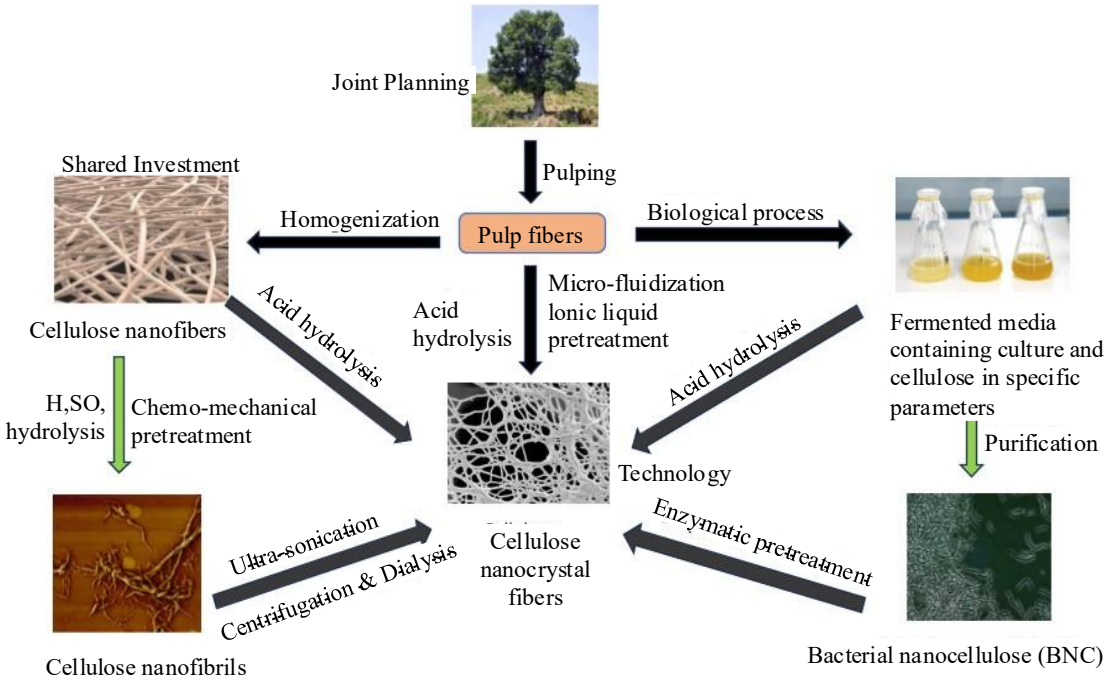

Figure 1. Methods of Preparation of Cellulose Nanocrystal Fibers

METHODOLOGY

Preparation of Nanocellulose for Polymer Composites

Nanocellulose is obtained by different mechanical and chemical-mechanical treatments that define its morphology, crystallinity, surface chemistry and finally its reinforcing effectiveness within polymer composites [21]. The preparation paths also control the fibrillation efficiency, aspect ratio, and surface functionality which play decisive roles in dispersion and interfacial adhesion in polymer matrices. (Fig. 2).

Mechanical Methods

The use of mechanical disintegration of cellulose fibers to nanocellulose is one of the well-used methods of nanocellulose production. High-pressure homogenization (HPH) [14, 22–23], microfluidization [24], grinding [24], cryocrushing [25–27], ultrasonication [28–29] and ball milling are commonly used methods to shorten the size of cellulose fibers into nanoscale dimensions.

High-Pressure Homogenization (HPH): High pressure exposes cellulosic suspensions to severe shear and impact forces, which make fibrils at the nanoscale in the absence of organic solvents [30]. Passing quantity is known to affect fibril diameter, surface area and reinforcement in composites [31].

Microfluidization: The fibers are defibrillated using a high-pressure pumping in interaction chambers leading to the generation of nanofibrils with high surface areas. More passes are beneficial to fibrillation but lead to aggregation that should be managed to achieve homogeneous composite performance.

Grinding: The rotating stones are used to mechanically grind the fiber, which makes it smaller and fibrillated. Grinding, when used with HPH cycles, increases the yield and aspect ratio of nanofibrils, which increases their effectiveness in loading transfer in polymer matrices.

Cryocrushing: The water saturated cellulose fibers are frozen in liquid nitrogen and broken under shear to form nanofibrils whose crystalline regions are disrupted, which enhance interfaces bonding of polymers.

High-Intensity Ultrasonication (HIUS): The shear forces caused by cavitation are used to separate fibrils, giving a uniform nanocellulose that is better dispersible. HIUS is commonly used with HPH to result in increased fibrillation and composite reinforcement.

Ball Milling: The nucleic acid is diminished to nanoscale sizes through the action of mechanical attrition. Ball size, milling time and moisture content parameters affect the fibril morphology and exposure of functional groups, which affect compatibility with polymers [32].

Chemical–Mechanical Methods

Although they are effective in a pure form of mechanical method, they are usually energy consuming and can lead to equipment blockage. Chemical pre-processing (e.g. oxidation, enzymatic hydrolysis or controlled surface modification) can lower the energy requirement and increase fibrillation efficiency. **Refining and HPH:** Microfibrillated cellulose (MFC) is obtained by combining refining and homogenization, which increases the surface properties of the material and leads to a high level of interfacial adhesion in composites.

Sonication with Chemical Pretreatment: Chemical activation, then, followed by HIUS needs less fibrillation energy and produces nanofibrils with reactive surfaces which enhance dispersion in hydrophobic polymer matrices [33–35].

TEMPO Oxidation

Through TEMPO-mediated oxidation of cellulose, selective oxidation of primary hydroxyl groups of cellulose to carboxyl groups, with negative charges appearing on the surfaces of the fibrils, induces electrostatic repulsion between cells and fibrils, and delamination of the fibrils. The result of the process is individualized nanofibrils that are more dispersible in aqueous and polar polymer systems, which promote interfacial adhesion and transparency in nanocellulose-based films and composites.

Acid Hydrolysis

The amorphous domains are broken off by acid hydrolysis to produce highly crystalline cellulose nanocrystals (CNCs). The dimensions and crystallinity of CNCs are regulated by parameters including temperature, concentration of acids and hydrolysis time. Sulfuric acid is the most frequently employed, which adds sulfate ester groups to increase dispersibility at the cost of thermal stability. CNCs are very rigid in nature and have a high aspect ratio, making them great reinforcements to polymer composites, especially in packaging and structural applications.

Enzymatic Hydrolysis

Enzyme hydrolysis use of oxidoreductases and hydrolases [36–38] is a mild and environmentally friendly method of nanocellulose manufacture. Processing has to be controlled, whereas the energy demand is reduced. Enzymes have the capability to modify surface chemistry of nanofibrils without altering morphology to allow a customized response to certain polymer systems, especially in biomedical and biodegradable composites.

Periodate Chlorite Oxidation

This will break down the secondary hydroxyls into aldehydes and eventually to carboxyl groups, leading to the decomposition of fibers. The resulting CNFs (3–5 nm diameter) are highly surface reactive and display a uniform morphology and enhance dispersion and bonding of polymer matrices.

Oxidative Sulfonation

The process of sulfonation attaches sulfonates to cellulose to hasten the process of fibrillation and produce CNFs with a diameter of 10–60 nm. This technique improves the dispersibility of water and can be used as a green pre-treatment of polymer nanocomposites that need a stable aqueous suspension.

Carboxymethylation

The carboxyl groups formed by carboxymethylation have a great effect on the dispersibility and interfacial bonding of CNF. The technique has the capability to make films based on optical transparent nanocellulose, which is appealing in flexible packaging, barrier coating, and optical composites.

Cationization

Cationization introduces quaternary ammonium groups and forms positively charged CNFs that possess antibacterial behaviour. Cationic nanocellulose promotes dispersibility of negatively charged polymers and is versatile in composites in terms of wound dressing, food/food packaging, and antimicrobial surface coating.

Deep Eutectic Solvent (DES) Pretreatments

DESs are created by using cheap and biodegradable materials (e.g., choline chloride -urea) to offer a good pretreatment to enhance fibrillation and surface modification. CNFs treated with DES are homogeneously morphologized and exhibit increased reactivity and provide a feasible and sustainable route to large-scale production of nanocellulose towards composites [39].

Chemical Hydrolysis (Other Acids)

Besides sulfuric acid, hydrochloric, phosphoric, hydrobromic, and nitric acids may be used to give CNCs of different crystallinities and surface charges. The aspect ratio of CNC is regulated by the conditions of hydrolysis and has a direct impact on the efficiency of reinforcement of polymer composites.

Alkaline Acid Pretreatments

Successive alkali and acid removals define NFCs of enhanced purity and crystallinity [40], with improved thermal stability and strength of the resulting composites. Repeated alkali and acid removals can remove lignin, hemicellulose and pectin [41], improving fibrillation and cellulose recovery.

Organic Acid Pretreatments

Formic and oxalic acid are organic acids that are used to soften cellulose and partially hydrolyze pulp. These treatments enhance dispersibility, thermal stability, and interfacial relationships to produce lignocellulose-based nanofibers that can be used as a composite reinforcement.

Solvent Assisted Pretreatments

Moderate alkaline pretreatments (e.g. NaOH solutions) eliminate impurities, improve fibrillation to form CNFs with better thermal degradation properties. The solvent pretreatments enhance fibrillation rates and are also compatible with hydrophobic polymers thus appealing to composite processing.

Ionic Liquids

Ionic liquids including 1-butyl-3-methylimidazolium chloride are beginning to be used as effective solvents in dissolving and regenerating cellulose. It is possible to homogenize pretreated cellulose fibrils recovered through ionic liquids into nanocellulose with a customizable surface chemistry to increase their incorporation into polymer composites [42].

Chemical and Pretreatment Methods for Nanocellulose in Polymer Composites

The chemical modifications and pretreatment are commonly used to enhance the fibrillation performance, lower the energy usage and add surface functionalities to nanocellulose. These mechanisms are essential to the achievement of the interfacial response of polymer matrices, thus improving dispersion, adhesion, and the total mechanical, thermal and barrier characteristics of polymer-nanocellulose composites. Surface modifications (acetylation, oxidation with TEMPO, silanization and grafting methods, etc.) are particularly significant to enhance the compatibility with hydrophobic synthetic polymers.

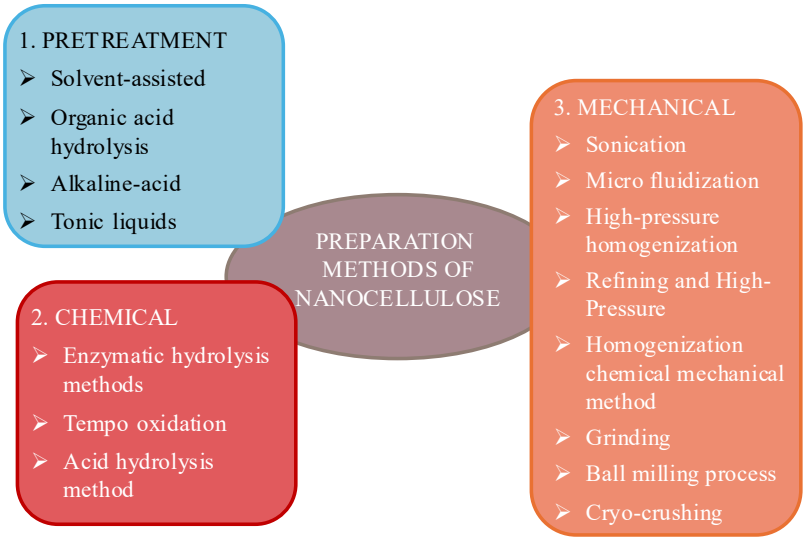

Figure 2. Schematic Representation of Nanocellulose Preparation Methods

In line with recent research that highlights surface chemistry and interfacial compatibility as crucial factors influencing mechanical performance, this review focuses on realistic nanocellulose preparation techniques and how they impact structure–property relationships when added to polymer matrices [5,7,8].

Characterization and Properties of Nanocellulose

Nanocellulose morphology, crystallinity and aspect ratio is highly dependent on extraction protocols, processing environment and the source of biomass. The recommendation of the World Health Organization that pharmaceutical formulations use herbal medicine and natural excipients be standardized is often supported by the pharmacopoeia standards of the countries concerned [43]. These are parameters that have a direct bearing on its reinforcing potential in composite matrices. The following methods are normally used to characterize.

- **Mechanical Testing:** Establishes the reinforcing strength of nanocellulose in polymer composites.
- **Density and Porosity:** Determined with the aid of helium pycnometry to determine the packing and dispersion of composite.
- **X-ray diffraction (XRD) and Crystallinity Index:** Applied to measure structural order, which is related to stiffness and thermal stability of composites. Increased crystallinity can be decreased by longer hydrolysis times, and thermal resistance can be enhanced by regulated surface modifications (e.g. disulfation or crosslinking).
- **Thermal Analysis (DSC, TGA, DTG):** Gives data on glass transition, melting, degradation profiles and processing stability. Processing temperatures are typically kept at lower than 200 C to prevent thermal degradation during compounding.
- **FTIR Spectroscopy:** Determines functional groups added to the carbon during chemical modification and proves interfacial bonding to polymers.
- **Optical Microscopy and SEM/TEM:** Examine the nanocellulose's morphology, dispersion, and particle size in composites.

Moisture Absorption and Hygroscopic Behavior

The sorption of moisture is an important study in predicting dimensional stability and barrier behavior of nanocellulose-based composites [44–45]. CNC and CNF are quite water-affinity so this property may adversely affect mechanical characteristics during wet environments. The processes of acetylation or silanization can greatly decrease the amount of moisture absorbed and prolong the ultimate stability of packaging and structural applications.

Ash Content and Thermal Residue

Ash content determination can be helpful in determining the purity of nanocellulose and inorganic matter left that may influence interactions between fillers and matrices. Analysis of ash and acid-insoluble residues [46] give a clue on the existence of non-cellulosic ingredients (e.g., silica, minerals) which determine thermal behavior and composite processing.

CNC Powder Flow Properties Characterization

Helium pycnometer can be used to measure nanocellulose density and porosity. The total pore volume and specific surface area are computed by N2 sorption analysis [47].

Crystallinity Index (CI)

The crystallinity of CNC can be analyzed by the use of a powder X-ray diffraction analysis (Bruker-AXS D8 Advance). The experiment involved the use of Cu radiation which had a wavelength of 1.5406A, an intensity of 25 mA and a voltage of 35 kV. The XRD of the pattern can be taken between 3 to 90 Degree (2the). The same diffractogram used in the determination of the crystallinity index is based on the peak height method described by Segal *et al*. [48].

Flow and Surface Properties

Nanocellulose powders have an influence on their dispersion in polymer matrices in the process of melt compounding or solvent casts due to their flowability. The analyses of the sample using Helium pycnometry, BET surface area, and pore volume can give data on bulk density, porosity, and surface area as the main parameters of filler loading optimization in polymer composites.

Thermal and Structural Behavior

Phase changes and degradation Characteristic Evaluation Phase changes and degradation Characteristic Evaluations are done by Differential Scanning Calorimetry (DSC) and Thermogravimetric Analysis (TGA). Such measurements aid in creating processing windows of polymer nanocomposites and demonstrate the importance of surface modification on the enhancement of thermal stability.

Quantitative and Qualitative Performance Metrics:

The addition of nanocellulose to polymeric matrices has been shown in numerous studies to result in quantifiable improvements in mechanical and functional properties. For instance, polylactic acid (PLA) reinforced with 5% weight nanocrystalline cellulose (NCC) showed a ~34% increase in strength and ~50% increase in stiffness, with a tensile strength that increased from ~58 MPa to ~78 MPa and a modulus that increased from 3.2 GPa to 4.8 GPa [49]. When 7 weight percent cellulose nanofibrils (CNF) and cellulose nanocrystals (CNC) were added to chitosan films, the tensile strength increased by 104% and 52%, respectively [50]. After treating nanocellulose with acetic acid, all-cellulose composites made from oil-palm empty fruit bunch (EFB) fibers shown an impressive ~279% increase in tensile strength [51].

CNF–nanoclay biohybrid films showed oxygen permeability as low as 0.07 cm^3 µm m^{-2} day^{-1} kPa^{-1} at 50% RH, and PLA/nanocellulose multilayer systems exhibited an approximately 80-fold reduction in oxygen permeability when compared to clean PLA in terms of barrier characteristics [52,53]. In comparison to untreated matrices, nanocellulose-reinforced all-cellulose composites have also shown enhanced biodegradation, exhibiting quicker weight loss in soil-burial studies [51,54]. These quantitative and qualitative measurements unequivocally demonstrate that nanocellulose is a sustainable, effective reinforcement that significantly enhances the mechanical integrity, barrier function, and environmental friendliness of polymer composites.

Dispersion and Interfacial Bonding Challenges

When added to hydrophobic polymer matrices like polyethylene, polypropylene, or polystyrene, nanocellulose's hydrophilic nature frequently results in poor dispersion and weak interfacial adherence,

despite its exceptional mechanical and reinforcing capabilities. Agglomeration of NC and restricted stress transport across the interface are the outcomes of these incompatibilities. Several surface modification methods, such as acetylation, silanization, and grafting functional groups compatible with polymers (such alkyl chains or maleic anhydride) onto nanocellulose surfaces, have been used to get around these issues. These changes result in composites with better mechanical, thermal, and barrier performance by strengthening interfacial bonding and improving dispersion uniformity [55–57].

Applications of Nanocellulose in Polymer Composites

Nanocellulose's high aspect ratio, crystallinity, surface functionality, and mechanical strength make it a versatile nanomaterial that improves the performance of polymer composites. Applications in the biological, structural, packaging, electrical, and environmental domains have been made possible by its special qualities. Figure 3 lists all further uses for nanocellulose.

In line with recent thorough reviews emphasizing the adaptable design of nanocellulose-based hybrid systems for advanced applications, particular attention is given to the multifunctional use of polymer–nanocellulose composites in structural materials, barrier films, biomedical scaffolds, drug delivery systems, aerogels, wound healing, biosensing, and bioimaging [6,8].

Biomedical and Biopolymer Composites

Nanocellulose is being used in biomedical scaffolds, wound dressings, tissue engineering, biosensors and implantable devices using biopolymer matrices. Cellulose nanocrystals (CNCs) due to their high surface to volume ratio and tunable surface chemistry, menace mechanical strength, controlled hydration and cell proliferation and as such, prove useful in biocompatible composites [58–63]. CNC-based, hydroxyapatite, alginate, and chitosan freeze-dried scaffold have great potential in implantation in bone tissue engineering [67]. CNCs are also good supporting matrices of bioactive molecules because they have non-immunogenic and greener properties [66].

Drug Delivery Systems (Subset of Biomedical Applications)

Controlled oral, transdermal, and local drug delivery with nanocellulose and polymer composites is under investigation which exploits the colloidal stability, surface charge and functionalizability to enhance solubility, release kinetics and therapeutic efficacy [64,65,68,69,70,73]. As an example, MCC/alginate and CNF/alginate composite beads are developed to deliver metformin hydrochloride in a sustained release, and CNC-based matrices are used to deliver anticancer drugs such as etoposide, paclitaxel and docetaxel [71–73].

Transdermal and Localized Delivery Systems

Transdermal and localized delivery platform Transcellulose-polymer composites have been investigated in the form of nanocellulosic nanocomposites because of their high permeability, biocompatibility, and mechanical stability. Bacterial nanocellulose (BNC) membranes have been explored in the delivery of active compounds and display rapid and controlled release just as commercial systems [74–75]. Likewise, biodegradable polymer nanocellulose composites can be used as mucoadhesive or site-specific delivery matrices which enhances drug efficacy with minimum dosage [76].

Tissue Engineering and Regenerative Medicine

CNC- and CNF-based composites offer flexible scaffolds in tissue engineering such as in cartilage, bone and in vascular grafts. This is made possible by their high surface area as well as mechanical stability in immobilization of bioactive molecules, enzymes, and growth factors to support cell adhesion and proliferation [77–79].

The use of functionalized nanocellulose hybrids in imaging and sensing biomedical applications has also been reported, including MRI-active scaffolds, and shown to have potential biomedical theranostic polymer composite usage [79]. The Tubular BNC with multilayered hydrogel has been used in

regenerative medicine such as nerve conduit, cartilage repair scaffolds embedded on the appropriate polymer matrix [80–81].

Hydrogels, Aerogels, and Nanocomposite Films

Hydrogels, aerogels, and films made of nanocellulose have proven to be multifunctional; i.e., in controlled release, antimicrobial action, and mechanical reinforcement. CNF-Ag+ composites, e.g., produce rigid hydrogels and aerogels that are applicable in polymer-nanocellulose biomedical devices, and responsive to environmental factors (temperature, pH, ionic strength, and light) [82–89].

Cardiovascular and Vascular Applications

BNC-polymer composites are especially example of promising towards vascular grafts and cardiovascular implants because of these properties: mechanical integrity, biocompatibility, and adjustable porosity. In the tissue engineering area, BNC has been tested in synthetic duct systems to complete medium- to large-diameter vessels, and it gives a template of polymer-reinforced scaffolds [90].

Summary of Functional Advantages

Overall, nanocellulose–polymer composites offer:

- High flexibility and mechanical strength
- Surface chemistry that can be adjusted to improve interfacial bonding
- Managed porosity and permeability for the delivery of nutrients and medications
- The possibility of regenerative applications and biocompatibility

In line with the goals of advanced polymer composites research, these multifunctional qualities establish nanocellulose–polymer composites as next-generation materials for structural, biomedical, and environmental applications.

Figure 3. Applications of Nanocellulose.

CONCLUSION

Overall, nanocellulose is a tremendously flexible and eco-friendly re-enforcement material in polymer and composite system with a distinctive collection of biodegradability, renewability, and outstanding mechanical as well as thermal qualities. It has become important to facilitate its synthesis and surface modification, and characterization, which has enhanced its ability to be compatible with a wide variety of polymer bases, allowing the creation of lightweight, high-performance, and environmentally friendly composites. This review identifies how nanocellulose can be used to improve the structural, functional and multifunctional characteristics of polymer-based materials and is associated with potential applications in packaging, biomedical equipment, coatings, and structural components. Elaborating on this, it will be important to further explore large scale production technique, surface functionalization and process optimization to realize the complete business potential of nanocellulose in the following generation polymer composites.

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Nil

Authors Contributions

All authors contributed equally.

Availability of Supporting Data

On reasonable request, the corresponding author will make the datasets used in this study available.

Conflict of Interest

Regarding the publishing of this paper, the authors hereby declare that they have no conflicts of interest.

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