International Journal of Science and Research Archive
eISSN: 2582-8185
Cross Ref DOI: 10.30574/ijsra
Journal homepage: https://ijsra.net/

(Review Article)

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Synthesis, characterization and application of nanocrystalline cellulose: A review

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International Journal of Science and Research Archive, 2023, 08(02), 234–243

Publication history: Received on 28 January 2023; revised on 22 March 2023; accepted on 24 March 2023

Article DOI: https://doi.org/10.30574/ijsra.2023.8.2.0217

Abstract

This review article summarizes the recent progress in the synthesis, characterization, and applications of nanocrystalline cellulose (NCC). NCC is a nanoscale material that can be extracted from renewable sources such as plants, and has attracted significant attention due to its unique properties, including high surface area, mechanical strength, and biodegradability. The review covers various methods for the synthesis of NCC, including acid hydrolysis, enzymatic hydrolysis, and mechanical methods. The characterization techniques for NCC, such as X-ray diffraction, transmission electron microscopy, and dynamic light scattering, are also discussed. Furthermore, the review provides an overview of the applications of NCC, including reinforcement in composites, paper and packaging, biomedical applications, and energy storage. Overall, this review provides a comprehensive understanding of the synthesis, characterization, and applications of NCC, highlighting its potential as a sustainable and versatile nanomaterial for various fields.

Keywords: Nanocrystalline cellulose; Food packaging; Cellulose; Acid hydrolysis

1. Introduction

Cellulose is a complex carbohydrate or polysaccharide that is found in the cell walls of plants, algae, and certain bacteria. It is the most abundant organic compound on Earth and provides structural support to plant cells, helping to maintain their shape and rigidity. Cellulose molecules are made up of long chains of glucose molecules that are bonded together by beta-1,4-glycosidic linkages [1]. These chains are arranged in parallel to form strong fibers that are insoluble in water and resistant to degradation by most enzymes. Nanocrystalline cellulose (NCC), also known as cellulose nanocrystals (CNC), are tiny crystalline structures that are derived from cellulose, which is the main structural component of plants. NCC is produced by breaking down cellulose fibers into smaller fragments using mechanical, chemical or enzymatic methods, and then subjecting them to acid hydrolysis. Acid hydrolysis is the most commonly used method due to its high yield and efficiency. However, it requires strong acid and high temperature, which may affect the NCC properties. Enzymatic hydrolysis and mechanical treatments, on the other hand, offer milder conditions and produce NCC with different properties and morphology. The resulting NCC has a high aspect ratio [2], meaning that they are very long and narrow, with diameters typically ranging from 5-20 nanometers and lengths up to several micrometers. Due to their small size and high surface area, NCC exhibits unique physical and chemical properties, such as high strength, stiffness, and transparency, as well as exceptional thermal and electrical conductivity. Nanocrystalline cellulose occurs in three forms: cellulose nanocrystals (CNC), cellulose nanofibrils (CNF), and bacterial nanocellulose (BNC) [3]. NCC has a high aspect ratio and surface area, which contribute to its unique properties. It exhibits high mechanical strength, stiffness, and thermal stability, making it a potential reinforcement material for composites [4, 5]. It also has excellent optical properties, such as transparency and birefringence, which can be used in various applications, such as packaging and optical devices. Furthermore, NCC has high water-absorption capacity, which makes it a potential material for drug delivery and wound healing applications. NCC has a wide range of potential applications in various industries, including

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biomedical, electronics, packaging, and construction. For example, it can be used to reinforce polymers, create transparent films, and even produce drug delivery systems [6]. Additionally, NCC is a sustainable and renewable resource, as it is derived from plant-based sources and can be produced using environmentally friendly processes. Characterization of NCC involves several techniques, such as X-ray diffraction (XRD), transmission electron microscopy (TEM), and dynamic light scattering (DLS). XRD is used to determine the crystallinity and crystal size of NCC, while TEM and DLS provide information on the morphology and size distribution.

Nanocrystalline cellulose (NCC) has emerged as a promising nanomaterial with unique properties that have attracted attention from various fields of research. In this review, we will summarize the recent advancements in NCC synthesis, characterization, and applications.

2. Cellulose

Cellulose is the most abundant natural polymer available on the earth and is the main constituent in the cell wall of trees and plants. It was discovered in 1838 by the French scientist Anselme Payen who isolated it from plant matter and determined its chemical formula [7].

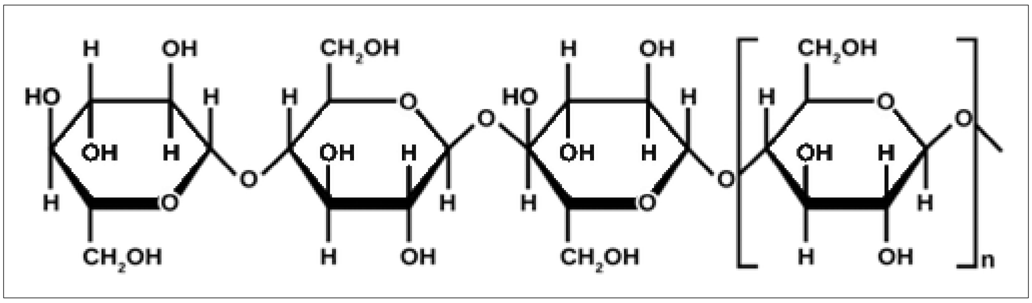

Figure 1 Structure of cellulose

2.1. Sources of Cellulose

The main sources of cellulose are plants; however, algae, bacteria and some sea animals [8] are also capable of producing cellulose in large quantities. Below is a brief description of the primary sources:

2.1.1. Plants

Cellulose is a key component of plant cell walls, providing structural support and rigidity to the plant. it is found in varying amounts in almost all plant materials, including trees, shrubs wood, cotton, flax, hemp, jute, and grasses [7]. In trees, cellulose is the primary component of wood and provides the necessary strength and stability for the tree to stand upright. Cotton is one of the most common sources of cellulose and has a cellulose content ranging from 80 to 90%, depending on the variety and growing conditions. Wood is the primary structural material in trees and has a cellulose content ranging from 40 to 50%, depending on the species and age of the tree. Flax, hemp or sisal, and jute which are plants commonly used as fibers have about 70 – 80% cellulose content. Some other cellulose plant sources include agricultural wastes such as wheat, rice husk, sugarcane bagasse, sawdust, corn cob etc.

2.1.2. Algae

Algae are a diverse group of photosynthetic organisms that can be found in a variety of aquatic environments, including oceans, lakes, and ponds. Some species of algae are known to contain significant amounts of cellulose in their cell walls, making them a potential source of cellulose for industrial applications. For example, [9] successfully extracted cellulose from Ulva lactuca, a green macroalgae for the production of microcrystalline cellulose. Various studies have reported the successful extraction of cellulose from red, green, and brown algae [10]. Among the various types of algae, green algae are the most preferred for cellulose extraction.

2.1.3. Bacteria

Bacteria are another potential source of cellulose for industrial applications. Although most bacteria do not naturally produce cellulose, certain strains have been genetically engineered to produce high levels of cellulose, making them a potential source of cellulose. One such strain is the bacterium *Komagataeibacter xylinus* (formerly known as Gluconacetobacter xylinus), which has been extensively studied for its ability to produce high-quality cellulose in a variety of conditions [11, 12]. This bacterium is capable of producing large amounts of cellulose in a relatively short period of time and can be cultured in large quantities using a variety of growth media [13]. Another bacterium with potential for cellulose production is *Acetobacter xylinum*, which is also capable of producing high-quality cellulose under the right conditions [11, 14]. This bacterium is commonly found in fermented foods such as vinegar and can be easily cultured in the laboratory.

2.1.4. Tunicates

Tunicates, also known as sea squirts, are a group of marine animals that are known to produce high-quality cellulose in their outer tunic layer. The tunic layer is composed of a complex matrix of cellulose fibers, along with other polysaccharides and proteins, which provide structural support and protection to the animal [15]. The cellulose produced by tunicates is of particular interest to researchers due to its high degree of crystallinity and its unique physical properties, such as high tensile strength and biocompatibility [16]. Tunicate cellulose has potential applications in a range of industries, including biomedical engineering, food packaging, and textile production. One tunicate species that has been extensively studied for its cellulose content is the sea squirt (*Halocynthia roretzi*). This animal is capable of producing large amounts of cellulose in a short period of time and has been shown to produce cellulose with a high degree of crystallinity and tensile strength [17].

2.1.5. Properties and Structure of Cellulose

Cellulose is a linear polysaccharide composed of repeating units of β-D-glucose, linked together by β(1→4) glycosidic bonds. The monomers of glucose are arranged in such a way that every other monomer is flipped upside down, resulting in a long, linear chain that is highly stable and resistant to degradation [18]. The properties of cellulose are largely determined by its unique structure. The β(1→4) glycosidic bonds are relatively strong, which contributes to the high tensile strength of cellulose. In addition, the linear arrangement of the glucose monomers allows for extensive hydrogen bonding between adjacent chains, resulting in a highly crystalline structure [19]. This crystalline structure contributes to the stiffness and rigidity of cellulose, as well as its resistance to swelling and solubility in water. The unique properties of cellulose make it an ideal material for a wide range of industrial applications, including the production of paper and textiles, as well as use in the food and pharmaceutical industries. Cellulose is also being investigated for use in the development of biodegradable plastics and other sustainable materials.

3. Polymorphism of cellulose

Polymorphism refers to the ability of a material to exist in different crystalline forms, or polymorphs. Cellulose is known to exhibit several different polymorphs, which can have significant impacts on its physical and chemical properties. So far, cellulose has been identified to exist in four different forms depending on native origin and treatment of cellulose [20]. These four forms are cellulose I (α and β), II, III and IV). Cellulose I (α and β) is the native form. Algae and bacterial cellulose have primarily Iα while tunicate, wood, cotton and ramie fibres have primarily Iβ [21]. The different polymorphs of cellulose are usually determined by x – ray diffraction and each gives a different diffraction pattern. The crystalline structure of cellulose can vary depending on factors such as the degree of polymerization and the presence of other compounds such as lignin, and also can be influenced by the solvent used, with some solvents promoting the formation of certain polymorphs over others [23].

4. Nanocrystalline cellulose

Nanocrystalline cellulose (NCC) is a type of nanomaterial that typically has dimensions in the nanometer scale, with crystalline particles that are typically a few nanometers in width and length, and a thickness of about 1 nm [24]. The aspect ratio (length to width) of NCC particles can vary depending on the source of cellulose and the processing method, but it is generally in the range of 5-50. Nanocrystalline cellulose are nanometre size range in all dimensions. It is a typically rod – shaped monocrystalline cellulose with tens to hundreds of nanometres in length and 1 – 100nm in diameter [25]. They are nanomaterials derived from natural sources such as wood or plants by breaking down cellulose fibers using chemical, mechanical or enzyme treatments. NCC is composed of small, crystalline particles that are typically a few nanometers in size, with a high surface area-to-volume ratio and unique mechanical, optical, and chemical properties. NCC has unique properties due to its small size and high surface area-to-volume ratio, including high strength and stiffness, high transparency, low thermal expansion coefficient, and high chemical stability. These properties make NCC an attractive material for a wide range of applications in various fields, including composites, coatings, films, and biomedical devices [24]. Plant cellulose comprises of crystalline and amorphous regions in different proportions which depends largely on the plant species. When lignocellulosic materials are subjected to various mechanical, chemical, or enzyme treatments the crystalline region can be isolated to give NCC. The chemical composition, and dimensions of NCC is determined by the type of plant, their origin, and the isolation methods used [26, 27].

4.1. Types of Nanocrystalline Cellulose

There are three general types of cellulose nanomaterials (CNs) that can be extracted from different sources. They are cellulose nanocrystals (CNCs), cellulose nanofibrils (CNFs), and bacteria nanocrystals (BNC) [20]. Cellulose nanocrystals can be referred to as cellulose nanowhiskers (CNW) or nanocrystalline cellulose (NCC) [28, 29] while cellulose nanofibrils can be referred to as nanofibrilated cellulose (NFC) or microfibrillated cellulose (MFC) depending on their size and extraction method [30]. CNF typically has dimensions in the nanometer scale, with diameters ranging from a few to tens of nanometers and lengths up to several micrometers. The aspect ratio (length to width) of CNF particles can vary depending on the source of cellulose and the processing method, but it is generally in the range of 10-100. CNF can be synthesized using various mechanical or chemical methods. Mechanical methods involve physically breaking down cellulose fibers into smaller fibrils, while chemical methods involve using chemicals to dissolve the amorphous regions of cellulose and leave behind the crystalline regions that can be further disintegrated into fibrils. Some of the most common methods for synthesizing CNF include high-pressure homogenization, microfluidization, cryocrushing, and acid hydrolysis. Bacterial nanocrystals are typically in the range of 30-100 nm in diameter, although the exact size can vary depending on the species of bacteria. The shape of the nanocrystals can also vary, with some bacteria producing nanocrystals that are elongated or bullet-shaped.

4.2. Synthesis of Nanocrystalline Cellulose

The methods of synthesis of nanocrystalline cellulose is categorized into three namely mechanical, chemical, and enzymatic hydrolysis.

The synthesis of NCC follows two steps [31]. The first step involves the pretreatment of the lignocellulosic biomass material to obtain pure cellulose, and the second step involves extraction of nanocrystalline cellulose.

4.2.1. Pre-Treatment Process

The aim of the pre-treatment process is to remove ashes, waxes, lignin, hemicelluloses and other non – cellulosic compounds. The types of pre – treatment applied on different raw materials such as plant, tunicate, algae and bacteria cellulose have been reported [32, 33]. The various pre – treatment processes that have been reported include alkaline delignification and organosolvation with acetic acid. Bleaching treatment with oxidizing agents is also considered as pre – treatment processes [34, 35].

Alkaline treatments are conducted when a more effective lignin, hemicelluloses and pectin solubilisation and removal is needed. Alkaline extraction needs to be controlled to avoid cellulose degradation [36]. A typical alkaline treatment involves impregnating of fibres in a 5% sodium hydroxide solution for about 4hrs at 55^{o}C to 75^{o}C. Sodium hydroxide reduces superoxide radicals wherein lignin and hemicelluloses are hydrolyzed [29]. However, if lignin content in the cellulose is high, the nanocellulose yield is low [37].

After biomass delignification, oxidative treatment is carried out to improve aging resistance, avoiding yellowing and brittleness as well as removing lignin. This pre-treatment method involves the use of an oxidizing agent to break down the lignin and hemicellulose components of the lignocellulosic material, leaving behind a more accessible cellulose substrate. The most commonly used oxidizing agents for oxidative pre-treatment include hydrogen peroxide, H_2O_2, chlorine dioxide, ClO_2, ozone, O_3 as well as sodium hypochlorite, NaClO.

4.2.2. Isolation of Nanocrystalline Cellulose

There are several methods of isolating nanocrystalline cellulose (NCC), which include acid hydrolysis, enzymatic hydrolysis, mechanical treatment. Each of these methods has its advantages and disadvantages, depending on the desired properties of NCC and the specific application. Acid hydrolysis is the most widely used method due to its high yield and efficiency, but it requires harsh conditions and can affect the properties of NCC. Enzymatic hydrolysis and mechanical treatment offer milder conditions but require longer processing times and can be more expensive.

Acid hydrolysis

This is the most commonly used method for synthesizing NCC. In this method, cellulose fibers are treated with a strong acid, usually sulfuric acid, phosphoric acid, and hydrochloric acid, under controlled conditions of temperature and time. The acid breaks down the amorphous regions of cellulose fibers, leaving behind the crystalline fragments that form NCC.

Nanocrystals isolated with hydrochloric acid are neutral with high dispersability in water compared to hydrolysis with sulfuric acid which gives more stable nanocrystals [38].

Enzymatic hydrolysis

This is a well-established method for isolating cellulose nanocrystals (NCC) from cellulose fibers using enzymes. This process involves the use of enzymes such as cellulases to selectively break down the amorphous regions of the cellulose fibers, leaving behind the highly crystalline NCC [39]. Enzymatic hydrolysis is milder than acid hydrolysis and can produce NCC with different properties and morphology. The hydrolysis step is typically carried out at a controlled temperature and pH to optimize enzyme activity. The final step involves the isolation and purification of the NCC which typically involves washing the NCC with water and centrifuging to remove any residual enzymes or other contaminants.

Mechanical treatment

This method involves subjecting cellulose fibers to mechanical forces, such as ultrasonication, high-pressure homogenization, and ball milling [31]. Mechanical treatment can produce NCC with varying sizes and shapes, depending on the processing conditions. Ultrasonication method involves the application of high-frequency sound waves to break down the cellulose fibers into smaller particles, which can then be further processed to obtain NCC. Ball milling is a mechanical process that involves the use of grinding balls and a rotating container to effectively break down cellulose fibers and release the NCC.

5. Methods of characterizing NCC

There are several methods of characterizing nanocrystalline cellulose (NCC), which include X-ray diffraction (XRD), scanning and transmission electron microscopies (SEM and TEM), fourier transform infra-red spectroscopy (FT-IR), dynamic light scattering (DLS), and thermogravimetric analysis (TGA). Each of these methods provides different types of information about NCC, and the choice of method depends on the specific properties of interest and the purpose of the analysis. For example, XRD and TEM provide information about the crystal structure, morphology, and size of NCC, while DLS and FTIR provide information about the surface charge and chemical composition, respectively. Combining multiple characterization techniques can provide a more comprehensive understanding of the properties of NCC.

5.1. X-ray diffraction (XRD)

X-Ray Diffraction (XRD) is a powerful technique for studying the crystal structure and degree of crystallinity of nanocrystalline cellulose (NCC) particles. In XRD, X-rays are passed through a sample, and the diffraction pattern produced by the interaction of the X-rays with the atoms in the sample is detected and analyzed to determine the crystal structure and degree of crystallinity. Nanocrystalline cellulose have shown to have high degree of crystallinity [40, 41].

5.2. Transmission electron microscopy (TEM)

Transmission Electron Microscopy (TEM) is a high-resolution imaging technique that is widely used to study the morphology and structure of nanocrystalline cellulose (NCC) particles. TEM works by passing an electron beam through a thin sample, which interacts with the atoms and produces a high-resolution image of the sample. It can be used to determine the size, shape, and distribution of the particles. TEM results from several researchers showed that NCC are needle-like or rod like in shape [42, 43].

5.3. Scanning electron microscopy (SEM

Scanning Electron Microscopy (SEM) is a powerful imaging technique that can be used to study the surface morphology and topography of nanocrystalline cellulose (NCC) particles. In SEM, a focused electron beam is scanned across the surface of a sample, and the electrons that are scattered from the surface are detected to create an image.

5.4. Dynamic light scattering (DLS)

Dynamic Light Scattering (DLS) is a technique used to measure the hydrodynamic size and size distribution of nanocrystalline cellulose (NCC) particles in suspension. DLS measures the Brownian motion of NCC particles in solution and provides information about the size and distribution of the particles based on the rate and intensity of their movement.

5.5. Fourier transform infrared spectroscopy (FTIR)

Fourier Transform Infrared Spectroscopy (FTIR) is a powerful technique for studying the chemical structure and functional groups of nanocrystalline cellulose (NCC) particles. In FTIR, a sample is exposed to infrared radiation, and the resulting absorption spectrum is analyzed to determine the types of chemical bonds and functional groups present in the sample.

5.6. Thermogravimetric analysis (TGA)

Thermogravimetric Analysis (TGA) is a technique used to investigate the thermal stability and decomposition behavior of nanocrystalline cellulose (NCC) particles. In TGA, a sample is heated in a controlled environment and the resulting mass loss is measured as a function of temperature, providing information about the thermal stability and decomposition behavior of the sample.

6. Applications of nanocrystalline cellulose

Nanocrystalline cellulose (NCC) has a wide range of potential applications due to its unique properties, including high strength, high aspect ratio, and high surface area. Here are some examples of the applications of NCC:

6.1. Bio composites

Nanocrystalline cellulose (NCC) has been extensively studied for its potential applications as a reinforcing agent in biocomposites due to its high mechanical strength, low toxicity, and biodegradability [4]. Biocomposites are materials composed of both natural and synthetic components that can be used in various biomedical applications. A study on the use of NCC as a reinforcing agent in chitosan-based biocomposites showed that the addition of NCC significantly improved the mechanical properties of the biocomposites, including their tensile strength, modulus, and toughness [5]. The researchers also observed that the NCC-reinforced biocomposites exhibited better thermal stability and lower water uptake compared to the control samples. [44] investigated the use of NCC as a reinforcing agent in polyvinyl alcohol (PVA)-based biocomposites. The researchers prepared NCC using ball milling method and incorporated it into PVA films. They found that the addition of NCC significantly improved the mechanical properties of the biocomposites, including their tensile strength, modulus, as well as the thermal properties of the composite. They also observed that the NCC-reinforced biocomposites exhibited better thermal stability and lower water uptake compared to the control samples. NCC has also been investigated for its potential applications as a reinforcing agent in bone tissue engineering. [45] in their study investigated the use of Poly(lactic acid) (PLA)/cellulose nanocrystal (CNC) composite scaffolds for bone regeneration. They found that the composite scaffold exhibited better mechanical properties, including higher stiffness and compressive strength, compared to the pure polymer. The researchers also observed that the NCC-reinforced scaffold promoted the adhesion and proliferation of osteoblast cells, indicating that it could be used as a promising material for bone tissue engineering applications.

6.2. Food packaging

The unique properties of NCC, such as its barrier properties, biodegradability, and high mechanical strength, make it an attractive material for use in packaging applications. NCC can be incorporated into food packaging materials to enhance their barrier properties against oxygen, moisture, and other gases. Studies have shown that NCC-based films exhibit improved gas barrier properties compared to traditional packaging materials such as polyethylene and polypropylene [46]. NCC can improve the mechanical strength of food packaging materials, making them more resistant to tearing and puncturing [5]. This can help to prevent food spoilage and reduce the amount of packaging waste generated. A study found that NCC-based films exhibited higher tensile strength and elongation at break compared to pure cellulose films. NCC has also been investigated for use as an antimicrobial agent in food packaging. One study published in the journal Food Chemistry investigated the use of NCC coatings containing silver nanoparticles as a method of controlling microbial growth on food surfaces [47]. The researchers prepared NCC coatings and incorporated silver nanoparticles into the coatings. They found that the NCC-silver coatings showed high antimicrobial activities against gram-positive and gram-negative bacteria when added to pulp fibers.

6.3. Biomedical applications

NCC has potential applications in drug delivery, wound healing, and tissue engineering due to its biocompatibility and biodegradability. Tissue engineering involves the use of biomaterials to replace or repair damaged or diseased tissue. NCC can be used to create scaffolds for tissue regeneration due to its high surface area-to-volume ratio, which provides a large surface for cell attachment and growth. Additionally, the stiffness and strength of NCC can be tailored to match that of the surrounding tissue, which can promote integration with the host tissue. A study carried out by [48] investigated the potential of NCC as a scaffold for bone tissue engineering. The researchers prepared NCC scaffolds and seeded them with bone-forming cells called osteoblasts. They found that the NCC scaffolds supported cell attachment and proliferation, and promoted the formation of mineralized bone tissue. In addition to tissue engineering, NCC can also be used for drug delivery. NCC nanoparticles can be used to encapsulate and deliver drugs to specific targets in the body, such as cancer cells [49]. The high surface area-to-volume ratio of NCC allows for a large drug loading capacity, and the biodegradability of NCC ensures that the material is safely cleared from the body once the drug has been released.

6.4. Water Treatment

Nanocrystalline cellulose (NCC) has been investigated for its potential application in water treatment due to its unique physicochemical properties, including its high surface area, high aspect ratio, and strong adsorption capacity. NCC can be used as an adsorbent to remove various pollutants from water, such as heavy metals, dyes, and organic compounds [50]. Nanocrystalline cellulose have been shown to have high adsorption capacity for heavy metals from water [51]. Tsade Kara [52] used modified NCC as adsorbent for the removal of methylene blue from waste water. They found that the modified NCC exhibited a high adsorption capacity for methylene dye, with maximum adsorption capacities of 90.91 mg/g. The researchers also observed that the NCC adsorbent was highly stable and could be easily regenerated, indicating its potential for practical applications in water treatment. devices.

6.5. Energy Storage

Another application of NCC is in energy conversion devices such as solar cells [53]. It can be used as a transparent conductive material in the electrodes of solar cells, improving the efficiency of energy conversion by increasing light transmission and reducing electrical resistance. It has also been investigated as a potential material for dye-sensitized solar cells [54], which are low-cost and high-efficiency solar cells that use dye molecules to absorb light and generate electricity. NCC can also be used as a component in energy-efficient materials such as insulation and coatings. NCC has high thermal insulation properties due to its low thermal conductivity, making it a potential additive for thermal insulation materials. NCC can also improve the barrier properties of coatings and films, reducing energy loss due to air and moisture infiltration.

7. Conclusion

Nanocrystalline cellulose (NCC) has gained significant attention in recent years due to its unique properties and potential applications in various fields such as biomedicine, electronics, and packaging. The synthesis of NCC involves the extraction of cellulose fibers from natural sources such as wood pulp or agricultural waste, followed by the mechanical or chemical treatment to obtain nanocrystals with high aspect ratio and crystallinity. The characterization of NCC is crucial to determine its properties and potential applications. Various techniques such as X-ray diffraction, electron microscopy, and spectroscopy have been employed to study the crystal structure, morphology, and chemical composition of NCC. NCC has demonstrated promising applications in various fields such as drug delivery, tissue engineering, and packaging. Its biocompatibility, high surface area, and mechanical strength make it a suitable material for biomedical applications. In summary, NCC has emerged as a promising nanomaterial with unique properties and potential applications in various fields. Further research is needed to optimize the synthesis and processing methods of NCC and explore its full potential in commercial applications.

Compliance with ethical standards

Acknowledgments

Authors would like to extend sincere gratitude to all those who contributed to the review one way or the other.

Disclosure of conflict of interest

There is no conflict of interest.

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