



Cellulose-Based Materials for The Consolidation of Archaeological Wooden Artifacts: Review Article

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HIGHLIGHTS

- Cellulose derivatives are highly compatible with wood and are commonly used to treat wooden artifacts.
- Cellulose nano filler possess outstanding properties compared with the original cellulosic fiber
- Cellulose nanocrystals are crystalline rod-like nanofibers obtained by acid hydrolysis.
- Cellulose nano fiber consists of pure cellulose from both crystalline and amorphous regions.
- Cellulose-based materials in composite systems present superior Mechanical properties.

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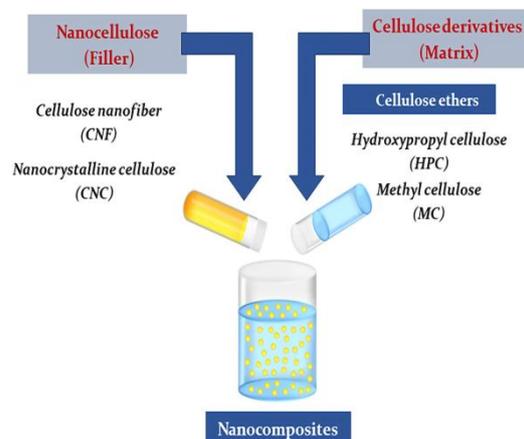
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GRAPHICAL ABSTRACT



ABSTRACT

There has been a continuous search for suitable polymers that could be used for the treatment of decayed archaeological wood, without causing further deterioration to the treated artifacts. Paraloid®, polyvinyl acetate, alum, Polyethylene glycol (PEG) or cellulose derivatives have been commonly used in the past, but the utilization of cellulose-based materials had been limited, due to their poor mechanical properties. Remarkable attributes including mechanical properties enhancement, optical appearance that may be adjusted, and consistent performance have been demonstrated by cellulosic materials in recent times.

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Cellulose derivatives, which are obtained by substituting some, or all, of the hydroxyl groups on the repeating unit in cellulose, based on the type of the desired property, are divided into two large categories, cellulose ethers and cellulose esters. These derivatives can be used as a matrix or filler in composites, and they are currently used in their nano size. The current interest in the nanocomposites is a result of the fact that the polymer matrix and nano filler interact with each other on molecular level, and can be used in the treatment of archaeological wood. In this article the general idea of cellulose-based materials and their nanocomposites is reviewed, aiming to focus on the possibility of expanding its use in the treatment and consolidation of decayed wooden artifacts.

1. Introduction

Since the early stages of human history, wood has been widely used in various fields of art and construction. Therefore, there is a large number of ancient wooden artifacts all over the globe that represent the cultural heritage of humanity. These artifacts constantly encounter various biological or chemical degradation factors, which in many cases results in reduced mechanical and chemical stability [1] [2] [3]. Wood also decays and loses its structural integrity under suitable temperature and humidity conditions [4]. To maintain the history of humankind, procedures to conserve and protect these artifacts is of utmost importance. Hence, the preservation and reinforcement of these degraded wood pieces of traditional wooden structures is essential [5].

Wooden artifacts are often affected by different degradation factors, such as microbiological infestation or exposure to weathering. Several studies have been conducted using Fourier Transform Infrared Spectroscopy (FTIR) spectroscopy to study the chemical changes that occur in some commonly identified softwoods [6], [7] and hardwoods [8] [9] [10]. Results showed that, depending on the decay factors, main components of wood such as cellulose and lignin are affected, thus resulting in different deterioration features leading to a change in physical appearance of wood and a decrease in its mechanical properties.

Wood loses its main components, namely carbohydrates and lignin, in different proportions, when it is attacked by cellulolytic microorganisms, the carbohydrate content decreases dramatically and a rise in lignin percentage occurs [11]. In a study on 11 different softwood samples, that had been affected by different decay factors, the decrease or

increase in the lignin/carbohydrate ratio was calculated and results indicated that it is extremely difficult to estimate the extent of decay in any type of wood without analysis [6].

Due to the aforementioned decay of wood, there was a continuous search for suitable polymers that could be used for wood treatment, and archaeological wood was often consolidated/reinforced with several materials such as Paraloid®, polyvinyl acetate emulsions, alum and PEG [12].

Paraloid B72 and polyvinyl acetate are the most common materials used in the last decades in the consolidation of various artifacts, including wooden objects. The results of earlier treatments with those consolidants were judged to be unsatisfactory, because they altered the surface appearance, attracted excessive amounts of dirt, usually contracted, embrittled and darkened on aging.

Recent research has highlighted challenges associated with the removal of Paraloid B72 from wooden surfaces, indicating difficulties in its extraction, while Paraloid B72 has shown success in treating both organic and inorganic materials, its application on wooden artifacts has faced scrutiny due to challenges in removal and potential long-term effects on the wooden substrates [13]

Polyvinyl acetate (PVAc) was also commonly used due to its adhesive properties, however, there are disadvantages associated with the use of PVAc in wooden artifacts. One significant issue is the potential for plasticizer loss over time, which can lead to degradation of the material [14]. Moreover, the use of PVAc in wooden artifacts may also pose challenges related to its stability and long-term preservation. For instance, the saponification of PVAc can lead to the formation of poly(vinyl alcohol) (PVA), which

may alter the properties of the material and impact its structural integrity [15]. Furthermore, the controlled radical polymerization of vinyl acetate, which is used to produce PVAc, can offer insights into the molecular weight control and structural features of the polymer [16]. It is increasingly evident that the use of acrylics like Paraloid B72 and PVA in the conservation of wooden artifacts may not be the most optimal choice. Research suggests that exploring alternative materials that are easier to remove, cost-effective, and provide comparable or enhanced consolidation properties could be advantageous for the long-term preservation of wooden objects.

Because cellulose derivatives are highly compatible with wood, conservators frequently utilized them to treat different kinds of wooden artifacts. Cellulose ethers were considered for use in the treatment because of their good aging properties and their versatile solubility in a range of organic and aqueous solvents. They play a crucial role in artifact conservation, particularly those based on cellulose and collagen such as paper, wood, parchment, and leather providing solutions for pH adjustment, consolidation, and protection of various cultural heritage objects. Their diverse properties and applications make them indispensable in the preservation of cellulose-based artifacts across different industries and conservation practices [17].

In an experimental study, several composites of gap fillers were evaluated, carboxy methyl cellulose in sodium salt samples gave better results than different acrylics like Paraloid B72, epoxy and poly vinyl acetate [18]. In another experimental study the application of methyl cellulose as a consolidant was evaluated on both sapwood and heartwood of an ancient part of a tree trunk, and scanning electron microscope images indicated that the secondary walls of the wood remained detached after treatment [19].

However, biocompatibility, customizable optical appearance, and stable performance are only a few of the intriguing qualities that cellulosic materials have demonstrated recently [20]. Conservators are unable to employ them for processes that require an ex-

tensive amount of mechanical support or for entirely and severely damaged objects due to the lack of significant mechanical properties [21]. For this reason, it was necessary to find a way to improve their mechanical properties [22].

The field of conservation and treatment of historical wood has embraced nanotechnology, which offers very promising ways to create nano-composites by adding nanomaterials as fillers. These fillers can improve the mechanical [23] and optical properties, depending on how they interact with the matrix [24]. It was introduced in the field of conservation and treatment of historical wood by focusing on the consolidation of wood samples, for antimicrobial protection, and deacidification (i.e., neutralization of acidic compounds). The shift towards using cellulosic materials in the conservation of wooden artifacts is driven by their renewable nature, enhanced mechanical properties, environmental friendliness, and effectiveness in protecting against degradation factors. These materials offer a sustainable and effective solution for preserving cultural heritage objects made of wood, paper, and fabric, ensuring their longevity for future generations.

Cellulose-based materials, such as cellulose nanocrystals and cellulose nanofibrils, have been found to significantly enhance the mechanical properties of composites, making them a preferred choice for fabricating materials from renewable resources [25].

The aim of this article is to review and discuss the uses of cellulose-based nanocomposites on wood, both as matrix and fillers in various fields of research, and specifically on wood by shedding light on the following main points:

- **Cellulose as source material**
 - **Cellulose derivatives (Matrix)**
 - **Cellulose ethers**
 - **Hydroxypropyl cellulose (HPC)**
 - **Methyl cellulose (MC)**
 - **Cellulose Esters**
 - **Cellulose Nitrate (CN)**
 - **Cellulose Acetate (CA)**
 - **Micro and Nanocellulose (Filler)**
 - **Types of cellulosic fillers according to size**

- Microcrystalline cellulose (MCC)
- Microfibrillated cellulose (MFC)
- Cellulose nanofiber (CNF)
- Cellulose Nanocrystal (CNC)
- Bacterial nanocellulose (BNC)
- Nanocomposites
 - Nonpolar polymer nanocomposites
 - Polar polymer nanocomposites

2. Cellulose as source material

Cellulose is considered the most common and renewable type of semi-crystalline natural polysaccharide. This biopolymer is an endless supply of natural fibers (NFs) and a valuable raw material for the synthesis of cellulose derivatives, nano-products, such as nanocellulose and microcrystalline cellulose (MCC), which are widely used in many fields of conservation especially organic artifacts [26].

Cellulosic materials have become a major area of study because of the increased need for items made from renewable resources that are biodegradable and pose no danger to the environment [27]. They possess distinguishing features including biocompatibility, stable performance, and adjustable optical appearance alongside with the high sorption capacity [28].

The building blocks of cellulose are hundreds to hundreds of thousands of β (1–4) linked glucose units, each of which has three carbon atoms connected to hydroxyl groups either directly or indirectly Fig (1). The hydroxyl groups in cellulose fibrils are in charge of combining and strengthening the crystalline matrix structure. [29]

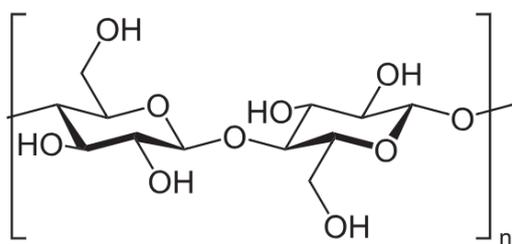


Fig. 1. Chemical structure of Cellulose [30]

3. Cellulose derivatives (Matrix)

Cellulose derivatives are produced from naturally existing raw materials using inexpensive chemical modification techniques. They are created by substituting some or all of the hydroxyl groups on the repeat unit in cellulose. This class of materials is seen to be very promising. A widely used technique for modifying cellulose chemically entails the well-known hydroxyl group oxidation, etherification, and esterification processes. Depending on the kind of required property, the derivatives are produced by substituting all or part of the hydroxyl groups on the cellulose repeating unit [31]. Two broad categories—cellulose ethers and cellulose esters—are used to group the derivatives [22].

Some cellulose derivatives can be classified as cellulose ether-esters, with some additional ester functions localized on the ether branches [32]. The degree of substitution (DS), defined as the average number of etherified hydroxyl groups in a glucose unit, can be controlled to a certain extent to obtain cellulose derivatives with given solubility and viscosity in water solutions [33].

3.1. Cellulose ethers

Cellulose ethers have been widely used as consolidant for almost a century [34]; especially cellulosic materials such as wood, either in consolidation [35], [12], [36] or gap filling [37], [38], [39].

The process of etherification of cellulose, which includes the interaction of cellulose's hydroxyl groups with organic species such as methyl and ethyl units, yields cellulose ethers [33]. Alkaline solutions are used for their manufacture, and one of the most commonly used methods is oxygen alkylation using alkyl halides [40] forming alkyl ethers [40] that are used as polymer coatings or film, e.g. hydroxypropyl cellulose (HPC), hydroxypropyl methylcellulose (HPMC), carboxymethylcellulose (CMC), and methylcellulose (MC) [23] [33].

They can form films, depending on the DS, and have moderate toxicity and excellent chemical stability [40] [23]. The degree and type of substitution, coupled with the placement of ether groups along the chain, can influence the solubility of cellulose ethers in water to some amount [40], while

native cellulose is a water-insoluble compound [23].

Because polysaccharides are naturally hydrophilic, they have poor mechanical and water vapor barrier qualities. Therefore a filler can be added to strengthen these weak characteristics and create a composite film [23].

3.1.1. Hydroxypropyl cellulose (HPC)

Klucel E [hydroxypropyl cellulose (HPC)], a specific type of cellulose derivative that lies under the category of cellulose ethers, is synthesized via the reaction of propylene oxide with alkali cellulose at high temperature and pressure to obtain a substituted cellulose ether where the cellulose backbone is hydroxy propylated [41] (Fig. 2).

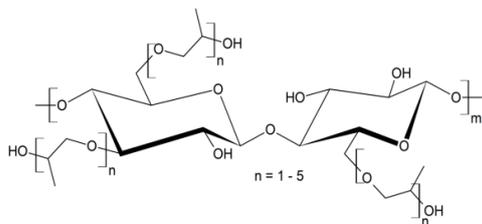


Fig. 2. Chemical structure of Hydroxypropyl cellulose [42]

Due to its remarkable combination of qualities, including substantial flexibility and resistance to sticky growth in the presence of humidity, it is thermoplastic; nonetheless, its mechanical properties are rather weak. This polymer exhibits both organic and water solubility, which is a significant advantage over cellulose, which is insoluble in a variety of solvents, including acetone, methanol, and ethanol [7].

Because hydroxypropyl cellulose is compatible with the chemical makeup of the artifacts, it is currently widely utilized in the field of wood conservation and other organic objects. HPC Klucel® J and G, like other cellulose polymers, do not seriously darken pigments, and have therefore been utilized for leather and pigment consolidation as a 2% solution in ethanol [43]. It also has been applied as a film to adhere between layers, to make a solvent-swollen poultice, to aid the removal of wax lining on paintings [44], in textile consolidation [45] and paper con-

servation [46]. Fulcher demonstrated how Klucel G, which can be produced using a range of solvents appropriate for certain artifacts, exhibited nearly all the qualities required as gap fillers for wooden artifacts when combined with paper pulp. It is possible to tune the adhesive's concentration to match an object's specific requirements by adjusting the hardness and strength [37] via changing the preparation solvents [47].

Although HPC is being widely used in the conservation and treatment of ancient cellulosic materials such as wood [35], [38], textile and manuscripts [48] there are insufficient reported results about its mechanical properties or any further modifications that can enhance its properties especially when employing it in the synthesis of nanocomposites.

3.1.2. Methyl cellulose (MC)

Methylcellulose has been widely used for many years to produce gels and fine chemicals in pharmaceuticals, foods, construction, paints, ceramics, detergents, agriculture, polymerization, adhesives, and cosmetics. It is a derivative of cellulose and can be produced from cotton cellulose, wood, and annual plant pulps. It is produced by chemical treatments via alkali cellulose using concentrated NaOH solution. Alkali-cellulose is then treated with methyl halide (or dimethyl sulfate) to produce MC [49] Fig (3).

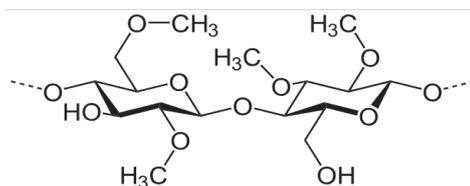


Fig. 3. Chemical structure of Methyl cellulose [30]

It is resistant to water and is the lowest hydrophilic cellulose derivatives [23], and shows good solubility in water at low temperature [49].

In paper conservation water soluble MC has a long record of usage as adhesive, size and as consolidant [50] [51] [52]. Its solutions have been used as a reversible facing on wall paintings, as a consolidant for water-

logged wood [53], for relining of canvas [21], and as an adhesive for textiles [54].

It was used in conservation either alone or as a bio-based additive because of its attractive properties such as transparency, non-toxicity [55], and sustainability. MC is also considered better than other alternatives in consolidation of plant-based materials as it is from the same resource and has the same component which makes it less vulnerable to cause dramatic changes or deterioration of archaeological materials.

Its “large molecular weight that results in high viscosity” reduces the amount of material that can enter the wood because of poor penetration, which results in poor treatment quality. Moreover, MC can easily be deteriorated due to its biodegradability [36]. Therefore, it was necessary to look for a method to improve its properties [56] [57] for example via incorporation of a reinforcing agent to make a (nano)composite. The use of fiber-like reinforcements to improve the properties of a matrix has been the subject of numerous scientific articles [58] [59].

3.2. Cellulose esters

In the field of conservation, cellulose esters were not frequently utilized. They are created by substituting organic or inorganic acids for the hydroxyl groups on cellulose. A wide range of properties can be obtained based on the kind, degree, and location of the substituents along the cellulose chain. Cellulose acetate (CA) and cellulose acetate propionate (CAP) are two examples of common cellulose esters [22]. Although several cellulose esters have been produced, only acetate and nitrate have been widely employed in conservation as follows:

3.2.1. Cellulose nitrate (CN)

Cellulose nitrate was one of the first cellulose derivatives and was discovered in 1833 [34]. It was prepared by soaking cellulose pulp in a mixture of concentrated nitric and (usually) sulphuric acid Fig (4).

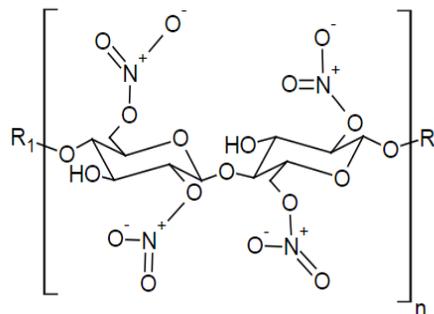


Fig. 4. Chemical structure of cellulose nitrate [60]

CN has a high glass transition temperature and has strong intermolecular polar and hydrogen bonds that produce higher solution viscosity, though it releases solvent more quickly than other polymers. It has retained its usefulness as a lacquer and adhesive because it rapidly releases solvents to form a dry, strong film [61]. Plasticized cellulose nitrate was first used for the conservation of objects during the late nineteenth century. It has been used for the coating of antiquities and strengthening of paper, and remained an adhesive, consolidant and coating in many applications for the first half of the twentieth century [44]. Typical uses were as a consolidant for stone, wall paintings, pigments, plants, wood and general organic materials. It was also used as an adhesive for pottery and as a coating for unstable glass and stained-glass paint. CN was replaced for some purposes during the 1920s and 1930s by cellulose acetate (CA) and poly vinyl acetate (PVAc) [61], as it proved to be an unstable material. CN degrades at room temperature by a combination of oxidation and hydrolysis, which are catalyzed by the presence of acid impurities and accelerated by light. There has been considerable disagreement over whether CN should be used in conservation and some conservation specialists do not recommend it [62].

3.2.2. Cellulose acetate (CA)

Cellulose acetate is manufactured by heating cellulose with acetic anhydride and a sulphuric acid catalyst. The chemical properties of CA change with increasing degree of substitution. The acetyl groups can be added at three places on the fundamental unit (Fig 5) [63].

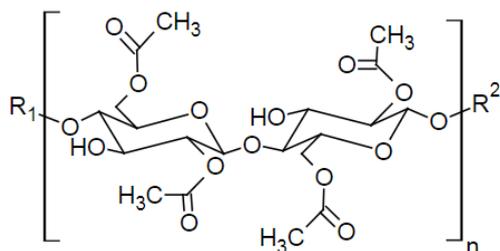


Fig. 5. Chemical structure of cellulose Acetate [60]

The polymer is soluble in acetone and similar solvents. Cellulose acetate became widely available during World War I as a coating for aircraft fabric, and replaced cellulose nitrate (CN), which was too inflammable. It was first used, in a solution in acetone for fixing pigments on paper and stone and as a consolidant for cloth and other materials. In 1937 it was rejected as a possible picture varnish, and it has been largely replaced for conservation uses by modern materials [44]. The greatest quantity of CA was used in the lamination of paper, starting around 1934, and has been widely used to copy cinema film images off old supports of CA or CN. However, much of the film stock used has deteriorated severely by metal-catalyzed degradation and hydrolysis of the polymer [64].

4. Micro and nanocellulose (Filler)

Nanocellulose (NC) is a cellulose derivative composed of a nanosized fiber network, which determines the product properties and its functionality. Plant-derived nanocellulose is derived from the top-down mechanical and/or chemical procedure of cellulosic precursors [65]. NC fibers are very interesting nanomaterials for production of cheap, lightweight, and very strong nanocomposites

[66]. A considerable amount of research has been done on the isolation of these nanofibers from plants for use as fillers in bio composites [49].

4.1. Types of cellulosic fillers according to size

Wood pulp or plant cellulose can be treated chemically or mechanically to obtain nanocellulose [65]. The term "nanocellulose" refers to cellulose fibril or crystallite that has at least one dimension in the nanoscale range. By varying their source, many types of nanocellulose can be produced, including bacterial, tunicate, algal, vegetal, and lignocellulosic materials [67]. There are different forms of nanocellulose depending on the preparation technique, such as (i) nano crystalline cellulose produced by acid hydrolysis and (ii) nano or micro fibrillated cellulose produced by mechanical means. These nanocellulose materials have been applied to polymer matrices as a reinforcing agent [65]. Generally, the family of nanocellulose can be divided in three types:

1. Cellulose nanocrystals (CNC), with other designations such as nanocrystalline cellulose, cellulose (nano) whiskers, rod-like cellulose microcrystals [68] [69].
2. Cellulose nanofibrils (CNF), with the synonyms of nanofibrillated cellulose (NFC), microfibrillated cellulose (MFC), cellulose nanofibers.
3. Bacterial cellulose (BC), also referred to as microbial cellulose [70] [71].

The Technical Association of the Pulp and Paper Industry (TAPPI) proposed standard terms and their definitions for cellulose nanomaterial WI 3021, 21, based on the nano cellulose size; microcrystalline cellulose (MCC), cellulose microfibril (CMF), cellulose nanofiber (CNF) and cellulose nanocrystal (CNC) [72].

Different cellulose sources give different characteristics and also different aspect ratios (L/d, where L is the length and d is the diameter). Almost particular crystals have a low aspect ratio (L/d = 1), while microfibrils can have a very high aspect ratio due to its small diameter (nm) and long fibrils (nm- μ m). The higher the aspect ratio, the higher is the reinforcement capacity when incorpo-

rated in composite materials. Also, the more surface area of the fillers, the more contact will the filler have with the polymer matrices [73] [74].

4.1.1. Microcrystalline cellulose (MCC)

Micro crystalline cellulose is typically derived from the acid hydrolysis of cellulose, which is a low cost production process. The acid hydrolysis preferentially removes the amorphous regions of cellulose [75]. The resulting MCC particles are porous, insoluble in water, possess high cellulose content, high aspect ratio (>1000), and high crystallinity [76].

MCC is insoluble in common solvents, however, it leads to the formation of colloidal suspensions when suspended in water [77]. It consists of large multi sized aggregates of nanocrystals that are bonded to each other. Commercial MCC can have spherical or rod-like particles with sizes of 10–200 μm [72] [78].

The high crystallinity of MCC delivers a strong reinforcing ability due to the high modulus. Therefore, MCC has been widely used as fillers in various polymers composite [79]. Since it is known that crystalline cellulose is much stronger and stiffer than the amorphous cellulose and cellulose itself, the production of MCC can be a better reinforcing agent than cellulose [80].

4.1.2. Microfibrillated cellulose (MFC)

It is determined that cellulose microfibrils are a type of fine cellulose with several elementary fibrils, such as the aggregated crystalline-amorphous region with a high aspect ratio and surface area [65].

The manufacture of MFC is generally performed by a mechanical treatment. The fundamental idea behind their techniques involves running diluted solutions of cellulosic wood pulp and water through a mechanical homogenizer. Another technique for creating nanofibers is called cryocrushing, which involves freezing fibers with liquid nitrogen and then applying strong shear stresses. More recently, wood pulp milling was also used to create NFC. By transferring the cellulose slurry between a static grind stone and a rotating grind stone throughout the grinding process, cellulose fiber fibrilla-

tion is achieved [81]. Reducing energy consumption is possible by combining the mechanical treatment with specific pretreatments like dewaxing, alkaline treatment, lignin hydrolysis, enzymatic bleaching process, and TEMPO-mediated oxidation to remove non-cellulosic materials like waxes, lignin, hemicellulose, and other impurities [82], [83], [84], [85].

The resulting MFC particles are made up of roughly individualized cellulose microfibrils and have a spaghetti-like form. They are also lengthy and flexible [72]. MFC can be utilized as reinforcing materials in composite films because of its strong web-like network formation ability and high elastic modulus [75]. The produced cellulose microfibrils are either used for transformation to CNC via acid hydrolysis or to CNF by mechanical processing using a variety of techniques, such as ball milling, high pressure homogenization, and ultrasonication. [85].

4.1.3. Cellulose nanofiber (CNF)

Comparable to MFC, CNF can form entangled networks since it is entirely made of pure cellulose from both crystalline and amorphous areas. The large range of size distribution observed in CNF is typically attributed to the energy required to break down the cellulose fibers and the pretreatment applied to speed up this process [86].

As reinforcing materials, MFC and CNFs are both predicted to have large aspect ratios and similar fibrils. However, CNFs have significantly thinner fibrils and complicated fibril networks thanks to protracted mechanical fibrillation, which MFC does not have [87].

CNFs, often referred to as nanofibrillated cellulose (NFC), are flexible structures that resemble fibers and have a diameter of less than 100 nm and a length of 100 nm to 500 nm or more [88]. They are acquired by means of particular methods that promote fibrillation during the process of mechanically refining plant and wood fiber [89].

Fibrillation, which yields finer particle diameters, is the mechanism that distinguishes NFC from MFC. However, there is occasionally confusion in the literature due to the

interchangeability of MFC and CNF terms [76] [90].

When there is a good dispersion of the nanofibers in the matrix, interactions between the nano sized elements produce a percolated network connected by hydrogen bonds, which is why cellulose nanofibers are acknowledged as being more effective than their micro sized counterparts to reinforce polymers [49]. Depending on their origin, the defibrillation process and the pretreatment have varying degrees of polymerization and chain lengths. Fiber-rich cellulose derived from wood has a length of approximately 180–200 nm, whereas cotton cellulose has a length of 100–120 nm, and tunicate cellulose has a length of up to 1000 nm [27]. Stretched bundles (aggregates) are constructed from alternating crystalline and amorphous domains that can be 20–50 nm in width and 500–2000 nm in length [72] [65], or can vary from 10 to 100 nm [91].

The mechanical breakdown of the original cellulose microfibers into nanofibers, which frequently requires numerous passes through the disintegration device, has a high energy consumption [70]. TEMPO-mediated oxidation (2,2,6,6-tetramethylpiperidine-1-oxyl radical), enzymatic hydrolysis, multi-pass high-pressure homogenization, and direct mechanical fibrillation represent a few of the methods that can be used to create fibers. CNF sizes and forms can differ significantly based on the degree of fibrillation and any pretreatment used [87].

Plant-based fibers are superior to other sources when it comes to treating archaeological wood because they require smaller fiber dimensions for better penetration [12]. The CNF features make them excellent candidates for usage in nanocomposites since they interact physically with the polymeric chains in addition to displaying intriguing mechanical and thermal properties [92].

4.1.4. Cellulose nanocrystal (CNC)

CNC represents the smallest, most fundamental building block in plant's cell wall. The crystalline content of cellulose in biomass ranges from ~25 to 75% [93]. The de-

gree of crystalline ordering is highest in the nanocrystal films (~87%), whereas the MFC exhibits a degree of crystallinity of about 60%, while the crystallinity of CNCs is generally > 80% [94].

A variety of biological materials, including wood, cotton, rice husk, wheat straw, bamboo, potato tubers, sugar beet, ramie, bacteria, and algae, are used to make CNCs [95] [96], in addition to other sources (e.g., bleached wood pulp, manila, tunicin, etc.) [87]. The particles are produced by hydrolyzing the nanofibrils with extremely concentrated acids (6–8M), which also break local crystalline connections between the nanofibrils [97], which include sulfuric, phosphoric, and hydrochloric acids, each of them donates particular functional groups to the surface of the nanoparticles that affect their colloidal stability [70]. The hydrolysis of glycosidic bonds occurs in the early stages of the process when the acid diffuses into the non-crystalline regions of the cellulose fiber. The hydrolysis of the polymer then proceeds to the more accessible glycosidic linkages, ultimately taking place at the surface of the nanocrystals and at the reducing end group [98] [74]. After this chemical process, strong mechanical or ultrasonic treatments are applied [99] to remove amorphous regions in the cellulose [71].

Cellulose nanocrystals, often known as "whiskers," are elongated crystalline rod-like nanofibers that are produced by acid hydrolysis at regulated temperatures and times. Colloid suspensions of distinct cellulose nanocrystals are produced by the acid hydrolysis, which is selective in its breakdown of amorphous holocellulose [100]. Geometrical dimensions of CNCs can vary widely [99], and it has been reported that as compared to CNCs made from wood or cotton, nanocrystalline particles isolated from tunicates and BC are often larger. This is due to the fact that while CNCs derived from pure cellulose materials show enhanced crystallinity, tunicates and BC are highly crystalline and have longer nanocrystallites [101].

Table (1): Length, Diameter and Ratio for nanocellulose according to source

Cellulose source	Length, L (nm)	Cross section, D (nm)	Axial ratio, L/D	References
Wood	180 nm +/- 80 nm	3.5	30-70	[103]
	100-300	3-5	30-70	[74], [104]
Cotton	100-400	7-15	10-20	[74], [104]
	170	15	10	[105]
	115	15		[74]
Bacterial cellulose	100 nm- μ m	5-10	N/A	[104]
Wheat	220	5		[74], [105]

The dimensions of a cellulose nanocrystal is determined by the type of source (Table 1); Mishra, Espinosa, Borjesson, Wu, Lin, and Dufresne describe CNCs as needle-shaped cellulose crystals that range from 10–20 nm in width and several hundred nanometers in length (100–250 nm) [65], [70], [74], [99], [102].

High surface area (150–250 m²/g) and a profusion of surface hydroxyl groups are just two of the numerous advantageous physical and chemical characteristics of CNCs [106], [107]. The benefits of CNC include low density, biocompatibility, and biodegradability. Several studies have shown that adding CNC to biopolymers can produce bio-nanocomposite materials with excellent mechanical, thermal, and barrier qualities [108]. Because there are many exposed hydroxyl groups, they have reactive surfaces and thermal stabilities up to about 300°C. These surfaces can be easily altered to provide a variety of surface characteristics, allowing for the control of interfacial properties in composites and the dispersion of a broad range of suspensions and matrix polymers [106].

CNCs are about 100% cellulose, with a high aspect ratio (3-5 nm wide by 50-500 nm long), and are extremely crystalline (54–88%) [76]. When processing nanocomposite materials with a polymer as the matrix, particle fillers (L/D) ranging from almost one to around one hundred can be employed [77].

Particles of nanocrystalline cellulose have superior mechanical qualities. It is calculat-

ed that a CNC along the cellulose chain axis has a theoretical Young's modulus of 167.5 GPa. Cotton CNCs have an experimental Young's modulus of 105 GPa, while tunicate CNCs have a modulus of 143 GPa. [109]. Dri et al. measured the Young's modulus of crystalline cellulose, which predicted the modulus of crystalline cellulose as high as 206 GPa [110] [70]. A wide variety of values were reported, but it is widely agreed upon that the crystalline cellulose's Young's modulus (assumed to represent the CNC's) should fall between 100 and 200 GPa. This range yields specific values that are comparable to those of Kevlar (60–125 GPa) and may even be stronger than steel (200–220 GPa) [70]. Crystalline cellulose is thought to be a superior reinforcing agent than amorphous cellulose since it is stiffer (Young's modulus) and much stronger (tensile strength 7500 MPa) than cellulose itself [111].

Over the past twenty years, cellulose nanocrystals and cellulose nanofibrils have been the primary subjects of investigation due to their intriguing mechanical characteristics [112] [113]; these materials demonstrate a high degree of mechanical resistance, with an estimated modulus of elasticity ranging from 50 to 145 GPa [114]. Because of its higher crystallinity than CNF, CNC, which has an elongated rod-like structure, has far less flexibility [97]. Their dimensions and crystallinity are the primary distinctions between CNC and CNF. While CNCs are extremely crystalline and typical-

ly have a length of less than 500 nm, CNFs are composed of mixtures of crystalline and amorphous cellulose chains that can reach several microns in length [107].

4.1.5. Bacterial nanocellulose (BNC)

Specific bacterial strains from the genus *Achromobacter alcaligenes* and *Gluconacetobacter*, specifically *Gluconacetobacter xylinus* and *Acetobacter G. xylinus* species, have been shown to directly produce BC at industrial production levels. These strains are the source of bacterial nanocellulose [115] [116].

A typical cross-section diameter of 20–100 nm and a degree of polymerization (DP) of 4000–10,000 nm are seen in BNC glucose chains, which have DPs ranging from 2000–8000 and diameters in the range of 20–100 nm [117]. Because of this, the cellulose chains in BNC are substantially longer than those in cellulose derived from plants. BNC is also purer than cellulose derived from plants, because it does not include any lignin or hemicelluloses. It has a high water content at least 90%—and a high degree of crystallinity. The material's crystalline structure gives it excellent mechanical qualities [22] and structured fibrils less than 100 nm wide that are made up of a bundle of even smaller nanofibrils (2 to 4 nm in diameter) make up the final cellulose network structure [118].

BNC can be made to order in an astounding variety of patterns, including fleeces, foils, spheres, tubes, fibrous aggregates, or pulp [119] [65]. It has been used in food products, textiles, cosmetics, and other industries. It also has great potential for use in healthcare applications [116].

5. Nanocomposites

A blend of two or more distinct components, one of which serves as a filler or reinforcer and the other as a resin or polymer matrix, with one or more phases that are bi-based or biodegradable, is sometimes referred to as a biopolymer composite material [120] [22]. PNCs (polymer nanocomposites) usually have higher thermal stability, lower gas permeability (higher barrier capacity) and improved mechanical properties, espe-

cially increased abrasion resistance in relation to matrix polymer [120].

In general, nanocomposites consist of two phases, where one of the phases has a minimum of one dimension within the nanometer range (1~100 nm) [121] [82]. The molecular interactions between the polymer matrix and nano filler are what give nanocomposites their appeal. Because of this, certain properties of the composite material may be significantly altered by adding a little amount of nanofiller—typically a few percent—to the matrix that has dimensions smaller than 100 nm [120].

Pickering emulsion, melt processing, solution casting, electrospinning, 3D printing, and in situ polymerization are the five to six techniques used to create cellulose polymer nanocomposites. Combinations of these two or three approaches may be employed in specific situations.

The most popular and straightforward method for creating cellulose nanocomposite is solution casting, fig (6). The interaction between the dispersed polar cellulose filler and the polar polymers is high for water soluble polymers. These two ingredients are mixed to create a suspension, which is then used to cast the mixture into a mold and evaporate the solvent to create a solid nanocomposite [84]

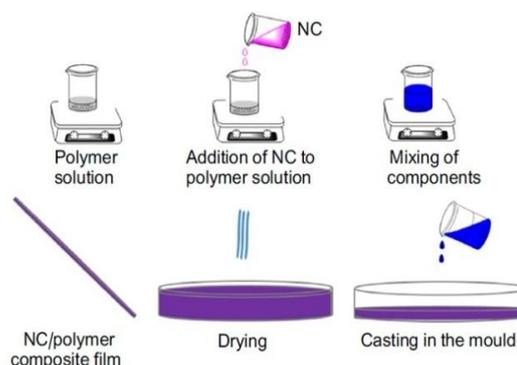


Fig. 6. Solution casting technique for preparation of Nanocomposite

In 1995, Favier et al. published the first study on the use of cellulose nanofibers as a reinforcing component in composite materials [122] [82]. The integration of cellulose fibers and derivatives as fillers and polymer

matrices into biopolymer composites and composite films is the current focus of interest in the utilization of these materials to generate cellulosic biopolymeric products worldwide [106]. When compared to standard composites, nanocomposite materials have better thermal, mechanical, barrier, recyclability, transparency, and low weight—even at low volume fraction of nanofibers [121] [82]. Nanocellulose's large surface area (150–250 m²/g), high tensile strength (7.5–7.7 GPa) with Young's moduli of 110–220 GPa [123], excellent optical properties, low density, and biodegradability have all contributed to its recent rise in popularity in the field of nano hybrids for the creation of bio-based nanomaterials and composites [124].

Additional studies investigated natural biopolymers to create environmentally friendly bio-based composites made of renewable resources, with the goal of improving the products' biodegradability, edibility, and sustainability while also making a significant contribution to the reduction of the impact of using fossil fuels [125]. Proteins, fatty components, and polysaccharides derived from both plant and animal sources are typically utilized for this objective [126].

5.1. Nonpolar polymer nanocomposites

The polarity and the tortuosity of nanocellulose make the composites resistant to non-polar organic solvents, a remarkable decrease in the diffusion coefficient and the equilibrium solvent absorption is noticed and this behavior increased with increasing the nanofiber content [127].

The possibility of dispersing cellulosic nanofibers in non-aqueous media has been investigated using surfactants or chemical grafting, and it opens other possibilities for nanocomposites processing. [128].

The presence of hydroxyl groups in the nanocellulose surface provides a platform for chemical functionalization reactions, such as oxidation, etherification, esterification, sialylation, and polymer grafting, enabling the nanocellulose to disperse more effectively in various types of polymer matrix materials. Thus, nanocellulose can be used as a nano filler [22], [76], [117].

Chemical modifications in nanocellulose are mainly performed to increase the incorporation of the nanocellulose with a polymer matrix [74]. There are several ways to change the surface of nanocellulose, particularly modification of nanocellulose by utilizing the polymer, chemical procedure, nanoparticles, employing an organic or even inorganic surfactant, biochemical procedure [129].

The surface chemical modification of cellulose whiskers is another way to disperse cellulose whiskers in organic solvents. It generally involves reactive hydroxyl groups from the surface of the whiskers [130], [131], [132].

5.2. Polar polymer nanocomposites

Several studies have been established to examine the effect of modifying different polar polymers with crystalline nanocellulose on the mechanical properties of those polymers.

Two aqueous solutions can be combined and cast to create solid nanocomposite films, and then the water can be evaporated [128]. Due to the great stability of aqueous cellulose whisker dispersions and the anticipated high degree of filler dispersion within the host matrix in the final composite film, water is the recommended processing medium. Because of this, the selection of the matrix is limited to water soluble polymers [131].

Bilbao-Sainz, Bras, et al proved that when whiskers were utilized as the filler, the elongation of the films at break was maintained while a 22% increase in tensile strength and a 55% rise in Young's modulus were obtained in HPMC. The composite films' ability to withstand water was further enhanced by the addition of whiskers. This result was ascribed to the composite films' decreased water affinity [69]. Patel, Dutab and Lim in 2019 made a nanocomposite of Nanocellulose/carboxy methyl cellulose (NC/CMC). Strong hydrogen bonding between the polymer matrix and the CNC facilitated effective stress transmission, which improved mechanical responses, including stiffness and strength [117]. Additionally, they observed that the addition of BNC to a poly(vinyl alcohol) (PVOH) matrix resulted

in an intriguing increase in thermal stability at higher temperatures when compared to pure PVOH, presumably as a result of the BNC's superior compatibility and greater dispersion in the PVOH matrix [117].

Kaboorani, et al. reported that in comparison to pure PVA, the hardness of CNC/PVA increased according to CNC loading to PVA, the higher the ratio of CNC to PVA, the greater the variation in hardness. Adding only 1% CNC to PVA increased average value of hardness by 70%. At 2% CNC loading, improvement decreased to 55%, and increased again to 155% in CNC loading at 3% [133].

Chaabouni and Boufi reported that adding CNF to PVA adhesive might contribute to improve the performance of wood joints bonded in humid conditions and at elevated temperatures. They also mentioned that a huge reinforcing effect and moderate enhancement in their resistance were noted with adhesive containing 3% CNFs. Also almost no effect on the shear strength of the adhesive at room temperature was noted for polymer with CNFs content up to 3% [134].

Huan et al. conducted a comparative analysis of the mechanical behavior of nanohybrids. The CNC loading concentrations were 0, 5, 10, 15, and 20 weight percent in proportion to the poly lactic acid (PLA) addition weight. This is attributed to more effective stress transfer from the PLA to the stiffer (at lower concentrations) nanocellulose; on the other hand, at higher concentrations, the CNC agglomerate, result in a decrease in the crystallinity [121]. They observed a significant enhancement in the tensile strength and Young's modulus at a low concentration of CNC (10 wt.%), and a deterioration of the same properties at higher concentrations with increased CNC contents [135].

According to Lu et al., there was an improvement in thermal stability when CNC is present in the PLA matrix. However, they also observed that the functionalization and preparation process of the CNC has a significant impact on thermal stability [136]. A number of nanocellulose based materials, including cellulose nanocrystals (CNC) and bacterial nanocellulose nanocrystals (CNC) and bacterial nanocellulose (BNC) were recently studied. Antonelli et al. [137] used cellulose materials like bacterial nanocellulose (BNC) and

cellulose nanocrystals (CNC) to consolidate the deteriorated cell walls of wet wooden artifacts, which produced successful results in terms of consolidation and interactions with the wood ultrastructure. In a study by Basile et al. [138], rotten wood samples of Norway spruce were treated with a suspension of CNC, either alone or in combination with lignin and/or siloxane derivatives. The results of the CNC treatment showed significant improvements in the samples' rigidity right away. A combination of CNF and CNC was suggested by Camargos et al. [139] to create new, stable composite coatings that are highly compatible with wood, able to maintain the morphology and roughness of the treated material, and able to stop wooden artworks from deteriorating. Additionally, reversibility of these coatings using water-loaded cleaning hydrogels was revealed by IR-analysis, which is another significant feature. Cataldi et al. [140] suggested using fillers based on nanocellulose as renewable reinforcing agents in a polymeric matrix of methacrylic-siloxane to protect wood against water absorption. The stiffness, thermal and dimensional stability and hydrophobicity of the initial matrix was enhanced by the cellulose nanocrystals.

6. Conclusion

It is possible to transform cellulose fibers into cellulose nanofibers (CNFs) or cellulose nanocrystals (CNCs), which have exceptional qualities in comparison to the original cellulosic fiber [74]. Thus, cellulose possesses exceptional mechanical qualities because of this crystalline portion [141]. Compared to 100% pure polymer material, nanomaterials added as fillers to composite materials demonstrated improved stiffness, resilience, durability, barrier properties, and flame retardancy [74]. Strong interactions between CNCs and the polymer resulted in the creation of a stiff network, which heated the composite to 227°C, the point at which cellulose begins to break down [122].

To create cellulose nanocomposites, cellulose nanoparticles must be well dispersed and distributed throughout the polymer; solvent casting is an easy and popular method for doing this [142]. Comparing cellulose

nanocrystals and nanofibrils to an empty polymer matrix, both significantly enhance the mechanical and thermal properties of composites [87] [65]. The tensile strength of the composite films may be lowered by a high concentration of nanocellulose in the polymer matrix, which could lessen the interaction between the fillers and the polymer matrix due to uneven stress distribution in the nanocomposite materials [143].

Studies have demonstrated that cellulose-based materials used in the composite system offer superior physical and chemical properties along with eco-friendly qualities, making them perfect materials for long-term practical use [65]. Nanocellulose based composites have already been used (applied) in the archaeological field, and several of those composites were modified to serve in other applications. In recent research the ability of CNF to improve the mechanical properties of Klucel E was investigated [144], and addressed the ability of CNC particles to enhance the mechanical properties of Klucel E [145]. The astonishing properties shown by these polymers after improving their properties by adding nanocellulose make it worthy of experimenting with them in the field of wood treatment.

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