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Nanofibrillated Cellulose from Natural Fibers for Food Applications

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Abstract

Interest in nanofibrillated cellulose has been growing rapidly due to its ease of preparation, high yield, specific surface area, strength, stiffness, lightweight nature, and biodegradability. Alfa fibers, with a cellulose content of over 45%, have been recognized as a sustainable source for producing cellulose nanofibers because of their renewability and availability. By subjecting raw Alfa fibers to alkali, bleaching, and chlorhydric acid treatments, nanofibers with high yields have been successfully produced. Microscopy tests have confirmed that Alfa nanofibers, with an average diameter of 12 µm, were obtained after bleaching treatments. Transmission electron microscopy has shown that these nanofibers have a needle-like shape, with an average diameter and length of 5 ± 3 nm and 400 ± 30 nm, respectively. These nanofibers will be further explored for synthesizing nanomaterials, as additives for paper and paperboard, in biomedical applications, and for food packaging purposes.

Keywords: Alfa ultimate fiber; Cellulose; Nanofibrillated; Food packaging

Introduction

Cellulose, the most abundant component of plant biomass, is primarily found in plant cell walls in nature. However, it is also produced by some animals, algae, and a few bacteria [1,2]. Cellulose is a semi-crystalline polysaccharide that appears in nature in the form of fibers with widths ranging from 5 to 20 µm and lengths ranging from 0.5 up to several millimeters.

In recent decades, natural cellulose materials have been utilized for various purposes such as energy sources, building materials, paper, textiles, and clothing [3]. Despite their versatility, cellulosic materials have limitations, such as incompatibility with hydrophobic polymers, which has hindered their use as reinforcement in polymers [4]. Nanocellulosics, which are classified into three main subcategories based on their dimensions, functions, and preparation methods, offer a potential solution to these limitations. The classification of nanocellulosics is dependent on the source of cellulose and the processing conditions. This classification is detailed in (Table 1).

Table 1: The family of nanocellulose materials classified in three main subcategories.

Type of nanocellulose	Selected references and synonyms	Typical sources	Formation and average size
Microfibrillated cellulose (MFC)	Microfibrillated cellulose [5], nanofibrils and microfibrils, nanofibrillated cellulose	cellulose (MFC) Microfibrillated cellulose [5], nanofibrils and microfibrils, nanofibrillated cellulose Wood, sugar beet, potato tuber, hemp, flax	Delimination of wood pulp by chemical or enzymatic treatment diameter: 5-60 nm length, several micrometers.
Nanocrystalline cellulose (NCC)	Cellulose nanocrystals, crystallites [6], whiskers [7], rodlike cellulose microcrystals [8]	Wood, cotton, hemp, flax, wheat straw, mulberry bark, ramie, Avicel, tunicin, cellulose from algae, and bacteria	Acid hydrolysis of cellulose from many sources' diameters: 5-70 nm length; 100-250 (from plant cellulose) 100 nm to several micrometers (from celluloses of tunicates, algae, bacteria).
Bacterial nanocellulose (BNC)	Bacterial cellulose [9], microbial cellulose [10], biocellulose [10]	Low-molecular weight sugars and alcohols	Bacterial synthesis diameter: 20 -100 nm; different types of Nanofiber network

The purity and properties of nanocellulose are influenced by the plant source, including the fiber dimension structure of the cell wall, as well as the relative percentage of cellulose, hemicelluloses, and lignin. Additionally, the extraction method used plays a crucial role in determining the final quality of the nanocellulose.

Experimental

Cellulose Nanofiber Preparation

Cellulose nanofibers hold great promise for a wide range of applications, especially as a reinforcement in the creation of nanocomposites. Numerous studies have been conducted on the isolation and characterization of cellulose nanofibers from different sources. These nanofibers can be extracted from cell walls using either simple mechanical methods or a combination of chemical and mechanical processes. In our study, the extraction of Alfa cellulose nanofibers involved two distinct steps: pretreatment/bleaching and the hydrolysis process. This method allows for the efficient isolation of high-quality cellulose nanofibers for use in various industries.

Pretreatment and Bleaching Process of Alfa

In order to achieve a high level of cellulose fiber purity, it is essential to undergo a pretreatment and bleaching process to eliminate hemicelluloses, lignin, and other impurities such as waxes and ashes. In this particular study, a combined approach involving the use of sodium hydroxide and hydrogen peroxide was employed. Interestingly, when esparto fibers were bleached solely with hydrogen peroxide, the lignin content remained unaffected. However, the introduction of sodium hydroxide into the bleaching solution proved to be effective in ensuring the complete destruction of lignin [11]. During the experimentation phase, 5 grams of dried esparto grass fibers were submerged in a solution containing 30g/L of sodium hydroxide and 35g/L of hydrogen peroxide. The fibers were treated for a duration of 90 minutes at a temperature of 120°C [12].

Hydrolysis of the cellulose Alfa fiber

The bleached pulp underwent treatment with a 10% HCl (1 N) solution and was mixed using an ultrasonicator at a temperature of approximately 60 ± 1°C for durations of 2 and 5 hours. Subsequently, the fibers were removed and thoroughly washed with distilled water to achieve a neutral pH before being dried. The fibers were then suspended in water and subjected to continuous stirring with a high-shear homogenizer for 15 minutes. This high-shearing action effectively breaks down fiber agglomerates, resulting in the formation of nano-fibrils (Figure 1).

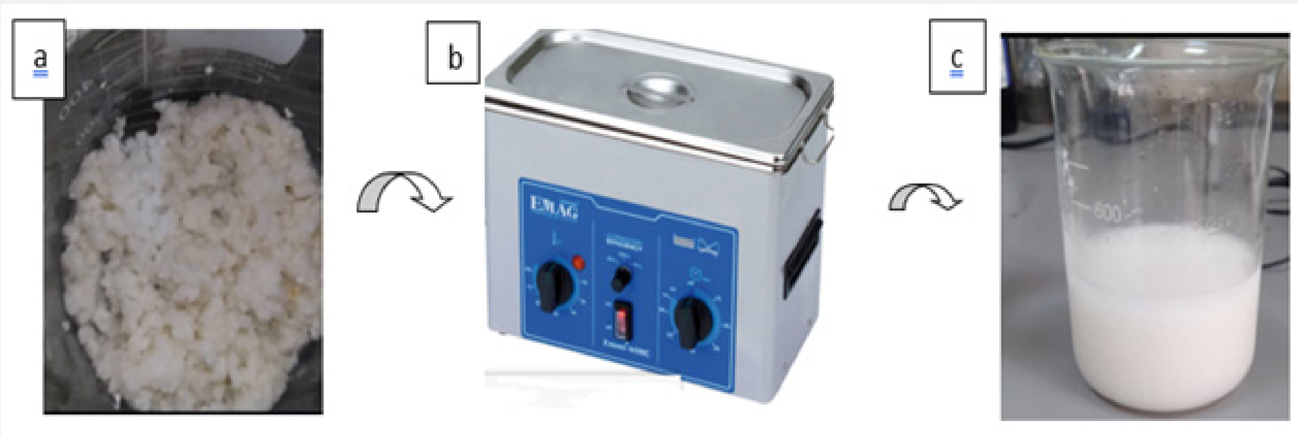

Figure 1: Hydrolysis method of Alfa fiber; (a): pretreated/ bleach fiber; (b): Ultrasonic cleaner; (c): solution of Alfa Nanofiber.

Results and discussion

Cellulose Alfa Nanofiber Preparation

The pretreatment and bleaching processes result in the production of purer and whiter Alfa fiber strands. During the pretreatment process, the use of HCL and temperature causes the fibers to explode, effectively removing hemicelluloses and lignin [13]. This process, known as steam explosion techniques, was originally developed by W. H. Mason in 1928. Different source materials require varying steam explosion temperatures and retention times. Some materials may need longer retention times and higher temperatures than others.

Previous research has shown that steam explosion effectively separates hemicelluloses and lignin, improving the subsequent processes following pretreatment [14]. Composition analysis using the Technical Association of the Pulp and Paper Industry method, as shown in (Table 2), confirms that a significant amount of hemicelluloses and lignin are removed during pretreatment. Acid hydrolysis of cellulose Nanofiber further removes hemicelluloses and lignin, resulting in a higher percentage of cellulose content in the final product.

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Table 2: Technical Association of the Pulp and Paper Industry method.

Component (%)	Untreated Alfa fiber	Pre-treated Alfa fiber	Cellulose Alfa nanofiber
Cellulose	48 [14]	79	87
Hemicelluloses	27	9,4	4,5
lignin	18	3,2	1,3

(Figures 2 & 3) displays the transmission electron microscopy image illustrating the morphology of the fiber after undergoing an acid hydrolysis process using hydrochloric acid. The fiber was broken down into nano-sized diameters ranging between 2-8 nm as a result of the acid treatment. The strong acid effectively cleaved the stack of cellulose microfibrils, resulting in the production of fibers with nano-sized diameters.

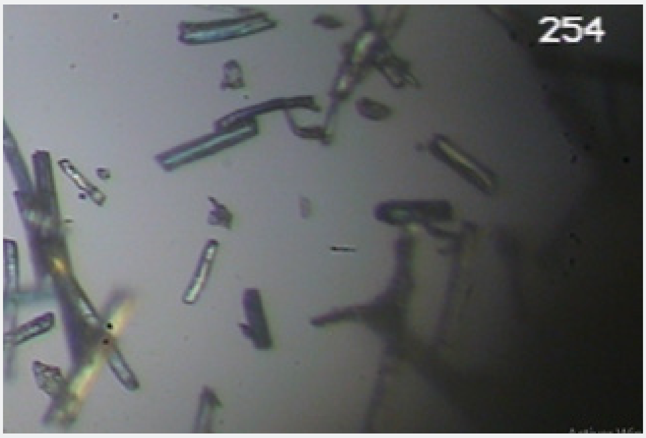

Figure 2: hydrolysis process (10% HCL, 5h).

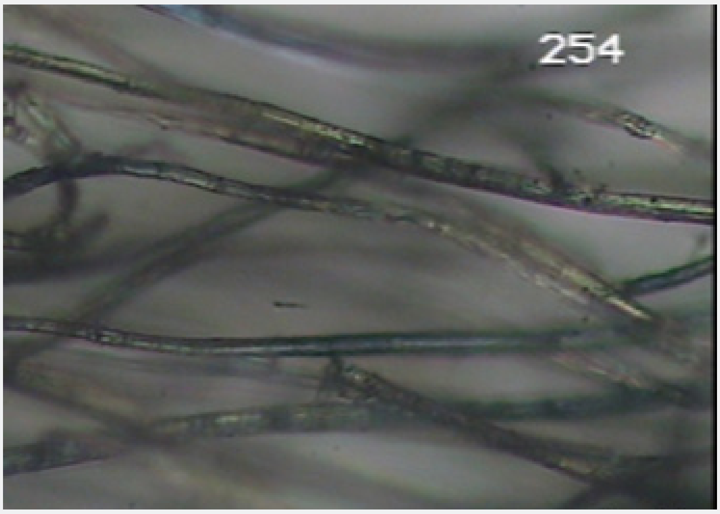

Figure 3: Hydrolysis process (10% HCL, 2h).

Conclusion

The pretreatment process was conducted to eliminate hemicelluloses, lignin, and other extractives in order to obtain a purer cellulose fiber. The cellulose nanofiber from Alfa was obtained through the hydrolysis process using hydrochloric acid. The optimal conditions for producing effective nanofibers were found to be using 10% hydrochloric acid for 5 hours. The purpose of this study was to assess the potential of Alfa nanofibers in creating new biopolymer-based nanocomposite films.

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Conflict of Interest

This research did not receive any specific grant from funding agencies in the public, commercial, or not-for-profit sectors.

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