

EXTRACTION OF CELLULOSE NANOFIBERS USING FOOD PACKAGING

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Abstract: Solid waste from food packaging is accumulated in urban waste in abundance. It is widely recognized that the composition of this packaging is 75% paperboard, 20% polyethylene and 5% aluminum; so it is necessary to look for solutions to minimize the impact of discarding these packets. Currently, there is a great motivation to study renewable materials, which have great potential for the development of nanomaterials due to their physical-mechanical properties, properties such as: high surface area, colloidal stability, mechanical resistance and low toxicity. Thus, this work aimed to characterize the cellulose nanofibers obtained from milk food packaging. For this, the cellulose fibers contained in the paperboard underwent pre-treatment, bleaching and then were subjected to acid hydrolysis. The resulting cellulose nanofibers were then characterized using Fourier transform infrared spectroscopy (FTIR), transmission electron microscopy (TEM), scanning electron microscopy (SEM), thermogravimetry (TG/DTG) and ray diffraction analysis. -X. The result of the FTIR spectra showed the efficiency of removal of non-cellulosic constituents. In addition, the cellulose nanofibers obtained were in the form of elongated needles, with a diameter between 6 and 9 nm, in addition to high crystallinity, good thermal stability and high dispersion.

Keywords: Characterization, Food Packaging, Cellulose Nanofibers, Nanotechnology.

INTRODUCTION

The natural crystalline fibril-like structure known as nano cellulose fiber (CNF) has emerged as a sustainable building element in advanced materials science [1]. The substance responsible for this characteristic is cellulose, an organic polysaccharide found in plant fibers and which is also the main component of plant cell walls [2], [3]. Cellulose fibrils are surrounded by a matrix of hemicellulose and

lignin. the NFC (*nanofibrillated cellulose*) or nanofibers are used to exploit the crystalline areas of this material, which provide high hardness and resistance. For this reason, the objective is to destructure the fibers in the amorphous areas to obtain nanofibers with high crystallinity [1], [4], [5]. There is a growing research on the extraction of new fibers and analysis of their properties in recent years [6]– [10].

Nanostructured cellulose has shown great potential as a sustainable alternative to conventional materials in several applications. Its unique properties such as high surface area, high mechanical strength and biodegradability make it a promising candidate for use in areas such as packaging, biomedical engineering and water treatment. Furthermore, its production from renewable sources further enhances its sustainability credentials [11], [12].

Cellulose nanomaterials (CNMs) are derived from various sources, including wood pulp and agricultural waste, and can be processed into different forms, such as nanofibrils and nanocrystals, with nanometer size. These materials have high mechanical strength, gas barrier properties and are biodegradable, making them a sustainable alternative for food packaging [3], [13], [14].

The composition of food packaging waste is a matter of concern due to the high accumulation of solid waste in urban areas. Typically, food packaging materials are made from cardboard, polyethylene, and aluminum, which account for about 75%, 20% and 5% of packaging waste, respectively [15],[16].

Several strategies were developed to avoid discarding packaging in landfills at the end of its useful life. Among them, the recovery of cardboard to be reused in paper production, the recovery of polyethylene-aluminium, the joint or separate recycling of materials and the pyrolysis of the entire package, which

eliminates the need for prior separation, stand out. To separate the cellulose fibers from the packaging, the hydropulping process is commonly used [15]–[18]. Despite these measures, the recycling rate of multilayer packaging is still quite low and it is necessary to invest in more efficient solutions to deal with the growing volume of packaging produced [15], [18].

Different approaches have been used for the preparation of cellulose nanofibers [19]–[26], with the literature suggesting several physical and chemical treatments, including pre-treatment, bleaching and hydrolysis to obtain cellulose nanocrystals [27]–[31].

In order to simplify the production of nanofibers, chemical processes were implemented to obtain cellulose nanocrystals, aiming to remove impurities such as lignin, hemicellulose, additive and residues, resulting in purer cellulose. However, the high consumption of water and energy involved represents a considerable environmental challenge. Currently, there is a strong motivation for researching renewable materials that have great potential for the development of nanomaterials, due to their physical-mechanical properties such as high surface area, colloidal stability, mechanical strength and low toxicity. It is important to emphasize that sustainability is not only an environmental issue, but also a social and economic one. Therefore, the objective of this study is to obtain and characterize cellulose nanofibers obtained from food packaging.

MATERIALS AND METHODS

To obtain cellulose nanofibers, it is necessary to follow a process that involves different stages, from the collection of the raw material (carton of milk) to the final product. The experiments were carried out at the Nanotec laboratory of ``Universidade de Brasília``, located at Faculdade do Gama.

To evaluate the morphological characteristics of the fibers, techniques such as X-ray diffraction, scanning electron microscopy (SEM), transmission electron microscopy (TEM), thermogravimetric analysis (TG/DTG) and Fourier transform infrared spectroscopy (FTIR). The flowchart shown in Figure 1 summarizes the steps of the project, from extracting the cellulose to obtaining the nanofibers.

The experiments were conducted using the technique for obtaining cellulose nanocrystals, which involved the treatment of cellulosic material in three chemical processes: pre-treatment, bleaching and acid hydrolysis [16], [31]. Two samples were prepared: sample “A” was not cooled in an ice bath and did not receive the addition of nitric acid during the bleaching process, while sample “B” underwent all chemical processes.

FEEDSTOCK

The technique for recycling milk cartons consists of sorting, which can be done manually or through automated processes, the separation is done by machines that use technologies such as magnetic and optical separation. Finally, the pulping process is carried out by crushing and mixing with water, resulting in a material that can be used in the manufacture of new products [15], [16].

To obtain the raw material, the manual sorting technique was adopted, collecting boxes of food such as milk, coconut water, juice, among others, which were found in residential waste. Then, the boxes were cleaned with water and neutral liquid soap, dried with plenty of water, cut in half and submerged in a container with water for seven days to facilitate manual removal of the paperboard, with the aid of a spatula.

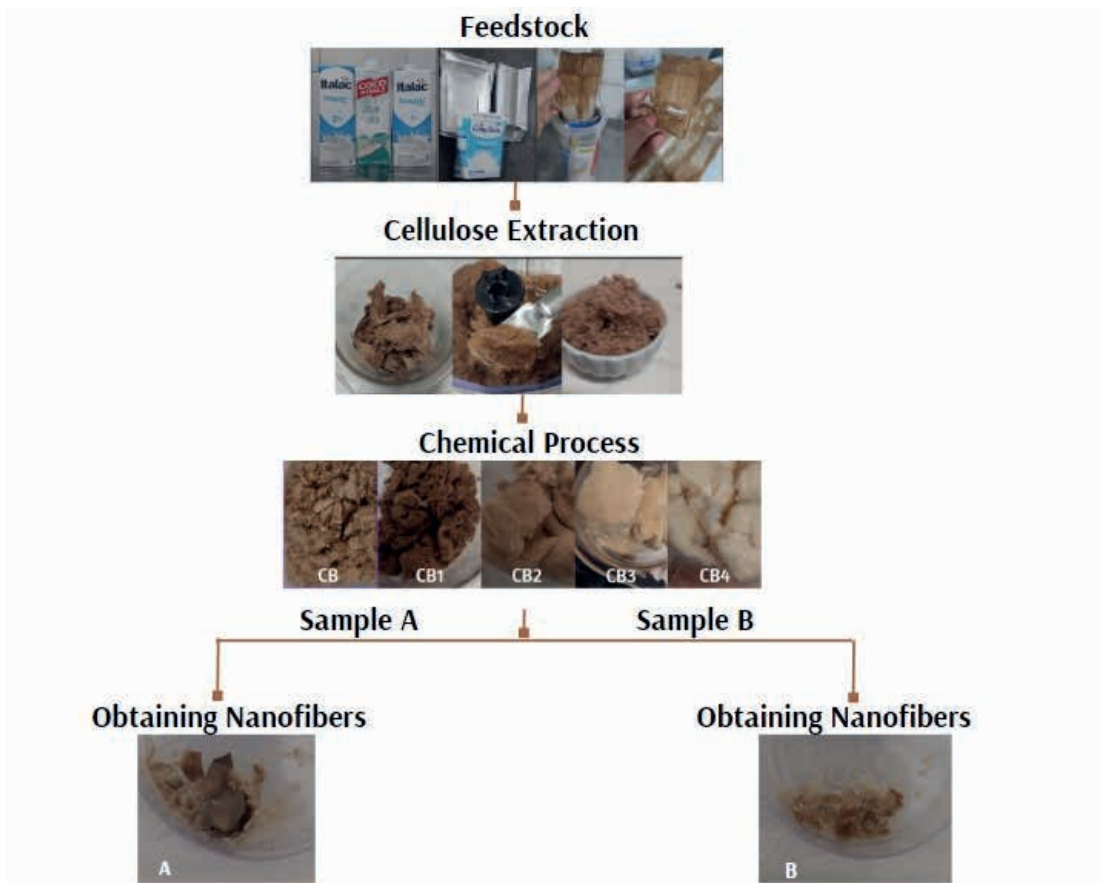


Figure 1: Scheme of the processes involved: cellulose extraction, bleaching process and obtaining nanofibers.

Source: Own author (2023)

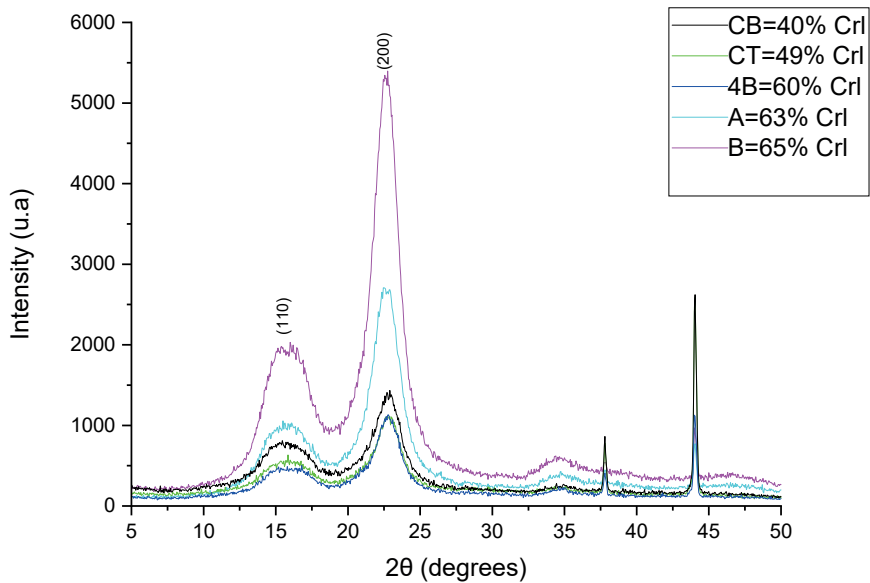


Figure 2: X-ray diffractograms with crystallinity index: crude cellulose (CB), pre-treatment cellulose (CT), bleached cellulose (4B), nanofiber A and nanofiber B.

Source: Own (2023)

EXTRACTION OF CELLULOSE NANOFIBERS

The extraction of cellulose nanofibers was performed following the methodology previously used in previous studies [16], [31]. Cardboard paper (CB), which was removed from the milk carton, underwent a grinding process in a processor and was subsequently dried in an oven at 50 °C for 24 hours. 10 g of BC was used for the first pre-treatment step, this material was washed with 250 ml of distilled water, sieved and stirred with heating at 50°C for 2 hours and was repeated once more. 500 ml of a 2% NaOH solution was added, which was kept on a magnetic stirrer with heating at 80°C for 2 hours, then filtered, and it was repeated once more. The fibers obtained were called pre-treated cellulose (CT).

The CT underwent a bleaching process, it was added to 300 ml of water, 3 g of NaClO₂ and 20 drops of glacial acetic acid, the solution was heated to 70 °C for one hour, then the sample was cooled in a bath of ice, filtered quantitatively and washed with cold water until reaching neutrality. For a more efficient delignification, the same process was repeated four consecutive times. Finally, a 0.05 M nitric acid solution was added to treat the pulps, sieved to a 0.212 mm mesh and washed with water to remove residues. The fibers obtained were called bleached cellulose (4B).

The bleached cellulose was subjected to acid hydrolysis, the chemical method used to obtain cellulose nanofibers. Cellulose is dissolved in concentrated sulfuric acid (64% by weight sulfuric acid in water) for 120 minutes at 50°C using magnetic stirring. In order to improve the quality of the hydrolyzed cellulose, the samples were centrifuged at 4,000 rpm for 10 minutes and the supernatant was removed, the precipitate was washed with distilled water several times until reaching neutral pH (pH=7). Finally, the nanofibers

called Samples (A and B) were separated from the solution, dried in an oven at 100 °C for 24 hours

SCANNING ELECTRON MICROSCOPY-SEM

A scanning electron microscope (SEM) model JEOL JSM-7001F, with power of 15 kW, was used at the Laboratory of Microscopy and Microanalysis (LMM) of the Institute of Biology (IB) of ``Universidade de Brasília`` (UnB), to analyze the morphology of samples CB, CT, 4B, A and B. SEM is a widely used technique to observe nanomaterials with high resolution and magnification, providing nanometric details. Diameter measurement is essential to verify fiber defibrillation. [27], [32].

TRANSMISSION ELECTRON MICROSCOPY – TEM

During the LMM of the IB at UnB, a TEM was used to examine the structure of samples A and B. An aqueous suspension containing nanofibers was deposited on a carbon microgrid (300 mesh) and allowed to dry. Then, the grid was stained with a 1% uranyl acetate solution and dried at room temperature. The images of the cellulose nanofibers were captured using a Transmission Electron Microscope model JEOL JEM 1011, operating at 80 kV [33]–[36]. In order to establish the measurements of the nanofibers, a total of 135 individual cellulose fibrils were randomly selected from different images to determine their average diameter using the Image J software.

X-RAY DIFFRACTION

At LabMatI, we performed X-ray diffraction using an XRD-6000 diffractometer, which swept the range from $2\theta = 5^\circ$ to 60° at a sweep rate of $0.5^\circ/\text{min}$, operating with a voltage of 40 kV/30 mA and using radiation from a Cu K α lamp (1.5406 Å).

To calculate the crystallinity index, we used Segal's method (Eq. 1), in which the arbitrary value of the peak intensity of the crystalline parameter (200 and 110) is represented by I (200), and the arbitrary value of the amorphous parameter is represented by I (AM) [37].

$$\text{CrI} = \frac{I_{200} - I_{110}}{I_{200}} \times 100$$

Eq. 1: Crystallinity index equation. Source: [37]

THERMOGRAVIMETRIC ANALYSIS (TGA)

Samples CB, CT, 4B, A and B were subjected to thermogravimetric analysis using a thermal analyzer model SDT Q600 (TA Instruments). An approximate amount of 10 mg of each sample was used, which were placed in an alumina crucible. The analysis was carried out in a nitrogen atmosphere, with a gas flow of 100 ml/min and a heating rate of 10 °C/min, ranging from room temperature to 600 °C [16], [38].

FOURIER TRANSFORM INFRARED SPECTROSCOPY (FTIR)

FTIR spectra of samples CB, CT, 4B, A and B were performed using a Fourier Transform Infrared Spectrometer (NICOLET IS10 from Thermo Scientific), with the aid of a diffuse reflectance accessory (DRIFT). The spectra were recorded in the range of 4000 to 400 cm⁻¹, with a resolution of 4 cm⁻¹ and 64 scans [39].

RESULTS AND DISCUSSION

X-RAY DIFFRACTION

The diffraction charts in Figure 2 show peaks approximately 15° and 22° that represent the crystallographic planes (200) and (110) respectively, according to the literature these peaks indicate the typical crystalline structures of cellulose. The crystallinity index

(CrI) intensifies according to the treatment processes that remove amorphous cellulose and impurities [40], [41].

FTIR SPECTROSCOPY ANALYSIS

The FTIR spectra obtained in Figure 3 show characteristic bands due to the bonds of the structure, mainly cellulose, thus a band was observed that corresponds to the axial vibration of the O-H bond at 3427 cm⁻¹. At the frequencies of 2970-2800 cm⁻¹, 1470-1350 cm⁻¹ and 750 cm⁻¹ the axial and angular vibration bands corresponding to the C-H bond were observed. The band at 1720 cm⁻¹ corresponds to C=O binding (hemicellulose), in addition, a band was observed at 1640 cm⁻¹ due to C=C binding (lignin), and a band at 1250-1100 cm⁻¹ due to the C-O bond (cellulose) [18], [27], [31].

SEM AND TEM ANALYZES

The SEM images show a more uniform morphology and fiber separation, as shown in Figure 4, of all samples (CB, CT, 4B, A and B) [18]–[20], [22], [27], [31], [38], [42]–[44]. In the CB sample, it is possible to notice that the fibers are still very aggregated, and that all non-cellulosic components are present. The structures are composed of fibers covered by a layer of non-cellulosic materials, which act as cementing materials. These materials include hemicellulose, lignin, waxes and mineral contents. It is observed that the CT fibers treated with NaClO₂ solution present a greater exposure of roughness in the fibers and a smoother and cleaner deagglomeration, due to the removal of lignin and other non-cellulosic components. Indeed, lignin plays a key role as a bridge between cellulose and hemicellulose molecules, binding the fiber components together. With acetosolv, the lignin was partially depolymerized from the internal parts of the fibers, which resulted in the disaggregation of the bundles as the main

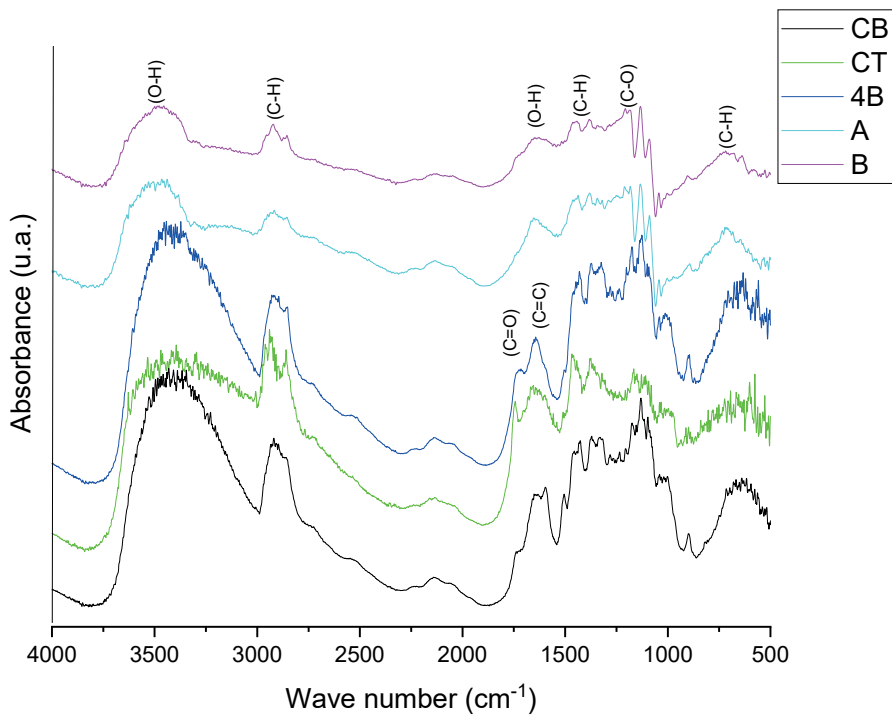
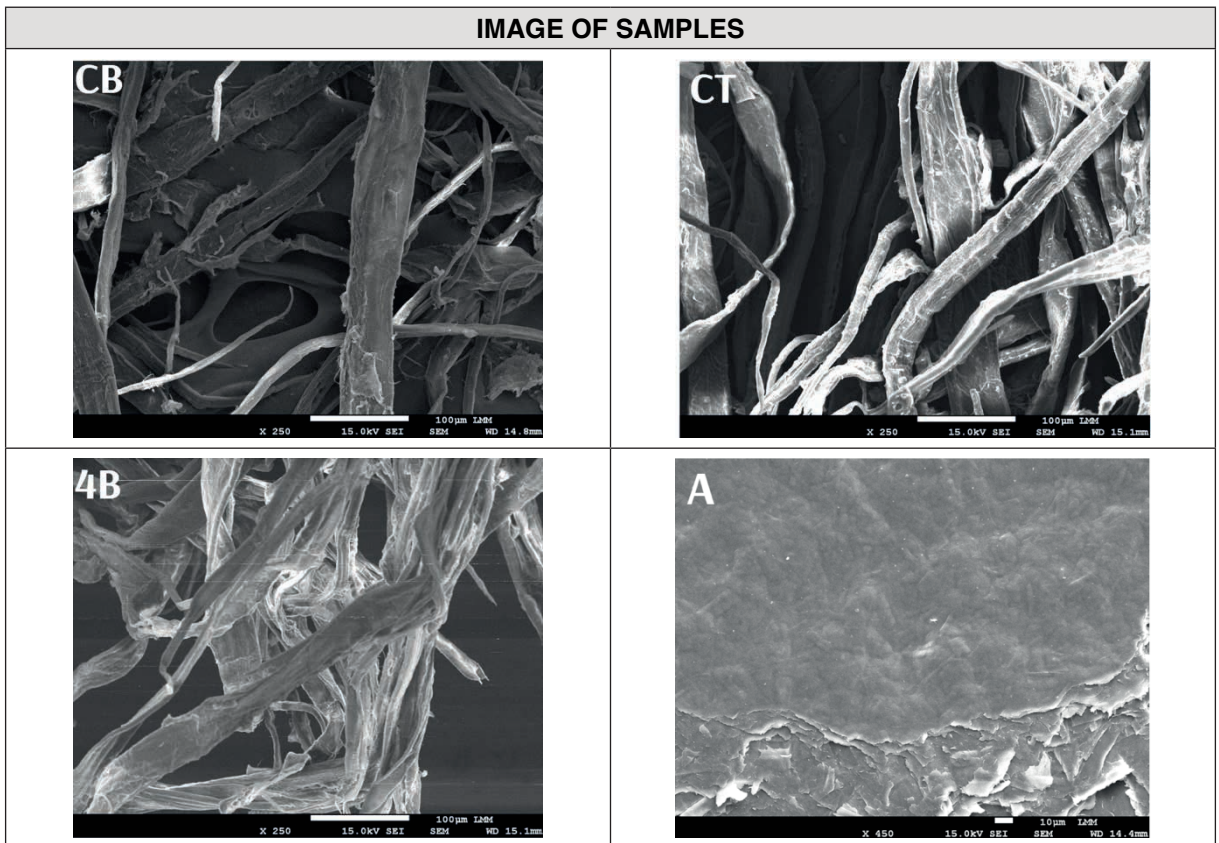


Figure 3: FTIR spectra: crude cellulose (CB), pre-treatment cellulose (CT), bleached cellulose (4B), nanofiber A and nanofiber B. Source: Own (2023)



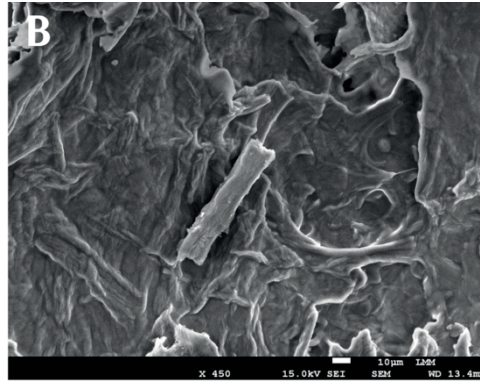


Figure 4: Crude cellulose (CB), pre-treatment cellulose (CT), bleached cellulose (4B), nanofiber A and nanofiber B.

Source: Own (2023)

MET IMAGE OF SAMPLES

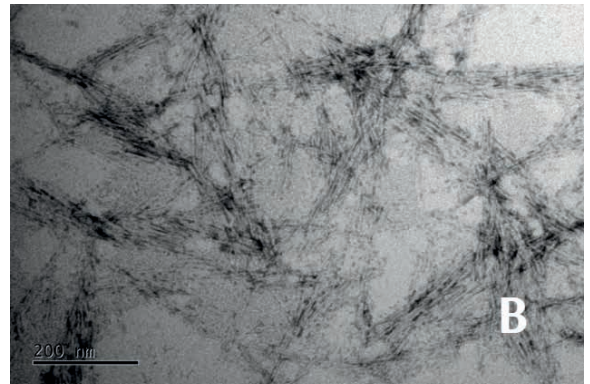
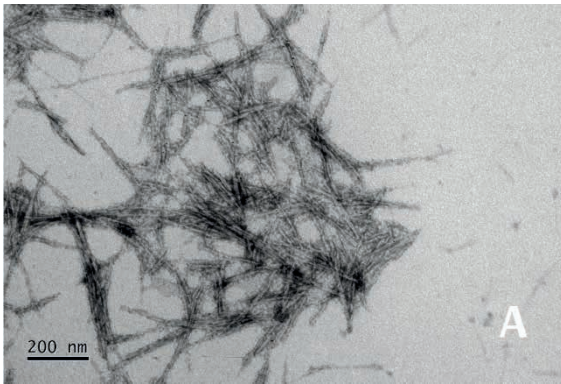


Figure 5: Micrographs obtained in the TEM of nanofibers: sample A and sample B.

Source: Own (2023)

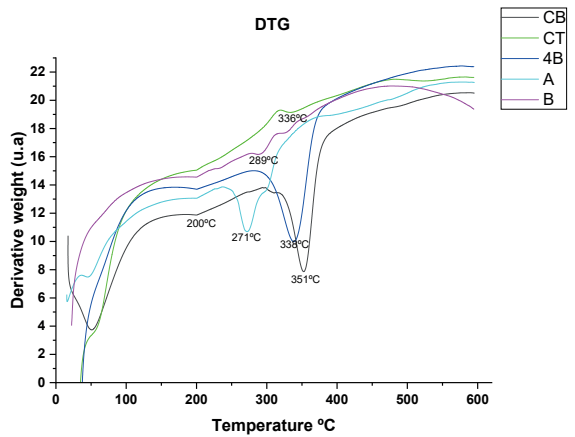
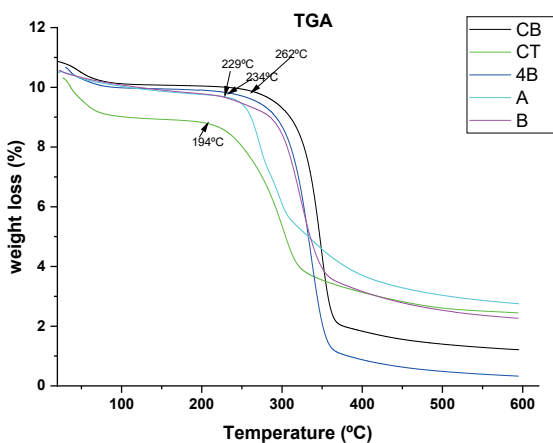
effect.

The bleaching treatment has an effect on the fiber structure. In sample 4B, a more intense defibrillation process was observed than in the other samples. The lignin underwent partial depolymerization in the inner parts of the fibers, which resulted in the separation of the fiber bundles. This process was the main cause of the deagglomeration of the bundles.

In sample "A" they present a non-homogeneous surface, on the edges encrusted superimposed layers were observed, on the other hand in sample "B" a rougher surface was observed with intercrossed fibers, on the edge's nanofibers were observed in a clear way.

In Figure 5, it presents the micrograph obtained in the TEM, in sample "A" nanofibers were clearly observed in the form of agglomerated needles with a diameter of 9 nm, in sample "B" the diameter of the nanofibers was smaller, being 6 nm, the images reveal a characteristic of thinner, elongated and agglomerated fibers [18]–[20], [22], [27], [31], [38], [42]–[44].

THERMOGRAVIMETRIC ANALYSIS (TG/DTG)



The first stage of mass loss occurs between 50 and 100°C, which corresponds to the vaporization of water in the sample. The second stage of mass loss presents peak degradation occurs between 194 to 389°C.

The thermal stability curve of the bleached pulp at 234 o C (sample 4B) is greater than that of the pre-treated paperboard (CT) at 194 o C. However, the cellulose nanofibers (sample A) at 229 o C and cellulose nanofibers (sample B) at 234 o C were less stable than bleached pulp. The lower stability of cellulose nanofibers can be explained by the replacement of hydroxyl groups by acid sulfate groups during hydrolysis, which reduces the activation energy for cellulose degradation, making the sample less resistant to pyrolysis, and leads to dehydration reactions that releases water and catalyzes the decomposition of cellulose [18].

The residues resulting from the analysis of the materials at the maximum temperature of TG 600 o C were approximately 0.5% for sample 4B, 2% for the raw material paperboard (sample CB), 3% for the treated sample (sample CT) and 3% for nanofibers (sample B) and 3.8% for nanofibers (sample A). Some factors can alter the number of residues, such as nanoparticles and a greater number of free bridges in the chain of cellulose nanofibers that decompose at low temperature.

The high crystallinity of cellulose nanofibers increases the proportion of carbon,

so the formation of residues increases as the carbon content increases, in addition, sulfates can act as flame retardants.

The thermal behavior differences between treated and untreated fibers and nanocrystals can be more clearly observed by the DTG curves.

The first loss of weight occurs due to the decomposition of the hemicellulose that occurs at 298 °C. The disappearance of this peak with the thermal treatment indicates that the amorphous constituents were removed due to the breaking of the carbon-carbon ether bonds.

The peak in the range of 351 °C which reaches the maximum rate of decomposition is attributed to glycosidic cleavage of cellulose; these results are in line with those presented in the literature [45].

The nanofibers show peaks at 271 °C (sample A) and 289 °C (sample B). For cellulose nanofibers, the Tonset (initial decomposition temperature) was reduced compared to treated paperboard (CT), bleached pulp (4B) and paperboard (CB). This is due to the insertion of sulfate groups on the surface of cellulose nanofibers during sulfuric acid hydrolysis.

CONCLUSION

Cellulose nanofibers were obtained from acid hydrolysis of cellulose extracted from cardboard from milk cartons, proving to be a promising material for future applications due to its ecological nature and potential reinforcement for composites. In addition, the effects of bleaching and hydrolysis time on the production process were investigated.

DRX and FTIR analyzes were effective in identifying amorphous and crystalline cellulose nanofibers. The characterization by SEM and TEM allowed observing the presence of agglomerated and needle-shaped cellulose nanofibers, with a length of 6-9 nm.

According to the characterization by TG/DTG, the cellulose nanofibers presented lower Tonset than the raw material used and the sample.

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