

Employment of Waste from the Textile Industry for the Production of Nanocomposites Aiming at the Generation of Thermal Shrinkable Films and the Non-Formation of Microplastics

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Abstract

With the increase of incorrectly discarded materials in the environment, including clothes, the need for recycling and reusing them becomes one of the alternatives to work around this problem, following the law of the 3 Rs. However, these polymeric materials which are deposited in the marine environment and land generate microplastics, due to their degradation/fragmentation due to the action of the weather, in these regions, including being carried by the air and transported from various regions. With the aim of reducing the impact of this inappropriate disposal and obtaining a material that does not generate microplastics, this study used waste from the textile industry, which would be unusable, and a biodegradable polymer. The tissue residues were submitted to acid hydrolysis with sulfuric acid, in order to weaken the interactions between the cellulose fibrils and promote a better dispersion of the latter when processed with the polymer, during the extrusion process, generating biodegradable polymeric nanocomposites. The results showed that the samples submitted to acid hydrolysis presented a higher degree of crystallinity and a greater number of interactions and lower mobility of hydrogen atoms. In addition, the samples treated for a longer time showed a small release of fibrils, suggesting that this treatment can help in the production of nanocomposites during their extrusion.

Keywords

Microplastic, Nanotechnology, Cellulose, Textile Industry, Biodegradable Polymer

1. Introduction

One sector that is not being given sufficient attention is the textile industry. On the planet, about 92 million tons of textile waste are discarded each year, which can generate an excess of microplastics, causing environmental problems. This is equivalent to a truckload of laundry being dumped into landfills every second. Furthermore, it is estimated that by 2030, the amount of textile waste discarded will be of 134 million tons [1]. A much-publicized example is that of the Atacama Desert in Chile, which over the years, has been receiving tons of clothes which are not consumed, causing great damage to the region [2] [3].

Many studies are being carried out aiming to evaluate the presence of microplastics in the oceans, marine life and coastal environments. Many of these studies indicate that, among the microplastics found, most of them are composed of microplastics formed by polyester, polyamide and cellulose, many of which come from textile materials [4] [5]. Wu *et al.* [6] found microplastics in fish in the West China Sea, where the major component was cellulose and cellophane. Wu *et al.* [7], using satellite remote sensing images, evaluated the effects of microplastics on aquaculture activities, among which were yellow croaker (*Larimichthys crocea*), oyster (*Ostrea denselamellosa*) and shrimp (*Parapenaeopsis hardwickii*). The authors observed that there was pollution in sediments and in aquaculture by microplastics. The majority of them were composed of cellulose and polypropylene; which changed the aquaculture activities present there. Finally, the authors suggest that the limited accumulation of microplastics in these beings may not imply harm to the people who consume them, being less harmful than imagined. Adams *et al.* [8] pointed out that, although cellulose fibers are obtained from natural sources, such as cotton, they should not be neglected. Therefore, it is necessary that studies continue being accomplished in order to observe the impacts that cellulose microplastics can bring to the environment.

Within the line of the 3 R's which is to reduce, reuse and recycle, the recycle of textile waste is a very viable proposal and would contribute to the reduction in the formation of microplastics. However, due to the lack of efficient recycling techniques, most clothes at the end of their useful life are discarded and are taken to landfills or incinerators or even discarded in fashionable dumps, being a great waste. Thus, incrementing awareness about protecting the environment and developing methods for recycling, these wastes are very important and should be enacted together. The cotton recycling method with fiber recovery through shredding has been done [9]. However, the material generated is of low quality and does not have the desired mechanical properties for reuse. In contrast, to obtain nanocellulose from these fibers becomes a good solution, as it eliminates most of the defects presented by recycled textile fibers, resulting in a high quality of nanomaterial, in addition to avoiding the formation of microplastics [10] [11].

Different types of nanoscale cellulose can be obtained, such as nanocrystals and cellulose nanofibers. The latter has a high aspect ratio, low density and has

hydroxyl side groups, facilitating their functionalization [11]. The use of nanofillers in polymeric matrices generates polymeric nanocomposites, materials that allow changes and improvements in physical and thermal properties, in a remarkable way, when compared to the composite with micrometer scale filler [12]. These properties can range from an increase in the degradation temperature to an improvement in barrier properties, greater resistance, changes in the degree of crystallinity, among others.

Many studies have been achieved using nanocellulose obtained from different sources and used in different polymers. Savadekar and Mhaske [13] studied the effect of these nanofibers obtained from cotton, together with thermoplastic starch. The results indicated that the films presented greater tensile strength when compared to isolated starch. Moreover, there was a decrease in the transmission of water vapor and oxygen. Fourati *et al.* [14] produced cellulose nanofibers obtained from cotton and incorporated them into thermoplastic starch and PBAT and observed that the addition of these nanofillers aided in the interfacial adhesion of starch with PBAT, resulting in an increase in the mechanical performance of the material. Kang and collaborators [15] developed biofilms hinged on gum arabic and cellulose nanocrystals, produced from the extraction of wheat bran and treatment in acid hydrolysis. The results showed that biofilms containing 4% (m/m) of cellulose nanocrystals showed tensile strength and elongation at rupture, as well as reducing oxygen and water vapor permeability, decreasing the loss of strawberry mass and showing that it is suitable for increasing the preservation time of fruits. Wang, Shankar and Rhim [16] produced cellulose nanocrystals from blackberry, for incorporation into the alginate matrix. The authors observed that the Young's modulus and the tensile strength of the obtained nanocomposite increased by 35% and 25%, respectively, compared to the pure alginate, however, there was no change in the permeability to water vapor. However, few studies have been performed obtaining nanocellulose from textile waste. Wang and collaborators [17] used an old patterned sheet composed of approximately 100% cotton. For the formation of cellulose nanocrystals, promote acid hydrolysis using sulfuric and hydrochloric acids. The ranges of length and diameter of the nanocrystals were from 28 to 470 nm and from 3 to 35 nm, respectively, being of potential use for biocomposites. Zhong and collaborators [18] used indigo-dyed denim fabric to form nanocellulose. The material was treated with sulfuric acid and the resulting nanocellulose was incorporated into the poly (vinyl alcohol) matrix. The results showed that the mechanical properties of the nanocomposite were better than those of the pure polymer, indicating the improvement provided by the nanocellulose. These studies presented show the potential of using nanocellulose in polymer matrices, with the source of cellulose being obtained from waste from the textile industry.

As stated previously, the objective of this work was to treat waste from the textile industry, intending to generate nanocellulose to be used in nanocomposites with poly (butylene-adipate-co-terephthalate) (PBAT), a biodegradable po-

lymer, to obtain heat shrinkable films which are friendly to the environment and the non-generation of microplastics, in view of the high volume of disposal of shrink films from pec of food and beverage products

2. Experimental

2.1. Materials

Fabric made from 100% cotton, obtained from SENAI CETIQ. Cotton marketed by the Apolo brand. PBAT, marketed under the name Ecoflex[®] F C1200, with molar mass (M_w) = 14.2×10^4 g·mol⁻¹, bulk density: 1.25 g/cm³, obtained from OEKO. Sulfuric acid (H₂SO₄), 95% - 98.5% purity, obtained from Sigma-Aldrich.

2.2. Pulp Treatment

The cotton tissue residues were cut into sizes of approximately 5 mm using scissors. The cut tissue was pulverized in the A11 Basic IKA analytical mill km in order to destrand it and facilitate the subsequent acid hydrolysis reaction, which took place in the presence of H₂SO₄. In a round bottom flask, 4 g of tissue were placed in 355 mL of water. From that point on, the following steps were performed with the suspension under vigorous mechanical agitation. To this system, H₂SO₄ was added slowly, with the aid of an addition funnel, in such a way that the concentration of the final acidic solution was 20% m/m. The reaction temperature was maintained at 25°C and the reaction time was started, ranging from 3, 6, 8, 10 and 12 days. At the end of the reaction, the suspension was poured into a 400 mesh sieve and washed with distilled water until constant pH. Then, they were kept in the hood for drying for 3 days and, at the end of the process, they were characterized. In order to assess whether the fabric was composed of cellulose, analyzes were made comparing it with commercial cotton.

Each sample was named according to the digestion time, as shown in **Table 1**.

3. Characterization

3.1. Scanning Electron Microscope with Field Emission Source (FEG-SEM)

The morphology of the samples was observed through MEV-FEG TESCAN, model MIRA 4th generation LMU (LowVac Mode UniVacTM of 1 - 700 Pa). The samples were evaluated in high vacuum, with a 5 keV beam, using SE detectors (Secondary Electron).

3.2. X-Ray Diffraction (XRD)

The X-ray diffraction was carried out by the RIGAKU X-ray Diffractometer-Model: Miniflex to obtain characteristics related to the organization of the cellulose. The variations in the diffraction peaks were made according to the following parameters: angle 2θ between 2° and 40°; 0.5°/min sweep rate; wavelength $\lambda = 0.154$ nm; X-ray generation source: copper.

Table 1. Sample code and formulations.

Sample	Digestion time (days)
Cotton	0
T	0
T3	3
T6	6
T8	8
T10	10
T12	12

3.3. Fourier Transform Infrared Spectroscopy (FTIR)

The spectroscopy was obtained using the Infrared Absorption Spectrometer (FTIR)—Perkin Elmer FT-IR Spectrometer, model 1720 X. The spectra were obtained in the range of 400 to 4000 cm^{-1} using a KBr tablet, with scan number equal to 40 and resolution equal to 4 for all spectra.

3.4. Nuclear Magnetic Resonance in the Time Domain (TD-NMR)

TD-NMR analysis was performed by determining the longitudinal relaxation time of the hydrogen nucleus (T_1H). The equipment used was the MARAN Ultra 0.54 T, from Oxford Instruments. Samples were placed on the probe in an 18 mm tube. The conditions used were: 1H core observation frequency: 23 MHz; pulse used: 90° ; sequence of pulses used: inversion-recovery, 40 points; temperature: $(30 \pm 2)^\circ\text{C}$; interval between recovery points: 0.1 to 5000 ms; receiver gain (dB): 40.

4. Results and Discussion

4.1. Scanning Electron Microscope with Field Emission Source (FEG-SEM)

Figure 1 displays the micrographs of the analyzed materials. Samples cotton (a) and fabric (sample “T”) (b) are presented at a magnitude of 1000x, while samples T3 (c), T6 (d), T8 (e), T10 (f) and T12 (g) are presented in three different magnitudes (200x, 1000x, 2000x and 10,000x), aiming at a better morphological visualization. T has a ribbon-like shape with circular bases, which conforms to the morphology of cotton. T3 showed few changes in its shape, showing that the digestion time did not have significant effects on its morphology. In T6, the beginning of the breaking of the cellulose polymeric chains was observed. In T8, it is possible to notice more significant effects in the cellulose morphology, with the opening of some fibrils, as it is also evidenced in T10, where the release of fibrils is observed. The sample that showed the greatest effect of acid hydrolysis was T12, in which the change in the morphology of the cellulose fibers and the greater breakage of their chains were observed, resulting in smaller chains, in addition to greater fibrillation.

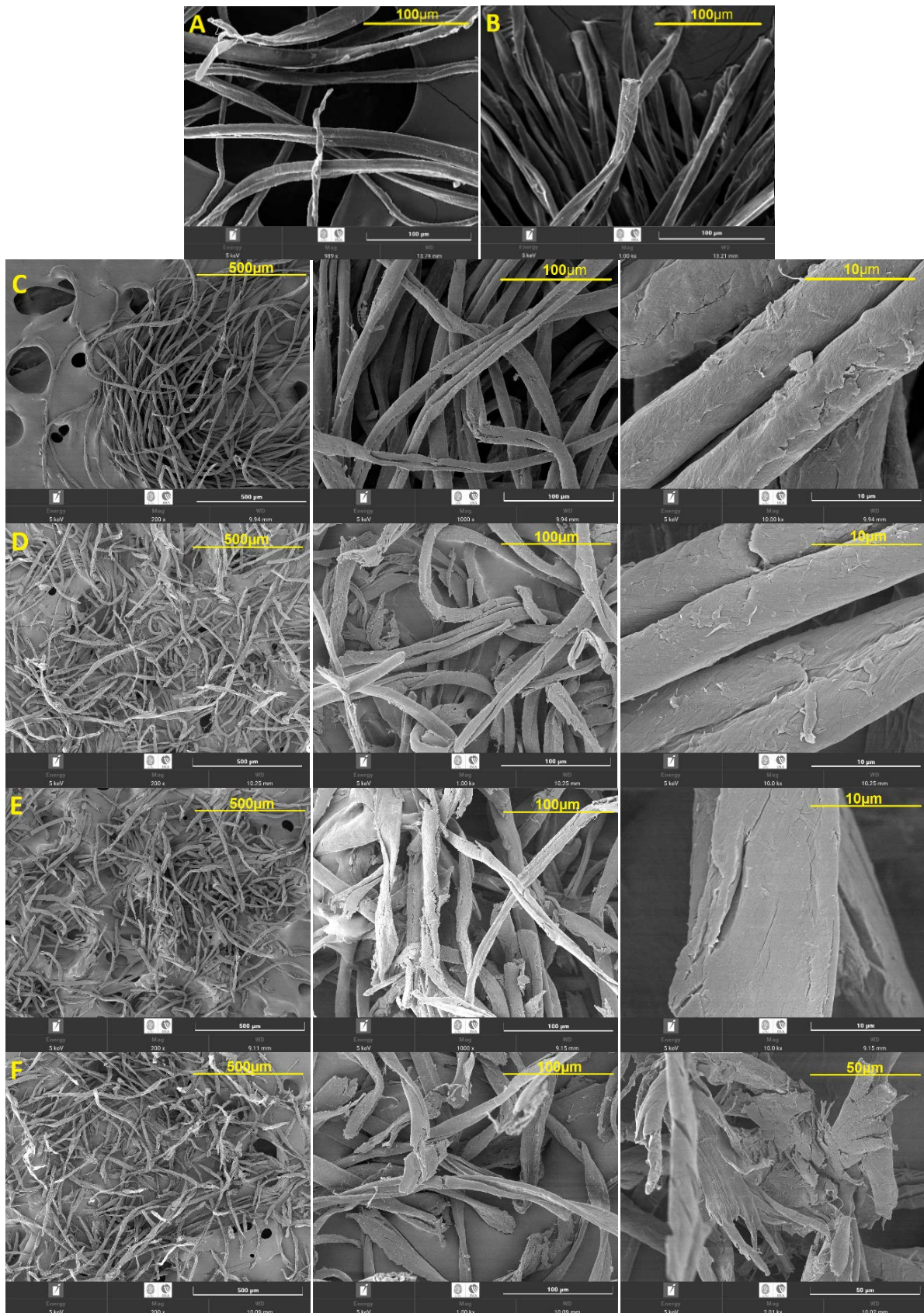


Figure 1. Images of cotton from FEG-SEM (a), T (b), T3 (c), T6 (d), T8 (e), T10 (f) and T12 (g).

4.2. X-Ray Diffraction (XRD)

Figure 2 shows the X-ray diffraction profiles of the samples. Well-defined peaks are observed both for T and for the samples after treatment with H_2SO_4 . The diffraction signals found in this analysis are close to $2\theta = 14.7^\circ$, 16.9° , 23.1° and 34.7° , which are in agreement with the literature [19] [20].

It is possible to observe a similar profile for T and the treated samples in relation to the peaks around $2\theta = 14.7^\circ$ and 16.9° . The increase in acid hydrolysis time generated peaks around $2\theta = 23.1^\circ$ narrower and more intense than the T without treatment. Furthermore, according to **Table 2**, the treated samples showed an X_c greater than T, remaining around 82% at T10 and T12. This indicates that the digestion process gave higher degree of crystallinity [19]. Taking into consideration the peak around $2\theta = 34.7^\circ$, no significant alteration was observed in relation to the T without treatment.

The degree of crystallinity (X_c), determined by XDR is listed in **Table 2**, was obtained from **Figure 2**.

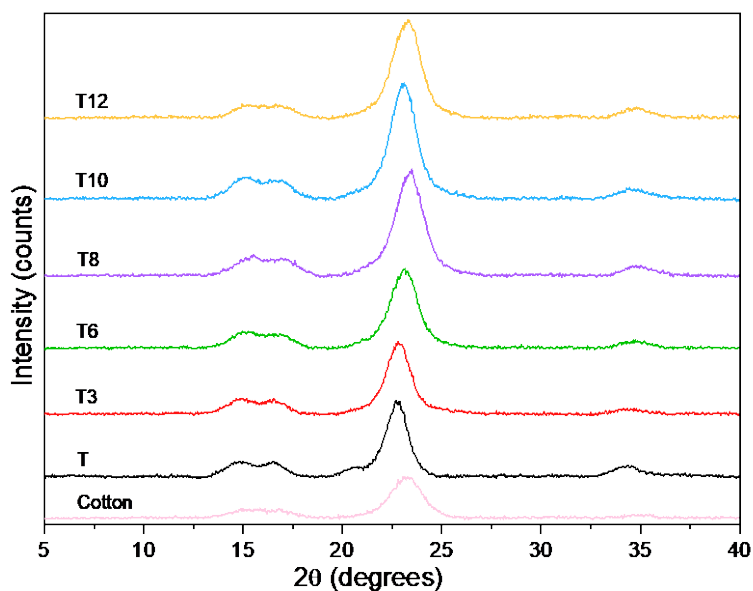


Figure 2. Images of cotton from XRD of T, T3, T6, T8, T10 and T12.

Table 2. Samples crystallinity degree.

Sample	X_c (%)
Cotton	83.0
T	63.2
T3	79.7
T6	83.0
T8	81.3
T10	82.2
T12	82.3

4.3. Fourier Transform Infrared Spectroscopy (FTIR)

FTIR analysis was performed in order to investigate possible changes in the chemical structure of cellulose fibers (Figure 3). The T samples and their derivatives presented spectra characteristic of cellulose, when compared with the spectra obtained from cotton and from the literature [18]. The absorption bands of the treated samples were similar to those of the untreated T, indicating that the treatment with H_2SO_4 did not significantly alter and/or degrade the chemical structure of cellulose [21]. It is observed that all samples presented a peak centered at 3333 cm^{-1} and another centered at 1645 cm^{-1} , which correspond to the axial and angular deformations of the intramolecular hydroxyl (O-H) group, respectively. The absorption band at 3333 cm^{-1} increased with increasing treatment time, except for t10, which showed lower absorption at this peak and at 1645 cm^{-1} . The following peaks were also observed: 3276 cm^{-1} , which represents axial deformations of intermolecular O-H bonds, 2897 cm^{-1} , which corresponds to axial deformation of C-H, 1427 cm^{-1} due to angular vibration $-CH_2$. The absorption band 1313 cm^{-1} attributed to C-O and C-H symmetrical angular strain, 1159 cm^{-1} represents the axial strain of the C-O-C asymmetric bond, 1028 cm^{-1} due to the C-OH axial strain and 897 cm^{-1} indicates the C-H angular strains [22].

4.4. Time Domain Nuclear Magnetic Resonance (TD-NMR)

The determinations of relaxation times by the TD-NMR technique permit to obtain information about the molecular dynamics of the material and also be able to infer about the mobility of the domains present in it. The longitudinal spin-lattice relaxation time of the hydrogen nucleus (T_{1H}) is used as an indicator of collective molecular motion, in which a lower value of T_{1H} corresponds to greater mobility, and often corresponds to moisture. Higher values of this parameter show changes in molecular mobility due to the generation of new or multiple intra and/or intermolecular interactions [23] [24].

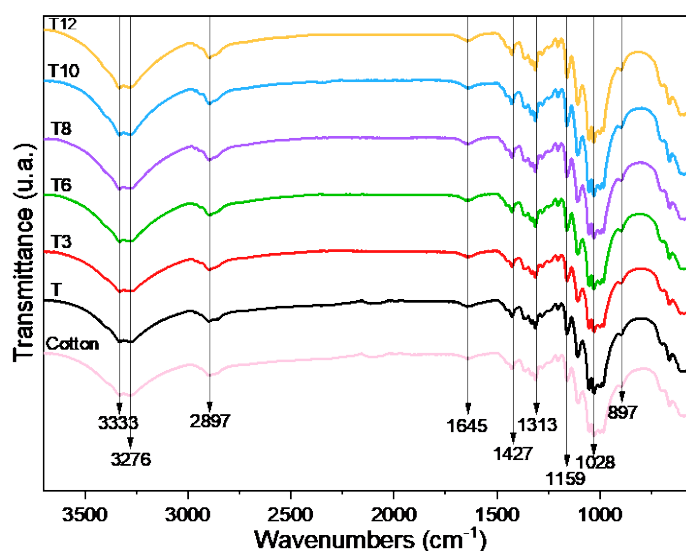


Figure 3. Images of cotton from FTIR, T, T3, T6, T8, T10 and T12.

The $T_1\rho$ values of each of the samples are listed in **Table 3**. Analyzing the $T_1\rho$ values for the most intense domain curve, it is observed that the cotton presented a $T_1\rho$ value equal to 97 ms. The T sample has a $T_1\rho$ value close to that of cotton, however, with a slight shift to the left. T6 showed a lower value of $T_1\rho$ than T, indicating that there was an increase in the molecular mobility of the sample, by the treatment process, causing a change in the structural organization. T8 showed a higher $T_1\rho$ value than T and T6, showing more intra and intermolecular interactions. The T10 sample revealed that the value of $T_1\rho$ is greater than that of T8, possibly pointing out stronger interactions between the components. T12 also followed this trend, being the sample that presented the highest value for $T_1\rho$, which shows that the digestion time directly influenced the $T_1\rho$ values, changing the molecular organization and, consequently, the intra and intermolecular interactions.

Figure 4 shows the distribution curves of $T_1\rho$ domains. The most intense peaks refer to the hydrogens of the cellulose chain, which, due to their rigidity, control the relaxation process. The smaller peaks observed in some samples refer to the presence of a water molecule, either adsorbed and/or absorbed, acting as a plasticizer; cellulose hydrogens are well plasticized. These domain curve peaks are smaller and wider because they have a smaller population of hydrogens and have a lower $T_1\rho$ value due to greater molecular mobility. The cotton sample showed a peak, whose base is widened, showing greater disorder and molecular heterogeneity. The T sample reveals a behavior similar to cotton, in addition to a smaller peak at 4 ms, referring to adsorbed water. T6 showed a more intense peak displacement to the left, and a slight narrowing of its base, which suggests a better molecular homogeneity/organization, and presented a smaller peak at 3 ms. The T8 peak was shifted to the right in relation to T. In addition, there was a

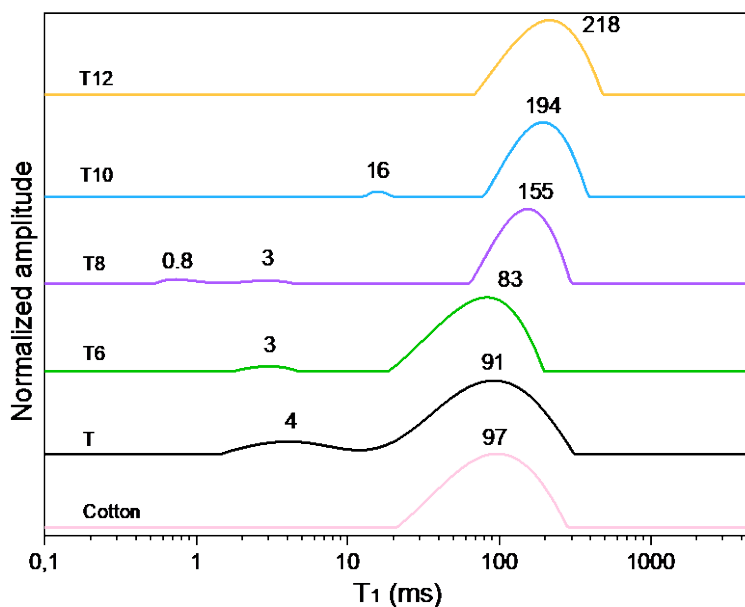


Figure 4. Distribution of relaxation domain curves for: T, T6, T8, T10 and T12.

Table 3. T₁H for cotton, T, T6, T8, T10 and T12.

Sample	T ₁ H (ms)
Cotton	97
T	91
T6	83
T8	185
T10	194
T12	218

greater narrowing of its base, which indicates that the cellulose molecules are better organized and differently from the other samples, generating a more homogeneous material, than those of T and T6. T8, unlike the rest of the samples, has 2 peaks of lesser intensity. T10 showed both a slight shift to the right and a slight widening of the base in relation to T8, designating that its molecules are less organized than T8. Following this same trend, there is an enlargement at the base of the T12 peak and a shift to the right, indicating that the molecules presented less organization of the cellulose molecules than the other treated samples, which may indicate that the acid hydrolysis reaction led to structural and organizational modifications of the cellulose molecules were more effective in this condition. This generates multiple interactions that caused an increase in stiffness, shifting the dominant relaxation time value to larger values [21] [25] [26]. A similar behavior was seen for the T10 sample, however with a smaller number or weaker multiple interactions, but which still caused a variation in the relaxation time to larger values indicating an increase in the sample stiffness.

5. Conclusions

The acid hydrolysis method was applied to weaken the cellulose fibrils and promote their openings during processing in the extrusion containing the polymer to generate biodegradable polymeric nanocomposites. The reduction in H₂SO₄ concentration and the absence of heating were treatment parameters used in order to reduce costs with the production of this type of material. Samples treated by digestion showed a higher X_c in relation to T, a higher molecular interaction and lower mobility of hydrogen atoms. In the microscopy images, samples T8, T10 and T12 showed fibrillation of the cellulose, being more significant for sample T12. The TD-NMR analysis showed that the samples that were treated showed a greater molecular organization and lower mobility in relation to T, which is in agreement with the XRD data. They present more crystalline structures in the treated samples than in T, thus generating more resistant materials with good properties, and biodegradability.

The results of this study are innovative. They show that this treatment method, reusing cotton waste from the textile industry that would otherwise be discarded, can be a great new option for the production of biodegradable mate-

rials. They can be produced with quality and applicability; avoiding the generation of microplastics. It would normally occur with the improper disposal of textile waste and plastics which are normally used for this type of material, thus reducing the impact on the environment.

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Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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