Composites Based on Jute Fibers and Phenolic Matrices: Properties of Fibers and Composites

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ABSTRACT: Composites based on phenolic matrices and both untreated and alkali and ionized air-treated jute fibers were prepared. Different fiber lengths and fiber content were used to reinforce the phenolic matrices. The jute fibers were characterized with respect to lignin, holocellulose, ash, and humidity contents and also to the crystallinity index. The mechanical properties of fibers were investigated by means of tensile analysis and the morphology by SEM. The untreated and treated jute fiber-reinforced composites were characterized as to water absorption. The mechanical property and morphological aspects of the composites were evaluated by impact strength and photomicrographs obtained from SEM. Among the jute fiber treatments considered in the present work, the treatment with a solution of 5% NaOH presented the best results because: (1) the fiber presented a higher tensile strength, and a larger percentage of elongation at break; (2) the composite reinforced with this fiber presented the highest impact strength results when this was the unique treatment (20% of fiber), as well as when it was combined with ionized air (30% of fiber); and (3) the composite that presented the lowest water uptake was that reinforced with this fiber. © 2003 Wiley Periodicals, Inc. J Appl Polym Sci 91: 1077–1085, 2004

Key words: fibers; thermosets; impact resistance; composites; mechanical properties

INTRODUCTION

Jute is a vegetal fiber made up of mainly cellulose, polyols, and lignin. In the plant, the jute fiber has the function of phloem; that is, it carries sap elaborated by the plant. It is located between the medulla and the bark.^{1–3} Therefore, the jute fiber represents a small portion of the plant, about 5–6% of the total weight of the green plant.⁴

There is a large group of composites prepared from polymeric matrices reinforced with vegetal fibers.^{5–10} These composites constitute a unique combination of materials, which may result in the improvement of properties such as strength to fatigue and impact. Natural fibers have attracted attention because of their availability, renewability, affordability, and their contribution to improve the mechanical properties of composites as reinforcing agents.

The high strength of jute fibers has not been intensively exploited in reinforced composites. This occurs, at least partially, because they are hygroscopic and their low wettability by resins. These characteristics result mainly from the presence of hydroxyl groups and other polar groups in several constituents of the fiber,⁸ mainly cellulose and lignin. Therefore, to enhance the use of jute and other vegetal fibers, a broader system of investigations is necessary to search for chemical modifiers and physical treatments that would alter these undesirable characteristics.

The treatment of lignocellulosic fibers with an NaOH solution (mercerization) leads to a decrease in the degree of fiber aggregation, which normally results in considerable benefits in their use as polymerreinforcing agents.¹¹ Fiber mercerization must be carried out with alkaline solutions in moderate concentration and temperature, so that the fibers do not degrade.

The treatment with ionized air can be considered as a "physical treatment" similar to corona discharge. Corona discharge can produce surface changes in lignocellulosic fibers when high-energy particles hit this region. The final effect will depend on the surface energy of the fibers, the viscosity of the resin used to prepare the matrix, the size, and quantity of surface irregularities.^{12–14}

The present study considers the characterization of untreated and treated jute fibers, as well as their use as reinforcing agents in phenolic composites. This work is part of a project dedicated to investigating the use of lignocellulosic fibers as reinforcing agents in composites^{15,16}; the study of derivatization of cellulose, the major component of lignocellulosic fibers, in a homogeneous medium^{17,18}; and the use of lignin, the second most abundant component of lignocellulosic fibers, as a partial substitute of phenol in the preparation of phenolic resins.^{19–22}

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EXPERIMENTAL

Prepolymer syntheses

A phenolic prepolymer was synthesized using phenol, formaldehyde, and potassium hydroxide (1.38:1.0: 0.06 w/w, respectively) with mechanical stirring, under reflux (70°C). The solution was then cooled to room temperature and HCl was added until neutralization was reached. Water was eliminated under reduced pressure.

Cure reaction and composite production

The thermoset and composites were obtained by mixing resorcinol (10% w/w) through mechanical stirring at 50°C for 30 min. Compression molding was carried out in a mold ($220 \times 99.5 \times 5$ mm) under pressure (50 kgf/cm²). The molding cure cycle (75°C/1 h; 85°C/2 h; 95°C/0.5 h; 105°C/0.5 h; 115°C/1 h; 125°C/1.5 h) was previously determined by DSC measurements.¹⁶

The jute fibers used in the preparation of the composites were previously extracted (Soxhlet) with cyclohexane/ethanol (1:1, v/v) over a period of 48 h, to extract low molecular weight substances [e.g., waxes, therpenes (natural impurities)], and then with water for 24 h, to extract inorganic contaminants. The fibers were dried in a circulating-air stove (60°C) until reaching a constant weight. As a consequence of cycloexane/ethanol and then water extraction, a loss of weight was observed near 2%.

Jute-reinforced composites (fiber length: 3.0, 3.5, 4.0, 4.5, 5.0 cm; 10, 20, 30% wt %) were obtained by adding the fibers to the prepolymers. The mixture was submitted to mechanical stirring (0.5 h, 50°C) before the cure step, which in turn followed the same route described for the preparation of the thermoset. The composites were prepared with randomly oriented fibers.

Composites were also prepared using fibers treated as described below:

- 1. The fibers were swollen using NaOH solutions of 10%, for 60 min, or of 5%, for 30, 60, and 120 min at 0°C. Then, the fibers were washed until the alkali was totally eliminated, after which they were dried in a circulating-air oven.
- 2. The fibers treated with 5% NaOH solution, 1 h, and the untreated fibers were subjected to ionized air. The fibers were placed in a system built using a metal box containing air-injection channels on its lid, connected to a high-tension generator. The fibers were submitted to an electric discharge (current: 5 mA; voltage: 7.8 kV) for 1 h and immediately used in the preparation of the composites.

Analyses

Fibers

Humidity content. Humidity content was determined according to ABNT (Associação Brasileira de Normas Técnicas, Brazilian Technical Standards Association), NBR9656, which consists in determining the percentage difference between the initial weight of the sample and that after 4 h of drying, at 105°C. A minimum of three samples of each material was tested and the average values are reported in the next section.

Ash content. Ash content was determined considering the percentage difference between the initial weight of dried fiber of the sample and that after calcination for 4 h at 800°C. A minimum of three samples was tested for each material and the average values are reported in the next section.

Crystallinity index. The crystallinity index (I_c) was determined by X-ray diffraction using a Rigaku Rotaflex model RU-200B diffractometer (Rigaku, Japan) operating at 40 kV, 20 mA, and λ (Cu–K_{α}) = 1540 Å. The crystallinity index was calculated using the Buschle–Diller and Zeronian²³ equation: $I_c = 1 - I_1/I_2$, where I_1 is the intensity at the minimum (2 θ value between 18 and 19°) and I_2 is the intensity of the crystalline peak at the maximum (2 θ value between 22 and 23°).

Klason lignin content. This method is based on the isolation of lignin by means of the polysaccharides acidic hydrolysis (sulfuric acid, 72%) [Technical Association of Pulp and Paper Industry (TAPPI) T13m-54]. A minimum of three samples of untreated and treated fibers was tested and the average values are reported in the next section.

Cellulose and hemicellulose content. The cellulose plus hemicellulose content (holocellulose content) was determined according to TAPPI T19m-54. A minimum of three samples of each material was tested and average values are reported in the next section.

Microscopy. Scanning electron microscopy (SEM) was carried out in a Zeiss–Leica model 440 apparatus (Zeiss, Jena, Germany), at electron acceleration of 20 kV. The fibers analyzed were covered with a thin layer of gold in a sputter-coating system.

Tensile strength. Tensile strength was analyzed using fiber bundles (15 mm long and 0.5 mm diameter), dried previously in a vacuum stove for 4 h, 100°C, and kept under vacuum, at room temperature. A dynamic mechanical analyzer (DMA model 2980; TA Instruments, New Castle, DE) in the tensile mode was used, under the following conditions: 25°C, 1 to 18 N/min. A minimum of 30 samples was tested for each material and average values are reported in the next section.

Composites

Impact strength. Ten unnotched samples were cut from each plate and shaped accordingly to ASTM D256

TABLE I			
Crystallinity Index (I _c) of Some Untreated and Treated			
Iute Fibers			

Fiber	I _c (%)
Untreated	50
Alkali-treated (NaOH 5% 1 h)	48
Alkali-treated (NaOH 10% 1 h)	46
Ionized air (1 h) treated	47
Alkali (NaOH 5%) and ionized air (1 h) treated	55

 $(63.5 \times 12.7 \times 4.0 \text{ mm})$. Impact strength was assessed using an Izod impact testor (Ceast Resil 25). Impact tests were carried out at room temperature with an impact speed of 4 m/s and incident energy of 5.5 J. As established in ASTM D256, in each experiment at least five measurements were used to calculate the average values reported in the next section.

Microscopy. SEM was carried out in a Zeiss–Leica model 440 apparatus, at electron acceleration of 20 kV. The fractured samples analyzed were covered with a thin layer of gold in a sputter-coating system.

Water absorption. The water absorption test was performed according to ASTM D570, which describes several types of tests. The selected test considers both water absorption during 24 h, and also up to saturation. The dimensions of the specimens for the water uptake experiments were $76.2 \times 25.4 \times 3.2$ mm.

RESULTS AND DISCUSSION

Characterization of the jute fiber

Humidity content of the jute fiber was 10.3 \pm 0.4%, which is consistent with values reported in the literature for lignocellulosic materials (7.7–10% humidity)²⁴ and particularly for jute (10–12% humidity).⁹

Table I displays the results concerning crystallinity index variations of jute as a function of the different treatments used in this work, by use of alkali (5 and 10%, 1 h of treatment) and ionized air treatments.

Not only can the NaOH treatment affect the crystallinity of the fibers, but it also usually alters the cellulose lattice from I to II. These changes depend on the tension applied during treatment. Lignin and hemicellulose removal can lead to an increase in the crystallinity index because it allows a better packing of the cellulose chains.^{10,11}

Although the alkali treatment removed the lignin and the hemicellulose of the jute fibers considered here, mainly the last (Table II), the results in Table I show that the fiber treatment with NaOH led to a small decrease in crystallinity. The diffractograms (not shown) also indicate that the cellulose lattice did not change from I to II, given that I_1 (the intensity at the minimum) and I_2 (the intensity of the crystalline peak at the maximum) had the same values of 2θ for both fibers, untreated and alkali treated.²³ In the present work, the cellulose was only swelled by an NaOH solution; that is, the treatment was softer than the mercerization process, in which the cellulose is usually suspended in the alkali solution.

Probably as a result of the conditions of alkali treatment used here, the crystallinity parameter did not change because of the treatment. It must be pointed out that in a previous work¹⁶ the lignocellulosic fiber was suspended in NaOH solution, instead of swelled. It was verified that the process adversely affects the fiber properties, as observed from the results of the impact strength of the composites reinforced with alkali-treated fibers.

Aggregation involving fibers can be related to electrostatic interactions. Charges may be generated by the friction between two surfaces. Elimination of electrostatic charges in nonconducting materials is not trivial and is possible only through specific treatments as, for example, the ionization of the molecules present in air. The fragments may neutralize the different charges present on the surface of the material. In the case of lignocellulosic fibers, it is presumed that at this point aggregation is minimized. In the present work, concerning the ionized air treatment, a small decrease in I_c occurred compared with the untreated fiber, possibly

TABLE II Composition of Untreated and Some Treated Jute Fibers

Fiber	Klason lignin (%)	Cellulose plus hemicellulose (%)	Ash (%)
Untreated	15.9	89.6	1.6
Alkali-treated (5%/30 min)	15.6	81.7	1.3
Alkali-treated (5%/1 h)	15.6	81.5	1.3
Alkali-treated $(5\%/2 h)$	15.7	81.0	1.3
Alkali-treated (10%/1 h)	14.2	81.7	1.2
Ionized air (1 h) treated	15.8	87.7	1.5
Alkali (5% $/1$ h) and ionized air (1 h) treated	14.1	82.1	1.9



Figure 1 Photomicrograph of untreated jute fiber (×10,000)

because this treatment affects only the upper layer of the cellulose fiber. It was the combination of NaOH and ionized air treatment that promoted the greatest alteration in I_c . The alkaline treatment separates the fiber bundles and removes natural and artificial impurities from the surface, allowing a more intense action of the ionized air, which in turn, apparently promotes a reorganization of the chains (e.g., through the formation of new hydrogen bonds among cellulose chains). This is facilitated by the previous removal of hemicellulose by the alkali treatment, thus increasing I_c .

The composition of jute fiber can vary because of several factors, such as weather conditions of the plantation site, type of soil, flood season (jute is grown in flood regions), manner and conditions of extraction, and the part of the plant from which the fiber is taken, as well as the treatments to which it may be submitted after extraction from the plant. Thus, it is convenient to make a detailed characterization of the material to be used. Table II shows the contents of Klason lignin, holocellulose, and ash for untreated and some treated fibers. For all the measurements, the reliability of the three trials was very good.

An analysis of the results of Table II reveals that the alkaline treatments changed the fiber composition concerning the three main components of the fiber (lignin, cellulose, and hemicellulose). The composition is less affected by the action time of NaOH, than to the concentration of the alkaline solution, mainly for lignin extraction.

Mohanty and Misra⁴ stated that jute lignin must behave as a bonding agent among the carbohydrates by two types of bonds: one sensitive to alkali and another resistant to alkali. The bonds sensitive to alkali are of the ester type, which result from the combination of the hydroxyl group of the lignin and the carboxyl present in the hemicellulose rings, as well as ether type bonds formed from hydroxyl groups of the cellulose. Because it is polyfunctional, lignin combines with several chains of cellulose and hemicellulose neighboring molecules, behaving as a bond structure between these chains.



Figure 2 Photomicrograph of jute fiber treated with a solution of 5% NaOH for 1 h ($\times 10,000$)

Gassan⁸ found that jute hemicellulose is much more sensitive to the action of NaOH at room temperature than lignin or α -cellulose. Taking into consideration that the percentage variation of holocellulose reflects more the extraction of the hemicellulose than of cellulose, the data in Table II can be regarded as confirmation that the alkali treatment extracts a larger percentage of hemicellulose than of lignin.

The treatment with ionized air led to only a slight decrease in holocellulose content.

Figures 1, 2, and 3 display SEM images of parts of untreated and alkali-treated fiber bundles.

Alkali treatment improves the adhesive characteristics of the fiber surface by removing natural (e.g., hemicelluloses, pectic substances) and artificial impurities (dust, inorganic material) from the surface. The surface energy, hence the wettability of the mercerized fibers, is higher, and this normally results in better bonding through a form of mechanical interlocking between the matrix and the roughened fiber surface. Alkali treatment also leads to fiber bundle fibrillation, that is, breakdown of the composite fiber bundle into smaller fibers, which increases the effective surface area available for contact with the wet matrix. If optimal conditions of alkali treatment for a particular fiber are achieved, the improvement of mechanical properties is then ensured.²⁵



Figure 3 Photomicrograph of jute fiber treated with a solution of 10% NaOH for 1 h ($\times 10,000$).



Figure 4 Photomicrograph of jute fiber immediately after treatment with ionized air (×10,000).

It may be observed from the photomicrographs (Figs. 1–3) that the increase in the concentration of the alkaline solution leads to a greater separation of the microfibrils. Therefore, in principle, the fiber treated with the 10% NaOH solution should be more appropriate for use as a composite reinforcement. However, jute submitted to a 10% NaOH solution for 1 h was not used in composites because it became brittle after treatment, a characteristic that did not allow its cutting to the length of 3 cm as used for all other composites. Apparently, the use of a more concentrated solution allowed the extraction of lignin in a greater proportion, and probably in a way that worsened the mechanical properties of the fibers. The reduction in the lignin content of the fiber can yield a fiber with a chemical structure less strongly bound, given that lignin connects the three-dimensional cellulose network as well as fibrils.²⁶

As previously mentioned, the treatment with ionized air can neutralize the static charges present on the surface of the fibers, reducing their attraction to each other, thus separating the bundles, and leaving them more exposed to impregnation by the polymeric matrix. Figure 4 shows the photomicrography of fibers subjected to this treatment.

Compared to untreated fibers (Fig. 1), it may be observed that the ionized air-treated fibers present increased surface roughness and the development of microfissures (Fig. 4), which can increase the contact area fiber/matrix in the composite.

Sun et al.²⁷ reported that the corona effect has a tendency to decrease as a function of time, attributed to factors that may stabilize charges, as for example, humidity and temperature variation.

Table III displays the average values of tensile strength and elongation at break of jute fibers untreated and treated with 5% NaOH, ionized air, and 5% NaOH plus ionized air. This analysis was repeated several times, as mentioned in the experimental section, and the values obtained in each experiment always confirmed the tendency shown in Table III.

It may be observed (Table III) that the fiber bundle treated only with NaOH presented larger tensile strength and elongation at break. Gassan et al.⁸ stated that the effect of mercerization on the improvement of tensile strength of jute is attributed to the removal of hemicelluloses. When hemicellulose is removed, the interfibrillar region becomes less tense, reducing stiffness and lending the fibrils a larger capacity of rearranging in the direction of deformation tension. The increased elongation characteristic is probably attributable to the increase in the degree of molecular orientation in the fibril as a result of the treatment.

The ionized air treatment produced an effect similar to the treatment with NaOH, and a larger tensile strength and elongation than those of untreated fibers have been observed.

The combination of the two treatments (ionized air/ NaOH) decreased tensile strength and elongation in relation to the proprieties observed when the fiber was submitted separately to the two treatments. The combination of the two treatments leads to a higher crystallinity index (Table I), which in principle should lead to a higher tensile strength. More detailed investigation must be carried out with these fibers for a better understanding of the effect of the combination of the two treatments on the fibers and its consequences on the proprieties observed.

In general, the results reflect the high jute fiber stiffness. This fiber is considered one of the stiffest among the lignocellulosic fibers. This propriety presents a limitation for the textile industry, although it is important for its use as a composite reinforcement.⁴

Characterization of composites

Water uptake

Figure 5 shows the mean water uptake. The reliability of three trials was very good.

Hemicelluloses are mainly responsible for the humidity uptake, although noncrystalline cellulose and lignin also play an important role in the process. The humidity swells the cell wall of the jute fiber until its saturation. Next, it goes on to occupy the void spaces of the fiber, and this free water does not cause further swelling.²⁸

TABLE III Tensile Strength of Untreated and Some Treated Jute Fibers

Fiber	Tensile strength (MPa)	Elongation at break (%)
Untreated	466	0.7
Alkali-treated (5%, 1 h)	589	1.6
Ionized air-treated (1 h) Alkali (5%, 1 h) and	547	1.1
ionized air-treated	401	0.8



Figure 5 Percentage water uptake of phenolic thermoset (PT) and phenolic composite (PC) reinforced with untreated, ionized air and alkali-treated jute fibers.

For the phenolic thermoset, which corresponds to the nonreinforced sample, the water uptake rate is larger from the beginning until about the fifth week, when it reaches a plateau.

Concerning the composites, it may be observed that the introduction of a hygroscopic fiber increases the absorbed water percentage compared to that of the thermoset, as expected. The first measurement, made after 24 h, already seems to correspond to the beginning of the plateau. The initial stage of faster uptake, which occurs for the thermoset until the fifth week for the composite, apparently occurs within 24 h.

Comparing the results obtained for the three composites, it may be observed that those reinforced with untreated fibers and fibers treated with ionized air present a similar behavior, whereas those reinforced with alkalitreated fiber present a smaller water uptake. Among the probable factors that may have led to this difference, the extraction of hemicellulose by the alkali treatment stands out because, according to what was mentioned earlier, hemicellulose is considered to be mainly responsible for water uptake, given that it is more accessible than the crystalline regions of the cellulose.

The lesser water uptake occurring when the fibers are treated with alkali can also be related to the fiber/ matrix interface. In this region, the larger the number of microcavities originated by an inefficient adhesion, the larger the number of water molecule clusters present.²⁹ As already mentioned in the SEM analysis, the alkali treatment can improve the fiber–matrix adhesion. It is proposed that this treatment generates additional mechanical interlocking sites, making possible a larger fiber/matrix interprenetration at the surface,³⁰ therefore reducing the number of microcavities and consequently reducing the quantity of water molecule clusters present.



Figure 6 Impact strength as a function of fiber length (fibers in composites: 30 wt %).



Figure 7 Impact strength as a function of weight percentage of fibers in composites (fiber length: 3 cm).



Figure 8 Impact strength as a function of fiber treatment time (NaOH 5%).

Impact strength Izod

Figure 6 shows the results of impact strength as a function of fiber length.

It may be observed that for the composite reinforced with 4.0-cm-long jute fibers, impact strength was maximized. Longer (4.5 and 5.0 cm) and shorter (3.0 and 3.5 cm) fibers present values similar to each other. Very short fibers can have a greater difficulty in adhering to the matrix, which may generate fissures and decrease impact strength in turn. Very long fibers make the homogeneous distribution of the fibers in the matrix difficult, which may lead to fiber/fiber interaction, thus decreasing impact strength.^{15,16} This effect also seems to be active when the percentage of fibers increases, given that impact strength increases up to 20% of fiber loading and decreases after that (Fig. 7). When the fiber fraction increases, it is more difficult to impregnate the fiber with prepolymer, which in turn can facilitate the fiber/fiber interaction and make the fiber distribution in the matrix less homogeneous.



Figure 10 Influence of fiber treatment on impact strength (fiber content: 30 wt %; fiber length: 3 cm). PT, phenolic thermoset; PC, phenolic composite.

Figure 8 shows impact strength as a function of alkali treatment time, considering a solution of 5% NaOH. Alkali treatment normally leads to an improvement in the mechanical properties for the reasons mentioned in the SEM analysis of the jute fibers. However, for this to occur, optimal process conditions must be attained. Impact strength increases considerably when treatment time reaches 60 min. If this time is doubled, however, this property noticeably diminishes. Probably, although the holocellulose does not vary significantly (see Table II), a longer treatment time allows the cellulose chains to degrade, thus affecting the mechanical property of the fiber, and consequently, its action as a reinforcing agent.

Figures 9 and 10 show the variation of impact strength as a function of the different treatments to



Figure 9 Influence of fiber treatment on impact strength (fiber content: 20 wt %; fiber length: 3 cm). PT, phenolic thermoset; PC, phenolic composite.

which the fibers used to reinforce the composites were submitted, in composites with 20 and 30% of fiber, respectively.

Both for the 20% and the 30% fiber content composites, the alkali treatment improves this property (Figs. 9 and 10). However, impact strength increases more significantly for the composite reinforced with 20% of fiber. The larger fiber distribution homogeneity, probably already present in the composite with untreated fiber, as mentioned before, must occur more intensely when the fiber is treated with alkali. The morphological changes caused by the treatment with NaOH can facilitate the resol prepolymer flux in the lignocellulosic network, leading to high interfacial strength, which can even be a consequence of reactions involving hydroxymethyl groups of the resin with hydroxyl groups, for example, with those of cellulose.³¹

Figures 11 and 12 support the previous proposition, because it is evident that the degree of adhesion is greater in the reinforced composite treated with alkali. The fibers fracture at the crack plane without the occurrence of pull-out (Fig. 12), whereas in the composite reinforced with untreated fiber, the pull-out mechanism can be observed (Fig. 11).

The treatment with ionized air for 1 h did not prove to be efficient for improvement of impact strength of jute fiber, except when combined with alkali treatment (composite with 30% of fiber). However, the composite reinforced with these ionized air-treated fibers (20 and 30% of fiber) presented a lower standard deviation in the impact strength measurement (Figs. 9 and 10). This result indicates that this treatment produced a more efficient separation of the fiber bundle, making possible a more homogeneous distribution in the matrix.



Figure 11 SEM photomicrograph of the impact fracture surface of untreated jute fiber phenolic composite (\times 1000) (fiber content: 20 wt %; fiber length: 3 cm). Pull-out mechanism occurrence indicated by arrows.



Figure 12 SEM photomicrograph of the impact fracture surface of alkali (5% NaOH) jute fiber phenolic composite (\times 1000) (fiber content: 20 wt %; fiber length: 3 cm).

CONCLUSIONS

Among the jute fiber treatments considered in the present work, the treatment with a solution of 5% NaOH presented the best results because of the following factors:

- The fiber presented a higher tensile strength, and a larger percentage of elongation at break.
- The composite reinforced with this fiber presented the highest impact strength results when this was the unique treatment (20% of fiber), as well as when it was combined with ionized air (30% of fiber).
- Among all composites, the one that presented the lowest water uptake was that reinforced with this fiber.

Further investigation of the application of ionized air is worthwhile because of its great advantages in relation to the treatment with the alkali, particularly because it is a "dry method." This characteristic eliminates the hard drying step of the hygroscopic lignocellulosic fiber. For this purpose, parameters such as increasing the fiber exposition time to ionized air should be varied, with the objective of producing materials with better properties compared to those obtained in this work when this treatment was used.

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