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Impact of alkaline hornification in jute fibers on the tensile strength

Abstract

Wetting and drying cycles are usually used in the paper and cellulose industry aiming to achieve a reduction in the water absorption capacity of lignocellulosic fibers. This procedure stiffens the polymeric structure of the fiber-cells (process known as hornification) resulting in a higher dimensional stability. Several authors have proposed treatments in natural fibers, including hornification, that modifies the surface of the fibers and increase the mechanical behavior. The present study presents a comprehensive analysis of the influence of alkaline hornification with calcium hydroxide 0.7% (1 cycle) on the structure modification, mechanical response, durability performance and bond behavior of jute fibers. The intrinsic changes on the fiber structure as well as their physical and chemical characteristics were evaluated through analytical techniques such as X-ray diffraction (XRD), Thermogravimetry (TGA), Fourier Transformed Infrared (FTIR) and Scanning Electronic Microscope (SEM), while their mechanical response was evaluated with direct tensile tests. The obtained results indicate that the hornification process removes partially the lignin and hemicelluloses from jute fibers, which changes the fiber properties, increasing their crystallinity, altering their morphology, by an increase in the thickness of the secondary fiber wall and reduction of the lumen, and increasing their mechanical resistance.

Keywords: jute fiber, chemical treatment, alkaline hornification, tensile strength, dimensional stability

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Abbreviations: XRD, x-ray diffraction; TGA, thermogravimetry; FTIR, fourier transformed infrared; SEM, scanning electronic microscope; EDX, energy-dispersive x ray spectroscopy; NaOH, sodium hydroxide

Introduction

Vegetables fibers are being used as reinforcement in cementitious composites not only for their reinforcement capability but also for other advantages, such as biodegradability, abundance, low cost, low health risk and the potential of economic development in the regions where they are cultivated. Nevertheless the use of vegetable fibers also presents some problems such as high water absorption and low durability in alkaline media and this can lead to fiber mineralization and low adhesion with cementitious matrices.[1–3] In order to overcome these problems some different strategies can be used, in an isolated or associated way, among them the most important are

i. The use of calcium hydroxide ($Ca(OH)_2$) free matrices, which is replacing some cement for some clay preventing excess of $Ca(OH)_2$ in the matrix

ii. The application of some chemical treatments such as acetylation, hornification, polymer impregnation, alkaline and thermal treatment.[4–6]

The treatment of vegetable fibers with sodium hydroxide (NaOH) is widely used to modify the cellulosic molecular structure. It changes the orientation of highly packed crystalline cellulose order and forms an amorphous region. This provides more access for chemicals to penetrate. In the amorphous region, cellulose and small molecules are separated, increasing distances and the space for water molecules to infiltrate. Alkali sensitive hydroxyl (OH) groups, present among the molecules, are broken down, which then react with water molecules (HOH) and move out from the fiber structure. The remaining reactive molecules form fiber–cell–O–Na groups between the cellulose molecular chains. Due to this, hydrophilic hydroxyl groups are reduced and the fibers moisture resistance property increases. It also reduces a certain portion of hemicelluloses, lignin, pectin, wax and oil covering materials. As a result, the fiber surface becomes clean and more uniform due to the elimination of microvoids and thus the stress transfer capacity between the ultimate cells improves. In addition to this, it reduces fiber diameter and thereby increases the aspect ratio (length/diameter) of the fiber. If the alkali concentration and/or exposition are higher than the optimum, the excess delignification of the fiber can take place, which results in weakening or damaging the fibers. Treated fibers have lower lignin content, a partial reduction of wax and oil cover materials and distension of crystalline cellulose order.[7–8]

In this study, jute fibers were submitted to one cycle of soaking and drying in an alkaline hornification treatment with calcium hydroxide ($Ca(OH)_2$) 0.7% w/v, to evaluate the impact on the fiber structure and the consequences in their stress-strain behavior. $Ca(OH)_2$ was selected because its presence in the matrix causes severe damage to the vegetable fibers, even in small concentration if the exposure was for long time. Thus, the main objective of this study is to understand better the mechanism that $Ca(OH)_2$ modifies the vegetable fiber structure and how these changes influence the stress-strain behavior.

Materials and methods

Manaus

Jute fibers came from the river Amazon between Manáus and Santarém (AM), Brazil. The calcium hydroxide P.A. was supplied

by Vetec. The used water was distillated in the own laboratory. All materials were used as received.

Vegetable fibers calcium hydroxide hornification

Vegetable fibers were soaked in 0.7% w/v of $Ca(OH)_2$ solution under controlled temperature (21±1°C) for 50 min. Literature[9] shows that soaking for periods from 30 to 60 min, in small alkali concentrations (0.5-1%) causes no degradation to the vegetable fibers. After that, the $Ca(OH)_2$ saturated fibers were dried in an air flow chamber at 40°C for 24h up to constant mass.

Characterization of jute fiber

FTIR spectra were performed using a Perking Elmer spectrometer, model Frontier FT-IR/FIR, and ATR with a ZnSe crystal. The range measured was from 4000 to 600cm^{-1}, with 4cm^{-1} of resolution and 60 accumulated scans. XRD diffractograms were carried out using a Bruker, model D8 Focus X ray diffractometer with the FT (fixed time) method and CuKα radiation, with wavelength of 0.1542nm. The used 2θ range was from 10 to 40°C with angular steps equal to 0.05°/s and the tube voltage and current were equal to 30kV and 35mA, respectively. The crystalline degree was calculated by the Ruland method[10] [Eq. 1]:

$$X_C = \left(\frac{A_C}{A_C + A_a} \right).100 \qquad \text{Eq (1)}$$

Where X_C is the crystalline degree, A_C is the crystalline area and A_a is the amorphous area. TGA analysis was performed using a TA instrument model SD 2960 with heating rate of 10°C/min under a nitrogen flow of 100mL/min and temperature range from 35 to 800°C. The initial sample was around 10mg in Pt pan. The morphology of the fibers was determined using a scanning electronic microscope (SEM) from Hitachi model TM3000, under vacuum with a secondary electron detector from Everhart-Thornley-ETD and voltage of 15kV. The use of energy-dispersive X ray spectroscopy (EDX) is associated with SEM.

Mechanical properties

The tensile strength tests of the fibers were carried out using an electromechanical device-Shimadzu AG-X100kN with a load cell of 1kN and a displacement rate of 0.1mm/min. The fibers, with a length of 50mm, were glued to a paper template, for a better alignment in the machine and a better gripping in the upper and lower jaws, in accordance with ASTM C1557.[11] In order to calculate the tensile strength of the fibers, their diameters were measured by analyzing the images obtained from SEM.

Results and discussion

Figure 1 presents the FTIR spectra of jute fibers before and after the alkaline hornification cycle. The spectra show same characteristic bands, typical for lignincellulosic materials. Stretching of-OH group can be seen around 3350cm^{-1}, while the C-H stretching appears around 2922, 2848 and 1363cm^{-1}. The characteristic band at 1742cm^{-1} is related to coupled stretching of C=O and the C=C bonds. Bending mode of -OH group from water in the hemicelluloses is observed around 1640 cm^{-1}, while the aromatic skeleton vibration of C-C bonds shows up at 1424cm^{-1}. Scissoring deformation in the plane of the ring of O-H bond is at 1324cm^{-1} and aromatic stretching of C=O is at 1246cm^{-1}. The band at 1161cm^{-1} is attributed to the C-O-C asymmetric stretching, and symmetric stretching of this bond appears at 1106cm^{-1}. Stretching O-C-C of appears at 1167cm^{-1} and 1038cm^{-1}. The characteristic band at 895cm^{-1} is related to C-H scissoring deformation out-of-plane in the aromatic ring.[12–14]

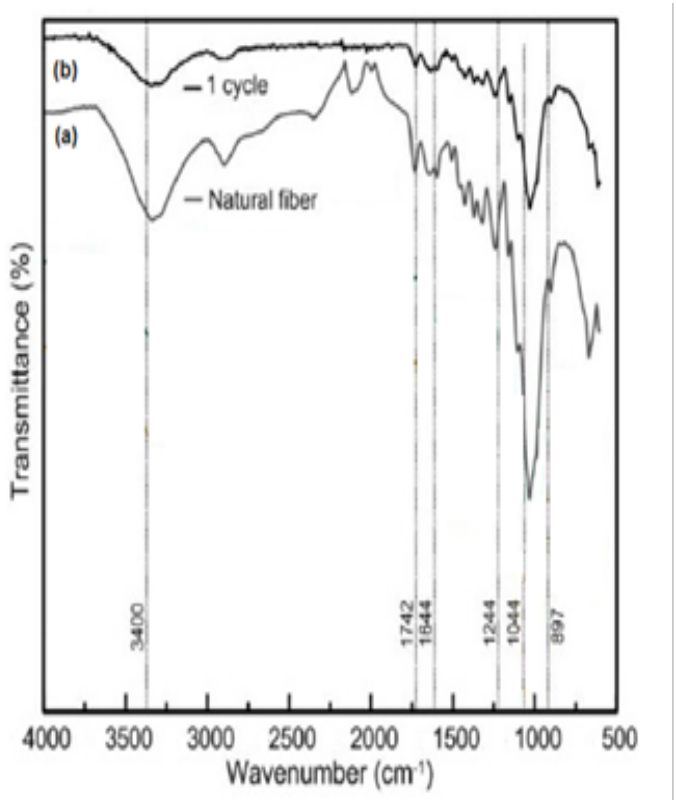

Figure 1 FTIR spectra of jute fibers: (A) natural, (B) 1 cycle $Ca(OH)_2$.

Hornification treatment caused a reduction in the intensity of jute fibers spectra, mainly in the characteristic bands associated with the aromatic compounds, in this case, the lignin, which proves the considerable removal of this component from the fiber by the treatment.

Figure 2 shows the XRD of jute fibers before and after the alkaline hornification cycle. It is possible to observe a typical diffraction pattern of ligniclellulosic materials, with 2 theta peaks at 16.6°C, 22.5°C and 34°C attributed to (101), (002) and (040) respectively.[15] As expected the removal of lignin and hemicelluloses, which are amorphous, causes an increment in the crystalline, as noticed by the higher intensity in the peaks signal. The calculated crystalline degree is presented in the Table 1.

Table 1 Calculated crystalline degree of jute fiber

Jute fiber treatment	Crystalline degree (%)
natural	30.1±1.5
1 cycle $Ca(OH)_2$	47.1±2.4

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Figure 3 presents the results to TGA/DTG of the jute fibers. The thermal degradation profile of this kind of fiber occurs in three stages of weight loss:

i. Evaporation of moisture, below 100°C

ii. Decomposition of hemicelluloses, from 250 to 350°C

iii. Degradation of cellulose, from 325 to 400°C.

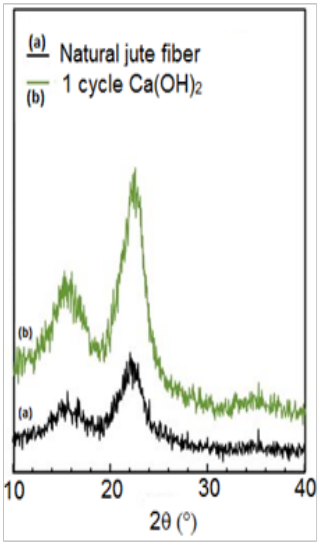

Figure 2 XRD of jute fibers: (A) natural, (B) 1 cycle $Ca(OH)_2$.

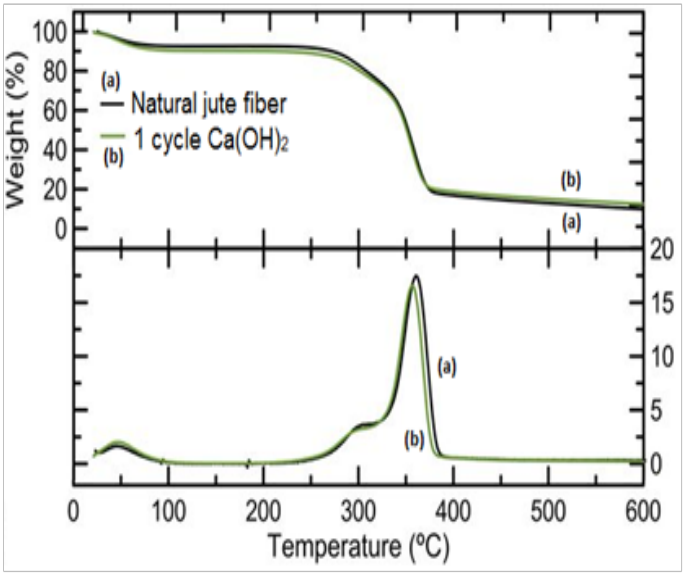

Figure 3 TGA/DTG of jute fibers: (A) natural, (B)1 cycle $Ca(OH)_2$.

The later one is major weight loss detected in this sample. Besides that, the lignin degradation occurs between 200°C and 600°C, with no evident degradation step, due to the way the lignin is spread in the cellulosic fiber structure.[16] It was noted that the hornification treatment reduces the amount of hemicelluloses present in the fiber.

Comparing the cross section of the fibers, before and after hornification treatment, shown in Figure 4, in this case an increase could be seen in the thickness of the secondary wall, like a swelling, causing a reduction of the fiber lumens. Figure 5 presents the side structure of jute fibers, before and after the treatment. It is possible to see some $Ca(OH)_2$ deposition on the fiber surface. None significant change was observed in the fiber after only one cycle of alkaline hornification.

Stress-strain behavior curves of the jute fibers are shown in Figure 6. Mechanical properties of these fibers, obtained from the curves presented in Figure 6, are presented in Table 2.

The hornification treatment produced an increment in the mechanical resistance of fibers. This increment for jute fiber was 70% in the max load, 176% in the tensile strength, 133% in the strain and 9% to the Young's modulus. As described in the literature[7–8] these kind of treatment removes partially the lignin and hemicelluloses from the fibers, leaving the cellulose, which is the crystalline phase and more resistant mechanically. Moreover, the lignin and hemicelluloses removal results in a reduction of fiber diameter, increasing their aspect ratio (length/diameter) and contributing to the improvement of the mechanical resistance.

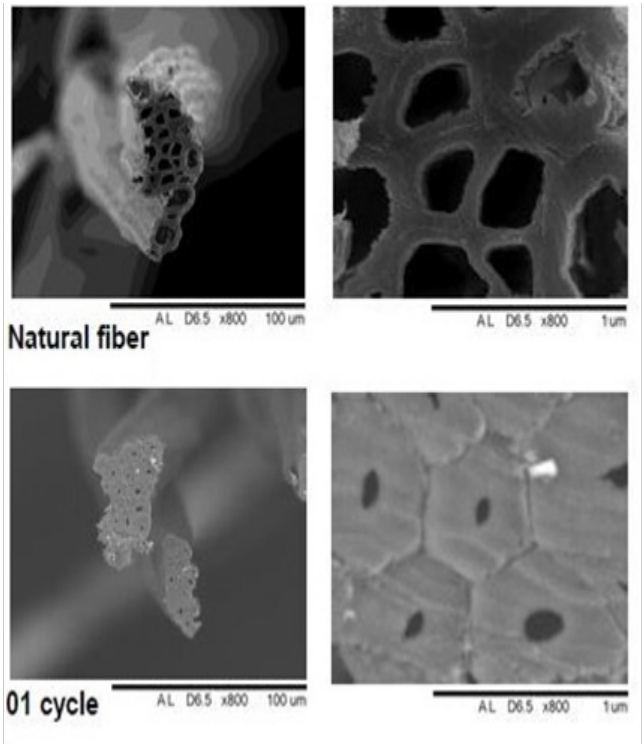

Figure 4 SEM images of cross section of jute fibers: natural and 1 cycle $Ca(OH)_2$.

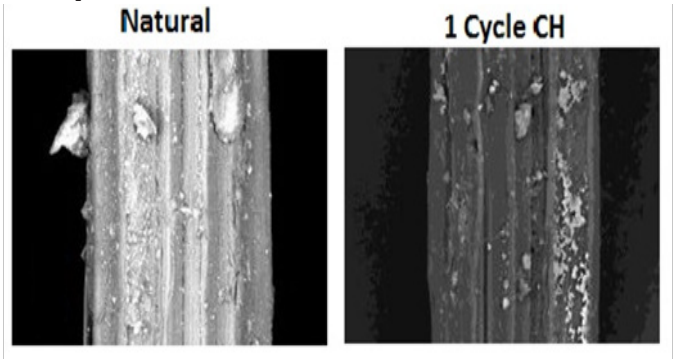

Figure 5 SEM images of side of jute fibers: natural and 1 cycle $Ca(OH)_2$.

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Table 2 Mechanical properties of tested jute fibers

Fiber	Treatment	Max load (N)	Tensile strength (MPa)	Tensile strain (mm/mm)	Young's modulus (GPa)
Jute	Natural	1.05±0.37	249±89	0.006±0.002	44±12
	1 Cycle $Ca(OH)_2$	1.78±0.16	687±134	0.014±0.003	48±14

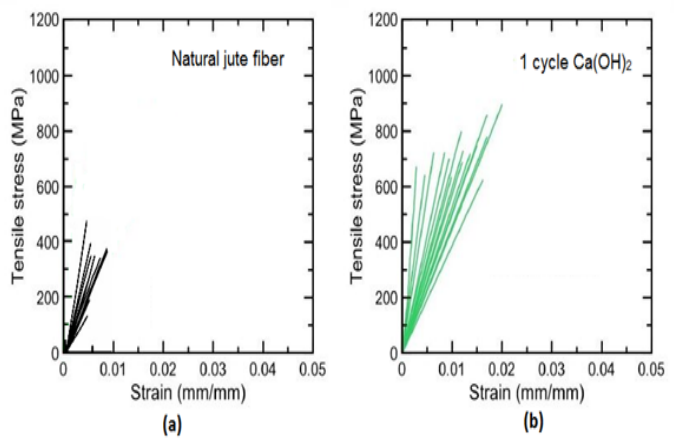

Figure 6 Stress-strain behavior curves of jute fibers: (A) natural and (B) 1 cycle $Ca(OH)_2$.

Conclusion

Alkaline hornification treatment with calcium hydroxide, even with only one cycle of soaking and drying, proved to be very efficient to remove partially the lignin and hemicelluloses from the jute fibers. This kind of treatment is able to increase the crystalline degree of fibers. Besides that, alkaline hornification also promotes changes in the fiber morphology, with an increase in the thickness of the secondary fiber wall and reduction of the lumen. With regards to the mechanical resistance, the alkaline hornification treatment with only 1 cycle has improved significantly the mechanical resistance by 70% in the max load, 176% in the tensile strength, 133% in the strain and 9% to the Young's modulus for the jute fiber.

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Conflict of interest

The author declares no conflict of interest.

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