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Technical note

Alkaline surface modification of sugar cane bagasse

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Abstract—One of the most important issues in producing composite materials is fibre sizing, due to the usual incompatibility between the fibres and the matrix. In the present work, the evaluation of an alkaline surface treatment of sugar cane bagasse fibres through Scanning Electron Microscopy(SEM), X-ray microanalysis, Fourier Transform Infrared Spectroscopy (FTIR) and Thermogravimetric Analysis (TGA) is reported. The results show that the treatment with calcium hydroxide [$Ca(OH)_2$] chemically modifies the surface of the bagasse fibre producing a calcium carbonate deposit on the surfaces, whereas sodium hydroxide [NaOH] has little or no effect. The main action of NaOH on the fibre is to remove the lignin binder of the cellulosic material.

Keywords: Sugar cane bagasse; surface studies; alkaline treatment; cellulosic materials; fibre sizing.

1. INTRODUCTION

Composite materials are usually based on high-strength, high-stiffness fibres and a relatively ductile supporting material called matrix, in which the fibres are embedded.

In producing this type of material, a very important factor is the chemical or physical interaction between the fibre and the matrix, if the load from the matrix to the fibre in a real strengthening process is to be achieved. In most cases, fibres are usually incompatible with matrixes, so fibre surface treatment (sizing) has been used normally to improve the compatibility between them. This approach has been applied in cases ranging from glass fibre sizing with silanols [1], carbon fibre sizing

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by oxidative etching or surface coatings [2] and cellulose fibre sizing by different methods such as delignification [3], surface acetylation [4], and coating by graft polymerization [5].

On the other hand, an interesting alternative for achieving low cost reinforcement of materials is through the use of natural fibres which grow abundantly in nature. Many attempts have been made to obtain practical applications. These include studies of fibres from rice husk [6], sugar cane bagasse [7], zacate [8], jute [3], sisal [4], and bamboo [9], among others.

Only a few of the above studies were dedicated to the production of cement-based composites [10–13]. For the case of cement-based composite materials, the most common treatment of the fibres involves the use of alkaline solutions. To our knowledge, no report has been published in the specialized literature related to the study of the effect of these treatments on the fibre surface. Accordingly, in the present communication, the effect on the sugar cane bagasse-fibres surface of alkaline treatment by sodium hydroxide or calcium hydroxide, is studied by SEM, X-ray microanalysis, FTIR and TGA.

2. EXPERIMENTAL

2.1. Materials

The sodium and calcium hydroxides used were purchased as Baker Co. reagent grade and the sugar cane bagasse was kindly supplied by the sugar cane industry 'Emiliano Zapata' in Zacatepec, Morelos, Mexico.

2.2. Alkaline treatment

50 g of sugar cane bagasse was mixed with a liter of either calcium hydroxide (0.5 M) or sodium hydroxide solution (1.0 M). Then, the mixture was heated to boiling. After 2.5 h of stirring, the fibre was separated by filtration and rinsed with distilled water until neutral pH was achieved (five washes, on the average). After rinsing, the fibre was dried in an oven for two days at 90°C.

2.3. Evaluation of the surface modification

2.3.1. Scanning Electron Microscopy (SEM). Samples of the treated fibres were placed in a vacuum chamber and a thin layer of gold was then deposited onto them to diminish charging effects. The SEM observations were carried out in a JEOL 5300 instrument, at 5 keV. Prior to the gold coating, the samples were observed with the SEM instrument at 10 keV and X-ray microanalysis using an Energy Dispersive System (KEVEX-EDS) was performed.

2.3.2. Thermogravimetric Analysis (TGA). 6 mg to 14 mg samples were evaluated from 20°C to 900°C under dry nitrogen flux (34 ml/min), at a heating rate of 10°C/min. Calcined alumina was used as a reference material. The instrument used in this characterization technique was a simultaneous thermal analysis equipment (STA-780 from Stanton Redcroft).

2.3.3. Fourier Transform Infrared Spectroscopy (FTIR). The powdered samples were mixed with KBr and pressed to pellets. The pellets were analyzed by the transmission mode with a resolution of 4 cm^{-1} and strong numerical apodization. The absorption range studied was from 4000 cm^{-1} to 400 cm^{-1} using a Perkin-Elmer spectrophotometer (FTIR 1600).

3. RESULTS AND DISCUSSION

Figures 1 to 3 show SEM images at two magnifications of the fibres untreated (Fig. 1) and treated with sodium hydroxide (Fig. 2) and calcium hydroxide (Fig. 3), respectively. As can be observed, the sodium hydroxide treatment only eliminates the lignin binder in the sugar cane bagasse providing the fibre surface with a greater roughness (Fig. 2). Besides this, the fibre surface does not have any apparent modification when compared with Fig. 1. In the case of calcium hydroxide treatment (Fig. 3), a clear surface modification has taken place all over the fibre, as can be seen from the appearance of small particles. These differences in surface modification are highlighted by the results of X-ray microanalysis (Fig. 4). While the elements present in both untreated (a) and NaOH treated fibres (b) are almost the same, in the $Ca(OH)_2$treated sample (c) a strong independent Ca-signal appears. Also, the relative intensity of the carbon-to-oxygen signal is higher for the untreated fibres as compared to the treated fibres, probably due to the lignin binder (mainly hydrocarbon), which is partially removed by the alkaline treatment. The FTIR-spectra shown in Fig. 5 confirms these results. The elimination of lignin binder in the fibre treated with sodium hydroxide (b) is again observed by the lack of some of the typical absorption bands of lignin [14] at 1600 cm^{-1},1509 cm^{-1} and 1252 cm^{-1} compared with spectra of the untreated fibre (a). The spectrum of the $Ca(OH)_2$ treated fibre (c) clearly shows new absorption bands, namely, 2507 cm^{-1} (OH of carboxylic acid), 1795 cm^{-1} (carbonyl) and 871 cm^{-1} (carbonate) along with a very strong band at 1423 cm^{-1} (ionic carboxylate), corresponding to the calcium deposit on the fibre surface.

The effect of the modification of the sugar cane bagasse surface on the thermal stability of the fibres was analyzed through thermogravimetric analysis. As can be observed in Table 1, the thermal decomposition of the fibres presents three stages. The first stage corresponds to the elimination of volatiles (average temperatures 52–59°C). The second stage is the main decomposition event of the cellulosic material (average temperatures 353–370°C) and the last stage corresponds to the final decomposition (temperatures above 600°C) resulting in a char of carbon and inorganic oxides [15]. The weight loss of the fibres up to 370°C is 71% for the untreated, 62.2% for the NaOH-treated and only 27.6% for the $Ca(OH)_2$-treated

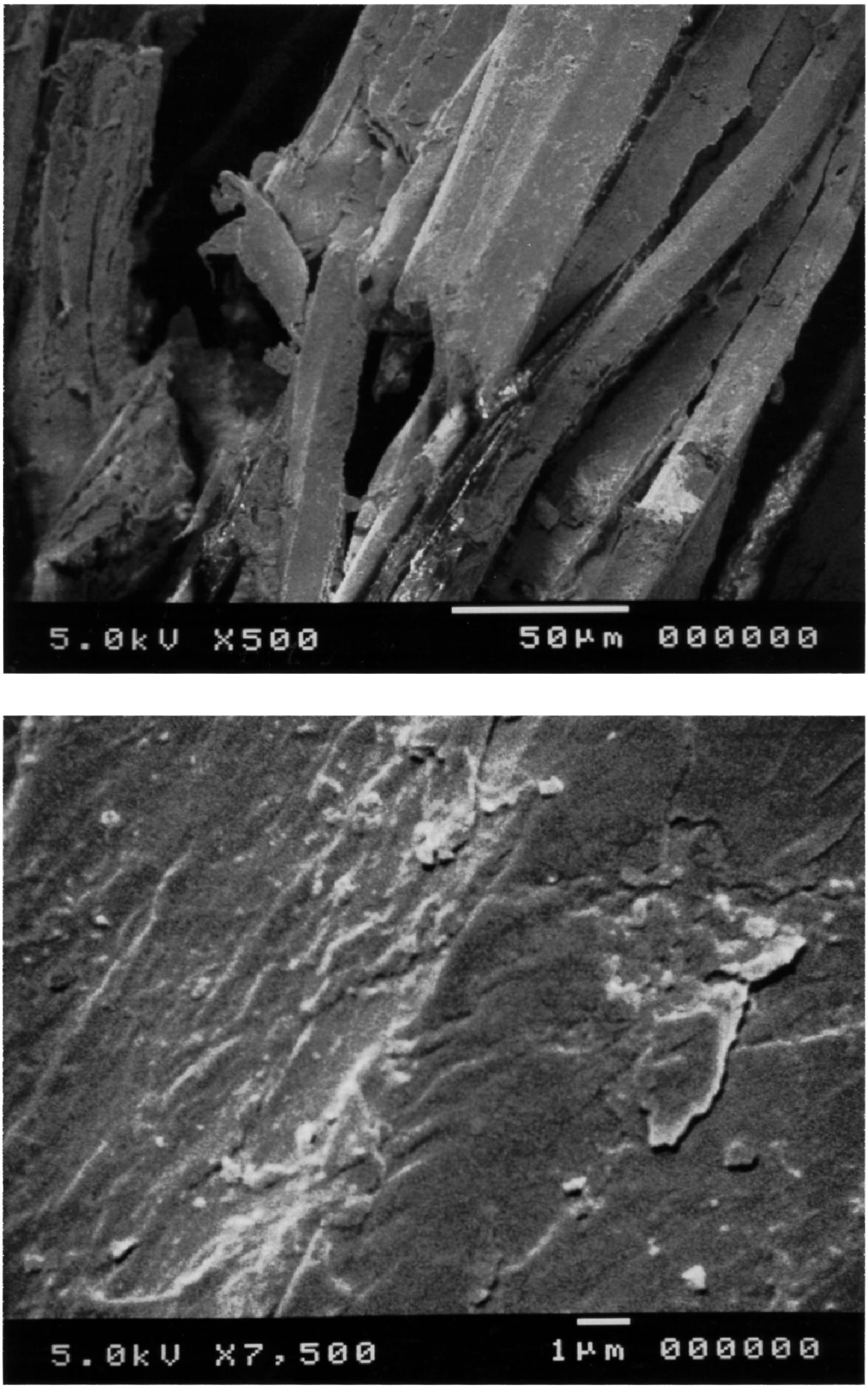

Figure 1. SEM images of untreated sugar cane bagasse fibres.

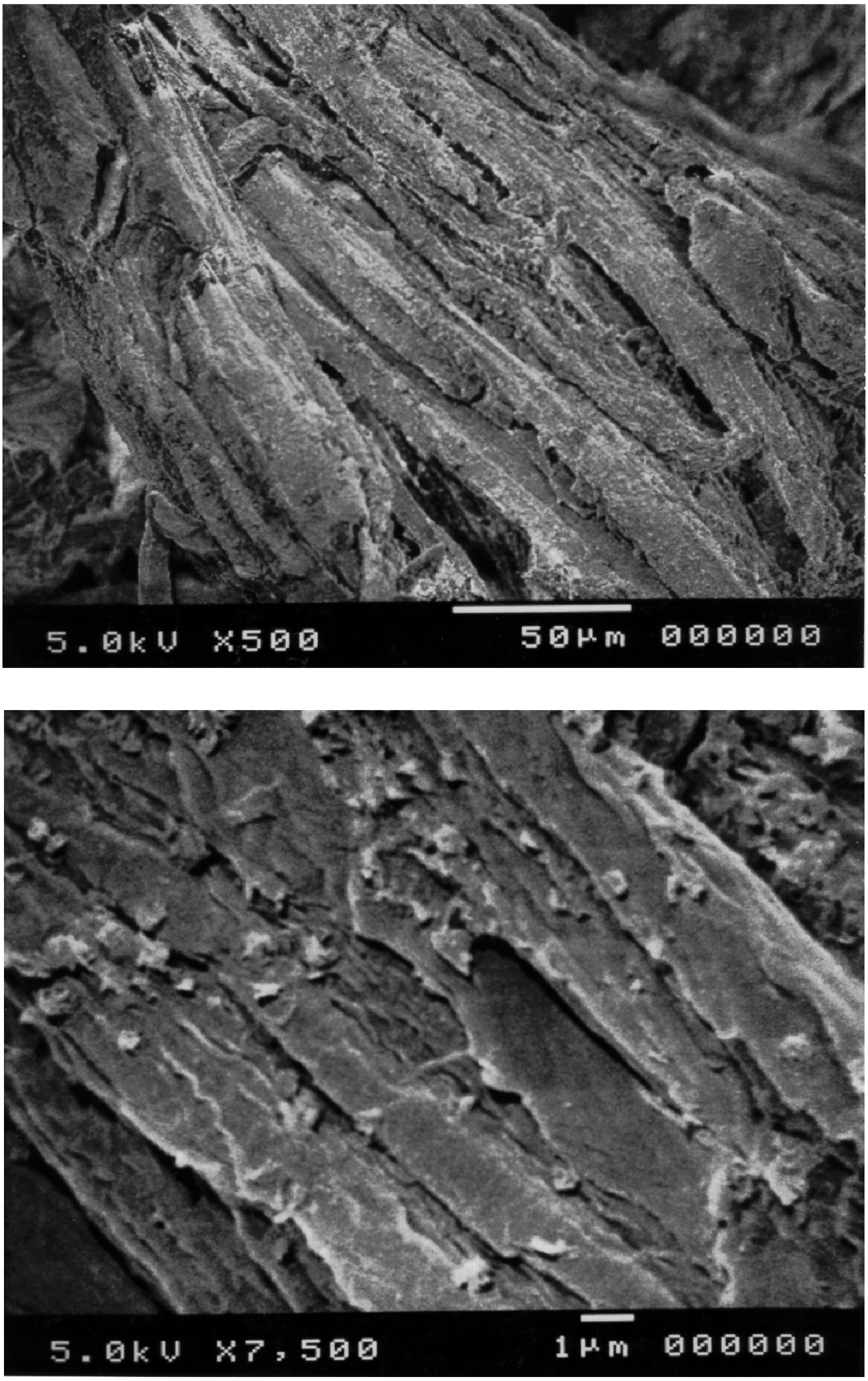

Figure 2. SEM images of NaOH treated sugar cane bagasse fibres.

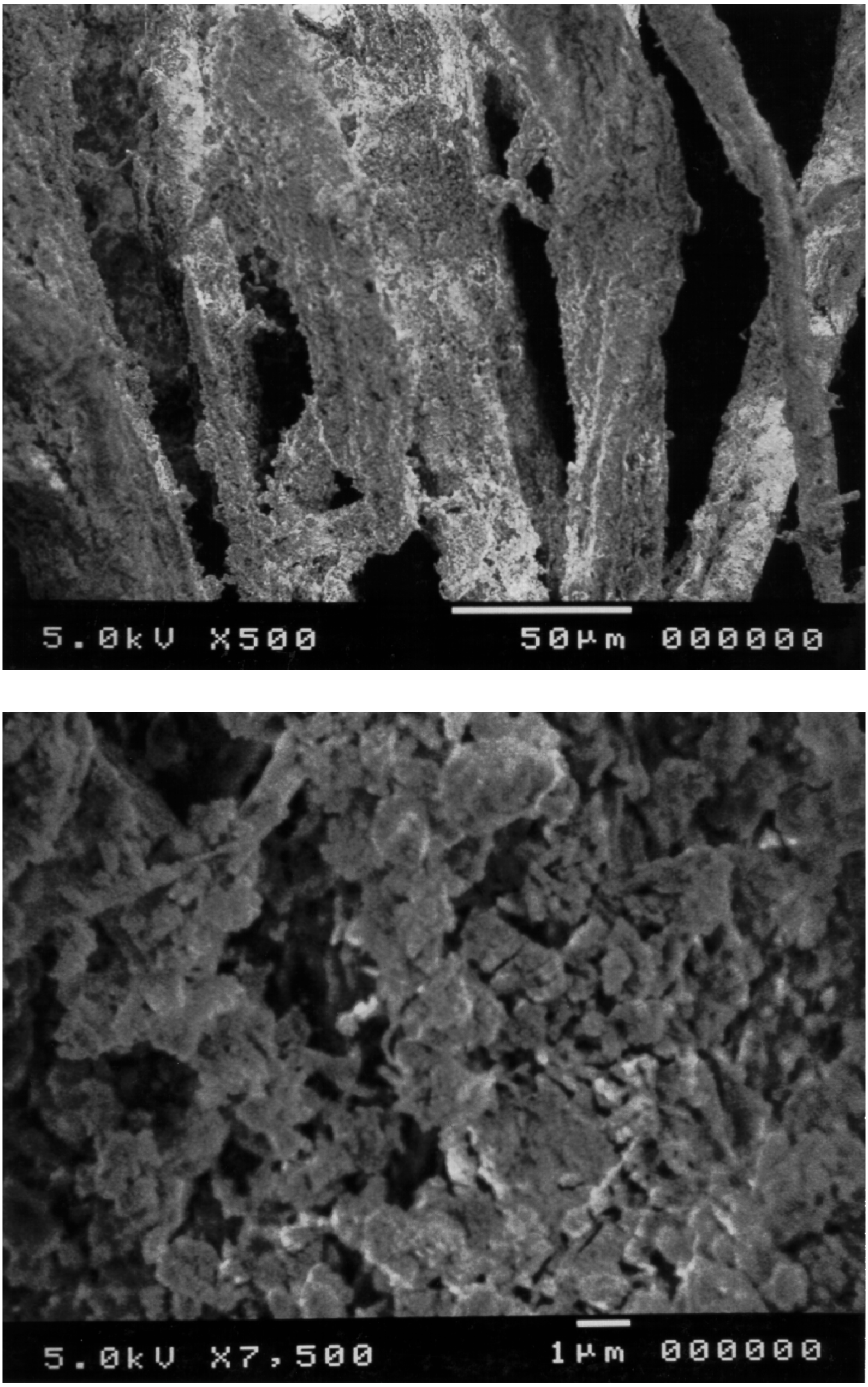

Figure 3. SEM images of $Ca(OH)_2$ treated sugar cane bagasse fibres.

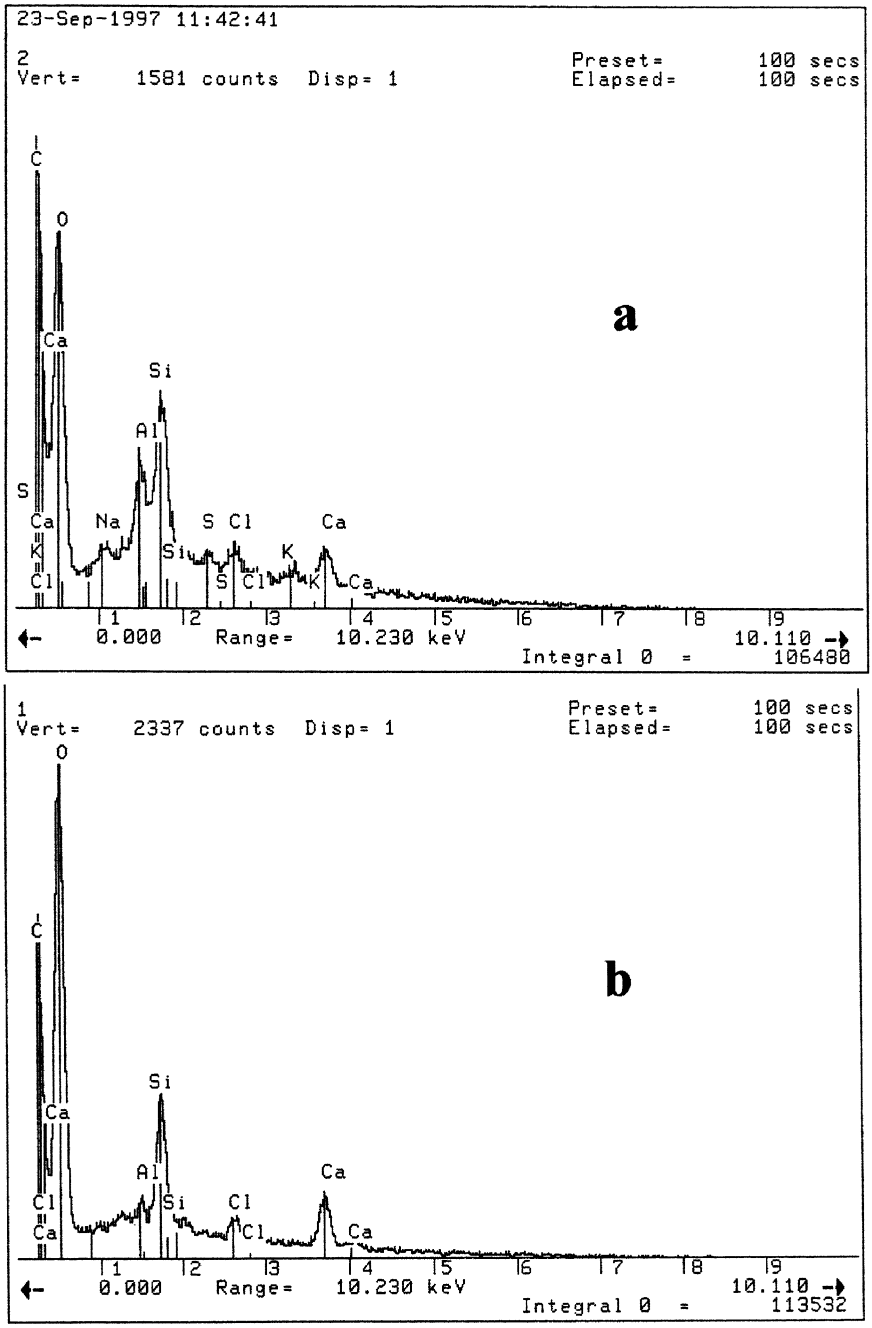

Figure 4. X-ray microanalysis results on sugar cane bagasse fibres. (a) Untreated; (b) NaOH treated; (c) $Ca(OH)_2$ treated.

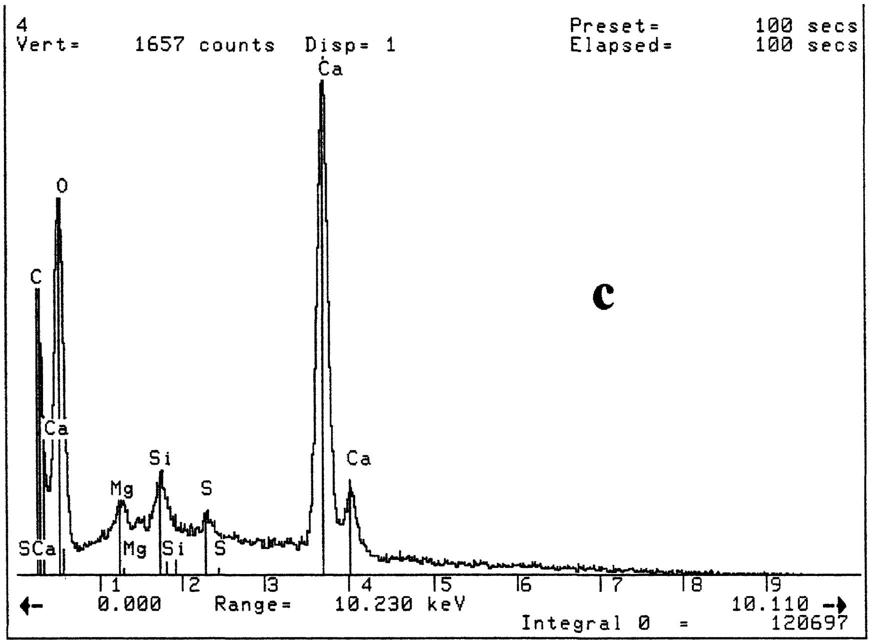

Figure 4. (Continued).

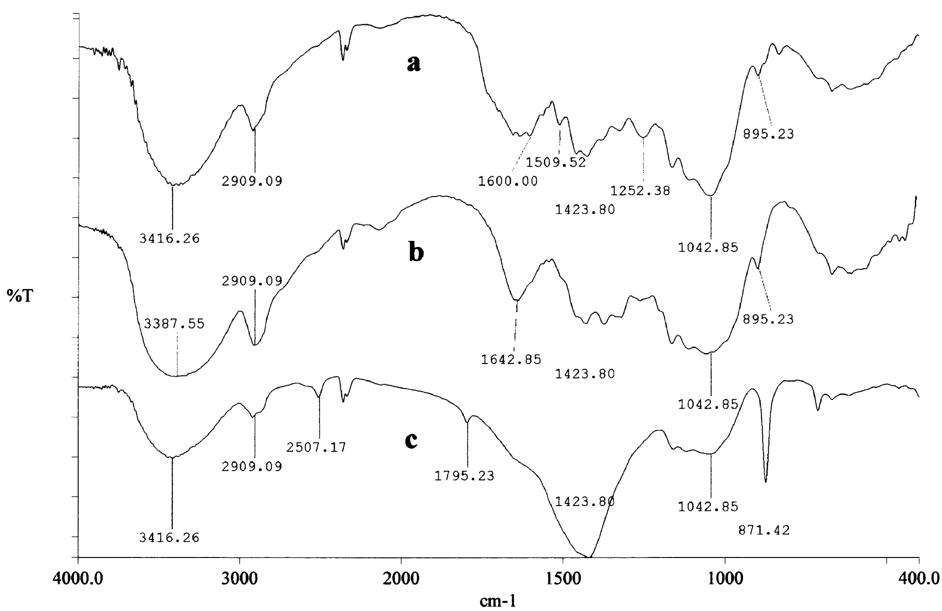

Figure 5. FTIR-spectra of sugar cane bagasse fibres. (a) Untreated; (b) NaOH treated; (c) $Ca(OH)_2$ treated.

Table 1.
Results of thermogravimetric analysis on sugar cane bagasse fibres.

Parameter	Untreated	NaOH treated	$Ca(OH)_2$ treated
T_{D1} [°C]	52	59	52
Weight loss (1) [%]	8.0	6.6	5.8
T_{D2} [°C]	353	362	370
Weight loss (2) [%]	71.5	68.5	30.3
T_{D3} [°C]	—	—	750
Weight loss (3) [%]	8.0	6.2	25.4
Weight loss at 370°C [%]	71.0	62.2	27.6
Residue at 900°C [%]	13.4	18.0	38.5

T_{D1}, T_{D2} and T_{D3} are the mean temperatures of the decomposition events.

fibres. Therefore, the thermal stability of the fibres treated with calcium hydroxide is higher than the one treated with sodium hydroxide and the one without treatment, providing further significant evidence of the bagasse surface modification.

4. CONCLUSIONS

The surface modification of sugar cane bagasse by an alkaline treatment has been studied by SEM, X-ray microanalysis, FTIR and TGA.

The calcium hydroxide treatment really modifies the sugar cane bagasse surface yielding a calcium carbonate deposit all over the fibres that results in an increased thermal stability of the treated fibres. Sodium hydroxide does not chemically modify the fibre surface; nevertheless it eliminates lignin, an inhibitor to the hydration of cement in composites based on natural fibres, increases the roughness of the fibre surface and also slightly increases the thermal stability of the treated fibres. The production and characterization of sugar cane bagasse/cement composites using calcium hydroxide treatment is now under study and will be reported separately.

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