THERMOGRAVIMETRIC ANALYSIS AND WEATHERING STUDY BY WATER IMMERSION OF ALKALI-TREATED BAMBOO STRIPS

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The effects of alkali treatment on the thermal degradation and weathering properties of bamboo strips were investigated in this work. Dried bamboo strips with average dimensions of 100 x 15 x (1.1-1.5) mm were mercerized with caustic soda solution (10%, 15%, 20%, and 25%) (w/v) at ambient temperature, maintaining a liquor-to-solids ratio of 15:1. All types of bamboo specimens were subjected to thermogravimetric analysis and accelerated weathering by water immersion and further characterized by flexural property measurements. Water absorption and dimensional changes were recorded at 100% humidity, room temperature, and atmospheric pressure. The results showed that the treated bamboo strips had a higher deterioration in flexural properties compared to the untreated ones. Thermogravimetric analysis of all the samples indicated better thermal properties of alkali-treated samples. The highest activation energy was observed with a 15% alkali-treated bamboo sample.

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INTRODUCTION

Nowadays there is a growing interest to properly utilize natural fibres, mainly due to their high specific modulus, light weight, low cost, and resistance to deforestation (as natural fiber by suitable modification can be used as a replacement of wood), in addition to the other usual advantages (Karmaker et al. 1996; Chand et. al. 1998; Varma et al. 1984; Jain et al. 1992; Jain et al. 1993; Umadevi et al. 1997; Avella et al. 1998). Bamboo has considerable potential as a reinforcing fiber, in view of its high strength.

Bamboo culm itself resembles a unidirectional fiber-reinforced composite with many nodes along its length. Like many other natural fibers, bamboo is not readily available in fine-fiber form, so bamboo is commonly used in strip, woven strip (mat), and dust form by different workers to fabricate composites and for evaluation of different properties (Jain et al. 1992; Jain et al. 1993; Amada et al. 1997; Das et al. 2006a; Das et al. 2006b). However, a handful studies are available related to the characterization of these fibers. Amada et al. (1997) have reported on the structural variation in bamboo with cross-sectional diameter and height. Jain et al. (1992) studied the mechanical properties of bamboo. The results of these studies reveal that the mechanical properties of bamboo vary along and across the orientation of the cellulose fibers. Despande et al. (2000) developed methods for extraction of bamboo fibers and evaluated their mechanical properties. Das et al. (2008) investigated the effect of mercerization on the mechanical properties of bamboo, thermal properties of bamboo (2006b), and fine structure and

morphology of bamboo fiber (2006a). They reported that the weight loss value of bamboo strips due to alkali treatment (0-50% alkali concentration) shows a maximum of 21.94% at 50% alkali treatment. The mechanical properties of bamboo strips increase steadily with increasing concentration of caustic soda, showing a comparable increased value at 15 and 20%, and then exhibiting a gradual fall (2008). XRD assessment on those treated and untreated bamboo samples in both strip and dust form by Das et al. revealed that during alkali treatment a lattice transformation from cellulose-I to cellulose-II took place. Morphological study of bamboo dust with scanning electron microscope indicates fibrillation at higher alkali concentration (2006a). Das et al also exposed the effect of alkali treatment on differential scanning calorimetry (DSC) and dynamic mechanical thermal analysis (DMTA) studies of untreated and alkali treated bamboo fibers.

Bamboo, like other natural fiber, is hygroscopic and exhibits a tendency to be in moisture equilibrium with the relative humidity of the surrounding atmosphere, either by taking up moisture from or giving out moisture to the atmosphere. They are very prone to swell / warp and shrink when exposed to moist and hot weather conditions respectively. However, for applications like composites, this aspect is detrimental so far as dimensional stability is concerned. Natural fibers absorb moisture as the cell wall polymers contain hydroxyl or other oxygenated groups that attract moisture through H-bonding. Taking advantage of the plentiful reactive groups present, modification of cell wall using proper surface modifier can be made to increase the scope of utilization of the natural fibers as reinforcement (Rao et al. 1981; Dash et al. 1999).

Mercerization is one of the most conventionally used treatments for natural fibers to modify its surface properties so that it can be successfully employed in composite formation (Gassan and Bledzki 1999). Available literature pertaining to alkali treatment of many natural fibers reveal that the process removes one of the cementing materials, hemicellulose, depending on the concentration of the alkali used, time and temperature of treatment, liquor ratio, etc. Thereby alkali treatment renders the changes in fibre properties by altering the fine structure and morphology of fibres (Gassan and Bledzki 1999; Prasad et al. 1983; Chand et al. 1986). Ray et al. (2001, 2002) also noted the changes occurring in jute fibers when treated with a 5% concentration of a NaOH solution for 0, 2, 4, 6, and 8 hours with respect to mechanical properties, weight loss, FTIR, X-ray TGA, DSC, and SEM measurements. The weight of the char residue at 600°C increased from 15% in the untreated fibers to 22% in all the treated fibers, with a lowering in the enthalpy values for α-cellulose degradation up to 4 hrs of treatment, followed by an increase in the enthalpy values in 6 and 8 hrs treated fibers due to increased crystallinity of these fibers. Similar types of thermal and weathering studies have been reported on many other natural fibers and their composites (Ray et al. 2002; Basak et al. 1993; Varma et al. 1988; Rajulu et al. 2002; Stamboulis et al. 2000; Rao et al. 1981). Although a large number of studies have been reported by different workers on the weathering behaviour and thermal stability of natural fiber and their composites, similar studies on bamboo are scanty (Rajulu et al. 2002).

The present paper aims at the evaluation of effect of alkali treatment on thermogravimetric analysis of bamboo fiber (in strip and dust form) and also on property deterioration under conditions of 100% humidity and water immersion for 5 days.

EXPERIMENTAL

Materials

Bamboo belonging to the variety *Bamboosa balcua* was supplied by FOSET (Forum of Scientists, Engineers and Technologists) , West Bengal. It was supplied in particulate form (30-36 mesh size) and also in strip form with an average dimension of (100x15x1.5) mm. This specimen was used throughout the study.

Method of mercerization

Dried Bamboo strips having average dimensions of 100 x 15 x (1.1-1.5) mm were soaked in caustic soda solution with varying concentration (10%, 15%, and 20%) (w/v) at ambient temperature, maintaining a liquor-to-solids ratio of 15:1. The fibers were kept immersed in the alkali solution for 1 hr. Then the fibers were copiously washed with distilled water to remove any traces of alkali sticking to the fiber surface and subsequently neutralized with 2% sulphuric acid solution. The neutrality was checked with litmus paper. Then the fibers were dried in a hot air oven at 105°C. Bamboo samples were designated as B-U, B-10, B-15, and B-20, where B-U: Untreated sample; B-10: 10% alkali treated sample; B-15: 15% alkali treated sample; B-20: 20% alkali treated sample

TESTING

Thermo-Gravimetric Analysis

All the bamboo samples were analyzed using a Perkin Elmer thermal analyzer. All the measurements were made under a nitrogen flow (30ml per min), keeping a constant heating rate of 10^{0}C per min and using an alumina crucible with a pinhole.

Weathering Test

Water uptake and thickness swelling due to exposure to humid conditions

Bamboo samples with unsealed edge were dried to constant weight and then placed in a closed desiccator containing distilled water. The relative humidity of the air surrounding samples was 100%. The weight gain was measured by weighing them periodically on a Mettler Analytical balance. Thickness change was also measured by a Mitutoyo thickness gauge.

Initial weight of sample= w_1;
Weight of wet sample=w_2 ;
% Water uptake= (w_2 - w_1)/ w_1 X 100
Initial thickness of sample= t_1; thickness of wet sample=t_2
% Thickness swelling= (t_2 - t_1) t_1 X 100

Water Absorption Test

The water absorption test of the bamboo specimens to determine the water uptake and thickness swelling was carried out as per the ASTM D-570 method. Rectangular specimens of length 60mm and width 12.5mm were cut from the bamboo strips longitudinally along the direction of the fiber axis. The initial thicknesses and weights of

all the samples were recorded after conditioning each sample for 24h in a desiccator. The samples were immersed in water for 5 days (120hrs) at ambient temperature. The samples were then removed from the water and dried with a cotton cloth.

Flexural Test

Three-point bend tests were performed with the weathered bamboo samples of dimensions $60 \times 12.5 \times 3$ mm^3, according to ASTM D790. A cross-head speed of 1.2mm/min. was used. The sample span length was 4 cm, and the thickness of the sample was 3 mm.

Analysis of Morphology of the Fractured Surface of Weathered Samples by Scanning Electron Microscope (SEM)

A JEOL scanning electron microscope (JSM 5200) was used to study the fracture surface of the weathered bamboo samples that were subjected to flexural testing. Prior to the analysis the samples were sputtered with Au/Pd alloy and stuck on a stub by adhesive tape.

RESULTS AND DISCUSSION

TG Analysis

The TGA curves of untreated and alkali treated bamboo dust (15% and 20%) are shown in Fig. 1 and the corresponding thermal characteristics are given in Table 1.
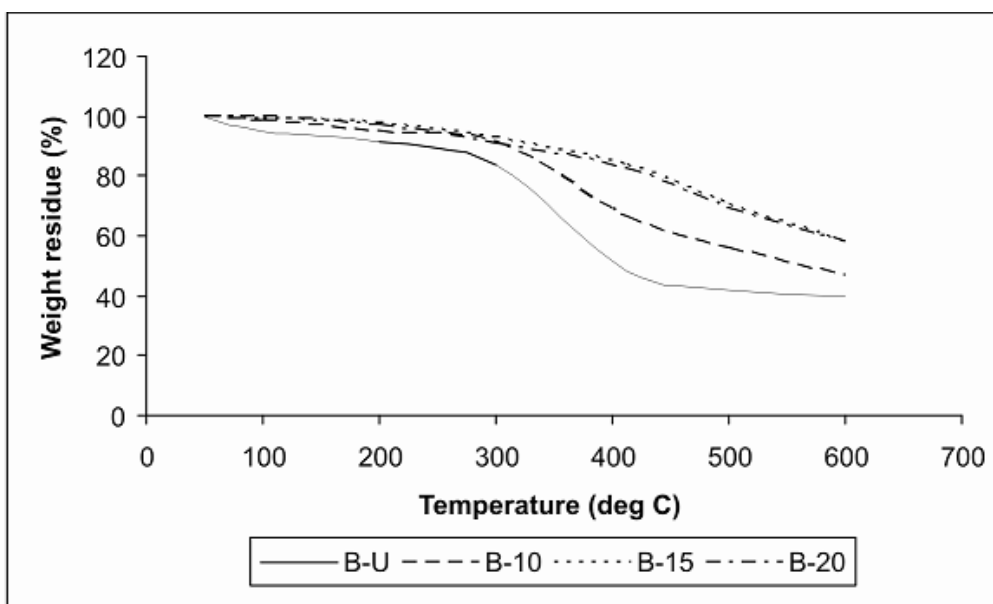

Fig. 1. TGA curve of untreated and alkali treated bamboo strips.

Table1. Results for TG Analysis of Untreated and Treated Bamboo Fiber

Sample Designation	No. of tran-sition	Transition Temperature (^{o}C)			Weight loss at corres-ponding transition (%)	Resid-ual weight (%) at 600°C	Activation Energy E_{act} J/^{o}K
		T_i	T_m	T_f			
B-U (Untreated bamboo fiber)	1	50	91.03	150	7.65	40	23.22
	2	150	342.89	426.11	52.35		
B-10(10% alkali treated fiber)	1	50	81.5	155.3	3.55	46.45	25.4
	2	155.3	241.7	287.5	7.5		
	3	287.5	349.6	178.3	42.5		
B-15(15% alkali treated fiber)	1	80	91.5	168.3	1.75	56.4	31.91
	2	168.3	223.31	276.64	4.6		
	3	276.64	359.9	409.99	17.25		
B-20(20% alkali treated fiber)	1	80	92	163.31	1.25	56.25	29.07
	2	163.31	224.64	250	3.5		
	3	250	346.77	389.96	20		

T_i : Initial temperature for transition; T_m : Maximum temperature for transition; T_f : Final temperature for transition.

The TGA curve of untreated bamboo showed two degradation steps in the temperature ranges of 50-150°C, and 150-426.11^{o}C, whereas the B-10, B-15, and B-20 samples showed three-step degradation following the temperature range as shown in Table 1. The untreated bamboo sample underwent 7.65% weight loss during the first step of degradation, which is attributed to moisture loss. But the weight loss percentages were 3.55, 1.75, and 1.25 for the B-10, B-15, and B-20 samples, respectively, for the first degradation step. The final temperature for this degradation was continuously shifted to a higher region from the B-10 to the B-20 sample. The amount of moisture absorbed continuously decreased with increasing treatment concentration of alkali. Again from TGA curves it is evident that the temperature at which moisture started to be liberated was higher for alkali-treated samples. This observation can be explained on the basis of changes occurring in the fine structure and morphology of bamboo fibers due to alkali treatment. From the earlier report on XRD analysis by Das et al. (2006a) it is clear that the crystallinity of alkali treated samples increases from B-10 to B-20, and in each case it is higher than untreated bamboo. Therefore, the tendency to liberate absorbed moisture upon heating will decrease, as moisture is strongly held within a tightly packed structure, leading to a higher finished temperature. The decreased amount of absorbed water might be the result of removal of increasing amounts of alkali-sensitive materials with higher alkali concentration, which are also active sites for moisture absorption.

Thermal degradation parameters of bamboo fiber reinforcement were studied by Rajulu et al. (2002), and it was concluded also that the thermal degradation of these fibers follows a two-stage process.

From Table 1 it is clear that there was no separate degradation for the components after moisture desorption in the case of the untreated bamboo samples, whereas it was evident for the treated samples. This is probably due to the artifact that bamboo itself behaves as a strong composite network, the structures of which are formed through inter-

and intra-molecular H-bonding in-between its component lignin, hemicellulose, and α-cellulose components. This behavior was also reflected during DSC analysis of untreated bamboo, by which a broad exotherm was observed by Das et al (2006b).

The final temperature for the second step of degradation decreased continuously from the B-10 to the B-20 sample. Basak et al. (1993) reported that the degradation temperature of hemicellulose of jute in the presence of nitrogen is 253°C. Therefore, the second degradation region probably corresponds to hemicellulose degradation present in the bamboo fiber, the final temperature of which has been shifted at a lower temperature from B-10 to B-20. Ray et al. (2002) also observed a shifting of the degradation peak of hemicellulose and α-cellulose in the case of jute fiber to lower temperature upon alkali treatment. The percentage degradation also decreased from the B-10 sample to the B-20 sample as the amount of hemicellulose leached out upon alkali treatment is higher with higher alkali treatment.

According to different authors (Basak et al. 1993; Varma et al. 1998) the temperature range for the last degradation peak temperature corresponds to α-cellulose degradation. The percent residual weight gradually increased from untreated to the alkali-treated sample, whereas B-15 and B-20 both were comparable. It can be then concluded that treated samples have higher thermal stability than the untreated, among which B-15 showed the maximum. Saha et al. (1991) reported a similar observation and explained that mercerization reduced the hemicellulose to a considerable amount, which leads to formation of lignin-cellulose complex, making the product more stable than the native one; hence the residual weight increased. Parker et al. (1969) reported a lowering of the degradation temperature and increase in char formation during the study of pyrolysis of treated cotton. They explained the results on the basis of an increased rate of formation of free radicals that are stabilized by condensed carbon ring formation in the char.
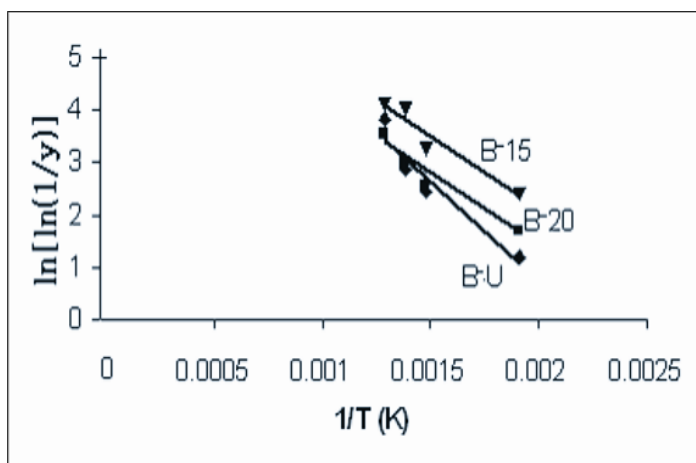

Fig. 2. Variation of ln[ln(1/y)] of bamboo strips with alkali concentration.

Analysis of the activation energy also can be helpful in reaching conclusions about the thermal stability of fibers. The larger the activation energy, the greater is the stability. The present calculation is based on the Broido equation (1969) and measured from the TGA thermograms of all the fiber samples considering the temperature range in between 250^{0}C- 400^{0}C. The equation is given below,

$$\ln[\ln(1/y)] = - E/R \{(1/T) + K\} \tag{1}$$

where R is the gas constant, T is the temperature in Kelvin, K a constant, y is the normalized weight (w_t/w_o), w_t denotes the weight of the samples at any time t, while w_o stands for its initial weights respectively. The energy of activation (E) can be obtained from a plot of $\ln[\ln(1/y)]$ vs $1/T$. The linear plots are shown in Fig. 2, and the value for activation energy is given in Table 1. The value of activation energy was at a maximum for the B-15 samples, for which thermal stability for the sample also was maximum.

Weathering Properties

It was found from Fig. 3 that the water uptake increased continuously from B-U to alkali-treated samples. On the contrary, thickness swelling decreased with increasing alkali concentration, and the flexural strength decreased as well (Figs. 4 through 6).
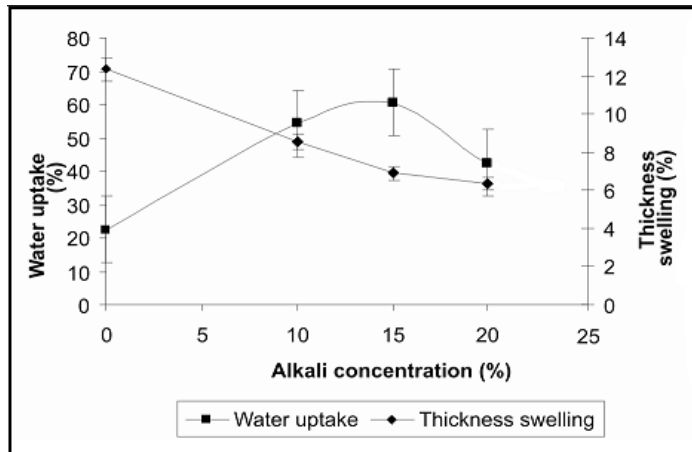

Fig. 3. Variation of water uptake and thickness swelling of bamboo strips with alkali concentration.
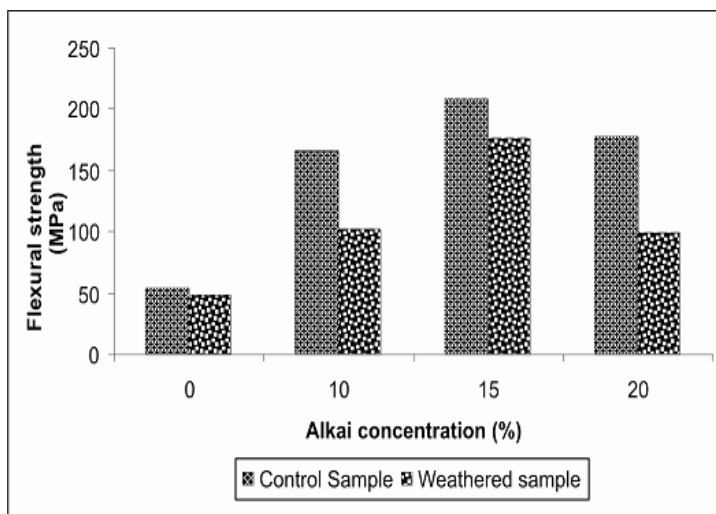

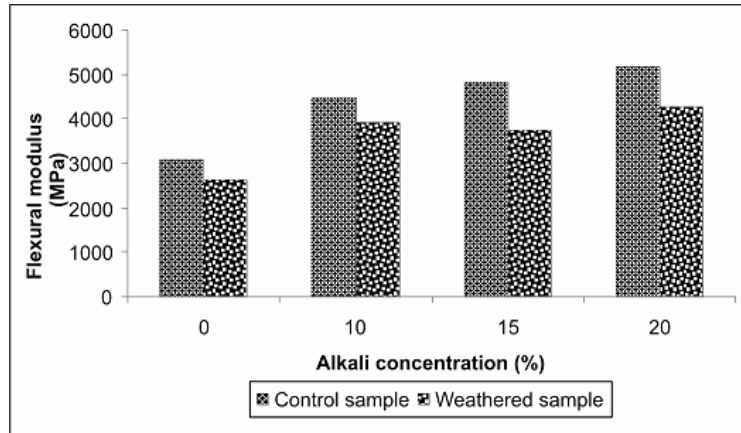**Fig. 4.** Variation of flexural strength of weathered bamboo strips with alkali concentration.

Fig. 5. Variation of flexural modulus of weathered bamboo strips with alkali concentration.
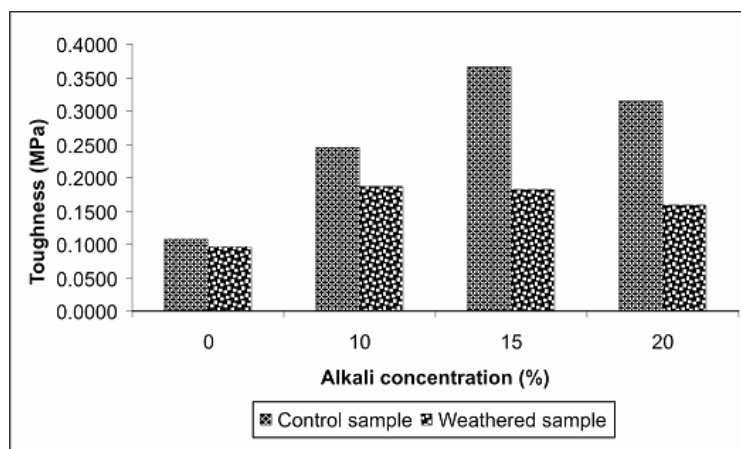

Fig. 6. Variation of toughness of weathered bamboo strips with alkali concentration.

Mukherjee et al. (1983) observed improved flexural strength and water uptake behaviour during the study of both natural and accelerated weathering performance of epoxy and polyester composites, using polyesteramidepolyol (PEAP)-treated jute fibers with nearly unidirectional as well as random orientation. These observations were explained on the basis of strong interfacial adhesion between fiber and matrix by formation of hydrogen bonding between the hydroxyl group of cellulose and PEAP.

It has been observed by Das et al. in their earlier work that there are more void spaces (created by removal of non-cellulosic material) in the bamboo structure after mercerization, and alkali-treated samples also have higher wetting ability (Das et al. 2008). Then the water molecules are now able to penetrate and spread into the cellulose network to a greater extent. They can enter into the capillaries, into the areas of the cellulose fibers where they are loosely bound with the matrix molecules, and into the void spaces. With increase in the extent of mercerization, the fibers are more fibrillated, so the water molecules get opportunity to move in between the fibrils, where sometimes they can attach themselves through chemical links to groups in the cellulose molecules. It has been found from different literature that water absorption of natural fibers depends upon the manner of retting and the way by which the cellulosic –OH groups can be modified (Stamboulis et al. 2000; Bismark et al. 2002). The water molecules force the

cellulose molecule apart, destroying some of the rigidity of the cellulose structure. This makes the cellulose molecules freer, more swollen, and more able to change shape easily with an application of force (Bismark et al. 2002). The absorbed water molecules can place themselves suitably in the void spaces created with the removal of non-cellulosic material upon alkali treatment, which leads to more deterioration with respect to mechanical properties. These facts lead to lower thickness swelling, flexural properties, and increased water uptake in the case of treated fibers. By contrast, if we consider the untreated bamboo as a uni-directional composite, the water molecules will get a lesser amount of space to move but can attach by chemical links with the matrix molecules. Then the cellulose chains will be forced more to accommodate properly the absorbed water molecule, and the thickness swelling is maximized. Stamboulis et al. (2002) reported during the study of water absorption of green and Duralin flax fiber, that the microscopic structure of cellulose shows a significant anisotropy, and the swelling is larger in a direction perpendicular to the chains than in a direction parallel to the fiber axis. Again, it was observed that at higher treatment concentration of alkali, percent thickness swelling attained a more or less constant value, whereas the percent water uptake value decreased. It was found that as the amorphous nature of the cellulose structure was increased with the inversion of cellulose-I to cellulose –II, the absorption of water decreased. These facts probably lead to reduction of amount of void spaces as well as the percent water uptake. This may be attributed to another fact that the hemicellulose component is largely responsible for moisture absorption, biodegradation, and thermal degradation of natural fiber (Bismark et al. 2002). Again, from the literature, it is known that alkali treatment leads to dissolution of hemicellulose. Hence as with higher alkali treatment, a higher amount of hemicelluloses has leached out; thus the amount of water absorption decreases with higher alkali treatment.
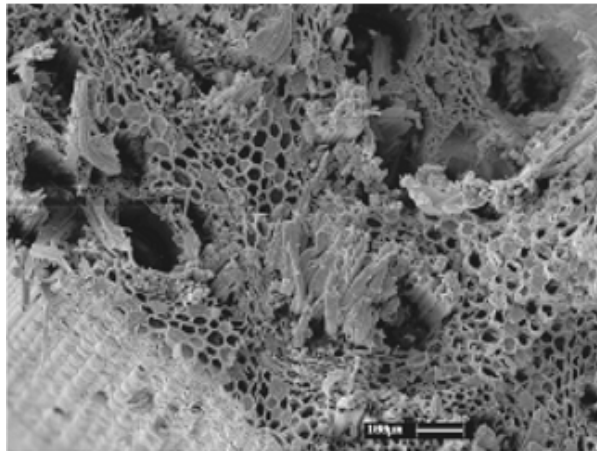

Fig. 7. Scanning electron micrograph of flexural fractured surface of weathered untreated bamboo strips.

From scanning electron micrographs (Figs. 7 and 8) of fractured surfaces of the weathered samples, it is revealed that in the case of the composite with mercerized fibers (15% alkali treated fiber), the fibers were more disentangled than the untreated fiber (Fig. 8).
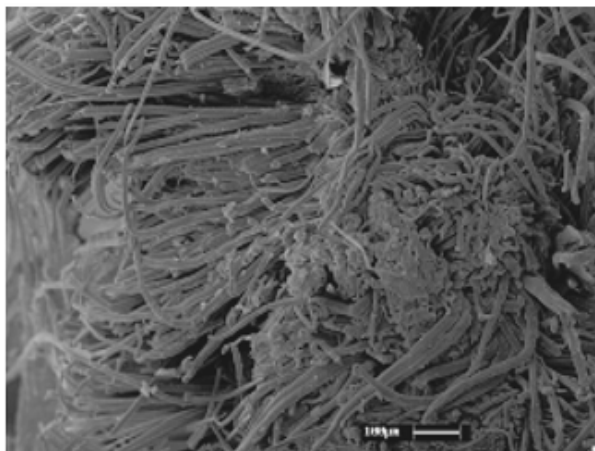
Fig. 8. Scanning electron micrograph of flexural fracture surface of weathered 15% bamboo strips.

The lumen of the fibers can be clearly seen in the micrograph of untreated bamboo sample, showing brittle fracture. This observation also indicates high interfacial tension between the fibrils and non-cellulosic binding material. Waxy material present in the surface may also restrict the water absorption in the case of untreated strips. As is known, alkali treatment removes noncellulosic materials and increases wetting ability of bamboo; hence, the treated fibers are more affected by weathering. It is evident from the micrograph of 15% alkali treated bamboo fiber that there was more pull-out of cellulose fibrils from the hemicellulose/lignin matrix of bamboo, indicating absorbance of more amount of energy during fracture as well as proving the ductile nature of fracture. The observed values for the weathering properties appear to be supported by the scanning electron micrograph.

To go beyond the scope of the present investigation, there is interest to evaluate degradation under sunlight or UV light. It is reasonable to expect that such factors play a role relative to the influence of mercerization on overall weathering.

CONCLUSIONS

1. Mercerization of bamboo fiber makes it thermally more stable over a certain temperature range during study. However, bamboo samples treated with 15% (B-15) and 20% alkali solution exhibit comparable percent weight residue at 600^0C. Activation energy was also highest for the B-15 sample, indicating higher thermal stability.

2. Water uptake increases continuously with increasing alkali concentration used for treatment. On the contrary, thickness swelling decreases with increasing alkali concentration. Decrease in flexural strength was observed in case of all samples, but the extent of deterioration was more for treated samples.

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