Alkali treatment of coir fibres for coir–polyester composites

S. V. PRASAD, C. PAVITHRAN, P. K. ROHATGI^{*} Regional Research Laboratory, (Council of Scientific & Industrial Research), Trivandrum 695 019, Kerala, India

Coir fibres were subjected to alkali treatment with a view to improving the wettability of coir fibres by a commercially available resin such as polyester. Tensile strength of the fibres increases by 15% when the fibres are soaked in 5% aqueous solution of NaOH at $28 \pm 1^{\circ}$ C for 72 to 76 h after which it shows a gradual decrease. This decrease is much more pronounced when the alkali is replenished after every 24 h. SEM observations showed the removal of cuticle and tyloses from the surface of coir as a result of alkali treatment, resulting in a rough fibre surface with regularly spaced pits. The debonding stress of alkali-treated fibres from polyester matrix was 90% higher than that of untreated fibres from the same matrix. Untreated fibres tended to float in the polyester whereas alkali-treated fibres were uniformly dispersed in polyester. Incorporation of 0.30 volume fraction of untreated and treated fibres in polyester resulted in composites having 11% and less 5% porosity, respectively. Flexural strength, modulus and impact strength of composites containing alkali-treated fibres were 40% higher than those containing the same volume fractions of untreated fibres. Longitudinal ultrasonic velocity and sound attenuation measurements indicated less fibre segregation and better fibre-matrix bonding in composites containing alkali-treated fibres.

1. Introduction

There have been several attempts to utilize the abundant and renewable resources of plant fibres in composite materials with a view to replace, at least partially, the use of expensive synthetic fibres and particulate fillers for a variety of consumer articles [1-3]. However, factors like the lack of wettability and interfacial bonding between natural fibres and well known commercial polymers like polyester have been the bottlenecks in these attempts. Experimental and theoretical studies over the past decade have shown that control of the fibre-matrix interfacial bond strength is a critical factor in obtaining the best mechanical properties of the resultant composite material [4].

The natural fibre under consideration here is coir which is extracted from coconut husk by a process called retting. During retting, the husk is soaked in water for 4 to 5 months to facilitate the removal of fibres from the pith which holds them together in the husk. In our previous study [5], we have reported the possibility of coating coir fibre with a thin layer (1.5 to $5.0 \mu m$ thick) of copper by electroless deposition to overcome the interfacial problem in coir/polymer composites. However, this method is relatively expensive and limits the use of composites made out of copper-coated coir to specialized applications like electromagnetic interference shielding where a definite electrical conductivity is an essential criterion. Therefore, any economically viable treatment to modify the surface of natural fibres so as to improve the wettability and bonding with commercial polymers will be highly desirable. Mercerization, essentially a simple alkali treatment, is a well known commercial process by which the lustre of textile

*Present address: Director, Regional Research Laboratory, Bhopal, MP India.

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fibres like cotton, and their reactivity towards dyes, are improved to a large extent. Mercerization also results in dimensional changes and an increase in the tensile strength of cotton fibres [6, 7]. In this investigation, we have subjected the coir fibres to an alkali treatment with a view to improve the wettability of these fibres with polyester resin. Fibre/matrix interfacial strength was studied from fibre pull-out tests. Since coir fibres are different from cotton fibres, both in dimensions and composition, we have studied the effect of alkali treatment on the microstructure, surface topography and tensile strength of coir fibre. Flexural, impact and ultrasonic properties of coir/polyester composites containing varying volume fractions of untreated and alkali-treated fibres are also reported.

2. Experimental details

2.1. Treatment

Retted coir fibres (0.15 to 0.30 mm diameter, 100 to 200 mm long) obtained from local sources in Trivandrum were used in the present study. These fibres were soaked in an aqueous solution of NaOH (5 wt%) for various periods of time. Approximately 30g of coir fibres was soaked in 700 ml of alkali solution. After each interval, the fibres were removed from the solution, washed

Figure 1 Macrophotographs of polyester blocks showing dispersability of coir fibres: (a) raw fibre, (b) 5S72 alkali-treated fibre and (c) raw fibre treated with lignin solution.

with fresh water several times and finally rinsed with distilled water containing a few drops of HCl to remove any excess NaOH sticking to the fibre surface. This series is designated by 5SX, 5S corresponding to soaking in 5% NaOH solution and X corresponding to the soaking time in hours.

In another series of experiments, the same procedure was followed except that the fibres were removed after every 24 h from the alkali bath and transferred to another bath containing freshly prepared solution of 5 wt % NaOH. This series is designated by 5SRX where R corresponds to replenishing the alkali solution after every 24 h.

2.2. Dispersability

A simple test was performed to characterize the dispersability of fibres in polyester resin. The fibres (in the as-received condition and 5S72 treated condition) were chopped to a length of 5 mm and mixed thoroughly in polyester resin (Bakalite Hylem India Limited, HSR 8111) to which were added 2% methylethylketone (MEK) peroxide and 2% cobalt naphthanate as initiator and accelerator, respectively. The resultant mix was allowed to set in a glass beaker.

Macrophotographs of polished sections of resin blocks containing untreated and 5S72 treated fibres were taken to demonstrate the role of the alkali treatment on the dispersability of fibres in polyester (Fig. 1).

2.3. Tensile and fibre pull-out testing

Tensile tests were carried out using an Instron testing machine at a cross-head speed of 20 mm



Figure 2 Schematic illustration of (a) the mould for preparation of pull-out specimens and (b) pull-out test.

 \min^{-1} . A gauge length of 50 mm was employed for the tensile testing. Ten fibres were tested after each treatment and the mean and standard deviations of the ultimate tensile stress and modulus were reported.

A mild steel mould of internal dimensions $15 \text{ mm} \times 50 \text{ mm} \times 10 \text{ mm}$ was used to prepare specimens for fibre pull-out testing. Fibres were introduced into the mould through the central holes as shown in Fig. 2a. The mould was placed on a glass plate and the sides were sealed with wax to prevent the leakage of resin. The mould was then filled with polyester resin mixed with initiator and accelerator. The resin block containing the fibres was taken out gently from the mould after the resin was set. The resin block was subjected to a post-curing treatment at 80° C for 8 h and cut into five pieces so that each piece contained a centrally embedded fibre. The embedded length of fibre in the resin was 10 mm.

A schematic illustration of the pull-out test is shown in Fig. 2b where one end of the fibre is gripped in the Instron. The resin block was placed on a jig fabricated with graphitic aluminium to reduce friction between the polyester disc and the jig (Fig. 2b). Fibre pull-out tests were carried out at a cross-head speed of 5 mm min^{-1} . Ten samples were tested after each treatment.

2.4. Mechanical and ultrasonic testing of composites

Coir fibres (treated and untreated) chopped to a length of approximately 10 mm were used for preparing composites. Weighed quantities of chopped fibres were placed in a die and a mat was prepared by pressing them against a wooden punch using a screw press. The polyester resin mixed with the accelerator and initiator was poured over the mat and squeezed using a flat piece of wood. A slight pressure was applied to keep the fibres in position until the resin was set. Specimens were subjected to a post-curing treatment at 80° C for 8 h. Volume fractions of coir were calculated from the weight fractions and densities of the fibres and polyester. Densities of the composites were determined from weight loss measurements in water. Porosities in composites were estimated from measured and calculated densities.

Specimens measuring $100 \text{ mm} \times 15 \text{ mm} \times 3 \text{ mm}$ were used for flexural testing and the tests were carried out using an Instron testing machine with a cross-head speed of 2 mmmin^{-1} . A span of 50 mm was employed conforming to the ASTM D 790 test procedure. Charpy-type impact tests on unnotched specimens of $50 \text{ mm} \times 6 \text{ mm} \times 4 \text{ mm}$ dimensions were performed using a pendulum impact testing machine. The pendulum load was 0.05 kg. Ultrasonic measurements were performed on cylindrical samples (35 mm diameter, 40 mm long) of composites at 1.25 MHz using a pulse echo apparatus with a piezoelectric transducer [8, 9].

2.5. Scanning electron microscopy

Cross sections and surface topographies of treated and untreated fibres were studied using a scanning electron microscope (SEM). Specimens for cross-



Figure 3 Typical stress-strain curves for raw fibre and 5S72 fibre.

sectional observations were prepared by mounting a bunch of fibres in polyester and polishing the resin block by conventional metallographic techniques. All specimens were sputtered with a 10 nm thick layer of gold prior to SEM observations.

3. Results

Typical stress-strain curves for untreated and 5S72 treated fibres under tensile load are shown

in Fig. 3. The modulus was calculated from the slope of the initial linear portion of the stressstrain curve as reported earlier [10]. The ultimate tensile strength of 5S and 5SR treated fibres is plotted as a function of treatment time in Figs. 4 and 5, respectively. The results were analysed by multiple regression analysis and the validity of regression was verified by "F" test. The superimposed curve shown in Figs. 4 and 5 were obtained using the following Equations 1 and 2, respectively.

$$\sigma_{\rm s} = 203 + 0.67t - 0.0038t^2 \tag{1}$$

$$\sigma_{\rm SR} = 207 + 0.846t - 0.00847t^2.$$
 (2)

These are significant at 5% level.

It can be seen from Fig. 4 that the tensile strength of coir fibres increased by about 15% when the fibres were soaked in alkali solution for 72 to 96 h. Prolonged soaking beyond 96 h resulted in a gradual decrease in the tensile strength of the fibres. This decrease is much more predominant in 5SR series wherein the alkali solution is replenished after every 24 h. Secondly, in the 5SR series, the fibres started losing strength after 48 h of treatment (Fig. 5). The Young's modulus of 5S treated fibres is plotted as a function of soaking time in Fig. 6. Statistical analysis was carried out as above and obtained the equation

$$Y = 4.062 + 0.03679t - 0.000194t^2.$$
(3)



Figure 4 Effect of soaking time on UTS of coir fibre. Fibres were soaked in 5% aqueous solution of NaOH (5S series).



Figure 5 Effect of soaking time on UTS of coir fibres. NaOH solution was replenished after every 24 h (5SR series).

This has significance level at 5%. The modulus increased by 40% when the fibres were soaked for 96 hours.

The effect of soaking time on the debonding stress is shown in Fig. 7 for 5S series. By the analysis of these data as in the previous cases the following equation was obtained



This is significant at 10% level.

The pull out stress increased from 83 to 145 $MN m^{-2}$ as the soaking time was increased from 0 to 72 h. Further increase in soaking time was found to result in a gradual decrease in pull-out stress. In the SR series wherein the NaOH concentration was replenished at 24 h intervals, the pull-out stress increased from 83 to 150 MN m⁻² (Fig. 8) as in the previous case. However, increase of soaking time beyond 96 h resulted in fibre failures in SR series (5SR 144) and no pull-out was recorded, apparently because the relative strength of the bond is now greater than the strength of the fibres. The breaking stress for 5SR144 fibres in Fig. 5 is in agreement with the UTS of these fibres (Fig. 8).

Scanning electron micrographs of a crosssection of coir fibre in the as-received condition are shown in Fig. 9 at different magnifications. Fig. 9a shows that the cross-section consists of cells embedded in a matrix. It may be worth recalling that in natural fibres like coir, the cells are crystalline cellulose arranged in a helical way in a matrix consisting of non-crystalline celluloselignin complex [6]. The central core, which is referred as "lacuna", can also be seen in Fig. 9a. The fibre surface can be seen to be covered by a thin and continuous layer. At a higher magnification (Fig. 9c) it can be seen that the thickness of the outer layer is 3 to $6\,\mu m$. Similar pictures of alkali-treated fibre (Fig. 10) show that the cells in the fibre soaked in NaOH for 72 h (5S72) are



Figure 6 Effect of soaking time on the modulus of coir fibre (5S series).

Figure 7 Effect of soaking time on the debonding stress (5S series).



more closely packed compared to those in the raw fibre (Figs. 9b and 10a).

After scanning the cross-sections of a number of fibres it was observed that the size of the central lacuna and lumen in alkali-treated fibres is much less than those of raw fibres indicating cell wall thickening and shrinkage of fibres during the alkali treatment. Diameters of twenty coir fibres (randomly selected) were measured before and after the alkali treatment using a microscope. Results indicate that a 5S96 treatment has resulted in an average 10% reduction in the diameter.

When the soaking time in NaOH was increased to 192h (5S192) the cells in the fibre were found to be ruptured (Fig. 10b). The fibre was covered with a thick ($10 \mu m$) layer (Fig. 10c).

An SEM picture of a typical coir fibre in the as-received condition shows the fibre surface to be very smooth (Fig. 11b). A series of globular particles of around $10\,\mu m$ diameter can be seen



Figure 8 Effect of soaking time on the debonding stress (5SR series).

20KU X200 1279 100.0U RRLSM

(a)

to be embedded in the fibre surface at regular intervals. These are identified as "tyloses" which cover the pits on the cell walls. On the surfaces of fibres soaked in NaOH for 24 h (5S24) most of the tyloses were intact but at a few isolated places it was removed creating holes (Fig. 12a). When the soaking time was increased to 72 h (5S72) the cells were exposed and a much greater proportion

Volume fraction of coir	Treatment given to coir	% Porosity	Flexural strength (MN m ⁻²)	SD*	Flexural modulus (GN m ⁻²)	SD	Charpy-type impact strength (10 ³ Nm m ⁻²)	SD
0 (Plain polyester)			48 5	4 7	3 077	0.30	8 33	0.86
(rialit polyester)			10.5		5.077	0.00	0.00	0.00
0.10	Untreated	3.71	33.5	8.78	2.792	0.75		
	5 S 72	0.65	34.0	4.33	3.026	0.52		_
	5 SR 144	0.50	34.5	2.85	3.004	0.16	_	
0.20	Untreated	9.17	33.0	3.90	2.497	0.38	7.44	2.38
	5S 72	1.60	42.3	4.60	3.380	0.24	11.33	1.92
	5 SR 144	0.89	42.0	5.60	3.390	0.22		_
0.30	Untreated	11.24	29.0	3.78	1.720	0.41	_	_
	5 S 72	4.50	41.5	3.98	3.334	0.17	_	_
	5 SR 144	2.43	40.5	3.60	3.380	0.16		

 $T\,A\,B\,L\,E\,$ I Mechanical properties of coir-polyester composites

*SD-Standard deviation.











4. Discussion

Before examining the results obtained in the present investigation, it may be worthwhile to give a brief account of the possible reactions between alkali and the different constituents of the fibre. Cellulose can absorb alkali from an aqueous solution and the absorbed alkali can be removed by soaking in water and subsequent drying resulting in a change in physical properties without altering the chemistry of the cellulose. Alkali can dissolve and leach out the fatty acids

Figure 10 Scanning electron micrographs of cross-section of alkali-treated coir fibres showing: (a) closure of lacuna(l) and cell wall (cw) thickening in 5S72, (b) and (c) rupture of cells and deposition (d) of dissolved materials, respectively, in 5S192.

and their condensation products which form the waxy cuticle layer of the fibre. Excessive alkali treatment under more severe conditions can also damage the fibrils by removing the lignin and hemicellulose which bind the cells in the fibre.

4.1. Effect of the treatment on UTS

From the results obtained so far, it appears that two opposing phenomena are simultaneously taking place during the alkali treatment. First, closure of lacunae as observed in SEM picture (Fig. 10a) and the reduction in the diameter of the fibres should contribute to an increase in the strength. Cell wall thickening (Fig. 10a) might also contribute to a further increase in the strength (similar observations on cell wall thickening were made on mercerized cellulose textile fibres [6]). Second, rupture of the cells (Fig. 10b) and complete exposure of the fibrils at the surface (Fig.

Volume fraction of coir	Treatment given to coir	Longitudinal velocity, V_1 (km sec ⁻¹)	Attenuation, α (Np cm ⁻¹)*
0 (Plain polyester)		2.50	0.41
0.1	Untreated 5872	2.37 2.45	1.30 0.94
0.2	Untreated 5872	2.25 2.38	1.45 1.12
0.3	Untreated 5S72	2.29	1.55

TABLE II Ultrasonic properties of coir-polyester composites

 $N_p = neper. 1 neper = 8.686 dB.$



Figure 11 Surface topographies of coir fibre: (a) unretted and (b) retted. Pith (P), tylose (T) and cuticle (C).

13a), presumably due to leaching out of the cuticle and the matrix material of the fibre, should lead to a decrease in the strength. During the initial stages, the first factor may be more predominant than the latter, resulting in an increase in strength with soaking time. However, at prolonged soaking times, the second factor is most likely to be over-riding the first factor, resulting on a gradual decrease in strength after the peak is attained (Fig. 4). It is worth noting that in the



5SR series, where the NaOH is replenished every 24h, the decrease in UTS after the maximum value is more predominant than in the S series. This is understandable because the fibre is regularly exposed to fresh NaOH solution and the tendency for fibril exposure (Fig. 13a) due to matrix leaching is much more than when NaOH is not replenished.

4.2. Fibre pull-out strength

The debonding stress should, in principle, depend on the surface topography of the fibre [11] and the chemical compatibility between the fibre surface and the resin matrix [4]. It has been shown that coir fibres in the as-received condition contain a continuous 3 to $5 \mu m$ thick cuticle layer which was presumed to be a waxy material consisting of fatty acids and their condensation products. Fatty acids are generally long chain

Figure 12 Surface topographies of alkali-treated fibres (5S series) showing: (a) partial removal of cuticle (c) in 5S24, (b) removal of cuticle and tylose (T) in 5S72 and (c) smoothing of fibre surface in 5S192 due to deposition of dissolved materials.







Figure 13 Surface topographies of alkali-treated fibres (SSR series). (a) 5SR72 treated fibre and (b) 5SR144 treated fibre showing leaching of intercellular binding materials.

aliphatic compounds which are not expected to be compatible with polyester. Thus, removal of this cuticle layer as shown in Figs. 12 and 13 will expose cellulose fibrils and lignin matrix to the polyester resin. Lignin, being a phenolic natural polymer, should be chemically compatible with polyester resin. Second, during the treatment, the "tyloses" were removed. Removal of the "tylose" and cuticle layer from the surface of the fibre during alkali treatment had led to a very rough fibre surface with regularly spaced pits of about 10 µm diameter (Figs. 12 and 13). In the pull-out tests, the pits could conveniently increase the mechanical interlocking between the fibre and resin and the removal of waxy cuticle layer increase the wettability of the fibre surface by the resin resulting in the observed increases in debonding/pull-out stress with soaking time. However, prolonged soaking beyond 72h in the 5S series resulted in a gradual decrease in fibre pull-out stress (Fig. 7). SEM pictures of fibres soaked for 192h (5S192) showed smoothing of the fibre surface (Fig. 12c) and deposition of a $10\,\mu m$ thick layer (Fig. 10c). The decrease in fibre pull-out stress could possibly be due to smoothing of the fibre surface. On the contrary, continued soaking of fibres in the 5SR series wherein the alkali concentration is replenished after every 24 h did not result in any decrease of fibre pullout stress (Fig. 8). Fibre pull-out data on these fibres (5SR 144) showed fibre breaking as opposed to fibre pull-out. SEM pictures of these fibres (5SR 144) showed complete exposure of fibrils and no smoothing of the fibre surface (Fig. 13). In fact, the concentration of dissolved materials may not be reaching a level favouring deposition

on the fibres in the "SR" series due to the periodical replacement of the alkali solution. Further, the drop in alkali concentration is much lower in the "SR" series than that in the "S" series, to 4.4 and 3.1%, respectively, after 72 h, which will reduce the rate of deposition of dissolved materials on the fibres in the "SR" series.

4.3. Dispersability

When the densities of the fibre and resin are similar, as in the present case (density of polyester resin = 1.12, density of coir in the as-received condition = 1.15), dispersability of fibres in the resin depends to a large extent on the wettability of the fibre surface by the resin. Lack of wetting leads to air being trapped at the interface resulting in the segregation of fibres at the top. Results of the dispersability tests showed that the fibres in the as-received condition when mixed thoroughly with polyester resin segregated at the top within two minutes (Fig. 1a) whereas 5S72 treated fibres remained in position as good dispersions until the resin was set (Fig. 1b). The dispersibility of 5S72 treated fibres could also be due to increased density (1.3). However, fibres dipped in lignin solution having similar density to raw fibres were also found to be well dispersed (Fig. 1c). These observations suggest that proper surface modifications of coir fibres could lead to better dispersion in the resin matrix. In practice, short fibres like coir are normally used in chopped strand mat forms to build FRP laminates by hand lay-up method, and prevention of the segregation of fibres at the top will solve a number of practical difficulties.

4.4. Mechanical properties of composites

Lack of proper wetting between the filler and matrix should, in principle, lead to the formation of voids at the filler—matrix interface. Incorporation of such a filler beyond a certain critical volume fraction in a matrix will be difficult owing to the rejection of the filler from the matrix. Any attempt to introduce high volume fractions of the filler by applying pressure would result in a highly porous structure and the strength of such a composite will be much lower than that of the matrix when a low modulus filler like coir is employed.

Flexural strength data on composites (Table I) shows that the introduction of 0.30 volume fraction of untreated coir fibre resulted in composites with 11% porosity and the flexural strength of composites was 40% less than that of polyester itself. On the contrary, composites containing 0.30 volume fraction of alkali-treated fibres (5S72 and 5SR144) had less than 5% porosity. At 0.30 volume fraction coir, flexural strength of composites containing alkali-treated fibres was only 15% less than that of the pure polymer without fibres. The flexural modulus of polyester was increased by 10% from 3.077 to $3.39 \,\mathrm{GN}\,\mathrm{m}^{-2}$ with the incorporation of 0.30 volume fraction of alkali-treated fibres (Table I). In contrast, incorporation of 0.30 volume fraction of untreated fibres in polyester resulted in a drastic decrease (40%) in flexural modulus of polyester from 3.077 to $1.72 \,\mathrm{GN\,m^{-2}}$. Similarly, Charpy-type impact strength of composites containing 0.20 volume fraction of alkali-treated coir was 50% higher than their counterparts containing untreated fibres and 35% higher than of polyester itself. This enhancement in mechanical properties of composites containing alkalitreated fibres over those of containing untreated fibres could be attributed to the improved bonding of treated fibres with polyester.

4.5. Ultrasonic properties of composites

The longitudinal ultrasonic velocity (V_1) decreased with increase in fibre content for both untreated and alkali-treated fibres (Table II). However, this decrease is much more predominant in composites containing untreated fibres than those containing alkali-treated fibres. In coir—polyester composites where the elastic modulus of coir are somewhat higher than those of the polyester matrix [3, 8], the effective modulus theories indicate an increase in V_1 with an increase in the fibre content. These theories, however, assume ideal conditions, uniform distribution of randomly oriented fibres in the matrix, absence of voids and a perfect interfacial bond between the fibres and the matrix. Interpreted against this background, the observed V_1 variations indicate, among other things, that the fibre segregation is less and fibre-matrix bonding is better in composites containing alkalitreated fibres than in those containing raw fibres. Ultrasonic sound attenuation (α) values increased with fibre content for both untreated and alkalitreated fibres (Table II). At the same volume fraction, α values were higher for composites containing untreated fibres than those containing alkali-treated fibres. This could possibly be due to higher porosity in composites containing untreated fibres. These data also emphasize the sound absorptive potential of these inexpensive composites.

5. Conclusions

1. Treatment of coir fibre with 5% aqueous solution of NaOH at room temperature $(28 \pm 1^{\circ} \text{ C})$ results in modifications of surface of coir fibres leading to increased wettability of these fibres with polyester resin. Alkali treatment also prevents the flotation and segregation of coir fibres in polyester-based composites.

2. Alkali treatment of coir fibre for 72 to 96 h results in a 10 to 15% increase in tensile strength, 40% increase in modulus and 90% increase in debonding strength from polyester.

3. Alkali treatment facilitates the incorporation of 0.30 volume fraction of coir in polyester with porosity below 5%. Incorporation of similar volume fractions of untreated coir in polyester results in 11% porosity.

4. Flexural and Charpy-type impact strength of composites containing alkali-treated fibres are about 40% higher than those of composites containing the same volume fractions of untreated fibres. Charpy-type impact strength of composites containing 0.20 volume fraction of alkali-treated fibres was 35% higher than that of polyester. The flexural strength of polyester was reduced by 15% with the incorporation of 0.30 volume fraction of alkali-treated fibres, however, an increase of 5 to 10% in the flexural modulus was observed.

5. Ultrasonic measurements indicate that incorporation of coir fibres leads to considerable

increase in the attenuation of polyester. Sound attenuation values were slightly higher for composites containing untreated fibres compared to alkali-treated fibres.

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