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# Effect of alkali treated jute fibres on composite properties

**DIPA RAY, B K SARKAR*, A K RANA[†] and N R BOSE[‡]**

Department of Materials Science, Indian Association for the Cultivation of Science, Kolkata 700 032, India
[†]Indian Jute Industries Research Association, Kolkata 700 088, India
[‡]Central Glass and Ceramic Research Institute, Kolkata 700 032, India

**Abstract. Jute fibres were subjected to a 5% alkali (NaOH) solution treatment for 0, 2, 4, 6 and 8 h at 30°C. An improvement in the crystallinity in the jute fibres increased its modulus by 12%, 68% and 79% after 4, 6 and 8 h of treatment respectively. The tenacity of the fibres improved by 46% after 6 and 8 h treatment and the % breaking strain was reduced by 23% after 8 h treatment. For the 35% composites with 4 h treated fibres, the flexural strength improved from 199·1 MPa to 238·9 MPa by 20%, modulus improved from 11·89 GPa to 14·69 GPa by 23% and laminar shear strength increased from 0·238 MPa to 0·2834 MPa by 19%. On plotting the different values of slopes obtained from the rates of improvement of the flexural strength and modulus, against the NaOH treatment time, two different failure modes were apparent before and after 4 h of treatment. In the first region between 0 and 4 h, fibre pull out was predominant whereas in the second region between 6 and 8 h, transverse fracture occurred with a minimum fibre pull out. This observation was well supported by the SEM investigations of the fracture surfaces.**

**Keywords. Vinylester resin; jute fibre composite; alkali; mechanical properties; fracture; fibre pull out.**

## 1. Introduction

In recent years, the natural fibre reinforced composites have attracted substantial importance as a potential structural material. The attractive features of the natural fibres like jute (Roe and Ansell 1985; Shah and Lakkad 1981), sisal (Bisanda and Ansell 1991), coir (Prasad *et al* 1983; Rout *et al* 1999) and banana (Pothan *et al* 1997) have been their low cost, light weight, high specific modulus, renewability and biodegradability. Naturally, composites reinforced with such natural fibres have thus been a subject of intense study for low strength, low cost application in contrast to the synthetic fibre reinforced composites. Since the interfacial bond between the reinforcing fibres and the resin matrix is an important element to realize the mechanical properties of the composites, studies have been focussed on the treatment of fibres to improve the bonding with resin matrix by several authors (Samal *et al* 1995; Gassan and Bledzki 1999a, b). Amongst the several natural fibres, jute constitutes a major area of investigation.

For the composite applications having good bonding between the fibre and the resin matrix, jute have been treated with alkali, a process known as mercerization, being commercialized for cotton fibres for superior reactivity with dyes (Ott *et al* 1954). Several authors have employed the technique on jute and the changes occurring in the fibre properties were investigated (Sarkar 1935; Mukherjee *et al* 1993). Sarkar (1935) and Samal *et al* (1995) have treated jute fibres with NaOH solution of concentration 1%, 8% for 48 h and 2% for 1 h and showed improvements in fibre properties by 130% and 13% respectively. Similar treatments were attempted by Gassan and Bledzki (1999a, b) on isometric jute yarns. They reported an improvement of 120% and 150% in the tensile strength and modulus of jute yarns respectively treated with 25% NaOH solution for 20 min and 60% improvement in the jute/epoxy composite properties reinforced with these treated yarns. The improvements have been attributed to the greater reactivity of the treated fibres with the resin administering superior bonding.

In this study, the effects of 5% NaOH treatment for short duration of up to 8 h on jute as reinforcing fibre material to vinylester resin have been investigated. Mechanical properties of the composites with varying weight % of fibres were determined. The change in properties were analysed with the help of microstructural analysis of the fractured surfaces.

## 2. Experimental

### 2.1 *Materials*

Jute fibres (white jute, *Corchorus capsularis*) were collected from Indian Jute Industries Research Association (IJIRA), Kolkata. Vinylester resin of grade HPR 8711, a Bakelite Hylam product was used. Methyl ethyl ketone peroxide (MEKP), Co napthenate and N,N-dimethylaniline were used as catalyst, accelerator and promoter respectively.

*Author for correspondence

### 2.2 *Alkali treatment*

The jute fibres were cut to 50 cm of length and were soaked in a 5% NaOH solution at 30°C maintaining a liquor ratio of 15 : 1. The fibres were kept immersed in the alkali solution for 2, 4, 6 and 8 h. The fibres were then washed several times with fresh water to remove any NaOH sticking to the fibre surface, neutralized with dilute acetic acid and finally washed again with distilled water. Final pH maintained was 7. The fibres were then dried at room temperature for 48 h followed by oven drying at 100°C for 6 h.

### 2.3 *Dispersability test*

A simple test was performed to observe the dispersability of the untreated and the alkali treated fibres in vinylester resin. Alkali treated jute fibres were found to have dispersed more uniformly in the resin than the untreated jute fibres.

### 2.4 *Composite processing*

The jute/vinylester composites containing the raw and alkali treated jute fibres were fabricated in the form of cylindrical rods of diameter 6 mm. Hollow cylindrical glass tubes of internal diameter of 6 mm were taken to act as a mould. The resin was mixed with accelerator, promoter and catalyst (1% each). The jute fibres were dried prior to use, soaked in the mixed resin and the wetted fibres were pultruded through the glass tube by hand.

The pultruded samples within the glass tube were cured at room temperature for 24 h followed by a post curing in an oven at 80°C for 4 h. The glass tubes were broken clean to release the composite rod. The composites with five different wt% (8, 15, 23, 30 and 35) of untreated and treated jute fibres were prepared for further investigation. In this case, both the wt% and vol% are similar as the density of the resin and the fibre are both equal to 1·3 g/cm$^3$.

### 2.5 *Test methods*

The fibres after different soaking times in 5% alkali solution were tested for their weight change. The loss in weight was calculated by weighing a fixed amount of dry and cleaned fibre ($W_1$). The amount of fibre was weighed again after soaking in alkali solution ($W_2$). The % weight loss was calculated as

$$\% \text{ Weight loss} = (W_1 - W_2)/W_1 \times 100.$$

It was observed that the fibres were somewhat leached and were finer. Fibre fineness was determined in terms of linear density by gravimetric method. The linear density was obtained from the weight of 100 single fibres of 60 mm length each.

Chemical analysis of untreated and treated fibres were made to estimate the cause for the loss in weight. The major constituents ($\alpha$-cellulose, hemicellulose and lignin) of the untreated and the alkali treated jute fibre samples were determined following the standard procedures (Chattopadhyay and Sarkar 1946).

Tensile tests of single jute fibres were carried out using an Instron 1195. A gauge length of 20 mm was employed with a cross head speed of 10 mm/min in accordance with ASTM D 3822-91. Fifty single fibres were tested after each treatment and the mean value was reported.

*Flexural Test*: Three-point bend tests were performed in an Instron 4303 machine in accordance with ASTM D790M-81 to measure the flexural strength of the composites. Test specimens were 120 mm long cylindrical rods having a diameter of 6 mm. A span of 100 mm was employed maintaining a cross head speed of 2 mm/min.

The flexural strength and modulus were measured using the following equations

$$\text{Flexural strength} = 8 \cdot F \cdot L/\pi \cdot d^3,$$

$$\text{Flexural modulus} = 4 \cdot m \cdot L^3/3 \cdot \pi \cdot d^4,$$

where $F$ is the load, $L$ the span, $d$ the dia. of specimen and $m$ the slope of the initial straight line portion of the load-displacement curve.

The breaking energy was calculated from the area under the load/displacement curve to the break point and the toughness was obtained by dividing the energy to break by the volume of the test specimen.

The flexural interlaminar shear strength (LSS) of the composites which is the maximum shear stress that a material can withstand before it ruptures, was calculated in the stress units based on the area of the sheared edge.

## 3. Results and discussion

Loss in weight was observed after the alkali treatment of the fibres, shown in figure 1, due to heavy dissolution of the hemicellulose content. The loss occurred primarily within 2 h of treatment. This had resulted in the drop of linear density of the fibres when treated between 2 and 6 h due to the creation of voids in the fibre structure, the strands became well separated and dispersed. The crystallinity of the fibres was observed to have increased only after 6 h treatment. By 8 h treatment, the $I_{002}$ peak height improved by 23·4% from the untreated fibre. The improvements in the properties of the untreated jute fibres and on alkali treatment is depicted in figure 2. The modulus of the jute fibres increased by 12% at 4 h of treatment, after which the increase was by 68% and 79% when treated for 6 and 8 h respectively. The tenacity at break point increased by nearly 46% after 6 and 8 h treatment, % breaking strain was reduced by 23% after

8 h of treatment (figure 2). It was imperative that the fibres became stiff and brittle on account of its high strength and low extensibility. Similar increase in strength of jute fibres on alkali treatment was reported by Sarkar (1935).

### 3.1 *Mechanical properties of untreated jute fibre reinforced vinylester composites*

The properties of jute/vinylester composites are given in table 1. For each data point, minimum five samples were tested and the value given is the mean of those results. The load/displacement curves for 23 wt% and 35 wt% composites with both untreated and 4 h alkali treated fibres are shown in figure 3. Heavy fibre pull out and breakage was observed for 23 wt% and 35 wt% fibre composites reinforced with untreated fibres. Shear fracture was predominant for alkali treated fibre composites, imperative of a better bonding at the interface between the fibre and the matrix.

In keeping with the general rule of mixtures, the improvements apparently have occurred linearly in modulus from 2·915 to 11·89 GPa, flexural strength from 120·70 to 199·1 MPa, for 8 to 35 wt% of fibres in the resin matrix. The initial lowering of the flexural strength with 8 wt% of fibre reinforcement than the properties obtained for the resin alone was in accordance with the rule of mixtures, i.e. when the volume fraction of reinforcing fibres is lower than the critical quantity, the fibres acted as flaws in the matrix (Milweski and Katz 1987). Such a drop in properties was observed by others also.

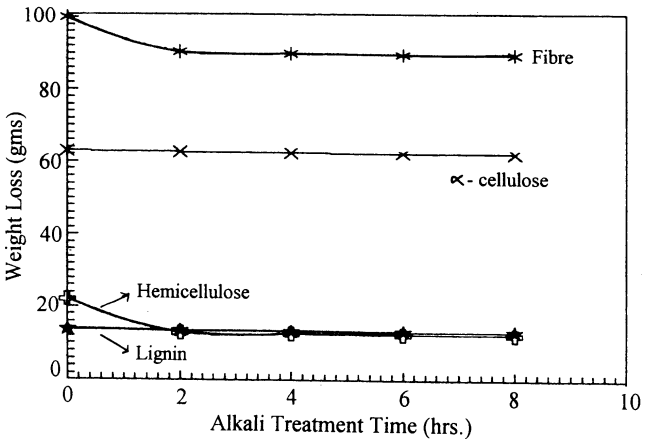


**Figure 1.** Loss of weight of the constituents of jute fibres after alkali treatment.

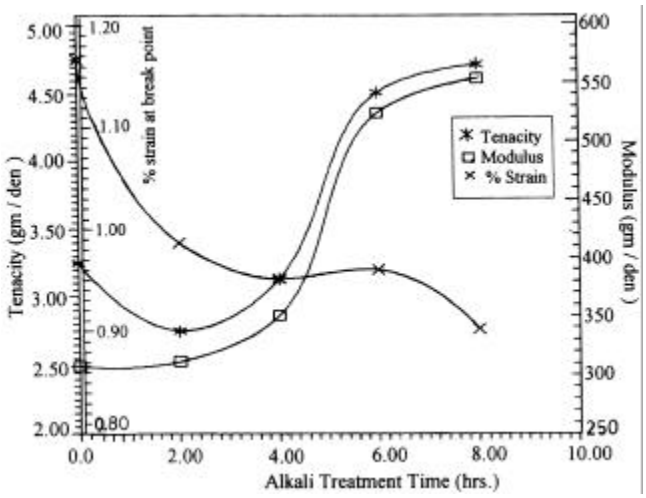


**Figure 2.** Tenacity, modulus and % breaking strain of jute fibres after alkali treatment with varying times.

### 3.2 *Mechanical properties of alkali treated jute fibre reinforced vinylester composites*

The mechanical properties of composites prepared with varying proportions of fibres (0 to 35 wt%) treated with alkali (5% NaOH solution) are included in table 1. The composites prepared with fibres treated for 4 h showed maximum improvements at all wt% fibre loadings. The improvement became more predominant with increasing fibre loading. The flexural strength properties of the com-

**Table 1.** Mechanical properties of untreated and alkali treated jute/vinylester composites.

| Jute (wt%) | Type of fibre | Modulus (GPa) | Flexural strength (MPa) | Breaking energy (J) |
|---|---|---|---|---|
| 0 | – | 2·915 | 120·70 | 0·8227 |
| 8 | untreated | 4·220 | 106·30 | 0·2948 |
| | treated 2 h | 3·446 | 96·27 | 0·2497 |
| | treated 4 h | 4·205 | 121·20 | 0·3634 |
| | treated 6 h | 3·967 | 101·80 | 0·2270 |
| | treated 8 h | 3·130 | 93·97 | 0·2488 |
| 15 | untreated | 5·544 | 128·60 | 0·3399 |
| | treated 2 h | 6·024 | 134·70 | 0·3530 |
| | treated 4 h | 6·539 | 146·50 | 0·4016 |
| | treated 6 h | 5·546 | 121·50 | 0·2569 |
| | treated 8 h | 5·337 | 127·60 | 0·3351 |
| 23 | untreated | 7·355 | 145·70 | 0·3531 |
| | treated 2 h | 8·065 | 157·70 | 0·4048 |
| | treated 4 h | 9·384 | 172·70 | 0·4198 |
| | treated 6 h | 8·542 | 155·40 | 0·3553 |
| | treated 8 h | 7·132 | 145·80 | 0·3762 |
| 30 | untreated | 10·030 | 180·60 | 0·4799 |
| | treated 2 h | 10·990 | 189·40 | 0·4816 |
| | treated 4 h | 12·850 | 218·50 | 0·5061 |
| | treated 6 h | 12·490 | 195·90 | 0·4319 |
| | treated 8 h | 11·170 | 197·50 | 0·5042 |
| 35 | untreated | 11·890 | 199·10 | 0·5543 |
| | treated 2 h | 12·700 | 205·20 | 0·4570 |
| | treated 4 h | 14·690 | 238·90 | 0·5695 |
| | treated 6 h | 14·890 | 232·00 | 0·5678 |
| | treated 8 h | 12·320 | 204·20 | 0·5099 |

posites at 35 wt% fibre loading after 4 h alkali treatment was 238·9 MPa in contrast to 199·1 MPa for the composites with untreated fibres. An improvement of 20% was measured. The improvements however, were 3% and 2·5% only for composites prepared with 2 and 8 h treated fibres respectively. The trend was similar for the lower loadings with the treated fibres. The improvement had occurred after 23 wt% fibre loading. Similar observations for the alkali treated coir reinforced polyester composites at 19 wt% loading was reported by Rout *et al* (1999). The trend was similar for the flexural modulus also. The maximum improvements were with 4 h treated fibre composites at 35 wt% reinforcements. For 4 h treated fibre composites at 35 wt% loading showed strengths in the range of $28{\cdot}34 \times 10^{-2}$ MPa. For the composites prepared with 8 wt%, 15 wt% and 23 wt% 4 h treated fibres, the breaking energy values increased by 23%, 19% and 18% respectively, whereas for the composites with 30 wt% and 35 wt% fibres, the increase were only by 5% and 3% respectively. Although 8 h treated fibres exhibited maximum strength properties, but the composites prepared with them showed lower strength values. Maximum composite strength was obtained from the 4 h treated fibres. The fibres became rigid and somewhat brittle afterwards owing to the development of crystallinity causing high strength and low extensibility. On application of stress, these fibres suffered breakage due to increased brittleness and could not take part in effective stress transfer at the interface, thus lowering the strength of the composites.

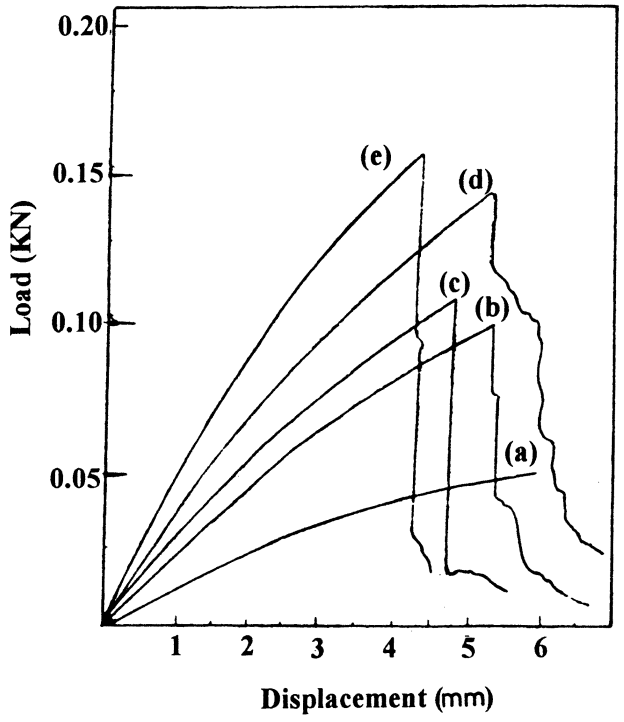


**Figure 3.** Load-displacement curves of jute reinforced composites: (**a**) vinylester resin; 23 wt% fibres, (**b**) untreated, (**c**) 4 h NaOH treated; 35 wt% fibres, (**d**) untreated and (**e**) 4 h NaOH treated.

The rates of improvements ($R$) in the flexural strengths and modulus of the composites with wt% of NaOH treated fibre reinforcements from 2 h to 8 h have been shown in figures 4 and 5.

The improvement occurred linearly with the increased wt% fibre content. The slope of the composite properties with 0–4 h treated fibres was maximum and became shallower with 4–8 h treated fibres. This can be expressed by the following relation

$$R = m \cdot W_f + C,$$

where $R$ is the rate of change of the composite properties, $m$ the slope and $W_f$ the weight of the fibre reinforcements. Taking the different slopes of '$m$' from figures 4 and 5, a plot of '$m$' against alkali treatment time was depicted in figure 6.

A deflection at 4 h treatment was apparent imperative of the occurrence of two different failure modes before and after 4 h of NaOH treatment. Taking the strength values of fibres from Bledzki and Gassan (1999) the com-

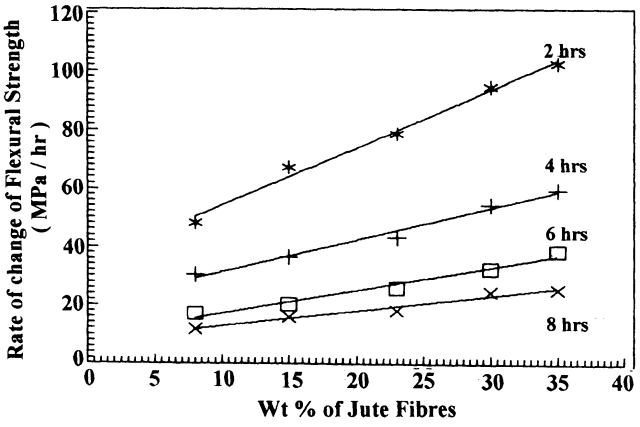


**Figure 4.** Variation of the rate of change of flexural strength of composites with varying wt% of alkali treated jute fibres.

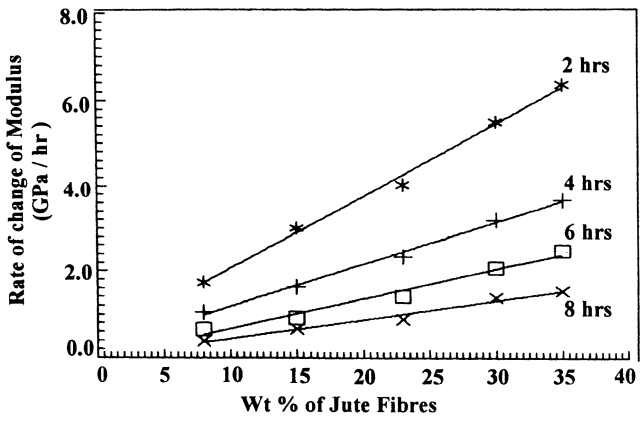


**Figure 5.** Variation of the rate of change of modulus of composites with varying wt% of alkali treated jute fibres.

posite strengths were estimated applying the rule of mixtures. This was compared with the experimentally obtained flexural strengths of the composites prepared with 4 h treated and untreated fibres. The variation of the property with wt% jute fibre reinforcement is shown in figure 7. A large difference between the estimated and actually measured values was observed. The improvements with alkali treated fibres for 4 h was apparent. The improvements in the experimental values were not linear, having the improvements more towards higher fibre loading. At 23 wt% fibre loadings, the composite flexural strengths were lower by 24% and 36% and for 35 wt% fibre loadings, they were lower by 16% and 29% from the estimated values for treated and untreated fibre composites respectively. Similar observations were made for the change in modulus also (figure 8).

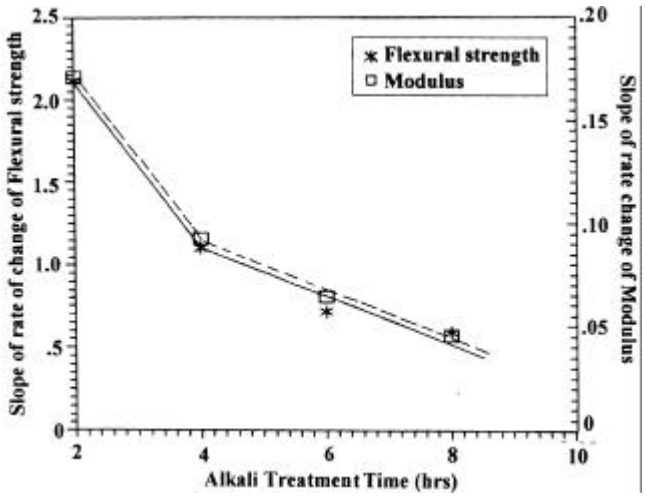


**Figure 6.** Effect of the alkali treatment time on the rate of change of flexural strength and modulus of the composites.

The composite modulus were lower than the estimated modulus by 37% and 51% for 35 wt% composites and by 31% and 44% for 23 wt% composites for the treated and untreated fibres respectively. The modulus value of the fibres for the estimation of modulus from the rule of mixtures was taken from Roe and Ansell (1985). The theoretical estimation of strength and modulus from the rule of mixtures considers defect free fibres and composites. The observed percentage differences of the composite properties between the estimated and measured values, would actually be the total amount of % defects in the composites incorporated during processing. Taking the relation

$$s_c = V_f (n_1 e^{-kd_1} - n_2 e^{-kd_2}) + n_2 e^{-kd_2},$$

modelled by Sarkar (1998) for the estimation of composite strength by modified rule of mixtures incorporating defects, where $s_c$ is the composite strength, $n_1$ and $n_2$ are fibre and matrix strengths at defect concentrations of $d_1$ and $d_2$, respectively, $k$ is a constant approximating 1 and $V_f$ the vol% of fibres, $d_1$ and $d_2$ were estimated. It was found that the total amount of defects in a 35 wt% composite was 30% against 29% found experimentally for composites prepared with the untreated fibres and 18·5% against 16% found experimentally for the composites having 4 h alkali treated fibres. The $d_1$ for untreated and 4 h treated fibres were 20% and 8·5% respectively, whereas $d_2$ was 10%. The defects in the composites with untreated fibres relates to the poor wetting characteristics with an improvement on alkali treatment.

The improved properties of the fibres with alkali treatment for a longer duration was the result of dissolution of hemicellulose and development of crystallinity and fibrillation thus created superior bonding with vinylester resin

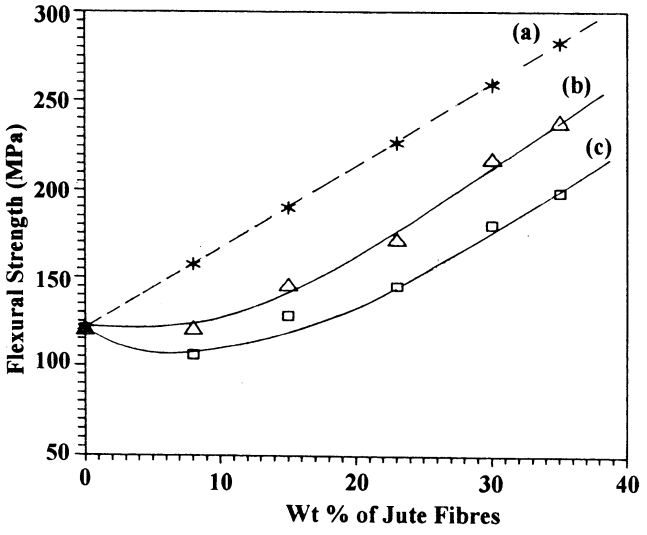


**Figure 7.** Variation of composite flexural strength with varying wt% of jute fibres: (**a**) theoretical, (**b**) experimental (4 h alkali treated fibres) and (**c**) experimental (untreated fibres).

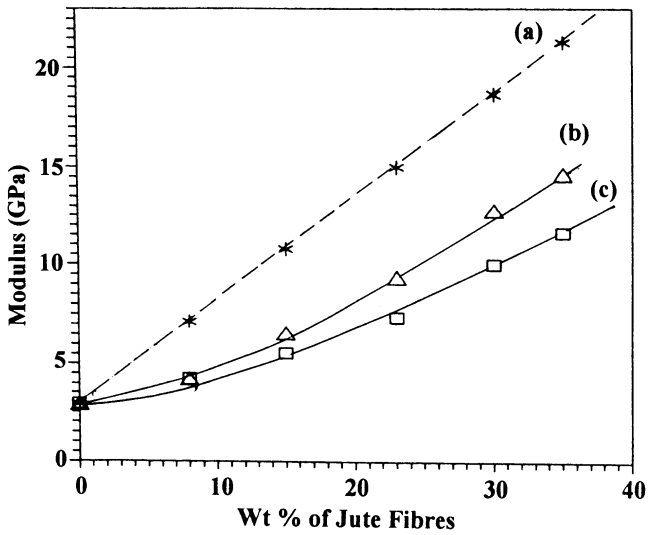


**Figure 8.** Variation of composite modulus with varying wt% of jute fibres: (a) theoretical, (b) experimental (4 h alkali treated fibres) and (c) experimental (untreated fibres).

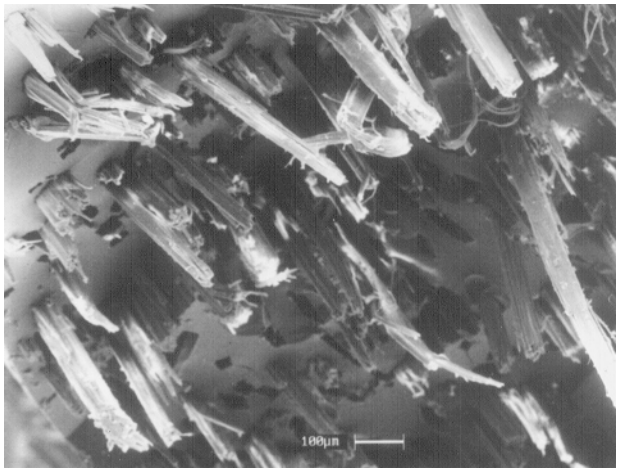

**Figure 9.** Fibre pull out and breakage in composites reinforced with 23 wt% untreated jute fibres.

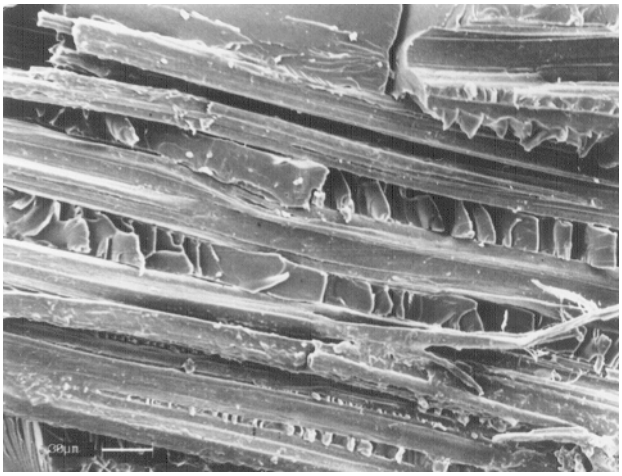

**Figure 10.** Matrix cracking and debonding of composites reinforced with 23 wt% 2 h alkali treated jute fibres.

matrix and resulted into superior properties of the composites. This was reflected in the fractured surfaces of the composites also when examined under SEM. The composites prepared with the NaOH treated jute fibres for shorter durations (2–4 h) had inferior bonding with the resin. The fibre breakage and pull out had been predominant (see figure 9), as demonstrated in load/displacement curve also. The matrix cracking and debonding of fibres at these stages were also prevalent (figure 10). The composites prepared with the alkali treated jute fibres for a longer duration (6–8 h) had a predominant transverse fracture with the minimum pull out (figure 11) of fibres, imperative of an increased load transfer characteristics from matrix to fibres as also revealed from the load–displacement curves (figure 3). Consequently, the deflection in the slope '*m*' indicated two different modes of fracture between the composites prepared with alkali treated jute fibre for the shorter and longer durations.

In contrast, composites prepared with untreated fibres for all sets of wt% of fibre reinforcement showed matrix failure followed by the fibre fracture and pull out irrespective of increased fibre loading due to the poor wetting characteristics by the vinylester resin.

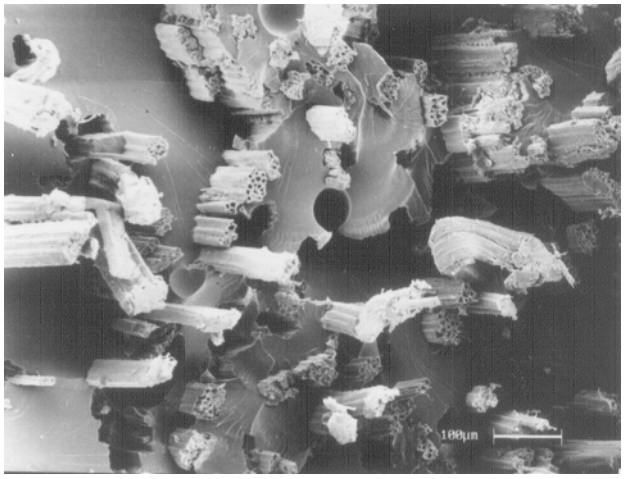

**Figure 11.** Transverse fracture of composites reinforced with 23 wt% 8 h alkali treated jute fibres.

## 4. Conclusion

The composites reinforced with alkali treated fibres showed improved mechanical properties. The improvement was maximum for the composites prepared with 4 h treated fibres at 35% fibre loading. The flexural strength improved by 20% and modulus by 23%. The strength and modulus of the composites were found to be lower than the values estimated from the general rule of mixtures. For the composites with a 35% fibre content, the strength was lower by 29% and 16% for the untreated and 4 h treated fibres and the modulus was lower by 51% and 37% for the untreated and 4 h treated fibres respectively. This reduction in strength was due to the incorporation of defects in the composites during processing. From the modified rule of mixture, the defects were estimated to be 10% in the matrix, 20% and 8·5% in the untreated and 4 h treated fibres respectively.

The rate of change of flexural strength and modulus were found to be linear with wt% of alkali treated fibres and the slope being maximum for 2 h and minimum for 8 h. The variation of slopes of the rate of change of flexural strength and modulus with alkali treatment time showed two separate zones with a deflection at 4 h treatment, indicating the possibility of two different failure modes before and after 4 h treatment. Fibre pull out was predominant up to 4 h and thereafter the transverse fracture occurred with minimum fibre pull out due to the superior bonding and improved tenacity and low extensibility of the fibres. A treatment time of 4 h was therefore optimal to get maximum strength of the vinylester composites reinforced with jute fibres treated with 5% NaOH solution at 30°C.

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