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Research Paper

EFFECT OF ALKALI TREATMENT ON MECHANICAL PROPERTIES OF *SISAL* -REINFORCED EPOXY POLYMER MATRIX COMPOSITE

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The present paper investigates the effect of fibre content and alkali treatment on tensile, flexural and impact properties of unidirectional *Sisal fibre* natural-fibre-reinforced epoxy composites which are partially biodegradable. The reinforcement *sisal fibre* fibre was collected from the foliage of locally available sisal bushes through the process of water retting and mechanical extraction. Commonly encountered problem between fibre and matrix is poor adhesion in natural-fibre-reinforced composites. To overcome this problem, specific treatments (physical and chemical) were suggested for surface modification of fibres by investigators. Alkali treatment is one of the simple and effective surface modification technique which is commonly used in natural fibre composites. In the present study both untreated and alkali-treated fibres were used as reinforcement in *sisal* epoxy composites and the tensile, flexural and impact properties were determined at different fibre contents. The alkali treatment is found to be effective in improving the tensile and flexural properties while the impact strength decreased.

Keywords: *Sisal* fibre, Epoxy resin, Alkali treatment, Mechanical properties

INTRODUCTION

The increase in the application of plant fibres as reinforcement for polymeric substrates has been stimulated by the environmental cost of manufacturing energy-intensive, synthetic fibres such as glass, carbon and kevlar. However, whereas synthetic fibres can be produced with engineered properties to suit particular applications this is not the case with naturally occurring plant fibres. Properties of the cellulose fibres depend mainly on the nature of the plant, locality in which it is grown, age of the plant and extraction method used. Today polymer composites are used for a wide range of applications. However, they do not undergo biodegradation easily, resulting in generation of solid waste, which causes environmental pollution. To meet this challenge, researchers are focusing their attention on producing bio-degradable composites with natural fibres/fabrics. Such composites are termed as green composites.

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L Y Mwaikambo, 1999) studied the effect of chemical treatment on sisal, Jute, hemp, Kapok etc., several green composites Shin *et al*., 1989) investigated the mechanical properties of bamboo-epoxy composites. Joseph *et al*., 2002) developed phenol formaldehyde com-posites reinforced with banana fibres and pineapple leaf fibre reinforced polyethylene composites have been de-veloped by George *et al*., 1998). Gomes *et al*., 2004) inves-tigated the influence of Alkali treatment on tensile properties of curaua fibre green composite.

Natural fibre composites are not only biodegradable and renewable but also possess several other advantages such as lightweight, low cost, high specific strength, high modulus, reduced tool wear and safe manufacturing pro-cess when compared with synthetic fibre composites .Nowadays polymer composite are used in construction, sports and recreational activities. Most of the parts of the automobiles, like door panels, trunk liners, seal backs, packages, speaker trays, engine and transmission covers, are made using natural fibre composites.

Despite several advantages they also possess few limitations such as poor wettability, poor adhesion incompatibility with some poly-meric matrices and high moisture absorption by the fibres (Batchiar *et al*., 2008). The greatest challenge in using the natural fibre as reinforcement in polymer matrix is the poor adhesion between natural fibre and matrix resulting in inferior strength of the composites. The main reason for the poor compatibility is that while polymer matrix is hydrophobic and non-polar, the natural fibres are hydrophilic and have polar groups in their structure. Moreover, natural fibres consist of several elementary fibres associated with cellulose, hemicellulose, pectin, lignin, etc. The mechanical properties of plant fibres are largely related to the amount of cellulose, which is closely associated with the crystallinity of the fibre and the micro-fibril angle with respect to the main fibre axis (Sreekala *et al*., 1997). Fibres with high crystallinity and/or cellulose content have been found to possess superior mechanical properties Hence, they can not be considered as the mono-filament fibres. To remove the unwanted elements from the fibre, specific treatments are necessary. Many investigators found and reported that the interfacial bonding can be enhanced by the surface modification of the fibre through alkali treatment and treatment with coupling agents, which in turn will enhance the overall per- Over synthetic composites. (Alexandre Gomes, 2004) studied the effect of alkali treatment on curaua fibres composites and reported improvement in tensile strength and fracture strain. Huda *et al*., 2008), in their investigation, pointed out that surface treated fibre-reinforced composites shown superior mechanical proper-ties as compared to the untreated fibre-reinforced composites. (Bisanda and Ansell., 1991) studied the effect of silanes on mechanical properties of sisal-epoxy composites and found considerable enhancement in compressive strength. (L Y Mwaikambo and M P Ansell, 1999) though Mercerisation and acetylation, have successfully modified the structure of natural fibres and these modifications resulted in improved performance of natural fibre composites by promoting better fibre to resin bonding.

(Theepopnatkul *et al*., 2009), Hildegardia fabric (Guduri *et al*., 2006) and oil palm fibres (Sreekala and Thomas, 2003). Valadez-Gonzalez *et al*., 1999) investigated the effect of fibre surface treatment on the fibre-matrix bond strength of natural fibre-reinforced composites and reported improvement in bond strength.

In the present study, *sisal fibres* are used as reinforcement. They are very-low-density fibres whose extraction from *sisal fibres* bush is very simple as its gum present in the sheath dissolves easily. The process is Economically feasible. *sisal fibres* are widely used in packaging and for making biodegradble bags due to their high strength and rigidity. In older references the genus of the tree was referred to as *Oreodoxa*. These bushes are normally found in all parts of india,Europe and north america, Central and South America,.Some of the basic properties of this fibre were investi-gated by (L YMwaikambo and M P Ansell *et al*., 2007). Epoxy was used as the matrix material in the present study. The role of the matrix is to keep the fibres in a desired location, orientation and to effectively transfer the stress to fibres. And also they protect the fibres from mechanical and chemical damage. Epoxy is most commonly used matrix material because of its desirable properties like high strength, low viscosity, low flow rate, low volatility during cure, low shrink rate, etc. The fibre-matrix inter-face plays a vital role in the transformation of load, from the matrix to the fibre. Even though mechanical properties of the natural fibres are much lower than those of synthetic fibres such as carbon, aramid, glass, etc., their specific properties, especially stiffness, are comparable. Natural fibres are very light in weight in comparison with the synthetic fibres. Most of the natural fibres are nearly 50% lighter than glass.

The present work investigated the effect of fibre con-tent in weight percentage and alkali treatment (with NaOH) on tensile, flexural and impact properties of *sisal* natural fibre-epoxy composites.

EXPERIMENTAL PROCEDURE

Materials

The matrix system used is an epoxy resin (Lapox-12) and hardener (k-8) supplied by Atul Limited, Gujarat, India. Lapox-12 is a liquid, unmodified epoxy resin of medium viscosity. Hardener k8 is a low-viscosity room-temperature curing liquid. It is generally preferred in hand-lay-up applications. Being reactive, it gives a short pot life and rapid cure at normal ambient temperatures. The reinforcement is a *sisal* fibre, which was collected from the foliage of locally available sisal bush through the water retting and mechanical extraction.

Fibre Extraction

From the foliage sheath, the leaves and leaf stem were removed and the sheath is dried for 2 days in shade. In the next step, it was immersed in water retting tank for 2 weeks, followed by hand rubbing and rinsing in water till the unwanted greasy material was dissolved and fine fibre was extracted. Finally, the extracted fibre once again washed thoroughly in plenty of clean water to remove the surplus waste. Continuous fibre was obtained with length up to 1.5 m. The obtained fibre was dried under sun for 6 days.

The average diameter of the fibre used for the composite preparation was between 0.2 and 0.3 mm. It contained Sisal Cellulose (wt%) 67-78, Lignin(wt%) 8-11, Hemicellulose (wt%) 10.0-14.2, Pectin (wt%) 10.0, Wax (wt%) 2.0, Moisture content(wt%) 11.0.

Alkali Treatment

Reinforcing fibers can be modified by physical and chemical methods. Physical methods, such as stretching, calandering, thermo treatment, and the production of hybrid yarns do not change the chemical composition of the fibers. The dry fibre was treated with 5% solution of NaOH for 2 h to remove the unwanted soluble cellulose, pectin, lignin, etc. from the fibre. The fibre to solution weight ratio was maintained at 1 : 20. After 2 h the fibre was washed thoroughly in distilled water containing 1% acetic acid to neutralise and remove excess amount of NaOH and dried at 50°C for 20 h. The fibres are dried to remove free water and placed in a glass container in a conditioning chamber.

Chemical Analysis for Alpha (α)-Cellulose

Chemical analysis was done for the determination of cellulose as per Indian standards (IS: 6213, PART III-1971) with 17.5% and 8.3% (by weight) sodium hydroxide reagents. The residual high-molecular-weight fraction was left behind when a mixture of fibre and 8.3% NaOH solution is filtered after the fibres have been initially swollen in a 17.5% NaOH solution. The residue was washed with distilled water followed by acetic acid solution at 20°C and soaked for 5 min. Again, it was washed with the distilled water to free it from the acetic acid and dried in the oven at 15°C. Next contents were transferred to a weighing bowl and dried to a constant weight.

% alpha (α)-cellulose can be calculated as follows:

$x = 100a/m$,

where x = % alpha (α)-cellulose by weight; a = weight of precipitate in grams; m = weight of fibre in grams calcu-lated on oven dry basis.

Composite Preparation

The mould box was made with the dimension of 180 mm (L) 130 mm (W) 3.0 (T) mm. The required equipment for making the mould for hand-lay-up process were glass, sticker, spacer frames and a transparent plastic film to keep on the top of the uncured composite before it was closed and compressed. The treated and untreated fibres were cut according to the mould size. Then, the matrix was prepared by mixing the hardener to epoxy. The epoxy and hardener ratio was maintained at 12 : 1. To get the well-cured and a standard-quality specimen, the epoxy and hardener is mixed smoothly and slowly for approximately 8 min. Initial layer of the mould was filled with epoxy resin mixture and then the appropriate quantity of fibres was placed such that epoxy mixture is completely spread over the fibres. Again, epoxy mixture is poured on the fibre. Thus, the starting and ending of the layers were of epoxy resin. The plastic releasing film was placed on the top of the uncured mixture. Before applying compression, efforts were made to remove all bubbles with roller. Finally, the compression pressure was applied uniformly to achieve the uniform thickness of 3 mm and

cured for 24 h at room temperature. The obtained composite laminates are of the size 180 130 3 mm^3.

Testing of the Composites

The composite specimens were tested as per ASTM standards. Tensile testing was done as per ASTM D 3039-76 with the help of FIE-model Universal Testing Machine at a crosshead speed of 10 mm/min. The temperature was conditioned at 24°C with the humidity of 50%. The specimen dimensions were 150 15 3 mm^3. Flexural testing was done as per ASTM D 5943-96 standards using three point bending method at a cross-head speed of 5 mm/min and at a temperature of 24 C with the humidity of 50%. The specimen dimensions were 100 15 3 mm 3. The impact testing was done as per ASTM D 256-88 by Izod impact machine with unnotched specimen. The specimen dimensions were 120 12. 2 mm^3. In each case, eight samples were tested and the average values were reported.

RESULTS AND DISCUSSION

Tensile strength, tensile modulus and % elongation at break of untreated *sisal* fibre (UTRF)-reinforced and alkali-treated *sisal* fibre (ATRF)-reinforced composites were presented in table 1 at different fibre contents (5, 10, 15 and 20% wt.). For correct understanding of the effect of fibre content on tensile strength, ten-sile modulus and percentage elongation at break separate charts have been plotted for untreated and alkali-treated types of composites as shown in figures 1-3, respectively. From Figures 1-3, it is evident that fibre content is directly proportional to tensile strength, tensile modulus and percentage elongation at

Figure 1: The Effect of Fibre Content on Tensile Strength of Untreated and Alkali-treated Composites

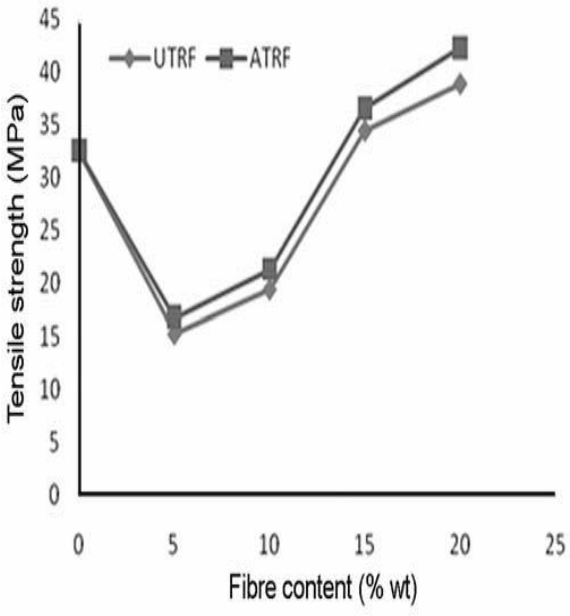


Figure 2: The Effect of Fibre Content on Tensile Modulus of Untreated and Alkali-treated Composites

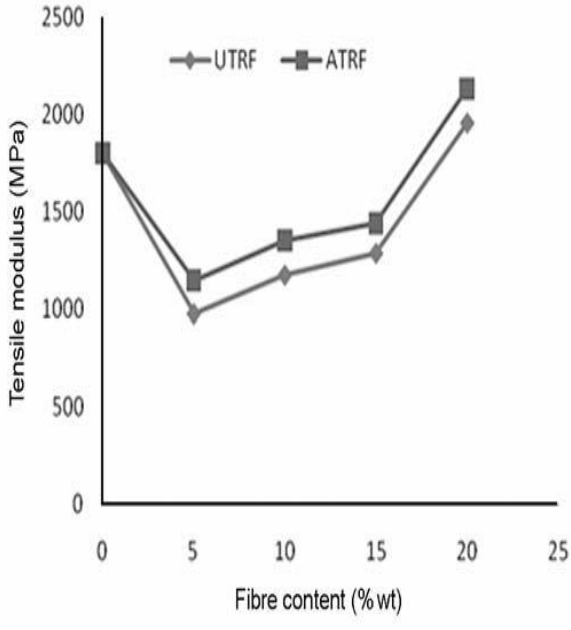


Table 1: Tensile Properties of Untreated and Treated Composites at Different Fibre Contents

Type of composite	Fibre content (% wt.)	Tensile strength (MPa)	Tensile modulus (MPa)	Elongation at break (%)
Epoxy without reinforcement	0	32.5	1800.00	1.6
UTRF	05	15.34	980.95	2.37
	10	19.47	1178.60	3.30
	15	34.47	1289.76	3.39
	20	38.89	1957.31	3.82
ATRF	05	16.78	1149.87	2.49
	10	21.29	1356.72	3.51
	15	36.56	1442.71	3.71
	20	42.29	2131.69	3.92

Table 2: Flexural Properties of Untreated and Treated Composites at Various Fibre Contents

Type of composite	Fibre content (% wt.)	Flexural strength (MPa)	Flexural modulus (MPa)
Epoxy without reinforcement	0	39.00	3507.40
UTRF	05	33.423	3705.18
	10	42.828	3997.78
	15	44.187	4158.25
	20	57.655	4568.25
ATRF	05	34.808	4550.80
	10	47.243	4790.02
	15	52.052	4910.00
	20	62.677	4945.00

Figure 3: The Effect of Fibre Content on % Elongation at Break of Untreated and Alkali-treated Composites

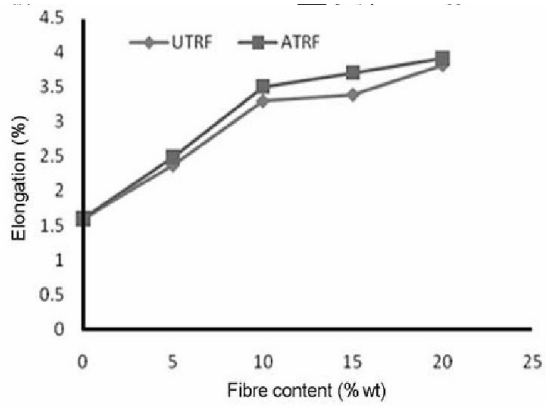
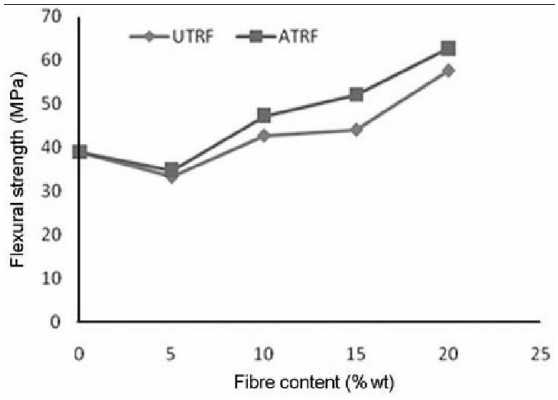


Figure 4: The Effect of Fibre Content on Flexural Strength of Untreated and Alkali-treated Composites



break for both treated and untreated fibre composites and peak value is at 20% fibre content. When com-pared with the untreated fibre composites, the alkali-treated fibre composites recorded 8% increase in tensile strength, 8.2% increase in tensile modulus and 2.6% increase in percentage elongation at break is at 20% fibre content.

Flexural properties of untreated and treated composites at various fibre contents were shown in Table 2 and separate charts for flexural strength and flexural modulus are depicted in Figures 4 and 5. From the figures it can be seen that fibre, content, is directly proportional to flexural strength and flexural modulus for both treated and untreated fibre composite and peak value is at 20% fibre content.

Table 3: Impact Strength of Untreated and Treated Composites at Various Fibre Contents

Type of composite	Fibre content (% wt.)	Impact strength (J/m)
Epoxy without reinforcement	0	30.6
UTRF	05	74.36
	10	92.35
	15	116.52
	20	148.94
ATRF	05	69.32
	10	87.94
	15	98.06
	20	116.43

Figure 5: The Effect of Fibre Content on Flexural Modulus of Untreated and Alkali-treated Composites

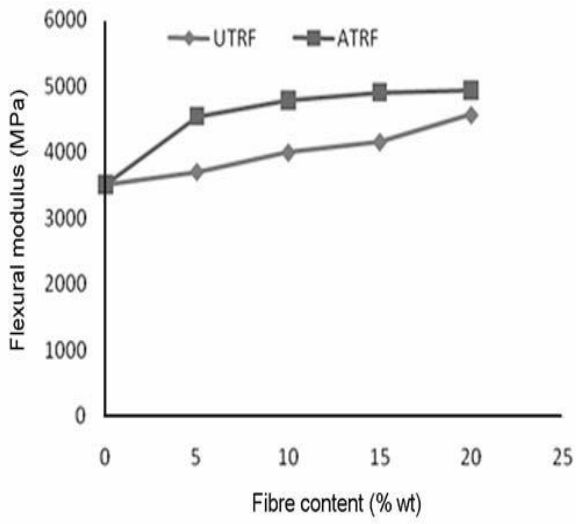
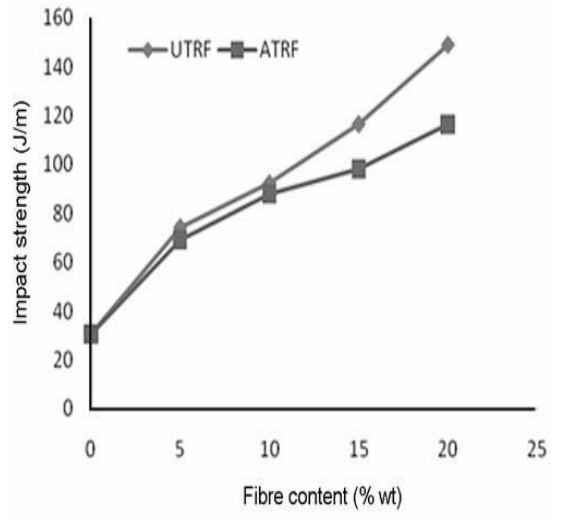


Figure 6: The Effect of Fibre Content on Impact Strength of Untreated and Alkali-treated Composites



untreated fibre composites, the alkali-treated fibre com-posites recorded 8% increase in flexural strength and 7.7% increase in flexural modulus at 20% fibre content. This increase in tensile and flexural properties of alkali-treated fibre composites could be due to the increased interface between matrix and fibre after treatment. This is evidenced by the fracture morphology of tensile and flex-ural composites before and after alkali treatment as shown in the figures 7a-d. The fibre damage is more in untreated fibre-reinforced composites when compared with the alkali-treated fibre-reinforced composites. The alkali treatment by removing hemicellulose and lignin contents from the fibre yields the higher percentage of α (alpha) cellulose in natural fibres Jayaramudu et al (2009). Chemical analysis revealed that after alkali treatment, the percentage of α-cellulose of *Roystonea regia* fibre increased from 58 to 65%. This will render the fibre surface coarser, leading to better interface between matrix and fibre. Alkalization also causes fibril-lation, i.e. breaking of fibre bundles in to smaller fibres, which would increase the effective surface area available for wetting by the matrix material (Yan *et al*., 2000). After fibrillation due to the reduced diameter of the fibre, the aspect ratio of the fibre increases and yields rough surface topography, which in turn offers a better fibre-matrix interface. This results in obtaining the enhanced properties. The effect of alkali treatment in modifying surface of fibre can be observed by scanning electron microscopy (SEM) as shown in the Figures 8a and b. The alkali-treated fibre has become coarser (see Figure 8b) when compared with the untreated fibre (see Figure 8a).

Izod impact test results were reported in table 3 for untreated and alkali-treated composites at various fibre contents and the corresponding chart was shown in the Figure 6. From the figure it is evident that although there is an increase in impact strength with increase in fibre loading, the values were decreased after alkali treatment and, at 20% fibre loading, the impact strength was de-creased by 22%. This could be mainly due to the reason that during the impact considerable part of energy absorp-tion takes place through the fibre pull-out process, but after alkali treatment, due to the removal of soluble greasy contents from the fibre, strong mechanical interlocking develops between fibre and matrix, and fibre pull-out is minimized. This, in turn, will decrease the impact strength.

Figure 7: (a) Tensile Fractured Surface of Untreated Composite (20% Fibre Content). (b) Tensile Frac-tured Surface of Alkali-treated Composite (20% Fibre Content). (c) Flexural Fractured Surface of Untreated Composite (20% Fibre Content). (d) Flexural Fractured Surface of Alkali-treated Composite (20% Fibre Content)

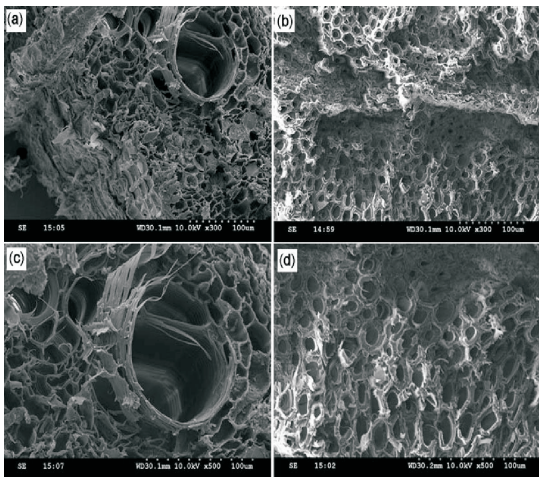
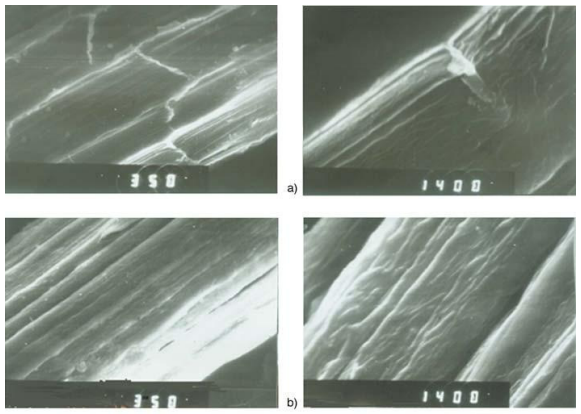


Figure 8: (a) Untreated Fibre Surface. (b) Alkali-treated Fibre Surface



CONCLUSION

Tensile strength, tensile modulus and percentage of elonga-tion of untreated and alkali-treated *sisal* natural fibre-reinforced epoxy composites were increased with increase in fibre content and are highest at 20% wt. fibre content. Alkali-treated fibre composites have shown superior tensile properties than untreated composites. Also flexural strength and flexural modulus of untreated and alkali-treated fibre composites were increased with increase in fibre content and are highest at 20% wt. fibre content. Alkali-treated composites have shown superior flexural properties than untreated fibre composites. For both untreated and alkali-treated fibre composites, there is an increase in impact strength with increase in fibre con-tent and found to be highest at 20% wt. fibre content. But, the impact strength of alkali-treated fibre composites was decreased at all fibre contents when compared with the untreated fibre composites. Study demonstrated that *sisal* natural-fibre-reinforced epoxy compo-sites could be successfully produced with good mechanical properties and the tensile and flexural properties can be further enhanced by alkali treatment. This study also reveals that maximum strength and maximum toughness cannot be achieved simul-taneously and optimum combinations of desired mechanical properties are possible only through careful design of composites.

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