# EFFECT OF SURFACE TREATMENT ON THE MECHANICAL PROPERTIES OF BAGASSE FIBER REINFORCED POLYMER COMPOSITE

Samir Kumar Acharya,<sup>a,\*</sup> Punyapriya Mishra,<sup>a</sup> and Suraj Kumar Mehar<sup>b</sup>

Bagasse is a by-product of the sugarcane milling process, and it also is an important fuel resource for that industry. In this study an attempt has been made to utilize this by-product to prepare a composite using epoxy resin. The fibers surface was modified by alkali treatment with 5% NaOH solution for 0, 2, 4 and 6 hrs. The effect of fiber surface modification on the mechanical properties such as flexural strength of the composites was investigated with the fibers as received from the milling process. It was found that alkali-treated bagasse/epoxy composites significantly improved the flexural strength of the composite. An SEM investigation also indicated that the surface modifications improved the fiber–matrix interaction.

Keywords: Surface modification; Bagasse fiber; Reinforcement; Environment; Flexural strength

Contact information: a: Department of Mechanical Engineering, N.I.T. Rourkela 769008, India; b: Department of mechanical Engineering, OPJindal Institute Of Technology, Raigarh 496001, India; \* Corresponding author: drsamirka@yahoo.com

# INTRODUCTION

Natural fiber composites are emerging as realistic alternatives to glass-reinforced composites in many applications. The attractive features of natural fibers such as jute (Roe et al. 1985; Sridhar et al. 1984; Kumar et al. 1986; Shah et al. 1981), sisal (Bisanda et al. 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. Natural fiber composites are also claimed to offer environmental advantages such as reduced dependence on non-renewable energy/material sources, lower pollutant emissions, lower greenhouse gas emissions, and enhanced energy recovery. These fibers are sensitive to temperature and moisture and usually have irregular crosssections. The main bottlenecks in the broad use of natural fiber in various polymer composites are poor compatibility between the fibers and the matrix, and the inherent high moisture absorption, which brings about dimensional changes in the lignocellulose-based fibers (Rout et al. 2001). Several authors have contributed to the development of polymer composites using different types of natural fibers. In this work (natural and chemically treated) sugarcane bagasse fibres have been utilized for similar study.

Bagasse is the solid lignocellulosic residue left after extraction of juice from the sugarcane stalk (Fig. 1). The principal use of bagasse is as a combustible material for energy supply in sugarcane factories, in countries such as Egypt, Cuba, etc. It also is used in pulp and paper industries and for fiberboard materials (Sefain et al. 1998). In general, the shortcomings of natural fiber-reinforced composites have been their high moisture

absorption, poor wettability, and poor fiber-matrix adhesion. These statements are also true for bagasse fiber; for instance if the bagasse fiber has not been subjected to any chemical surface treatment, it certainly contributes to a weak interface between the bagasse and the resin matrix. This is because the lignin content of sugarcane bagasse is high (21% on average) (Cadenas et al. 1990). A high lignin content has been proposed as a factor preventing good surface wettability between polymer matrix material and natural fiber (Navarro et al. 1991), although it increases the resistance to chemical and microbial attack (Godfried et al. 1975).



Fig. 1. Bagasse

In the present study an attempt has therefore been made to prepare a bagasse fiber composite by treating the surface of the fiber with alkali to increase the adhesion between the fiber and the resin matrix and to study its mechanical properties, subjecting the composite samples to different environmental treatments. The improvement in the mechanical properties (viz., flexural strength) of the specimen treated at different weathering conditions is also reported in this study. Furthermore interfacial studies were made with SEM to ascertain the effects of the surface treatment on the mechanical properties of the composite.

# EXPERIMENTAL

#### Materials

Table 1 shows a typical bagasse composition obtained after chemical analysis. In general, the results obtained from the analysis are in accordance with the previous work (Shibata Shinichi et al. 2005). However, these proportions in a fiber depend on the age, source of the fiber, and the extraction conditions used to obtain the fiber. Fresh bagasse fibers were collected after they were crushed for extracting juice by using a hand crushing machine. After extracting the juice, the fibers were spread on a waterproof sheet to reduce the moisture content. From the available long fibers (rind portion only), small size (12x1x1 mm) fibers were cut with a pair of scissors. Small size fibers were then cleaned via pressurized water for about 1 hr. This procedure removes fine bagasse particles, sugar

residues, and organic materials from the samples. The fibers were then dried with compressed air at a pressure of approximately 145kPa at 108°C. The required drying time was determined by weighing a trial sample every ten minutes until the measured mass became constant. A drying time of 40 min was established to provide sufficient drying of the fiber.

ITEMS	PERCENTAGE (%)		
Moisture	49.0		
soluble solids	2.3		
fiber cellulose hemicellulose lignin	48.7 41.8 28.0 21.8		

**TABLE 1.** Average Bagasse Composition

#### Alkali Treatment of Bagasse Fiber

Alkaline treatment or mercerization is one of the most used chemical treatments for natural fibers that are to be used to reinforce thermoplastics and thermosets. The bagasse fibers were soaked in a 0, 1, 3, and 5% (w/w) NaOH solution at room temperature, maintaining a liquor ratio of 15:1 (w/w). The fibers were kept immersed in the alkali solution for 2, 4, and 6 hrs. The fibers were then washed several times with fresh water to remove any NaOH sticking to the fiber surface, neutralized with dilute acetic acid, and finally washed again with distilled water. A final pH of 7 was maintained. The fibers were then dried at room temperature for 48 hrs, followed by oven drying at 100°C for 6 hrs.

# **COMPOSITE PREPARATION**

#### Fabrication

The type of epoxy resin used in the present investigation was Araldite LY556, which was used together with hardener HY 951, both supplied by Ciba-Geigy of India Ltd. A wooden mold of dimensions 120x100x 6mm was used for casting the composite sheet. A usual hand lay-up technique was used for preparation of the samples. A mold-release spray was applied at the inner surface of the mold for quick and easy release of the composite sheet. A calculated amount of epoxy resin and hardener (ratio of 10:1 by weight) was thoroughly mixed with gentle stirring to minimize air entrapment. After keeping the mold on a glass sheet (coated with mold release spray), a thin layer ( $\approx$ 2mm thickness) of the mixture was poured into the mold. The bagasse fibers were then distributed uniformly over the mixture, and the remainder of the mixture was then poured into the mold. Pressure was then applied from the top, and the contents of the mold were allowed to cure at room temperature for 72 h.

#### **Experimental Design**

The composite systems outlined in Table 2 were manufactured to investigate varying properties such as fiber volume fraction and chemical treatment of fibers. From the first group of samples (Group 1) 10, 20, 30, and 40% volume fraction, the initial results from flexural testing and the environmental treatment indicated that 30% fiber volume fraction was the optimum level, yielding the maximum strength. Based on this result the second group of samples of 30% fiber volume fraction was selected for further experimentation (Group 2). The second group of samples involved 30% volume fraction of fibers treated with varying concentration of NaOH, as indicated in the table. The maximum improvement in the flexural strength of the composite was observed for 5%(w/w) NaOH-treated fibers. The third group of samples was prepared with 30% volume fraction of fibers soaked in a 5% (w/w) NaOH solution for 2, 4, and 6 hr, from which it was found that 4 hrs was the optimum. Considering these results, the fourth group of samples involved varying fiber surface treatment, namely (i) unwashed bagasse, (ii) unwashed and treated with alkali (5% concentration, treated with 4 hrs).

# EXPERIMENTAL PROCEDURE

These composite samples were treated in various environmental conditions including subzero (at -25°C), steam (at 160°C), and saline water (at room temperature). The effect of environment on the flexural strength of the composites has been studied.

#### Flexural Strength

The composites, after being exposed to various weathering conditions, were subjected to the three-point bend test. This test was carried out in an UTM 201 machine in accordance with ASTM D2344-84 to measure the flexural strength of the composites. All the specimens (composites) were of rectangular shape, having lengths varying from 100 to 125 mm, breadth of 100 to 110 mm, and thickness of 4 to 6 mm. A span of 100 mm was employed, maintaining a cross-head speed of 10 mm/min.

#### SEM Studies

To characterize the morphology of untreated and treated fiber surfaces and the mode of material removal, the samples were observed under a scanning electron microscope (SEM) Joel JSM-6084LV. The samples are mounted on stubs with silver paste. To enhance the conductivity of the composite samples, a thin film of platinum was vacuum evaporated onto them before the photographs were taken.

# **RESULTS AND DISCUSSION**

The variations in flexural strength for the 10, 20, 30, 40% fiber composites in natural, steam, saline, and subzero environments are shown in Fig. 2. The plot shows that the samples with 30% fiber volume fraction gave the highest strength in the normal

condition and also in steam, saline, and subzero conditions. Based on these results, for further experimentation 30% fiber volume fraction of composite was taken, as indicated in Table 2.



**Fig. 2.** Flexural strength of 10, 20, 30, and 40% fiber volume fraction of composites under normal steam, saline and subzero conditions

0	0	0/ 1	<b>T</b>	D a second a		
Group	Sample	% volume	туре	Remarks		
		fraction				
Group1	1	10	Washed bagasse fiber	30% Optimum		
Volume	2	20	Washed bagasse fiber			
Fraction	3	30	Washed bagasse fiber			
Analysis	4	40	Washed bagasse fiber			
			Conc. of alkali (%)			
Group2	5	30	0			
Fiber surface	6	30	1	5% Optimum		
Treatment	7	30	3			
	8	30	5			
Group3			Hrs of Treatment			
Fiber surface	9	30	2	4hrs Optimum		
Treatment	reatment 10		4			
	11	30	6			
	12	30	Unwashed bagasse fibers			
Group4	13	30	Unwashed bagasse fibers			
Fiber surface			treated with alkali			
Treatment	14	30	Washed bagasse fiber treated			
			with alkali			

TABLE 2.	Types of	Badasse	Samples	Used in	the F	Research
	1 9 0 0 0	Duguooc	Gumpico	0000 111		Cocuron

The effect of alkali treatments (0%, 1%, 3%, 5%) of bagasse fibers on the flexural strengths were examined using treated fiber composites. As seen from Fig. 3, the greatest improvement in the flexural strength of the composite was observed for 5% NaOH treated fibers. It was believed that better interfacial adhesion along with better fibrillation of these fibers contributed effectively to the enhancement in the flexural properties.

# bioresources.com



Fig. 3. Effect of alkali treatment on mechanical properties of composites











Figure 4 (a-d) shows the flexural strength of the composite for unwashed treated and washed treated fibers with 5% (w/w) alkali for 2, 4, and 6 hours after subjecting them to different environmental conditions. It is evident from figure that in all cases the flexural strength was the highest for composites that had been both washed and treated for 4 hrs. The increase in flexural strength was about 7.67%, 23.34%, 17.43%, and 17.56% for normal, steam, saline and subzero conditions respectively. Hence for rest of the investigations, composites were prepared with 4 hours alkali treated fibers for analysis.

Figure 5 shows the flexural strength of unwashed, washed treated, and unwashed treated composites samples. It is clear that flexural strength of washed and 4 hrs alkali (5% conc.) treated composites was maximum for all cases as compared to unwashed and unwashed treated fiber composites.



Fig. 5. Comparison of flexural strength of unwashed, unwashed treated and washed treated fiber composites under steam, saline and subzero conditions

The important modification done by alkaline treatment is the disruption of hydrogen bonding in the network structure, thereby increasing surface roughness. This treatment removes a certain amount of lignin, wax, and oils covering the external surface of the fiber cell wall, depolymerizes cellulose, and exposes the short-length crystallites. Addition of aqueous sodium hydroxide (NaOH) to natural fiber promotes the ionization of the hydroxyl group to the alkoxide (Agrawal et al. 2000).

Fiber – OH + NaOH  $\rightarrow$  Fiber – O – Na +H<sub>2</sub>O

Thus, alkaline processing directly influences the cellulosic fibril, the degree of polymerization and the extraction of lignin and hemicellulosic compounds (Jähn et al. 2002). It is reported that alkaline treatment has two effects on the fiber:

- 1) It increases surface roughness resulting in better mechanical interlocking, and
- 2) It increases the amount of cellulose exposed on the fiber surface, thus increasing the number of possible reaction sites (Valadez et al. 1999).

Consequently, alkaline treatment has a lasting effect on the mechanical behavior of bagasse fiber, especially on fiber strength and stiffness.

#### FRACTOGRAPHIC ANALYSIS

Figure 6 shows the fiber surface before and after alkali treatments. It was observed that the filaments in the untreated fiber were packed together but became split after the alkali treatment. This phenomenon is termed fibrillation, and it involves breaking the untreated fiber bundle down into smaller ones by the dissolution of the hemicellulose. The fibrillation increases the effective surface area available for contact with the matrix and hence increases the interfacial adhesion which leads to increase in the flexural strength of the composite.



Untreated

5% NaOH treated

Fig. 6. SEM micrograph of a baggsse fiber

Most of the fibers, as shown in Fig. 7 (a), had come out from the matrix without breaking during fracture for the composite subjected to steam treatment. Probably dissolution of cellulose constituent in alkali creates voids in the fiber structure, which increases swelling and makes the fiber weaker. Destruction of the network and splitting of fibers in to filaments might have occurred during treatment, which is responsible for this type of failure in the composite.

Figure 7(b) shows the micrographs of the samples subjected to subzero condition. It clearly indicates the breaking down of fiber bundles into smaller ones. This increases the effective surface area available for wetting by the resin. When subjected to subzero conditions, the absorption of water is less, as explained earlier; hence leading to higher flexural strength.

Figure 7(c) shows the micrograph for the saline exposed sample. The same types of features were seen as for the subzero condition. The breaking of fibers due to fibrillation is clearly visible, helping to explain the higher strength.

(h



100

(a



(c

**Fig. 7.** Fracture surface features of the samples treated with alkali subjected to (a) steam, (b) subzero, and (c) saline treatment

# CONCLUSIONS

- 1. Sugarcane residue bagasse, an underutilized renewable agricultural material, can successfully be utilized to produce composite by suitably bonding with resin for a value-added product.
- 2. By comparing the flexural strength of the composites with fiber treatment, the best mechanical property results are obtained with bagasse fibers that have been both washed and treated with alkali. The increase in flexural strength was about 7.67%, 23.34%, 17.43%, and 17.56% for normal, steam, saline, and subzero conditions.
- 3. Fibrillation in the fiber bundles was observed when the fibers were treated with alkali by the dissolution of hemicellulose. This also increased the effective surface area available for contact with the matrix, and hence the interfacial adhesion was improved which finally improves the flexural strength of the composite.
- 4. In general, fiber pull-out is the predominant mode of failure for natural fiber composites. In our case the morphology of the fractured surface (treated under different environment) for the alkali-treated fiber implied that fiber breakage was the predominant mode of failure. Washed and treated samples with alkali improved the fiber matrix bonding, increasing composites' flexural strength.

# **REFERENCES CITED**

- Agrawal, R., Saxena, N. S., Sharma, K. B., Thomas, S., Sreekala, M. S. (2000). "Activation energy and crystallization kinetics of untreated and treated oil palm fibre reinforced phenol formaldehyde composites," *Material Science Engg: A* 277(1), 77-82.
- Bisanda, E. T. N., and Ansell, M. P. (1991). "The effect of saline treatment on the mechanical and physical properties of sisal–epoxy composites," *Comp. Sci. Tech.* 41, 165-178.
- Cadenas, G. A., Mitrani, R. B., Pena, C. G., Munilla, M. H., and Correa, J. L. (1990). In: Silverio, H. N. (ed.) *Manual de los Derivados de la Cana de Azucar*, 2nd Ed. (in Spanish). GEPLACEA, Mexico City, Mexico, p. 47.
- Godfried, L. M. (1975). Unsaturated Polyester Resin. Its Production and Reinforcement with Natural Organic Fibers, Fokker-VFW B.V. Report, The Netherlands.
- Jähn, A., Schröder, M. W., Füting, M., Schezel, K., and Diepenbrock, W. (2002). "Characterisation of alkali treated flax fiber by means of FT Raman spectroscopy and environmental SEM," *Spectrochim. Acta A: Mol. Biomol. Spectrosc.* 58, 2271-2279.
- Kumar, P. (1986). "Mechanical behavior of jute fiber and their composites," *Indian J. Tech.* 24, 29-32.
- Navarro, R. F., Medeiros, J., Mariz, T. F., and Maia, D. F. (1991). "Determination of cellulose and lignin content from luffa cylindrical fibers (in Portuguese)," *Proceedings of the 1<sup>st</sup> Brazilian Polymer Conference*, Sao Paulo, Brazil, 2, 679-681.
- Pothan, L. A., Thomas, S., and Neelakantan, N. R. (1997). "Short banana fiber-reinforced polyester composites: Mechanical, failure and aging characteristics," *J. Reinforced Plastics*, 16, 744-765.
- Prasad, S. V., Pavithran, C., and Rohatgi, P. K. (1983). "Alkali treatment of coir fibers for coir–polyester composites," *J. Mater. Sci.* 18, 1443-1454.
- Roe, P. J., and Ansell, M. P. (1985). "Jute-reinforced polyester composites," J. Mater. Sci., 20, 4015-4020.
- Rout, J., Mishra, M., Nayak, S. K., Tripathy, S. S., and Mohanty, A. K. (1999). "Effect of surface modification of coir fiber on physichomechanical behavior of coir-polyester composite," In: Ghose, A. K. (ed.) *Polymers Beyond AD2000*, 489-491.
- Rout, J., Misra, M., Tripathy, S. S., Nayak, S. K., and Mohanty, A. K. (2001). "The influence of fiber treatment on the performance of coir-polyester composites," *Compos. Sci. Technol.*, 61, 1303-1310.

Sefain, M. Z., Fadt, N. A., and Qakha, M. (1998). J. Appl. Chem. Biotechnol. 29, 79-84.

- Shah, A. N., and Lakkad, S. C. (1981). "Mechanical properties of jute-reinforced plastics," *Fibre Sci. Tech.*, 15, 41-46.
- Shibata, S., Cao, Y., and Fukumoto, I. (2005). "Press forming of short natural fiber reinforced biodegradable resin: Effect of fiber volume and length of flexural properties," *Polymer Testing* 24, 1005-1011.
- Sridhar, M. K., Basavarappa, G., Kasturi, S. G., and Balasubramaniam, N. (1984). "Mechanical properties of jute/polyester composites," *Indian J. Tech.*, 22, 213-215.

Valadez-Gonzalex, A., Cervates-Uc, J. M., Olayo, R., and Herrera Franco, P. J. (1999). "Chemical modification of henequen fibers with an organosilane coupling agent," *Compos B: Eng* 30(3), 309-320.

Article submitted: Feb. 5, 2010; Peer review completed: Feb. 15, 2011; Revised version received: March 8, 2011; Accepted: June 7, 2011; Published: July 6, 2011.