Influence of Alkali-Treated Fibers on the Mechanical Properties and Machinability of Roselle and Sisal Fiber Hybrid Polyester Composite

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In this work, the alkali-treated roselle and sisal fibers were used as reinforcement fillers for thermosetting matrix with aim of obtaining better mechanical properties and machinability of natural fiber hybrid polyester composite. However, their mechanical properties and machinability were compared with untreated fiber composites. The roselle and the sisal fibers were subjected to a 10% sodium hydroxide solution treatment at different duration of 2, 4, 6, and 8 h. Besides, the fractured surfaces of composite specimen were investigated using scanning electron microscopy. Drill hole profiles were analyzed using profile projector and machine vision inspection system. An improvement in strength and stiffness combined with high toughness was achieved by treating the fibers using 10% NaOH solution. POLYM. COMPOS., 31:723–731, 2010. © 2009 Society of Plastics Engineers

INTRODUCTION

A decade ago, the natural fiber reinforced composites have shown a growth of interest due to their high environmental friendliness, recyclability, and their high specific properties. Shah and Lakkad [1], Roe and Ansell [2], Lilholt and Bjerre [3], and Mohanty and Misra [4] investigated a diverse usage of natural fibers as reinforcement with considerable interest. Roe and Ansell [2] and Shah and Lakkad [1] investigated the natural fiber likes jute reinforced composites that have attractive features like low cost, light weight, high specific modulus, renew ability, and biodegradability. Bisanda and Ansell [5] studied the sisal fiber reinforced composites and its attractive features like low cost, light weight, etc. Prasad et al. [6] and Rout et al. [7] studied the coir reinforced composites that have attractive features high specific modulus, renew ability, and biodegradability, etc. Sebe et al. [8], Chawla and Bastos [9] and Harikumar et al. [10] reported that the composites reinforced with the natural fibers have been used in contrast to the synthetic fiber reinforced composites in low strength and low cost application. Several thermoplastic and thermoset matrices were used with cellulose fibers. Ray et al. [11], Mishra et al. [12] and Singh et al. [13] reported that the polyester and phenolic resins gave the best results for natural fibers, despite the multiuse of thermoplastic and thermosetting resins as matrixes. Samal et al. [14], Gassan and Bledzki [15], and Gassan and Bledzki [16] reported that because the mechanical properties of the composites mainly depends up on the interfacial bond between the reinforcing fibers and the resin matrix, several authors turned their studies on the treatment of fibers to improve the bonding with resin matrix. Dash et al. [17] Investigated the weathering behavior of natural fiber reinforced composite as well as the influence of water absorption on the mechanical properties of the laminated composites. Ray and Sarkar [18], De Albuquer-

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que et al. [19], and Semsarzadeh and Amiri [20] studied a numerous surface treatments like slivers bleaching, for example, with alkali (NaOH) or silane coating to reduce the sensitivity of natural fiber composites to weathering. Sarkar [21] and Mukherjee et al. [22] investigated the changes of properties of jute fibers during surface treatment. Samal et al. [14] and Sarkar [21] reported that the alkali-treated jute fibers with NaOH solution of 1%, 8% for 48 h and 2% for 1 h, respectively, showed improvements in fiber properties by 130% and 13%, respectively. Gassan and Bledzki [15] and Gassan and Bledzki [16] investigated the alkali treatment of isometric jute yarns. An improvement of 120% and 150% in the tensile strength and modulus of jute yarns was achieved with 25% NaOH solution for 20 min while there was 60% improvement in the jute/epoxy composite properties reinforced with treated yarns. The improvements have been attributed to the greater reactivity of the treated fibers with the resin administering superior bonding. Ott et al. [23] used the alkali treatment for jute fiber to obtain good bonding between the fiber and the resin matrix. This work reports the influence of alkali treated fibers on mechanical properties like tensile, flexural and impact strength, and machinability of hybrid polyester composites. The results were compared with untreated roselle and sisal fiber hybrid polyester composite material.

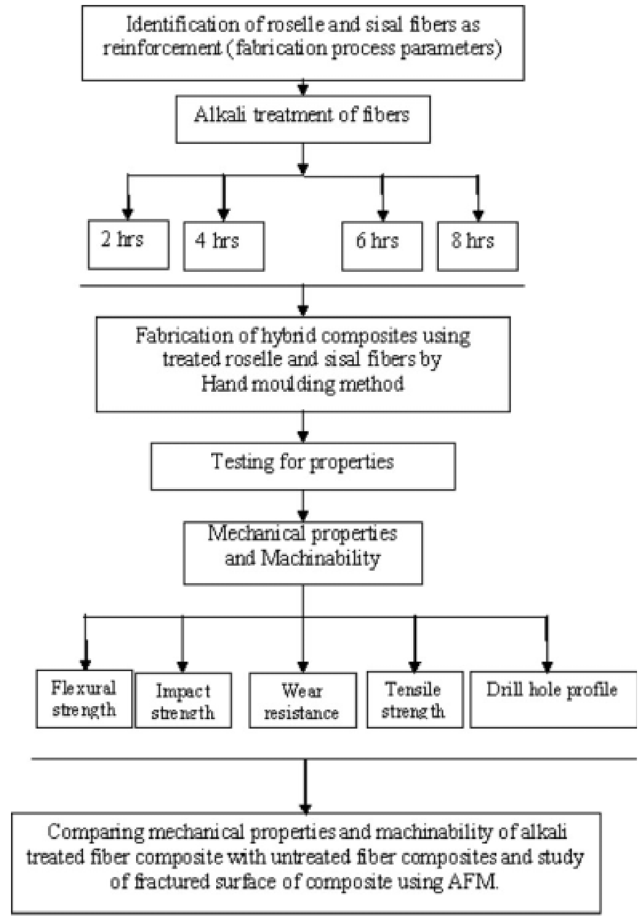

FIG. 1. Methodology of analysis of roselle and sisal hybrid polymer composite material.

EXPERIMENTAL PROCEDURES

Materials and Treatment of Fibers

The roselle and the sisal fiber in dry condition were taken as reinforcement fillers. The matrix material used in this investigation was based on commercially available polyester, Trade name Satyan Polyester supplied by GV Traders.

Because the interface acts as a binder and transfers stress between the matrix and the reinforcing fibers, the fiber-matrix interface plays a major role in determining the quality of a fiber reinforced composite. A good wetting of the fibers with the matrix is obtained by interfacial bonding and the formation of a chemical bond between the fiber surface and the matrix. By imparting hydrophobicity to the fibers by mechanical treatments, surface treatments and chemical treatments, the mechanical properties and environmental performance of composites are developed.

Mwaikambo and Ansell [24] reported that a change in the surface topography of the fibers and their crystallographic structure was achieved by the alkalization or acetylating of fibers. The treatment with sodium hydroxide is before washing of the fibers. The sodium hydroxide opens up the cellulose structure allowing the hydroxyl groups to get ready for the reactions. During washing with sodium hydroxide, the wax, cuticle layer, and part of lignin and hemi cellulose were removed. The major reaction takes place between the hydroxyl groups of cellulose and the chemical used for the surface treatment.

Graft copolyesterization is one of the effective methods of chemical modification of natural fibers. It is a process by which matrix polyesters can be grafted directly onto the fiber surface to provide better fiber and matrix bonding. It involves both etherification and esterification reactions, but it is different from either the mono or the difunctional modification. The reaction is initiated by free radicals of the cellulose molecule. The cellulose is treated with an aqueous solution with selected ions and then followed by exposure to a high-energy radiation. Then the cellulose molecule cracks and radicals are formed. The radical sites of the cellulose are treated with a suitable solution compatible with the polyester, such as vinyl monomer, Gassan and Bledzki [25].

In this contribution, the roselle and sisal fibers were cut to 10 cm of length and were soaked in a 10% NaOH solution at 30°C. The fibers were kept immersed in the alkali solution at different duration of 2, 4, 6, and 8 h. The fibers were then washed with fresh water and again with distilled water to remove any NaOH sticking to the fiber surface and to neutralize with dilute acetic acid at

TABLE 1. Effect of alkali treatment duration on tensile strength.

Fiber content in wt%	Treatment duration in Hrs	Tensile strength in MPa
5	Un-T	30.1
	T-2	32.9
	T-4	34.3
	T-6	38.9
	T-8	36.7
10	Un-T	32.7
	T-2	33.1
	T-4	35.8
	T-6	40.1
	T-8	38.6
20	Un-T	38.9
	T-2	41.3
	T-4	43.6
	T-6	49.1
	T-8	42.7
30	Un-T	41.6
	T-2	42.3
	T-4	47.5
	T-6	50.9
	T-8	46.1

several times. The roselle and sisal fibers were then dried at room temperature for 48 h followed by oven drying at 80°C for 8 h.

Preparation of Specimens

The composites specimens were prepared with four different wt% (5, 10, 20, and 30%) of treated roselle and sisal fibers. The wt% of treated roselle and sisal fibers was calculated on dry basis in order to obtain the correct reading. This fiber contents was chosen to observe the effect of alkali-treated fibers with constant fiber length of 10 cm on the mechanical properties and machinability. The fibers were evenly arranged in a mould measuring 36 cm × 36 cm × 0.3 cm. The resin was degassed before pouring and air bubbles were removed carefully with a roller. The closed mould was kept under pressure for 24 h. The samples were cured at room temperature for 24 h followed by a postcuring in an oven at 80°C. The composites were fabricated in the form of flat sheets of thickness 3 mm. The length, width, and the thickness of each sample were ~15 cm × 2 cm × 3 mm, respectively. The research methodology of this investigation is shown in Fig. 1.

Material Characterization

The roselle and sisal fibers were soaked in 10% NaOH solution with different time period and tested for their weight change. By weighing a fixed amount of dry and cleaned fiber (M_x), the loss of weight was calculated. The amount of fiber was weighed again after soaking in alkali solution (M_y). The percentage weight loss of treated fibers was calculated using following equation: The percentage weight loss is $(M_x - M_y)/M_x \times 100$. It was observed that the fibers were somewhat leached and finer condition. By using gravimetric method, fiber fineness was determined in terms of linear density. The linear density was obtained from the weight of 100 single fibers of 100 mm length each.

Mechanical Properties

For all test, four specimens were tested to get an average value. In tensile test, the composite specimens were measured with Universal Testing Machine. The tensile strength was determined after conditioning for 2 days at 25°C and 50% relative humidity. The tensile strength of the composites was measured in accordance with the ASTM D 638 procedure. The flexural tests were performed using the 3-point bending method according to ASTM D 790. The specimen was freely supported by a beam and the maximum load was applied in the middle of the specimen. In impact test, the strength of the samples was measured using an Izod impact test machine. All test samples were notched. The procedure used for impact testing was ISO 180. The test specimen was supported as a vertical cantilever beam and was broken by a single swing of a pendulum. The pendulum strikes the face of the notch.

Machinability

For machinability, the composite specimens are cut in the rectangular shape of 5.0 cm × 4.0 cm and it is slid against a rotating abrasive wheel. A constant load of 0.5 N was applied during the wear test for all the samples. The weight loss was measured for the specified time intervals such as 4, 8, and 12 min. Besides, the machining properties of the fiber composites are studied in drill hole profile analysis. In this work, the drill hole is made in the composite material by using 8 mm HSS drill bit. The hole profile is analyzed by using profile projector and machine vision inspection system with RAPID I software. The magnified hole is measured for its diameter in different orientation. The holes are selected randomly and studied and then their average diameter is analyzed for various fiber content specimens.

RESULTS AND DISCUSSION

Effect of Fiber Content and Alkali Treatment of Fibers on Mechanical Properties and Machinability of Composite Materials

As shown in Table 1, the maximum improvements were with 6 h-treated fiber composites at 30 wt% fiber content in tensile tests. It is found that the increase of treated fiber content resulted in a significant increase in

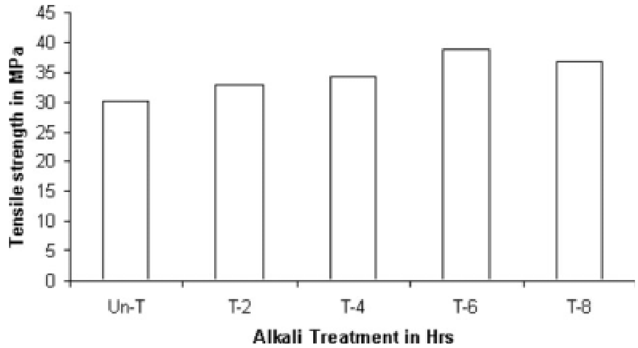

FIG. 2. Effect of fiber content and alkali treatment of fibers on tensile strength of composite material with 5 wt% fiber content.

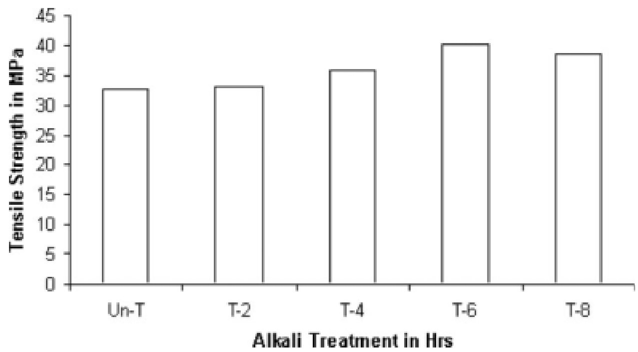

FIG. 3. Effect of fiber content and alkali treatment of fibers on tensile strength of composite material with 10 wt% fiber content.

tensile strength. This behavior was observed in the entire composite with all wt% of fiber content as shown in Figs. 2–5. The composites fabricated with 6 h-treated fibers showed maximum tensile strength at all wt% fiber content. The tensile strength of the composites at 30 wt% fiber content for 6 h treatment was 50.9 MPa, in contrast to 41.6 MPa for the composites with untreated fiber content. An improvement of 22.4% was obtained. The improvements in tensile strength however, were 2%, 14.2% and 11% only for composites fabricated with 2, 4, and 8 h-treated fibers, respectively. The improvement had occurred from 5 wt% fiber content. The rate of change of

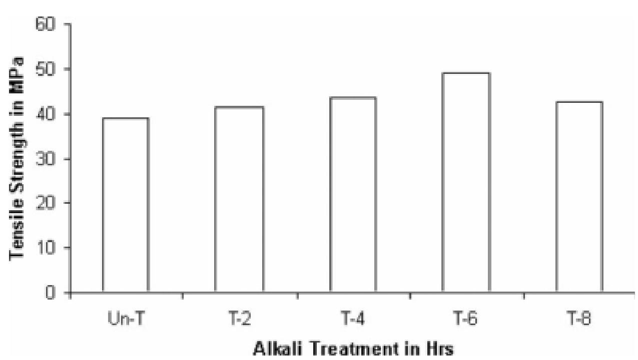

FIG. 4. Effect of fiber content and alkali treatment of fibers on tensile strength of composite material with 20 wt% fiber content.

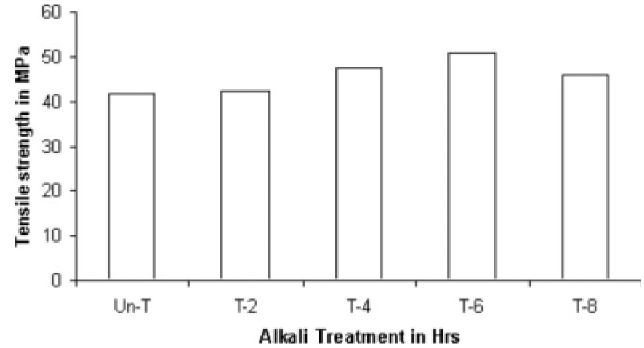

FIG. 5. Effect of fiber content and alkali treatment of fibers on tensile strength of composite material with 30 wt% fiber content.

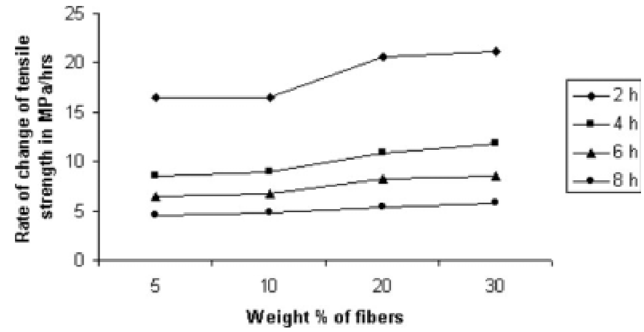

FIG. 6. Variation of the rate of change of tensile strength of composites with varying weight percentage of treated fibers.

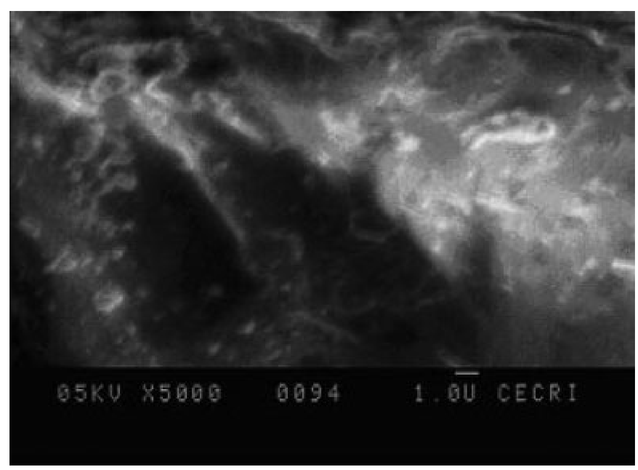

FIG. 7. SEM image of the fractured surface of the hybrid composite with 30 wt% fiber content and 6 h treatment in tensile test.

tensile strength was found to be nonlinear for 2, 4, and 6 h-treated fiber content except 8 h-treated fiber content. The rate of change of improvement in the tensile strength of the composites with all wt% of fiber content treated for 2–8 h is shown in Fig. 6. The scanning electron microscopy (SEM) image of the fractured surface of composite with 30 wt% fiber content and 6 h treatment is shown in Fig. 7.

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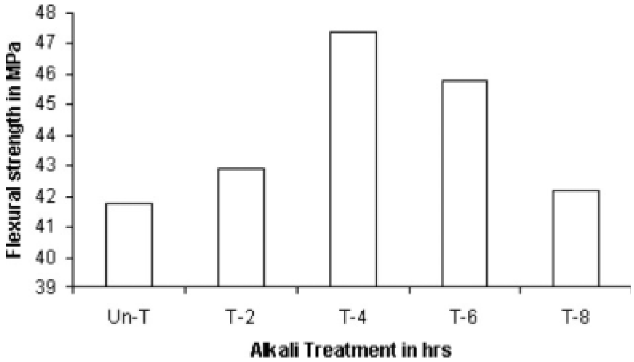

FIG. 8. Effect of fiber content and alkali treatment of fibers on flexural strength of composite material with 5 wt% fiber content.

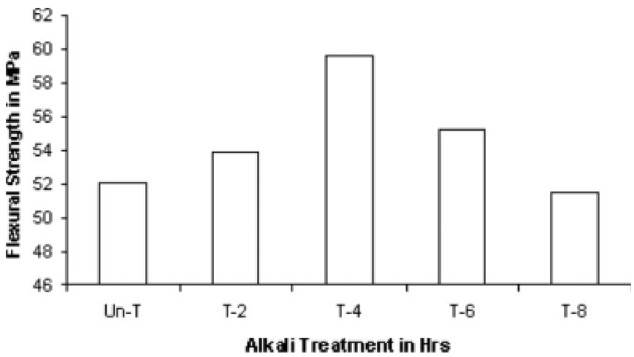

FIG. 9. Effect of fiber content and alkali treatment of fibers on flexural strength of composite material with 10 wt% fiber content.

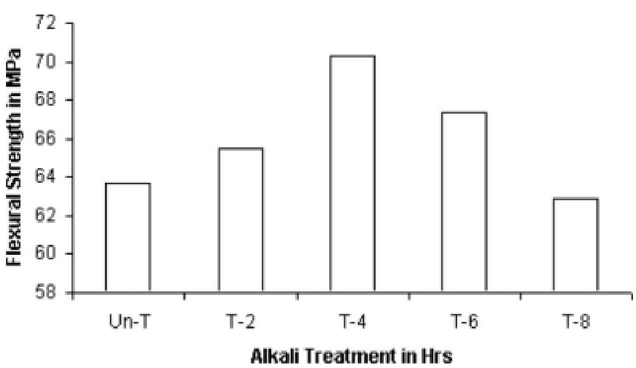

FIG. 10. Effect of fiber content and alkali treatment of fibers on flexural strength of composite material with 20 wt% fiber content.

It is clear from Figs. 8–11 that the flexural strength of the composites is increased with increasing the fiber content and the alkali treatment time. As shown in Table 2, the composites fabricated with 4 h-treated fibers shows the maximum flexural strength at fiber content of 30 wt%. The composites prepared with 4 h-treated fibers showed maximum flexural strength at all wt% fiber content. The flexural strength of the composites at 30 wt% fiber content for 4 h treatment was 88.9 MPa, whereas the flexural strength of the composites with untreated fiber content was 72.7 MPa. The improvement of flexural strength was

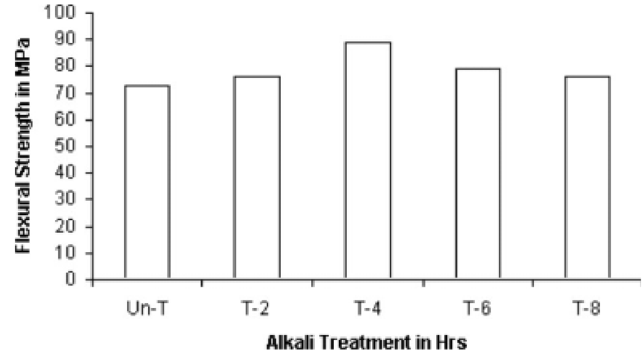

FIG. 11. Effect of fiber content and alkali treatment of fibers on flexural strength of composite material with 30 wt% fiber content.

22.2%. The improvements in flexural strength, however, were 5.1%, 9%, and 4.4% only for composites prepared with 2, 6, and 8 h-treated fiber, respectively. The rate of change of the flexural strength was found to be linear for 2, 6, and 8 h-treated fibers composites except 4 h-treated fiber composites. The rate of change of the flexural strengths of the composites with all wt% of fiber content treated fiber for 2–8 h is shown in Fig. 12. The SEM image of the fractured surface of composite with 30 wt% fiber content and 4 h treatment in the flexural testing is shown in Fig. 13. From Figs. 7 and 13, it is identified that the time taken to break the roselle fibers is larger than that of sisal fibers. It shows that the roselle fibers are more responsible for tensile and flexural strength than sisal fibers.

The impact resistance was not improved by increasing the treated fiber content as shown in Table 3. The highest impact strength (1.39 KJ/m^2) was achieved in the com-

TABLE 2. Effect of alkali treatment of fibers on flexural strength.

Fiber content in wt%	Treatment duration in Hrs	Tensile strength in MPa
5	Un-T	41.8
	T-2	42.9
	T-4	47.4
	T-6	45.8
	T-8	42.2
10	Un-T	52.1
	T-2	53.9
	T-4	59.6
	T-6	55.2
	T-8	51.5
20	Un-T	63.7
	T-2	65.5
	T-4	70.3
	T-6	67.4
	T-8	62.9
30	Un-T	72.7
	T-2	76.4
	T-4	88.9
	T-6	79.2
	T-8	75.9

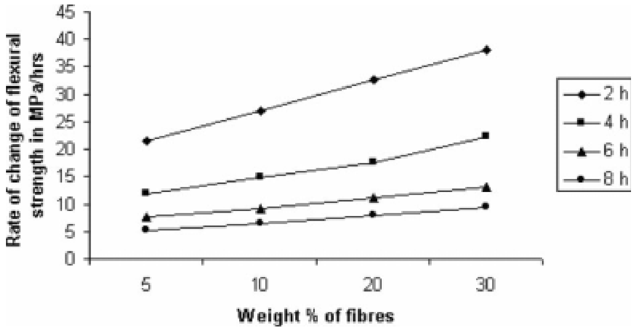

FIG. 12. Variation of the rate of change of flexural strength of composites with varying weight percentage of treated fibers.

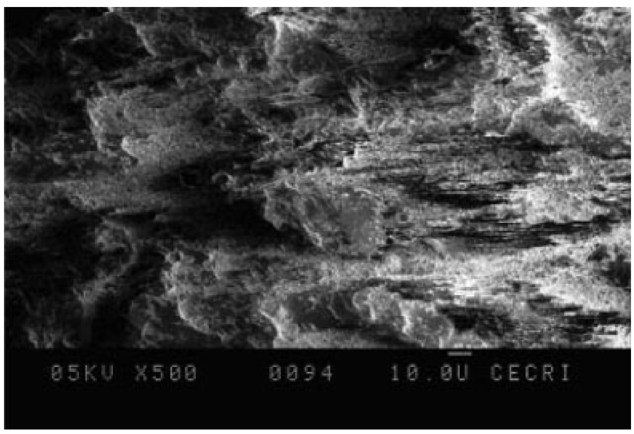

FIG. 13. SEM image of the fractured surface of hybrid composite with 30 wt% fiber content and 4 h treatment in the flexural test.

posite with at 20 wt% fiber content and 2 h treatment, in contrast to lowest impact strength (1.2 KJ/m^2) for 8 h treatment at 5 wt% fiber content as shown in Figs. 14–17.

TABLE 3. Effect of alkali treatment of fibers on impact strength.

Fiber content in wt%	Treatment duration in Hrs	Tensile strength in MPa
5	Un-T	1.37
	T-2	1.3
	T-4	1.29
	T-6	1.23
	T-8	1.2
10	Un-T	1.41
	T-2	1.4
	T-4	1.37
	T-6	1.32
	T-8	1.3
20	Un-T	1.4
	T-2	1.39
	T-4	1.31
	T-6	1.28
	T-8	1.22
30	Un-T	1.38
	T-2	1.35
	T-4	1.3
	T-6	1.27
	T-8	1.25

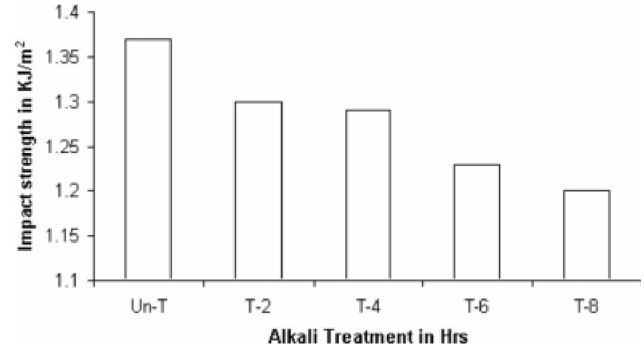

FIG. 14. Effect of fiber content and alkali treatment of fibers on impact strength of composite materials with 5 wt% fiber content.

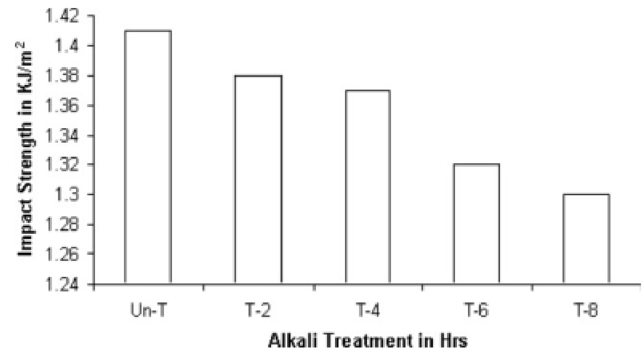

FIG. 15. Effect of fiber content and alkali treatment of fibers on impact strength of composite materials with 10 wt% fiber content.

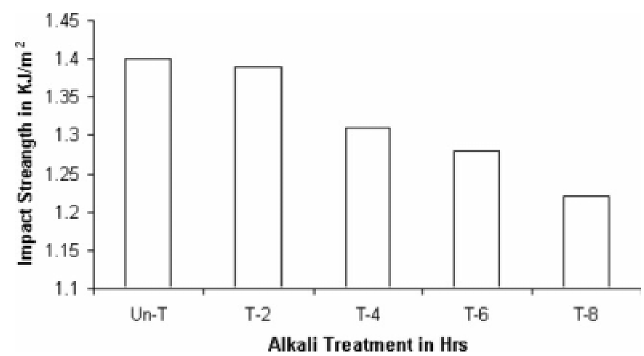

FIG. 16. Effect of fiber content and alkali treatment of fibers on impact strength of composite materials with 20 wt% fiber content.

The SEM image of the fractured surface of composite with 20 wt% fiber content and 2 h treatment in the impact testing is shown in Fig. 18 in which it is identified that the time taken to break the sisal fibers is larger than that of roselle fibers. It shows that the sisal fibers are more responsible for impact strength than roselle fibers. Generally, 6 and 4 h-treated fiber content should exhibit maximum strength properties. Maximum tensile and flexural strength were obtained from the 6 and 4 h-treated fiber content. On application of stress, these treated roselle and sisal fibers suffered breakage due to increased brittleness

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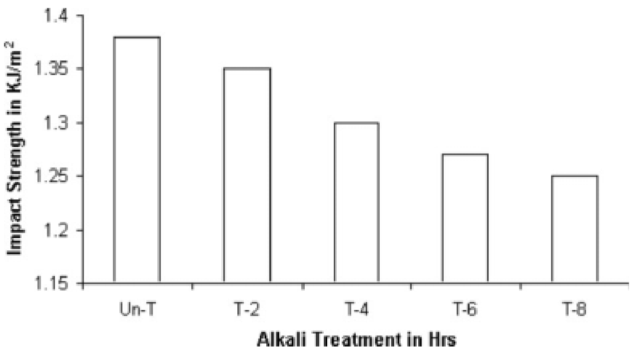

FIG. 17. Effect of fiber content and alkali treatment of fibers on impact strength of composite materials with 30 wt% fiber content.

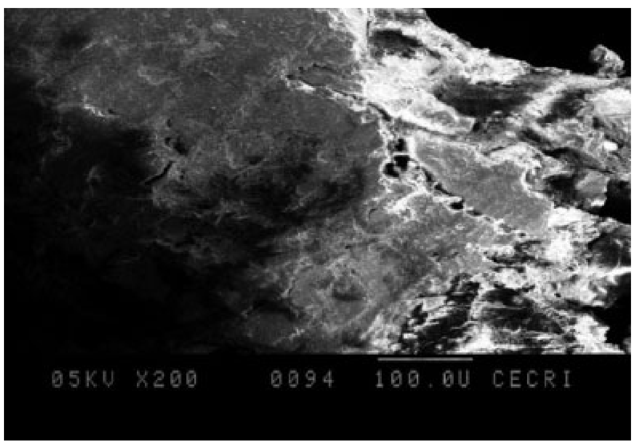

FIG. 18. SEM image of the fractured surface of roselle and sisal hybrid composite with 20 wt% fiber content and 2 h treatment in the mpact test.

and could not take part in effective stress transfer at the interface, thus lowered the strength of the composites. The improvements in mechanical properties such as tensile and flexural were achieved linearly with the increasing of fiber content. The improved properties of the composites fabricated with treated fibers for a longer duration were the result of dissolution of hemi cellulose and development of crystallinity and fibrillation and thus were created superior bonding with polyester resin matrix and resulted into superior properties of the composites.

Wear test was performed on the rectangular specimen by applying a constant load of 0.5 N against a rotating abrasive wheel. The weight loss of the specimen at three intervals such as 4, 8, and 12 min were observed. The effect of wear test time on the weight loss of the composite materials is shown in Figs. 19–22. Gradual weight loss is observed in all the cases. The composites fabricated with 8 h treatment and fiber content of 30 wt% was found to be better wear resistant than 5, 10, and 20 wt% fiber content composite. The wear tests conducted for the alkali-treated composite samples showed superior wear performance when compared with the untreated composite

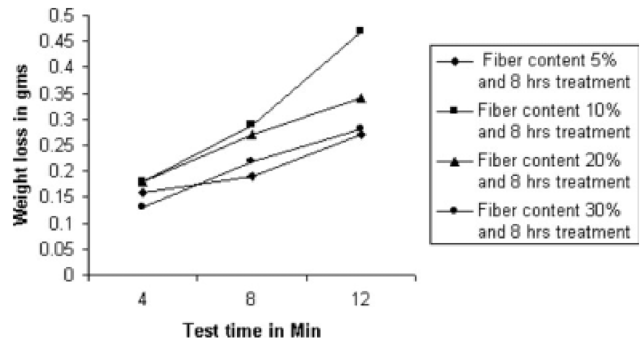

FIG. 19. Effect of wear test time on weight loss for 8 h treatment of fibers.

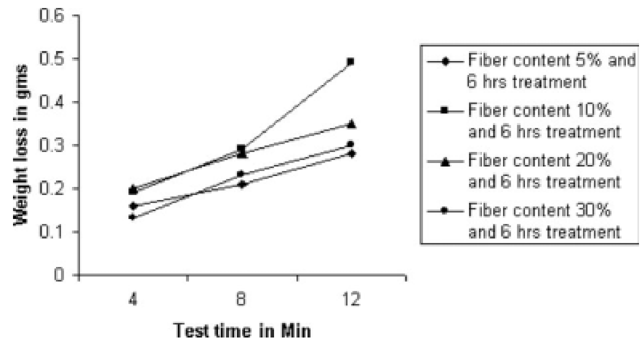

FIG. 20. Effect of wear test time on weight loss for 6 h treatment of fibers.

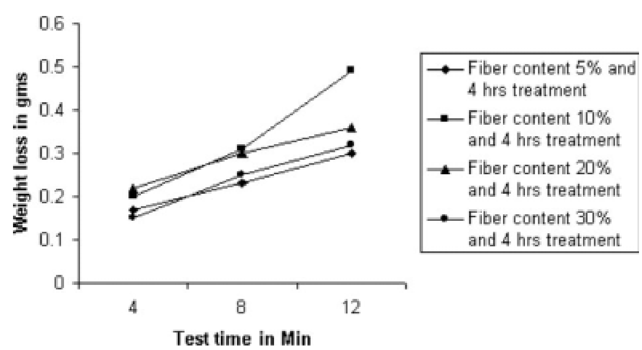

FIG. 21. Effect of wear test time on weight loss for 4 h treatment of fibers.

samples in all wt% of fiber content. The interfacial bonding strength between reinforcement and matrix is increased considerably by removing the moisture of fibers using alkali treatment. The hole profile of the treated roselle and sisal fibers hybrid polyester composites varies drastically due to the tensile nature of treated fibers as shown in Figs. 23–26. The composite with 5 wt% fiber content and 2 h treatment re-bounces in smaller amount. The composite specimen with 30 wt% fiber content and 8 h treatment shows better dimensional accuracy than other fiber content and treatment composites. CCD image of drill hole profile of hybrid composite is shown in Fig. 27.

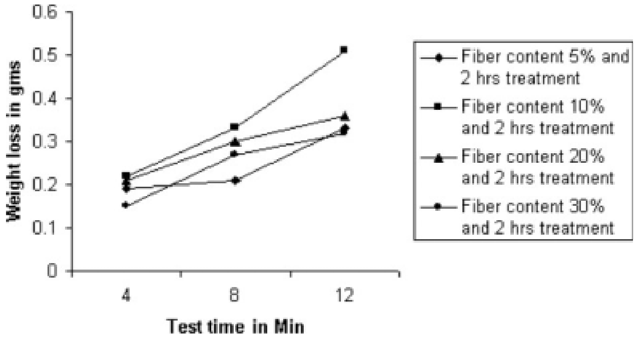

FIG. 22. Effect of wear test time on weight loss for the composite with 2 h treatment of fibers.

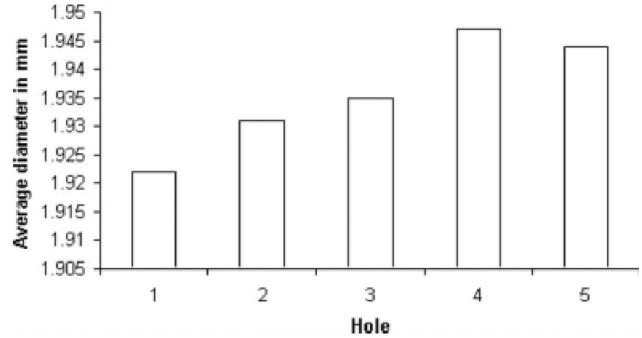

FIG. 25. Drill hole profile analysis of the hybrid composite with 20 wt% fiber content and 8 h treatment.

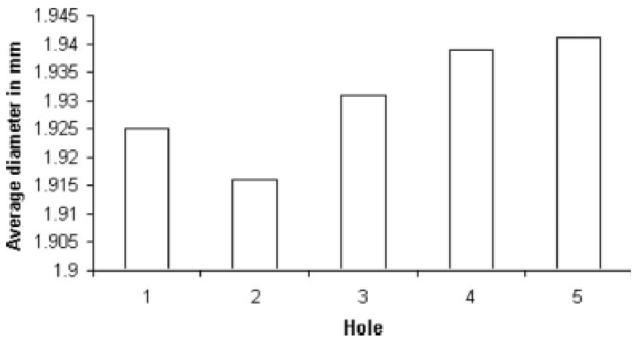

FIG. 23. Drill hole profile analysis of the hybrid composite with 5 wt% fiber content and 8 h treatment.

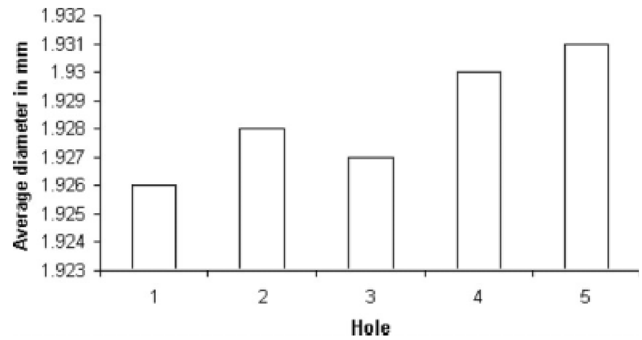

FIG. 26. Drill hole profile analysis of the hybrid composite with 30 wt% fiber content and 8 h treatment.

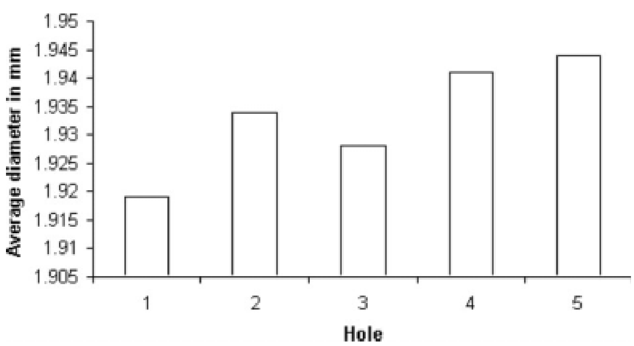

FIG. 24. Drill hole profile analysis of the hybrid composite with 10 wt% fiber content and 8 h treatment.

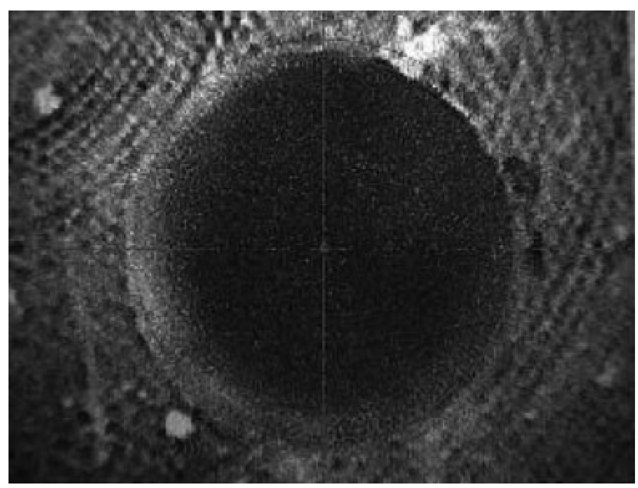

FIG. 27. Machine vision image of drill hole profile of hybrid composite specimen with 30 wt% fiber content and 8 h treatment.

CONCLUSIONS

It was observed that the composites with alkali-treated fibers showed the highest mechanical properties such as tensile and flexural strength except impact strength. The tensile strength improved by 22.4% in 6 h treatment and 30 wt% fiber content composite, whereas the flexural strength improved by 22.2% in 4 h treatment and 30 wt% fiber content composite. The rate of change of tensile strength was found to be linear for 8 h treatment. The rate of change of flexural strength was found to be linear for 2, 6, and 8 h treatment. The highest impact strength was achieved in 2 h treatment and fiber content of 20 wt% composite, in contrast to the lowest strength for 8 h treatment and fiber content of 5 wt%. The alkali treatment time of 6 and 4 h at 30 wt% was optimal to get maximum tensile and flexural strength of the composites reinforced with roselle and sisal fibers. In impact test, the alkali treatment of 2 h at 20 wt% was optimal to get maximum impact strength. In wear test, the composite specimen with alkali-treated fiber content gives a better wear resist-

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ance than untreated fiber content composite. In drill hole analysis, the composite specimens with 30 wt% fiber content and 8 h treatment show better dimensional accuracy than the composite specimen with other fiber content and treatments. The mechanical properties and machinability of the roselle and the sisal fiber hybrid polyester composite were improved significantly after alkali treatment.

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