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السلوك الميكانيكية والنحر لمركب الفينيل استر الغير معالج والمقوى بالألياف

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الخلاصـة

في هذا البحث، تأثير المعالجة بالمرسرة أو القلوية (هيدروكسيد الصوديوم) على السلوك الميكانيكي والنحر لمركب الفينيل استر (CRFRV) والمقوى بالألياف تم دراستها بوصفها وظيفة من وظائف محتوى الألياف. وأظهرت النتائج بأن الشد والمرونة وتأثير قوة الصدمة لمركبات الألياف غير المعالجة أو المعالجة زادت مع زيادة محتوى الألياف لتصل إلى نسبة 40٪ من الوزن ثم تراجعت. ولوحظ بأن أفضل محتوى الألياف (نسبة الوزن) لخصائص الميكانيكية هي 40٪ من الوزن لكل من مركبات الألياف المعالجة أو الغير المعالجة. ومقاومة التآكل لمركب الألياف المعالجة كانت أفضل من المواد المركبة غير المعالجة.

Mechanical and wear behaviors of untreated and alkali treated Roselle fiber-reinforced vinyl ester composite

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ABSTRACT

In the present communication, the effect of mercerization or alkali (NaOH) treatment on mechanical and wear behavior of chopped Roselle fiber-reinforced vinyl ester (CRFRV) composite have been studied as the function of fiber content. The results show that the tensile, flexural and impact strength values of untreated and treated fiber composites increased with increasing fiber content up to 40 wt% and then dropped. It was observed that the better fiber content (weight percentage) for better mechanical properties is 40 wt% at both the untreated and treated fiber composites. The wear resistance of the composites fabricated with treated Roselle fibers was found to be of better wear resistance composites fabricated by untreated fibers.

Keywords: Composite material; mechanical properties; mercerization treatment; natural fiber; wear resistance.

INTRODUCTION

In recent years, natural cellulose fiber-reinforced polymer composites have received widespread attention among material engineers and researchers to replace synthetic fibers in various applications, like structural, aerospace, automotive etc, due to advantages such as abundant availability of renewable resources, nontoxicity, biodegradability, nonabrasiveness to processing equipments, low cost, low density and good mechanical properties (Athijayamani *et al.,* 2010; Shen *et al.,* 2012). Despite the various advantages, these fibers had a number of disadvantages, when used as reinforcements, such as lower impact strength, higher moisture absorption and poor thermal stability (Athijayamani *et al.,* 2009; Ghasemi & Farsi, 2010; Robertson *et al.,* 2013). However, the applications of natural fibers as reinforcement for polymer composites were reduced by the hydrophilic nature of natural fibers. The poor moisture resistance and poor wet ability of natural fibers with hydrophobic polymers, affected the interface bonding between the fiber and matrix (Rana *et al.,* 1997). By using a suitable surface treatment method, the resistance to moisture absorption and the better mechanical properties of natural fibers had been achieved (Bisanda & Ansell, 1981). To reduce moisture sensitivity and biological decay and to optimize the properties of the fiber matrix interface, the natural fibers used in polymer composite materials were modified by chemical and physical methods (Gassan & Bledzki, 1999). By treating the fibers with suitable chemicals, the reinforcing efficiency of the fibers in the composite and the interfacial adhesion between fibers and most polymer matrices can be solved (Li *et al.,* 2000; Martins & Joekes, 2003). Among the various natural fibers, the Roselle fibers are one of the most important fibers used in polymer composites recently. Roselle (*Hibiscus sabdariffa L*) plant is cultivated to safeguard other plants in the Southern Region of Tamilnadu, India. The fiber from the Roselle plant is in great demand and also used for various applications. Keeping all the above in view, in this study, chopped Roselle fibers were treated with alkali (NaOH) in order to obtain better adhesion of these fibers with the vinyl ester resin. The effects of treated fiber content on mechanical properties such as tensile, flexural and impact of composites were observed. In addition to that the wear resistance of the composites made of treated and untreated fibers was tested. Fractographic study of the treated, untreated fibers and fractured surface of treated fiber composites have also been carried out using SEM.

EXPERIMENTAL PROCEDURES

Materials

Roselle fibers were cut into 15 mm length and used as reinforcements in the commercially available vinyl ester resin (Satyen Polymers Ltd., Bangalore). Methyl ethyl ketone peroxide, cobalt 6 % naphthenate and *N–N* dimethyl aniline were used as an accelerator, catalyst, and promoter respectively. All chemical agents used in this study were supplied by GVR Enterprises, Madurai, Tmailnadu, India.

Mercerization or alkali treatment of Roselle fibers

Mercerization treatment is carried out on natural cellulose fibers to activate the OH groups of the cellulose and lignin in the fiber and to change the surface topography of the fibers and their crystallographic structure. In the present study, the chopped Roselle fibers with an average length of 15 mm were immersed in 10% NaOH solution for 2 hours at room temperature and then washed with running water and allowed to dry at room temperature for 48 hours. The major reaction takes place between the hydroxyl groups of cellulose and the chemical used for the surface treatment, which is shown by the following equation (Sreenivasan *et al.,* 1996):

$$\text{Fiber} - \text{OH} + \text{NaOH} \longrightarrow \text{Fiber} - \text{O} - \text{Na} + H_2O \tag{1}$$

Preparation of composites

The chopped Roselle fibers were evenly spread in a mould measuring 150 mm x 150 mm x 3 mm using a mechanical roller. The accelerators and promoters were mixed with resin and the air bubbles were removed by degassing. The Catalyst was added and poured into the mould. Then, the mould was closed and kept under atmospheric pressure for 48 h. Composite plates were further cured by post curing in an oven at 80°C.

Characterization of composite specimens

Composite specimens were cut as per ASTM and ISO standards. The tensile strength of the composites was measured with a computerized FIE (Fuel Instruments & Engineers Pvt. Ltd.) universal testing machine in accordance with the ASTM D 638-10 (ASTM D 638-10, 2010) procedure at a crosshead speed of 2 mm/min. The flexural tests were performed on the same machine, using the 3-point bending fixture according to ASTM D790 -10 (ASTM D 790-10, 2010) with the crosshead speed of 2 mm/min. The impact strength of the samples was measured using an impact test machine as per ISO 180:2000 (ISO 180:2000, 2000). For statistical purpose, a total of five samples of each case were tested and the average values were recorded.

The fractographic studies were carried out in detail on the fracture surfaces of composites using SEM (Hitachi S-3000N).

Wear testing

In the present study, the two-body abrasive wear test was carried out according to ASTM G99-04a (ASTM G99-04a, 2004) standards using Pin-on-Disc machine. The rectangular specimen of 10 mm x 10 mm x 3 mm was sliding against a rotating abrasive wheel. Three different load conditions (5, 10, and 20 N) were applied during the wear test for all the samples. The weight loss was measured at a constant sliding distance of 27 meters. This conventional method was selected as the wear study was restricted only to compare the wear performance among the samples prepared with treated and untreated fibers.

RESULTS AND DISCUSSION

Effect of fiber content on mechanical properties of untreated fiber composites

The effect of fiber content on the tensile strength and modulus is given in Table 1. From Table 1 it is observed that composite reached the tensile strength and modulus of the cured un-reinforced resin sample at 20 wt%. The tensile strength increased continuously from 10 wt% to 40 wt% and then dropped. The maximum tensile strength was observed at 40 wt% composite, which is 44.8 % higher than the cured neat resin sample. The tensile strength value of 30 wt% composite was higher than that of 50 wt% composite. The tensile modulus values increased linearly with fiber content. 50 wt% composite showed the maximum tensile modulus value.

The flexural strength of the vinyl ester composite increased with Roselle fiber content as given in Table 1. The maximum flexural strength was obtained in composite having fiber content of 40 wt%, which is 52.17% higher than cured un-reinforced resin sample. 50 wt% of composite showed the flexural strength of 58.4 MPa, which was lower than that of 35 wt% composite. The flexural modulus values also showed the same trend like in tensile modulus values. 50 wt% composite showed a maximum flexural modulus of 1609.8MPa, which was 29.99% higher than the cured neat resin sample.

Impact strength or energy observed by composites due to impact loading for various fiber weights was obtained by impact testing machine as given in Table 1. Composites reached the impact strength of the cured neat vinyl ester resin sample at 20 wt%. The impact strength values increased continuously up to 40 wt% and then dropped. An improvement of 45.28% was achieved due to the reinforcement of 40 wt% fibers with the vinyl ester resin matrix.

It has been observed that the strength values of untreated Roselle fiber-vinyl ester composite increased with the increasing fiber content up to 40 wt%, after that they started to decrease. It could be attributed to the fact that the increase of the fiber content above a certain limit lead to the poor wetting of the fiber by resin, which resulted in loss of the mechanical properties. In composites having the high fiber content, the aggregation of fibers took place instead of dispersing; therefore resin could not wet the fibers properly. It was also clearly observed that the fiber pull-out and weak interfacial bonding were identified on the fracture surface of untreated CRFRV composite. This may be attributed to the low adhesion between the Roselle fiber surfaces and vinyl ester resin. Hence, the mechanical properties of untreated fiber composite were low.

Table 1. Average mechanical properties of CRFRV composite.

Fiber content (wt%)	Tensile strength (MPa)	Tensile modulus (MPa)	Flexural strength (MPa)	Flexural modulus (MPa)	Impact strength in (kJ/m²)
0	29.6	1088.63	33.1	1127.3	0.87
10	19.8	989.7	21.4	1105.7	0.82
15	25.5	1035.2	28.8	1134.3	0.86
20	31.2	1063.5	35.5	1171.8	0.91
25	37.4	1089.8	43.1	1212.7	1.12
30	42.8	1124.4	52.4	1249.5	1.25
35	47.5	1155.3	60.7	1271.3	1.37
40	53.7	1207.5	68.2	1319.1	1.59
45	48.6	1262.2	61.5	1287.4	1.52
50	39.2	1321.4	58.4	1249.8	1.48

Effect of mercerization treatment on mechanical properties of composites

The increase of treated fiber content resulted in a significant increase in tensile strength and modulus composites. This behavior was observed in the entire composite with all weight percentages as shown in Figure 1. Composite reinforced with treated fibers reached the tensile strength of unreinforced composite specimen at 15 wt%. The high tensile strength value was obtained at 40 wt% composite. 62.86 % of the improvement was achieved in tensile strength, when compared with the neat resin composite specimen. The tensile modulus values increased linearly from 10 wt% to 50 wt%. 40wt% composite shows the tensile modulus value of 1469.6 MPa, which is 25.92 % higher than the cured neat resin sample.

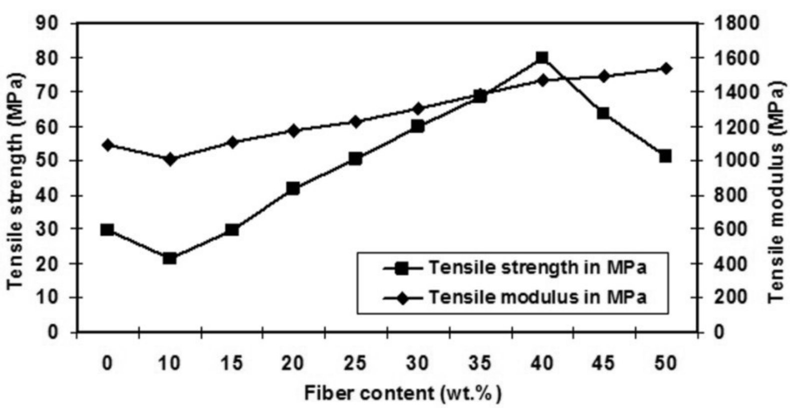


Fig. 1. Effect of mercerization treatment on the tensile properties of CRFRV composites.

It is clear from Figure 2 that the flexural properties of composites increased with increase of the treated fiber content. The flexural strength values continuously increased up to 40 wt% like in tensile strength values and then dropped. Composites having the fiber content of 35 and 45 wt% show almost same value. 50 wt% composite shows the flexural strength value of 80.9 MPa, which is 15.9 % lower than 40 wt% composite.

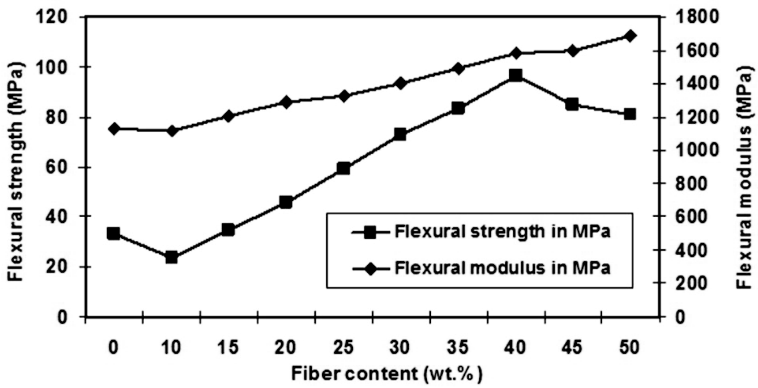


Fig. 2. Effect of mercerization treatment on the flexural properties of CRFRV composite.

The impact strength of composite was improved by increasing the treated fiber content as shown in Figure 3. The high impact strength was identified at 40 wt% fiber content, after that, it decreased. Composite reaches the impact strength of a neat resin specimen at 15 wt%. An improvement of 56.1% was achieved in 40 wt% composite, when compared with the neat resin composite specimen. The impact strength value of 50 wt% composite was 1.73 KJ/m^2, which is 12.63 % lower than 40 wt% composite. After careful study of the CRFRV composite at untreated and treated conditions, it was observed that the better fiber content for the best combination of mechanical properties is 40 wt%. This better fiber content may vary with the nature of fiber, matrix, fiber length, fiber content and degree of fiber matrix adhesion etc.

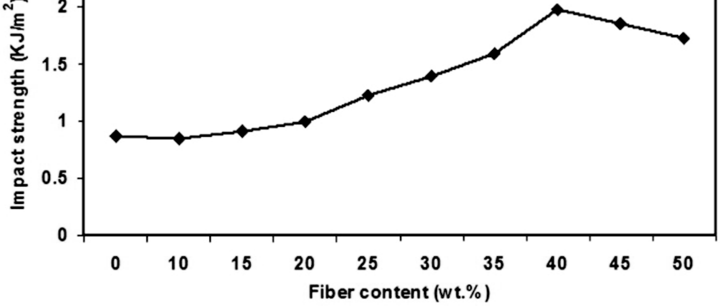
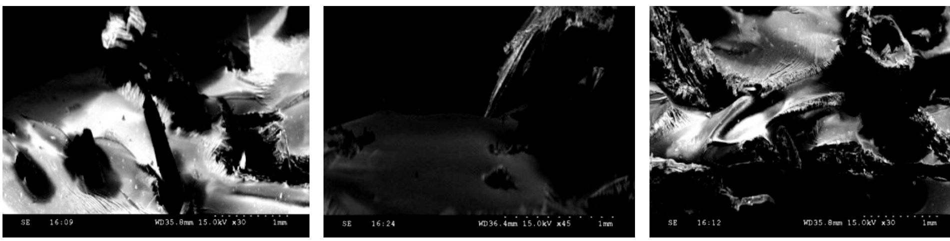


Fig. 3. Effect of mercerization treatment on the impact strength of CRFRV composite.

Figure 4a is an evidence for above explanation and shows the less fiber pull-out and brittle fracture in the fractured surfaces of the composite specimen after the tensile test. Figure 4b shows the SEM image of the fracture surface of composite after the flexural test. Here also, the less fiber pull-out and the brittle fracture on the fracture surface were identified. Figure 4c shows the SEM image of the fracture surface of composite after impact test. It can be seen that a good bonding strength between the fiber and matrix is identified. Here also, the less fiber pull-out and the brittle fracture of composite were identified.



(a) (b) (c)

Fig. 4. SEM images of fractured surfaces of treated fiber composites (40 wt%) after the (a) tensile test, (b) flexural test, and (c) impact test.

Percentage of improvement in mechanical properties

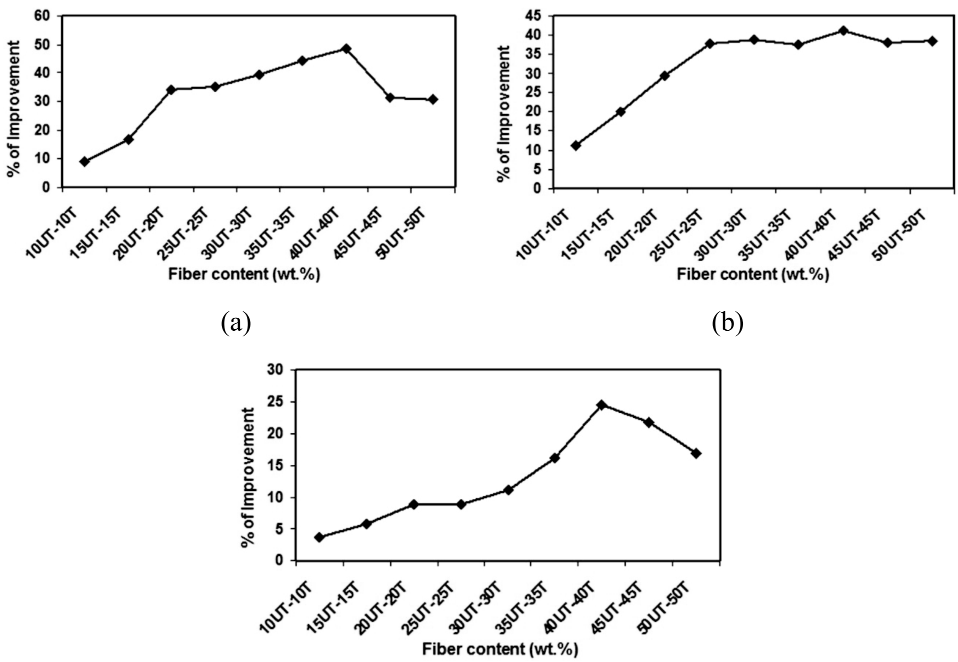
Percentage of improvement in mechanical properties due to the mercerization treatment of Roselle fibers was determined by the strength of the samples fabricated by treated fibers relative to the strength of untreated samples as follows:

$$\text{Percentage of improvement } (\%\ IM) = \frac{\sigma_{tr} - \sigma_{ut}}{\sigma_{ut}} \times 100 \quad (2)$$

where σ_{tr} and σ_{ut} are the strength of the composites fabricated by treated fiber and untreated fiber.

The maximum percentage of improvement in all mechanical properties was obtained for composites having 40 wt% of the treated fiber, when compared with composites reinforced with untreated fibers (Figure 5a). The percentage of improvement had occurred from 10 wt% of fiber content. 48.4 % of improvement was achieved in tensile strength, when comparing the composite having 40 wt% treated fiber with respective untreated fiber composite. This is the highest level of improvement in tensile strength. 45 and 50 wt% of composites almost show the same level of improvement. Composite having the fiber content of 40 wt% shows the highest level of improvement in flexural strength, when compared with respective untreated fiber composites (Figure 5b). 40 wt% of composite shows the maximum level of improvement in impact strength (Figure 5c). 20 and 25 wt% of composites show the same level of improvement.

Finally, it was observed that the mechanical properties increased with mercerization treated Roselle fibers. The reasons for that being, the impurities on the Roselle fibers are removed during mercerization treatment, so the level of interfacial adhesion increased between the fiber and the matrix.



(a) (b)

(c)

Fig. 5. Percentage of improvement in (a) tensile strength, (b) flexural strength, and (c) impact strength when comparing the untreated and treated fiber composites

Wear resistance

Composite specimen for wear test was cut from the prepared composite plate with a size of 10 mm x 10 mm x 3 mm. Wear test was performed by applying a constant sliding distance of 27 meters on composite specimen against a rotating abrasive wheel (abrasive paper with 400 grade). After each 10 minutes run, composite test pieces were removed from the machine and measured to determine the loss in weight. The weight loss of the specimen, at three different load conditions such as 5, 10 and 20 N were measured. The weight loss of each test was calculated by following equation:

$$\text{Weight loss } (W_l) = (W_a - W_b) \quad (3)$$

where W_l is the weight loss in grams and W_a and W_b are the weight of the sample after and before the test in grams. Abrasive wear rate and the specific abrasive wear rate are calculated by determining the weight loss after each test by using the following equations:

$$WR = \frac{W_l(grams)}{\rho\left(\frac{grams}{cm^3}\right)\times S_d(m)} \quad (4)$$

$$SWR = \frac{W_l(grams)}{\rho\left(\frac{grams}{cm^3}\right)\times S_d(m)\times F(N)} \quad (5)$$

where *WR* is the wear rate in cm^3/m, *SWR* is the specific wear rate in cm^3/Nm, W_l is the weight loss in grams, ρ is the density of composite sample in grams/cm^3, S_d is the sliding distance in *m*, and *F* is the applied load in *N*.

Table 2 depicts the effect of applied load conditions on the weight loss of composite materials prepared by untreated and treated fibers. A gradual weight loss was observed in all the cases.

It was observed that the weight loss in all composites increased, when increasing the load. The weight loss decreased linearly with the addition of untreated Roselle fibers up to 30 wt% in all load conditions, after that it increased. But, in case of treated fiber composites, the weight loss decreased linearly up to 40 wt% in all load conditions and then increased. The maximum level of weight losses is identified in 20 N at both the cases. Irrespective of load conditions, the weight loss increased due to the agglomeration of fiber in case of composites having higher wt% in both cases. It was also observed that the treated Roselle fibers are more effective in improving the wear resistance of vinyl ester.

Table 2. The variation of the weight loss in CRFRV composite at various applied load conditions (5 N, 10 N, and 20 N).

Fiber content (wt%)	Weight loss (grams) at 5 N		Weight loss (grams) at 15 N		Weight loss (grams) at 20 N	
	Untreated	Treated	Untreated	Treated	Untreated	Treated
10	0.39	0.37	0.42	0.39	0.43	0.41
15	0.37	0.35	0.39	0.37	0.41	0.39
20	0.34	0.32	0.36	0.34	0.39	0.37
25	0.31	0.29	0.33	0.32	0.37	0.35
30	0.28	0.27	0.3	0.28	0.35	0.33
35	0.3	0.23	0.32	0.26	0.38	0.36
40	0.32	0.21	0.34	0.25	0.39	0.37
45	0.34	0.23	0.36	0.28	0.4	0.4
50	0.36	0.25	0.38	0.32	0.41	0.42

The variation of the specific wear rate (SWR) with three different loading conditions for constant sliding distance of 27 meters is given in Table 3. It was observed that for all load conditions the specific wear rate decreases with the increase of fiber content up to 30 wt% for composite prepared with untreated Roselle fibers. It was up to 40 wt% for treated fiber composites. The highest specific wear rates were identified in the composite having the fiber content of 10 wt% for both the cases. It was also observed that the material removal is less under low load conditions (5 and 10 N) because of less penetration of abrasive particle on the surface of composite. By applying higher load, the abrasive particles penetrate deeper into the surface of composites and deeper grooves are formed, which causes the higher wear rate.

It may be concluded that composites fabricated using treated fibers are found to be of better wear resistance compared to untreated fiber composites. The reduction in the wear resistance of composites at untreated conditions could also be expected due to the poor mechanical interlocking between the fiber and matrix. Composites prepared using untreated fibers absorbed high level of moisture compared to treated fiber composites. The moisture absorbed by composite is damaging the fiber structure in composite. This is also the reason for the reduction in wear resistance of composites at untreated condition.

Table 3. The variation of SWR in CRFRV composite at various applied load conditions (5 N, 10 N, and 20 N).

Fiber content (wt%)	SWR (cm^3/Nm) at 5 N		SWR (cm^3/Nm) at 15 N		SWR (cm^3/Nm) at 20 N	
	Untreated	Treated	Untreated	Treated	Untreated	Treated
10	0.0025	0.0024	0.0027	0.0025	0.0027	0.0026
15	0.0023	0.0022	0.0025	0.0023	0.0026	0.0025
20	0.0021	0.002	0.0023	0.0021	0.0024	0.0023
25	0.0019	0.0018	0.0021	0.002	0.0023	0.0022
30	0.0017	0.0017	0.0019	0.0017	0.0022	0.002
35	0.0018	0.0014	0.002	0.0016	0.0023	0.0022
40	0.002	0.0013	0.0021	0.0015	0.0024	0.0023
45	0.0021	0.0014	0.0022	0.0017	0.0024	0.0024
50	0.0022	0.0015	0.0023	0.0019	0.0025	0.0025

SWR = Specific wear rate

CONCLUSIONS AND FUTURE RESEARCH

From this study, it was observed that composites prepared by treated Roselle fibers show improved mechanical properties compared to composites prepared by untreated Roselle fibers. The tensile, flexural and impact strength of the untreated and treated fiber composites increased with increasing fiber content up to 40 wt% and then dropped. It was identified that the better fiber content for the better mechanical properties is 40 wt% in both the cases. The weight loss decreased linearly with the addition of untreated Roselle fibers up to 30 wt%. But, in case of treated fiber composite, the weight loss decreased linearly up to 40 wt% in all load conditions and then increased. The wear test results show that composites fabricated using treated Roselle fibers are found to be of better wear resistance compared to untreated fiber composites. Finally, it was concluded that the mercerization treatment with this much of concentration and treatment duration have a significant effect on mechanical and wear properties of CRFRV composites. In future research, we plan to study the effect of coupling agent and impact modifier on mechanical and machining (drilling and turning) behaviors of chopped Roselle fiber-reinforced vinyl ester composites.

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