FATIGUE STUDIES OF UNTREATED AND NaOH-CLAY TREATED SISAL FIBER POLYPROPYLENE AND EPOXY COMPOSITES

K. Kanny*, T. P. Mohan

Composites Research Group, Department of Mechanical Engineering, Durban University of Technology, Durban – South Africa
** kannyk@dut.ac.za*

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Abstract
This paper presents the effect of moisture on fatigue and dynamic mechanical properties of untreated and NaOH-clay treated sisal fiber reinforced epoxy and polypropylene (PP) composites. Sisal fibers were reinforced in epoxy and PP composites at the loading level of 0 wt.% to 50 wt.% and result shows that due to water content, the amount of reduction of fatigue life cycle, glass transition temperature (T_g) and storage modulus of epoxy sisal fiber composites was higher than that of polypropylene sisal fiber composites. About 10^2 times fatigue life cycles were reduced due to the moisture effect in epoxy-sisal fiber composites; however, this difference in reduction is minimal in polypropylene-sisal fiber composites. Among the composite series, improved properties are observed in NaOH-clay treated sisal fiber composites.

1 Introduction
The natural fiber reinforced composites are finding increasing engineering applications due to their environmental friendly, low cost and comparable thermal and mechanical properties in certain applications to glass fiber reinforced polymeric composites. In particularly, the chemical treatment of natural fibers has resulted in increased applications with enhanced mechanical and thermal properties. Even though large number of natural fibers, namely, sisal, kenaf, bamboo, kemp, coir, jute, flax, etc based composites were studied, some of the properties of natural fibers, namely, fatigue, creep, visco-elastic and time-dependent behavior of these fibers are not well understood. Also their high level of moisture uptake and debonding characteristics are the primary concern of the natural fiber reinforced composite materials [1-4].

In this paper we examined the fatigue (tension-tension) characteristics of sisal fiber reinforced composites and the influence of moisture content on fatigue. Two polymer matrices were chosen for this study, namely, thermoset epoxy (E) polymer and thermoplastic polypropylene (PP) polymer. Untreatead and chemically treated (NaOH-clay) sisal fiber from 0 wt.% to 50 wt.% were reinforced in polymer matrix.

2 Materials and testing methods

2.1 Raw materials

PP pellets of melting point 168°C were procured from Chempro, South Africa. Diglycidyl ether of bisphenol-A (DGEBA) resin and amine based hardener (LR 281) were purchased from CIBA, Basle - Switzerland. Untreated Na^+ montmorillonite (MMT) clay was purchased from Southern Clay Products, USA supplied under the trade name Na^+-Cloisite and NaOH pellets were purchased from Merck Chemicals-SA. Sisal in the form of short fibers (length 2.5 cm and diameter varying from 0.1 to 1 mm) was procured from AMT Composites, Durban-South Africa.

2.2 Fiber treatment and Composite preparation

In NaOH-clay treatment of sisal fiber, 40g of NaOH was added in 100 ml distilled water and agitated in the magnetic stirrer at 60°C for few minutes and then 5g of sisal fiber was placed into the solution with weight of clay twice to that of sisal fiber. The fiber-NaOH solution was agitated using thermally controlled magnetic stirrer at 60°C for 4 hours. The treated fiber was taken out and dried in an oven at 60°C for 4 hours.

10 wt.% to 50 wt.% of treated and untreated sisal fibers were reinforced in PP and epoxy polymer. In preparation of PP-sisal fiber composites, the PP and fiber mixture were mixed by melt extrusion method. In this method, the polypropylene pellets and sisal fibers (0 to 50 wt.%) were combined in a Reiffenhaeuser single screw extruder. The extruder has a 40 mm diameter single rotating screw with a length/diameter ratio (L/D) of 24 and driven by a 7.5 kW motor. Extruder has three heating zones along the length of the screw as follows: Zone 1 (Hopper or pellet loading end), Zone 2 (centre region of screw) and Zone 3 (extrusion end). The melt mixing conditions of all the composite specimens were kept at constant temperature of 190°C (Zone 1), 230°C (Zone 2) and 230°C (Zone 3), and the screw speed of 80 rpm.

Epoxy - sisal fiber composites were manufactured using resin casting method. In this method, 300 ml of epoxy resin was taken in a beaker and sisal fiber (0 wt.% to 50 wt.% to the weight of uncured epoxy resin) was added to the resin and the solution was mixed well using magnetic stirrer. Thereafter 30 wt.% of hardener (equivalent to epoxy weight) was added into the epoxy-fiber solution and the mixture was gently stirred using a glass rod. This mixture was cast by resin casting method. The resin casting was performed in a closed glass molds. Two 30 cm X 30 cm X 3 mm square glass plates were used as moulds to fabricate casting. The 3 mm rubber gasket was used between the glass plates and fastened by clips. The rubber gasket covers the three sides of the mould and the remaining one side is sued to pour the resin-clay mixture through the runner made in the mould. The wax was used as a mold release agent, and was applied at faces of glass plates and rubber gasket for easy removal of cast products. The cured epoxy resin was removed after two days.

3.3 Characterization and testing

Fatigue test was performed on the sample using servo hydraulic MTS machine. The test was carried out using tension-tension mode. Throughout the test, the static stress (S) of 90%, 60% and 30% of tensile strength of the respective sample was used and the fatigue life cycle was represented by plotting S-log N where N represents the number of cycle to failure. The stress ratio and the frequency were kept at 0.1 and 1 Hz respectively throughout the test and for the entire test specimens. Three samples were tested for each series and the average S and N values were considered. The fracture surface of the fatigue samples was examined using Zeiss Environmental Scanning Electron Microscopy (ZEISS EVO-HD 15) operating at the 20 kV with secondary electron image.

Water uptake property of composite (sisal fiber reinforced PP and epoxy) sample was carried out by making sample dimension of 2.5 cm X 2.5 cm X 0.3 cm. Three specimen of each composite series were placed in a distilled water bath medium and left until equilibrium water uptake was reached. All specimens were taken out at regular time interval, dried using paper towel and then weighed using an electronic balance. The weighed sample was then immediately transferred into the water bath. The water content *(Wc)* in the sample was measured as % weight increase in the sample. *Wc* was measured until the composite specimen attained equilibrium water uptake content (ie., no further uptake of water in specimen or very minimal uptake). Equation 1 is used to calculate the water content *Wc* in the composite specimen.

$$Wc = \frac{(Wt - Wo)}{Wo} \times 100 \tag{1}$$

where *Wt* is the weight of specimen at time *t* and *Wo* is the initial weight of the sample before placing in water. Dynamic mechanical analysis (DMA) was carried out at a frequency of 10 Hz with 3-point bending mode using the Netzsh model Q800 under atmospheric conditions. The specimens (5.5 cm X 1 cm X 0.3 cm) were tested before and after placing in a water medium.

3 Results and discussion

Figures 1 and 2 shows the fatigue result of 50% untreated (UT) sisal fiber reinforced PP and Epoxy composites respectively at constant stress ratio (R=0.1) condition. The life cycle of PP and epoxy composites is different from each other, however, it shows a similar failure pattern, i.e., as the stress decreases the fatigue life cycles increases. Tables 3 and 4 shows the fatigue life cycle of PP and epoxy sisal fiber (treated and untreated) composites respectively at 90% stress level. The result shows that the fatigue life cycle of treated fiber composites is increased to the order of about 10^2 than that of untreated sisal fiber composites. The fracture surface of the representative specimen was examined and shown in Figure 3. The fracture surface shows the brittle type failure in both the epoxy and PP composites.

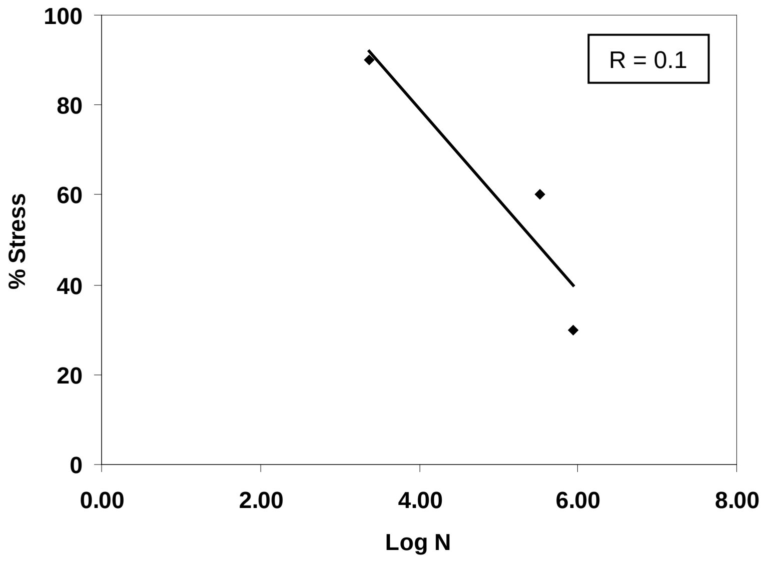

Figure 1. Stress-life fatigue result of PP-untreated fiber (50%)

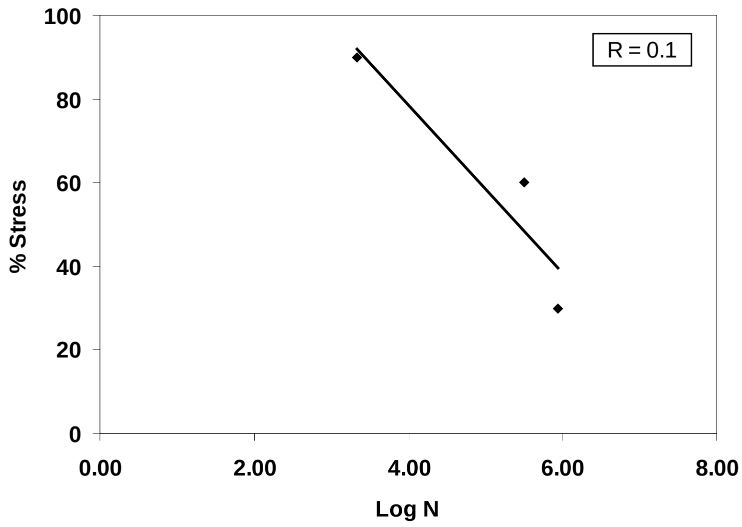

Figure 2. Stress-life fatigue result of Epoxy-untreated fiber (50%)

To study the effect of moisture on fatigue properties of the composites, the fatigue test was performed after soaking the composite sample in the water medium. Figure 4 shows the water mass uptake of UT sisal fiber reinforced epoxy composites. The result shows that the water mass uptake of composites were continuously increased as the fiber content continuously increase in epoxy polymer and similar result was observed in the PP based composites. Tables 1 and 2 show the equilibrium water mass uptake of the PP and epoxy sisal fiber reinforced (treated and untreated) composites. The result also shows that the chemically treated fiber reinforced composites resulted in reduced water mass uptake than that of untreated fiber composites (at the respective fiber content).

The fatigue result of water immersed PP and epoxy composite samples is shown in Tables 3 and 4 respectively. The result shows that the moisture content adversely affect the fatigue properties of the composites. Even though the fatigue life of chemically treated and water immersed sisal fiber composite is reduced than the composites before immersing into water medium, the magnitude of reduction of the fatigue life cycle is lower than that of untreated composites when placed in the water medium. Another observation shows that the fatigue life cycle of epoxy sisal fiber composites was severely affected due to moisture content than that of PP sisal fiber composites. Basically PP polymer is a hydrophobic polymer (due to their non-ionic molecular structure) and hence resulted in lower water uptake than that of epoxy polymer which is a hydrophilic polymer (ionic molecular structure) [5].

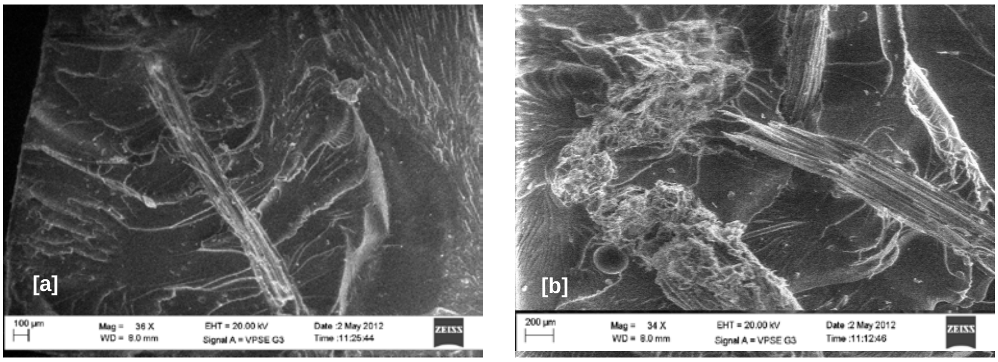

Figure 3. Fatigue fracture surface of (a) epoxy and (b) PP sisal fiber composites

Material	Untreated Fiber Composites	Treated Fiber Composites
Epoxy	62.6	62.6
E + 10%	65.6	54.6
E + 20%	68.3	59.6
E + 30%	77.1	63.4
E + 40%	85.3	68.3
E + 50%	92.6	72.1

Table 1. Equilibrium water uptake (%) of epoxy and epoxy-fiber composites

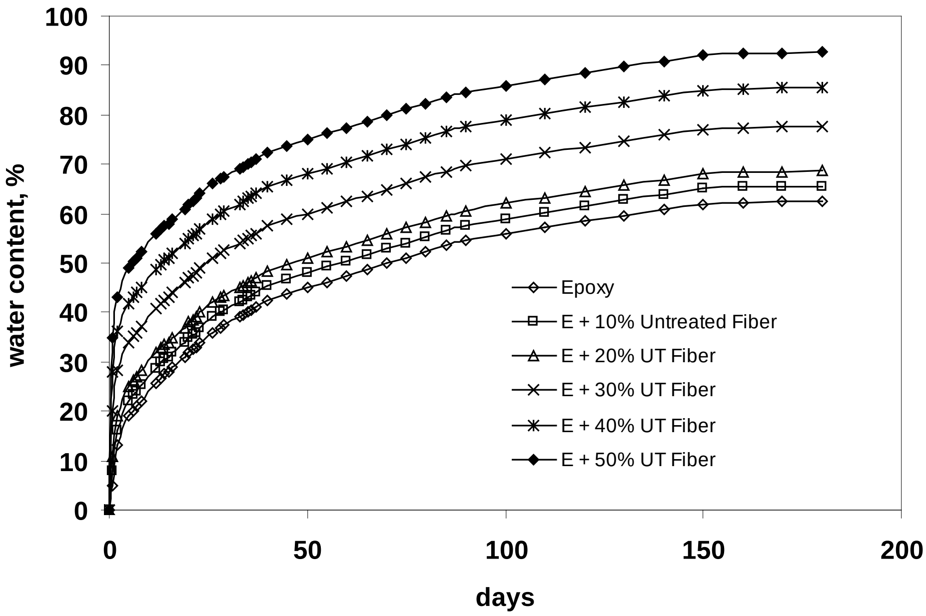

Figure 4. Water uptake curves of Epoxy-untreated fiber series

Material	Untreated Fiber Composites	Treated Fiber Composites
PP	3.0	3.0
PP + 10%	3.7	3.2
PP + 20%	4.0	3.3
PP + 30%	4.2	3.5
PP + 40%	4.6	3.7
PP + 50%	5.3	3.8

Table 2. Equilibrium water uptake (%) of PP and PP-fiber composites

Material	Untreated Fiber Composites		Treated Fiber Composites	
	Before	After	Before	After
PP + 10%	2400	2252	2501	2356
PP + 20%	2451	2286	2533	2401
PP + 30%	2632	2315	2743	2435
PP + 40%	2853	2368	2901	2503
PP + 50%	2891	2453	2983	2589

Table 3. Fatigue life of PP-fiber composites

Material	**Untreated Fiber Composites**		**Treated Fiber Composites**	
	Before	**After**	**Before**	**After**
E + 10%	2160	1803	2182	1963
E + 20%	2182	1853	2253	1979
E + 30%	2206	1795	2295	2004
E + 40%	2321	1771	2393	2023
E + 50%	2388	1763	2459	2067

Table 4. Fatigue life of Epoxy and Epoxy-fiber composites

The cause of the reduced fatigue life cycle of composites due to water content was examined by studying the dynamic mechanical properties of the composites before and after placing in the water medium. Figure 5 shows the DMA properties of pure PP and epoxy polymer. Figure 5a and 5b shows the variation of storage modulus and loss modulus respectively of polymer as the function of temperature. The temperature corresponding to the maximum peak of loss modulus is called the glass transition temperature (T_g) of the polymer. T_g of pure PP and epoxy is 5°C and 55°C respectively, and the effect of T_g of the composites before and after placing in the water medium is shown in the Figures 6 and 7 respectively. The result shows that the T_g of untreated sisal fiber composites is highly affected than that of treated sisal fiber composites due to water content. The presence of water could have caused the plasticization effect to the polymer matrix or could have reduced the strength of fibers resulting in the reduced fatigue properties. Table 5 and 6 shows the storage modulus of PP and epoxy sisal fiber (treated and untreated) composites, before and after placing in the water medium. The result shows that the storage moduli of composites are affected due to water content. Better improvement in modulus was observed in treated fiber reinforced composites.

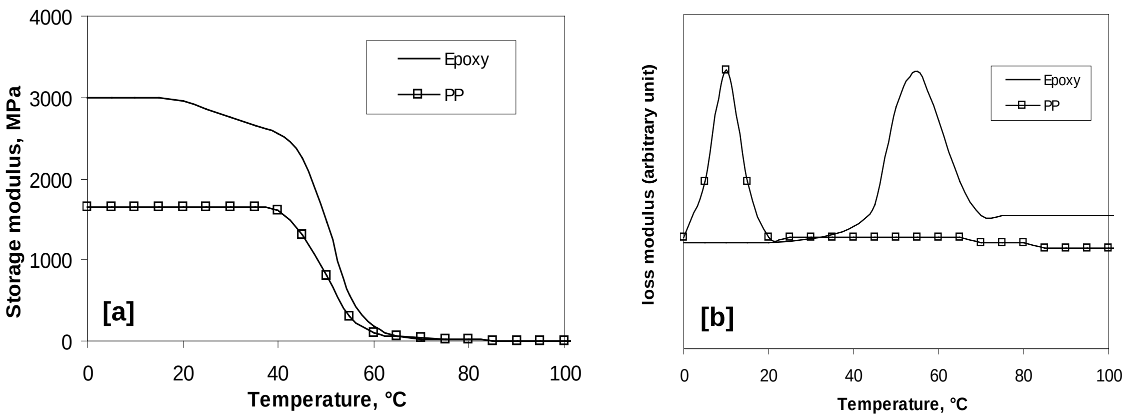

Figure 5. DMA curves of epoxy and PP polymer (a) storage modulus and (b) loss modulus

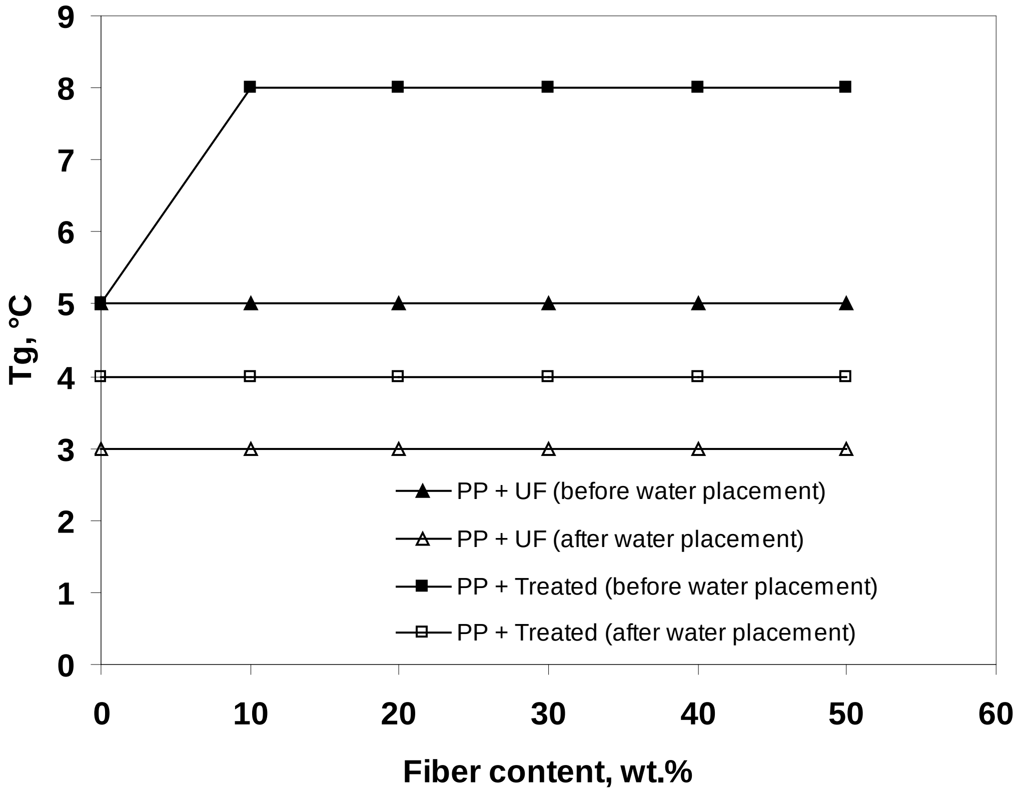

Figure 6. Effect of Tg due to moisture content in PP-fiber composites

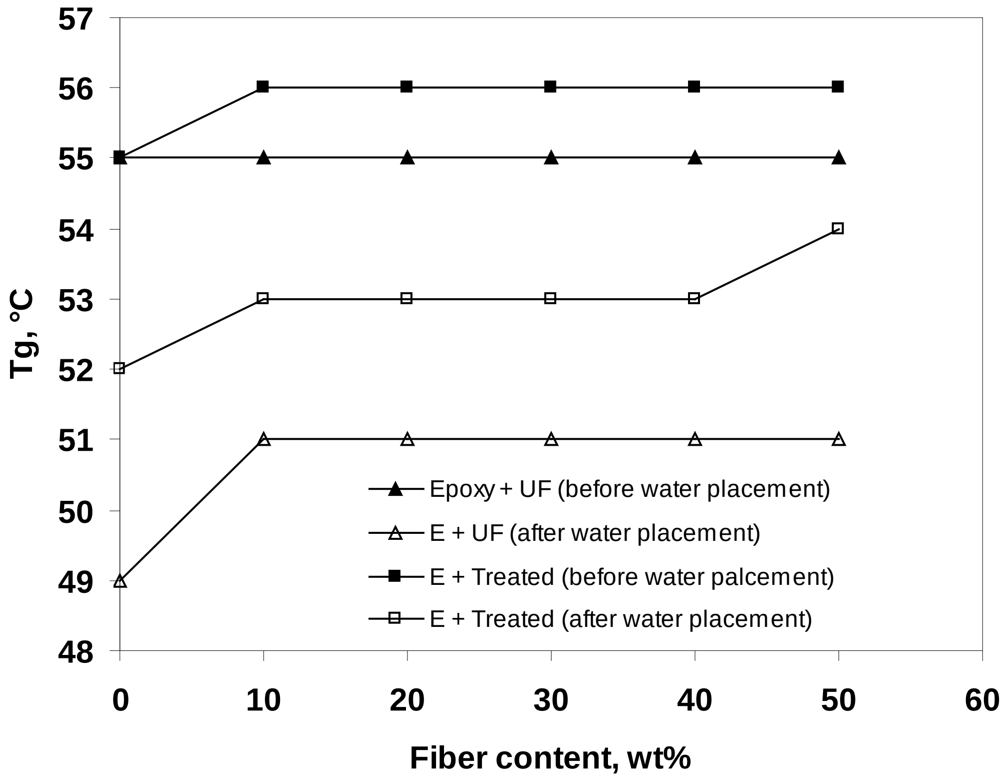

Figure 7. Effect of Tg due to moisture content in Epoxy-fiber composites

Material	**Untreated Fiber Composites**		**Treated Fiber Composites**	
	Before	**After**	**Before**	**After**
PP + 10%	1569	1401	1603	1523
PP + 20%	1543	1463	1589	1514
PP + 30%	1529	1429	1583	1537
PP + 40%	1536	1346	1611	1549
PP + 50%	1436	1127	1627	1587

Table 5. Storage modulus (MPa) of PP-fiber composites at room temperature

Material	**Untreated Fiber Composites**		**Treated Fiber Composites**	
	Before	**After**	**Before**	**After**
E + 10%	3000	2286	3000	2836
E + 20%	3011	2253	3053	2901
E + 30%	3023	2178	3057	2963
E + 40%	2937	2164	3089	2961
E + 50%	2843	2113	3113	2923

Table 6. Storage modulus (MPa) of Epoxy and Epoxy-fiber composites at room temperature

4 Conclusions

The effect of moisture on fatigue properties of sisal fiber (treated and untreated) reinforced PP and epoxy composites were examined in this work. The result shows that the moisture content reduces the fatigue life cycle of the composites. NaOH-clay treated sisal fiber reinforced composites shows better and improved fatigue properties that that of untreated sisal fiber composites, when examined in before and after water placement conditions. Untreated sisal fiber composites shows about 10^2 times reduced fatigue life cycle and 10^1 times reduced water content than that of treated sisal fiber composites. It is also observed that the T_g and storage modulus of composites were reduced after placing in the water medium and thereby reducing the fatigue properties. NaOH-clay treated fiber composites shows positive result in DMA parameters (T_g and storage modulus) than that of untreated composites before and after water placement conditions.

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