Original Article

Performance of *Zea mays* fiber reinforced epoxy composites

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Abstract

The tensile properties of unidirectional and randomly oriented short fibre lengths from agricultural based plant stems *Zea mays* (poaceae) fibre/epoxy composites (ZMFE) are described for the first time in this work. Composites were fabricated using raw *Zea mays* fiber (ZMF’s) with varying fibre weight percents viz. 25, 30, 35, 40 and 45wt %. The tensile parameter such as maximum stress, Young’s modulus and elongation at break were determined using the universal testing machine (UTM). Wet hand lay-up technique was used for the preparation of the composite. Effect of alkali treatment (with and without 10 % NaOH solution) of ZMFE composites were also studied on the tensile properties. ZMFE composites showed a regular trend of an increase in properties with fibre weight percent until 40% and afterwards a decrease in properties for composites with greater fibre weight percent. It was observed that the increased performance was attributed for unidirectional fabric was due to the narrow interface between the fabric and matrix, there by stress transfer between increased. The analysis of the tensile parameters of short ZMFE composites displayed an optimum fibre weight percent at 40wt %. Scanning electron microscope (SEM) studies were carried out to evaluate the fibre/matrix interactions. DSC, TGA and FT-IR spectra of treated and untreated ZMFE composites were also studied.

Key words: tensile parameter, *zea mays* fibre, composites, SEM, FTIR

INTRODUCTION

Natural fibers are gaining progressive account as renewable, environmentally acceptable, and biodegradable starting material for industrial applications, technical textiles, composites, pulp and paper, as well as for civil engineering and building activities. The fibers of the plants, such as flax, hemp, linseed, jute, sisal, kenaf, yucca, abaca, or ramie, have outstanding mechanical properties [1-10]. The first-rate mechanical characteristics of these natural fibers permit the substitution of synthetic, glass, and carbon fibers in a wide range of industrial products. The weight of the natural fibers is about two thirds and the consumption of primary energy for their production is only one third that of glass fibers at a comparable strength. Therefore, natural fibers embedded in plastics will soon compete strongly with conventional reinforcing fibers. Remarkable strength, high stiffness, and dimensional stability of light constructional elements make it possible to essentially reduce the weight of aircraft, trains, trucks, and cars. Technical designers have long been talking about the “end of the metal age,” and a silent revolution will take place in the construction of aircraft and vehicles during the next ten years. The industrial application of natural fibers

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requires making high quality fibers continuously available in large quantities at competitive prices and independently of weather conditions and annual yields. Conventional processing technologies cannot meet the strict demands of modern industries. Consequently, new technologies have to be developed in order to successfully set up powerful process plants for natural bast fiber. Therefore natural fibers can serve as reinforcement not only by improving the strength and stiffness and also reducing weight of the resulting composite materials, although the properties of natural fibers vary with their source and treatment. In order to improve the performance of these composites, the matrix or the reinforcement often needs to be modified. Many aspirants worked on modification of surface of lingo-cellulose fibers and fabric by reinforcing or by short and long fibers or by unidirectional and randomly oriented by coated with coupling agent[11-27] with different polymers, research with Zea mays reinforced composites are scanty.

In the present research, plant stem of Zea mays (poaceae) stalks were reinforced into the epoxy resin as a short fiber at different weights viz. 25, 30, 35, 40 and 45wt. %. Unidirectionally oriented fiber orientations were fabricated using short fibers. The tensile parameter such as maximum stress, Young’s modulus and elongation at break were determined using the universal testing machine (UTM). Scanning electron microscope (SEM) studies were carried out to evaluate the fibre/matrix interactions on treated and untreated surfaces using alkali solution. TGA, DSC and FT-IR spectra of treated and untreated ZMF composites were also studied.

METHODOLOGY
The epoxy and hardener (Araldite-LY 556 and Amine Hardener- HY 951) employed in this study was supplied by Ciba-Geigy. ZMF was extracted from Z.may plant stalks was obtained from the Enumuloddi forest, near Kalyanadurg, Anantapur (AP) India. Fiber peeled from the stalks after in was kept under the sun over a period of one month. This way 90 % of cellulose materials are separated easily. Different stages of the ZMF extracted were shown in the Figure 1. Then ZMF were treated with a 5% NaOH solution and allowed to soak in the solution for about half an hour. The fibres were washed with water to remove the excess quantity of NaOH sticking to the fibres. Finally the fibers were washed with distilled water and dried in a hot oven at 70°C for half an hour. Stalks were kept under compression during drying to keep the shrinking at bay. For short fibers width and thickness was maintained about 3mm and 0.2 mm was maintained uni-randomly oriented samples respectively. Stalks were rubbed with emery paper on the outer and inner surfaces to attain the rough surface. Then the fibres were cut into sort fiber lengths (30-33mm). Short fibers have cut with sharp scissors and kept in unidirectional orientations in the glass mould prior to pour into the epoxy modified epoxy mixture. ZMF fibers were placed in the mould in unidirectional manner, and then epoxy/hardener mixer (100:10) ratio was stirred about 15minutes then poured in to the mould. Samples were cast in a glass mould and cured for 24 hours at room temperature followed by 1hour at 80°C.

Procedure was applied for randomly oriented composite preparation. Tensile tests were conducted using universal testing machine (Instron, Series-3369) with across head speed of 5mm/min. In each case, five samples were tested and average value tabulated. Authors were used 10KN load cell for testing further the sample sizes are 100mm x 20mm x 3mm and supported span length of 50mm. Tensile samples were cut on par with ASTM D638 taking specimens of dimensions (165x13x3) mm³. Tests were carried out at room temperature. All the readings were taken 10s after the indenter made firm contact with the specimen. The test was repeated about 10 times for every sample and the average values were taken. The thermal characteristics TGA, DSC is measured on ZMF reinforced epoxy composites using SDT Q600 TGA/DSC (TA Instruments) at a rate of 10°C/min under nitrogen flow. Measurements were carried out at 25°C temperature, 45% relative humidity. The sample undergoes a sinusoidal oscillation at a fixed frequency. At least three tests were carried out for each case. Scanning electron microscopy (SEM) studies of the fractured surface of the tensile specimen were carried out on a Jeol (6380LA, Japan). The specimen was sputter-coated with gold to increase surface conductivity. The digitized images were recorded. The FTIR spectra measurements of the untreated and alkali treated samples were run on an ABB-Bomem FLATA-2000 model spectrophotometer using KBr pellets. The concentration of the fabric powder was maintained at 1% in KBr.

RESULTS AND DISCUSSIONS
Tensile properties uni and randomly oriented fiber of before and after treatment are shown in the Table 1 as a function of fiber loading. Maximum stress, Young’s modulus and elongation at break were gradually increases as fiber loading increases from 25wt.% to 40wt.%, but decreases in further increase to 45wt.%. As always treated fiber gets going due to

<table>
<thead>
<tr>
<th>Table 1</th>
<th>Fiber Loading (wt%)</th>
<th>Maximum Stress (MPa)</th>
<th>Young’s Modulus (GPa)</th>
<th>Elongation at Break (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>25</td>
<td>26.3</td>
<td>0.94</td>
<td>0.37</td>
<td>8.7</td>
</tr>
<tr>
<td>30</td>
<td>27.5</td>
<td>1.07</td>
<td>0.56</td>
<td>11.2</td>
</tr>
<tr>
<td>35</td>
<td>28.8</td>
<td>1.15</td>
<td>0.78</td>
<td>13.8</td>
</tr>
<tr>
<td>40</td>
<td>29.3</td>
<td>1.22</td>
<td>0.96</td>
<td>15.5</td>
</tr>
<tr>
<td>45</td>
<td>29.8</td>
<td>1.29</td>
<td>1.13</td>
<td>17.2</td>
</tr>
</tbody>
</table>

Figure 1 Different stages of Z.mays (a) bottom stem segments (b) cylindrical cellulose material (c) fiber stalks after peeling and (d) treated fiber.
Table 1: Tensile parameters of treated (T) and untreated (UT) ZMF’s composites as a function of fiber weights.

<table>
<thead>
<tr>
<th>Fiber weight (wt.%)</th>
<th>Max. Stress (MPa)</th>
<th>Tensile properties</th>
<th>Young’s modulus (GPa)</th>
<th>Elong. at break (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>UT(T)</td>
<td>UT(T)</td>
<td>UT(T)</td>
<td>UT(T)</td>
</tr>
<tr>
<td>25</td>
<td>55.81(56.33)</td>
<td>53.91(54.26)</td>
<td>0.58(0.64)</td>
<td>0.60(0.65)</td>
</tr>
<tr>
<td>30</td>
<td>60.32(62.97)</td>
<td>59.81(60.12)</td>
<td>0.64(0.70)</td>
<td>0.63(0.69)</td>
</tr>
<tr>
<td>35</td>
<td>66.84(66.98)</td>
<td>65.09(66.07)</td>
<td>0.85(0.90)</td>
<td>0.84(0.98)</td>
</tr>
<tr>
<td>40</td>
<td>75.22(76.03)</td>
<td>75.00(76.23)</td>
<td>1.05(1.10)</td>
<td>1.00(1.02)</td>
</tr>
<tr>
<td>45</td>
<td>70.43(71.34)</td>
<td>66.02(67.90)</td>
<td>1.01(1.20)</td>
<td>0.91(1.08)</td>
</tr>
</tbody>
</table>

Note: Values shown in the bracket is treated values for ZMFE composites.

Figure 2: TGA micrograms for short Zea mays fiber before and after alkali treatment with 5% NaOH solution long fibers.

Figure 3: DSC micrograms for short Zea mays fiber before and after alkali treatment with 5% NaOH solution long fibers.

The removal of white cellulose and lignin, in building interface between fiber matrixes. Tensile strength modulus and elongation at break was improved by 34.9%, 71.8 and 79.9% respectively for treated/unidirectional fiber orientation at 40% fiber loading when compared with 25wt. % fiber loading. Tensile strength modulus and elongation at break was improved by 40.4%, 47.8 and 96.6% respectively for treated/randomly oriented fiber orientation at 40% fiber loading when compared with 25wt. % fiber loading. Creating rough surface by removing all white, grease, lignin, cellulose materials would certainly promotes excellent performance. Unidirectional fiber orientation left less overlapping of fibers there by considerable amount of reduced pull outs, fiber agglomerations, on other hand, in randomly oriented fiber composites, lot of gap can be seen between the fiber and matrix that really makes bulk gaps between them, as a result of that remote chance of hiking performance. In other words, more chances of uniform fiber distribution in the former case, where as less chances of uniform filling in the later case and yet more chances of overlapping. Another reason is more chances of air entrapment when fiber becomes overlapped, there by crack tip initiation is quiet easy and that makes poor stress transfer. The TGA curves of treated and untreated fibers were shown in the Figure 2. The TGA curves of ZMFE composites at different time intervals gave two distinct temperature regions were the samples experience significant weight loss. The slight weight loss below 100°C is due to the moisture present in the untreated fiber. Weight loss was constant for untreated fiber up to 250°C, and from there on to 450°C decomposition was occurred for untreated. Treated samples were turned out good weight loss was occurred slightly above the untreated fiber. Weight loss was constant for untreated fiber up to 300°C and from there on to 500°C decomposition was occurred for untreated. Generally fibers become stiffer (modulus) after removal of organic material in the form of lignin, cellulose. This might be reason in increase in decomposition temperature (Ashok Kumar 2011c). DSC thermograms on treated and untreated ZMFE composites were shown in the Figure 3. The glass transition temperature (Tg) of composite was observed at a temperature of 70°C for
untreated; 70.5°C for treated. However, with fiber loading the Tg values do not shift appreciably. From the above figures it was clearly noted that 0.5°C rise in glass transition temperature was observed. Crystallization temperature was observed for untreated was 390°C, for treated it was improved significantly about 405°C. An endothermic peak was observed at beyond 500°C both SVFE treated and untreated (which is not shown in the figure) composites. In order to investigate the structural changes in the fiber before and after alkali treated the FTIR spectra of the samples were recorded. The spectra of untreated (Figure 4a) and alkali treated (Figure 4b) showed similar bands (corresponding to the lignin and cellulose) except the disappearance of the band at 1700 cm⁻¹ when fiber was treated with alkali solution. This band corresponds to the CO stretching of the hemicellulose present in the untreated fiber. Further no appreciable changes in the spectra of the fiber in the absence of alkali solution were noticed except weak additional bands around 850 cm⁻¹. The remarkable reinforcing ability can be accounted for efficient hydrogen bonding between the fibers, which are basically composed of cellulose (composed of hydroxyl group) and the polymeric matrix (composed of NH and isocyanate terminated groups). Analysis of the stretching vibration of epoxy and the composites showed that there is a

Figure 4 FTIR spectra of short fiber ZMFE composites before and after alkali treatment with 5% NaOH solution long fibers.

Figure 5 SEM micrograms for Zea mays fiber before and after alkali treatment with 5% NaOH solution (a) untreated; (b) treated.
very prominent band at approximately 1620 cm⁻¹ attributed to free C=O epoxy group. In addition, there appears an additional shoulder at about 1700 cm⁻¹ ascribed to hydrogen bonded C=O band present in the composite samples. This shoulder is not evident in the pre-polymer. The observed prominent absorbance of the NCO group 2150 cm⁻¹ of the epoxy pre-polymer and broad absorbance of the hydroxyl group 3320 cm⁻¹ present in the treated fiber all decreased to zero absorbance in the composite. The disappearance of the –NCO and –OH bands is due to the formation. These functional groups could engage in the in covalent and hydrogen bonds formation.

The scanning electron macrographs of fiber surfaces of the untreated and treated fibers were shown in the **Figure 5a & Figure 5b** respectively. Significant changes were observed in each case. For example, the content of white components belonging to the hemicellulose in the untreated fiber (figure 5a) decreased on the alkali treatment (figure 5b). This indicates the elimination of some surfaces held hemicellulose by the NaOH solution. It can also observed from the graphs after treatment surface were become rough that may be sound great in building strong interface. It can also observe that the white layer (corresponding to hemicellulose) is decreased considerably upon alkali treatment. This is as expected since the hemicellulose is soluble in aq NaOH solution. A rough and cellulose removed surface can be seen in the later figure, where as white cellulose flakes can be seen on the former micrograph was the clear indication of the decrease in composites behaviour.

**CONCLUSIONS**

The effects of untreated and treated and unidirectional and randomly oriented short fibers of ZMFE composites were studied. FTIR studies indicated that the formation of functional groups could engage in the in covalent and hydrogen bonds formation. SEM surfaces decoded that removal of cellulose and lignin would create rough surface as a result more interaction between fiber and matrix which played vital role in building performance. Short fiber unidirectional orientation fiber builds interfacial strength due to more uniform spreading of fiber. Elimination of amorphous hemicellulose by alkali treatment and filling up the rough surfaces with polymer may be the responsible for this behaviour. It was observed that, performance of unidirectional/treated short fibers was optimized at 40wt. % when treated with alkali solution. 50°C rise in decomposition temperature and 4°C rise in glass transition temperature was observed in TGA and DSC measurements.

**REFERENCES**


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