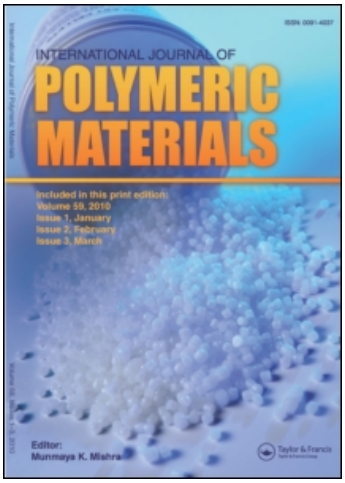

International Journal of Polymeric Materials
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713647664

The Mechanical Properties and Chemical Resistance of Short Glass-Fiber-Reinforced Epoxy Composites

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To cite this Article Kaushik, Anupama , Singh, Paramjit and Kaushik, Jyoti(2006) 'The Mechanical Properties and Chemical Resistance of Short Glass-Fiber-Reinforced Epoxy Composites', International Journal of Polymeric Materials, 55: 6, 425 — 440
To link to this Article: DOI: 10.1080/009140390970404
URL: http://dx.doi.org/10.1080/009140390970404

International Journal of Polymeric Materials, 55:425–440, 2006

ISSN: 0091-4037 print/1563-5333 online
DOI: 10.1080/009140390970404

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The Mechanical Properties and Chemical Resistance of Short Glass-Fiber-Reinforced Epoxy Composites

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Epoxy–short glass fiber composites were prepared by directly blending two-pack system of Araldite (CY-230) and hardner (HY-951) with short glass fibers. The short glass fiber content was varied from 2% to 10% by weight of the total matrix. These composites were then characterized for morphology using scanning electron microscopy, mechanical properties, that is, tensile and flexural properties and resistance toward various chemicals. The epoxy-glass fiber composites showed improved tensile and flexural properties but increased dispersion among the properties with increasing fiber content. Several reasons to explain these effects in terms of reinforcing mechanisms were discussed. These composites were stable in most chemicals but were completely destroyed in concentrated sulfuric acid, nitric acid, and pyridine.

Keywords: short glass fibers, epoxy composites, chemical absorption, mechanical properties

INTRODUCTION

During the last decades, there has been a tremendous growth in the use of composite materials in various fields of application, ranging from sporting goods to structural components for the automotive and aerospace industries, where the long-term properties are of primary

Received 31 March 2005; in final form 11 April 2005.
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importance. High-performance polymer composite materials are being used increasingly for engineering applications under hard working conditions. The materials must provide unique mechanical and tribological properties combined with a low specific weight and a high resistance to degradation in order to ensure safety and economic efficiency.

Epoxy resins are widely used for matrix composites due to their excellent adhesive, dielectric, and mechanical properties [1–5] and alone provide most of the properties required for composites. However, because the polymer matrix must withstand high mechanical and tribological loads, it is usually reinforced with fillers. These fillers can be chosen either as fibers (glass, carbon, and aramid) or particles such as ceramic powders. These fillers favorably stiffen the material and may also increase the strength under certain load conditions.

The fibrous composites, especially glass fiber–reinforced epoxy composites in which glass fiber is the primary load carrying element, are being increasingly used in military and aerospace applications owing to several desirable properties including high specific strength, high specific stiffness, and controlled anisotropy. The mechanical properties of composites are most important, because they can be influenced by parameters such as type of filler, type of matrix, filler concentration, filler dispersion, alignment of fibers, length of fibers, aspect ratio of fibers, the fiber matrix interphase properties or adhesion between fiber and resin matrix [6–9]. Glass fiber reinforcement can be of several types, such as rovings, mats, woven fabric, hybrid fibers, multiaxial fabrics, and the most important and widely used short glass fibers. The short-fiber reinforced polymer (SFRP) composites are very attractive because of their ease of fabrication, economy, and superior mechanical properties. By combining short fibers with an appropriate polymer matrix and by controlling the production process it is possible to make composite materials featuring large specific properties. In addition, the constituents, that is the polymer matrix and the glass fibers, are usually inexpensive and easily processed. As a consequence of these technical and economical reasons, the short-fiber reinforced polymer composites are finding more and more industrial applications where high performance per weight at a reasonable price is required.

For short fiber materials there are many more microstructural variables that affect the elastic and thermoelastic properties as compared to the continuous fiber materials: these include distributions of fiber to fiber separation, fiber length, and fiber orientation. Tensile strength is one such important property. One of the basic motivations for the use of composite materials as engineering materials is the high tensile strength that can be achieved by incorporating high-strength fibers

into a matrix since the fibers carry most of the load. As indicated earlier, failure initiation and the fracture process of SFRPs depend to a large extent on the fiber volume fraction, the fiber aspect ratio, and the ratio between failure strain of fiber and matrix. In short-fiber reinforced composites, tensile strength of composites increases with increasing aspect ratio of reinforcing fibers [9–14]. The orientation and length of fibers also affects the mechanical characteristics of composites'; the studies show that the tensile strength and ultimate strain are found below the valves, that are obtained for continuous composites. Longitudinal strength and strain increases with increase in fiber length [11,15].

The fiber content also affects the composite failure strain. It has been observed that the composite failure strain decreases with the increase in fiber content [16–19]. The studies on failure mechanisms [18,20] have shown that under loading of tensile stress, the cracks start at the fiber ends and propagate along the fiber–matrix interface or cross through the matrix and finally failure takes place. Fiber ends have been shown to substantially concentrate the stress in the adjacent matrix [18], producing stress magnifications of ten or even higher.

Generally, polymer-based materials are not water-soluble but they are capable of absorbing various amounts of water and other chemicals/solvents depending on their chemical nature and formulation, as well as on the humidity and temperature of the environment to which they are exposed. Despite the application advantages, composites are susceptible to heat, moisture and other chemicals when operating in harsh and changing environmental conditions. When exposed to humid environments, glass–epoxy composites absorb moisture and undergo dilatational expansion (swelling). The diffusion of moisture in polymer systems depends on the polymer microstructure, morphology, and cross-link density, which are functions of degree of cure, stoichiometry, molecular chain stiffness, and cohesive energy density of polymer [13]. The presence of moisture and the stresses associated with moisture-induced expansion can result in lowered damage tolerance, with an adverse effect on long-term structural durability [21].

In the present research work, the epoxy/short glass fiber composites were made by varying the fiber content from 2 to 10% keeping the length and fiber radius constant. Mechanical properties (tensile and flexural strength), moisture absorption, effect of various chemicals, and their absorption have been studied for these composites. The fiber volume fraction was limited to 10% because of difficulty in processing beyond this concentration due to increased viscosity.

EXPERIMENTAL

Materials Used

The E-glass fibers used were in the form of short glass fibers (3 mm) supplied by Saint Gobain, India were mixed with epoxy resin (Araldite CY-230), which was cured with a suitable amount of hardner (HY-951), the ratio of hardner to resin used was 10:1. Other chemicals that were used included toluene (Qualigens), DMF (HPLC grade, Qualigens), and NaCl (E-Merck). Table 1 shows the composition of various samples used in the study.

Specimens Preparation

Short E-glass fiber/epoxy composites were prepared by compression molding method. Fibers were mixed with resin using a stirrer and degassed to avoid air entrapment in the composite; the hardener was mixed for 10 to 15 min and was poured into the mold. The composite sheet was then cured for 24 h at room temperature, and post-cured at 70°C for 9–10 h. Specimens according to standard dimensions were then cut according to ASTM D-638. The sample was dumb-bell shaped with the total length of 150 mm, span 80 mm, width 10 mm, and crosshead speed of 2 mm/min.

Flexural strength was measured using Instron 4466 following ASTM D-790 standard. The sample dimensions were 80 mm × 10 mm × 2.2 mm, the span was 65 mm and crosshead speed was 2 mm/min. The sample dimensions are shown in Figure 1. Maximum bending load (P_{max}), maximum deflection (δ_{max}), Young modulus in bending (E_b), and flexural strength (σ_b) were determined.

TABLE 1 Sample Identification Depending on Fiber Loading for Various Samples of Glass Fiber Reinforced Epoxy Composites

Sample identification	Amount of resin (gms)	Amount of hardner (gms)	Amount of glass fiber (gms)	% of resin based on resin fiber mixture (gms)	% of fibers based on resin fiber mixture (gms)
P1	80	8	0	80	0
P2	100	10	2.02	98	2
P3	100	10	4.58	96	4
P4	100	10	7.02	94	6
P5	150	15	14.35	92	8
P6	150	15	18.33	90	10

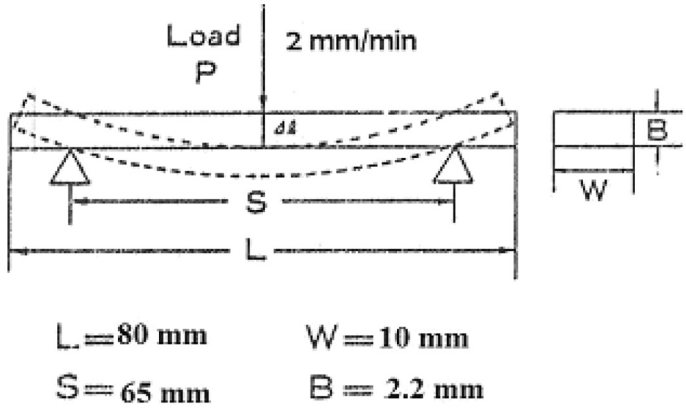


FIGURE 1 Dimensions of flexural testing specimen of short glass fiber reinforced epoxy composites.

Characterization of Short Glass Fiber Reinforced Epoxy Composite Sheets

Morphology

Scanning electron micrographs were obtained on a JSM 6100 CX (JEOL) electron microscope. The sample preparation technique used was based on Kato's osmium tetraoxide staining technique and Matsuo's two step sectioning method. The SEM of the composite samples was done to characterize the composite sheets and to analyze the changes before and after fracture. The samples prepared of short glass fiber reinforced epoxy composites were analyzed and effect of stresses induced during testing were investigated. The interfacial bonding and distribution of voids in the composite can also be studied using this technique.

Chemical Resistance and Absorption

The aim of chemical absorption studies was to test whether the composites were capable of withstanding exposure to a variety of organic solvents including highly solvating species. Acids, alkalis, and various solvents were chosen for this purpose. The initial weight of the polymer samples was determined by weighing on a highly sensitive electronic balance with a least count of 0.0001 g. The samples were then kept immersed in penetrant taken in stoppered diffusion bottles at $50 \pm 0.1^{\circ}C$ (Julabo, VC-5, with cooling as well as heating arrangements). The swollen samples were taken out after 48 h, wiped free of adhering solvents, and weighed on an electronic balance. Each

weighing was completed within 30 s, so as to keep the error due to solvent escape from the surface minimum. The solvents used for chemical absorption study included toluene, xylene, HCl (conc.), HCl (54%), dibutyl amine, CCl_4, NaOH (1.67 N), distilled water, DMF (dimethyl formamide), ethanol, sulphuric acid (conc.), formaldehydes, nitric acid, pyridine, and acetone. The percent absorption was calculated using Eq. 1.

$$A = \frac{m_t - m_i}{m_i} \times 100 \tag{1}$$

where, m_t is the mass of sample at time t (48 h in this case) and m_i is the dry weight of sample.

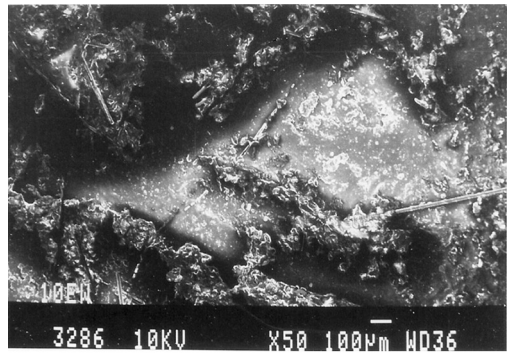
RESULTS AND DISCUSSION

Morphology

Figure 2(a) shows the dispersion of fibers in resin containing 6% fibers by weight and Figure 2(b) shows the dispersion of fibers in the resin sheet containing 10% fibers by weight. It can be clearly observed from the two micrographs that there is good dispersion of fibers up to 10% with a little agglomeration of fibers.

Figures 2(c) and 2(d) shows the scanning electron micrograph of fractured surface of tensile test specimen (containing 6% fibers by wt.) of the glass/epoxy specimen failed at room temperature. The micrographs clearly indicate the effect of stresses induced during testing on the surface of fibers and at resin matrix. Figure 2(c) reveals that there is a good adhesion between resin matrix and fibers. Figure 2(d) reveals that the resin sticking to fibers showing good wetting of fibers by the resin and good adhesion properties. Figures 2(e) and 2(f) represent the effect of stress induced during tensile testing of the 10% glass fiber reinforced epoxy composite after fracture. Holes due to fiber pullout and stress on the resin matrix can also be observed.

FIGURE 2 Scanning Electron Micrographs of short glass fiber reinforced epoxy composites. (a) SEM photograph of sample containing 6% glass fibers; (b) SEM photograph of sample containing 10% glass fibers; (c) SEM photograph of fractured sample containing 6% glass fibers showing random orientation of fibers; (d) SEM photograph of fractured sample containing 6% glass fibers showing good fiber matrix bonding; (e) SEM photograph of fractured sample containing 10% glass fibers showing fiber breakage at yield point; (f) SEM photograph of fractured sample containing 10% glass fiber showing fiber breakage and fiber pullout.



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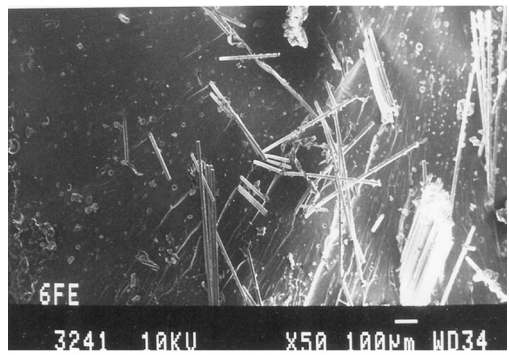

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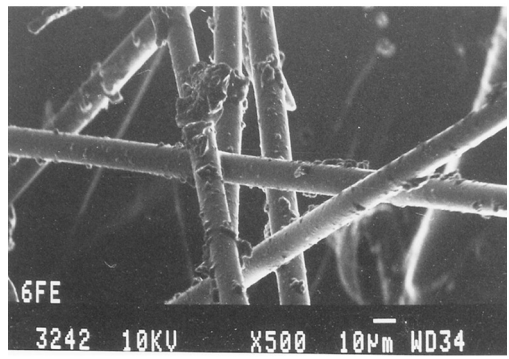

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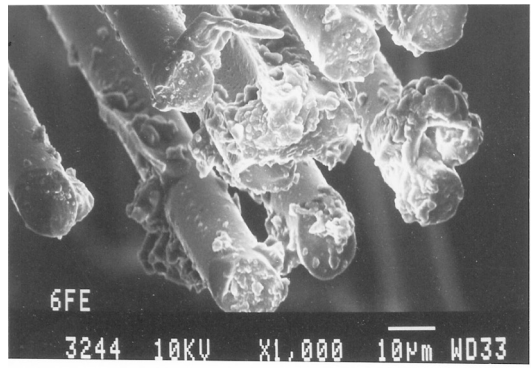

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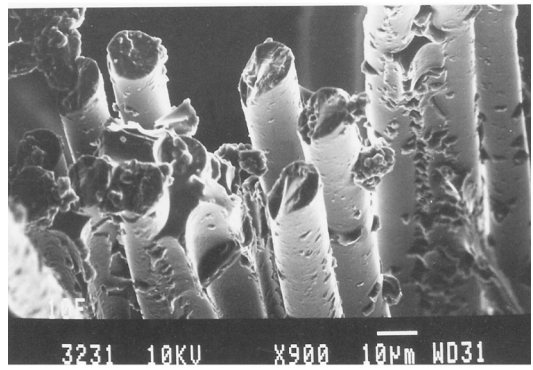

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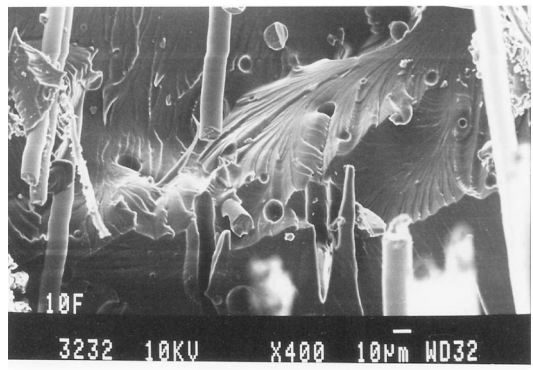


(f)

FIGURE 2 Continued.

Mechanical Properties

Mechanical properties such as the strength and modulus of short fiber–reinforced composites have been shown to depend critically on the microstructure [22–24]. For short fiber–reinforced polymer composites, the microstructure is referred as fiber length, fiber orientation, fiber wetting, and fiber distribution. The fracture toughness is also one mechanical property depending on the microstructure, but it is not so easy to predict this in theory as it is to predict the composite strength and tensile modulus [6,9,15,25]. On the one hand, fiber end micro-cracks reduce the fracture toughness, whereas on the other hand, fiber pullout, interfacial debonding, and fiber fracture increase the fracture toughness [13,26].

Fiber-reinforced composites show a lot of dispersion in mechanical properties. This may be due to random alignment of fibers at a particular fiber volume fraction, very much evident from the electron micrographs. Samples drawn from the same sheet differ in their properties to some extent. Due to this reason four samples were tested for each fiber concentration and mean values have been reported. As the fiber content increases, this dispersion decreases. Highest and lowest values of tensile strength have also been indicated. Figures 3 and 4 illustrates the tensile strength and break displacement of

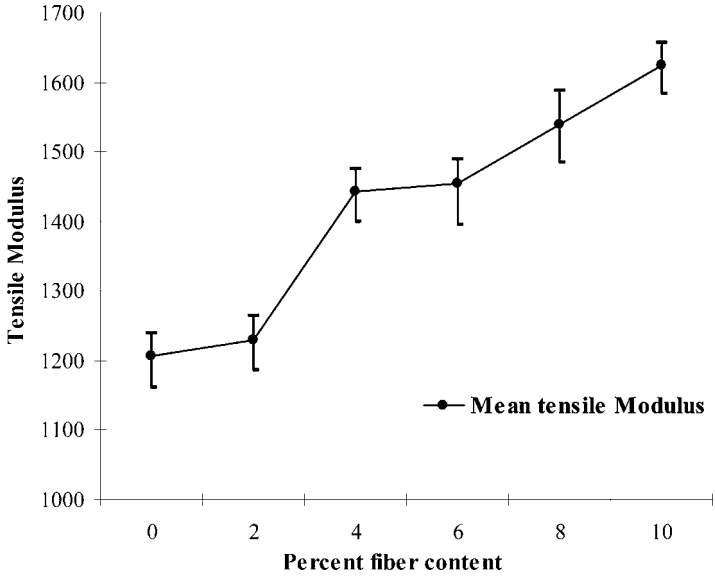


FIGURE 3 Tensile strength versus fiber content of SGFR epoxy composites.

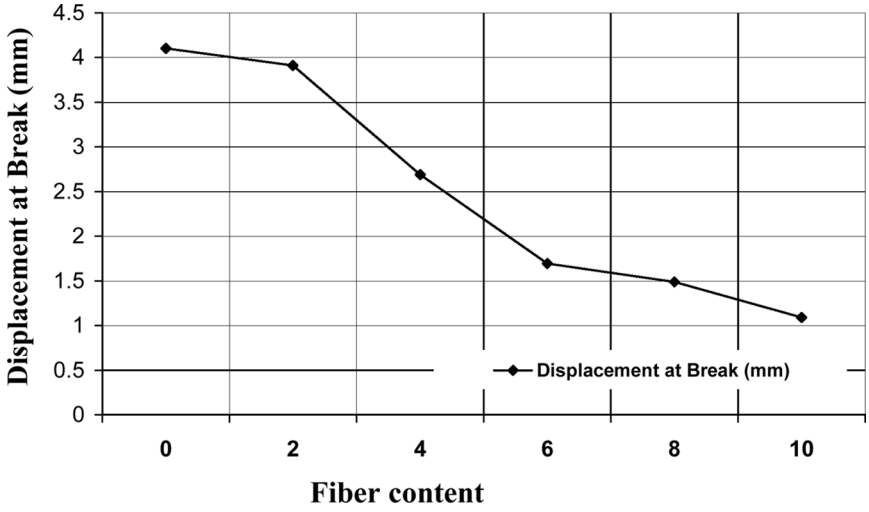


FIGURE 4 Mean value of break displacement versus fiber content of SGFR epoxy composites.

various short glass fiber reinforced epoxy composites with different fiber content. Results indicate that the tensile strength increases with fiber content (Figure 3) and a synergistic relationship exist between the two [27]. It is obvious that the strength enhancement is derived from the high strength of the glass-fiber reinforcement. For the configuration used, the tensile modulus increases from 1207 MPa for the neat sample to 1624 MPa for sample containing 10% fibers.

Visual examinations on the fracture surfaces of short glass fiber–filled epoxy resins by scanning electron microscope methods can often reflect detailed information on the cause and location of failure. Some representative samples were chosen to study the deformation mechanisms responsible for the reinforcement. Resulting pictures of fractured samples can be seen in Figure 2 (c–f) for 6% and 10% short glass fiber content after subjecting them to tensile load. Several mechanisms of crack propagation and failure are commonly known to be applicable for filled epoxy resins [28]. On one hand, cracks may pass through the fiber if the fillers are weak, known as trans-particulate fracture, or they may pass around if the fibers are strong enough [29]. On the other hand, failure may occur by interfacial debonding or by cohesive failure of the matrix. Figure 2(f) shows a fracture surface for the epoxy matrix with 10% glass fibers, which reveals a brittle behavior characterized by large smooth areas, large hyperbolic markings, ribbons, and fracture steps in the direction of crack propagation. The figure also shows scattered holes indicating fiber pullout.

TABLE 2 Variation of Young Modulus in Bending, Maximum Displacement, Maximum Bending Load, and Modulus of Rupture with Varying Amount of Fiber Content

Sample identification	Young's modulus in bending (MPa)	Maximum displacement (mm)	Maximum bending load (KN)	Modulus of rupture (MPa)
P1	966.2	9.50	0.098	181.67
P2	1243	9.00	0.10	186.51
P3	1261	8.50	0.11	209.01
P4	1285	8.20	0.13	236.16
P5	1546	7.32	0.13	236.99
P6	1846	1.74	0.131	244.34

Contrary to tensile strength, there is a decrease in break displacement or failure strain with rising fiber content. Even low fiber loadings can cause a dramatic drop in the fracture strain. One has to recall that the composite is part filler and part matrix. Due to the rigid nature of the fillers, most of the deformation comes from the polymer. The actual deformation experienced only by the polymer matrix is much larger than the measured deformation of the sample, with the result that the polymer reaches the failure strain limit at a lower total deformation. Hence, the total composite strain-to-break decreases. The reduction in the failure strain is also caused by an embrittlement effect as the stiffness of the composites is improved when the fiber volume fraction is increased. The cause of this effect has been identified as matrix crack formation at the ends of the reinforcing fibers. Subsequently, as the strain is increased more cracks form progressively at the ends of shorter fibers. Initially this cracking can be accommodated by load transfer to adjacent fibers that "bridge" the cracked region. Final failure occurs when the extent of cracking across the weakest section of a specimen reaches a critical level when the surrounding fibers and matrix can no longer support the increasing load. For epoxy short glass fiber samples prepared in this study, the break displacement decreases from 4.1 mm for the neat resin sample to 1.09 mm for sample containing 10% fibers.

Maximum bending load (P_{max}), maximum deflection (δ_{max}), Young modulus in bending (E_b) and flexural strength (σ_b) are reported in Table 2. The modulus of rupture (MOR) of the material was calculated using Eq. 2 for beam theory.

$$MOR = \sigma_{max} = \frac{3PS}{2WB^2} \tag{2}$$

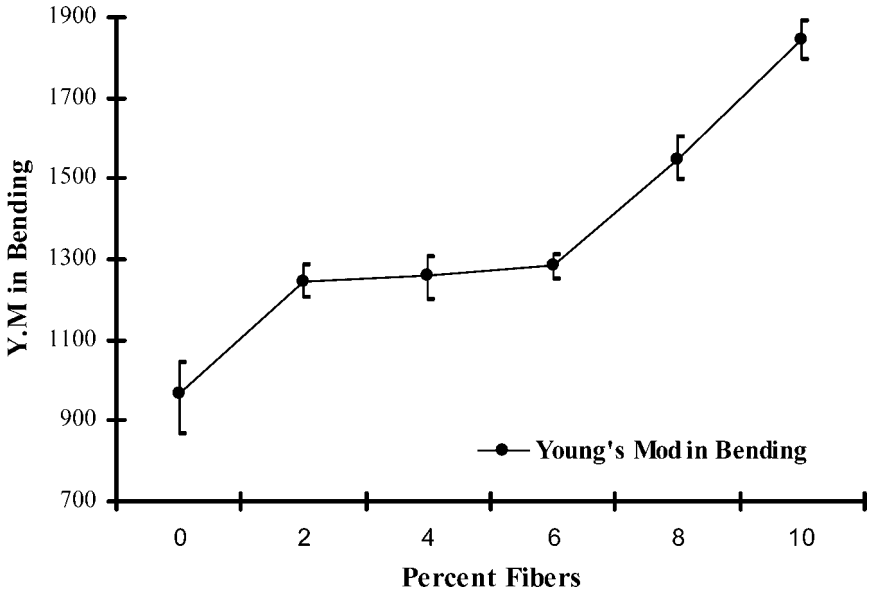
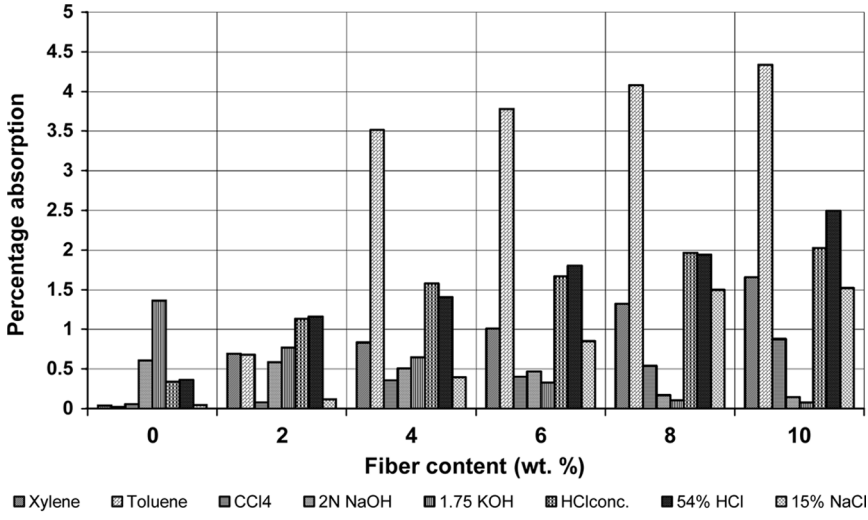


FIGURE 5 Variation of fracture toughness with fiber content for short glass fiber reinforced epoxy composites.

where P is the critical fracture load, W and B are the width and thickness of the specimen, and S is the span of the flexure supports. For the specimens used in this study, $S = 65\,\text{mm}$, $B = 2.2\,\text{mm}$ and $W = 10\,\text{mm}$. Figure 5 shows the variation of fracture toughness with fiber content for short glass fiber reinforced epoxy composites.

It is interesting to note that fracture parameters including maximum bending load, Young's modulus in bending, and Modulus of rupture increase steadily with increasing fiber-volume fraction. In the presence of fibers, all fracture parameters are improved as a result of a combination of fiber-related micro-mechanisms such as fiber bridging and pull-out, and an increase in fracture strength, the latter being associated with additional load bearing capability of the fibers [26]. The flexural modulus also follows the same increasing trend as that of tensile modulus, that is, minimum for resin sheet (966 MPa) and maximum for the sheet containing 10% glass fiber (1846 MPa). The fracture toughness of these specimens is nearly two times higher than non-reinforced specimens. It should be mentioned here that the addition of glass fibers to epoxy matrix also increases the viscosity of the wet mix, thus reducing workability. For this reason the maximum fiber content of the mix is limited to about 10 wt%.



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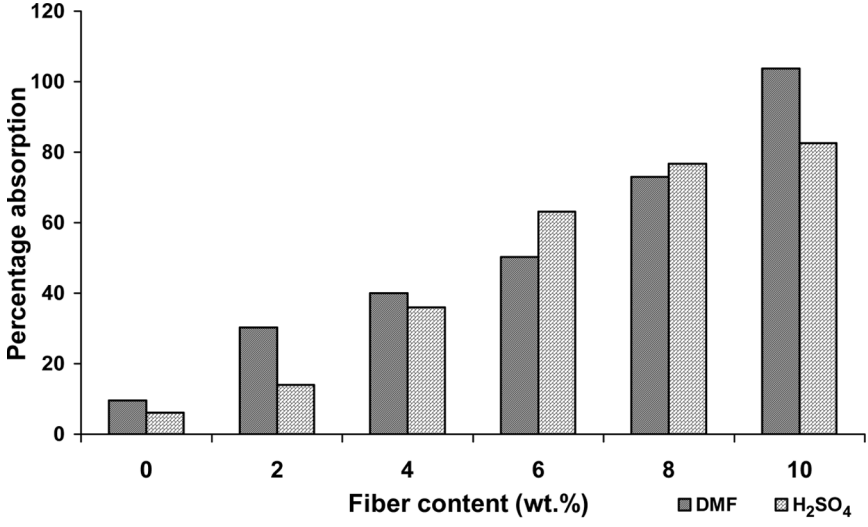

(b)

FIGURE 6 (a) Percentage absorption of low absorbing chemicals on different epoxy composite samples. (b) Percentage absorption of high absorbing chemicals on different epoxy composite samples.

Chemical Absorption

The chemicals can be divided into two types low absorbing and high absorbing, depending on their absorption within the composite. The low absorbing chemicals were characterized with percentage absorption ranging from 0.023 to 4.34 include xylene, toluene, CCl_4, 2 N NaOH, 1.75 KOH, conc. HCl, 54% HCl, and 15% NaCl, whereas high absorbing solvents were characterized with percentage absorption of 9.5 to 105% and include DMF and conc. sulphuric acid. Absorption for the low absorbing and high absorbing solvents for fiber reinforced epoxies is shown in Figures 6(a) and 6(b), respectively.

During the absorption period the samples dipped in Dimethyl Formamide (DMF) showed the maximum swelling, which increased with increase in fiber content. The absorption in DMF was 9.52% for the neat sample and 103.71% for the sample containing 10% fibers. DMF was followed by sulfuric acid where the absorption ranged from 6.01 to 82.54%.

Samples dipped in acetone and methylethylketone showed cracks on the surface, which may be due to the crazing effect. The samples were completely destroyed in pyridine, nitric acid, and conc. sulphuric acid. The other chemicals did not show any significant effect on the surface of the composites and the absorption was intermediate and limited to 4.34%.

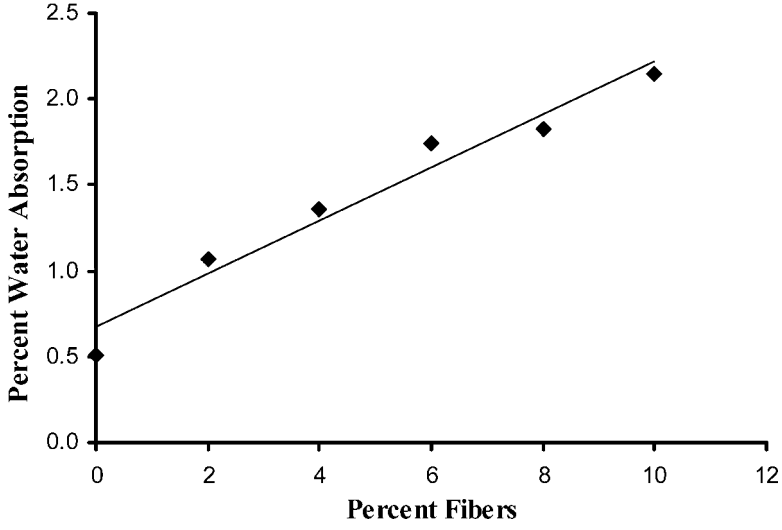

FIGURE 7 Percentage absorption of water on different epoxy composite samples prepared.

Chemical absorption increased with increasing amount of glass fibers. The reason can be attributed to the increased clustered fibers and void spaces with increasing fiber content, thus resulting in decreased interfacial bonding between the fibers and matrix. As the fiber content increases, viscosity of polymer matrix increases, resulting in possibility of air entrapment in the matrix (although this possibility was ruled out in SEM micrographs). The voids in the matrix act as host for the water and chemical pockets in the composites, thereby increasing the absorption values.

The resistance to moisture depends on a number of factors including chemical nature of the polymer matrix, fillers used in composite manufacturing, degree of cure of the sheet [30], voids, and porosity. Water sorption behavior is also a function of the loading of fillers and additives, activity of water in the matrix and concentration of water soluble and water insoluble low molecular weight compounds [31]. The water absorption by glass/epoxy composite samples has been reported separately in Figure 7 and it observed the same trend as that for other chemicals. The highest absorption was in the sample containing 10% glass fiber content. Pure resin sheet absorbs minimum amount of water, that is, 0.51%, which increases up to 2.15% for the sheet containing 10% fibers.

CONCLUSIONS

In this study, the effect of chopped strand glass fiber content on mechanical and chemical absorption behavior of the polymer composites was investigated and the following conclusions were derived:

1. Tensile strength and flexural modulus increases with increase in fiber content and was maximum for 10% fibers. The study could not be extended beyond this fiber fraction because of limitations in processability.
2. Scanning electron micrographs reveal good bonding and uniform dispersion of glass fibers within the matrix.
3. According to absorption studies, absorption increases with increase in fiber fraction and it was maximum for DMF. The samples were completely destroyed in conc. sulfuric acid, pyridine, and nitric acid.

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