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FABRICATION AND CHARACTERIZATION OF TiO_2 PARTICULATE FILLED GLASS FIBER REINFORCED POLYMER COMPOSITE

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Abstract. A new, hybrid polymer composites were fabricated with random oriented e- glass fiber in unsaturated polyester resin matrix. Titanium oxide (TiO_2) was used as reinforcement material and kept 10 wt.% constant. The fiber content was varied as 20 wt.%, 25 wt.%, 30 wt.%, 35 wt.%, and 40 wt.%. Unsaturated polyester resin was used as bonding agent. The composites were prepared with two different fiber lengths of 3 cm and 5 cm by hand layup method. Experiments were conducted to determine tensile strength, impact strength, hardness and chemical resistance. The results showed that the combined reinforcement effect yield the better mechanical properties with increased fiber length and particulate material. The chemical resistance of the composites was analyzed by weight loss method. It was found that the chemical resistance was more pronounced in 5 cm fiber length composites. The SEM image observation of the cross section has found the pull effect to cause the defect of the fiber in the polyester resin matrix.

1. Introduction

Composite is a heterogeneous material created by the synthetic assembly of two or more materials, one a selected filler of reinforcing material and the other a compatible matrix binder. The binder and the filler have two very different properties but when combined together form a material with properties that are not found in either of the individual materials.

The matrix is responsible for the surface finish of composite materials and its durability. Its main function is to bind the reinforcement together and act as a medium to distribute any applied stress that is transmitted to the reinforcement. Fibers are widely used in aerospace, automotive [1], marine and industries. Sometimes fibers were used in house hold applications [2]. The main reason is the cost Vs properties always good to prefer. Fiber components may be fabricated by different methods like resin transfer molding [3] or hand layup method.

The particles are generally added to reduce the wear rate and improve the bonding strength of composites. The properties of the composites were improved with addition of filler materials [4-8]. Noticeable improvements were obtained by addition of inorganic minerals of little weight percentage in polymer matrix composites [9-10]. Adhesion of particulate in the matrix was improved with coupling agents and the properties of composites were considerably improved due to higher bond strength [11]. The results were analyzed that sudden improvements in mechanical properties can be achieved by the addition of even small weight percentage of particulate in polymer matrices [12]. The structural properties can be improved with increase in particulate loading [13]. $CaCO_3$ was filled with the composites and the toughness was improved linearly [14].

In this work, short 3 cm and 5 cm e-glass fiber reinforcement in unsaturated polyester resin matrix was produced with titanium oxide (TiO_2) filler material. The tensile strength, impact strength, hardness and chemical resistance tests were carried out and their performances were evaluated. The micro structure of the composites with good tensile strength and poor tensile strength were studied by SEM images.

2. Experimental details

2.1. Materials. The E-glass fiber with random orientation was used as a reinforcing material in the unsaturated polyester resin matrix. The unsaturated polyester resin was used for ease of handling applications and enhances the curing property [15]. Polyester resins are unsaturated resins formed by the reaction of dibasic organic acids and polyhydric alcohols. The properties of unsaturated polyester resin are shown in Table 1.

Table 1. Properties of polyester resin.

Sl. No.	Properties	Value
1	Appearance	Colorless to pale yellow liquid
2	Specific gravity	1.12+0.01 g/cm^3
3	Viscosity	450+50
4	Tensile Strength	22 MPa
5	Flexure Strength	40 MPa

Fiber and resin were procured from Seenu and Company, Coimbatore, Tamilnadu, India. Methyl ethyl ketone (MEK) ($CH_3C(O)CH_2CH_3$) and cobalt (ii) naphthenate were used as accelerator and catalyst respectively. Titanium oxide was used as filler material in this hybrid composite. Physical properties for titanium oxide are given in Table 2.

Table 2. Physical properties of titanium oxide.

Sl. No	Properties	
1	Molar mass	79.866 g/mol
2	Appearance	White solid
3	Density	4.23 g/cm3
4	Melting point	1843 °C
5	Boiling point	2972 °C

2.2. Specimen preparation. The mould was cleaned, polyester resin was mixed with accelerator (1.5 wt.%) and catalyst (1.5 wt.%). The titanium oxide (TiO_2) particulate was added to this mixture and stirred well. The resin mixture was laid up uniformly on to the mould. Composites of five different compositions with various content of e glass fiber of 20 wt%, 25 wt.%, 30 wt.%, 35 wt.%, and 40 wt.%, along with particulate content (10 wt.%) with resin matrix were made with 3 cm fiber length and 5 cm fiber length. The mould was closed and the composite material was pressed uniformly for 24 hours at room temperature for curing. After curing, composite specimens were cut according to the ASTM standards. The designations of composites are shown in Table 3.

Table 3. Designation of composites with different wt.% of fiber content.

Sl. No	Designation of composites	Fiber content, wt.%	Matrix, wt.%	Particulate, wt.%
1	C1	20	70	10
2	C2	25	65	10
3	C3	30	60	10
4	C4	35	55	10
5	C5	40	50	10

2.3. Mechanical characterization. Tensile test was performed on a Shimadzu AG-IS 50 KN Autograph Universal testing machine according to ASTM D638. The impact test was performed according to ASTM D256. Hardness of composite materials was measured using Brinell hardness tester. For statistical purpose, a total of three samples for each tests were carried out at room temperature. The chemical test was carried out in 2N of concentrated H_2SO_4 and HCL.

3. Results and discussion

3.1. Tensile strength. Tensile strength of the composite materials having fiber lengths of 3 cm and 5 cm are shown in Fig. 1 as per the designations given in Table 3. The tensile strength of the composites increases with increase in weight % of fiber and also is changing length of the fiber with constant particulate loading up to 35 wt.% and after that it decreased. It may be for the matrix materials which are strongly interacting with fibers and filler materials. Break and peak load of the composite materials having a fiber lengths of 3 cm and 5 cm are shown in Tables 4 and 5. The tensile test results shows that the hybrid composite made using fiber having fiber length of 3 cm and 5 cm length possess highest tensile strengths of 0.105 GPa and 0.143 GPa at 40 wt.% and 35 wt.%, respectively.

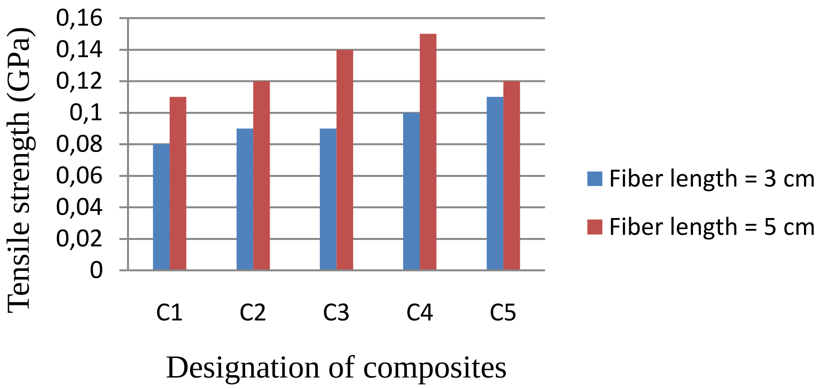


Fig. 1. Tensile strength of the composite fiber lengths of 3 cm and 5 cm.

Table 4. Tensile strength values for 30 mm fiber.

Designation of composites	Peak load in KN	Breaking load in KN
C1	4.69	4.6
C2	5.31	5.2
C3	5.46	5.35
C4	5.82	5.7
C5	6.33	6.2

Table 5. Tensile strength values for 50 mm fiber.

Designation of composites	Peak load in KN	Breaking load in KN
C1	6.32	6.2
C2	7.04	6.8
C3	8.3	8.1
C4	9.2	8.9
C5	7.36	7.2

3.2. Impact strength. The impact energy values were directly found out from charpy impact test machine. The impact strength for the specimens is given in Table 6. It shows that impact strength of hybrid composites improves with increase in fiber and particulate loading. The impact test results show that the hybrid composite made using fiber having a fiber length of 3 cm and 5 cm length posses highest impact strengths of 138.3 J/sq.cm and 186.6 J/sq.cm at 40 wt.%. Table 7 shows the energy required for breaking the composite specimens.

The result reveals that the impact strength of the 5 cm fiber length composites were quite appreciable than 3 cm fiber length.

Table 6. Impact strength values of composite having 30 mm and 50 mm fiber length.

Designation of composites	Impact strength values for 3 cm fiber length, J/sq.cm	Impact strength values for 5 cm fiber length, J/sq.cm
C1	66.6	127.5
C2	86.6	140
C3	111	161.6
C4	116	173.3
C5	138.3	186.6

Table 7. Impact values absorbed from charpy impact test machine.

Designation of composites	C1	C2	C3	C4	C5
Energy required for brake the specimen in Joules (Average of three samples)	3 cm fiber lenth				
	40	52	67	72	87
	5 cm fiber length				
	78	84	97	108	112

3.3. Hardness. The Brinell hardness values for 3 cm length of fiber and 5 cm length of fiber are shown in Fig. 2. The hardness of the composite increases with increase in weight % of fiber and particulate, it may be for the polyester resin made tight bonding with the fiber and titanium oxide powder to resist the penetration. The result reveals that the hardness increases with increased weight % of fiber and particulate loading in the e-glass based polymer composite specimens up to 40 %.wt. The composite made using fiber having an aspect ratio of 3 cm and 5 cm fiber lengths posses the highest hardness values of 25.6 BHN and 35.3 BHN, respectively.

3.4. Chemical resistance analysis. The Chemical resistance test was carried out by weight loss/gain method with H_2SO_4. The values obtained from the test was recorded and examined. The weight loss of various combinations of 3 cm fiber length and 5 cm fiber length of hybrid composites with 2N of concentrated hydrochloric acid is given Table 10 and Table

11. The result reveals the 5 cm fiber length of polymer composite has more chemical resistance with HCL.

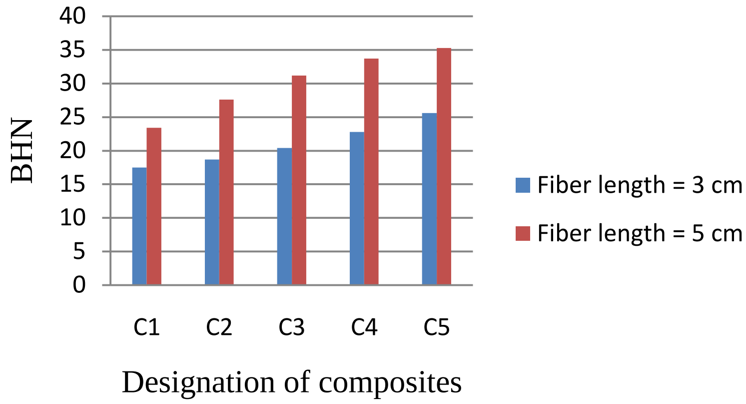


Fig. 2. Brinell hardness values for 3 cm length of fiber and 5 cm length of fiber composites.

Table 10. Chemical test of the fiber composite with 3 cm length of fiber in H_2SO_4.

S No.	Designation of composites	Weight before the chemical test, g	Weight after the chemical test, g	Weight loss, g
1	C1	16.636	16.523	0.113
2	C2	17.130	16.981	0.149
3	C3	17.651	17.456	0.195
4	C4	18.252	17.966	0.286
5	C5	18.687	18.506	0.181

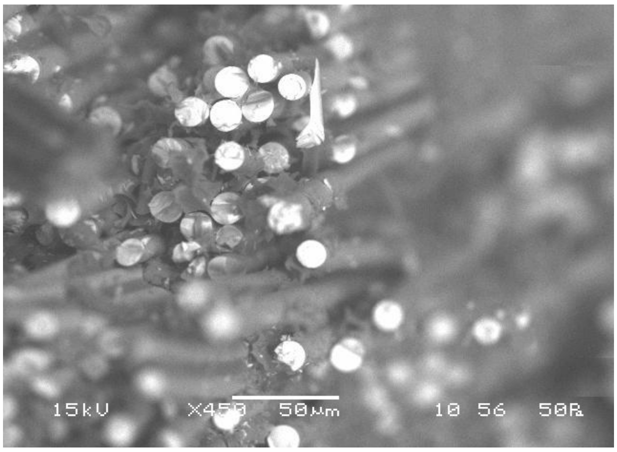
Table 11. Chemical test of the fiber composite with 5 cm length of fiber in H_2SO_4.

S No.	Designation of composites	Weight before the chemical test, g	Weight after the chemical test, g	Weight loss, g
1	C1	17.432	16.545	0.887
2	C2	17.677	16.553	1.124
3	C3	18.078	17.235	0.843
4	C4	18.481	17.663	0.818
5	C5	19.829	18.645	1.184

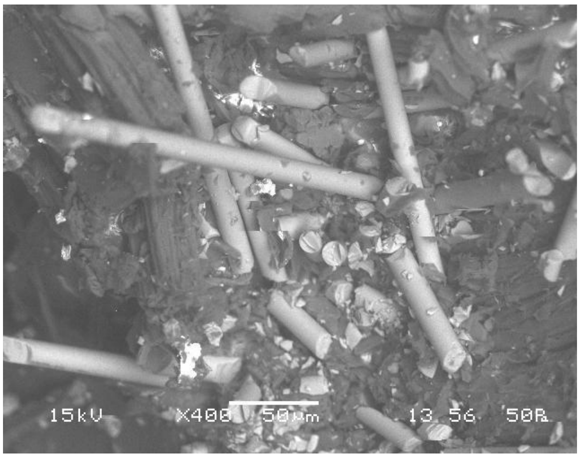
The results from the chemical test showed that the resistance to chemicals of GFRP hybrid composites improved with increase in fiber and particulate loading. The characterization of the composites reveals that the chemical resistance increased with the increase in the particulate content in the hybrid composites. The dual reinforcement and characterization with all these properties, it can be implemented in automotive engine components. Also it is noticed that there is significant improvement in the mechanical properties of composites with the increase in fiber and constant particulate loading.

SEM analysis. In scanning electron microscope (SEM), micrographs have a large depth of field yielding a characteristic three-dimensional appearance useful for understanding the surface structure of a sample.

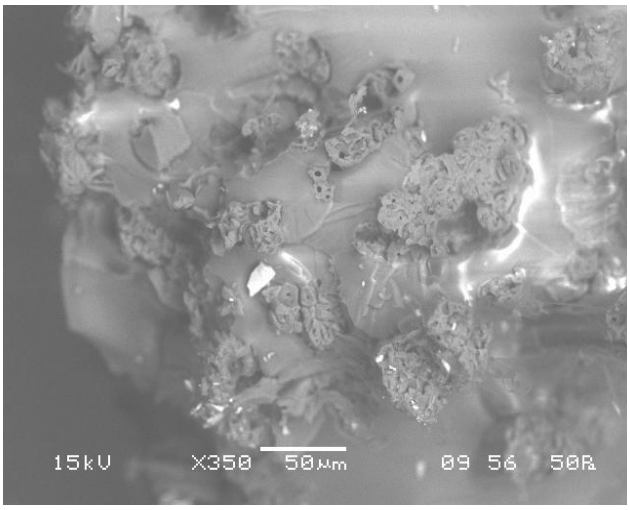
Glass fiber composite in Fig. 3 (a) has shown enormous amount of fiber pull out. This was mainly because of the weak bonding between the constituent glass fiber and the polymer resin matrix. The fabrication process that was used could be the reason for uneven distribution of the stress being applied. The Figure 3 (b) shows the weak bonding between the matrix and the fibers for glass fiber polymer composite. It shows the cracking of the matrix as well as the deboning between both the phases. This could be due to the residual stress present while curing as well as due to the fabrication techniques used. The Figure 3 (c) shows the large extent of the fiber fracture. The amount of stress being applied was unable to be sustained by the fibers when distributed on them by the matrix due to the inability of the formation of strong interfacial bonds.



(a)



(b)



(c)

Fig. 3. SEM image on (a) pull out failure of composite, (b) weak bonding of composite and (c) extent of the fiber fracture.

The glass fibers were in aptly held by the matrix which was followed by the rupture of the fibers. It may be due to the localized stress and strain fields in the fibrous composite.

4. Conclusion

The fabrication of hybrid reinforced polymer composites were carried out by hand layup method. The experimental investigations on the composite specimens were carried out to determine the tensile strength, impact strength, hardness and chemical resistance. It was observed that the inclusion of titanium oxide and increase of fiber length resulted in composites with increased tensile strength, impact strength, hardness and chemical resistance. The effect pull load on the composites was studied with scanning electron microscope images.

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