Mechanical and wear properties of epoxy matrix composite reinforced with varying ratios of solid glass microspheres

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Abstract. This research work is carried out to study the mechanical and wear behaviour of inorganic particle filled polymers. Epoxy matrix filled with varying ratios of Solid glass microspheres (SGM) were fabricated using mechanical stirring process. The weight percentage of SGM were varied from 0 – 30, with a step size of 5wt%. Effect of weight percentage of SGM particles on bending strength, compression strength and compression modulus of the composites was evaluated. The Wear resistance of the composites against En-32 steel disk, was evaluated in terms of mass loss for various ratios of SGM using pin-on-disk test rig. Besides these, density of the composites was also evaluated. Mechanical and wear properties of the composites were improved by adding suitable percentage of the filler. Moreover density increased with the increase in percentage of SGM particles in the composites.

1. Introduction
The usage of polymers especially epoxy resin is of considerable significance in the engineering field for a substantial period of time. Several components which are made of materials based on epoxy have demonstrated outstanding properties like thermal conductivity, mechanical properties and electrical properties [1]. Mechanical properties of these composites are of major significance as these aspects play quiet an important role in material and industrial refurbishing in copious fields, such as electronics, aerospace, automotive, and biotechnology applications. Epoxies, generally, have small shrinkage upon cure, good mechanical and thermal properties, significant chemical and corrosion resistance, good electrical insulating properties and exceptional adhesion to various substrates. Also, it is the ability of epoxies to be processed under a variety of conditions which make it apposite candidate as matrix materials for superior composites applications [2]. The curing reaction of the epoxy occurs on adding a hardener into the epoxy resin. For the duration of curing, the molecules form cross-links with others and grow in a 3-dimensional network that ultimately forms a rock-solid epoxy resin [3].

The exploit of particulate fillers to alter the properties of a material for instance a epoxy resin, is well recognized and variety of fillers are there including carbon blacks, mineral powders and graphite, ceramic particles and metallic powders. Study of the effect of inclusion of inorganic particulates on the composite’s mechanical properties is crucial for designing the particulate reinforced composites. Furthermore, to study the hybrid organic – inorganic materials, an understanding of inorganic particulate reinforced polymers is important [4].

Fracture behaviour and mechanical properties of the modified polymers including epoxy have been previously investigated [5]. The adhesion between the matrix and the particle strongly influence the toughness of the polymers reinforced with particles. The inclusion of these solid particulates results in a constant rise in the modulus and toughness values. Adding the particle filler of the spherical shape – glass beads improves the adhesive bond strength [6]. The studies based on mechanical properties of syntactic foams are mainly focussed on incorporation of high content of glass microspheres as it
improves specific strengths [7,8]. Authors have studied the strength of the glass/epoxy composites and its relation with the density, with varying volume fraction of glass particles [9]. Epoxy resin has poor temperature resistance and the soda-lime-borosilicate glass particles possess excellent resistance to temperature because of their chemical composition[10].

The addition of solid glass microspheres to epoxy resin results in the improvement of elastic modulus, yield strength (compressive), and fracture surface energy but on the other side it decreases the strength, both compressive and bending, in comparison to the matrix without the glass particle [11]. Glass micro beads have a very high recyclability potential and can substitute many metals or composites in various industries. Thus, the use of glass micro-bead reinforced composite material does not impose any potential harm to the ecosystem and could be considered as a green material [12]. The bending strength of the glass microspheres reinforced epoxy composites increases when the particles are added in small fraction, but the property decreases with increase in particulate loading. Further the interaction and adhesion between the matrix and solid glass particles (the reinforcement) can be enhanced by using a suitable silane coupling agent [13].

Hence, the present study focuses on the effect of the contents ratio of SGM on bending and compression strength, compression modulus, wear and density of the composites, and to have better understanding of the fabrication with a view to finding out the optimum percentage by weight of the glass microspheres that can be added to the composites.

2. Experimental

2.1. Raw Materials

The matrix used for the study is a DGBEA based epoxy matrix LY556 cured by using an amine hardener HY951. The resin and the hardener were mixed in 10:1 ratio as suggested by the supplier (Singhal Chemicals, India). Density of the resin is 1.1-1.2. g/cm³ and that of the hardener is 0.98 g/cm³. The curing time of the epoxy/hardener mixture was 24 hours at room temperature.

For the present study, the reinforcing material was solid glass microsphere (soda lime glass) with an average particle size of 125 μm supplied by Quality spare centre, India. Density of the glass particles used is 2.52 g/cm³ and Young’s modulus is 72 GPa. SGM is considered a promising reinforcement for the development of epoxy based composites due to its isotropic properties, uniform shrinkage in all directions, very low stress concentration around the particles owing to their spherical shape, and relatively small increase of the flow viscosity of polymers [14].

2.2. Mould Preparation

Steel plate was used for the preparing moulds for the fabrication of the epoxy/SGM composites of desired shape and size. Cavities were made in the steel plate for preparing samples for bending and compression test according to according to ASTM standards (table 1).

For wear test, the specimen was in the form of a cylindrical pin with 6mm diameter and 30mm length (ASTM G99-95).

Table 1. The dimensions, of specimens for bending and compression test

| Test       | Length (mm) | Width (mm) | Thickness (mm) | ASTM     |
|------------|-------------|------------|----------------|----------|
| Flexural   | 80          | 13         | 5              | D790-2015|
| Compression| 10          | 10         | 5              | D695     |

Mould was designed by using Auto-Cad software. The design is later transferred to develop Computer Numerical Control (CNC) codes which were used in the machining of the steel plate. The mould after machining and polishing processes is shown in figure 1. The finished mould required high consideration to ensure a smooth and easy removal of the fabricated composite when it gets cured in
the mould. Initially six layers of wax were required to make sure that the surface is completely covered. After that only one or two layers of wax were adequate to easily remove the cured composite from the mould.

2.3. Fabrication of Composites
Different composites were prepared by varying the weight percentage of the filler materials (table 2). The fabrication of composites consists of following steps:
1. Mixing the Epoxy resin and hardener in the ratio 10:1 using a mechanical stirrer (1200 rpm).
2. Mixing the SGM particles with the epoxy resin/hardener mixture.
3. Pouring the mixture into mould.
4. Curing of the mixture at room temperature for 24 hrs.
5. Heating the cured composites in vacuum oven at 75°C for 7 hrs.

![Figure 1. Mould for bending and compression specimens](image)

Table 2. Compositions of Epoxy-SGM composites

| Set of specimens | SGM content in composite, wt% |
|------------------|-------------------------------|
| 1                | 0                             |
| 2                | 5                             |
| 3                | 10                            |
| 4                | 15                            |
| 5                | 20                            |
| 6                | 25                            |
| 7                | 30                            |

The epoxy was first mixed with the hardener in the 10:1 ratio using a mechanical stirrer at 1200 rpm for 4 minutes at room temperature. SGM particles were then added to the mixture which was again mixed by stirrer for 4 minutes. The mixture was then poured into the mould and left for curing at room temperature for 24 hrs. Cured composites were then exposed to elevated temperature (75°C) for 7 hours in vacuum oven.

3. Results and Discussion
To study the mechanical and wear behaviour of the fabricated SGM/Epoxy composites bending, compression and pin-on-disk wear tests were performed.

Figure 2 illustrates the effect of weight fraction of SGM on the bending strength of the composites. Three point bend test was conducted to determine the bending strength using UTM (Bliss) with strain.
rate of 2mm/min. The bending strength of cured resin used in this study was 65 MPa which was greater than all the composites with different weight percentage of SGM but for the 5 wt % composition the strength was more (66.26 MPa). The strength was reduced to 64.91 MPa at 10% by weight of SGM and retained the same strength value upto 15 wt% composition. On further increasing the particulate loading to 20wt% the bending strength reduced to 59.94 MPa and it remained so up to 30wt% composition. In general, the bending strength reduced with increasing the weight percentage of SGM particles. Thus, the highest values of bending strength for the composite in this study were obtained with 5% particulate loading. This could be because the resin could encapsulate the small amount (up to 5 wt %) of SGM particles easily. Similar results were obtained by Ku et al.[13].

Zee et al. [15] used epoxy resin (EPON 828) and alumina powder (XA 3500) to prepare epoxy composites by varying the volume percentage of the alumina powder. For curing the resin, different anhydrite hardener was used. The bending strength of neat epoxy resin varied from 96.6 MPa to 117.3 MPa. For the 10% particulate loading the values of bending strength slumped to a range of 55.2–62.1 MPa but restored to values close to those of the neat resin. The bending strength of cured neat resin used in the study by Zee et. al. was more when compared with that of the epoxy used in this study. The trend of the curves of the bending strength of the composites of the two studies was different and this may be due to the different fillers used. This may be due to the degassing and devolatization processes used by Zee et al.

![Figure 2. Effect of wt% of SGM on bending strength of SGM/epoxy composites](image)

Figure 3 shows the compressive strengths of epoxy composites with varying weight percentage of SGM. The compression behaviour of the composites was opposite to that of bending. The compressive strength of the cured neat resin was 112 MPa, which increased with addition of 5wt% of SGM particles (115.921 MPa) of composite. The strength values were reduced when the particles were added in 10wt%. But on further addition of the fillers, the compressive strength increased with increase in percentage of the particles. The maximum value of the compressive strength was obtained for 30 wt % of the glass particles. Table 3 shows the values of compressive and bending strength mentioned above.

Compression modulus of the composites was also evaluated for the SGM/epoxy composites (table 3). The modulus for neat epoxy was 1.38 GPa which was reduced on addition of the 5wt % SGM particles. The value of the modulus rose to 1.47 GPa on adding of 10wt % particles. On addition of 15 wt% particles, the modulus value was reduced and came close to that of neat epoxy, which was further reduced on increasing the wt % to 20. The maximum value of the modulus for the SGM/epoxy composite used in this study was for the 25wt % composition of SGM.

![Figure 3. Effect of wt% of SGM on compression strength of SGM/epoxy composites](image)
Table 3. Effect of wt% of SGM on bending and compressive strength of SGM/epoxy composites

| Sample Number | Wt% of Epoxy | Wt% of SGM | Maximum Bending Strength, MPa | Max. Compressive Strength, MPa | Compressive modulus, GPa |
|---------------|--------------|------------|-----------------------------|-------------------------------|--------------------------|
| 1             | 100          | 0          | 65 ± 3                      | 112 ± 4                      | 1.38 ± 0.8               |
| 2             | 95           | 5          | 66.26 ± 2                   | 115.92 ± 2                   | 1.35 ± 0.7               |
| 3             | 90           | 10         | 64.91 ± 4                   | 115.78 ± 5                   | 1.47 ± 0.8               |
| 4             | 85           | 15         | 64.48 ± 2                   | 117.86 ± 2                   | 1.34 ± 0.6               |
| 5             | 80           | 20         | 59.94 ± 5                   | 119.86 ± 3                   | 1.27 ± 0.7               |
| 6             | 75           | 25         | 59.89 ± 3                   | 121.30 ± 2                   | 1.68 ± 0.9               |
| 7             | 70           | 30         | 59.80 ± 4                   | 121.66 ± 4                   | 1.57 ± 0.5               |

Effect of weight percentage of glass particulates on the wear resistance and friction coefficient was evaluated using pin-on-disk dry wear test. A 6 mm diameter cylindrical specimen was used for the pin. Test conditions were a speed of 955 rpm, with an applied load of 20 N, sliding distance was 1000 m and track diameter was 10 mm. For the wear test the En-32 was used for the disk. The wear resistance was calculated by weight loss according to ASTM G99-95 standard. The friction coefficient was measured using the same standard. Highest weight loss was for the 5wt % composition of SGM (figure 5). With increase in weight percentage of SGM the loss reduced and the minimum weight loss was observed with 15wt% of SGM particulates. Further addition of the particles increase in weight loss was observed. Similar trends were observed for the friction coefficient also. The highest wear resistance and minimum coefficient of friction was therefore observed with 15 wt % composition of the composites. This could be because of better anchorage of particles with the matrix for this particular composition.

![Figure 4. Compression modulus of SGM / epoxy composites with varying wt% of SGM](image)

![Figure 5. Effect of wt% of SGM on wear rate and friction coefficient of SGM/epoxy composites](image)

The density of glass-epoxy composite is calculated by rule of mixture:

\[
\text{Density} = \rho_m W_m + \rho_r W_r
\]

Where, \(\rho\) and \(W\) are density and weight fraction respectively. Subscripts \(m\) and \(r\) represents the matrix and reinforcements respectively.

The density of epoxy used in the study (LY556) was 1.15 g/cm\(^3\) and that of SGM particles was 2.52 g/cm\(^3\).
| Wt % of SGM | Density, g/cm³ |
|-------------|----------------|
| 0           | 1.15 ± 0.03    |
| 5           | 1.2185 ± 0.02  |
| 10          | 1.287 ± 0.04   |
| 15          | 1.3555 ± 0.01  |
| 20          | 1.424 ± 0.02   |
| 25          | 1.4925 ± 0.04  |
| 30          | 1.561 ± 0.03   |

Density of LY = 1.15 g/cm³, SGM 2.52 g/cm³

Table 4. Effect of wt% of SGM on density of SGM/epoxy composites

The effect of SGM on the density of the prepared samples as a function of wt% of SGM is shown in table 4. As can be seen from the table, the weight percentage of SGM particles has significant effect on the density of the prepared composites. Increase in weight percentage of the glass particles increases the density of the composites (figure 6). The maximum density (1.561 g/cm³) of the composite was obtained with 30% of SGM.

Figure 6. Effect of wt% of SGM on density of SGM/epoxy composites

4. Conclusion
Epoxymatrix composites reinforced with SGM were synthesized successfully using mechanical stirring method. Mechanical properties like bending strength, compression strength, compression modulus, wear properties (wear resistance and coefficient of friction) and density has been evaluated for the epoxy composites reinforced with varying weight percentage (0-30) of solid glass microspheres. For all the tests, five samples of each composition were tested.

The following are the significant observations made from the above investigation:

- The bending strength of the SGM/epoxy composites decreased with increasing particulate loading, except at 5 wt %, and the trend was similar to other studies with epoxy resins but different fillers. This behaviour is because resin encapsulated the small amount (5 wt %) glass powder particles easily.
- Compressive strength of the composites increased with increasing weight percentage of the SGM particles in the composites. Maximum value of compression strength was obtained with 30 wt% composition (8.6% above the compressive strength of the cured epoxy).
• The modulus of compression however varied differently with increasing weight percentage of glass particles, with maximum value obtained at 25 wt%.
• Density varied linearly with the weight percentage of particles. Maximum value of density was 1.561 g/cm³ for 30 wt% composition.
• Maximum wear resistance and minimum coefficient of friction was observed for the composition with 15 weight percentage of the particles.

Thus, the mechanical and wear properties of the epoxy can be improved by including solid glass microspheres in suitable percentage.

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