CHAPTER IV

EPOXY FLY ASH AND EPOXY CENOSPHERE PARTICULATE COMPOSITES

Epoxy – Fly ash (EF) and Epoxy – Cenosphere (EC) particulate composites were prepared using casting method with varying amount of fly ash and cenosphere from 0 to 17.5 wt %.

Fly ash particulates in desired ratio were added to epoxy resin. The mixture was stirred for half an hour followed by ultrasonication for a period of 20 minutes. Hardener K6 was added drop-wise with gentle stirring. Mixture was then slowly poured into the mould. It was left to cure at room temperature for 15–20 h followed by post curing at 80°C for 4 h. The cured rigid composite was released from the mould and the edges were machined. These composites were characterized for their physical, morphological, mechanical and frictional properties. The present chapter includes results of prepared composites.

PART – A

EPOXY FLY ASH PARTICULATE COMPOSITES

IV.A1 Density of Epoxy-Fly ash composite

The apparent densities of the epoxy-fly ash composites are shown in figure IV.1. Density of EF composites is found to increase from 1.18 to 1.27 with increase in fly ash percentage from 0 to 17.5%. Since fly ash has higher apparent density as compared to neat epoxy rigid block. Overall density of the epoxy-fly ash composites gets increased with percentage of fly ash.
IV.A2 Rockwell Hardness of Epoxy-Fly ash composite

The hardness of regid blok epoxy and E-F composites was evaluated using Rockwell hardness testing machine with 100 kg load and ¼ inch diameter steel ball indenter. The dwell time for the hardness measurement was 10 seconds. Hardness of the composites depends on the percentage of reinforcing particles present in them. Figure IV.2 shows the hardness of the pure epoxy and E-F composites in cast condition. As the amount of fly ash is increasing the hardness of the composite is increasing. This increase was observed from 109.5 HRL for pure epoxy to 113.5 HRL for 17.5 % E-F composite. This could be due to the presence of fly ash particulates which consist of major alumina and silica, both hard in nature.
IV.A3 Compressive strength of E-F composite

Figure IV.3 shows the variation in compressive strength with the percentage of fly ash. For as such epoxy the strength was found to be 197 MPa. Addition of fly ash increases the compressive value but for certain amount of percentage only as shown in figure IV.3. As, the percentage of fly ash increase from 2.5% the strength tends to decrease. This can be explained with the contrast that the higher amount of fly ash particles hinders the bonding between fly ash and epoxy which also leads to slight increase in porosity. Further as amount of fly ash particles increases, the possibility of agglomelation is higher, which may lead to less surface area for the bonding with matrix. This agglomeration phenomena may lead to more paricle – particle adhesion as compared to particle – matrixadhesion and hence premature failare occurred during the measurement.
Figure IV.3 Compressive strength of Epoxy-Fly ash composite

IV.A4 Friction coefficient of composites

Tribological measurement and tribological study was carried out for as such epoxy, EF composite with different percentage of fly ash at different load conditions. Load is varied as 1N, 2N and 5N.

Figure IV.4 shows change in friction coefficient of neat cured epoxy with increase in load condition. It is found that the friction coefficient decrease with increased load. This decrease is due to soft nature of epoxy materials.

When load increases, more and more friction force is applied on epoxy material. As there is no filler material, epoxy gets removed easily from the surface resulting in decrease in friction value. For 1 N load condition, friction value of epoxy was found to be 0.161 whereas for 5 N load condition, the value decreased to 0.129.
Figure IV.4 Friction coefficient for as such epoxy at 1N, 2N and 5N load

Figure IV.5 Friction coefficient values for different % of fly ash composite at 1N, 2N and 5N load
Figure IV.5 shows the coefficient of friction of Fly ash – epoxy composites with increase in fly ash content. As seen from the Figure, coefficient of friction value tends to increase from 0.161 to 0.323 with percentage of fly ash. Fly ash generally consists of SiO₂ and Al₂O₃ particles which are hard ceramic particles. As the percentage of fly ash increases, the percentage of ceramic constituents also increases which act as barrier for the static partner to slide easily on the surface of the epoxy matrix. Therefore, presences of hard fly ash particles tend to change friction value [1].

For higher load condition from 2 N to 5 N there is a decrease in coefficient of friction value from 0.149 to 0.129. The reason for decrease in friction value is due to the wear of epoxy matrix from the surface at higher rates. This leads to decrease in epoxy content of the epoxy-fly ash composites which increases with increase in fly ash percentage. As load increases, more and more fly ash particles come in direct contact with static partner. To show the relative coefficient of friction for varying load, the results are compiled in Figure IV.5.

At the end of each test, fly ash particles became smaller in size due to rubbing against mating surfaces. Once the smaller sized particles of fly ash were formed and spread in between the mating surfaces, a film of uniform thickness of these particles started to form, which caused approximately steady weight loss of epoxy matrix[2]. It was also found that the generation of fly ash particles in between the sliding surfaces and the wear rate of materials depends on the amount of fly ash incorporated in the epoxy resin. This is because of more shearing and ploughing of the surface.

It is also observed from Figure IV.5 that, there is some fluctuation in coefficient of friction at high load. This is because the surface of the sample revealed that soft epoxy matrix in the composites were plastically deformed and smeared over the contact surface by generating excessive adhesion on the counter surface, resulting
in a smooth transfer film. If applied load increase to higher value, friction layer gets removed from the surface, thereby increasing friction between specimen composite surfaces.

The reason for this reduction in coefficient of friction is attributed to the presence of the smeared epoxy fly ash layer at the sliding surface which may act as a solid lubricant. This smeared layer becomes thicker with the increase in applied load and become highly adhering and compacted [3].

Figure IV.6 shows friction curve value of different epoxy-fly ash composites with 5N load condition. As it can be seen from the Figure for as such epoxy material the friction coefficient is very low as compared to epoxy-fly ash composite. The initial period of the curve shows some non-linear variation which is due to the surface roughness or micro-cracks present on the surface of the epoxy-fly ash composites. All the curves tend to increase for initial few times and after that it acquires a steady path. Only for epoxy-fly ash composites containing 5 % of fly ash shows little heap-type mark which can be due to improper polishing or machining of epoxy – fly ash composites prior to friction test. Lastly, from the curve it can comment that as the percentage of fly ash increases, coefficient of friction increases.
Figure IV.6 Friction co-efficient values for different wt% of fly ash composites at 5 N load

For EF 15 % fly ash a heap type slight curve was seen during initial stage. As the fly ash particles wore, not only the surface contact between the composite and rotor changed but also did the asperities. Stable $\mu$-value was not seen after some time of friction test.

It was seen that after some time of friction test the curve tends to a linear behavior. This may be due to the removal of upper epoxy phase in that area.
IV.A5 Worn surface of Epoxy-Fly ash composite

![Image of worn surface of Epoxy-Fly ash composite]

**Figure IV.7** Optical images of after friction coefficient for 5 % EF composite

During measurement of coefficient of friction on tribometer there is sliding of Cr6 ball on surface of fly ash filled epoxy composite. At greater sliding distance, smooth surface of composite converted into rough pattern. Figure IV.7 shows optical micrograph of sliding direction and frictional pattern of epoxy-fly ash composite. In 5 % epoxy-fly ash composites the friction pattern gets changed due to fly ash particles. It also shows a magnified view of 5 % epoxy-fly ash composites.

Friction test were performed on EF5 composite with 5N load condition, linear speed and 15 c/s and sliding distance of 200m. The EF5 contains a lot of asperities on the surface. It consists of only two phase, i.e. matrix (epoxy) and reinforcement (fly ash). Therefore, all the friction affects or alterations are due to these two phases. During the initial stage of friction test, friction force is applied on the epoxy phase from the static partner. As, static partner is made of steel cr6 ball which is hard, it tries to eradicate the epoxy matrix. This leads to interaction of fly ash particles with static partner. This creates a smooth surface on the EF5 composite. The nature of surface...
depends on the percentage of fly ash particles present in that friction area. Higher the fly ash percentage more will be the roughness.

As shown in Figure IV.8, the worn surfaces of EF composites are very smooth. The worn surfaces are covered with fine lines of aligned parallel to the sliding direction. During the friction process, epoxy matrix of composites that enwrapped the fly ash is first ground off and exposed on the worn surface. The fly ash exposes on the surface bare the load. They could inhibit the cutting action of micro-convexity on the surface of counterpart to the matrix of composites [4]. Fly ashes strengthen the polymer matrix and protrude out of the rubbing surface during sliding, so that fly ash epoxy composites present good tribological properties [5, 6].

![Figure IV.8 Optical micrograph of 10 % EF Composite](image.png)
IV.A6 Scanning electron microscopy (SEM)

In SEM analysis it has been found that fly-ash particles have been uniformly segregated. These revealed that there is uniform distribution of fly ash particles in the base matrix. It also clearly shows that there was less voids and discontinuities observed in the composite and also there was a good interfacial bonding between the fly ash particles and matrix materials.

Figure IV.9 (A) shows SEM image of as such fly ash. It contains cenosphere. Figure IV.9 (B) shows the as such epoxy after compressive strength. It shows river type pattern. After addition of fly ash compressive strength of E-F composites improves. In figure IV.9 (C) presence of fly ash particles in the matrix inhibit the crack propagation during compressive loading.

Evidence of plastic deformation of epoxy resin can be seen very clearly by the river pattern formation in the matrix (figure IV.9 (B)). The stepped appearance at this end is due to the change in the plane of fracture when it approaches the free surface. The fly ash particles play a key role in the stage of crack propagation. To examine the role of ash particles a SEM micrograph of the composites is shown in Figure C, where the debonding and subsequent displacement of larger sized ash particle is seen. This photo clearly can prove that the debonding is favoured by larger sized particles.
Figure IV.9 (A) As such fly ash

(B) As such hard epoxy after compressive strength

(C) Epoxy-fly ash composite after compressive strength
PART - B

EPOXY CENOSPHERE COMPOSITES

IV.B1 Density of Epoxy-Cenosphere composite

The cenospheres are spherical particles filled with air inside during its formation in coal – combustion unit in thermal power plant. Due to its emptiness, cenospheres are light in weight and less denser than water. The addition of cenospheres replaces volume of epoxy resin and hence the density decreases with addition of cenospheres from 2.5 to 17.5 %. The density of rigid epoxy block is around 1.195 g/cc while addition of 2.5% cenospheres bareing it around 1.185 g/cc i.e. 0.75 % decrease in density.
further addition of cenosphere laead to linears decrease in density up to 15 % then lead to constant value.

**IV.B2 Rockwell Hardness of Epoxy-Cenosphere composite**

Prior to the hardness measurement, the samples surfaces of the composites were polished flat using a sequence of 600, 800, 1000 and 1200 grit silicon carbide papers to produce a flat surface for the indentation test and after polished by alumina powder for shining.

The hardness of regid blok epoxy and E-F composites was evaluated using Rockwell hardness testing machine with 100 kg load and ¼ inch diameter steel ball indenter. The dwell time for the hardness measurement was 10 seconds. Hardness of the composites depends on the percentage of reinforcing particles present in them. Figure IV.11 shows the hardness of the pure epoxy and E-C composites in cast condition. As the amount of cenosphere is increasing the hardness of the composite is decresing. This increase was observed from 114.2 HRL for pure epoxy to 105.7 HRL for 17.5 % E-C composite. This could be due to the presence of cenosphere particulates which consists of majority of the alumina and silica which are hard in nature but it is hollo nature so, hardnesh is decreases. Cenospheres and epoxy resin bonding are not so good. This is evident from Fig. IV.154.
IV.B3 Compressive strength of E-C composite

Compression testing was performed on the test coupons with the help of INSTRON-8300, computer-controlled testing machine. The machine crosshead was programmed to apply the compression load at a constant strain rate of 1.50 mm/min throughout the test by using the built-in software. A minimum of three samples, as
stated earlier, were used for compression tests on any similarly processed samples of different compositions.

Result showed that the increase in the strength of the material is very sharp when filler volume fraction is low. With increase in filler volume fraction, it decreases [7].

Additions of 2.5 wt % cenosphere increase the compressive strength of composite. After addition of more wt % cenosphere the compressive strength of composite decreases may be due to poor bonding of cenospheres with epoxy resin and presence of fraction cenosphere. This is evident from Fig. IV.14. Also the density of composite decreases after addition of more wt % cenosphere.

At such a small volume fraction of cenosphere, when the size of the particle is considerably small, it does not offer any substantial barrier to the deforming epoxy during compressive loading of the specimen. Evidence of plastic deformation of epoxy resin can be seen very clearly by the river pattern formation in the matrix. For load bearing purpose, at the small volume fractions cenosphere particles seem to be ineffective. Dispersion of these particles is good in the structure. Substantial elastic and then plastic deformation has taken place in the matrix materials and small particles in such a low proportion are not able to resist them. On the contrary, due to their regular spherical shape, they may be able to move around easily with the matrix. On the entire fractured surface, broken particle pieces can be seen abundantly.
IV.B4 Scanning electron microscopy (SEM)

Figure IV.13

a. As such epoxy like a river pattern type shape (X 35)

b. As such epoxy like a river pattern type shape (X 150)

c. Epoxy Cenosphere composite
Figure IV.14 SEM micrograph of the epoxy-cenosphere composite after compressive test
In SEM analysis (Fig. IV.14) it has been found that cenosphere particles are uniformly distributed. SEM image of EC composite after the compressive strength. It shows homogeneous mixing of cenosphere particles with epoxy matrix. Cenosphere particles are of different particle size and are evenly distributed in epoxy matrix. There are only two phases present in the figure IV.14. The continuous phase is epoxy matrix and the particles which are embedded in the epoxy matrix are cenosphere particles. Particles show hollow type structures which are light weight cenosphere particles consisting of silica/alumina.

Evidence of plastic deformation of epoxy resin can be seen very clearly by the river pattern formation in the matrix (figure IV.13). The stepped appearance at this end is due to the change in the plane of fracture when it approaches the free surface. The cenosphere particles play a key role in the stage of crack propagation.
Chapter IV

IV.C Bibliography


