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Effect of Mixing Time on Mechanical Properties of Epoxy-Fly Ash Composite

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Abstract

Particulate reinforced polymer composites have been designed to improve the (polymer) composite properties and with reduction of cost. In the present study, fly-ash is used as a filler material. Samples are made with different mixing times i.e. 5, 10, 15, 20, 25 and 30 min. Ultrasonic and magnetic mixing are the two ways to mix the particulate as fillers. The resulting mechanical properties such as flexural strength, tensile strength and impact strength of the composites are evaluated. The SEM and other investigations are been studied.

Keywords: *Fly ash, epoxy, ultrasonic, magnetic*

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INTRODUCTION

Among polymer composites, particulate filled polymer composites are highly accepted due to their light weight, good mechanical properties and low cost. Particulate filled composites find their uses in the chemical, automobile and furniture manufacturing industries [1]. Through reasonable control of fortifying strong particulate stage, choice of network and suitable handling method, composites can be arranged to tailor the properties required for any particular application. In past two decades, ceramic particulate filled polymer composites have emerged as a subject of extensive research. In any case, because of the high cost of routine ceramic fillers, it has ended up critical to investigate the capability of shoddy materials like metallic minerals and mechanical squanders for use in planning molecule fortified polymer composites [2]. Low cost and attractive properties such as light weight, high strength, corrosion resistance and ability to be tailored to specific engineering applications are responsible for the dramatic surge in the use of particle reinforced composite products. The order of the day is to look for more low-cost fillers and where possible, to meet the recycling requirements. Fly ash, a fine powder generated during combustion of coal in thermal power plants is a suitable material to be used as a filler material [3, 4]. Fly-ash is the reinforcement material that is now widely used in the metal matrix composites because of its advantages like low density, low cost, and high wear resistance [5, 6]. Most mineral fillers used in thermoset and thermoplastic composites are ground into fine particles with relatively low aspect ratio. Almost any powdered material can be used as filler, but fly ash on the other hand composed of aluminium and calcium and silicon oxides is harder than other powder particles available at low cost [7]. Various researchers have used fly ash with different thermoset and thermoplastic matrix materials and evaluated the mechanical properties but no attention has been given towards the mixing time and mixing process for preparing the samples.

The vast majority of the inflexible fillers are made of ceramic materials, which have higher thickness than the polymer network. Mechanical properties of these particulate composite systems depend on the matrix properties, filler characteristics and polymer/matrix interface properties. The properties of the interface is dependent on the size of the filler or more specifically its specific surface area [1, 3]. Filler nature or its

constituents, size, shape and particle size distribution significantly influence the properties of composite [8]. The mixtures of particulate filled composite were made by hand stirring. Slight modification in mixing condition is helpful for improvement in mechanical properties. In this paper, mechanical properties of the composite have been studied with respect to mixing condition and mixing time. The results are compared depending on the mixing condition i.e. ultrasonic mixing and magnetic mixing.

MATERIALS AND METHODS

Epoxy resin is chemically named Diglycidyl Ether of Bisphenol A (DGEBA) (LAPOX L-12). A room temperature hardener K-6 (Triethylene Tetra Amine) supplied by Atul India Ltd, Kolkata; is used as the matrix material. It is a medium viscous fluid at room temperature. The density of cured neat resin, as given by the supplier, is 1120 kgm^{-3}. The ratio for mixing epoxy resin to hardener is pre-defined by the supplier as 10:1. The utilization of fly ash as an additive component in polymer composites have received increased attention recently, particularly for price driven/high volume applications [9]. Fly ash collected from Rourkela Steel Plant, Odisha; is used as the filler material. This class 'C' fly ash mainly consists of spherical particles with random diameters that are observed with scanning electron microscope. Particle size analysis has been done by the MALVERN (Mastersizer) and average size is found to be 27.26 µm. The detail chemical analysis revealed that SiO_2 and Al_2O_3 are the major constituents of the fly ash. ASTM D 638, ASTM D 790 and ASTM D 256 have been followed for making the designs for tensile, flexural and impact testing specimens respectively, using CATIA V5R19 software. Patterns were made in rapid prototype machine. It is an additive layer manufacturing technology that is usually used to fabricate a scale model of a physical part or assembly using three-dimensional computers-aided design (CAD) data. The patterns were made by hard plastic material (ABS Plastic) which has a glass transition temperature around 110°C, and is much higher than the reaction temperature during polimerisation of the epoxy resin at room temperature. The split patterns were assembled together with a gummed tape, and a negligible amount of grease is applied to the inner corners of the pattern. Then the mould release spray is used for easy removal of the solidified samples.

The fly ash and epoxy resin are taken by weight% in a small beaker and mixed by ultrasonic and magnetic mixing. In the case of the ultrasonic mixing, the mixture is placed in a sonicator for the desired time with a pulse time of 5 sec and frequency of 10 KHz. In the case of the magnetic stirring, a steel ball is placed inside the beaker and placed over the steel plate that helps in proper mixing of epoxy and fly ash. The mixture of fly ash and epoxy resin is prepared. Pre-weighed hardener is stirred in the mixture for 1 min very gently to avoid the formation of bubbles. Then the freshly prepared mixture is gently poured inside the pre-designed pattern. The hardening time depends on the atmospheric temperature. As we are not maintaining a predefined condition, so it is allowed atmospherically for 24 h for curing. Once the solidification is completed, the tensile, flexural and impact test samples were removed from the pattern and carefully placed inside the polythene packs for further use. The polythene packs help in preventing moisture absorption. Samples of six different mixing times (5, 10, 15, 20, 25, and 30 min) with both ultrasonic and magnetic mixing conditions were made. The mixing time is constrained to 30 min, due to overheating of the mixture that results in the formation of the bubbles sometimes.

Mechanical properties viz. tensile strength, flexural strength and impact strength are evaluated for the above-prepared samples. Three specimens from each category are assessed, and the mean value is presented. Both tensile and flexural test are done in universal testing machine (INSTRON 8800R, United Kingdom) with a cross-head speed of 1 mm/min. The UTM is a dynamic servo-hydraulic testing system with data storage. Samples of dog bone shape are used for tensile testing, and flat rectangular samples are used for (3-point bend test) for flexural test. Utmost care has been taken during clamping of the tensile specimens as the material shows a high amount of brittleness. In flexural testing, the span length is fixed to be 40 mm. The impact test was done in a VEEKAY TLVS4 impact

tester. The pendulum when released from an angle 150 degrees, can provide a maximum energy of 21.4 J. Microscopic observation is undertaken to have a better perspective of interactions arising from differing aspect ratio bearing reinforcements in the epoxy matrix [8]. Samples subjected to mechanical testing are examined in a JEOL 6480 LV scanning electron microscope. It is difficult to show all the SEM images in a single paper, so SEM images are given only for the samples giving the highest values of strength in each category. In this particular work, emphasis has been given on the distribution of load. The samples were magnified at 250X with acerating voltage of 15 KV to show the directional flow of stress. A "Shimadzu", IR presige-21, automatic infrared microscope with a wavelength range 350 to 7800 cm^{-1} is used for structural characterization of the composite. FTIR spectroscopic technique is used to understand the chemistry of surface for fly ash in thermally active state along with epoxy resin. In order to prepare pellet, a little quantity of potassium bromide, (KBr) was segregated with powder sample and after that pressing of mixture was done at 6 t of load in a hand press. Analysis of that pellet was done using FITR by keeping the pellet in a sample holder. First the % transmittance of only KBr is measured and then for the KBr mixed samples so that the graph can be plotted between % transmittance and wave number of Infrared frequency.

A "Mettler Toledo" (Model No-822) differential scanning calorimeter is used to determine the glass transition temperature. The temperature range of the instrument is from 100 to +400°C. The melting temperature range of phase changing materials is usually measured by a differential scanning calorimeter with a dynamic measurement method. The instrument is also equipped with an external liquid nitrogen cooling accessory. Normally polymer samples with flat surfaces and weight between 10 to 15 mg are placed in an aluminium crucible for proper testing. The samples were scanned from a temperature range from 30 to 250°C. Experiments were repeated twice to ensure the reproducibility of the date and the error is minimized. Here the endothermic peak shows the amount of heat absorbed for the total curing of the material.

RESULTS AND DISCUSSION

Epoxy polymers are classified as a brittle material because these materials usually fail in tension at relatively low values of strain [10]. Figure 1 shows the tensile strength of the composite prepared at six different mixing times. It is seen that the properties go on increasing with the increase in mixing time.

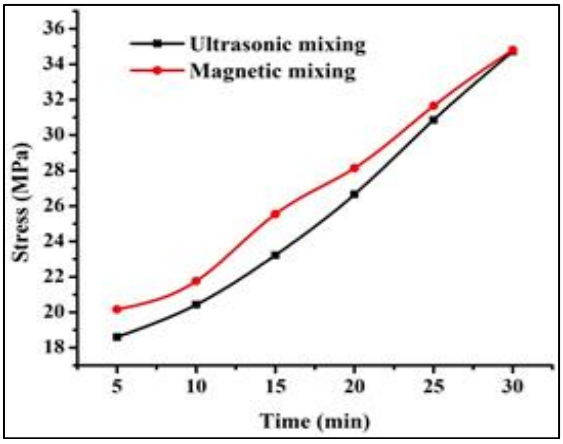
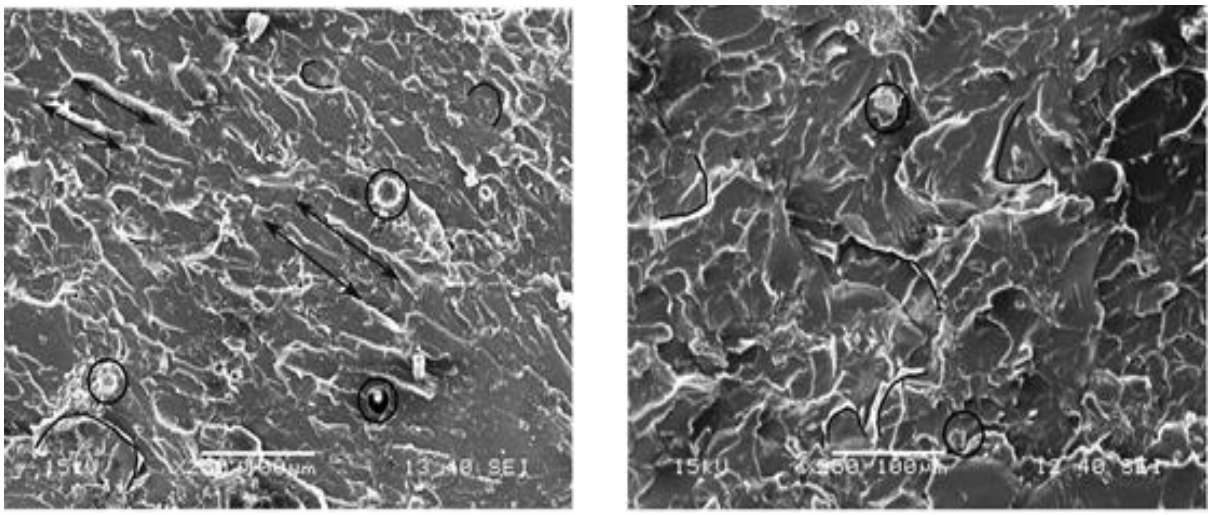


Fig. 1: *Effect of Mixing Variations on Tensile Strength.*

It is clearly evident from the graph that, the tensile strength for the ultrasonic mixing samples is slightly lower than the magnetic mixing condition in the beginning. But as the mixing time increases both tend to a near same result. As the tensile load increases, the matrix tries to elongate in its usual way. However, the filler particles resist deformation [10].

Lines with arrow marks show the directional flow of stress. Circles marks show the fly ash particles entrapped which results in the points/regions for stress concentration. In Figure 2(a) an air bubble is seen trapped on the lower side of the figure. In Figure 2(b) no such directional flow of material is seen but more dispersion of fly ash particles is seen which helps in increase in strength. Semi-circular marks in the figure show the flow layers of stress in the material. Here the SEM micrograph is taken only for samples having highest values of tensile strength, in both ultrasonic and magnetic mixing conditions. From the microstructure, we can see that, in magnetic mixing condition the reinforcement

particles are more dispersed than in ultrasonic mixing condition. In addition, directional flow is observed in the case of ultrasonic mixing condition where as more protrude portions are seen in magnetic mixing state.



(a) ***(b)***

Fig. 2: *SEM Micrograph of Fracture Surface of Tensile Testing.* ***(a)*** *Magnetic Mixing;* ***(b)*** *Ultrasonic Mixing.*

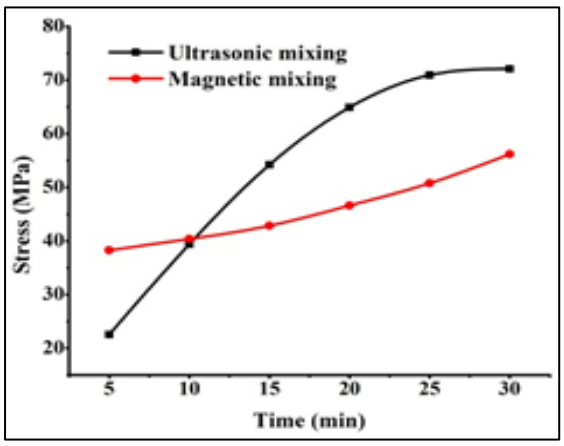
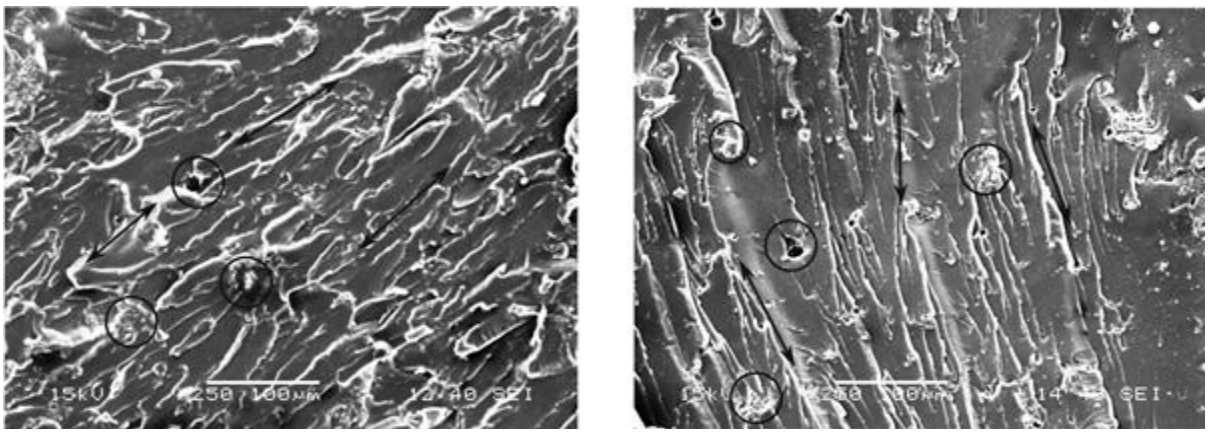


Fig. 3: *Effect of Mixing Variations on Flexural Strength.*

The 3-point bend test is a stress-strain test whereby the specimen is deformed under bending conditions [11]. Figure 3 shows the flexural strength of two different mixing conditions.

It is seen that, at less time of mixing i.e. for 5 min, the flexural strength in ultrasonic mixing is less than that of flexural strength in magnetic mixing. But, near about at 10 min, both the strengths are remaining same and as the mixing time increases flexural strength of ultrasonic mixing is increasing than the magnetic mixing condition.



(a) ***(b)***

Fig. 4: *SEM Micrograph of Fracture Surface of Flexural Testing.* ***(a)*** *Magnetic Mixing;* ***(b)*** *for Ultrasonic Mixing.*

In both the cases, it is seen that, the load is transmitted in the matrix, and it is smoother in case of ultrasonic mixing condition. But there are some fly ash particles still embedded in the matrix that is the main cause of stress concentration in the composite. Circle marks in the Figure 4(a) show the stress concentration zones and arrow marks show the directional flow of stress, which is more prominent in Figure 4(b).

It is evident from Figure 5 that, the impact strength in both the conditions of mixing is increasing. At the lower time of mixing i.e. at 5 and 10 min, the impact strength in magnetic mixing is more than the ultrasonic mixing. As the time of mixing increases, the impact strength increases. It is seen from the graph that the impact strength for both the conditions remains same at around 17 min of mixing. After 20 min, the impact strength in ultrasonic mixing is quite more than the magnetic mixing.

The Figure 6 shows that the intercalated structure of fly ash creates high localized stress in the matrix. A weak fly ash-epoxy interface restricts the ability of transferring the energy and results in plastic deformation [12]. Circle marks in the Figure 6 show the entrapped fly ash particles that increase the stress concentration. Arrow marks show the linear directional flow of stress. SEM micrographs show very uniformly mixing in ultrasonic mixing condition. In Figure 6(a) river pattern is clearly visible that shows that the impact energy is being transmitted from one part to another which is ultimately responsible for the breaking of the sample. Magnetic mixing SEM micrograph shows some more amount of entrapped fly ash particles (stress concentration zones) which may be the reason for the decrease in impact strength.

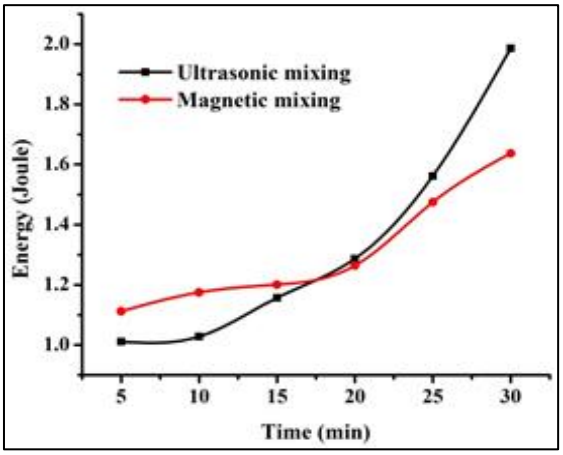
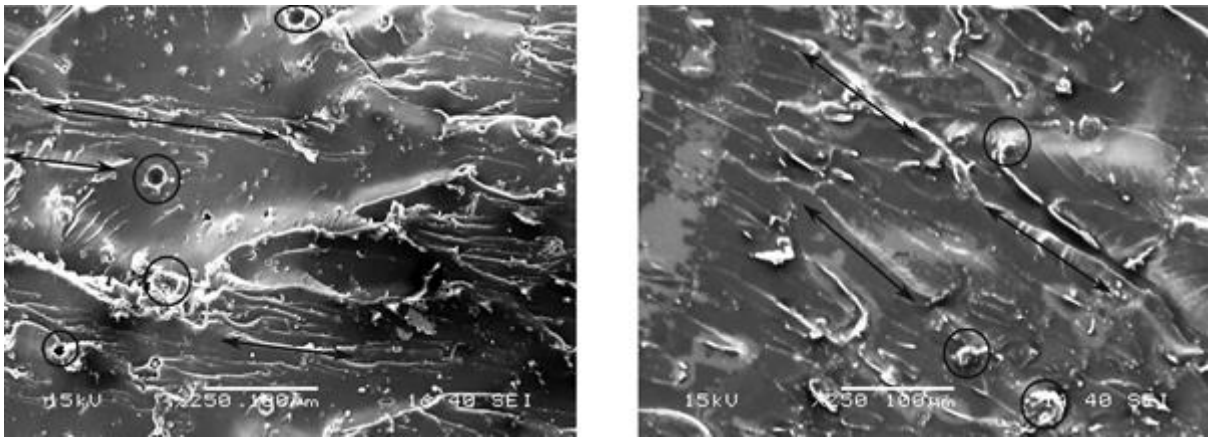


Fig. 5: *Effect of Mixing Variations on Impact Strength.*



(a) ***(b)***

Fig. 6: *SEM Micrograph of Fracture Surface of Impact Testing.* ***(a)*** *Magnetic Mixing;* ***(b)*** *Ultrasonic Mixing.*

FITR spectrometer is used to calculate the transmission percentage of infrared as shown in Figure 7. Here the sharp peaks are obtained at wave number of 3400, 2900, 1600, 1530, 1250, 1150, 1050, 850, 550 cm^{-1}. By correlating the above graph with the IR chart, we can conclude the bonds formed during the mixing time. The wave numbers like 2900, 1600, 1530, 1250, 1150, 1050, 550 cm^{-1} represent presence of strong bonds like CH_2,

R-C-O, and C-O-H. Wave numbers like 3400 and 850 cm^{-1} represent presence of $–NH_2$ medium strength bond and isolated aromatic C-H weak bond respectively. FTIR analysis of different mixing time for a same composition of material show almost same result as the composition remains the same i.e. 10%FA + 90%EP.

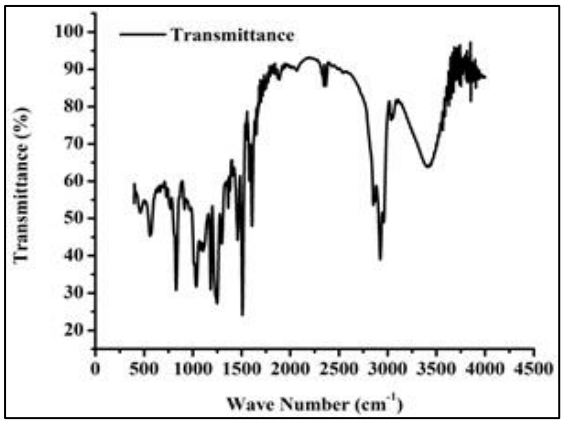


Fig. 7: *IR Spectra of the 10% Fly Ash And 90% Epoxy Resin.*

DSC results are dependant of the heating rate and the mass of the sample [13]. Both the sample and the reference are maintained at the same temperature throughout the experiment. The temperature programme for a DSC analysis is designed such that the sample holder temperature increases linearly as a function of time.

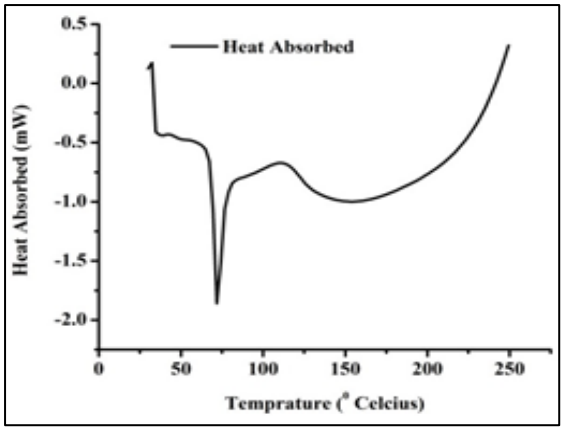


Fig. 8: *DSC Thermograph of 10% Fly Ash and 90% Epoxy Resin Mix.*

DSC analysis of 10% fly ash and 90% epoxy composite at different mixing times was carried out in order to find the thermal stability of the material. But the DSC curve for all these samples remains almost same as the composition remains same. There is a variation of 2°C in glass transition temperature, may be due to variation in sample weight or due to machine error. So, one DSC graph is represented as Figure 8. The DSC thermograph shows an endothermic peak with an onset temperature at 68.28°C. The midpoint of the thermograph lies at 68.70°C giving the glass transition temperature. The endothermic peak is attributed to curing of the sample by absorbing the heat [13]. The melting temperature range increases with an increase in heating rate [13].

CONCLUSIONS

The present work deals with the intention of finding out the best suitable way of mixing filler particles to the high viscous epoxy resin. Various conclusions drawn from the above experiment are listed below;

1. The strength is increasing with an increase in mixing time.
2. In tensile test, the strength is remaining same at 30 min for both ultrasonic and magnetic mixing conditions and in other cases it is lower than the magnetic mixing condition.
3. Normally in polymer composites the flexural strength is important, so we can conclude that ultrasonic mixing condition is more suitable than the magnetic mixing condition for sample preparation. It may be because of, as polymer is a nonmagnetic material, magnetic mixing has very little impact.

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