Tribological Properties of Fly Ash Blended Polymer Composites

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ABSTRACT
In this study, it was studied that the abrasion resistance and characteristics of polymer composites with fly ash (FA). The epoxy based polymer composites are produced with resin and FA as mineral additive. Mixtures of different ratio by replacing the FA were added to the resin from 0 to 30% by weight. Polymeric samples were cured in air conditioning and they were taken from the molds after 24 hours. Polymeric samples gain ultimate strength after 7 days. Therefore, abrasion tests were performed on 7 aged specimens. Abrasion characteristics of polymer composites were defined by pin-on-disc test for 500 m under a dry friction condition and room temperature. Three types of loading conditions were carried out as 5, 10, 15 and 30 N. The hardness and wear resistance values increased with the increase in the content of fly ash. Showing the relationship between wear rate and hardness, an equation with parameters dependent on load was provided. There was an increase in the friction coefficient with an increase in the surface roughness values. In addition, dynamic friction was as a function of the wear rate. The wear surfaces of the polymer composites were analyzed using scanning electron microscopy. It was observed that the wear rate of the polymer composites and pure epoxy samples ranged from 17.82 to 172.96 mm³/Nm.

Keywords: Fly ash; polymer composite; characterization; wear; friction.

1. INTRODUCTION
Fly ash produced during coal burning for energy production is an industrial side considered as an environmental contaminant [1]. It is generated in million tons worldwide due to heavy industrial growth and a very few percentage is reused in different purposes. This large amount of industrial waste can cause significant environmental and ecological problems if not properly handled. Due to the ecological and economical importance, research has been conducted around the world on the potential reuse and effective use of these wastes. [2]. The problem of fly ash disposal is expected to get worse as the demand for energy grows. The main use of fly ash is in making of Portland cement, road embankment and fly ash bricks. Beyond these large scale applications, fly ash hasn’t been used in product development and automobile industries which may play an important role in its reuse. Epoxy resin is a cheap semi viscous fluid which becomes a strong matrix material when a ceramic filler like fly ash is used in it. Different types of ceramic, metal and bio materials are used as reinforcement in form of fibre or particle reinforcement [3]. Filler material helps in increasing mechanical, thermal and tribological properties and simultaneously helps in reduction of cost of the component. Various researchers have used alumina and silica in polypropylene and polyethylene. But very few attempts have been made to use the cheap materials like rice husk, coir, jute fibre and even chicken feather [4, 5].

Tribology is an important parameter while characterizing any material as it is related to the loss of material at the application area. The presence of hard reinforcement phases, particulates, fibres or whiskers has endowed these composites with good wear behavior properties. Under the loading condition the wear rate increases rapidly as the temperature due to frictional force matches with the material’s melting temperature [6, 7]. Hence, to enhance the life of the part there must be lowest possible amount of wear during the use of the material. So, higher loading condition produces higher working temperature with increment in wear and lead to speedier substitution of parts [8]. Light weight polymer matrix composites like glass fiber and carbon fiber are the most suitable materials for weight sensitive application in aerospace and automobile industries
due to its high strength and low erosion rate [9]. In this view, the present work has been focussed on fabricating fly ash epoxy composite and its sliding wear test has been carried out at various treatment conditions to evaluate at which condition the product is sustaining minimum wear. Wear test has been carried out in three different treatment conditions like normal atmospheric condition, oven treatment and micro oven treatment condition. Duration of curing in oven or micro oven has been determined by varying the temperature and duration of curing in a trial basis and optimized condition is chosen taking mechanical strengths into consideration. Prior to the tribological experiment, the optimized oven treatment condition is found to be 30 minutes at 1200c in oven and 5 min in micro oven at 60% of power flow. The DSC analysis curve shows the variation in enthalpy due to the treatment. In this present research work, effort has been made to increase the surface and overall property of the polymeric sample and compare it with the normal curing conditioned sample. Various researchers have explained the impact of fortification volume percentage, sliding conditions (time, distance, speed etc.) and applied load that impacts the dry sliding wear of the composites [10, 11].

In general, studies take into account to enhance the tribological properties of polymer materials. XU et al. [12] described that the sliding is mainly influenced by contacting parts and tribological behavior of the materials. To make a suitable wear resistant composite, one has to evaluate the relation between varying parameters and wear rate. This technique has effectively sought parametric evaluation in different wear procedures of an extensive variety of polymer composite. The addition of fly ash in vinyl ester matrix has increased the wear characteristics of the composite [13]. Treated fly ash particles reinforced in epoxy resin exhibited better compressive strength [14]. Increased fly ash content in the hybrid reinforcement of graphite and fly ash to aluminium matrix contributed to the decrease in the wear rate [15]. Fly ash and glass fabric reinforced epoxy resin had increased impact strength and compressive strength [16]. PATTANAIK et al. [17] examined the dry shear wear behavior of the epoxy fly ash composite with Taguchi optimization. As a result of the research, they demonstrated that particle reinforcement plays an important role in increasing wear rate. BROSTOW et al. [18] a commercial epoxy diglycidylether of bisphenol-A (DGEBA) was modified by adding fluorinated poly(aryl ether ketone) fluoropolymer and in turn metal micro powders (Ni, Al, Zn, and Ag) and studied wear behavior. They obtained the best wear result in composites with Al additive. RAJa et al. [19] effective utilization of industrial waste (fly ash) to enhance the properties of polymer composites for mechanical components were examined and They found that composites filled with 15% fly ash are superior to other composition in terms of wear. Epoxy resins are the most commonly used thermosetting plastics in polymer matrix composites and do not give reaction products when they are hardened and cause low hardening tensile strength. They also have good adhesion to other materials, good chemical and environmental resistance, good chemical properties and good insulation properties. In this study, particularly, it was investigated that the influence of fly ash addition as filler on wear and friction characteristics of epoxy composite.

2. METHODS
2.1 Materials and sample production
The fly ash (FA) used in the study was supplied from the Tunçbilek Thermal Power Plant in Tavşanlı/Kütahya (Turkey). It’s maximum particle size was 113.78 µ. Chemical component of FA was presented in Table 1.

| Oxide | CaO₂ | SiO₂ | Al₂O₃ | Fe₂O₃ | ZnO | Na₂O | MgO | LOI |
|-------|------|------|-------|-------|-----|------|-----|-----|
| Content, % | 6.66 | 47.4 | 19.8  | 11.8 | 62  | 0.57 | 4.76 | 6.39 |

Epoxy resin which is commercially available along with hardener was used as matrix material in production of different samples. Epoxy resin has modulus of elasticity of 3.42 GPa, and density of 1100 kg/m3. Mixing ratio (by weight) was used as indicated for the processing of 4 parts of epoxy resin and 1 part of hardener (Table 2). The required mixture of resin components were made by mixing them in parts in a beaker by stirring the mixture in a beaker by a rod. The matrix composites were mixed at room temperature. The prepared mixture was transferred to the mold which was covered with the separator. Steel molds of Ø50 mm size were used for casting of polymer matrix composite specimens. The curing was carried out at laboratory condition for about 24 hours. After hardening, the specimens were removed from the molded.
Table 2. Composition of epoxy based polymer composites

| MIXTURE CODE | EPOXY RESIN*, % | FLY ASH, % |
|--------------|----------------|-----------|
| FA0          | 100            | -         |
| FA10         | 90             | 10        |
| FA20         | 80             | 20        |
| FA30         | 70             | 30        |

*Epoxy resin was used with hardener (4:1)

2.2 Wear Tests on polymer composites

To perform friction and wear tests on epoxy-based samples, scratch tests were performed by using a ball-on-disc test instrument. In the abrasion tests, WC-Co balls with a diameter of 8 mm and an average hardness of 1895 HV were used. By using a separate abrasive element in each experiment, faults resulting from the corrosion that may occur on the surface of the abrasive element have been removed. The abrasion tests carried out in the ball-disk system were made under dry friction conditions, at room temperature, under loads of 5, 10, 15 and 30N, at a shear rate of 0.3 m/s and a distance of 500 m. At the end of the wear test, the abrasion volumes of the specimens were calculated with data aid obtained from the so-called rugosimeter; is obtained at the end of the multiplication of the wear cross-sectional areas with the circumference of the formed wear trace. The rate of wear was calculated by dividing the amount of worn volume of the applied load and the slip distance on the polymer composite sample. Friction coefficients of worn samples depending on sliding distance were obtained by a friction coefficient program.

3. RESULTS AND DISCUSSION

3.1 Characterization

The SEM microstructures of the epoxy composite samples containing various FA contents used in this study are shown in Figure 1. As can be seen from Figure 1, the surface around of fly ash grains has covered with the densely hardened polymer matrix. This case showed that there is a good bond between fly ash grains and polymer matrix. The hardness values of the composites were increased proportionally with the increase of the amount of FA in Figure 2. The hardness of the pure epoxy resin is 84 Shore D (H_D). The hardness values of 10% FA, 20% FA and 30% FA blended polymer composite samples were obtained as 86 H_D, 88 H_D and 91 H_D, respectively. The hardness value of 30% FA blended composite sample was obtained 8.33% higher than the control sample (0% FA).

Figure 1. SEM micrograph of FA polymer composites: (a) 10% FA, (b) 30% FA matrix / fly ash.
3.2 Surface roughness

The surface roughness values of no added the waste material and added polymer composites were presented in Figure 3. The roughness values were obtained close to each other in samples that control and with low FA content. However, in samples with high volume of FA, the surface roughness value is lower than FA0 specimen. The surface roughness of the samples produced from fly ash-added polymer composite materials are respectively: Pure epoxy -0.38 µm, 10% FA -0.419 µm, 20% FA-0.474 µm and 30% FA-0.521 µm.

3.2 Friction coefficient and wear rate

Friction coefficients at different load are presented in Figure 4 for control and FA containing samples. While the lowest friction coefficient is obtained in FA0 samples as 0.39, it was increased to 0.53 in 30% of FA containing series. Use of FA in polymer composites provides higher friction coefficient when compared to control series. The friction coefficient of all the series are increased by increasing of load. The friction coefficient increased the friction coefficient in the samples with high additives, by removing the particles / additives from the sample during wear and in the wear zone (between the pin and the sample) during wear. Low friction coefficient is generally one of the best indicators of wear resistance [20]. An increase in the friction
coefficient was found with the increase in the additive ratio in the material. Srinath and Gnanamoorthy [21] found that the friction coefficient values increased as the nanoclay rate added to the structure increased. BHIMARAJ et al. [22] observed in their study that the amount of nanoclay added to the matrix increased and the friction coefficient increased. It is believed that this is due to polymer chains that become weaker with the increased amount of additive materials.

Wear rate of polymer composites are given in Fig. 5 for 5, 10, 15 and 30N depending on FA content. As seen from the Fig. 5, wears of composites decreased by increasing the content of on FA. The highest wear rates were obtained on pure epoxy series at each loading conditions. The range of wear varied from 59.64x10^(-4) to 17.82x10^(-4) mm^3/Nm at 5N, from 80.13x10^(-4) to 36.75x10^(-4)mm^3/Nm at 10N, from 119.14x10^(-4) to 42.87x10^(-4) mm^3/Nm at 15N, from 172.96x10^(-4) to 60.12x10^(-4) mm^3/Nm at 30N depending on fly ash content. In other words, there was about 2.87 times increase in wear resistance of fly ash composites at 30 N by increasing of waste content. Increase of FA addition ratio resulted with decrease of wear rate. This means that the polymer material become harder, and also surface character of polymer composites change the by addition of FA. Wear strength was higher in high FA ratio than in low FA ratio due to more homogeneity distribution of grains with high resist to abrasion. On the other hand, when the wear loading value was considered, the wear rate for all the samples increased with load regardless of FA ratio. The wear rate increased with an increase of load from 5N to 30N due to increase friction of and abrasion on surface of fly ash samples. CHANG et al. [23] investigated the wear behavior of the polymeric composite material by adding short carbon fiber, graphite particles, TiO_2, ZnS into the polyether ether ketone (a colorless organic polymer thermoplastic used in engineering and medical fields). They determined that there was an increase in wear rates with the increase of the applied load amount. UYGUNOGLU et al. [24] in their study, added boron wastes to pure epoxy in certain proportions and examined their abrasion resistance. With the increase of the waste boron ratio added to the samples up to %50, they achieved increases in abrasion resistance. BHIMARAJ et al. [22] stated that in their study with the addition of poly (ethylene) terephthalate (PET) alumina nanoparticle, the rate of more than 1% nanoparticle reduced the rate of wear.
One of the interesting findings of this study was presented in Fig. 6 as relation between hardness and wear rate for FA containing polymer composites. The hardness values of the samples increase with increasing FA content. As seen, wear resistance increases with an increasing hardness. The related equations were given in the appendices in Fig. 6. The coefficient of correlation is very close to 1.0 for each loading test, and the highest value being 30 N.

The calculated regression functions can be seen in Fig. 7 to facilitate the analysis of the surface quality on the tribological properties of the examined epoxy-based polymer composites. This figure shows the friction coefficient depending on the surface roughness. The relationship between surface roughness and friction coefficient are lower at 5N (R=0.948) than 30N (R=0.994) loading condition. These results show that there is better relation between friction and surface roughness at high loadings.
For FA blended epoxy-based polymer composites, the relation between the friction coefficient and wear rate was presented in Fig. 8. Suitable linear relation is developed between friction coefficients and wear rate, regardless of waste material content. The relation was found with correlation coefficient (R) of 0.927. As a function of wear rate, friction coefficient of FA blended polymer composites can be determined by:

\[ F_c = 0.0103W_r + 0.164 \]  

(1)

The wear scanning electron microscopy (SEM) micrographs of polymeric composites after tribometry for various FA content were shown in Figure 9. The hardness values of the polymer composites were increased by increase of FA content. So, the wear resistance is increased when compared to control specimens due to high degree of hardness values of FA blended polymer composites (Fig. 9a-d).
Figure 9. SEM micrograph and cross-sectional surface of the worn-out surfaces of epoxy composites after tribometry for various FA content: (a) for pure epoxy, (b) for pure epoxy CS, (c) for 10% FA, (d) for 10% FA CS, (e) for 20% FA, (f) for 20% FA CS, (g) for 30% FA, (h) for 30% FA CS.

Figure 9 shows the wear surfaces, and the cross-sectional surface (CS) of the wear mark obtained from the wear region by analyzing multiple profilometry surface line scans using a Nanovea ST-400 non-contact optical profiler. It was observed that the depth and the width of the wear trace on the surfaces of the samples decreased with an increase in the contribution rate FA (Figures 9b, 9d, 9f and 9h). Figure 9a shows the SEM micrographs of the wear surfaces and Figure 9b shows the cross-sectional surface (CS) of the wear
mark obtained from the wear region of the FA0 polymer composites. Increase of load provides that the ball moves over a wear border. The wear border makes the movement of the ball more difficult at high loading condition (see Fig. 9); now the indenter moves across the entire surface of each region [25 - 27]. As a result of the wear of the pure epoxy sample, tearing on the surface occurred (Fig. 9a). The wear tests in the added fly ash specimens, scratches, delamination wear, abrasive and adhesive wear occurred on the surface. As the fly ash additive ratio increases, the wear depth decreases. A high friction results, in fact even higher than friction values for pure components [26 - 28]. The hardness values of the specimens were increased by increase of FA content. So, the wear resistance is increased when compared to control specimens due to high degree of hardness values of FA blended polymer composites. BARPANDA et al. [29] compared the sliding wear behavior of neat epoxy with the epoxy-containing different volume fraction of fly ash. Obtain results showed the different magnitudes of wear loss for neat epoxy and epoxy/fly ash composite. OZSOY et al. [30] investigated and compared the tribological and mechanical performance of nano and micro epoxy composite nanofillers such as Al$_2$O$_3$, TiO$_2$ and clay and micro fillers like fly ash, TiO$_2$ and Al$_2$O$_3$. On pin-on-disc arrangement, wear test was examined under dry atmospheric conditions values 5, 10, 15 N, sliding speed 0.8 m/s and sliding distance by 2000 m. They concluded that the tribological and mechanical performance of micro filler epoxy composite was found better than nanofiller epoxy composite.

4. CONCLUSIONS
The purpose of this report is to explain the tribological properties (friction and wear characterization) of FA-containing polymer composites, as well as how FA additives effect to polymer composite properties. From the present study, the following conclusions may be derived:

- The hardness values of FA containing composites were increased by using fly ash when compared to control sample. The fly ash ratio increased surface roughness values.
- The hardness value of 30% FA blended composite sample was obtained 8.33% higher than the control sample (0% FA).
- The roughness of surface values was obtained close to each other in specimens that without and with low FA content. Use of FA in polymer composites provides higher friction coefficient when compared to control series. The friction coefficient of all the series are increased by increasing of load.
- The addition of 30% fly ash increased the surface roughness of the pure epoxy sample by 37%.
- The range of wear varied from 59.64x10$^{-4}$ to 17.82x10$^{-4}$ mm3/Nm at 5N, from 80.13x10$^{-4}$ to 36.75x10$^{-4}$mm3/Nm at 10N, from 119.14x10$^{-4}$ to 42.87x10$^{-4}$ mm3/Nm at 15N, from 172.96x10$^{-4}$ to 60.12x10$^{-4}$ mm3/Nm at 30N depending on fly ash content.
- Wear strength is higher in high FA ratio than in low FA ratio due to more homogeny distribution of particles with high resist to abrasion. There is about 2.87 times increase in wear resistance of in samples with added fly ash at 30 N by increasing of fly ash content.
- A good correlation has been obtained between surface hardness and wear rate; roughness coefficient of friction; for epoxy based and FA blended polymer composites, friction coefficient and friction coefficient are obtained respectively.
- The relationship between surface roughness and friction coefficient are lower at 5N (R=0.948) than 30N (R=0.994) loading condition.
- Increase of load provides the ‘border effect’ on the surface. The border effect leads to increase of friction coefficient at higher loading conditions.
- In microstructure studies, FA particles were found homogeneously distributed in the epoxy matrix. The wear tests in the added fly ash specimens, scratches, delamination wear, abrasive and adhesive wear occurred on the surface. As the fly ash additive ratio increases, the wear depth decreases.
Consequently, it was obtained that notable improvement in the wear resistance, friction coefficient and surface quality by using of FA in epoxy based polymer composites.

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