Indian Journal of Engineering & Materials Sciences
Vol. 22, June 2015, pp. 354-362

Fly ash cenospheres as reinforcement in different polymer composites – a comparative study of physical and mechanical properties

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Received 20 May 2014; accepted 10 December 2014

The work reports the preparation of fly ash cenospheres bearing polymer composites, using various polymer matrix materials namely, low density polyethylene, high density polyethylene, polystyrene and polymethylmethacrylate followed by evaluation of properties. The composites are synthesized by including about 18% by weight fly ash cenospheres, into various polymer matrices using brabender facility in the temperature range 120-160°C and at a mixing pressure of 50 MPa. Subsequently, they are cast into sheets through compression moulding. The test samples, made from the sheets, are characterized for physical as well as mechanical properties such as density, hardness, compression strength, impact response, wear and friction. The investigation reveals that the addition of fly ash cenospheres to various polymer matrices results in reduction of density. Further, improvements in the slide wear resistance and decrease in the co-efficient of friction values are noticed. As for interpreting the slide wear data, recourse to examination under scanning electron microscope is made in this paper. As regards the mechanical properties, hardness increases while the compression strength and impact energy decreases with inclusion of cenospheres in all the four types of samples investigated.

Keywords: Thermoplastics, Fly ash cenospheres, Compression strength, Impact energy, Hardness, Slide wear, Friction

The use of polymer based composite materials for many engineering applications is on the increase due to their displaying attractive properties[1-7]. Hence, a study of the various properties in polymers has gained importance. The one property that has received attention is the tribological response of glass fiber or filler reinforced polymer systems[8-10]. Fiber re-inforced display high strength and stiffness typically needed for aeronautical and aerospace applications[11].

With the inclusion of appropriate fillers, the polymers are found to exhibit better properties required for specific end-use applications. Thus, references to compression strength parameter[12] and a study of impact response owing to presence of rubber as additional filler material[13] are found in the literature. The inclusion of fillers can also promote lower friction values as reported elsewhere[9,14,15].

In one instance, graphite inclusion has been looked into from the point of strength variation unlike the friction properties normally associated with this filler. The work takes into account the moisture effect in the graphite-bearing systems on compression strength[16]. Not only the slide wear, friction and compression properties, but even the response to erosion gets influenced by the presence of fillers [17-19]. The erosion response of another filler material, namely, glass microspheres has been investigated in another work[20], wherein the emphasis is on correlating erosion loss with the angle of impact. The possibility of working with fly ash fillers from tribological point of view can be found in literature[21-23]. This filler as a material to alter the wear response has been investigated[21] as the ash is inexpensive and hence the cost of the product can be made low. In another study[22], the slide wear characteristics of glass-epoxy (G-E) composite, filled with either rubber or oxide particles were evaluated. The lower wear was recorded for oxide at lower loads where as this trend was displayed by rubber bearing ones at higher loads. The G-E composite systems had rubber in one case and graphite of two differing levels in the other[23]. The higher graphite-filled samples showed lower co-efficient of friction for any combination of load and velocity. These data have been accounted for by the scanning microscopic features seen on the worn surface of the samples[23]. Akinci *et al*.[24] have studied the effect of basalt as

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filler with low density polyethylene (LDPE) as matrix material on the physical, mechanical and morphological behaviour. The investigation revealed that the content of basalt filler affected the structural integrity and mechanical properties of composites. Unal *et al*.[25] have studied the influence of speed and load on the friction and wear behavior of different composite systems.

From the above literature coverage, it is seen that greater impetus need be given to the technological advancements of processing of polymer composites followed by evaluation of properties. There are two ways of attempting to develop composites. The first one, a well attempted by many investigators is to employ fillers of various shapes, sizes, types and compositions with a given matrix so that the overall performance of the system is improved. The other route is to explore by keeping the filler or reinforcement material the same and varying the polymer matrices. The latter approach is less explored and hence attempted in this work.

The present work reports the influence of load application on the friction and wear behavior of four matrices, namely, low density polyethylene (LDPE), high density polyethylene (HDPE), polystyrene (PS) and polymethylmethacrylate (PMMA) containing fly ash cenospheres as filler material. The friction and slide wear measurements for different loads were carried out on a pin-on-disc arrangement in dry condition at room temperature. Also, catalogued are the mechanical and physical properties of the newer composites.

Fly ash cenospheres has been chosen as filler material as they occur as a by-product of the burning process of coal in coal-fired power plants. They provide the advantages of reduction in weight owing to their low densities. Besides, utilizing them in newer products will aid the process of finding an appropriate use for the ash particles as they are considered to be environmentally unfriendly.

Experimental Procedure

Materials and processes

The composites were prepared as stated above, using fly ash cenospheres, having density of about 400 kg/m^3 and 100 µm as the average particle size. The matrices used were LDPE, HDPE, PS and PMMA supplied by M/s Shah Polymer, Bangalore, India.

The polymer composite was prepared using a plasticorder machine (brabender make No,/Country/ Type) for blending of thermoplastic polymers with filler material. It consists of a small internal mixer with interchangeable rotors. The mixing chamber is jacketed for operation at constant temperature and provided with a rotor assembly coupled to a torque meter to record the force during the mixing cycle. The two blades are made to rotate inside the chamber for proper and uniform mixing of polymer matrix with the filler material. Subsequently, a calculated amount of filler is incorporated to the matrix material. In the present case, fly ash cenospheres filler to the tune of about 18% by weight, has been used. The plasticorder has a free volume of 100 mL in which two sigma blades rotate against each other in opposite directions with variable speed in the range 0 to 100 rpm under molten conditions of the polymer at a temperature of about 240°C. The blending was optimized with lower mixing times to reduce the possibility of breaking of fly ash cenospheres in the matrix.

The composite specimens were fabricated by the compression molding method. For this, the previously blended material is taken from the machine and kept in between the pressing plates. A layer of polyester film was provided in between the plate and composite surface for easy release and to obtain smooth and uniform surface on the composites. Subsequently, sheets of size 150 × 150 × 2.0 mm^3 were prepared from the blended material using a hydraulic press at a pressure of 0.4 MPa and temperature of about 40°C. The hot pressed sheets were cooled to room temperature through chilled water circulation around the plate. The composite samples were cut using a diamond-tipped cutter to yield wear test coupons of size 6 mm × 6 mm. The test samples were then glued using an adhesive to a steel pin of size 6 mm diameter and 23 mm length. The details of the sample designation for the different polymer matrices with and without fillers used in this work are cited in Table 1.

Characterization techniques

The processed composites were characterized for physical (density), mechanical (compression/hardness/ impact energy) and tribological (slide wear and friction)

Table 1 – Designation of un-filled and cenospheres filled polymers

Sl.No.	Materials processed	Designation
1	Low density polyethylene	LDPE
2	LDPE + 18 wt% FAC	LDPE-C
3	High density polyethylene	HDPE
4	HDPE + 18 wt% FAC	HDPE-C
5	Polystyrene	PS
6	PS + 18 wt% FAC	PS-C
7	Polymethylmethacrylate	PMMA
8	PMMA + 18 wt% FAC	PMMA-C

properties. The density of the composites was calculated using weight measurements in a high precision digital balance, first in air and then immersed in distilled water (as per the guidelines in ASTM D792 Standard). As regards evaluation of fly ash content is concerned, the difference in the weight of the sample before and after the burn out test was made. The hardness measurements of the composite were carried out using Shore D Durometer. The compression strength was measured using electromechanical computerized universal testing machine (UTM, Instron Series 3360). The impact strength was determined using falling weight type Dynatup 8500 (USA) impact test machine. The scanning electron microscope (SEM; LEICA 440I, UK) has been used to interpret the worn surface features of the test samples. In all the mechanical evaluation methods, data were gathered on four representative samples and the average value obtained has been reported and used for the analysis.

Slide wear and friction measurements

The photograph of the slide wear test set-up, widely known as Pin-on-Disc (POD) machine is shown in Fig. 1. The set up is used for wear measurement at room temperature wherein the test specimen in the form of a pin having the dimensions 6 mm ϕ and 25 mm length is used as a test sample by fixing it to a sample holder. The sample runs against a counter face material of 60 HRC alloy steel disc for test duration of 10 min. Both before and after the tests, the samples weight is noted using a sensitive high precession electronic balance. The test set up has the facility to suspend dead weights which yields force ranging from 20 to 40 N. A string, a pan and pivoting arrangement helps in this process. The disc can be rotated at any given rpm by changing the gear box parameters. For the present work, a sliding speed of 3.14 m/s corresponding to 400 rpm (sliding distance of 722 m) has been chosen. For the frictional value readings, the load cell attached to the pin assembly is used. By dividing the friction load by the normal load, the coefficient of friction can be obtained as a number.

Fig. 1 – Photograph of pin-on-disc machine

The slide wear tests were done three times on the sample and using another sample of the same history, additional three test data were collected. The average value obtained from such readings is used in the plot of weight loss vs. applied load. It is in important to note that an analysis of the co-efficient of variation yielded a value of about 10%, which is below the acceptable value of 15% mentioned in the ASTM standard. The bar charts and graphs plotted in respect of the results in this work have been incorporated with error bars taking in to accounts the variations in the data observed.

Results and Discussion

Physical property

Density

The density values measured are shown in Table 2. It decreases with the addition of fly ash cenospheres for all the polymer materials. From the data, it is noticed that the values for LDPE/LDPE-C, as a set, is the least. The fillers, possessing a value of about 400 kg/m^3 for the density, which is less than half of the matrix values for all the four chosen system, reduces the value of the resulting composite system.

Mechanical properties

Hardness

The hardness data are shown in Fig. 2 (a-d). It is observed that the hardness values, generally stated, increases by a marginal level with the addition of fly ash cenospheres. LDPE-based ones show lowest set of values for the hardness.

Table 2 – Density of un-filled and cenosphere filled polymers

Sl. No.	Sample identification	Density (kg/m^3)
1	LDPE	930
2	LDPE – C	850
3	HDPE	960
4	HDPE – C	890
5	PS	1050
6	PS – C	960
7	PMMA	1170
8	PMMA – C	1030

Compression strength

The compression strength results are recorded in Fig. 3 (a-d), and they show a decrease, for LDPE, HDPE, PS and PMMA of about 33%, 43%, 43% and 42%, respectively, owing to the presence of fly ash fillers. Like in the recording of hardness values, the

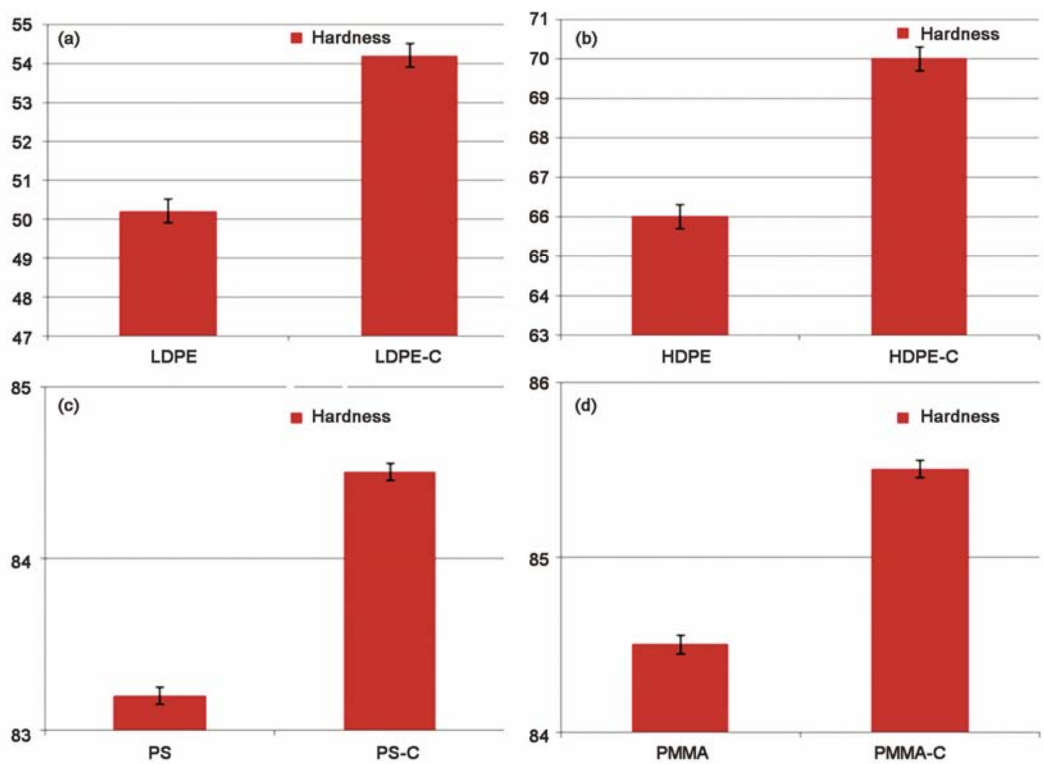

Fig. 2 – Hardness of unfilled and filled (a) LDPE, (b) HDPE, (c) PS and (d) PMMA

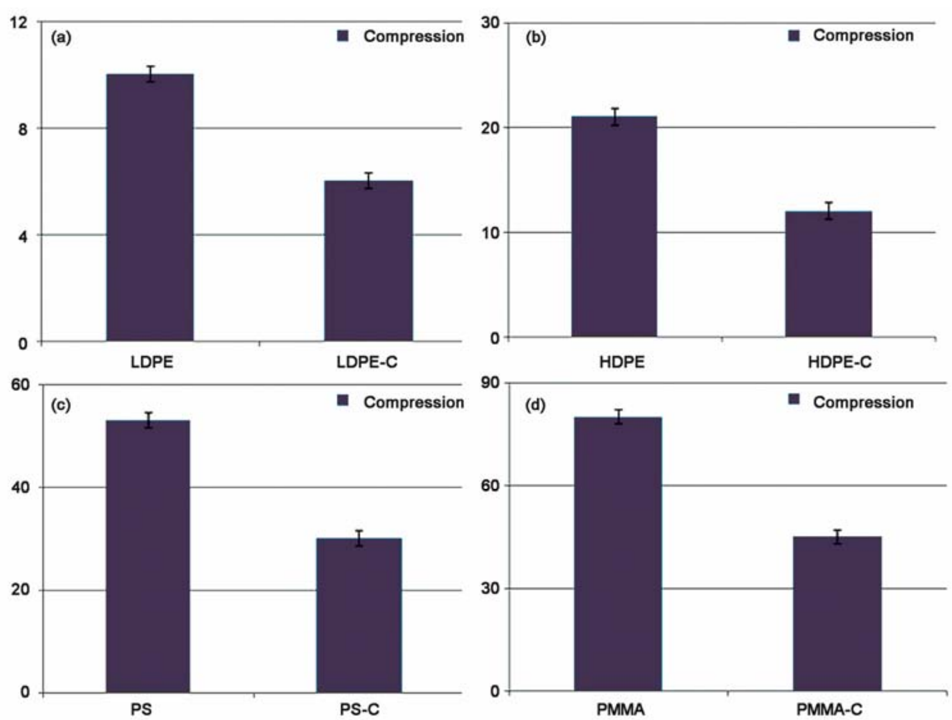

Fig. 3 – Compression strength (N/mm^2) of filled and unfilled (a) LDPE, (b) HDPE, (c) PS and (d) PMMA

compression strength of the unfilled LDPE is the least where as it is highest for the PMMA.

Impact energy

The impact energy data are displayed in Fig 4 (a-d), they show that there is a marginal decrease in values. Further, it is seen from the bar charts that the impact energy of HDPE/HDPE-C as a group is the highest and for PS/PS-combination, it is the least.

Tribological properties

Slide wear

The slide wear data are presented in Fig. 5 (a-d) for unfilled and fly ash cenospheres-filled LDPE, HDPE,

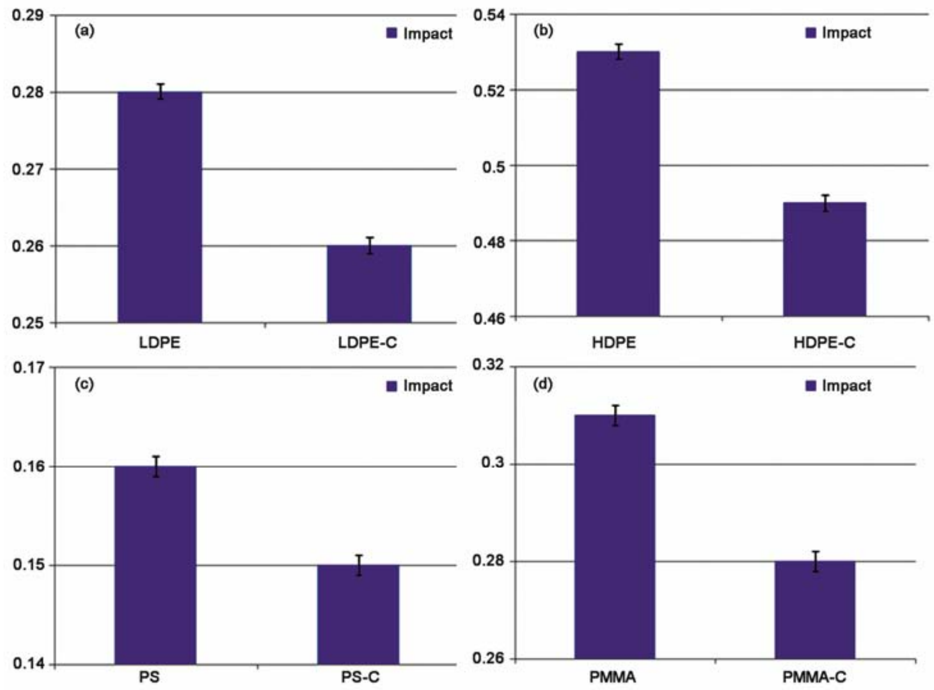

Fig. 4 – Impact energy (joules) values of filled and unfilled (a) LDPE, (b) HDPE, (c) PS and (d) PMMA

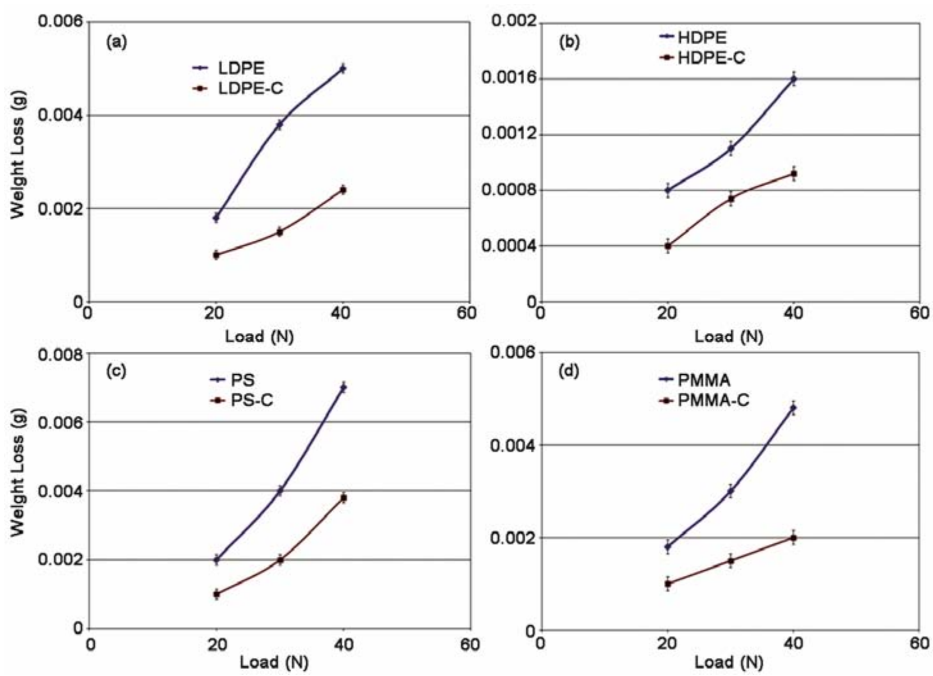

Fig. 5 – Plot of wear loss versus normal load of unfilled and cenosphere filled (a) LDPE, (b) HDPE, (c) PS and (d) PMMA

PS and PMMA composites for 20 N, 30 N and 40 N loads, respectively. It is evident that irrespective of the type of sample used, i.e., unfilled or the filled composite, the wear loss increases with increase in the applied load. The unfilled LDPE, HDPE, PS and PMMA, respectively, exhibit considerably higher wear loss as compared to their fly ash cenospheres-filled counter parts. The improvement in the slide wear characteristics (in terms of weight loss) of HDPE, PMMA, LDPE and PS composites are in the descending order. It is evident from the slide wear data that the HDPE - C shows the least wear loss compared to the other materials studied.

It is observed from the data in Fig. 5 (a-d) that higher the test load, higher is the wear loss whether in composite form or in plain polymer form. However, the unfilled showed steep gradient for the data compared to the filled ones thus highlighting the unique role of fillers in all the matrices tested.

The study on the slide wear of aluminum-based silicon carbide composites, reported by Singla *et al.*[26], revealed that the composites show much lower wear losses compared to the pure aluminium sample. They have attributed this trend to the higher hardness recorded for the composites. Anand and Kishore[27] also found that oxides in the form of corundum particles, to the extent of 5 wt% in Al based system reduced the wear loss and this reduction increased till about 30%. In a different investigation involving introduction of again a hard alumina but this time in an epoxy matrix[28], it was noted that as filler quantity in the matrix increased, the wear loss values decreased. In another work too, the distinct differences in surface features due to wear in epoxy in one case and glass fiber bearing in the other for identical test conditions have been highlighted using optical microscopy[29]. In the present case also the polymer composites exhibit lower wear losses, as the hardness of the composites are higher than that of the unfilled polymer samples. Working with thermoset (epoxy)-based system containing fly ash, Kishore *et al.*[21] observed that wear loss decreases with ash content. In the present case the matrix materialsemployed are thermoplastics and the content of filler is fixed unlike the earlier case involving epoxy based ones subjected to sliding wear with varying amounts of ash content.

Reference to transfer of film involving metal and polymer is available in the literature[30,31]. The importance of formation of thin and uniform transfer of film on the counter surface during sliding has been emphasized in these reports[30,31]. This aspect of film formation and correlating it to the wear rate requires a detailed experimental effort for the presently studied systems.

Coefficient of friction

The data in respect of coefficient of friction for all the samples evaluated are given in Table 3. It is observed that the coefficient of friction of LDPE composites at three different loads are less compared to unfilled LDPE. For other samples also, i.e., HDPE, PS and PMMA, similar trends are noticed. The difference in the friction values observed between LDPE and LDPE – C is more than that of the other set of materials investigated in this work. It is also observed from Table 3 that the co-efficient of friction increases with increase in load (20 N to 40 N) for all the four samples. Among the filled composites, HDPE-C has shown the lowest coefficient friction at all the loads. A noticeable point is, for an identical load, for the eight samples employed in this work, the HDPE–C shows the least co-efficient of friction values. In this context, it may be recalled that a similar increasing coefficient of friction values with load has been reported by Suresha *et al.*[32] working on fiber reinforced vinyl ester composites.

Further on, its relevant to recall the inferences drawn by Chauhan and Thakur[33] working on micro size cenospheres in vinylester system, wherein they have opined that ‘under dry sliding conditions increasing applied normal load (10 to 70 N) and sliding speed (1.88, 3.14. 4.39 and 5.65 m/s) increases the temperature at the interface’. They further pointed out ‘this increase in temperature causes thermal penetration to occur, which results in weakness in bond at the filler-matrix interface. They further state that ‘filler become loose in the matrix and shear easily

Table 3 – Co-efficient of friction of un-filled and filled polymers

	Sample designation							
Load	LDPE	LDPE – C	HDPE	HDPE – C	PS	PS – C	PMMA	PMMA – C
20.8 N	0.47	0.39	0.22	0.18	0.27	0.23	0.30	0.20
30.6 N	0.52	0.44	0.24	0.2	0.29	0.27	0.31	0.22
40.5 N	0.58	0.51	0.26	0.22	0.31	0.29	0.33	0.25

due to axial thrust and this results in reduction in co-efficient of friction value. The overall range in co-efficient of friction values reported for 15% filler addition to the matrix is from 0.4 to 0.55 for a sliding speed of 3.14 m/s and load in the range 30 to 50 N[33]. In the present investigation for similar sliding speed and load conditions, the coefficient of friction of the polymer composites with a slightly higher level of cenosphere addition, i.e., 18%, the value obtained is in the range 0.3 to 0.5. The agreement as regards the range in friction between the present effort and earlier one[33] is a notable point.

In another work[34], it is reported that addition of cenosphere of about 20% resulted in decrease of co-efficient of friction from 0.40 to 0.34 for a load of 16 N and a sliding speed of 1.2 m/s. In the present case also, the cenosphere addition of 18% resulted in the friction levels in the range 0.22 to 0.18 at 20.8 N load and a sliding speed of 3.14 m/s. Hence, the present data are in broad agreement with the published data trends.

Microscopic studies

The slide wear data presented earlier are now discussed based on the scanning electron microscopic (SEM) features. The arrow marks shown in the SEM pictures represent the direction in which the sliding has taken place. For microscopic studies, the loads selected were 20 and 40 N. The two loads were selected as they represent the minimum and maximum loads tried in this investigation as well as based on the earlier work on polymer composite systems. The SEM pictures to examine the worn surfaces of unfilled and filled systems of LDPE and LDPE-C are shown in Fig. 6 (a) and (b), respectively. They are pertaining to the test conditions of 40 N load and sliding speed of 3.14 m/s. Thus, unfilled LDPE shows (Fig. 6a) higher wear out features on the surface such as, deeper grooving, clear detachment of material from the surface leading to crater formation as for instance at arrow makes G & H and some and some faintly visible cracks on the surface (example; arrow mark I). LDPE - C (Fig. 6b), on the other hand, shows less of such wear out features. The microscopic features thus corroborate the wear data presented earlier in Fig. 5(a).

The SEM pictures of HDPE and HDPE-C, shown in Fig. 7(a) and (b), respectively, for the test conditions of 20 N, 3.14 m/s are now considered.

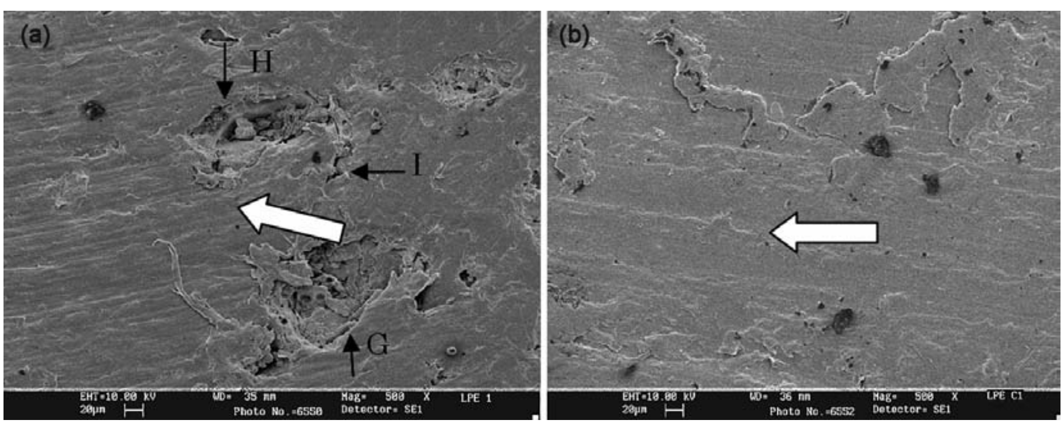

Fig 6 – Worn surface SEM picture of (a) unfilled LDPE and (b) LDPE-C at 40 N load

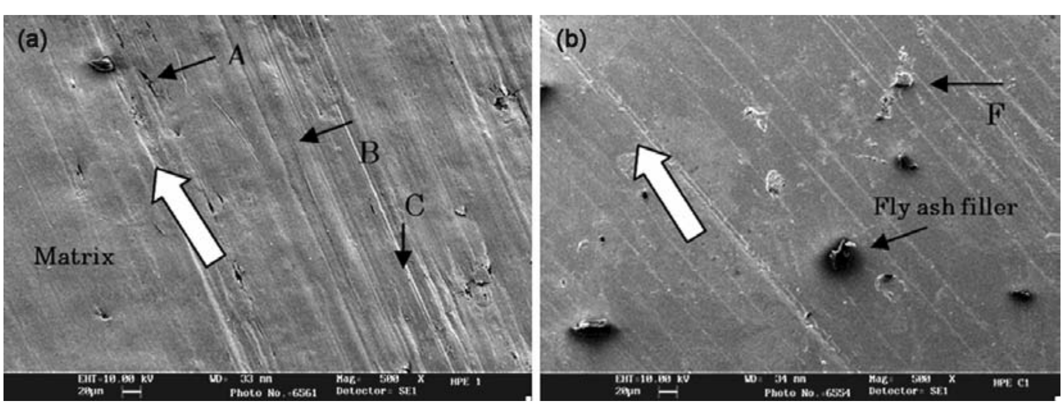

Fig. 7 – Worn surface SEM picture of (a) unfilled HDPE and (b) HDPE-C at 20 N load

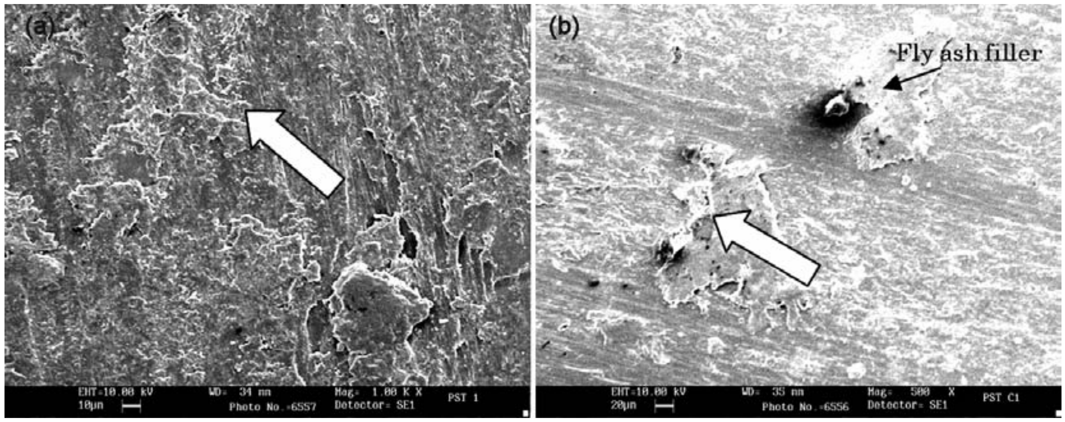

Fig. 8 – Worn surface SEM picture of (a) unfilled PS and (b) PS-C at 40 N load

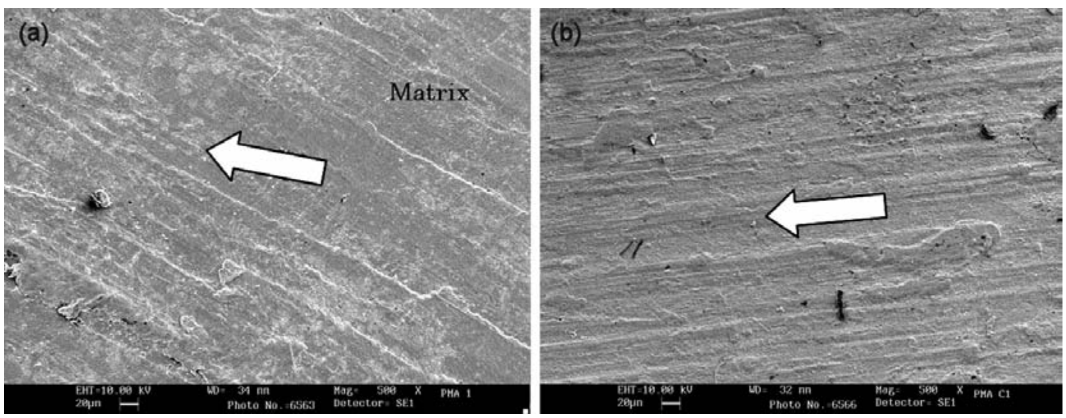

Fig. 9 – Worn surface SEM picture of (a) unfilled PMMA and (b) PMMA-C at 20 N load

Figure 7(a) shows the worn surface of unfilled HDPE, where in faint cracks along the sliding direction (shown by arrow at A), matrix displaying grooving tendencies (arrow mark at B) and a slight tendency for material removal (arrow mark at C) leading to small pits are observed. In Fig. 7 b, the fly ash bearing region is shown at arrow mark F. In HDPE-C such tendencies are less (Fig. 7b) thus supporting the experimental wear test data presented in Fig. 5b.

The SEM features of unfilled PS subjected to 40 N load and sliding velocity of 3.14 m/s are shown in Fig. 8 (a) and (b), respectively. The unfilled sample (Fig. 8a) shows more wear and debris formation on the surface, grooving and some tendency for matrix cracking. The PS-C on the other hand, shows (Fig. 8b) less of such features supporting the wear data trend in Fig. 5c.

The SEM features of unfilled PMMA and PMMA-C samples subjected to 20 N load and sliding velocity of 3.14 m/s are shown in Fig. 9(a) and (b), respectively. These features support the wear trend seen in Fig. 5(d). The unfilled PMMA shows (Fig. 9a) more surface distortion compared to cenospheres filled PMMA (Fig. 9b).

An investigation on various polyaryletherketone composites with glass fibers by Harsha *et al.*[17] showed improved tensile and flexural strength properties for PEK composite as compared to PEEK composite. In that work[17], the volume fractions of fibers in PEK and PEEK have been kept constant. Good correlations have been reported in respect of erosion, mechanical properties and SEM worn surface features. In the present work also, one can observe good structure property correlation amongst wear, mechanical properties and SEM features for all the four different types of composites studied.

Conclusions

The following conclusions are emerged from this investigation.

The density value decreases when the fly ash cenospheres are added to the thermoplastic polymers.

The hardness increases following the inclusion of cenosphere filler.

The compression and impact strengths decrease with the addition of fillers. The numerical value of reduction of compression strength is least for LDPE. As regards impact energy, its numerical value change is least for the PS system.

The slide wear resistance of cenosphere based composites is better compared to the un-filled ones. Further, the wear loss of both unfilled and filled samples increases with increase in load.

The co-efficient of friction values of polymer samples with cenospheres are slightly lower than that of unfilled counterparts. Further, the friction co-efficient increases with increase in load irrespective of the type of sample tested. The inclusion of cenospheres has thus contributed significantly to the reduction of friction and increasing the wear resistance thereby indicating a possible end use in an industry for the systems.

Among the polymer materials investigated, HDPE seems to be the best choice for wear resistance applications in view of their improved slide wear characteristics, decrease in coefficient of friction, less percentage decrease in respect of compression strength and impact energy compared to the other polymer materials studied.

Acknowledgements

The authors wish to thank the management of Sambhram Institute of Technology, Bangalore and Central Power Research Institute, Bangalore for according permission to publish this paper. Also, the help rendered by R. Manjunatha in the preparation of the manuscript is gratefully acknowledged.

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