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## Study of tribological properties of cenospheres filled HDPE composites

M.V.Deepthi<sup>1</sup>, R.R.N.Sailaja<sup>1\*</sup>, P.Samathkumaran<sup>2</sup>, S.Vynatheya<sup>2</sup>, S.Seetharamu<sup>2</sup><sup>1</sup>The Energy and Resources Institute, 4<sup>th</sup> Main, 2<sup>nd</sup> Cross, Domlur 2<sup>nd</sup> Stage, Bangalore-560 071, (INDIA)<sup>2</sup>Central Power Research Institute, Bangalore-560 080, (INDIA)Received: 1<sup>st</sup> November, 2009 ; Accepted: 10<sup>th</sup> November, 2009

### ABSTRACT

High Density Polyethylene (HDPE) composites have been made using light weight cenospheres as filler material in different volume fractions along with the incorporation of HDPE-g-DBM compatibilizer for improved interfacial adhesion and tribological characteristics. Cenospheres are hollow ceramic based microspheres found in fly ash, a natural by-product of coal combustion during the generation of electric power. Cenospheres are easy to handle and provide a low surface area-to-volume ratio. Utilization of cenospheres reduces the cost of manufacturers' raw materials and can lead to the development of lightweight composites. The study comprising of preparation and evaluation of various tribological properties of HDPE with cenospheres as filler material at 3 levels and HDPE-g-DBM as compatibilizer has been reported since similar kind of work could not be cited in the literature. The tribological studies have been done using a Pin-on-Disc type of wear test rig with the pin as the HDPE composite material sliding over a hard disc. The damage assessment of the worn out samples has been carried out using Scanning Electron Microscopy (SEM). The results showed that as the cenosphere content is increased, both slide wear loss and coefficient of friction values decreased. The HDPE material without any filler showed the highest wear loss. The coefficient of friction of HDPE matrix in comparison with HDPE-cenosphere based composites exhibited mixed trends. Compatibilization favored wear resistance although the excessive compatibilizer decreased tribological properties of the composites.

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### KEYWORDS

HDPE;  
Cenospheres;  
Compatibilizer;  
Tribological properties;  
Coefficient friction.

### INTRODUCTION

In recent years, the usage of polymeric composites have increased in industrial applications including thermal power stations. Thermoplastics composites

are known for possessing good mechanical and tribological properties due to addition of filler or reinforcements. With different types and contents of filler material such as organic and inorganic types, tailor made properties can be achieved. The type of matrix or filler

contents or reinforcements yield good properties such as high load bearing capability, improved wear resistance, higher strength etc. depending upon the type of application the material is subjected to.

Various researchers<sup>[1-4]</sup> have reported that the wear resistance of polymers is improved by the addition of fillers such as Kaolin, Talc, CuO, fly ash, Hydroxyapatite etc. Studies by Wang<sup>[2]</sup> showed that, Hydroxyapatite [HA] reinforced high density polyethylene [HDPE] composites have lower coefficients of friction than unfilled HDPE under certain test conditions and also exhibited less severe fatigue failure marks than HDPE. It has been reported by Palabiyik et.al.<sup>[3]</sup> that the additions of CuO and polytetrafluoroethylene [PTFE] in HDPE showed lower wear loss and coefficient of friction. In a recent review<sup>[4]</sup>, Yang reported that by reinforcing the multi wall carbon nanotubes (CNT) into ultra-high molecular weight polyethylene (UHMWPE) and high density polyethylene (HDPE) polyblend yielded improvement in the wear performance of the composites. The specific wear rate of the composites decreased with increasing CNT content. Hung et.al.<sup>[5]</sup> evaluated the tribological and mechanical properties of silane treated fly ash as plastic filler and the investigation revealed that the polymers containing flyash as filler material improved the wear properties. A similar work in the non ferrous area where in flyash particulates used as filler in aluminum based metal matrix<sup>[6]</sup> showed improvement in resistance to wear increases with increase in flyash content.

Introduction of third component as compatibilizer has found to improve the tribological behavior of the polymer composites<sup>[7,8]</sup>. Jiansong et.al.<sup>[7]</sup> studied the tribological behaviour of compatibilized UHMWPE/Liquid crystalline polymer composites with PE-g-Maleic anhydride. It is reported that the inclusion of compatibilizer helped in improving the wear resistance of the composites.

The use of HDPE as a matrix material with hollow light weight cenosphere as filler along with the incorporation of suitable compatibilizer has not been reported in the literature so far. Hence, the present work aims at studying the physical and tribological properties of HDPE / Silane grafted cenosphere composites at 3 levels prepared in the laboratory.

## EXPERIMENTAL

### Materials used

HDPE [grade 24FS040 with melt flow index 10g/10 min. from Reliance Petrochemical Ltd. India] is used as matrix. Cenosphere used as filler material having low density and particle size of 100-350 microns. It is treated with 3-Amino propyl triethoxy silane [APTS] obtained from sigma Aldrich (USA).

### Silane grafting

Silane grafted cenospheres can be synthesized (as done earlier by Hongping et.al.<sup>[9]</sup>) by grafting APTS onto cenospheres by silanation process. The grafting reaction is carried out in a mixture of water/ethanol [20:80 by volume] and the procedure is given below.

A quantity of 100ml of APTS is first introduced into 1000ml of the mixture of water/ethanol at 80°C. Then 50g of cenospheres is added to the above mentioned solution and continuously stirred for 5-6 hr at 80°C. Then the reaction products are filtered and washed many times using H<sub>2</sub>O/ethanol mixture and oven dried. The resultant product thus obtained is characterized for silane coating using Fourier transform infrared spectroscopy (FTIR).

### Synthesis of the compatibilizer

HDPE-g-DBM can be synthesized<sup>[10]</sup> by the dissolution of HDPE in 0-dichlorobenzene at 120°C; 0.2% of the dicumyl peroxide initiator is then added along with DBM. The reaction is carried out for 3h at 120°C with continuous stirring. The product is then cooled and precipitated in methanol. Further, the precipitate obtained are filtered, washed several times with methanol and finally rinsed with acetone and dried.

### Test samples preparation

Polymer composites of HDPE, Cenospheres (Silane treated) and HDPE-g-DBM was melt mixed in various proportions at 270°C using Brabender (CMEI, Model-16CME SPL). The test specimens were then molded using a hot press. The amount of compatibilizer added is expressed as the weight percent of cenospheres. The test samples are subjected to hardness and wear testing.

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### Wear test method

A pin-on-disc type of wear test set-up is used for abrasive wear experiments with a pin sliding over a hard disc fixed with 800 grade SiC paper. The surface of the sample (6mm×6mm× 3mm) glued to a pin of dimensions 6mm diameter and 22 mm length and comes in contact with a hard counter surface of HRc 62. The test is conducted on a track of 115 mm diameter hard disc by selecting the test parameters namely, test duration, load and speed.

The pin assembly is weighted initially using a digital electronic balance. The test is performed under a set of identical conditions by applying normal load (N) and run for a constant sliding distance at a constant speed of 200rpm. At the end of the test, the pin assembly is again weighted. The difference between the initial and final weights is a measure of wear loss. The test is done on three repetitive samples and the average value is reported. The friction force at the sliding interface of the specimen is measured. The co-efficient of friction is obtained by dividing the frictional force with the applied normal force. The test runs are conducted at a load of 16 N and sliding velocity of 1.2m/s. The examination of the samples subjected to slide wear tests is carried out using scanning electron microscopy (SEM). Prior to this, a thin layer of gold coating on the surface of the samples is done.

## RESULTS AND DISCUSSION

The tribological properties of HDPE samples with cenospheres at 3 levels (10-30 wt %) and compatibilizer at 4 levels (0-15 wt% of cenospheres) have been examined for wear properties. Figure 1 shows the FTIR data of untreated and silane treated cenospheres. A sharp peak at 2925 is recorded in the FTIR spectrum. This corresponds to C-H, stretching mode of APTS, indicating the existence of silane in the grafted products<sup>[9]</sup>.

### Wear loss

Figure 2 shows the wear loss (g) of the HDPE/Cenospheres composites with the addition of cenospheres at a load of 16 N and sliding velocity of 1.2m/s. It is observed that, as the cenospheres content

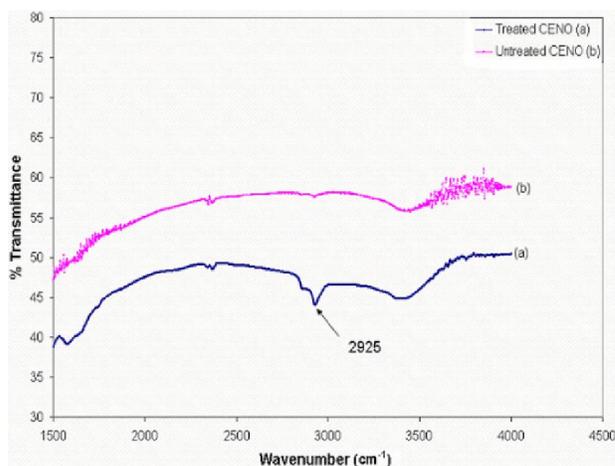


Figure 1 : FTIR spectrograms of untreated and silane treated cenospheres.

is increased from 0-30%, the wear loss decreases. Composites with 30% cenospheres shows significantly lower wear loss compared to composites with 20% and 10% additions. Similar results have been reported by Aurrekoetxea et.al.<sup>[11]</sup>. It shows wood reinforced polypropylene composites shows better wear performance than its neat constituent (PP).

It is also noticed that the incorporation of compatibilizer improves the wear resistance of the composites. Minimum wear loss is obtained for composites containing 10% compatibilizer. The amine groups of silane treated cenospheres can react effectively with the ester group of the compatibilizer, thereby, anchoring HDPE and cenospheres particle together. However, with further addition of compatibilizer beyond 10% (i.e.15%), there is an increase in the wear loss value by 10% which might be due to the saturation of reactive sites. This finding shows that the addition of treated cenospheres to HDPE contributed to the improvement in wear characteristics significantly and also further enhanced by compatibilization.

### Coefficient of friction

The variation in coefficient of friction with varying cenospheres and compatibilizer content is shown in figure 3. Incorporation of cenospheres to HDPE matrix gives raise to lower coefficient of friction compared to pure HDPE. It is observed that, as the cenospheres content is increased, the coefficient of friction decreased irrespective of the presence of compatibilizer. The introduction of compatibilizer up to 10 % has good influ-

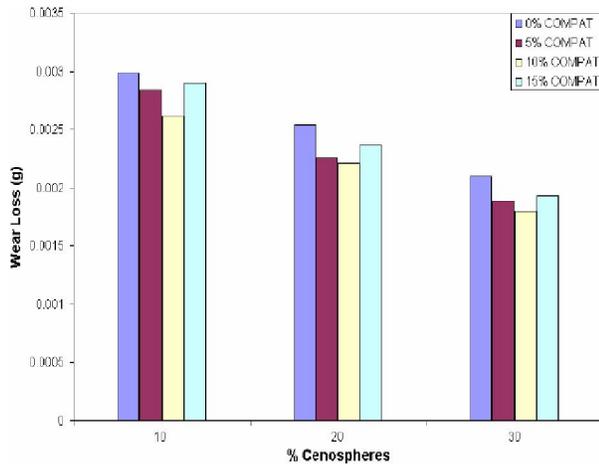


Figure 2 : Weight loss versus weight percent cenospheres for different loadings of compatibilizer.

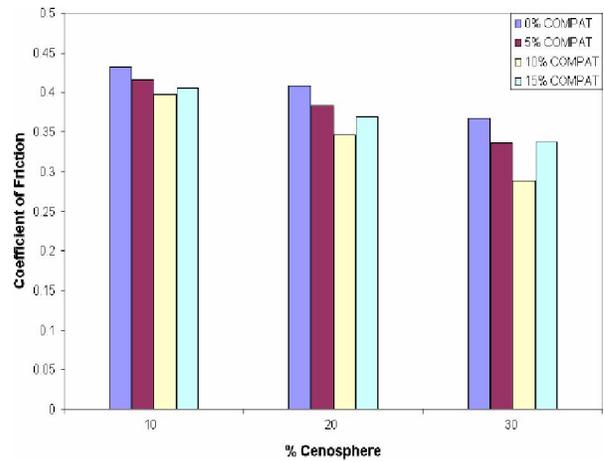


Figure 3 : Coefficient of friction versus weight percent cenospheres for different loadings of compatibilizer.

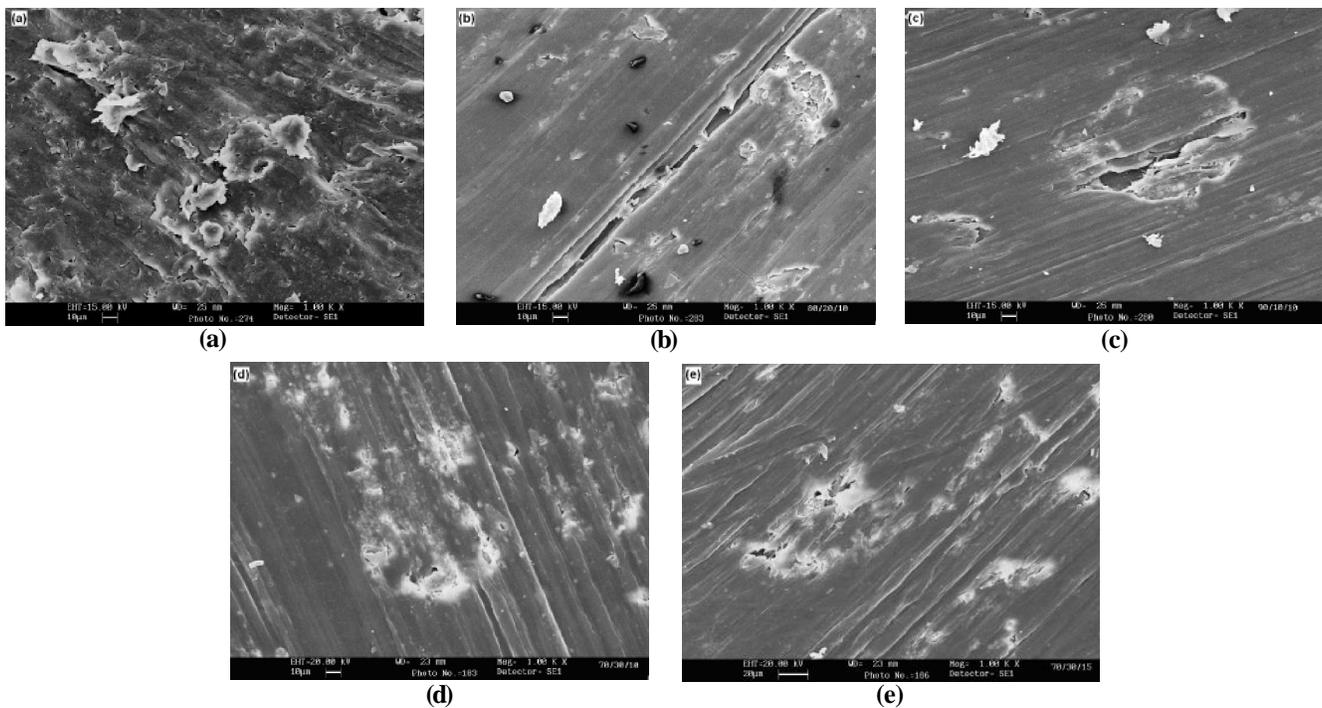


Figure 4 : SEM photographs of worn HDPE/Cenospheres composites containing, (a) 10% cenospheres and 0% compatibilizer, (b) 10 % cenospheres and 10% compatibilizer, (c) 20% cenospheres and 10% compatibilizer, (d) 30% cenospheres and 10% compatibilizer, (e) 30% cenospheres and 15% compatibilizer.

ence on the coefficient of friction since its value decreases. However, further addition of compatibilizer beyond 10% resulted is detrimental for wear properties of the composites. These data trends are corroborated with SEM features and the same is covered below.

### SEM observations

Figure 4(a) shows the worn surface features of uncompatibilized HDPE/Cenospheres composites con-

taining 10% cenospheres loading run for 0.345m at a load of 16 N and sliding velocity of 1.2m/s, wherein the highest wear loss is recorded. From this figure, it is evident that the debris formation is more compared to samples having higher cenospheres and compatibilizer concentrations, since the matrix is distorted due to higher wear out. The compatibilized (with 10% compatibilizer) counterpart [Figure 4(b)] for the above composites shows a lower wear loss compared to that shown in Figure 4(a). Here, breaking up of matrix along a length

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and less debris formation is noticed [Figure 4(b)]. An increase in cenospheres content from 10 to 20%, further reduced the debris formation as exhibited in the SEM micrographs shown in figure 4(c). In this case, onset of shearing of the matrix is observed along with the debris formation in the vicinity of the tearing points. When compared to all, the least wear loss is exhibited by the composites containing 30% cenospheres with 10% compatibilizer as seen from the figure 4(d). The compatibilized composites have improved matrix-filler adhesion. Hence, the formation of debris is accompanied by severe matrix deformation indicating resistance for the removal of cenospheres particles during wear. A similar observation is seen in figure 4(e) with increased compatibilizer content (at 15%) although the debris formation is now localized. The increased compatibilizer content has influenced the wear process by way of tearing of the matrix at different locations and appearance of grooving marks throughout the matrix along the wearing direction.

## CONCLUSIONS

The tribological properties of silane treated cenospheres/HDPE based composites are dictated by the filler material i.e., cenospheres and also the HDPE-g-DBM compatibilizer contents. The enhanced interfacial adhesion properties are seen for an optimum concentration of 30% cenospheres containing 10% compatibilizer, where as highest wear loss and coefficient friction are obtained for the pure HDPE sample. Also, HDPE composites with 30% cenospheres shows significantly lower wear loss and coefficient of friction compared to composites with 20% and 10% loadings. All the wear and friction deductions are substantiated by the SEM pictures through worn surface features. For low friction and wear applications, it is desirable to employ high filler addition with 10% compatibilizer.

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