

Effects of Silica Ceramic Particle Sizes on the Properties of Recycled Polyethylene Composites

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Abstract

Particulate filled polymeric composites are becoming attractive because of their wide applications and lower production cost. To evaluate the possibility of using waste silica based ceramic materials as reinforcing filler in recycled low density polyethylene (LDPE) composite, the effect of ceramic particle (PC) sizes on the mechanical, wear and thermal behaviours of LDPE composites were investigated at (2wt% filler) and grain sizes (40 – 150 μm). The wear behaviour was characterized using analysis of variance (ANOVA) and linear regression to determine the main and interactive effects of the process parameters such as speed, load and time on the wear behaviour of the fabricated low density polyethylene reinforced with ceramic particles (LDPE-PC) composites. Test results show that the decreasing of the ceramic particles 40 – 150 μm improved the mechanical, wear and thermal properties of the LDPE-PC composites. Factorial design of the experiment can be successfully employed to describe the wear behaviour of the samples and developed linear equation for predicting wear rate with in selected experimental conditions.

Key words: Polymer matrix composites; Mechanical; Wear and thermal properties.

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INTRODUCTION

In recent times, new composites are being developed in the continual quest to improve material performances^[1]. Thus material scientists, engineers and scientists are always striving to produce either improved traditional materials or completely new materials. Recent investigations of polymer based composite materials have developed many methods for polymer formulations and have allowed the manufacture of new products with optimal properties for special applications^[2-4]. Polymer matrix composites are increasingly finding industrial application because of their unique combination of mechanical, electrical and thermal properties. Typically, they have high specific strength and modulus, excellent fracture toughness and fatigue properties and good corrosion, thermal and electrical resistance properties.

Polymers are being used in a wide variety of tribological applications such as dry bearings, seals, piston rings, slides, prosthetic joints, gears, tires and brake shoes. As polymers become more widely used as bearing and sliding materials, their friction and wear properties become of greater interest and importance. Despite several potential advantages, there are still many challenges which remain for the effective and economic use of polymers for specific tribological applications^[4]. For example, the wear rate can be extremely high if contact pressure is slightly raised, and low friction is not necessarily associated with low wear rate. Although virgin polymers have shown excellent tribological properties in some very uneconomical cases, they are rarely used as monolithic one-phase materials; not least for economic reasons as fillers are often relatively less expensive than the polymer.

An addition of second phase in the form of particulate making composites has many structural advantages that has not be extensively utilised. It is possible to improve polymer tribological performance by certain change in the bulk constituents or the surface conditions of the material^[5-8]. The main advantage of adding hard and

strong phases to soft matrix is in increasing the stiffness of the matrix; there may be corresponding loss of toughness or fatigue life. The effects of several different types of fibres on the friction and wear properties of polymer have been studied extensively^[9–13]. A summary of some of these results can be usefully presented in a friction and wear map format^[12]. Studies on polymer matrix composites subjected to sliding and abrasive wear indicate that wear resistance depends on the detailed properties of the material as well as the external wear conditions such as applied pressure and contact velocity^[4].

Despite some progress in the understanding of polymer, further research is needed to tap the great potentials of using polymers for wear applications. The use of adding hard and strong phases to soft matrix to improve the stiffness of the matrix without affecting the mechanical properties of the bulk of the polymer appears to have some great potential. While the friction is controlled by the self lubricating property of the matrix, wear resistant is improved because the load carrying capacity of the composite is basically increased due to the presence of the hard and strong second phases^[13]. Good examples of this kind of polymer will be those with glass or carbon fibers in thermoplastic or thermosets matrices. Several kinds of hardphases such as ceramic oxides are also added to polymer matrix with varying effect. This study therefore seeks to develop a new polymer composite from recycled polyethylene (matrix) reinforce silica based ceramic oxide. The wear characteristic, thermal and mechanical properties of the developed composites were evaluated and studied with the aid of scanning electron microscope (SEM) and statistical tools. The significant of the main and interactive effects of the process parameter (load, speed, time and reinforcer) were carefully studied in relation to the wear behavior.

1. METHODOLOGY

The silica ceramic was grinded into powder and sieved in accordance with BS1377:1990 standard^[4]. The chemical composition of the silica ceramic used in this work is shown in Table 1. The various particles sizes of the PC and the LDPE were blended together using a two-roll rheomixer at 50°C and a rotor speed of 60 rpm. The sizes of the filler (PC) in the matrix were varied from 40 to 150 μm to produce five different samples. Compression of the composites was carried out with a Wabash machine V200 hot pressing machine for 7 minutes under controlled pressure (30 tons) at 150°C. Each of the samples was cooled to room temperature under sustained pressure before it was removed from the press. Before testing all samples were conditioned for 72 hours at a temperature of $25 \pm 2^\circ\text{C}$ and a relative humidity of $(55 \pm 5)\%$ ^[8].

Scanning Electron Microscope (SEM), model EVO-MA10 LaB6 Analytical VP-SEM was used to determine the morphology of the samples at 20kV. The results of the SEM micrographs are presented in Plate 1 – 3.

The tensile test of the composite samples was conducted on an Instron testing machine with a strain rate of $2 \times$

10^{-3}s^{-1} as specified by the American Society for testing and Materials^[6,7].

The hardness of the samples were determined according to the provision of America Society of testing and Materials using Rockwell hardness tester on B scale (Frank Welltest Rockwell Hardness Tester, model 38506) with 1.56 mm steel ball indenter, minor load of 10 kg, major load of 100 kg and hardness value of 101.2 as the standard block^[6,7]. Before the test the mating surface of the indenter, plunger rod and test samples were thoroughly cleaned by removing dirt, scratches and oil. The calibration of the testing machine was done using the standard block. The samples were placed on anvils, which acts as a support for the test samples. A minor load of 10 kg was applied on the sample in a controlled manner without inducing impact or vibration and zero datum position as established, and then major load of 100 kg was then applied. The reading as taken when the large pointer came to rest or had slowed appreciably and dwelled for up to 2 seconds. The load was then removed by returning the crank handle to latched position and the hardness value read directly from the semiautomatic digital scale. Average of three readings was taken for the hardness test.

The impact test of the composite samples was conducted in accordance with ASTM D256-93^[6–8] using a fully instrumented Avery Denison test machine. Charpy impact test was conducted on notched samples.

A pin-on-disc apparatus was used to investigate the dry sliding wear behaviour of the polymer composite. 150 μm mesh emery paper with 180 mm diameter surface was mounted on the pin-on-disc apparatus. The experiment was carried out with varied speed, time and load. The weight of the samples were taken before the wear test and after the test with an electronic scale with accuracy 0.001 mg; the difference between the two weights was calculated which indicated the amount of composite material that wore off. After each test, the worn out samples were cleared from the emery paper with a compressed dry air blower. A parameter referred to as wear coefficient, which define the wear severity was calculated using Equation (1.1). Experiments were conducted in accordance with standard 1.8 orthogonal array, with a view to investigate which of the design parameters; speed, load and time most significantly affect dry sliding wear and which combinations of these parameters^[2, 4].

$$\text{Wear Rate} = \frac{\text{Wear volume loss}}{\text{Sliding distance} \times \text{applied load}} \quad (1.1)$$

Factorial design and linear regression methods a common tools used in engineering analysis was used in the data acquisition. Two levels of each of the four factors were used for the statistical analysis. The levels for the four factors are entered in Table 2. The model equation was obtained by representing the wear value by W , as a function process parameters speed (A), time (B), load (C) and the particle sizes (D) (Equation (1.2)).

$$W = f(A, B, C, D) \quad (1.2)$$

Table 1
Chemical Composition of Ceramic Particles

Ceramic Particles	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	Na ₂ O	K ₂ O	P ₂ O ₅	TiO ₂	MgO	MnO	PF
Percentage (%)	62.36	19.20	2.51	1.85	0.23	1.80	0.05	0.81	2.15	0.01	7.1

Table 2
Process Parameter For the Wear Test

Factor	Name	Low level	High level
A	Speed/(m · s ⁻¹)	1.18	2.36
B	Time/s	60	120
C	Load/N	6.5	14.2
D	Reinforcer/μm	40	150

The general model is represented as shown in Equation (1.3)

$$\begin{aligned}
 W = & \beta_0 + \beta_1A + \beta_2B + \beta_3C + \beta_4D \\
 & + \beta_5AB + \beta_6AC + \beta_7AD + \beta_8BC + \beta_9BD + \beta_{10}CD \\
 & + \beta_{11}ABC + \beta_{12}ABD + \beta_{13}ACD + \beta_{14}BCD + \beta_{15}ABCD
 \end{aligned}
 \tag{1.3}$$

Where β_0 is average response of W and $\beta_i (i = 1 - 15)$, are coefficients associated with each variable A, B, C, D and interaction.

Thermal decomposition (TA) was observed in terms of global mass loss by using a TA Instrument TGA Q50 thermogravimetric analyzer. The apparatus detects the mass loss with a resolution of 0.1 as a function of temperature. The samples were evenly and loosely distributed in an open sample pan of 6.4 mm diameter and 3.2 mm deep with an initial sample weight of 8 – 10 mg. The temperature change was controlled from room temperature (25 ± 3) to 700 °C with a heating rate of 10 °C/min^[14-17].

2. RESULTS AND DISCUSSION

2.1 Mechanical Properties

The stress-strain curve of the LDPE matrix is showed in Figure 1(a). In Figure 1(a), it is clear that the material started necking at 0.4 mm/m. From Figures 1(b)-1(f), it was observed that the materials become progressively tougher (increased strain) as the particle size of the reinforcer becomes finer. The strain at which the maximum tensile strength occurs are 0.5 mm/mm for Figure 1(b), 0.45 mm/mm for Figure 1(c), 0.5 mm/mm for Figure 1(d), 0.6 mm/mm for Figure 1(a) and 0.8 mm/mm for Figure 1(f) respectively. These results are in par with the works of other researchers^[5-8]. These observations captured in one sentence, is that the smaller the particle size, the tougher (increased strain) the composite becomes except of course, in the case of Figure 1(b) where the material showed some level of high brittleness. Figure 1(b) resembles the stress-strain curve for brittle material which is typically linear

over a range of strain, eventually terminating in a fracture without appreciable plastic flow.

From Table 3, shows the extrapolated values of the mechanical properties from a stress-strain curve of two extreme particles size from the fabricated LDPE reinforced composites. As the particle size of the filler increase, the mechanical properties of the composites change. From Table 3, the yield-stress, UTS, strain at UTS and strain at fracture all reduce with increasing particle sizes this is in line with the research of^[1,6]. The high values of tensile strength observed as the particles size of the ceramic particles decreased may be due to the good distribution and dispersion of the silica ceramic particles in the polymer matrix resulting in strong-particles-matrix interaction. This good particles dispersion improves the particles-polymer matrix interaction and consequently increases the ability of the reinforcer to restrain gross deformation of the polymer matrix. The tensile strength obtained in this study remained within acceptable levels for car bumper^[5-8].

Figure 2, represents the hardness values of LDPE matrix and the LDPE-PC composites against the grain size of the reinforcer (ceramic particle). It can be observed that the hardness values of the material increased as the particle size of the reinforcer increased. The increase in hardness can be attributed to the hard ceramic reinforcer. This is due to increase in the hard and brittle phase of the ceramics body in the polymer matrix. In comparison with the unreinforced matrix, a substantial improvement in hardness values was obtained in the reinforced polymer matrix. This is in line with the earlier work of^[8]. From Figure 2, the impact value of the LDPE-PC composites increase as the particles size of the second phase decreases.

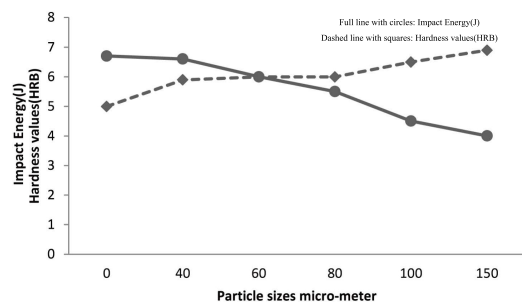


Figure 2
Stress-strain Curve for Samples at Different Wave Lengths

High strain rates or impact loads may be expected in many engineering applications of polymer composite materials. The suitability of a polymer composite for such applications should therefore be determined not only

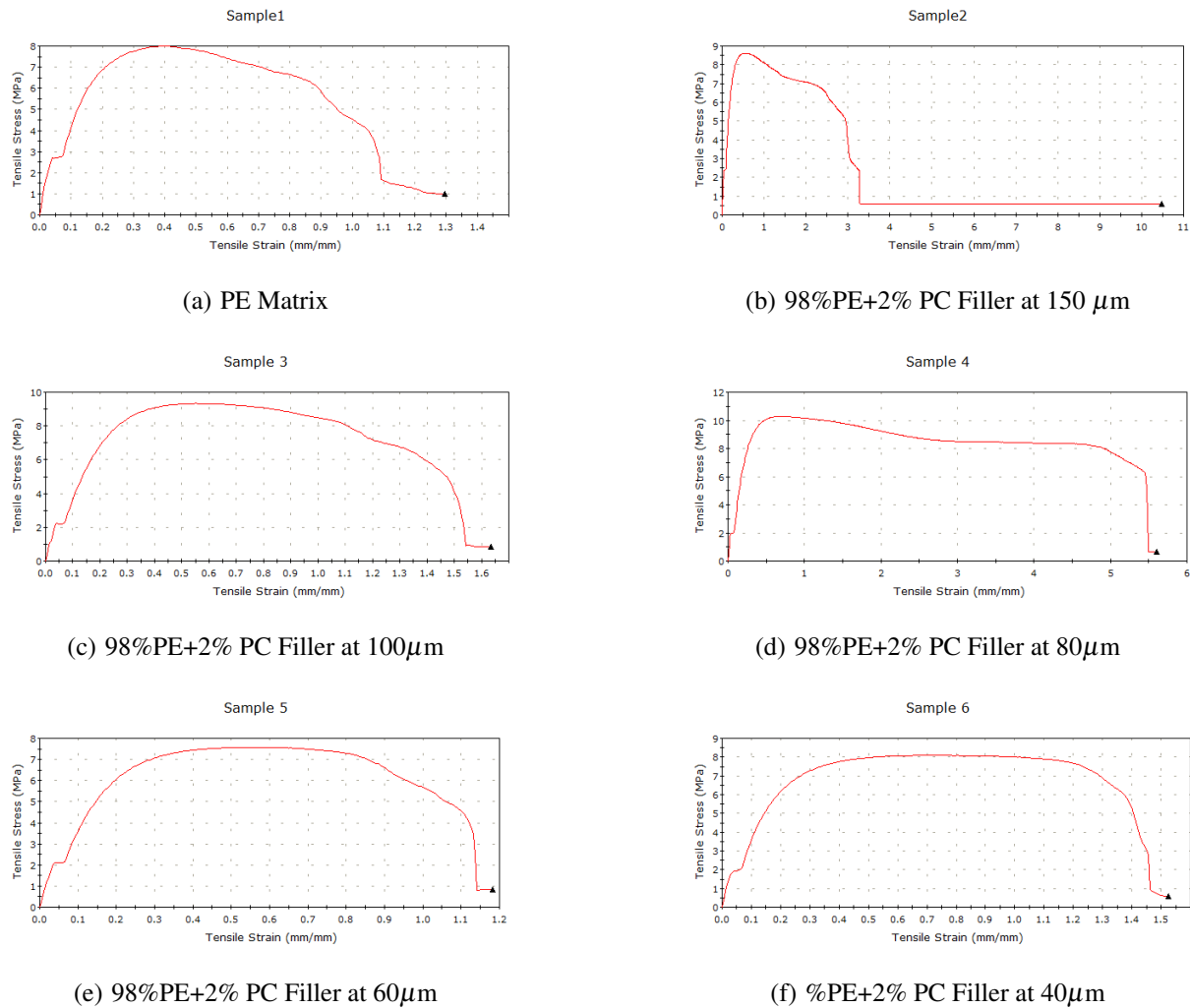


Figure 1
Stress-strain Curve for Samples at Different Wave Lengths

by usual design parameters, but by its impact or energy absorbed^[4–8]. The steep increase in the impact strength of the composites as the particle sizes of the reinforcer decreased could be attributed to the presence of particles well bonded by the polymer matrix^[4]. This factor leads to increase in impact energy (see Figure 2).

The steep improvement in the impact energy of the composites could also be attributed to good interfacial bonding between the matrix and the particles. This is achieved due to large surface area of the reinforcer as the particles size decreased. These results are in agreement with the work of other researchers^[4–8]. The increases in impact energy with increasing particles loading might also be due to the increased deformability of a rigid interface between the particles and matrix^[8].

2.2 Wear Test Result

The experiments were conducted with an aim of relating the influence of sliding speed (A), Time (B), applied load (C) and particle sizes (D) with dry sliding wear of both the

composites under study. On conducting the experiments as per the factorial design, the dry sliding wear results for various combinations of parameters were obtained and shown in Table 4. The presence of ceramic particles in the polymer matrix lowers the wear rate (see Table 4). This may be attributed that the composites materials are harder than the polymer matrix (see Figure 2). Because of increase in the hardness values of the materials the wear rate decreases obviously. The various researchers^[9–13] have predicted that the presence of hard filler materials in polymer materials increases the dry sliding wear resistance. Also the decrease in wear rate of the composites may also be attributed to higher load bearing capacity of hard reinforcing material and better interfacial bond between the particle and the matrix reducing the possibility of particle pull out which may result in higher wear^[12,13].

The analysis of variance was used to investigate which design parameters significantly affect the wear characteristic. It was accomplished by separating the total variability of the wear result, which is measured by sum of the squared deviations from the wear rate obtained, into contributions

Table 3
Mechanical Properties of LDPE-PC Composites at Different Compositions

Grain size / μm	Yield stress / MPa	UTS / MPa	Strain at UTS	Strain at fracture
0	2.7	8	0.4	1.1
40	2	8	0.8	1.45
60	2.2	7	0.6	1.15

Table 4
Design Layout and Response Data for Wear Study

Standard order	Speed/($\text{m} \cdot \text{s}^{-1}$)	Time/s	Load/N	Grain Size/ μm	Wear-rate/g
1	2.36	120	14.2	150	0.309
2	2.36	60	14.2	40	0.105
3	1.18	120	14.2	40	0.045
4	1.18	120	6.5	150	0.46
5	2.36	120	6.5	40	0.031
6	1.18	60	6.5	40	0.024
7	2.36	60	6.5	150	0.023
8	1.18	60	14.2	150	0.027

by each of the design parameters and the errors (see Table 4). The speed, load and interactions between speed-time, speed-load and speed-particle sizes have significant effect on the wear behaviour of the composites. The model F-value of 175.23 implies the model is significant. There is only a 0.57% chance that a Model F-Value this large could occur due to noise. Value of Prob F less than 0.0500 indicate model terms are significant. In this case A, C, AB, AC, AD is the significant model terms. This model can be used to navigate the design space.

Substituting the coded values of the variables for any experimental condition in Eqn. (3), the wear rate of polyethylene and the LDPE-PC composites at different ceramic particle sizes can be calculated. The final linear regression equation for the wear rate of the LDPE-PC composites when tested against a pin on disc set up can be expressed as follows:

$$W = 0.01 + 0.01A + 0.018C + 0.049AB + 0.072AC + 0.051AD \quad (2.1)$$

The regression coefficients associated with the variable i.e., load and speed is positive which indicate that wear rate increases with increasing load and speed. Equation (2.1), was used to predict the wear rate of the developed composites. The regression coefficient calculated for this model was of 0.9461, i.e., reasonably close to unity, and thus acceptable. It was concluded that 94.61% of the data can be explained by this model. It shows that this model provides reasonably good explanation of the relationship between the independent factors and the response. The coefficient of sliding speed (A), applied load(C) in the regression equation is positive. These also support the earlier conclusion that sliding speed, applied load increased the wear rate, while decreased the particles sizes of the reinforcer decreases the wear rate of the materials.

Confirmation tests were conducted with parameter levels that were same from those used for the analysis. The different parameter levels chosen for the confirmation tests are shown in Table 6. The results of the confirmation test were obtained and a comparison was made between the actual values and the predicted values obtained from the

Table 5
Analysis of Variance Table to Identify Significant Factors Influencing Wear Rate of the Composites

Source	Sum of Squares	DF	Mean Square	F Value	Prob>F
Model	0.085	5	0.017	2175.23	0.0057
A	0.00151	1	0.00151	15.59	0.0586
C	0.00266	1	0.00266	27.47	0.0345
AB	0.019	1	0.019	194	0.0051
AC	0.041	1	0.041	424.58	0.0023
AD	0.021	1	0.021	214.52	0.0046
Residual	0.000194	2	0.000097		
Cor Total	0.085	7			

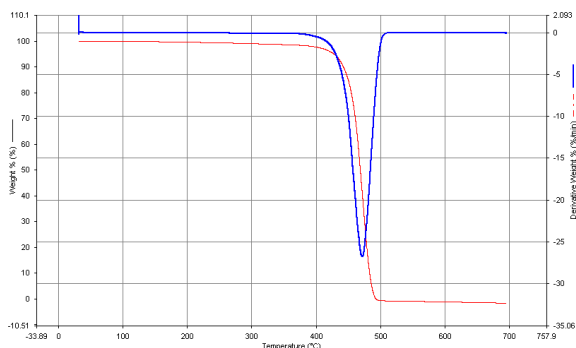
regression model (Table 6). The error associated with the relationship between the actual values and the predicted values from the regression model for developed composites was very low.

Table 6
Comparison of Actual and Predicted Model Values
Result

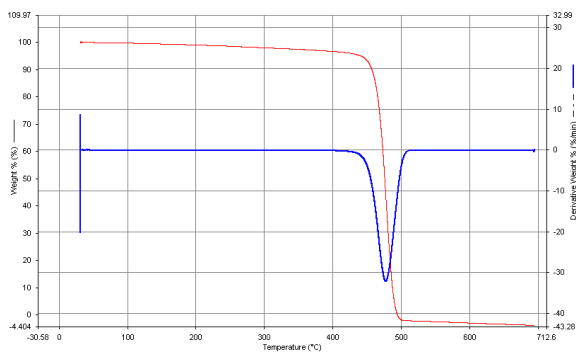
Order	Actual Value	Predicted Value	Residual
1	0.24	0.24	-0.0025
2	0.023	0.029	-0.0065
3	0.046	0.044	0.0025
4	0.031	0.025	0.0065
5	0.027	0.034	-0.0065
6	0.11	0.11	-0.0025
7	0.045	0.038	0.0065
8	0.31	0.31	0.0025

2.3 Thermogravimetric Analysis

TGA/DTA curves (Figures 3(a)-3(b)) of the thermal decomposition for of the polymer matrix and the developed composite with 40 μ m particle sizes exhibit only one dominant decline of the residual weight, indicating random scission of the polymer matrix main chains as the prevailing decomposition reaction(see Figures 3(a), 3(b)).



(a) DTA/TGA Analysis of the Polymer Matrix



(b) DTA/TGA Analysis of the Composite with 4 μ m Particle Sizes

Figure 3
DTA/TGA Analysis of Samples

The pronounced single endothermic effects observed in the DTA curves correspond to the oxidative degradation process and the release of volatile matters^[14-17]. From Figures 3(a)-3(b), the temperature of maximal decomposition and destruction of the matrix and composite fall in between 450 to 500°C. The presences of endothermic effects in the samples as a result of three processes: intermolecular dehydrogenation, vaporization and solid state decomposition of some additives^[14-17]. The total burning and degradation of the residual polymer backbone took place at temperatures interval of 400 – 500°C. In the last temperature interval the mass loss was minimal; this last step is due to the degradation of the ceramic particles material in the composite^[17]. The result indicated that the composite biomass show less percentage of decomposition. At temperature above 710°C, the residual weight stabilized agrees with the silica and carbon content earlier reported in literature^[16].

From Figures 3(a)-3(b), it was observed the addition of ceramic particles into polymer matrix increased the thermal decomposition temperatures and the residual yields of the composite. This result indicated that the presence of ceramic particles lead to the stabilization of polymer matrix resulted in the enhancement of the thermal stability of composite. In the composite, the ceramic particles act as physical barriers to prevent the transport of volatile decomposed products out of composite during thermal decomposition^[14,15]. The phenomenon is attributed to the physical barrier effect of the incorporated particles which act as the mass and heat transfer barriers^[16].

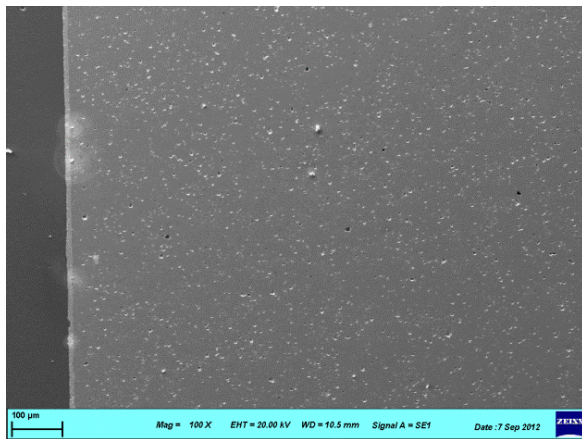
2.4 Microstructure Result

The SEM micrograph for the control sample (PE matrix) without the addition of any filler is presented in Figure 4(a). This micrograph shows uniform composition of the polyethylene matrix.

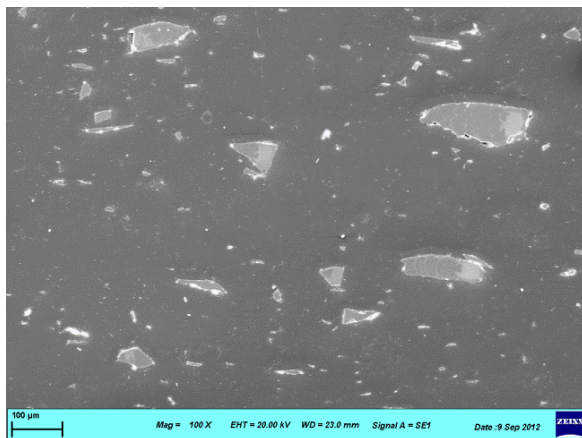
Figure 4(b), shows the distribution of ceramic granules at 150 μ m for the LDPE-PC composite. It is observed from the micrograph that the distribution of the second phase ceramic particles is heterogeneously. Furthermore, the shape of the second phase particle is irregular and non-spherical in nature. The heterogeneous nature of the second phase particles is presumed to be responsible for the brittleness of the LDPE-PC composite as observed from the stress-strain curve in Figure 1(b).

The brittle nature of the composite can also be attributed to poor transfer of stress between the LDPE matrix and the particulate ceramic filler despite the higher loading carrying capacity of the filler. On the other hand, Figure 4(c) shows the SEM micrograph and the distribution of ceramic granules at 40 μ m LDPE-PC composite. The second phase particle is a mix of fewer non-spherical and more fairly spherical in shape. The associated stress-strain curve in Figure 4(c) showed that improved mechanical properties of the LDPE-PC composite when compared to Figure 4(b) and Figure 1(b) composite of the same composition.

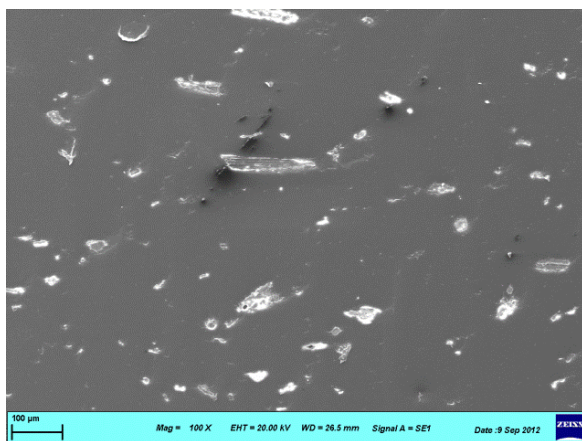
It appears the shape and the distribution of the second phase particles has a direct influence of the toughness of



(a) Without Ceramic Oxide Particle



(b) Composite at 150µm Ceramic Oxide Particle



(c) Composite at 40µm Ceramic Oxide Particle

Figure 4
SEM Micrograph of LDPE-PC Samples

the LDPE-PC composites. As the grain size of the filler become finer, the shape and the distribution of the second phase particle changes from non-spherical, non-coherent to a more compact fairly mixed fewer non-spherical and more-spherical coherent shapes. This may have been responsible for the better transfer of stress and interfacial bonding between the PE-matrix and the ceramic second

phase particles. As the grain size becomes finer, the strain at which necking start for LDPE-PC composites increased thereby resulting tougher composites.

CONCLUSION

From the data and various observations, it can be concluded that the silica based ceramic can be used as fillers for the LDPE-PC composite. The grain size of the ceramic affects the mechanical properties of the composite i.e. the smaller the grain size, the tougher the material becomes and also, the higher the impact values.

The wear property of the composite is significantly improved. The process parameters such as load has the most significant effect on the wear behaviour of the LDPE-PC composite so also the interaction effect of the process parameters such as speed-time, speed-load and speed-reinforcer have a characteristic effect on the wear behaviour of the polymeric composite (LDPE-PC composite).

The incorporated silica based ceramic particles play a crucial role in improving the thermal stability by acting as effective physical barriers against the thermal decomposition in the polymer composites. The fairly uniform distribution of the silica ceramic particles in the microstructure of composites is the major factor responsible for the improvement in the thermal and wears properties.

Factorial design of the experiment can be successfully employed to describe the wear behavior of the samples and develop linear equations for predicting the wear rate with in selected experimental conditions.

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