#### EFFECT OF PARTICLE SIZE ON THE PROPERTIES OF BLACK DATE SEED PARTICLE-REINFORCED HIGH DENSITY POLYETHYLENE COMPOSITE

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#### ABSTRACT

The effect of two particle sizes  $90\mu$ m and  $180\mu$ m on the physical and mechanical properties of HDPE reinforced with black date seed particles was investigated. Samples were cut for the physical properties (Density and Water absorption) and mechanical properties (Tensile Strength, Hardness and Impact). From the results of the physical properties, it was observed that the density for all the particle sizes decreased from  $0.930g/cm^3$  to  $0.686(90\mu)$  and  $0.699g/cm^3$  ( $180\mu$ m) as the percentage of the filler increased, the water absorption for all particle sizes increased as the percentage of the filler increased from 0.322% to  $0.583(90\mu)$  and  $0.656\%(180\mu)$ . Results from the mechanical properties tests show that the tensile strength for all the particle sizes decreased from 44MPa of 0wt% black date seed to 22MPa and 23MPa of 40wt% black date seed particulate for  $90\mu$ m and  $180\mu$ m respectively. The hardness values increased upon increase in particulate for both sieve sizes from 0 to 20wt% black date seed before decreasing at 30 and 40wt%. The impact energy values of the composites increased as the percentage of the filler increased.

*Keywords:* High density polyethylene (HDPE); Black date seed (BDS), particle size, polymer composite

#### **1.0 INTRODUCTION**

The composite industry has begun to recognize that the commercial application of composites promises to offer much larger business opportunities to the aerospace sector due to the sheer size of transportation industry. Thus, the shift of composite application from aircraft to other commercial uses has become prominent in recent years. With the introduction of polymer resin matrix material and high performance reinforcement fibers such as glass, carbon and aramid, the penetration of this advanced material has witnessed a steady expansion in uses and volume (Fuad et al, 1998).

High performance can now be found in such diverse applications as composite armoring designed to resist explosive impact, fuel cylinder for natural gas vehicle, windmill blades, industrial drive shaft and support beams of highway bridges and even paper making rollers. For certain applications, the use of composites rather than metals has resulted in saving of both cost and weight. Some examples are cascade for engines, curried fairing and fillets, replacement for welded metallic parts, cylinders, tubes, and ducts, blade containment band. The need for composites for lighter construction material and more seismic resistant structure has placed high emphasis on the use of what not only decreases dead weight but also absorbs the shock and vibration through tailored microstructures (Ahmed et al, 1999).

Polymer composites as engineering materials have shown steady development since 1942. In view of their versatility, they are used in aerospace application (where the reduction of weight was the principal irrespective objective of the cost). automobile, electrical, constructions and equipment sport industries. Polymer composites involving natural fibers or particles have additional advantages such as environmental friendliness, abundance, low cost, biodegradability and renewability (Roe and Ansell, 1995).

Polymeric composite materials can offer a very high strength and require excellent properties like high strength to weight ratio. Besides that, they can last longer in the long run and can be manufactured easily as compared to other materials such as metals. Many researchers have developed composites with fiber reinforcement and between 1955 and 1970, polymer matrix composites, such as fiber glass, carbon fiber and aramid fiber were used for applications in a variety of structures like aircraft, space craft, submarines and automobiles (Roe and Ansell, 1995).

# 2.0 EXPERIMENTAL PROCEDURES2.1 Materials

The materials used in this research are: Black date seed particles [Atili] ( $90\mu$ m and  $180\mu$ m) sieves obtained from Terminus market now Abuja market, Jos. Petroleum jelly, foil paper, and High density polyethylene which was obtained from Nigerian Institute of Leather and Science Technology (NILEST), Samaru, Zaria.

#### 2.2 Preparation of composite and sample

The black date seeds (BDS) were washed with water and detergent to remove sand particles and other dirt from its surface and was handpicked to separate it from stones. This process was carried out until the black date seed were free from stones and sand particles before sun drying. The black date seeds were ground in a ball mill at Mineral Resources Engineering Department, Kaduna Polytechnic, Kaduna, after which the black date seeds were sieved into 2 different sieve sizes of 90µm and 180µm.

Sample preparation was done in two stages, namely composite preparation and preparation of sample for tests. To produce the composite blocks, the ratios of the HDPE to the black date particles were measured out in the metallurgical laboratory at Ahmadu Bello University, Zaria. The ratios HDPE (resin) and black date (filler) particles measured and were 90/10, 80/20, 70/30, and 60/40. The measurement or weighing was carried out using electronic digital weighing balance (ADAM with precision of 0.001g, maximum capacity of 180g, power of 184KW and model of AE43134). The two roll mill machine was warmed for about an hour at a temperature of 150°C. After this, the HDPE was compounded first and thereafter the black date seed particles were mixed with the HDPE. Foil paper was attached to the mould and petroleum jelly was rubbed round the

foil paper. The hot compounded sample was collected from the two roll mill machine and then immediately placed in the mould. The composite was covered using a flat sheet of metal and then placed on a hydraulic hot press machine for pressing. The material was pressed at a temperature of 100°C for 8minutes. After eight minutes, the material was removed and then allowed to cool, and then the composite was removed from the mold. The procedure was repeated for all the compositions and sieve sizes of particulates.

## 2.3 Density Test

The densities of the specimens were obtained using the relationship

$$\rho = \frac{m}{n} \tag{1}$$

Where  $\rho$  = density (g/cm<sup>3</sup>); m = mass of the samples in grams; V = volume of the samples in cm<sup>3</sup>.

# 2.4 Water Absorption Test

Certain weights of the composites were cut off and weighed (say  $W_1$ ) before immersing in water for 24hours. The samples were then removed from the water and weighed again (say  $W_2$ ). The weights of moisture absorbed by the composites were calculated by the formula in equation (ii):

% Moisture Absorbed =  $\frac{W_2 - W_1}{W_1} \times 100$  (2)

Where  $W_1$  = initial weight of the material before soaking.

 $W_2$  = final weight of the material after soaking.

## 2.5 Determination of Tensile Strength

The universal testing machine (Instron LRX) with capacity of 2.5KN was employed for the tensile test according to ASTM D3379-75. The cross head speed was 50mm/min at gauge length of 100mm.

The composites were cut into a rectangular shape of dimensions  $20 \times 2 \times 100$ mm. The edges of the samples were placed in between the grips of the machine before applying the load increasingly until the sample fractured. The tensile loads together with the corresponding extensions were recorded. The test was carried out at National Research

Institute for Chemical Technology (NARICT).

#### 2.6 Hardness Values Determination

The hardness values of the samples were Rockwell obtained using the Indenter universal hardness testing machine. The test type is Rockwell hardness with scale F (HRF). the indenter was a 1/16 inch steel ball, the minor and major loads were 10 and 60 Kgf respectively. The samples were placed on the anvils and the minor load was applied to the samples and the zero datum position was established and then the major load was applied. This loading process was repeated on three different positions on each sample and the average value was obtained from the digital display on the machine as the hardness value of each sample.

#### 2.7 Impact Energy Determination

The samples used for the impact energy determination were cut dimensions to 10×10×100mm. The pendulum was raised to the test height and held there. The sample was mounted in the machine and the door of the machine was closed. The pointer for reading the impact energy value on the calibrated scale was adjusted to zero before the pendulum was released by means of a handle on the door of the machine. The pendulum falls from the height, breaking the sample and hitting the pointer to the test energy value. This process was repeated till all the average values were recorded for 2 samples each of the different compositions and black date seed particulate sieve sizes.

#### 2.8 Microstructural Examination

The micrographs of the composites produced were obtained by means of a

metallurgical microscope connected to a computer system.

# 3.0 RESULTS AND DISCUSSION

#### 3.1 Density

The results for the density are shown in Figure 1, for the various sieve sizes and compositions of the composites produced.

For a given dimension which is the same as the dimension of the control sample, it is observable from Figure 1 that,

1. As the composition of reinforcement increases for both particles size  $(90\mu m \text{ and } 180\mu m)$ , the density of the composite decreases due to the density of the reinforcement which is much lower than the density of the matrix.

2. As the composition of reinforcement increases for both particle sizes (90 $\mu$ m and 180 $\mu$ m), the number of interfacial spaces or pores increases which results in decrease in density.

From the result, it was observed that 180µm particle size had slightly higher density values than 90µm particle size due to difference in grain size. The density values for all the samples fall between 0.930g/cm<sup>3</sup> and 0.686g/cm<sup>3</sup>. The decrease in density of the composite is a great achievement, since low density is one of the advantages of composites. This observation is in line with earlier research by Erhuvwu (2012) in his research work, in which he noticed that as the Carbonized maize stalk particulate (CMSp) additions increased the density of the developed composites decreased.



Fig. 1: Variation of density with %composition of Black Date Seed particulate for 90 and 180µm sieve sizes.



Fig. 2: Variation of water absorption with %composition of Black Date Seed particulate for 90 and 180µm sieve sizes.



Fig. 3: Variation of Tensile strength with %composition of Black Date Seed particulate for 90 and 180µm sieve sizes.

#### **3.2 Water Absorption**

The results obtained from the water absorption test are plotted against percentage composition as shown in Figure 2. The results show that the water absorption for all the particle sizes increased progressively as the percentage of the filler increased. The values of water absorption obtained for the 180µm size were more than those for 90µm. This can be explained by the increasing composition of the reinforcement resulting in increasing number of interfacial spaces or pores giving room for water absorption more as composition increased. The water absorption value for all the samples ranges from 0.322% to 0.656%. In general it can be said that as the percentage of filler is increased the water absorption also increases. This is in line with the research carried out by Espert et al, (2004), in which they observed that as the filler content increases given the same average particle size, the interfacial area increases. The interfacial area can be a pathway for movement of water molecules. This is also as a result of the hydrophilic nature of the black date seed.

#### **3.3 Tensile Properties**

For 90 $\mu$ m sieve size of black date seed, the tensile strength demonstrated with the plot in Figure 3 was found to decrease from 44MN/m<sup>2</sup> (0wt% black date seed) to 25MN/m<sup>2</sup> (10wt% black date seed) before increasing to 26MN/m<sup>2</sup> (20wt% black date seed) and then down to 22MN/m<sup>2</sup> (40wt% black date seed). For the 180 $\mu$ m sieve size the value decreased from 44MN/m<sup>2</sup> (zero wt% black date seed) to 26MN/m<sup>2</sup> (10wt% black date seed) before dropping to 1MN/m<sup>2</sup> (30wt% black date seed). The reason the

drop to 1MN/m<sup>2</sup> (30wt% black date seed) is not known; it is possible that an area of weakness was gripped during the tensile test. It then subsequently increased to  $23MN/m^2$ (40wt% black date seed). This is in agreement with Husseinvah and Mostapha (2011), in the research work that the tensile strength of the composite decreased with addition of 15% filler content and then started to increase with increasing filler content. It can be seen in Figure 3 that the smallest value of tensile strength was obtained at 180µm sieve size (30wt% black date seed) which is in agreement with what was discussed above, about the increase in porosity due to increased retention of gas in the composite.

#### **3.4 Hardness Values**

The hardness values for both sieve sizes of 90 and 180µm increased progressively from 0wt% black date seed to 23.5 and 29.2 HRF respectively at 20wt% black date seed. The hardness values then dropped after 20wt% addition of filler before picking up at 40wt% even though the values were less than at 20wt% black date seed. However, this is in agreement with what was obtained by Ishidi et al (2011) in their research work, in which they noticed that hardness values increased upon increase in PKS and decreased at 60wt%PKS.



Fig. 4: Variation of hardness with %composition of Black Date Seed particulate for 90 and 180µm sieve sizes.



Fig. 5: Variation of impact energy with %composition Black Date Seed particulate for 90 and 180µm sieve sizes.

### **3.5 Impact Values**

All the composites made possessed impact values greater than the impact strength of the unreinforced High density polyethylene which has impact strength of 0.5J (joules).

From the results obtained, it was observed that the impact values increased as the wt% of reinforcement increases for both particle sizes. At 10wt% of reinforcement, the impact value was 0.6 for both particles sizes of 90µm and 180µm. The impact values increased respectively as the wt% of reinforcement increased to 2.4 and 6.13 of particle size of 90µm and 180µm at 60/40wt% reinforcement. Impact energy values increased with increase in percentage black date seed which is in agreement with what was obtained by Ishidi et al (2011), in which they reinforced HDPE with 30,40 and 60wt% PKS and then discovered that the unnotched tensile-impact strength of composites increased as filler loading increased.

# 3.6 MICROSTRUCTURES OF THE DEVELOPED COMPOSITE

The micrographs of the composites produced are shown in Plates A1-A9. The white sections represent the suspected spherulite structure of HDPE and the dark section which is uniform represents the spacing of linear boundary.

The thicker dark portion represents the flaws due to over or under compacting during the processing and A1 shows the microstructure of the control sample.

Micrographs A2 to A5 show microstructures of HDPE/BDS composites with BDS sieve size of 90µm. Micrograph A2 shows the structure of HDPE, with the white section representing HDPE structure. The reinforcement particle is dispersed averagely in the structure. A3 shows a structure of HDPE with black date seed (dark region) present in small amount. A4 shows a denser HDPE with more of black date seed. A5 shows cluster of black date seed (dark region) in small amount within the HDPE matrix.

Micrographs A6 to A9 show microstructures of HDPE/Black date seed composites with black date seed sieve size of 180µm. Micrograph A6 shows black date seed almost uniformly distributed in small amount within the HDPE matrix. Micrograph A7 shows a partially arranged structure of HDPE. Also, micrograph A8 shows less arranged structure of HDPE with increase in black date seed (dark region). Micrograph A9 shows distinct phases of both the matrix and reinforcement and also an arranged structure of HDPE.



Plate. A1: Microstructure of unreinforced HDPE (x100)



Plate. A2: Microstructure of 10wt% BDS (90µm) reinforced HDPE (x100).



Plate. A3: Microstructure of 20wt% BDS (90µm) reinforced HDPE (x100).



Plate. A4: Microstructure of 30wt% BDS (90µm) reinforced HDPE (x100).



Plate. A5: Microstructure of 40wt% BDS (90µm) reinforced HDPE (x100).



Plate. A6: Microstructure of 10wt% BDS (180µm) reinforced HDPE (x100).



Plate. A7: Microstructure of 20wt% BDS (180µm) reinforced HDPE (x100).



Plate. A8: Microstructure of 30wt% BDS (180µm) reinforced HDPE (x100).



Plate. A9: Microstructure of 40wt% BDS (180µm) reinforced HDPE (x100).

#### 4. CONCLUSIONS

The effect of Black date seed particle size on the mechanical properties, physical microstructure properties and of HDPE/Black date seed particulate composite was investigated in this research by the use of Universal testing machine, Indenter hardness testing machine and Charpy impact testing machine to determine the mechanical properties of the composites, and metallurgical microscope to determine the microstructures of the composites which were produced in the order of 10, 20, 30, 40wt% Black date seed of 2 particle sizes: 90µm and 180µm.

The following conclusions are made from the results of the investigations.

- (i) For all sieve sizes, the density decreased with increase in weight percent Black date seed.
- (ii) The values of water absorption increased with increase in wt% of BDS for both sieves sizes of 90 and 180μm
- (iii) The tensile strength values decreased with increase in wt% black date seed for both 90 and 180µm sieve sizes until it got to 30wt% black date seed before it started to increase.

- (iv) The hardness values increase upon increase in particulate for both sieve sizes from 0 to 20wt% black date seed before decreasing at 30 and 40wt%. When producing a composite of high hardness, the reinforcement should be maintained at 20wt% black date seed
- (v) The impact energy values also increased as the percentage of the filler increased.
- (vi) 180µm sieve size black date seed had higher values for all the tests carried out, and the composite produced has low density which means it can be used in automobile industries.

#### REFERENCES

Ahmed, E.M., Sahari,B. and Pederson P. (1999): Non-linear behaviour of unidirectional filament wound COTFRP, CFRP, and GFRP composites. In: Proceedings of World Engineering Congress WEC 99, Mechanical and Manufacturing Engineering, Kuala Lumpur: 1999. pp 537-543.

Erhuvwu Joseph Oghenevweta (2012): The potentials of Carbonized maize stalk particulates as reinforcement in polyester matrix composites, an MSc thesis, Department Metallurgical and Materials Engineering, Ahmadu Bello University, Zaria, unpublished. Espert , A., Vilaplana. And F. karlsson, S. (2004). Comparison of water absorption in natural cellulosic fibers from wood and one-year crops in polypropylene composites and its influence on their mechanical properties. *Composite Part:A Applied Science and Manufacturing*. Vol. 35 No. 11 pp 1267-1276.

Fuad MYA, Rahmad S, Azlan MRN. (1998) Filler content determination of bio-based thermoplastics composite by thermo gravimetric analysis In: Proceedings of the Fourth International conference on Advances in materials and Processing Technology 98, Kaula Lumpur; 1998, pp 268-275

Husseinsyah, S. and Mostapha, M. (2011): The effect of Filler Content on Properties of Coconut Shell Filled Polyester Composites, *Malaysian Polymer Journal*, Vol. 6, No. 1 pp 87-97.

Ishidi, E. Y., Adamu, I. K., Kolawale, E. G., Sunmonu, K. O. and Yakubu, M. K. (2011): Morphology and Thermal Properties of Alkaline Treated Palm Kernel Nut Shell – HDPE Composites, *Journal of Emerging Trends in Engineering and Applied Sciences*, Vol. 2 No. 2, pp 346-350.

Roe, P. and Ansell. M. (1995). Jute Reinforced Polyester Composite. J. Mater, vol. 20, pp 4015-4020