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Assessment of carbonized cherry/mango seed endocarp on the mechanical properties of low density polyethylene

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Abstract
Assessment of carbonized hybrid cherry/mango seed endocarp on the mechanical properties of low density polyethylene (LDPE) was investigated. Samples of cherry/mango seed endocarp were carbonized at 700^0C for 3hours, milled to fine powder, characterized and were used in compounding LDPE. The characterization results of carbonized cherry endocarp (CCSE) and carbonized mango seed endocarp (CMSE) present excellent values of pH (CCSE; 8.75 and CMSE; 8.09), moisture content (CCSE; 0.37% and CMSE; 0.83%), bulk density (CCSE; 0.52g/ml and CMSE; 0.58g/ml) and loss on ignition (CCSE; 69.50% and CMSE; 73.25%). The mechanical properties of the filled LDPE composites present excellent tensile strength (CCSE; 21.46–31.8MPa, CMSE; 19.05–30.50MPa and CCMSE; 20.63–31.09MPa), tensile modulus (CCSE; 153.39–228.00MPa, CMSE; 149.56–224.06MPa and CCMSE; 151.48–227.17MPa), elongation at break (CCSE; 320.79–273.89%, CMSE; 318.18–267.16% and CCMSE; 318.89–271.10%), hardness (CCSE; 51.88–77.95Shore A, CMSE; 50.75–69.49Shore A and CCMSE; 51.07–69.89Shore A), abrasion resistance (CCSE; 57.00–39.68, CMSE; 53.42–33.91 and CCMSE; 52.04–34.01) and impact strength (CCSE; 169.03–137.80 J/mm, CMSE; 167.08–131.60J/mm and CCMSE; 165.34–133.81J/mm). These excellent mechanical results showed that hybridized carbonized filler has the potential improving and enhancing polymer matrices for better product quality, serviceability and performance.

Keywords: Hybrid, endocarp, samples, carbonized, matrices

Introduction
Research innovations in the field of material science led to many and advanced materials (Ismail *et al*., 2010) [5]. Composites are one of them which are adopted in various engineering applications. Distinctive size dependent properties of particles often exist which are mainly due to their large surface area. Due to these, a lot of physical properties of powdered particles are quite different from bulk materials yielding a wide variety of application such as carriers for delivering drug molecules (Manmode *et al*., 2009) [6]. Powdered particles show different mechanical property relative to micro-particles and bulk materials providing more effective options for surface modifications of many devices in mechanical strength to improve the quality of products. The extensive use of composites in these areas is basically due to their combined properties of resilience, high strength and stiffness to weight ratios, corrosion resistance and good damping properties. The most commonly used filler for rubber compounding are carbon black, calcium carbonate, china clay, silica etc. which are used as reinforcement or as cheapeners. Carbon black is mostly used for rubber products except where colouration is prioritized. Due to the expensive nature and non-renewability of carbon black, there arises the need to source for locally modified fillers (Yue *et al*., 2006) [12]. Most agricultural renowned wastes are melon husks, palm font and kernels, saw-dust, guinea corn husks, sugarcane baggase, castor seed shell, cherry seed endocarp, mango seed endocarp, groundnut shell etc. This desecrate materials are one of the problem facing producing countries which so far has no ultimate resolution (Xie *et al*., 2010) [11]. To reduce the quantity of these squander materials, they can be burned in the open air which creates noteworthy environmental effluence. As a result, the use of such materials has motivated the growth of research into the value-added potentialities of these waste products derived from agriculture and these waste materials are very low cost and cheap. Composites play significant role as engineering materials and their use has been increasing day by day due to their specific properties exhibited in their tear and wear resistance as well as modulus (Xie *et al*., 2010) [11].

Scientific research on agro-waste is intense as they have many potential applications in rubber industries.

Objectives of Study

i. To carbonized and characterize cherry/mango seed endocarp powdered fillers.
ii. To study the effects of carbonized cherry/mango seed endocarp powdered on the mechanical properties of low density polyethylene (LDPE) matrices.
iii. To compare the properties of the filled LDPE composites produced.

Materials and Methods

Materials

The various raw materials used in this research are presented in Table 1.

Table 1: Materials and their Sources

Materials	Sources
Mango Seed Endocarp	Auchi Metropolis and Environment
Cherry Seed Endocarp	Leventis Farms, Agenebode
Low Density Polyethylene	Vulnax International, England
Processing Aid	Vulnax International, England

Equipment and Machines Used

The equipment and models used include

1. Muffle Furnace METTm-525, Elektron Technology Series, UK.
2. Universal Tensile Tester, manufactured by British Company Limited, England was used for tensile tests properties.
3. Wallace Hardness Tester, Elektron Technology Series, UK was used for hardness test.
4. Hydraulic press, Elektron Technology Series, UK was used for moulding composite.
5. Mevler Zeta analyzer for particle size analysis was used in determining the filler sizes.
6. Wallace Akron Abrasion Tester, Elektron Technology Series, UK was used for abrasion test.
7. Two Roll Mill, manufactured by British Company Limited, England mills were used in mixing the rubber composite.
8. Retch new planetary mill series (Pm100) with planetary balls made of harden steel as small as 0.1mm (< 0.004inch) in diameter.

Methods

Preparation of Powdered Fillers

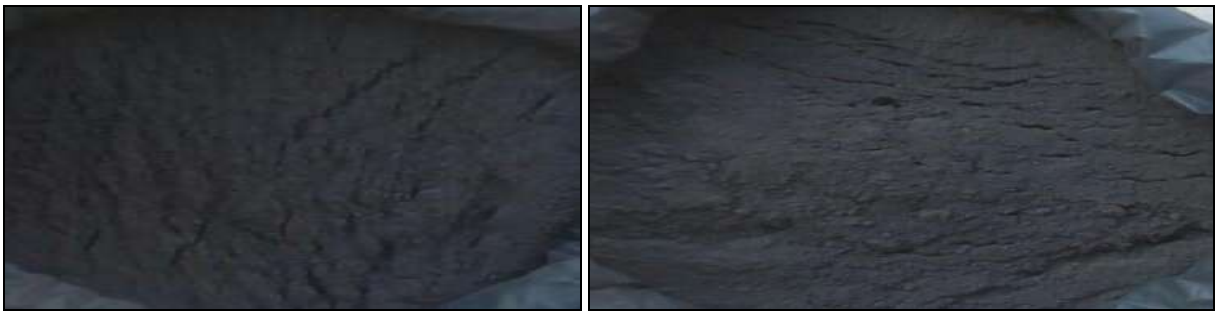
Cherry and mango seed endocarps were crushed and ground reduce the particle size and further subjected to ball milling using the PM100 series. The material and the media are agitated by a shaft with arms which rotates at high speed. This causes the media to exert both shearing and impact forces on the material resulting in optimum size reduction and dispersion. An internal cascading effect reduces the material to ultra-fine powder (Hussain *et al*., 2010) [4]. In general, the particle size decreases with increasing reaction time and temperature. Consequently, the particle size could be controlled by systematic adjustment of the reaction conditions including time, temperature and concentration of stabilizers.

(a) **(b)**

Fig 1: Cherry (a) and Mango (b) Seed Endocarp Powder

Filler Carbonization

Carbonization is a process of converting an organic compound to high carbon content by heating it in the absence of oxygen (inert). Carbonization of fillers used in this research work was carried out using a laboratory muffle furnace set at 700 °C by creating an inert environment via the passage of nitrogen gas for 3 hours each and milled to fine powder.



(c) **(d)**

Fig 2: Carbonized Cherry (c) and Mango (d) Seed Endocarp Powder

Characterization of Filler

Particle Size Analysis

The size of powder filler sample was determined using Malvern Zetasizer. The powder was dispersed in a cuticle using methanol as the suitable dispersant to obtain a medium capable of been focused by dynamic light scattering system and reflecting on a transducing medium which help to measure the aggregation of the particles. As the light passes through the sample, image is formed by reflection on the transducing medium which detects the resultant size of the material by volume of aggregates and the level of light intensity that passes through the medium. The peaks and percentage volume and intensity are measured for particle data.

Moisture Content

The method determines the percentage of water in a sample by drying the sample to a constant weight. The moist sample was weighed and recorded as initial weight of sample; the wet sample was dried to a constant weight at a temperature of 125ºC in an oven. The sample was allowed to cool, reweighed and recorded as the final weight of the sample.

$$Moisture\ \ Content\ (\%) = \frac{Initial\ Weight\ -\ Final\ \ Weight}{Initial\ \ Weight} \times 100$$

pH

The pH of the powdered samples were determined using ASTMD 1512 method by immersing 1.0g samples in 20.0ml of deionized water in a 250ml beaker. The mixture was stirred for 15minutes and the pH meter was then inserted into the solution to obtain reading directly.

Bulk Density

Bulk density of the various samples was determined by the tapping procedure. Accurately weighed samples were poured into a uniform cylinder of cross sectional area and were then tapped several times until there was no change in the volume occupied. This volume was then recorded and the bulk density calculated and recorded.

Loss on Ignition

Loss on ignition refers to the mass loss of a combustion residue whenever it is heated in an air or oxygen atmosphere to high temperatures. The loss on Ignition of the various samples was determined gravimetrically, in accordance to the procedure described in ASTMD 7348. 10g of the sample was place in a tarred, pre-ignited crucible and placed in a furnace for some time (about 2hours). It was removed from the oven, cool and weighed. It was then put in the oven and heated until the weight is constant.

Processing of CCSE/CMSE Hybridized LDPE Composites

The formulation and compounding of low density polyethylene (LDPE) with the carbonized mango and cherry seed endocarp is presented in Table 2.

Table 2: Formulation for Compounding.

Samples	LDPE	CMSE	CCSE	CCSE/CMSE
1	100	-	-	-
2	90	10	10	5/5
3	80	20	20	10/10
4	70	30	30	15/15
5	60	40	40	20/20
6	50	50	50	25/25

Mixing Procedure

The polyethylene mix was prepared on a laboratory size two roll mill in accordance to the formulation presented in Table 2.

(e) (f) (g) (h) (i)

Fig 3: Mixing Stages (e, f, g and h) and Compounded Samples of Low-Density Polyethylene (LDPE)

Moulding

The molding of tests samples was done in a compression moulding machine at a temperature of 170 °C for 5 minutes in each case.

Mechanical Properties of Composite

Tensile Properties

Tensile properties were determined on a universal tensile tester at a cross speed of 060.00mm/min using dumbbell test pieces of dimension (100 × 15× 5mm). The test samples were tested in the machine giving straight tensile pull, without any bending or twisting. The machine measures both the tensile stress and the tensile strain. The tensile stress is the strength of pull in the area between the notch marks based on original cross-sectional area while the tensile strain is a measure of how the test sample has been stretched by the pull.

Abrasion Resistance

It is the loss of properties by surface scratching and wearing. The Wallace Akron abrasion tester was used. The angle between the test sample and the wheel was adjusted to 15º. The abrasion was carried out per 100 revolutions and the material loss for each run was noted. The specimen was re-

reweighed between each test run and the mean revolutions of the abrasive wheel calculated.

$$Abrasion\ \text{Re}sis\tan ce = \frac{Weight\ \ Loss\ \ of\ \ the\ S\tan dard}{Weight\ \ Loss\ of\ \ the\ Sample} \times 100$$

Hardness

The hardness of the samples was determined by adopting the standard dead load method. The test was carried out using the Wallace Hardness Tester in accordance with shore A.

Impact Test

The impact test was carried out by supporting the sample at both ends placed horizontally with the impact applied midway between the two supports. A pendulum of a known mass was allowed to fall through a known height and strike the specimen as it continues swinging. The difference between the heights was proportional to the energy absorbed. The difference between the initial height and the height reached after impact on the test sample was recorded as the impact strength of the sample.

Results and Discussion

Results

The results of this research work are presented in Tables 3 - 5 and Figures 4 - 10 respectively.

Table 3: Particle Size Distribution by Volume and Intensity Analysis

Parameter	Volume		Intensity	
	Peak 1	Peak 2	Peak 1	Peak 2
Percentage (%)	75.80	24.20	66.00	34.00
Size (d.nm)	389.10	1086	1731	4444
Z-Average (d.nm)	2257		2257	
PDI	0.406		0.406	

Table 4: Characteristics of the Powdered Fillers

Moisture Content (%)	pH of Slurry	Bulk Density (g/ml)	Loss on Ignition (%)
(0.37), [0.83]	(8.75), [8.09]	(0.52), [0.58]	(69.50), [73.25]

Table 5: Mechanical Properties of LDPE filled Composites

Property	Filler Loading (g)					
	-	10	20	30	40	50
Tensile Strength (MPa)	17.05	(21.46) [19.05] {20.63}	(23.61) [22.93] {22.99}	(27.65) [27.02] {27.48}	(31.49) [29.80] {29.89}	(31.87) [30.50] {31.09}
Modulus (MPa)	138.04	(153.39) [149.56] {151.48}	(178.95) [172.03] {175.59}	(201.69) [201.05] {201.58}	(227.39) [223.17] {222.03}	(228.00) [224.06] {227.17}
Elongation @ Break (%)	353.05	(320.79) [318.18] {318.89}	(305.58) [301.69] {304.12}	(297.57) [296.71] {296.98}	(294.74) [269.00] {265.73}	(273.89) [267.16] {271.10}
Hardness (Shore A)	47.64	(51.88) [50.75] {51.07}	(57.03) [54.38] {56.23}	(68.09) [63.92] {65.12}	(72.03) [67.37] {66.09}	(77.95) [69.49] {69.89}
Abrasion Resistance (mm^3/100rev)	62.10	(57.00) [53.42] {52.04}	(52.65) [49.04] {50.42}	(48.94) [42.08] {43.06}	(43.81) [37.68] {35.93}	(39.68) [33.91] {34.01}
Impact Strength (J/mm)	183.01	(169.03) [167.08] {165.34}	(164.37) [156.02] {159.60}	(145.73) [141.34] {142.65}	(139.53) [138.35] {137.62}	(137.80) [131.60] {133.81}

Key: Carbonized Cherry Seed Endocarp (CCSE), Carbonized Mango Seed Endocarp [CMSE] Carbonized Cherry/Mango Seed Endocarp {CCMSE}

Size Distribution Report by Volume

v2.2

Malvern

Sample Details

Sample Name: CSS
SOP Name: Dispersant.sop
General Notes: Average result created from record number(s): 1 2 3

File Name:	CSS.dts	**Dispersant Name:**	Methanol
Record Number:	4	**Dispersant RI:**	1.326
Material RI:	1.59	**Viscosity (cP):**	0.5476
Material Absorbtion:	0.010	**Measurement Date and Time:**	24 September 2017 08:4...

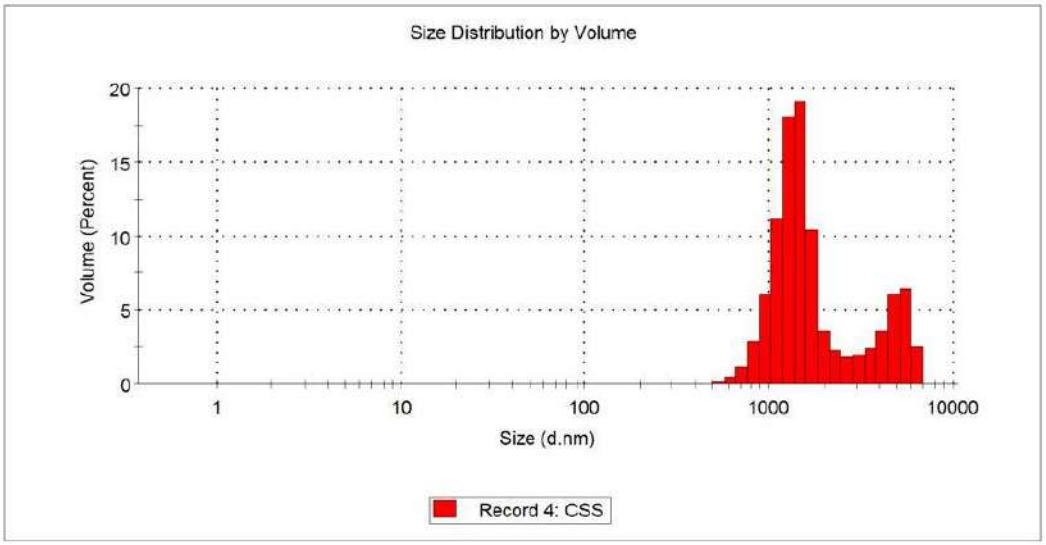
System

Temperature (°C):	25.0	**Duration Used (s):**	60
Count Rate (kcps):	387.3	**Measurement Position (mm):**	4.65
Cell Description:	Disposable sizing cuvette	**Attenuator:**	10

Results

			Size (d.n...	% Volume:	St Dev (d.n...
Z-Average (d.nm):	2257	**Peak 1:**	1404	75.8	389.1
PdI:	0.406	**Peak 2:**	4657	24.2	1086
Intercept:	0.400	**Peak 3:**	0.000	0.0	0.000

Result quality Refer to quality report



Zetasizer Ver. 7.01
Serial Number : N/A

File name: CSS
Record Number: 4
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Fig 4: Size Distribution by Volume

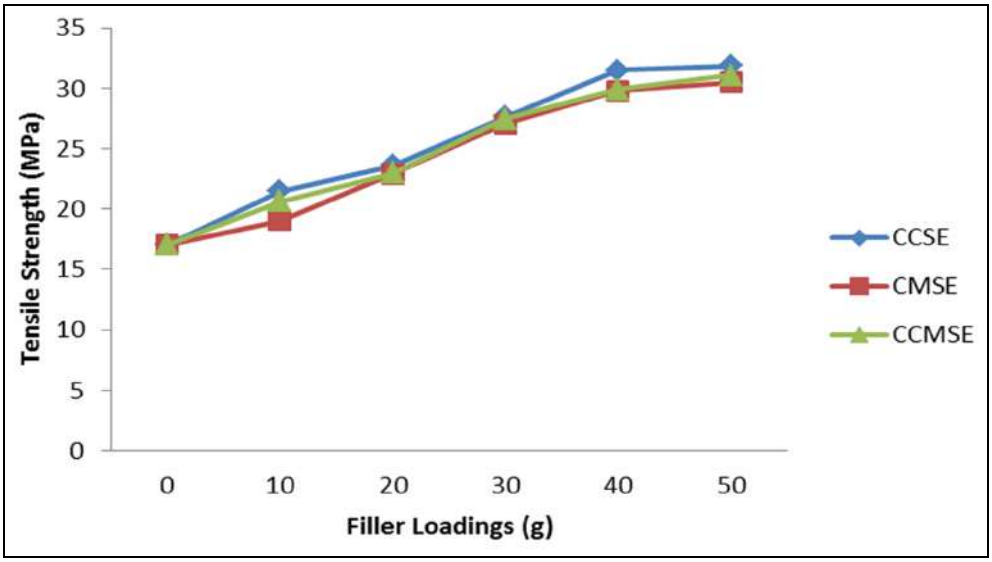

Fig 5: Tensile Strength of filled LDPE Composites

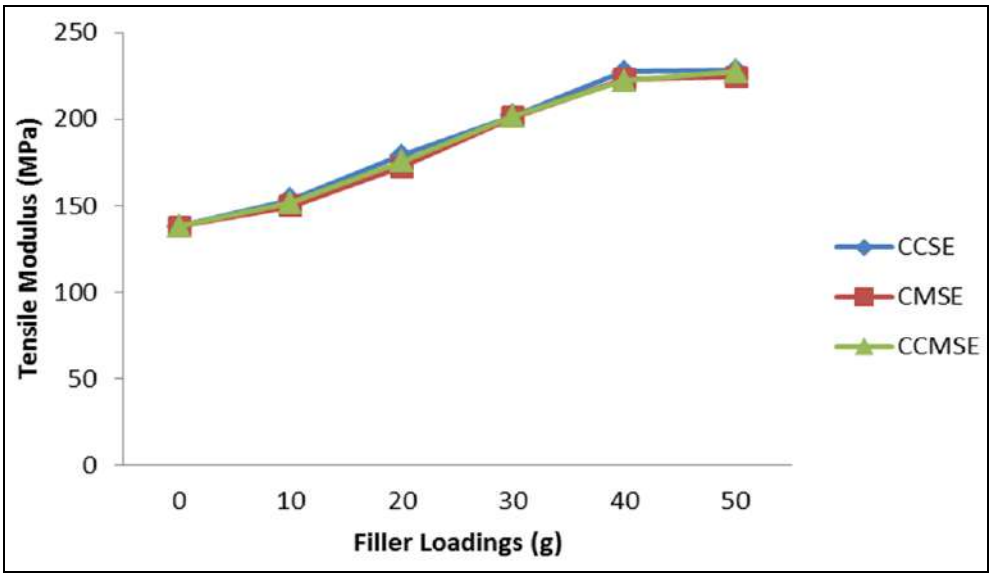

Fig 6: Tensile Modulus of filled LDPE Composites

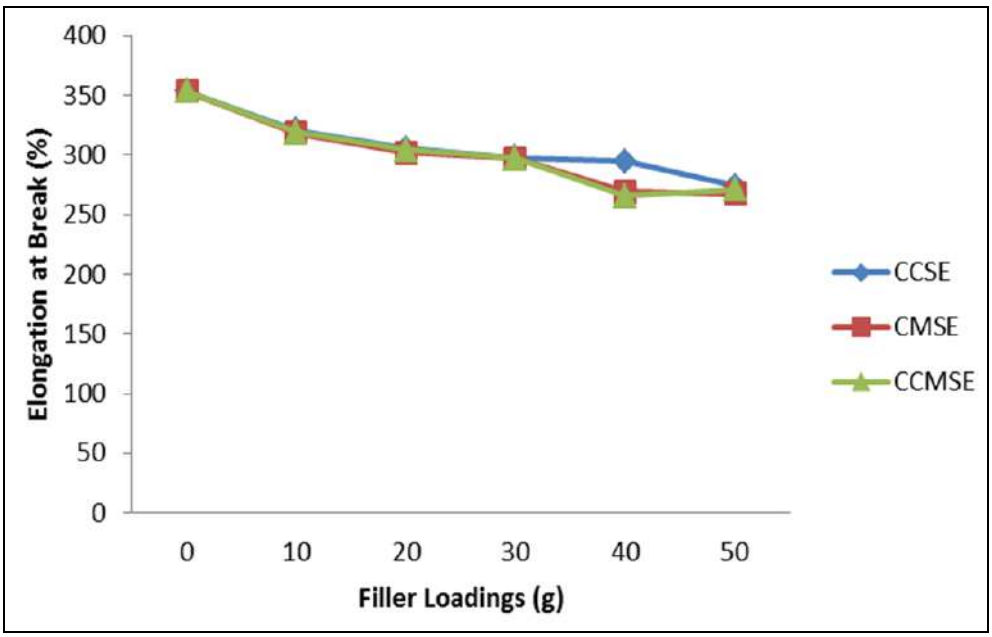

Fig 7: EaB of filled LDPE Composites

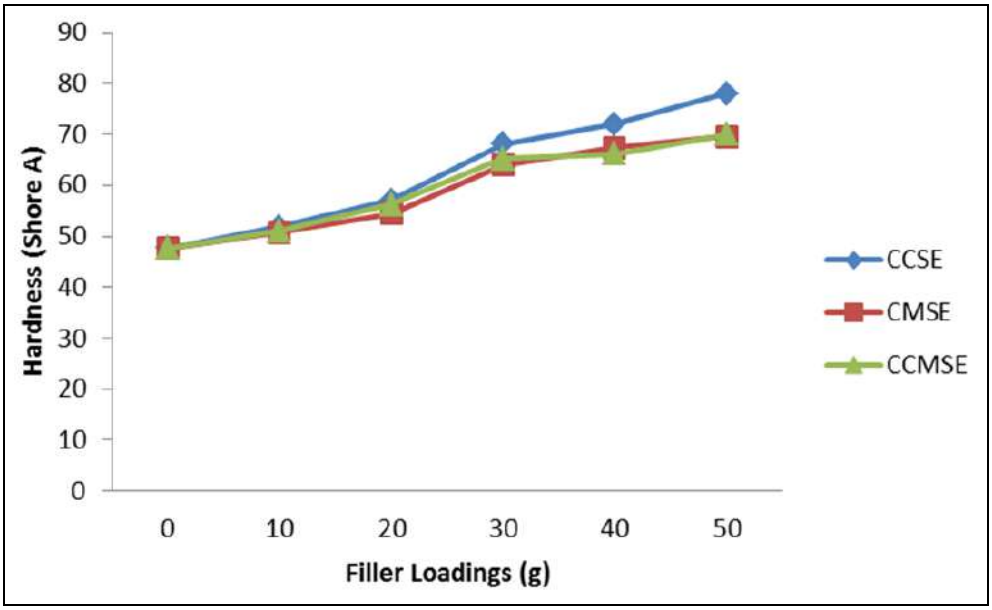

Fig 8: Hardness of filled LDPE Composites

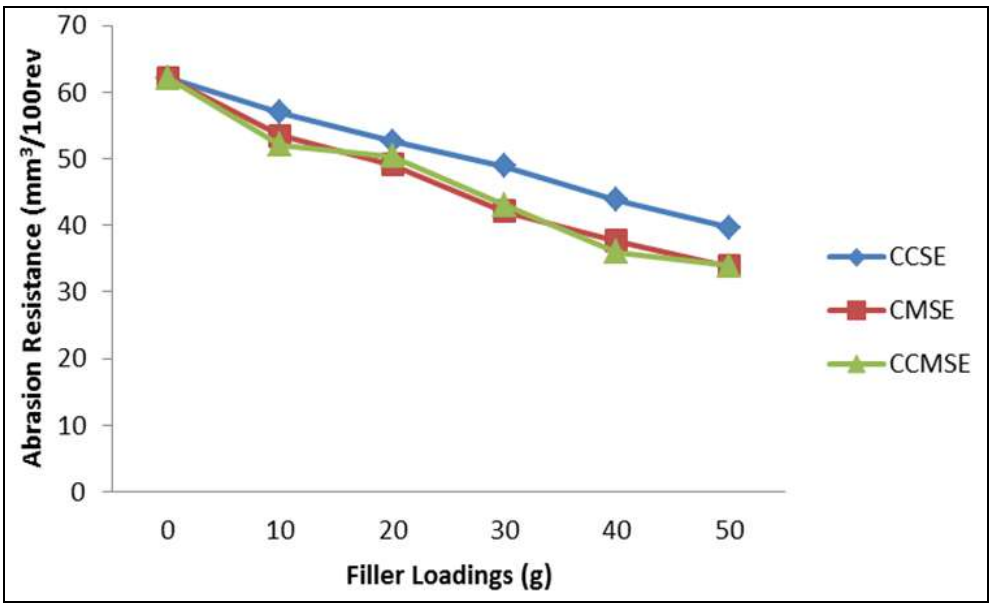

Fig 9: Abrasion Resistance of filled LDPE Composites

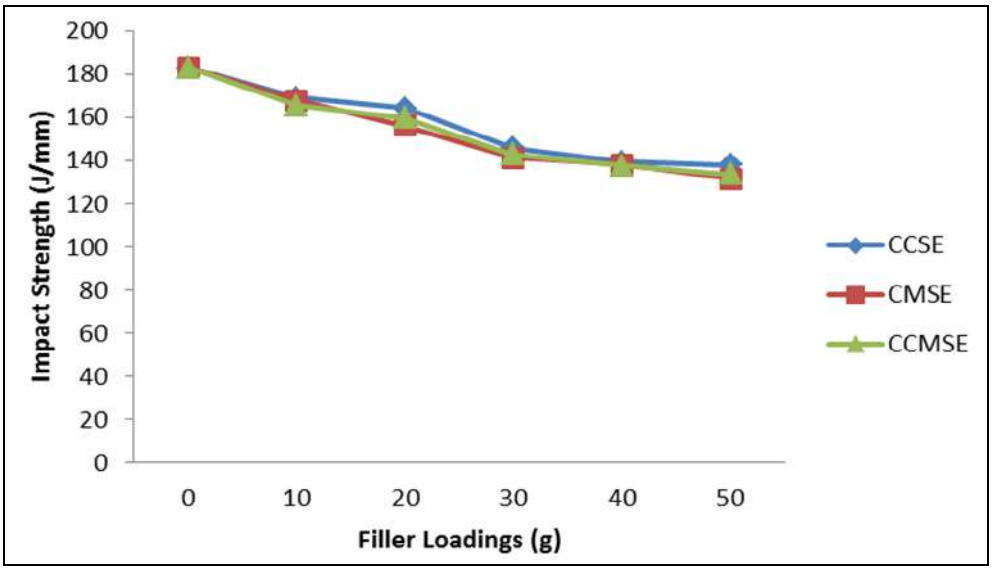

Fig 10: Impact Strength of filled LDPE Composites

Discussion

Results of Filler Characterization

Table 3 and Figures 4 showed the result of particle size of the carbonized filler studied in this research work. The powdered carbonized filler was adequately dispersed using methanol as the dispersing medium. The result showed that the particle sized distribution by volume of sample was 75.80% in the first peak and 24.20% in the second peak while the intensity of light reflectance on the sample was 66.00% in the first peak and 34.00% in the second peak which showed a very high level of dominance of particles within the nanometer range over the quasi or coarse particles in the aggregate. However, it revealed that an aggregation is often formed with powdered particles (Akbari *et al*., 2011) [1]. The result showed that the powdered particles were of nanometer sizes.

Table 4 showed the characteristic results of the powdered carbonized fillers. The relationship between moisture contents of the carbonized fillers is presented in Table 4. The moisture content for CCSE and CMSE were 0.37% and 0.83%. However, CMSE present higher value when compared with CCSE, thus showing a high tendency for CMSE to absorb moisture when exposed. The moisture content of the filler is often used to predict the degree of defects arising from shrinkage during curing and moulding of polymer materials particularly for products processed at elevated temperatures since water boils at 100^0C.

The loss on ignition for CCSE and CMSE presented in Table 4 were 69.50% and 73.25% respectively. However, the loss on ignition for CCSE is higher than that of CMSE suggesting a high amount of carbon present and hence better reinforcement and compactibility with the polymer matrix. The loss on ignition values at 700 °C may have been caused by almost complete evaporation of the volatile matter at high temperatures. At high temperature, the porosity reduces, thus, some cavities have been burnt and the corresponding surface area reduced (Tenebe *et al*., 2013) [9]. The surface area value is much smaller when carbonizing at low temperature (100 °C) due to the low porosity resulting from incomplete carbonization (Ayo *et al*., 2011) [2].

The bulk density of CCSE and CMSE samples showed in Table 4 were 0.52g/ml and 0.58g/ml respectively. Bulk density is principally influenced by the particle size and structure of the fibre and the lower the particle size, the lower the bulk density and therefore better the interaction between the polymer matrix and the reinforcing fibre which will thus enhance the processing and improve quality of the final product as desirable properties for fibre include excellent tensile strength and modulus, high durability, low bulk density, good mouldability and recyclability (Ayo *et al*., 2011) [2].

The pH of the carbonized powdered fillers presented in Table 4 was 8.75 for CCSE and 8.09 for CMSE. The results showed a progressive increase in pH from acidic to alkalinity with carbonization temperature. This is possible because residuals materials are being lost on combustion, leading to the alkalinity. However, pH at acidity level tends to slow cure rate and hence reduce the inter-links within the polymer matrix (Tenebe *et al*., 2016) [10].

Results of Mechanical Properties

The results in Table 5 and Figure 5 showed an increase in tensile strength with increased carbonized filler loadings which were due to large surface area of both CCSE and CMSE fillers suggesting better polymer filler interaction and hence enhanced better tensile properties. However, CCSE showed better and improved tensile Strength property when compared with CMSE and CCMSE filled LDPE composites. The surface area of CCSE filler was larger than CMSE which aid the adherence and compactibility of the CCSE filler in the polymer matrix. The factors that affect the reinforcing potentials of fillers include filler dispersions, surface area, surface reactivity, bonding capacity (quality), particle size between the filled matrix (Tenebe *et al*., 2013) [9].

Table 5 and Figure 6 showed that modulus of filled composites depend on the level of filler dispersion in the polymer matrix. The reduction in particle size accounts for the improved moduli since fine particle size increase modulus because it impedes filler interaction which consequently decreases the ability of the filler to restrain gross deformation of the composite matrix (Bakker *et al*., 1995) [3]. The modulus of CCSE, CMSE and CCMSE-filled composite increased with filler loadings. However, CCSE was higher than CMSE and CCMSE-filled LDPE systems which had been explained as a result to large surface of CCSE filler.

Table 5 and Figure 7 showed that elongation at break (EAB) decreases with increasing fillers loadings for CCSE, CMSE and hybridized CCMSE-filled LDPE composites due to a high interaction between the filler and polymer matrix leading to stiffening of the chains with highly stressed chains break first and their loads are then distributed to other chains forcing them to either break or slip so as to relieve the stress on them (Zhang *et al*., 2010) [13]. A decrease in EAB has been explained in terms of adherence of the filler to the polymer phase leading to the stiffening of the polymer chain and hence resistance to stretch when the strain is applied (Ayo *et al*., 2011) [2].

Table 5 and Figure 8 showed the hardness values for CCSE, CMSE and hybridized CCMSE-filled LDPE composites respectively. The hardness increases with increasing filler loadings for both Carbonized fillers. This result is expected because as more filler particles get into the matrix, the more stiffen are chain and thus increased hardness. Since hardness measures small deformation at the surface of the polymer matrix and an approximate index of stiffness, longer shelf service life may be expected of products (Ismail *et al*., 2010) [5]. However, the hardness of CCSE was higher than CMSE and hybridized CCMSE when compared.

The trend of abrasion resistance presented in Table 5 and Figure 9 showed a regular pattern of decreased with increased the filler loadings of CCSE, CMSE and hybridized CCMSE-filled LDPE composite samples. This indicates that filler loading is a function of the measured parameter attributed to the degree of dispersion of the fillers (Sung *et al*., 2004) [8]. The abrasion resistance of a solid body is defined as its ability to withstand the progressive removal of the material from its surface as a result of the mechanical action of rubbing, scraping or erosive nature. However, CCSE-filled composite has higher percentage resistance when compared with CMSE and hybridized CCMSE-filled LDPE composite.

The impact result presented in Table 5 and Figure 10 revealed that as the moulded LDPE filled with CCSE, CMSE and hybridized CCMSE material gets stiffened there is the tendency that they require more energy to break it or to cause failure within the composite material. Thus impact

is the resistance to indentation by sudden blow or fracture in a material. The result also predicts the higher the filler loading the lower the average degree of energy requires breaking the sample (Peng *et al*., 2007) [7]. Thus, CCSE-filled LDPE samples showed higher impact strength when compared with CMSE and hybridized CCMSE-filled LDPE composite.

Conclusion

The essence of this research is to examine how the carbonized cherry/mango seed endocarp hybrid fillers may affect its characteristics properties and hence the mechanical properties of LDPE composites. The use of carbonized hybrid cherry/mango seed endocarps (CCSE, CMSE and CCMSE) powder in LDPE composites has showed that carbonized filler influences the mechanical properties of filled composites. The result showed that carbonized CCSE and CMSE can serve as reinforcing filler for polymer composites. The results indicate that mechanical properties of composites are greatly influenced by filler loading. The results also showed that the use of carbonized fillers in polymer matrix reinforcements are great importance in ensuring that such polymer products meet specification and service requirements.

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