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Effects of carbonization on the physical and mechanical properties of coconut shell particle reinforced polyester composite

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Abstract

In this research work, the effect of carbonization on the flexural strength, hardness, water absorption and density of coconut shell/unsaturated polyester composite was studied. A portion of the coconut shell was carbonized at 700°C in a heat treatment furnace with a soaking time of three hours while the other portion was uncarbonized. They were ground and sieved into two particle sizes. Flexural strength, hardness, density and water absorption properties of the developed composites were determined. The 30wt% of the 1700microns uncarbonized coconut shell particle reinforced composite sample absorbed more water. Maximum flexural strength was obtained at 20wt. % for the 425microns sample reinforced composite while that of hardness was at 30wt.%. The density of the composite decreased with increase in filler content. The SEM showed the distribution of coconut shell particles within the composite. The carbonized coconut shell particle reinforced composite recorded the least density value, therefore, this composite can be used in applications requiring light weight material.

Keywords: polyester composite, carbonized coconut shell, flexural strength, hardness, density, water absorption, SEM.

1. Introduction

The interest in natural particle-reinforced polymer composite materials is rapidly growing both in terms of their industrial applications and fundamental research. They are renewable, cheap, completely or partially recyclable, and biodegradable (Singh, *et al*, 2013) [1]. Plants, such as flax, cotton, hemp, jute, sisal, kenaf, pineapple, ramie, bamboo, banana, etc., as well as wood, used from time immemorial as a source of lingo-cellulosic particles, are more and more often applied as the reinforcement of composites.

Most of these materials according to Adeosun *et al*, 2015 [3] are lignocellulosic and could be used as reinforcements in developing composite materials. Lignocellulosic fibres are hydrophilic in nature and absorb moisture when exposed to the atmosphere. There is a large amount of hydrogen bonds (hydroxyl groups -OH) present between the macromolecules in the plant fibre cell wall and when moisture from the atmosphere comes in contact with the fibre, the hydrogen bond breaks and hydroxyl groups form new hydrogen bonds with water molecules (Savita Dixit and Preeti Verma, 2012) [4]. Hydrophilic fibre reinforced with hydrophobic resin results in fibre swelling within the matrix when exposed to moist environment. This, according to Sarani and Poh, 2002 [6], causes weak bonding between fibre and matrix, dimensional instability, matrix cracking and poor mechanical properties of the composites. The moisture absorption of natural fibres can be reduced by different chemical treatment such as alkali, silane, acetylation, benzoylation and peroxide treatment on the fibre surface to remove hydrophilic hydroxyl bonds (Wang *et al*, 2007) [7]. Also, (Savita Dixit and Preeti Verma, 2012) [4] stated that removal of moisture from fibres by heat treatment at 150°Cis an essential step before the preparation of composites as it will help to check weak bonding between fibre and matrix, dimensional instability, matrix cracking and poor mechanical properties of the composites.

Natural fillers reinforced polymeric materials according to Durowaye *et al*, 2014 [5], provide materials engineers with a new group of materials that offer exceptional combinations of mechanical properties that make them equivalent to steel applications. Their availability, renewability, low density, and price as well as satisfactory mechanical properties make them an attractive ecological alternative to glass, carbon and man-made particles used for the manufacturing of composites. As was observed by (Prakash, 2009) [2], natural particle-containing composites are more environmentally friendly, and are used in transportation (automobiles, railway coaches, aerospace), military applications, building and construction industries (ceiling paneling, partition boards), packaging, consumer products, etc.

Coconut shells are natural particle-reinforcement materials and are available in abundance in tropical countries as a waste product after consumption of coconut water and meat. Such abundance can fulfill the demand of natural particle-reinforced polymer based composites while reducing waste and promoting healthier environment. As an agro waste, its residues during harvest seasons as is obtainable in most African countries are enormous and in most cases constitute environmental eye-sore.

Carbon black according to Ojha *et al*, 2016 [8], can be derived from various biomass waste products such as bamboo, jute, cotton, flax, which are carbonaceous in nature and received raised attention as alternative fillers because of their low cost and abundance. Such biomass, according to Khalil *et al*, 2007) [9] which are rich in lignocellulosic fibers can produce biocarbon (carbon black) after carbonization because they have high fixed carbon content.

Chanap, 2012, reported that coconut shell contains about 65 – 75% volatile matter and moisture which are removed largely during carbonization process which involves converting the coconut shells to char (charcoal) by heating in the absence or limited amount of air to a temperature sufficiently high to dry and volatilize substances in the coconut shell. During the carbonization of coconut shells, volatiles amounting to 70%. of the mass of coconut shells on dry weight basis are released to the atmosphere, yielding about 30% of coconut shell mass of charcoal. The volatile released during the carbonization process is methane, CO_2 and wide range of organic vapors. The carbonization temperature ranges between 400°C and 850°C sometimes reaches 1000°C. The shell of the coconut is one part of the waste after removing the husk, which is very hard lignocellulosic and can be converted into carbon black. Coconut shell has the potential to be used as a filler material in polymer composite due to its high carbon content (Ojha *et al*, 2016) [8].

This research work investigated the use of agro-based waste, coconut shell particles in carbonized and uncarbonized form as a reinforcement material in polyester matrix to develop a polymeric composite. The essence of the carbonization which was carried out at 700°C with a soaking time of three hours was to reduce drastically the volatile matter content of the coconut shell and enhance its structural strength for better mechanical properties. The volatile matter contains the OH group which prompts water absorption in coconut shell. This was responsible for the reduced water absorption of the carbonized coconut shell reinforced polyester composite.

2. Materials and Methods

2.1 Materials

Materials used in this research work were coconut shell bought from Ogbete main market in Enugu; unsaturated polyester resin (matrix), methyl ethyl ketone peroxide (catalyst), cobalt Naphthanate (accelerator) were supplied by Ndidiamaka Trading Company in Enugu.

2.2 Methods

2.2.1 Drying of coconut shell

The coconut shell was sundried eight hours per day for six days. It was divided into two portions and one portion carbonized and the other uncarbonized.

2.2.2 Coconut shell processing (Carbonization)

The coconut shell that was sundried for 48 hours was packed in an earthen pot, covered with a lid and heated in electric resistance furnace model KGVB kohaszat gyarepito vallalat, Type –Koo 80/50-120 "Temperature -950°c 513-4124 -0730/B at a temperature of about 700°C with a heating rate of 5°C per minute and a soaking time of three hours to form carbonized coconut shell. This was crushed to powder using a pulverizing machine. A particle size analyzer in accordance with ASTM standard was used to obtain two filler sizes of 425 µm and 1.70mm.

2.2.3 Grinding of coconut shell

Both samples of the coconut shell i.e. carbonized and non-carbonized samples were crushed to different particle sizes using a locally fabricated grinding machine at new market, in Enugu.

2.2.4 Sieving of ground coconut shell

The pulverized coconut shell particle samples were sieved using a set of sieves arranged in descending order of fineness. Sieve Model 567924/173281Endecotts Test Sieves, ltd. London, England was used to sieve the ground coconut shell to different particle sizes. The collected powder was sieved to different mesh sizes according to ASTM standard. About 1000g of CSP was put over the sieve shaker and shaken for 15 minutes to have homogenous reinforcement-matrix interfacial mixture.

2.2.5 Weighing of the materials for composites sample production

The unsaturated polyester resin was weighed using a digital scale-precisa XB6200D weighing machine with the use of the beaker placed on the petty dish.

The polyester resin was added gradually into the beaker until the weight indication observed is equal to the required polyester amount needed for that particular mixture with reinforcement particulates. The same process was repeated for other polyester amount needed for the other particulate mixture. The catalyst (methyl ethyl ketone peroxide) was weighed using the electronic machine with the use of beaker placed on the weighing machine. With the help of a syringe, the quantity of the catalyst needed was taken and poured by drops into the beaker containing the reinforcement-matrix mixture. With the indicator on the weighing machine, the pouring by drops of catalyst was stopped once 1g of catalyst is obtained. The same procedure was used for the accelerator (cobalt Naphthanate) but 0.5g was used.

2.2.6 Chemical characterization of the coconut shell

Proximate Analysis was used to determine the chemical composition of the coconut shell particles. The Proximate Analysis provided information on moisture, ash, volatile matter and fixed carbon content on dry or weight base. This is one of most characterization methods to analyze bio-fiber material like coconut shell. It provides information on moisture, ash, volatile matter and fixed carbon content on dry weight base. In this instance, the proximate analysis was carried out in observance of ASTM standards E-871, E-1755, E- 872 for moisture at 110°C, ash at 715°C and volatile matter at 925°C using a muffler furnace. The fixed carbon content was determined by subtracting the sum of the values of weight percent of moisture, ash and volatile matter from 100%. It was carried out at Scientific Equipment Development Institute, (SEDI) Akwuke, Enugu.

$$Fixed\ Carbon\ (FC)\ (\%) = 100 - (Ash + Moisture + Volatile\ Matter)\% \ldots\ldots \quad (1)$$

2.2.7 Composite sample preparation

A mold of 420 mm × 50 mm × 15 mm having a base of glass and sides of wood was used for casting the composite sheets. About 2000ml measuring cylinder was used to measure out the coconut shell particulate and the corresponding volume of the polyester resin. The volume fraction percents of coconut shell particles carbonized and un-carbonized (i.e. 10, 20 and 30 weight %), were mixed with the matrix material consisting of polyester resin in different volume fractions (1800ml, 1600ml, and 1400ml), accelerator and catalyst. The accelerator and the catalyst were mixed in the ratio of 1:2.

Care was taken to avoid formation of air bubbles during pouring and the mixture was covered to avoid buckling and allowed to cure at room temperature for 24 hours. For quick and easy removal of the molded composite sheet, a mold release sheet was kept over the glass plate and wood sides. After curing, the laminate produced was trimmed and cut into required size using a hack saw for water absorption test. The above process was repeated for the carbonized and un-carbonized coconut shell.

2.2.8 Density Measurement

Specimens of size 10x10x15 mm^3 were taken from the cast composite sheet. The weights of these samples were measured using a digital scale-precisa XB6200D weighing machine. Density calculation was done using the formula:

$$Density = mass/volume\ of\ sample \qquad \text{…. (2)}$$

2.2.9 Hardness test procedure

Hardness refers to a material ability to resist permanent or plastic deformation. Rockwell hardness test was carried out by indenting the specimen.

This method is the best for achieving the bulk or macro-hardness of materials particularly those materials with heterogeneous structure.

The machine model used was Type DVRB-M 220/240 V. The hardness tests were performed according to ASTM D785 standard using Rockwell Scale K hardness testing machine.

The proper indenter ball 1/8” for scale K was installed. The indenter was put into the pressure shaft so that flat part of the indenter cylinder was in front of the Allen screw. The Allen screw was slightly tightened and the machine switched on by the main switch in front of the panel. Loading force of 100kgwas selected using the lever force on the right side of the machine. The test specimen was placed on the anvil and lifted against the indenter. The anvil was lifted with the test specimen carefully until the green light in front of the panel came on. The red light on the panel signaled the completion of the test and the machine switched off automatically.

2.2.10 Flexural test procedure

Flexural test was performed using Universal Testing Machine model TUE-C-100, according to ASTM D790.The composite samples were tested at a three-point bending test at a cross head speed of 5 mm/min. In each case, three samples were taken and average value of the flexural force was recorded.

The flexural stress was computed using the following equation:

$$\sigma\text{max} = \frac{3P_{max}L}{bh^2} \qquad \text{….. (3)}$$

Where, P_{max} is the maximum load at failure (KN), L is the span (mm); b and h are the width and thickness of the specimen (mm) respectively.

2.2.11 Water absorption test procedure

This test was done at Standards Organization of Nigeria, Enugu. The composite samples were ovum dried at 60ºC for 2 hours, weighed and immersed in distilled water at room temperature. The water absorption was determined by weighing the samples at regular intervals of twenty-four hours for five days i.e. one hundred and twenty hours.

The specimens were periodically taken out of the water, wiped with tissue paper to remove surface water and weighed. Two specimens each for the carbonized and un-carbonized samples were used. A digital scale-precisa XB6200D was used to weigh the samples. The percentage of water absorption, % W_a was calculated by:

$$\% Wa = \frac{W1 - W2\ x\ 100\%}{W1} \qquad \text{…… (4)}$$

Where

W_a is percentage of water absorbed after one hundred and twenty hours; W_1 and W_2 are original dry weight and weight after exposure i.e. after one hundred and twenty hours *respectively.*

3. Results and discussions

Table 1: Proximate Analysis Result (wt %)

	Fixed Carbon	Volatile Matter	Ash	Moisture
Un-carbonized coconut shell	18.23	73.92	2.55	5.3
Carbonized coconut shell (700°C)	58.60	31.80	6.2	3.40

3.1 Chemical analysis result

The ash content is an indication of the presence of carbon compounds and inorganic components in the form of salts and oxides in the shell of coconut. The fixed carbon content of the carbonized coconut shell is higher than the un-carbonized coconut shell sample as can be seen in Table 3.1. The increase in carbon content was as a result of carbonization. Therefore, carbonization increased the carbon content of coconut shell. Furthermore, the volatile matter content of the carbonized coconut shell sample is lower than that of the un-carbonized coconut shell sample (Table 3.1). This is due to the removal of non-cellulosic filler components and increased surface roughness of the coconut shell leading to better bonding of treated coconut shell fillers with unsaturated polyester resin and also would increase mechanical interlocking with unsaturated polyester resin as was observed by (Salmah *et al*, 2013) in their work, “Treated Coconut Shell Reinforced Unsaturated Polyester Composites’. The hydrophilic nature of coconut shell makes it prone to moisture absorption, limiting the exterior application of the developed composites. The moisture absorption of the lingo-cellulosic fillers may cause dimensional change of the resulting composites and weaken the interfacial adhesion. This leads to induced stress, micro-cracking and ultimate failure in service of the composite. In proximate analysis, the pyrolysis of hemicellulose and cellulose occurs quickly; while the weight loss of hemicellulose mainly happenes at 220–315 °C, that of cellulose is at 315–400 °C. However, lignin is more difficult to decompose, as its weight loss happens in a wide temperature range (from 160 to 900 °C) and the generated solid residue very high. Also, lignin and cellulose contain most of the polar hydroxyl (OH) group in coconut shell. Therefore to reduce drastically the polar hydroxyl (OH) group in lignin that will ensure poor moisture absorption of the coconut shell particle in a composite, a carbonization temperature of 700ºC used in this research work was apt.

In addition, the important parameters that determine the quality and the yield of carbonized products are rate of heating, final temperature and soaking time; therefore, the need for a heating rate of 5ºC per minute, soaking time of three hours and 700ºC final temperature that were used in this research.

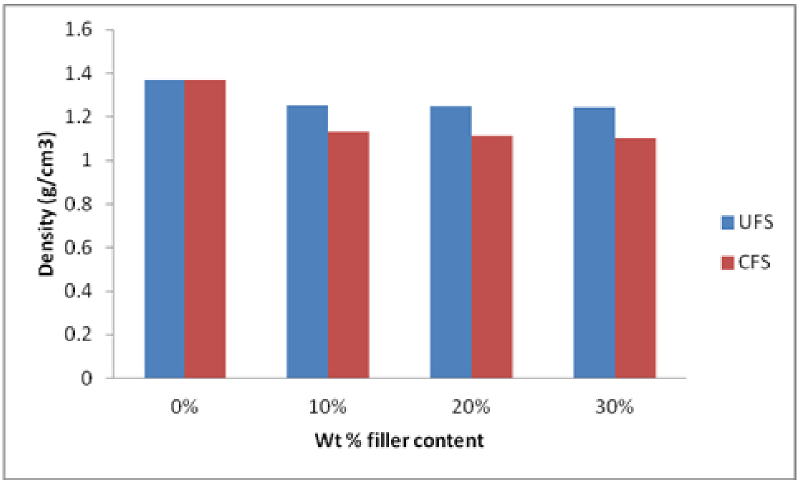


Fig 1a: The effects of particle carbonization on density of coconut shell fine particle reinforced unsaturated polyester composite

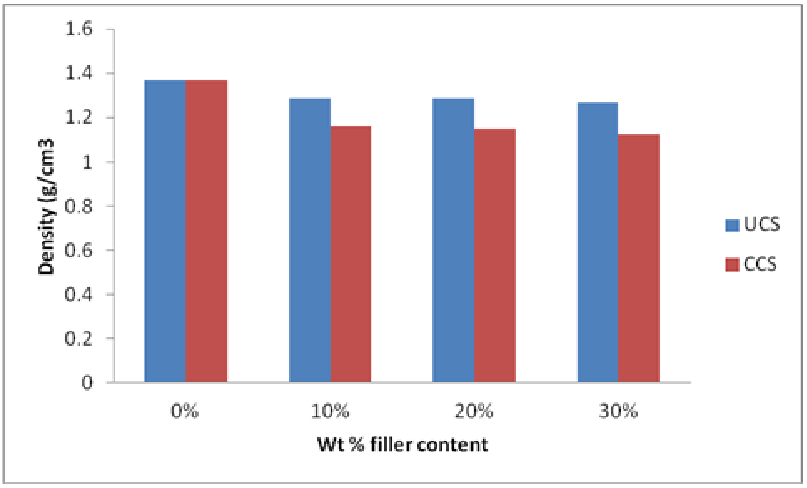


Fig 1b: The effects of particle carbonization on density of coconut shell coarse particle reinforced unsaturated polyester composite

3.2 Density

The unreinforced polyester had the highest density value of 1.367g/cm^3 as can be seen in Figure 1a and Figure. The density of the composites decreased as the quantity of the reinforcement increased for both uncarbonized and carbonized fine and coarse coconut shell particles as illustrated by figures 1a and 1b respectively. The coarse coconut shell particle reinforced composite samples had the highest density values of 1.288g/cm^3 and 1.164g/cm^3 at 10wt. % for the uncarbonized composite sample while the fine coconut shell particle reinforced composite sample had the lowest density value of 1.241g/cm^3 and 1.10g/cm^3 at 30wt. % for the carbonized composite sample. The carbonized coconut shell reinforced composite sample had the lowest density value in the than the uncarbonized sample. This may be likened to substantial loss of volatile matter and moisture during carbonization. Also, the fine coconut shell particle reinforced composite sample for the uncarbonized and carbonized coconut shell had lower density value than the coarse coconut shell reinforced composite. However, the carbonized fine coconut shell particle reinforced composite recorded the least density value among all the composite samples. It may be argued that reduction in particle size resulted in lower density value. Generally, the observed decrease in density with increase in weight percent of the reinforcement may be attributed to the light weight of coconut shell particles. They also occupied substantial amount of space in the polyester matrix.

3.3 Flexural Test Result

Table 2: Flexural Test Results of Uncarbonized Coconut Shell Composite Samples

Volume fraction of particulate	Sample Designation	Flexural force	Sample Designation	Flexural force
0%	Control	2.148	Control	2.148
10%	UFS	3.905	UCS	0.663
20%	UFS	4.440	UCS	3.210
30%	UFS	3.690	UCS	3.205

Table 3: Flexural Test Results of Carbonized Coconut Shell Composite Samples:

Volume fraction of particulate	Sample Designation	Flexural force	Sample Designation	Flexural force
0%	Control	2.148	Control	2.148
10%	CFS	4.110	CCS	2.470
20%	CFS	5.440	CCS	0.615
30%	CFS	4.015	CCS	2.240

The results of uncarbonized and carbonized fine and coarse particle coconut shell reinforced polyester are as shown in Tables 2and 3.

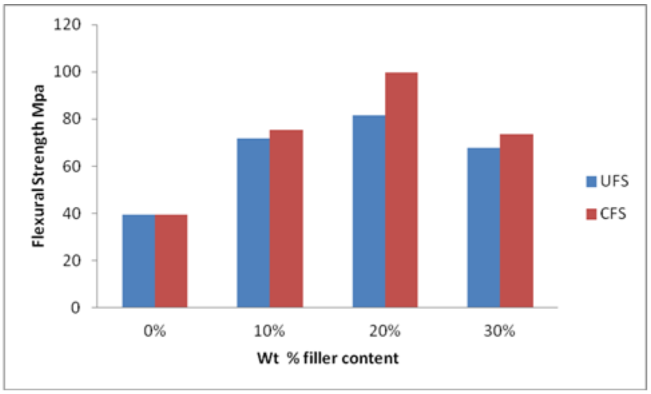


Fig 2a: The effects of particle carbonization on flexural strength of coconut shell fine particle reinforced unsaturated polyester composite.

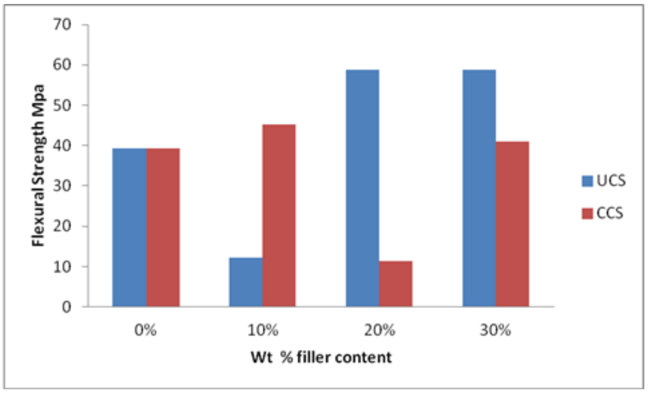


Fig 2b: The effects of particle carbonization on flexural strength of coconut shell coarse particle reinforced unsaturated polyester composite

The flexural properties of composites depend critically on the microstructure of the composite and the interfacial bonding between the reinforcement and the matrix (Subita Bhagat and Pardeep Kumar Verma, 2013) [10]. From the Figures 2a & 2b, it could be observed that the flexural strength of the composites generally increased with increase in reinforcement content at 10wt. %. This increase could possibly be due to strong interfacial adhesion between the particles and the matrix which enhances load transfer as was indicated by (Swain, 2013) [13]. However, the 10wt. % coarse un-carbonized sample was an exception to this increment in flexural strength. This could be as a result poor distribution of the reinforcement particles within the matrix. The fine particles of both the carbonized and un-carbonized samples showed a better flexural strength than the coarse samples which is as a result further reduction in particle size (Tables 2 & 3). The 20wt. % carbonized fine particle reinforced sample had a higher increase in flexural strength than any other sample and exhibited the maximum flexural strength of 99.733MPa (Table 3).

This may be as a result of carbonization which enhanced the structural strength of the coconut shell particle to withstand excessive particle crumble during use (Manocha, 2003) [11]. (Sapuan *et al*, 2003) [14], in their work Mechanical Properties of Epoxy/Coconut shell Filler Particle Composites examined the flexural properties of epoxy - CS composites and reported an increase in flexural strength and modulus up to 15 wt. % CS within the weight fractions considered. Also, the later decrease in flexural strength as the weight percent of the filler coconut shell increased as observed in the 30wt% was due to agglomerate formation at higher concentrations of the reinforcement. The carbonized and un-carbonized coarse particle reinforced samples showed the lowest flexural strength which is due to reduced surface area of the reinforcements in the matrix.

3.4 Hardness Test Result

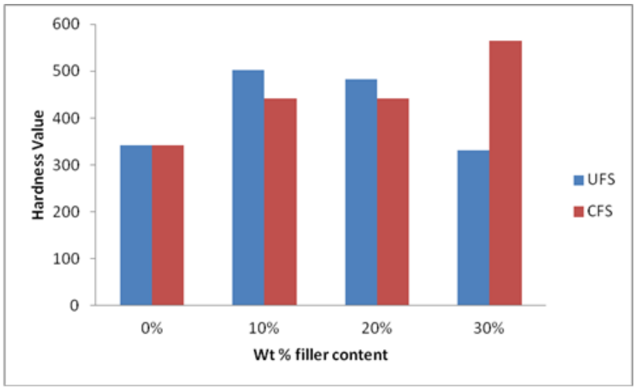


Fig 3a: The effects of particle carbonization on hardness value of coconut shell coarse particle reinforced unsaturated polyester composite

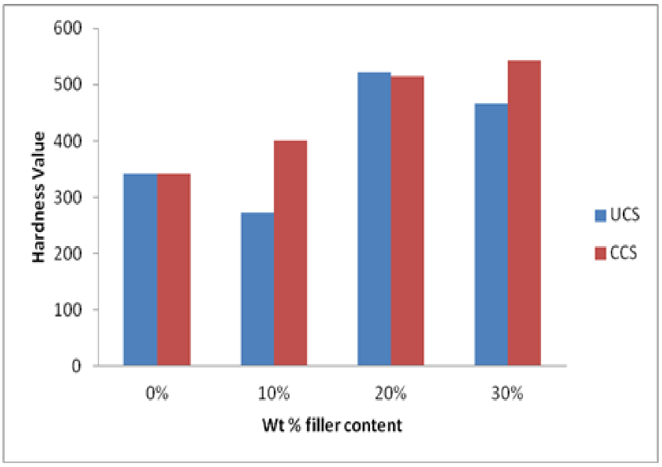


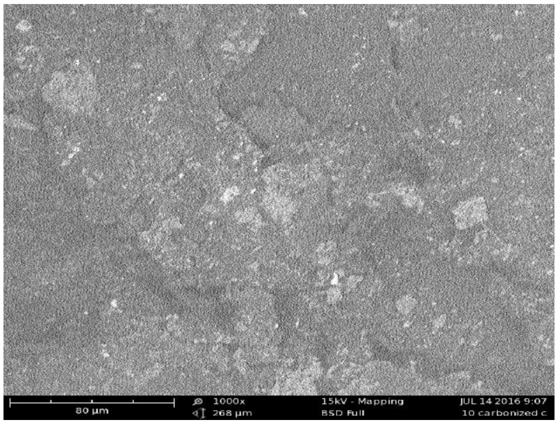
Fig 3b: The effects of particle carbonization on hardness value of coconut shell coarse particle reinforced unsaturated polyester composite

The hardness response of the composite is shown in Figures 3a & 3b. For the carbonized particle reinforcement, the hardness value increased up to a maximum value of HRN564 in the fine particle reinforced sample at 30wt% weight percent, Figure 3a, while the lowest hardness value of HRN 400 was observed in the coarse particle at 10wt%. Both samples showed an increase in hardness value as the particle volume fraction increased. For the un-carbonized particle sample, Figure 3b, the highest hardness value of HRN 522 occurred at 20wt% of the coarse particle reinforcement and the lowest of HRN 272 was observed at 10wt% of the same particle size. Generally, the carbonized samples showed a better hardness behavior than the un-carbonized samples which could be attributed to increase in carbon content of the coconut shell while the fine particle size had a better hardness than the coarse particle which also could be seen as a result of better dispersion of the particle in the composite.

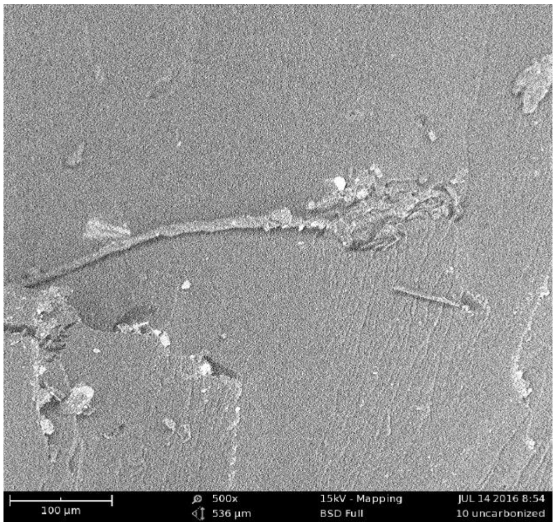
The observed increase in hardness value is related to the increasing amount of particulate and brittle coconut shell particle in the polymer matrix and strong interfacial bond between the particulate and the matrix. The hard and brittle particulates will resist deformation due to indentation.

3.5 Scan Electron Microscope (SEM) Result

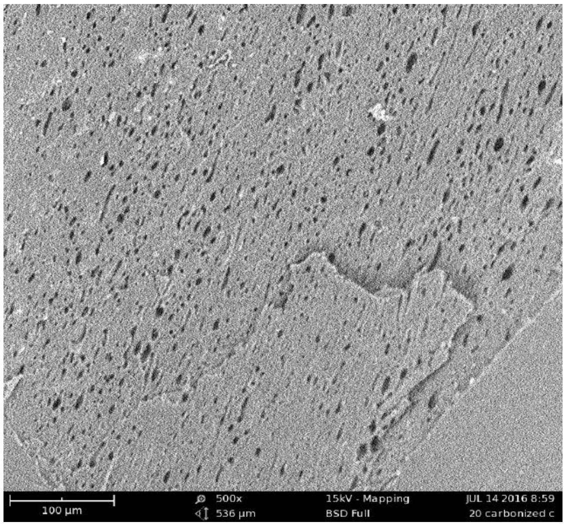
(A)



(B)



(A)



(B)

Fig 3: SEM Micrograph of reinforced polyester composite. (a) Unreinforced polyester resin (b) 10% carbonized coarse coconut shell particles (c) 10% uncarbonized fine coconut shell particles (d) 20% carbonized coarse coconut shell particles (e) 20wt.% carbonized fine coconut shell particle. (f) 30wt. % uncarbonized fine coconut shell particle

The micrographs of the uncarbonized composites figures (c) fine particle and (f) coarse particle show some detachment of filler from matrix. This is an indication of insufficient bonding between matrix and coconut shell particles and as a result, less adhesion occurred between them. The micrographs of carbonized composites in plates (b) coarse particle and (e) fine particle show that the coconut shell particles were better dispersed in the polyester matrix and this accounted for less detachment and agglomeration of coconut shell particle in polyester matrix, thus enhanced interfacial bonding between coconut shell particles and the polyester matrix. This explained the better flexural strength in the 20wt% and hardness value in the 30wt. % of the carbonized composite sample. Pores observed in the carbonized coarse sample, figure (3d) may be as a result of carbon segregation in the matrix.

3.6: Water absorption result analysis

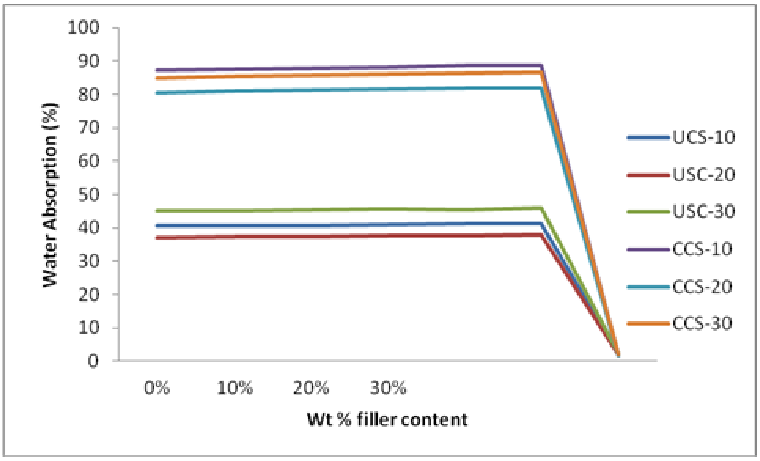


Fig 4a: The effects of particle carbonization on Water Absorption of coconut shell coarse particle reinforced unsaturated polyester composite

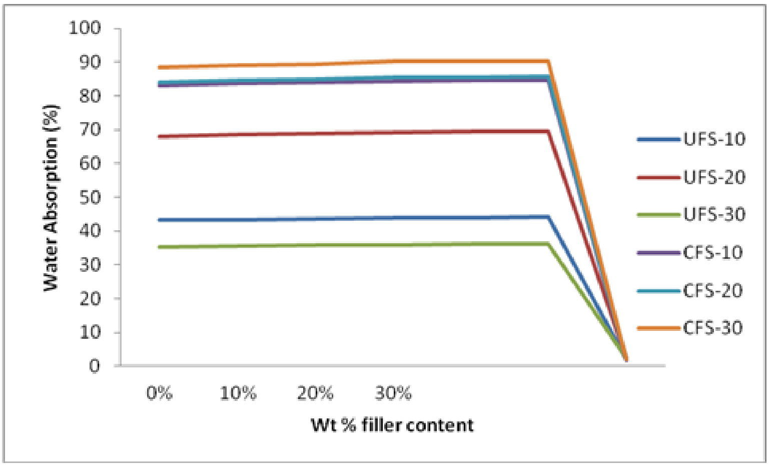


Fig 4b: The effects of particle carbonization on Water Absorption of coconut shell Fine particle reinforced unsaturated polyester composite

Most of the composites showed a similar pattern of water absorption where initial sharp water absorption uptake was followed by a gradual increase for the one hundred and twenty hours of immersion in water. From the water absorption chart in Figures 4a and 4b, maximum water absorption occurred in the composite prepared with 30wt. % uncarbonized fine particle coconut shell sample while minimum water absorption occurred in the 10wt. % carbonized coarse coconut shell particle composite sample. This may be due to removal of volatile matter and moisture containing the polar hydroxyl group (OH) during carbonization. The polar hydroxyl group is responsible for water absorption in coconut shell. Figure 4a and 4b also revealed that the percentage of water absorbed increased with increase in the quantity of coconut shell particle for both composite samples. In terms of particle size, the fine particle coconut shell composite sample absorbed more water than the coarse particle sample in both uncarbonized and carbonized composite samples. However, the uncarbonized fine coconut shell particle composite had a higher water uptake which could be likened to the presence of polar hydroxyl group in the walls of the uncarbonized coconut shell. Higher water absorption witnessed in the fine coconut particle reinforced composite for both samples may likely be as a result of increased surface area.

4. Conclusion

Unsaturated polyester resin reinforced with carbonized and uncarbonized coconut shell particles where studied for effect of carbonization on selected mechanical and physical properties and the following conclusions drawn.

1. Carbonization lowers the moisture and volatile matter contents of coconut shell which contain the shell's hydroxyl group responsible for water absorption. As a result, water absorption in a carbonized coconut shell/polyester composite was drastically reduced.
2. Carbonized coconut shell particle reinforced polyester composite is lighter than the uncarbonized coconut shell/polyester composite.
3. Carbonized fine coconut shell particle reinforced composite had a better flexural strength of 99.733MPa at 20wt. %.
4. Carbonized fine coconut shell particle reinforced composite had a better hardness value of 564HRN at 30wt. %.
5. The percentage of water absorbed increased with increase minimally with quantity of coconut shell particles.
6. The density of the composites decreased as a result of carbonization.

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