Effect of Carbonization and Particle Size on the Impact Strength and Water Absorption Properties of Coconut Shell /Polyester Composite

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Abstract— This research work investigated the effect of carbonization on the impact strength, water absorption and density of coconut shell/unsaturated polyester composite. A portion of the coconut shell was carbonized at 700ºC in a heat treatment furnace with a soaking time of three hours while the other portion was uncarbonized. The coconut shell was carbonized at 700ºC with a soaking time of three hours, ground and sieved into two particle sizes of 425micron and 1.70micron referred to as fine and coarse samples. Water absorption and impact strength of the developed composites were determined. The 20wt% un-carbonized coarse coconut shell reinforced composite sample absorbed more water while the 10wt% carbonized fine coconut shell composite sample absorbed the least quantity of water. Maximum impact strength was obtained at 30wt. % for fine coconut shell particle reinforced composite sample. This composite can be used in automobile and aerospace industries or applications requiring light weight.

Keywords— Polyester composite, Carbonized coconut shell, water absorption, Impact strength, SEM.

I. INTRODUCTION

Coconut shells 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 filler based composites while reducing waste. Procurement and processing of coconut shell powder is cost effective than other artificial fillers. Previously, coconut shell was burnt as a means of solid waste disposal which contributed significantly to CO_2 and methane emissions (Singh, et al., 2013).

Because natural particle-reinforced polymer composite materials are renewable, cheap, completely or partially recyclable, and biodegradable (Singh, et al., 2013), there is a growing interest in such materials both in terms of their industrial applications and fundamental research.

Presently in Nigeria, as was observed by Agunsoye et al., 2012, recycling of waste products and other agricultural by-products suitable for the invention and characterization of new materials has been attracting much interest.

Monteiro et al., (2008), and Wang and Huang, (2009), put the annual harvest of coconuts at approximately 33 billion worldwide with only 15% of these coconuts being utilized for fibers and chips. This therefore, suggests that there is considerable room to reduce this kind of environmental pollution and enhance the efficiency of using natural resources.

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Coconut shell contains about 65 – 75% volatile matter and moisture which are removed largely during the carbonization process (Chanap, 2012). The carbonization process involves converting the coconut shells to char (charcoal). The charring process (making of charcoal) is known as pyrolysis, which is chemical decomposition of the shell by heating in the absence of oxygen. During the carbonization of coconut shells, between 400ºC and 850ºC, volatiles amounting to 70% of the mass of coconut shells on dry weight basis are released to the atmosphere, yielding 30% of coconut shell mass of charcoal.

Schröder et al., (2011) produced charcoal from different waste biomass including coconut shell. They generated activated carbon in a two-step process of pyrolysis at 600°C and steam activation at 900°C. Jun et al, (2011), concluded that surface modification of coconut fiber by plasma treatment enhanced the interfacial adhesion between the coconut fibers and poly lactic acid (PLA) matrix.

Ayrilmi, et al., (2011), were of the opinion that particulate composite have distinct properties rather than to improve strength, it contains large amounts of coarse particles that do not block slip effectively.

Husseinsyah and Mostapha, (2011), were of the view that increase in coconut shell content increases the tensile strength, Young's modulus and water absorption rate but reduces the elongation at break of coconut shell filled polyester composites.

This work, studied the use of agro-based coconut shell particles in carbonized and uncarbonized form to reinforce polyester matrix to develop a composite. The essence of the carbonization which was carried out at 700ºC with a soaking time of three hours was to reduce drastically the volatile matter content of the coconut shell and enhance the structural strength of the coconut shell.

II. MATERIALS AND METHOD

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 Enugu, Nigeria.

2.2 Method.

2.2.1 Coconut shell processing (Carbonization)

The coconut shell was sundried for 48 hours and 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. 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. A particle size analyzer in accordance with ASTM standard was used to obtain two filler sizes of 425microns and 1.70microns

2.2.2 Chemical characterization of the coconut shell

Proximate Analysis 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 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. 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.

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

2.2.3 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. 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.

2.2.4 Impact test

The impact tests were performed according to ASTM D256 standard using Impact testing machine model EXT94064/6705CE, 300 J capacity. The test machine made use of a pendulum-testing machine which determined the Charpy impact strength of the composite. The impact velocity of the pendulum is 5.24m/s. The impact toughness of a composite is determined by measuring the energy absorbed in the fracture of the specimen.

2.2.7 Water absorption test

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:

$$\% \, Wa = \frac{W1 - W2 \, x \, 100\%}{W1} \quad (3)$$

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.

III. RESULTS AND DISCUSSIONS

3.1 Chemical Analysis Result

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

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 Iloabachie et al., (2018). The fixed carbon content of the carbonized coconut shell is higher than the un-carbonized coconut shell sample as can be seen in Table 1. The increase in carbon content may be attributed to carbonization. Therefore, carbonization increases the carbon content of a material. The volatile matter content of the carbonized coconut shell sample was lower than that of the un-carbonized coconut shell sample (Table 1). This may be 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). 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

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to decompose, as its weight loss happens in a wide temperature range (from 160 to 900 °C) Iloabachie *et al*., (2017) and the generated solid residue very high. Also, lignin and cellulose contain most of the polar hydroxyl (OH) group in coconut shell.

TABLE 2. Impact Test Results of Carbonized Composite Samples.

Volume fraction of particulate	Sample Code	Impact Energy (J)	Impact strength (J/m^2)	Sample Code	Impact Energy (J)	Impact strength (J/m^2)
0%	Control	0.20	2000	Control	0.20	2000
10%	CFS	0.65	6,500	CCS	1.00	10,000
20%	CFS	1.00	10,000	CCS	0.30	3,000
30%	CFS	2.00	20,000	CCS	0.45	4,500

3.2 Impact Strength Test Result

As observed in Table 2, the carbonized fine coconut shell reinforced composite sample had the best impact strength value of 2J at 30wt. % of the particulate. This represents 900% improvement over the virgin unsaturated polyester resin.

Fig. 1. The effect of carbonized fine and coarse particles coconut shell sample on the impact strength of coconut shell reinforced unsaturated polyester composite.

TABLE 3. Result of Water Absorption.

S/N	Composite composition	Initial mass (g)	Mass after 24hrs (g)	Mass after 48hrs (g)	Mass after 72hrs (g)	Mass after 96hrs (g)	Final Mass after 120hrs (g)	Water Absorption (%)
1	10wt.% Carbonized Coarse	87.40	87.70	87.90	88.30	88.70	88.80	1.602
2	20wt.% Carbonized Fine	84.00	84.50	84.70	85.00	85.30	85.40	1.667
3	10wt.%Un-carbonized Fine	40.50	40.60	40.70	40.90	41.20	41.30	1.975
4	20wt.%Un-carbonized Coarse	37.10	37.30	37.50	37.60	37.80	37.90	2.156

The variation in impact strength for different weight percent of carbonized coconut shell is as shown in figure 1. The Impact strength chart of Figure 1, illustrates that the maximum impact strength was obtained for the 30wt. % carbonized fine particle composite while the lowest was for the 20wt. % of carbonized coarse particle. The impact strength value increased with increasing particle weight percent in the carbonized fine particle composite. The increase in impact strength of the composites is due to increase in elasticity of the composite thereby increasing the deformability of the matrix. This is in line with the work of (kaundal et al., 2012). As the quantity of the reinforcement increased in the coarse particles reinforced composites, it was observed that the impact strength decreased. This could be as a result of poor interfacial adhesion between the polymer matrix and the particles resulting into occurrence of micro-cracks at the point of impact which decreased the impact strength. The decrease in impact strength in the carbonized coarse particle composite sample could be attributed to the inability of the reinforcements to block the crack propagation resulting in reduction of the impact strength, therefore, corroborating the work of (Thomas et al, 2012).

3.3 Water Absorption Behavior

The water absorption behavior of the different composite samples was observed and recorded in Table 3. All 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
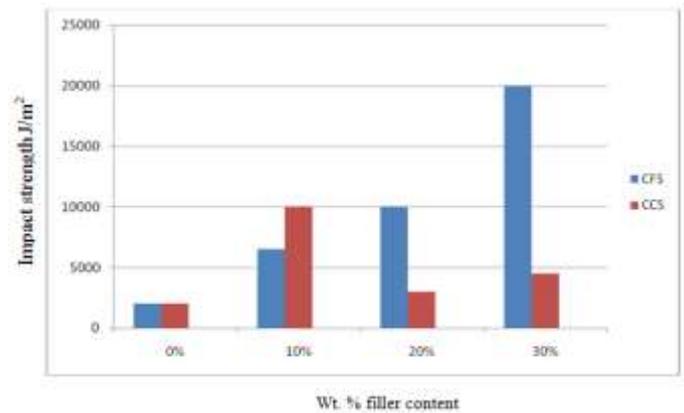
immersion in water. The water absorption chart Figure 2 illustrates that maximum water absorption occurred in the composite prepared with un-carbonized coconut shell sample while minimum water absorption occurred in the composite prepared with carbonized coconut shell sample. It was also evident from Figure 2 that the percentage of water absorbed increased with increase in the quantity of coconut shell for both composite samples.
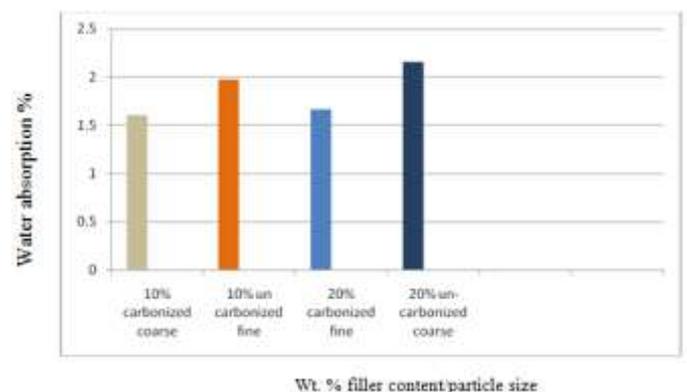
Fig. 2. The effects of carbonization and particles size on the water absorption behavior of coconut shell particle reinforced unsaturated polyester composite.

The 20wt% un-carbonized coarse coconut shell reinforced composite sample absorbed more water while the 10wt% carbonized coarse coconut shell composite sample absorbed the least quantity of water. As was stated by (Savita Dixit and

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Preeti Verma, 2012) Water absorption property is higher in untreated coconut coir than chemically treated coir. This is due to the more water soluble portion (may be hemicelluloses) present on the surface of the untreated coconut coir. Also, as was observed by (Singh et al, 2013 and Husseinsyah and M. Mostapha, 2011) the maximum water uptake increased as the natural filler content increased in a coconut shell polymer composite. The lower water absorption capacity observed in the carbonized coconut shell particle reinforced composite sample may be attributed to volatile matter containing the water soluble hydroxyl group and the moisture which was removed largely during the carbonization process as was observed by (Chanap, 2012). In terms of particle size, the carbonized fine particle reinforced composite absorbed more water at higher filler content compared to the coarse particle reinforced composite which absorbed less water at lower filler content. 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). Also, (Savita Dixit and Preeti Verma, 2012) stated that the removal of moisture from fibres by heat treatment at $150^{o}C$ is an essential step before the preparation of composites as it will help to check weak bonding between fiber and matrix, dimensional instability, matrix cracking and poor mechanical properties of the composites. Therefore, the observed lower water absorption of the carbonized coconut shell particle reinforced composite could be attributed to its carbonization at $700^{o}C$. 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.

IV. CONCLUSION

Coconut shell can be carbonized at $700^{o}C$. Carbonization of coconut shell at this temperature lowers the moisture and volatile matter contents of coconut shell which in turn affects the shell's hydroxyl group responsible for water absorption. As a result of this, water absorption in a carbonized coconut shell/polyester composite was drastically reduced. Particle size of the carbonized coconut shell affected the quantity of water absorbed in a coconut shell/polyester composite. The percentage of water absorbed increased with increase in the quantity of coconut shell. The 20wt% un-carbonized coarse coconut shell reinforced composite sample absorbed more water while the 10wt% carbonized coarse coconut shell composite sample absorbed the least quantity of water. Therefore, when carbonized coconut shell particle will be less prone to water absorption if used as a reinforcement material in a polyester resin to develop a polymeric composite. Impact strength increased with increase in particulate content in the fine particle carbonized composite sample while for the coarse

particle composite, impact strength decreased with increasing filler content.

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