

Mechanical, Abrasion and Water Absorption Characteristics of Coconut Shell Ash and Charcoal Based Polyester Composites

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Abstract

This research presents the mechanical, abrasion and water absorption properties of composites made from coconut shell particulates and unsaturated polyester resin. Particulate coconut shell was processed into ash and charcoal as potential reinforcement materials, respectively. The composites were fabricated by adding coconut shell particulates in predetermined proportions to the polyester. The cured samples were tested to ascertain the response of the materials to the selected properties. Experimental results showed that tensile and flexural properties of the composites increased as the coconut shell ash (CSA) particle content increases while these mechanical properties decrease as the content of coconut shell charcoal (CSC) increases. The abrasion resistance of the composites decreases as the reinforcement contents increase for both CSA and CSC composites. However, CSA samples showed least resistance to abrasion compared to CSC while the control sample displayed the highest wear rate. Similar trends were observed for water absorption response where initial sharp water uptake was followed by gradual increase until saturation of water content was achieved. SEM and EDX revealed the dispersal and elemental constituents of the fabricated composites, respectively.

Keywords: coconut shell, particulate, reinforcement, ash, charcoal, polyester, composites

1. Introduction

The development in science and technology required a variety of polymer with good properties and low cost. Therefore, polymer composites are considered to be among the more promising approaches to yield new materials and have been investigated extensively. In recent years, many studies have been dedicated to utilize lignocellulosic fillers such as coconut shell, wood, pineapple leaf, palm kernel shell and many more as fillers in order to replace synthetic fillers through exploitation of natural fillers as reinforcement in thermoplastic and thermosetting polymer composites. This is usually carried out in an attempt to minimize cost, increase productivity and improve mechanical properties of products [1].

Today, the growing environmental awareness throughout the world has triggered a paradigm shift from synthetic fibers and their composites to-wards composites made from natural reinforcing constituents which are more environmentally friendly. In the light of this, researchers have focused their attention on composites composed of natural or synthetic resins, reinforced with mineral particulate fillers or natural fibers in manufacturing of high-performance engineering materials from these renewable resources [2]. Many of the plant fibres find application as a resource for industrial materials [3].

Polyester resins are group of general purpose thermoset having average mechanical properties, low resistance to temperature, higher co-efficient of expansion and low cost. Nowadays, the cost is one of the most important factors. The overall cost can be reduced by blending the polymer with low cost filler materials like coconut shell powder [4].

Coconut shell (CS) is one of the most significant lignocellulosic materials which grow in tropical countries such as Malaysia, Sri Lanka, Thailand and Indonesia. In an attempt to reduce the abundance of these agricultural by-products, new applications are urgently needed for CS to be more useful. Therefore, the use of CS as lignocellulosic fillers in polymer composites becomes more pleasing due to their high strength and modulus properties [1].

Coconut shells are cheap and readily available in high quantity. It contains about 65-75 % volatile matter and moisture which are removed largely during the carbonization process. The carbonization process involves converting the coconut shells to char. The charring process is known as the Pyrolysis, which is chemical decomposition of the shell by heating in the absence

of oxygen. 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 30 % of coconut shell mass of charcoal. The volatile released during the carbonization process is Methane, CO₂ and wide range of organic vapors. The carbonization temperature range is usually within 400 - 850 °C. The product of carbonization is usually referred to as the ash. Research by Imoisili *et al*, (2012) [5] revealed that coconut shell ash (CSA) is a potential candidate for the development of new composites because of their high strength and modulus properties. Composites made from CSA can be used for a broad range of applications such as furniture, house hold appliances and building constructions.

Many researchers have made efforts to produce carbon black from agricultural by-products such as coconut shell, apricot stones, sugarcane bagasse, nutshells, forest residues and tobacco stems. Coconut shell is suitable for preparing carbon black due to its excellent natural structure and low ash content. Conversion of coconut shells into activated carbons which can be used as adsorbents in water purification or of industrial and municipal effluents would add value to these agricultural commodities, help reduce the cost of waste disposal, and provide a potentially cheap alternative to existing commercial carbons.

In this research, carbonized coconut shell was referred to as coconut shell ash (CSA) while the non-carbonized was referred to as coconut shell charcoal (CSC). The CSA and CSC particulates selected for this research were chosen so as to investigate their influence on polyester matrix, which is the most widely used among the thermoset plastics. The work was to examine the influence of charcoal and ash of CS since many researchers have established the use of CS particulate in an untreated and ash forms. In this research, the possibility of using CSC as reinforcement in polyester matrix composites fabrication in order to expand the scope of the areas of applications for the material was the main focus.

2. Materials and Methods

2.1. Materials

The materials utilized for this work includes: Coconut shell, unsaturated polyester resin, Methyl ethyl ketone peroxide (catalyst), Cobalt 2% in solution (accelerator), Ethanol and polyvinyl acetate (mould releasing agent).

2.2. Methods

2.2.1. Procurement and preparation of coconut shell particulate

Coconut shells were procured from a coconut chips producer in Ado Ekiti in Ekiti State, South-West, Nigeria. The coconut shell was dried in open air and burnt into charcoal in an enclosure. The charcoal was divided into two and one part was kept as charcoal while the other part was subjected to carbonization at 650 °C in a muffle furnace for 3 hours and furnace cooled. The collected char and the charcoal were grinded into powders with the aid of laboratory ball mill followed by sieving with sieve shaker to obtain 150 µm. The production process products are as shown in Plate 1. Carbonization is the production of charred carbon from a source material. The process is generally accomplished by heating the source material usually in the absence or limited amount of air to a temperature sufficiently high to dry and volatilize the substance. Therefore, CSC was produced by burning the coconut shell in the presence of air while CSA was produced by burning the coconut shell in the absence of air.



Plate 1. Production process products: coconut shell – coconut shell particulate

2.2.2. Fabrication of composites

The composites were developed using open mould technique shown in Plate 2 after mixing the materials in the proportions presented in Table 1. Unsaturated polyester, accelerator and coconut shell particulate were first blended together before the catalyst is added in order to initiate the polymerization reaction. The mixture was stirred thoroughly until homogeneous mix was achieved and was poured into the mould. The average time of mixing was about 10 minutes.

Table 1. Formulations for the polyester/CS composites

Materials	Composition of the developed samples	
Polyester (php)	100	php - parts per hund reds of total polymer
Coconut shell particulate (php)	1, 2, 3, 4, 5.	
Cobalt 2% solution (g)	4	
MEKP (g)	6	



(a) Tensile



(b) Flexural



(c) Wear

Plate 2. Samples inside the moulds: (a) Tensile. (b) Flexural. (c) Wear.

2.2.3. Property Test

Measurement of Tensile Properties

Tensile test was carried out in accordance to American Standard Testing and Measurement Method D412 (ASTM D412 1983) on Instron Universal testing machine. Composite samples with 3 mm thick and of gauge length 150 mm were used. Three identical samples were tested for each weight fraction from where the average values were used as the representative values.

Measurement of Flexural Properties

Three point bend tests were performed in accordance to ASTM D 790 M to measure flexural properties using Instron Universal testing machine. The samples were of 150 x 50 x 3 mm. Three samples were tested for each weight fraction used and the average values were taken to represent the actual values.

Measurement of Abrasion Property

This involves mounting the specimen to a turntable platform that rotates at a fixed speed. The samples were measured using an analytical weighing balance to take the initial weight of the samples. The weighed samples was mounted on turntable platform and rotated at 1000 r.p.m. for 20 minutes each. Thereafter, the weight was measured as the final value and the difference between the initial and the final value was noted and recorded against each samples. The average values were used like that of the mechanical properties.

Water Absorptivity Test (Swelling Behavior)

The composite samples were dried at room temperature for six weeks and immersed in distilled water at room temperature. The water absorption was determined by weighing the samples at regular intervals. The specimens were daily taken out of the water, wiped with tissue paper to remove surface water and weighed. The percentage of water content (W_t) was determined using equation 1:

$$\% W_t = \frac{W_t - W_o}{W_o} \times 100 \quad (1)$$

Where W_t is the weight of sample at time t and W_o is the initial weight of the sample.

3. Results and Discussion

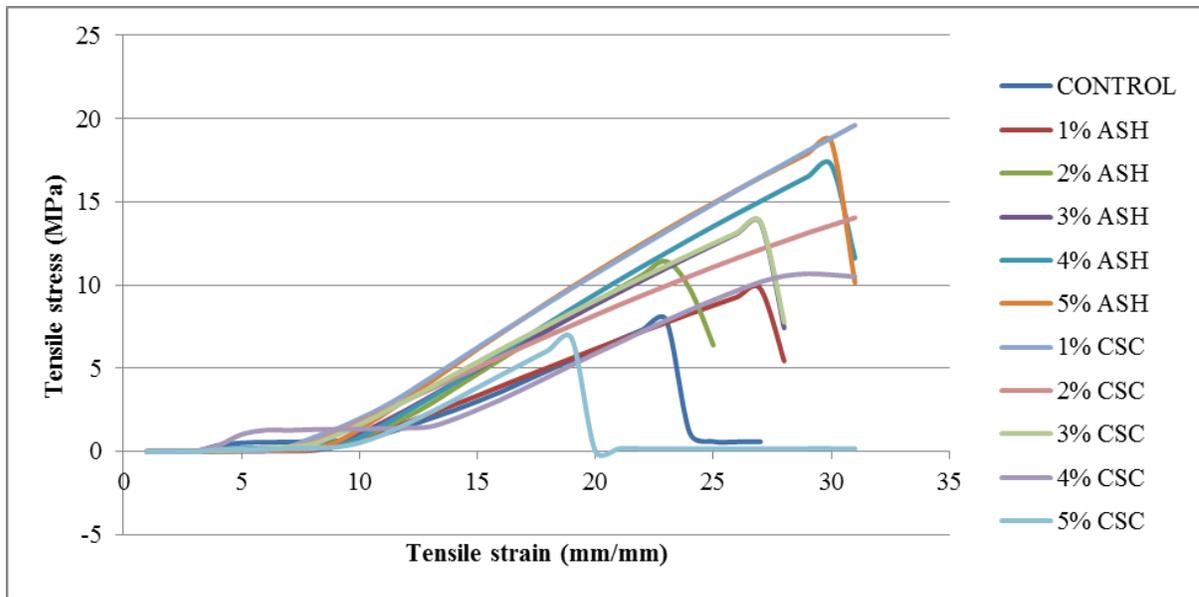


Fig. 1. Stress-Strain Curve for Coconut Shell Ash and Charcoal as well as the Control Samples

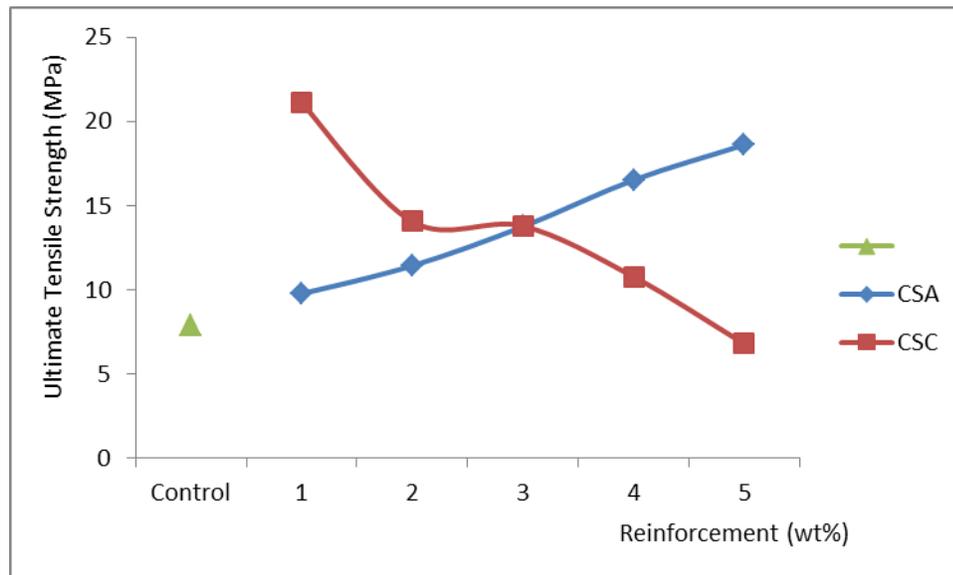


Fig. 2. Variation of Ultimate Tensile Strength with the Composites and Control

Fig. 1 showed the stress-strain curve for the response of the materials to tensile behavior while Fig. 2 depicts the ultimate tensile strength (UTS) of the materials. The UTS represent the maximum load or stress the materials can withstands before fracture and this was seen in Fig. 1 as the points where there is drop in the curves for each sample. The materials exhibit brittle

failure mode being a thermosetting based, and the reason for the observed sudden drop in tensile strength at the peak value.

From Fig. 2, the UTS of the developed composites were enhanced by the addition of the CS particulates. The selected particulates were noticed to influence the matrix in an inverse trend which was one of the reasons for this investigation. The UTS was observed to increase as CSA increases while it decreases as CSC increases. The increase obtained in CSA composites was as a result of strong particles–matrix interaction which increases the ability of the particles to restrain gross deformation of the matrix. Conversely, the decrease in ultimate tensile strength observed in CSC composites may be due to increase in discontinuity between the particles surface and the matrix as the reinforcement content increases. However, the best ultimate tensile strength potential was achieved with the addition of 1 wt% CSC which has a value of 21.12 MPa compared to other samples. Virtually all the reinforced samples had higher ultimate tensile strength compared to the control sample that has a value of 7.89 MPa. This implies that, the UTS can be enhanced with about 167 % with the use of CSC. The observed trend of results can be due the influence of the elemental contents on the weight fractions as well as the interfacial bonding strength. The increase in tensile strength of the composites was due to the ability of the particulates of the reinforcement contents used to support stress transfer from the polyester matrix.

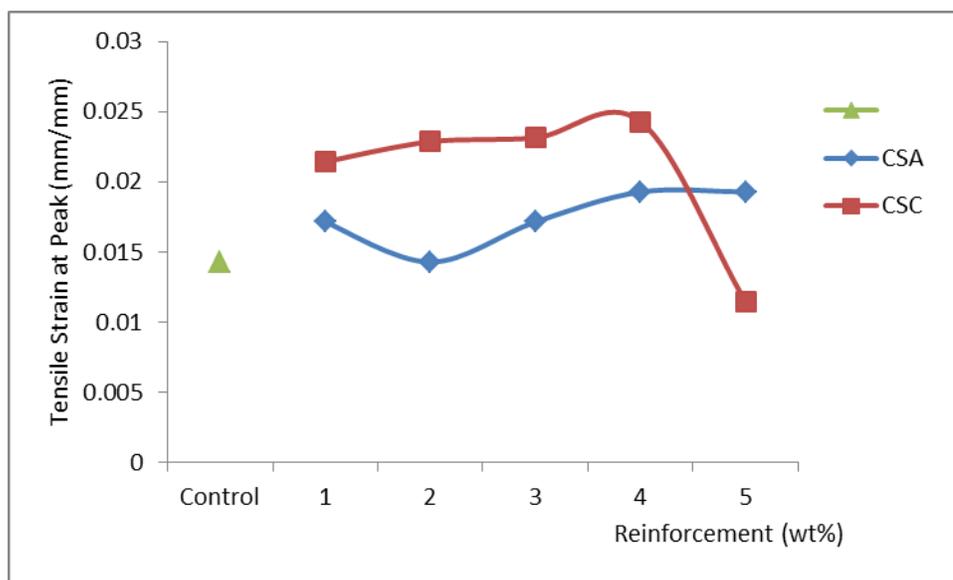


Fig. 3. Variation of Tensile strain at maximum load with the Composites and Control

As shown in Fig. 3, there was almost gradual increase in the tensile strain at maximum load as the ash content increases up to 4 wt% followed by a decrease at 5 wt% for CSA samples while a slight decrease was followed by an increase as the charcoal content increases for the CSC samples. Both composites show increasing strain (ductility) as reinforcement content increases. From the results, CSA sample possess better tensile strain property than CSC and the control for all the reinforcement content except at 5 wt%. Increase in reinforcement content of CSA above 4 wt % reduces the elasticity of the matrix. This enhances rigidity and causes a decrease in tensile strength and ductility as a result of restriction in the polymer matrix movement. The ductility at break decreased upon filler addition for composites regardless the nature of the filler. At higher filler content, the domination of filler-matrix interaction can be expectable to subside and being substituted by filler-filler interaction (Salmah *et al*, 2013).

The best performance was obtained at 4 wt% with a value of 0.024 mm/mm compared to the control with a value of about 0.014 mm/mm which culminated to about 71 % enhancement. This result was in agreement with the UTS results in Fig. 2 where 1-4 wt% addition of CSC was seen to possess better strength than the control. The results confirmed the capability of the samples with CSC to withstand higher strain than others within 1-4 wt%.

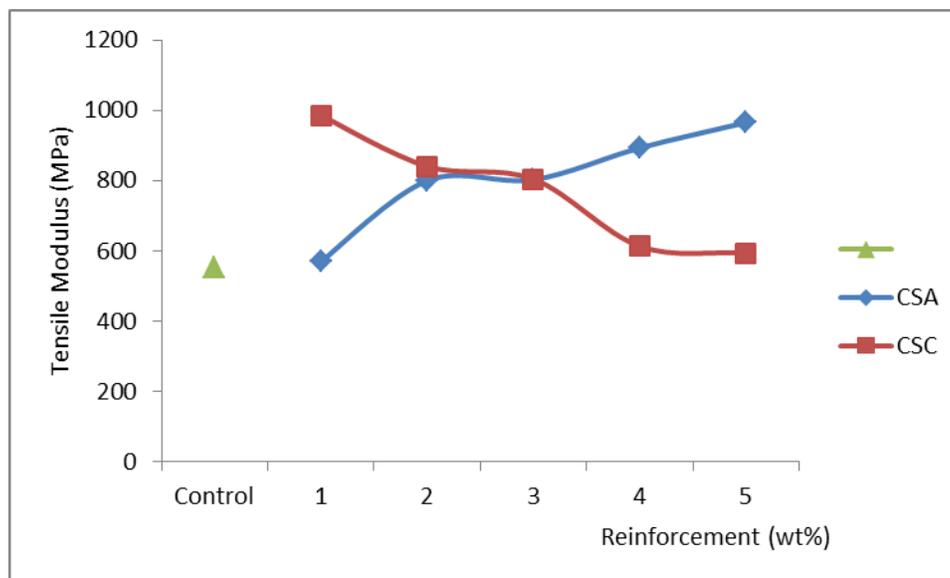


Fig. 4. Variation of Modulus of elasticity with the Composites and the Control

Fig. 4 show the response of the materials to tensile modulus. Similar trend to UTS in Fig. 2 was obtained, though; slight changes in trends were noticed for both CSA and CSC between 2-3 wt% reinforcement. The moduli for both samples at those weight fractions were almost the same. From the results, it was seen that 1 wt% gave the best tensile modulus result with a value of 985.39 MPa. The moduli of the developed composites were higher than the control sample which has a value of 552.74 MPa. The enhancement in percentage when compared with the developed composites that has the best result was 78 %. The result was in agreement with previous research findings in which it was stated that the addition of particles from lignocellulosic source increases the tensile modulus of the polymer composites. The incorporation of hard phase in particulate form tends to restrains the motion of the matrix phase in the proximity of each particle which consequently contributes to the enhancement in moduli and stiffness of the composites. The presence of the volatile materials as well as moisture which differentiate the CSC from the CSA may be responsible for the enhanced tensile properties obtained with the use of CSC when compared with the use of CSA and the control samples.

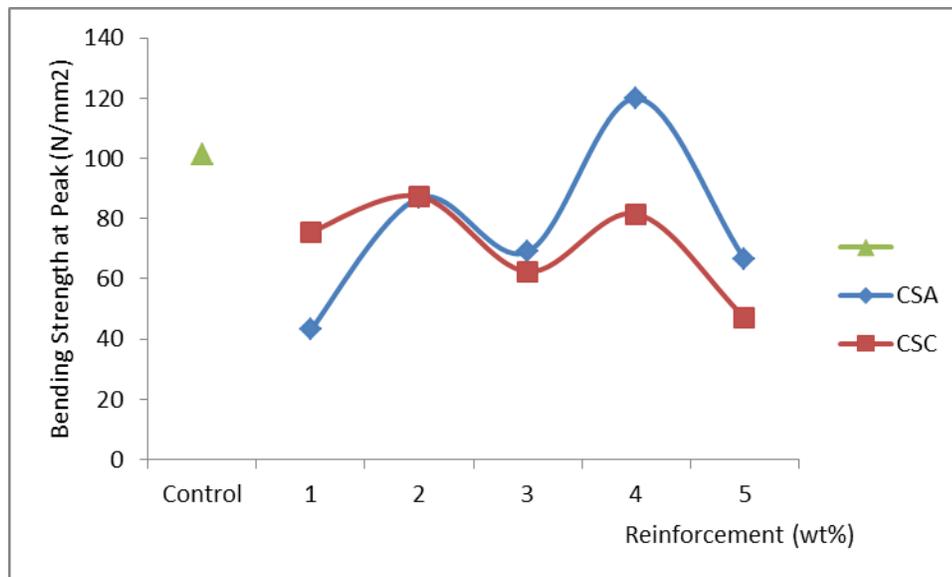


Fig. 5. Variation of Bending Strength at peak with the Composites and Control

Fig. 5 show the behavior the materials with respect to bending strength at peak. All the composites with the exception of the sample with 4 wt% CSA have less bending strength at peak value than the control. This observed situation only suggest that there is optimum value of CSA addition that will give better enhancement of the bending strength at peak for the matrix.

Though, the responses revealed an irregular trends but it was observed that, the addition of CSA tends to enhance the bending strength at peak as the weight content increases up to 4 wt% followed by a decrease while the addition of CSC tends to reduce the bending strength at peak as the weight content increases. However, it was seen that both reinforcements gave enhancement within the intervals of even numbers while the odd numbers gave reduction. This encourages the use of even numbers at higher weight fraction in the fabrication of composites for bending strength at peak application using CSA. The highest bending strength at peak was achieved at 4% ash particulate with a value of 119.91 MPa compared to the control with a value of 101.30 MPa.

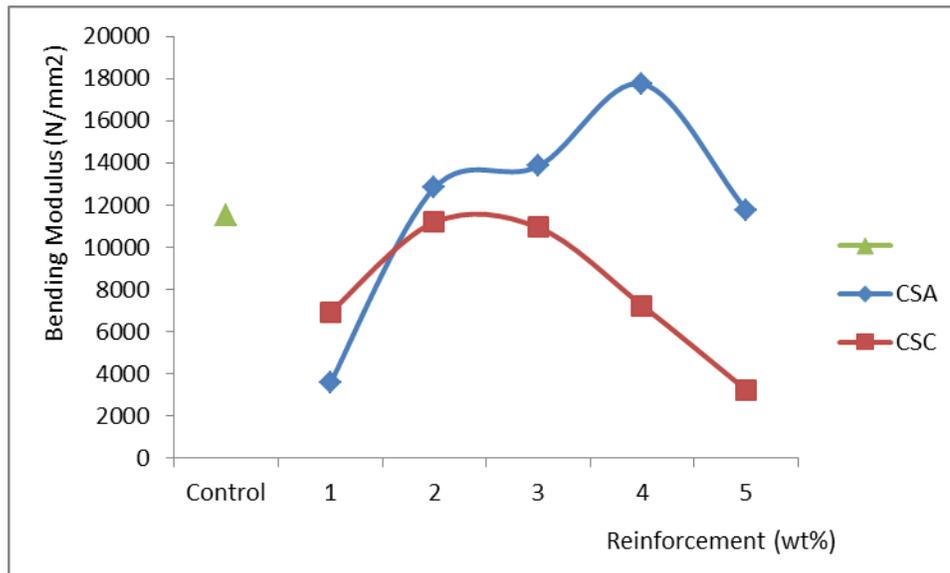


Fig. 6. Variation of Bending Modulus with the Composites and the Control

The response of the materials to bending modulus was shown in Fig. 6. All the CSA samples with the exception of 1 wt% reinforcement gave better bending moduli compared to the control which has a value of 11517 MPa while none of the CSC developed composites gave enhancement. Coconut shell ash with the best enhancement was 4 wt% with a value of 17742 MPa which correlate to about 54 % enhancement.

The flexural properties of composites depend critically on the microstructure of the composite and the interfacial bonding between the reinforcement and the matrix [6]. In Figs. 5 and 6, both

flexural strength at peak and modulus of the CSA composites were better enhanced at 4 wt %. This was due to the strong interfacial adhesion/bonding between the particles and the matrix which enhances load transfer [7].

It was observed in all the mechanical properties examined that similar trends were seen for the responses of CSA and CSC reinforced polyester composites. The properties tends to increases as the reinforcement content increases for CSA composites while it tends to decreases as the reinforcement content increase for CSC composites. Since the responses show a consistency in the tendencies of the behavior of the composites, it therefore implies that, both elemental constituents and weight fraction of these particles have influence on the mechanical properties of the developed polyester based composites.

Previous work by Oladele *et al*, (2013) confirmed the possibilities of using agro waste as reinforcement in polyester material. The work revealed that different agro waste particles can be used to enhance different mechanical properties for unsaturated polyester.

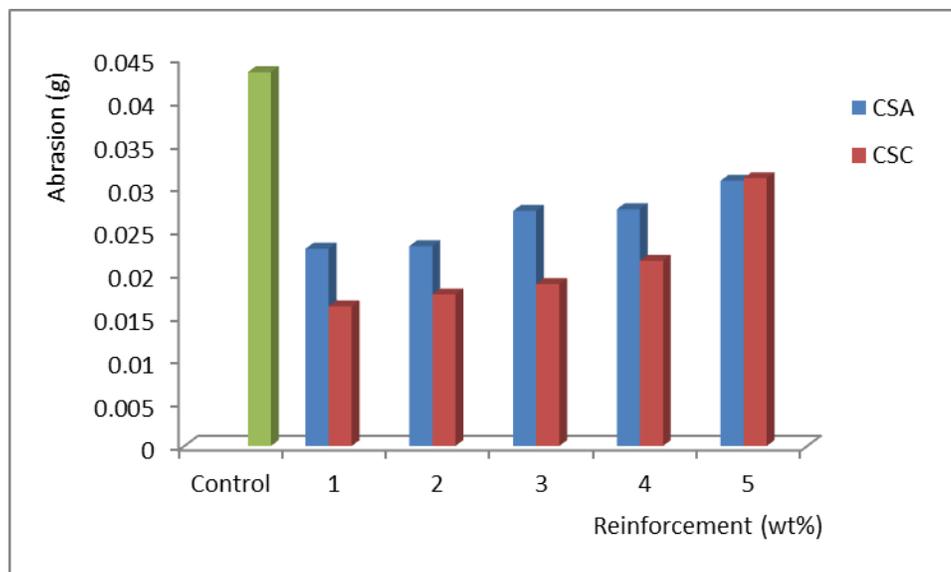


Fig. 7. Variation of Abrasion property with the Composites and the Control

The variation of the samples with abrasion was shown in Fig. 7. From the Figure, similar trends were seen for the two sets of reinforcement used where, it was observed that the wear resistance

reduces as the reinforcement content increases. The wear resistances of the developed composites were higher than the control sample for all the composites. However, it was seen that, the wear resistance of the charcoal was higher than that of the ash. This may likely due to the presence volatile materials that aid good interfacial bonding strength. The control sample showed the highest wear rate with a value of 0.0434 g while the sample with the best wear resistance was 1 wt% CSC with a value of 0.0162 g.

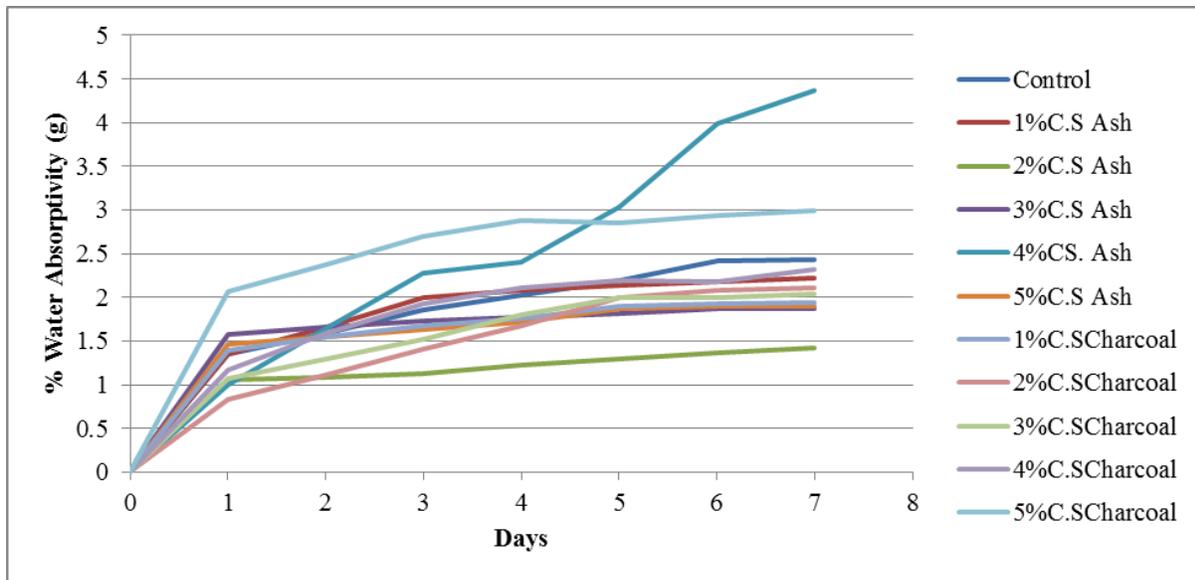


Fig. 8. Variation of % water absorption property with the Composites and Control

The relationship between water absorption and reinforcement content for Polyester/CS composites at different content of CS was shown in Fig. 8. The Figure displayed the rate of water absorption on daily basis for 7 days from where it was seen that 2 wt% CSA reinforced sample demonstrated the best water resistance potential. All the composites show a similar pattern of water absorption where initial rapid water uptake was followed by gradual increase until

gh the water absorption potentials seem alike for the sites, however, it was observed that most of the control. Though CS is from agro-fibers which are groups (-OH) in their structures and, this is usually treatment have eliminate this groups and therefore,

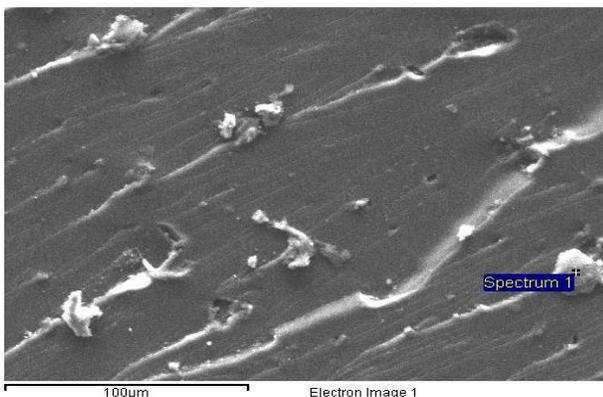


Plate 3. SEM and EDX of CSA reinforced composite

Table 2. Spectrum processing: Peaks possibly omitted : 1.045, 9.161 keV Processing option : All elements analyzed (Normalised)

Element	Weight%	Atomic%
C K	67.99	77.38
O K	23.74	20.28
Al K	0.86	0.43
Si K	1.04	0.51

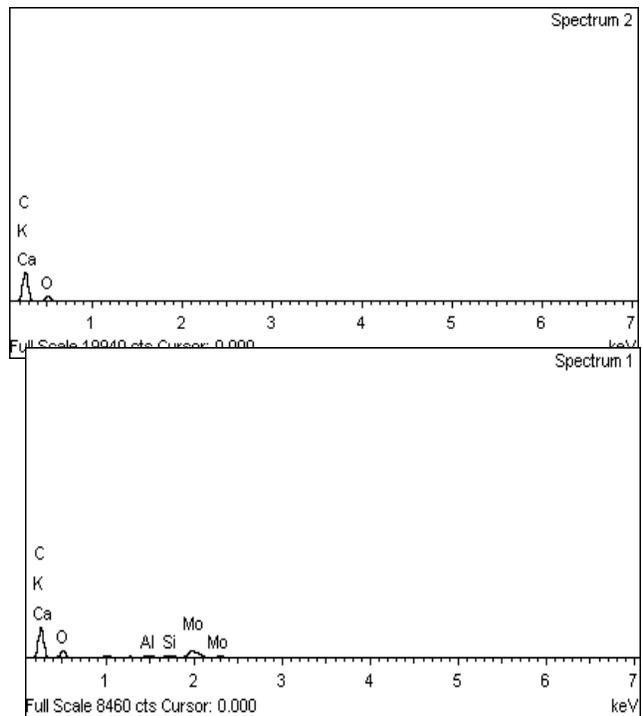
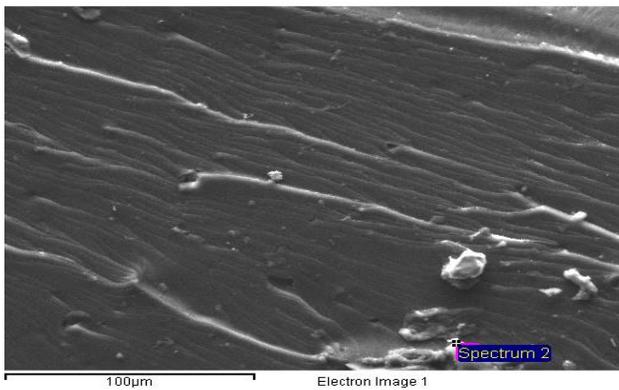


Plate 4. SEM and EDX of CSC reinforced composite

Table 3. Spectrum processing: No peaks omitted Processing option: All elements analyzed (Normalised)

Element	Weight%	Atomic%
C K	71.81	78.45
O K	24.12	19.78
Na K	0.67	0.38
Al K	0.65	0.31
Si K	0.50	0.23
P K	0.43	0.18
S K	0.30	0.12
Cl K	0.68	0.25
K K	0.38	0.13
Ca K	0.46	0.15
Totals	100.00	

Plates 3 and 4 displayed the SEM of the fractured surfaces of CSA and CSC reinforced composites, respectively while Tables 2 and 3 presented their elemental constituents. The SEM revealed that the CS particles were well dispersed within the polyester matrix and this support the reasons why the composites possess better properties compared to the control samples.

The elemental compositions shown by the EDX from the two processed CS particles revealed that the pyrolysis actually removed some elements like Sodium, Phosphorus and Sulphur which were present in the CS charcoal while the ash particles were noticed to contain Molybdenum which was not present in the charcoal. The elements that were present in the two particles were observed to either increased or decreased after the treatment. While Carbon, Oxygen and Chlorine were reduced after the treatment, Aluminium, Silicon, Potassium and Calcium increases. Therefore, the presence of these increased elements was responsible for the enhancement obtained in flexural properties of the CSA composites compared to others.

4. Conclusion

It was discovered from the results that reinforced polyester composites exhibited better properties than the unreinforced polyester matrix. While CSC composites possess better results in terms of tensile and abrasion properties, CSA possess better properties in terms of flexural and water absorption properties.

There were consistent trends on the influence of the CS particles on the composites mechanical, abrasion and water absorption properties which confirmed that the effect of these particles on these composites properties were real. Nevertheless, reinforcement content with the optimum perform for CSA composites was 4 wt% while 1 wt% gave the optimum yield in CSC composites.

The EDX confirm the elimination of some elements like Sodium, phosphorus and Sulphur as well as alterations in the quantity of the elements that were present after pyrolysis treatment. The results confirmed the possibility of inclusion of these particles into the polyester for enhanced performance.

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