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Development of Green Composites Based on Castor Bean Shell (*Ricinus communis*) as Filler in Epoxy Resin Polymer

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Abstract. *The present study was carried out to develop composites using waste castor bean shell (CBS) as a filler and epoxy resin as a matrix. The composites were produced by varying the weight fractions of filler in the range of 5 to 35 wt%. The effects of castor bean shell filler weight percentages (0, 5, 10, 15, 20, 25, 30 and 35) on the tensile, flexural, impact and hardness properties of the epoxy/castor bean shell composites were evaluated using universal testing machine, hardness tester and scanning electron microscopy (SEM). The study revealed that the tensile strength, tensile modulus, flexural strength and impact strength increased with the increasing of the castor bean shell filler content. The highest tensile, flexural and impact properties of the CBS loaded epoxy composite were achieved at a filler content of 15wt%. The hardness increased with the increasing of the castor bean shell filler content. The water absorption and the thickness swelling increased with the increasing of the castor bean shell filler content. The swelling rates of the castor bean shell-epoxy polymer matrix composites are low during the initial stages of moisture absorption due to the visco-elasticity of the polymer matrix. The density decreased with the increasing of the castor bean shell filler content. SEM showed improved interfacial adhesion of the castor bean shell filler-epoxy resin matrix at 15wt% CBS content. The study has shown that castor bean shell filler is a viable reinforcement for manufacturing green composites from the viewpoint of their physical and mechanical properties.*

Keywords: *green composite, epoxy resin, tensile strength, castor bean shell, scanning electron microscopy.*

1. Introduction

In our day-to-day activities polymer bio-composites have found diverse implementations owing to their exceptional attributes. Bio-composite performance is consequent upon selection of individual elements. Bio-fibers addition into a polymer is known to cause substantial alterations in physical and mechanical properties of the resulting composites. Recent years have witnessed

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increasing enthusiasm for bio-fillers as alternative to glass, carbon and man-made fibers due to their lightweight, inexpensive, sustainable, as well as environmentally friendly [1].

The flexibility in producing composites using bio-fillers is that the materials is sourced effortlessly from biomass wastes, therefore, composites are easily developed. Because natural fibers can be replenished their accessibility is sustainable. Yet, natural fibers possess dissimilar sizes, thermally unstable, and prone to water uptake. Normally, composites are applied in areas where materials' performance is of critical concern as attempts have been made to use bio-filler composites for lightweight applications. The alternative processing of bio-composites is an important task to scientists, and such method is the application of powder impregnation technology for the fabrication of biomass waste composites [2]. Among the top ten castor bean producers globally, India has the biggest production volume of 830,000 tons seconded by China with 210,000 tons. The figures are favorable compared to the overall global output 1,209,756 tons [3]. Recent innovation has also found the product adequate for bio-diesel manufacturing [4].

Processed castor bean serves as food and spice in south-eastern part of Nigeria. The seed of the plant utilized totals to huge volumes of solid waste due to improper utilization and ultimately discarded as waste. Waste castor bean shells are ubiquitous, and usually incinerated or arbitrarily discarded. This formed the major motivation to this study.

Epoxy is a thermosetting resin polymer having two or more epoxy groups. It has high mechanical properties due to its low shrinkage, exhibits high resistance to alkali, acids and solvent resistance. However, in many applications the common resin system cannot satisfy high mechanical properties. Cured epoxy systems generally exhibit good dimensional, thermal stability and resistance to fungi with excellent moisture barriers, exhibiting minimal water uptake and moisture transport. Recent quests for high performance composites have increased the usefulness of epoxy resins as structural adhesive, and as the matrix resin for advanced composites. The applications require toughness, rigidity, and good bonding properties for epoxy resins [5]. The blending of castor bean shell with epoxy resin would be an attractive method to develop new composites materials, and data obtained from this study might be useful for the effective utilization of waste castor bean shell thereby reducing the environmental impact. Recently, environmental issues caused by non-biodegradable plastics obtained from petroleum resources have aroused interest in developing green composites [6].

Improvement on the tensile strength and modulus with the increasing of peanut shell powder content with optimal tensile properties using 7% alkali bio-filler loading at 15wt% was reported by

Prabhakar *et al*. [7]. Arecanut husk filler treated-epoxy resin composites were fabricated by Muralidhar *et al*. They reported enhancement in the elastic properties with the increasing of the bio-filler loading frequencies [8]. Okonkwo *et al*. showed that the tensile strength and modulus of bambara nut shell reinforced polyester composites increased with the increasing of the bambara nut shell loading from 8-12 wt% [9]. Shih observed increase in the mechanical properties of bamboo husk reinforced epoxy composites at 10wt% coupling agent addition [10]. The effects of particle size and filler loading using hazelnut shell, walnut shell and sunflower husks agro-wastes by Barczewski *et al*. They found that the best mechanical properties were achieved with the addition of the hazelnut shell powder [11]. Nwigbo *et al*. studied the mechanical properties of castor seed shell-polyester composites, and reported that the inclusion of the filler added strength to the composites [12]. Ogah *et al*. studied the effect of immersion temperature on the mechanical, water absorption and morphological properties of sodium hydroxide modified fluted stem fiber reinforced polyester biocomposites. They found that the highest improvement in tensile, flexural, compressive and impact strength was obtained at 30wt% filler loading and temperature of 45ºC [13]. Effect of nanoclay on combustion, mechanical and morphological properties of recycled high-density polyethylene/marula seed cake/organo-modified montmorillonite nanocomposites was reported by Ogah *et al*. [14]. The mechanical and morphological properties of fluted pumpkin stem fiber (Telfaira occidentalis) recycled high density polyethylene nanocomposites were reported by Ogah *et al*. [15]. Physical and mechanical properties of agro-waste filled recycled high density polyethylene bio-composites were investigated by Ogah *et al*. [16]. Ogah and Timothy studied the mechanical behavior of agricultural waste fibers reinforced vinyl ester bio-composites [17]. The physicomechanical properties of agro waste filled high density polyethylene bio-composites were reported by Ogah and Chukwujike [18]. Anselm Ogah studied the characterization of sorghum bran/recycled low density polyethylene for the manufacturing of polymer composites [19]. The physicomechanical properties of groundnut shell flour-filled maleic acid anhydride compatibilized natural rubber (NR)/low-density polyethylene blends were investigated by Ogah and Onwu [20]. The characterization and comparison of mechanical behavior of agro fiber filled high-density polyethylene bio-composites were studied by Ogah and Afiukwa [21].

A water absorption test is a fundamental test conducted to assess the porosity and permeability of composites. This test proffers valuable information about the composite's ability to absorb and retain water which is essential for understanding its durability, structural integrity, and performance in various environments. Thickness swelling (TS) is an important property that indicates the stability performance of the composites. Generally, the swelling rates of polymer matrix composites are low during the initial stages of moisture absorption due to the visco-elasticity of the polymer matrix. Thickness swelling refers to the increase in thickness of a material such as wood particle-polypropylene composite when it absorbs water. The water absorption, thickness swelling and rheological properties of agro fiber/HDPE composites were investigated by Ogah *et al.* [22].

The use of fillers with percentage limits (0, 5, 10, 15, 20, 25, 30 and 35) in composites has been reported by Eze *et al*. [23]. They found that mechanical properties of composites were greatly influenced by the volume fraction of bambara nut shell filler in the epoxy matrix. In their study, the optimal weight percentage of filler for achieving the best mechanical properties in the composites was attained at 15wt%. They observed a decrease in the mechanical properties of the composites above 15wt% up to 35wt% filler loading. Apart from the study on adding natural fibers (castor bean shell) into epoxy resin matrix to develop green composite materials, several other researches use banana fiber reinforcement for other polymers, usually poly (lactic acid), with varying concentrations: 10%, 20%, and 30% weight [24]; 20, 40, and 60wt% [25]; and 10, 20, 30, and 40% by weight [26].

These studies demonstrate the utilization of biomass wastes as fillers with polymer matrix composites and suggest that they could be used in a variety of applications. Therefore, further studies are needed to evaluate properties and performance of these composites. While there are no studies specifically investigating the development of green composites based on castor bean shell as filler in epoxy resin matrix polymer. There is the possibility that similar results could be obtained if castor bean shell particles were added to the epoxy matrix polymer. The main aim of this research was to study the effect of waste castor bean shell filler content on the tensile strength, tensile modulus, flexural strength, impact strength, hardness, density, water absorption and thickness swelling properties of the epoxy matrix. This may create a new opportunity for the beneficiation of epoxy and waste castor bean shell for high-value products. In the epoxy matrix, castor bean shell can be utilized as filler, which will lower costs and improve the environment.

2. Methods

2.1. Materials

The waste castor bean shell was collected from a dumpsite located at Awka metroplolis, Anambra State. Forward Response Technology Limited provided the epoxy resin (Epotech 2005004) and the hardener (Epotech 105003) and were used as received.

2.2. Processing of Castor bean shell

The castor bean shells were washed with distilled water to remove impurities and other unwanted materials. Thereafter, they were sun dried for three days and ground to powder using a grinder. The powdered castor bean shells were passed through 75 µm mesh sieve. They were subsequently oven dried at 100ºC for 24 h to gain humidity content of <2%, and bagged in an airtight black polythene bag to avoid moisture absorption prior to further processing. Figure 1, Figure 2 and Figure 3 represents the castor bean, the castor bean shells and the castor bean shell powder respectively.

Figure 1. Castor bean

Figure 2. Castor bean shell

Figure 3. Castor bean shell powder

2.3. Bio-composite production

The castor bean shell powder of varying contents of 5, 10, 15, 20, 25, 30 and 35 wt% were mixed with a corresponding wt% of epoxy resin. After thorough mixing, the hardener was added to the epoxy/filler mix at a ratio of 2:1, Table 1, Table 2. Subsequently, the epoxy/hardener/filler mixes were poured into metal molds (180x180x5mm dimensions), smeared with paraffin wax as a mold release agent to prevent the composites from sticking to the mold. The composites were allowed to cure for 24h at room temperature before de-molding.

Density of the epoxy resin (ρ) = 1.1 g/cm^3

Volume of the mold (V) = 180 x 180 x 5 mm = 162000mm^3/1000 = 162 cm^3

Mass of the epoxy resin = ρ x V = 1.1g/cm^3 x 162cm^3 = 178.2 = 200g

Table1. Composite formulation

Composite code	Castor bean shell powder (wt %)	Epoxy resin/hardener (wt %)
A	0	100
B	5	95
C	10	90
D	15	85
E	20	80
F	25	75
G	30	70
H	35	65

Table 2. Formulation of castor bean shell filler, epoxy resin and hardener composites

Sample code	Castor bean shell filler (wt %)	Epoxy loading (wt %)	Mass castor bean shell filler (g)	Mass epoxy (g)	Ratio (2:1)		Total mass (g)
					Epoxy (g)	hardener (g)	
A	0	100	0	200	133	67	200
B	5	95	10	190	127	63	200
C	10	90	20	180	120	60	200
D	15	85	30	170	113	57	200
E	20	80	40	160	107	53	200
F	25	75	50	150	100	50	200
G	30	70	60	140	93	47	200
H	35	65	70	130	87	43	200

2.4.1. Density of the composites

The ASTM D2395-17 (2022) [27] was applied to determine the density of the composites. Three replicates of each composite (A-H) with the dimensions of 180 x 180 x 5 mm were weighed on a digital weighing balance (Mettler 5000) at the Polymer Engineering Laboratory, Nnamdi Azikiwe University, Awka, Nigeria. The densities of the composites were calculated according to equation 1:

$$\text{Density, } (\rho) = \text{mass (g)/volume (cm}^3) \qquad 1$$

2.4.2. The water absorption test

The percentage water absorption of the composites was determined according to the ASTM D570-98 [28]. Three replicates of each sample (A-H) with the dimensions of 50 x 40 x 5 mm were cut, weighed and then immersed in distilled water. The composites were removed from the water and cleaned with a clean white cloth every 24 h for a period of 5 days. The percentage water absorption of the composites was calculated using equation 2.

Percentage water absorption = (w-d)/d x 100 2

Where d and w are the weights of the dry and the wet samples respectively.

2.4.3. The thickness swelling test

The percentage thickness swelling of the composites was determined following the DIN EN 317 [29]. The thickness of each original sample was first measured and recorded using a micrometer screw-gauge. Then, the samples were soaked in distilled water. The samples were removed from the water and cleaned with a clean white cloth every 24 h for a period of 5 days. The thickness of the soaked samples were then measured and the percentage of thickness swelling was calculated using equation 3.

Percentage thickness swelling = $(t-t_o)/t_o$ x 100 3

Where t_o and t are the thickness of the dry and the wet samples respectively.

2.4.4 Mechanical properties

2.4.4.1 Tensile properties

The tensile strength and tensile modulus of the composites samples (A-H) was determined according to the ASTM D638-10 [30]. Tensile test samples was cut and stored for 48 hours at a temperature of 23±2ºC and relative humidity of 50±5%. Test was carried out on a Universal Instron Testing Machine (Model 4466) at a crosshead speed of 5 mm/min and a load cell of 15 kN.

2.4.4.2 Flexural strength

The flexural strength of the composites samples (A-H) was conducted according to the ASTM D7264M-07 [31]. Flexural test samples measuring 100 x 35 x 10 mm was cut and stored for 48 hours at a temperature of 23±2ºC and relative humidity of 50±5%. Test was carried out on a Universal Instron Testing Machine (Model 4466) at a crosshead speed of 5 mm/min and a load cell of 8.5 kN.

2.4.4.3 Impact strength

The Un-notched Izod impact strength of the composites samples (A-H) was conducted according to ASTM D256-10 [32]. Test samples measuring 60 x 12 x 5 mm was cut and stored for 48 h at a temperature of 23±2ºC and relative humidity of 50±5%. The test was carried out using a pendulum impact tester.

2.4.4.4 Hardness

The hardness of each composite sample was determined according to ASTM-D2240 [33] utilizing the Durometer hardness tester model number 5019 on Shore A scale.

2.4.4.5 Scanning Electron Microscopy

To confirm the surface morphology of the composite materials, (SEM) investigations were performed using a JSM 6400 SEM (JEOL Ltd., Akishima, Tokyo, Japan). For the composite samples, the requisite condition is that they must be moisture free. The samples were mounted onto a substrate with a conductive adhesive. Coating with a thin film of conducting materials is foremost requirement for all non-conducting samples to analyze in SEM. The image results were evaluated to study the distribution of the castor bean shell bio-filler and their interactions within the epoxy resin polymer matrix. The samples were coated with gold by vacuum evaporation with the acceleration voltage of 5-8 kV.

3. Results and discussion

3.1. Density

Figure 4 shows the variation of density with castor bean shell filler loading. The control sample A (pure epoxy with hardener without castor bean shell filler) gave the highest density of 1.21 g cm^{-3}. The addition of 5wt% castor bean shell filler content gave a density value of 1.19 g cm^{-3}. The addition of 35 wt% castor bean shell filler content gave a density value of 1.07 g cm^{-3}. From the figure it can be observed that the density of the composites with the addition of castor bean shell filler decreased. The porous nature of the filler might have reduced the densities of the composites, making them lighter with the increasing filler contents [15], [16], [34]-[36].

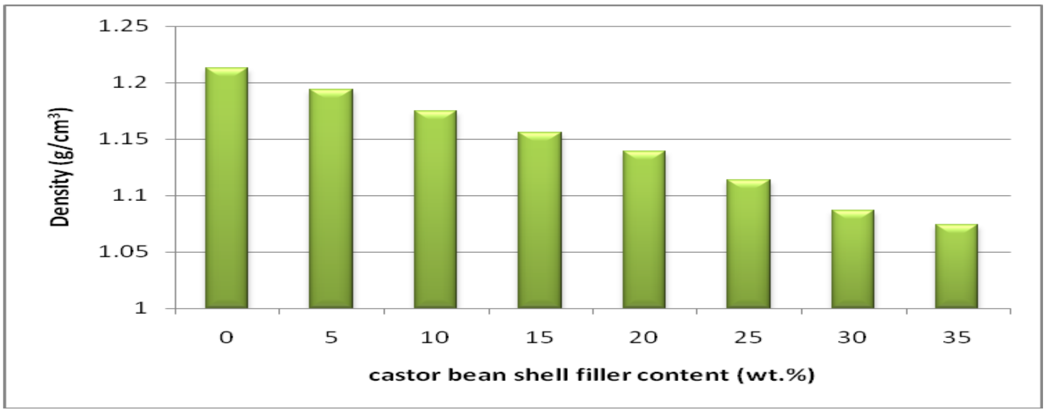

Figure 4. Density of the castor bean shell filler/epoxy composites

3.2 Water absorption

The results of Figure 5 showed increasing percentage of water absorption of the composites with the increasing castor bean shell filler content from 0 to 35wt% for the immersion period of 4 days. The sample A (containing 0wt% filler loading) gave the lowest water absorption of 6%. This was attributed to the viscoelasticity of the epoxy resin polymer matrix. The sample B (containing 5wt% filler loading) gave water absorption of 0.9%, and the sample H (containing 35wt% filler loading) gave water absorption of 2.4%. The increase in the percentage water absorption of the composites with the increasing castor bean shell filler content is due to the hydrophilic nature and high surface area of the filler [15], [16]. The results show that water absorption is related to the epoxy resin content. As this content decreases, the volume fraction of the filler increased and more water is absorbed. As epoxy content decreased, less amount of the epoxy was available to wet the filler, resulting in more substantial filler water absorption, causing to increase the water absorption percentage of the composite [37].

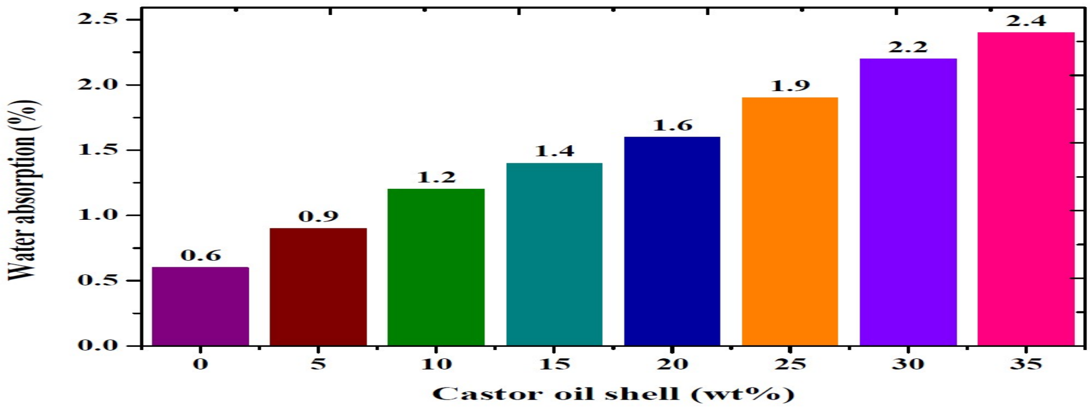

Figure 5. Water absorption of the castor bean shell/epoxy composites

3.3 Thickness swelling

A thickness of swelling test was conducted on the reinforced samples composites to analyze the changes in dimensional stability of the composites. Figure 6 shows the increase in the thickness swelling of the composites with the increasing filler loading from 0-35wt%. The results showed that the swelling rates of the castor bean shell filler epoxy resin polymer matrix composites are low during the initial stages of moisture absorption due to the visco-elasticity of the polymer matrix. It can be seen that with the decrease of the epoxy weight percentage content the thickness swelling of the composites increase, where composites with 35% filler showed the highest values [39], [40].

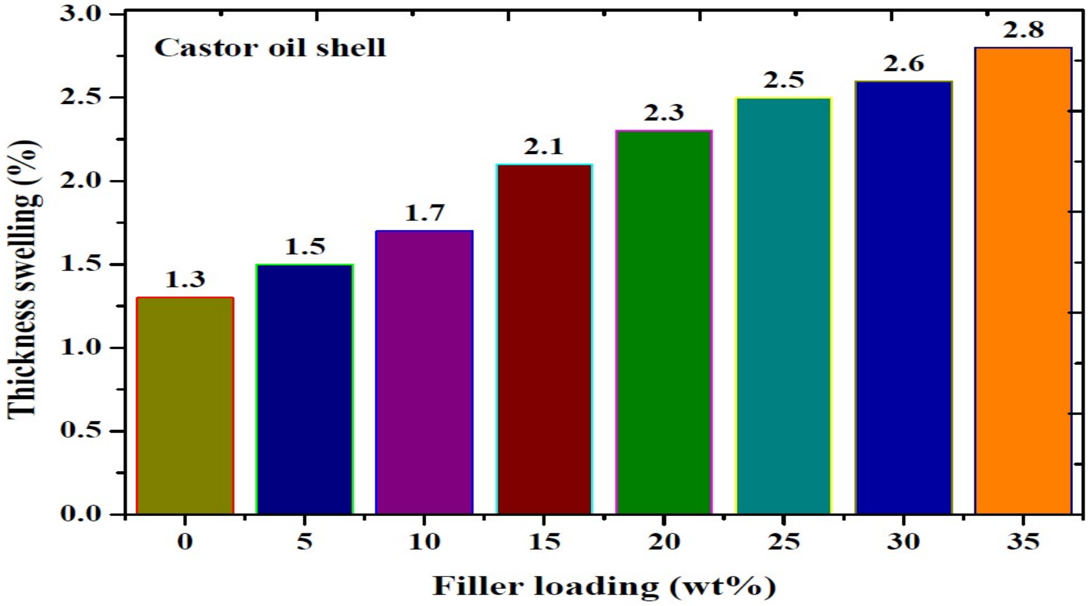

Figure 6. Thickness swelling of the castor bean shell/epoxy composites

3.4. Mechanical properties

3.4.1Tensile strength

Figure 7 shows the result of the tensile strength of the castor bean shell/epoxy composites at varying filler contents. The tensile strength of the control sample A was found to be 20MPa. The additions of the castor bean shell filler into the epoxy matrix increased tensile strength from 25MPa for 5wt% to 30MPa for 10 wt% and to 50MPa for 15wt% filler loadings respectively. But, there was a decrease in the tensile strength of the composites beyond the 15wt% filler loading. The increase in tensile strength was due to the polymer-filler interaction. The decrease in tensile strength was due to the filler-filler interaction and the inability of the epoxy resin to wet the filler at the increased filler content [41]-[43].

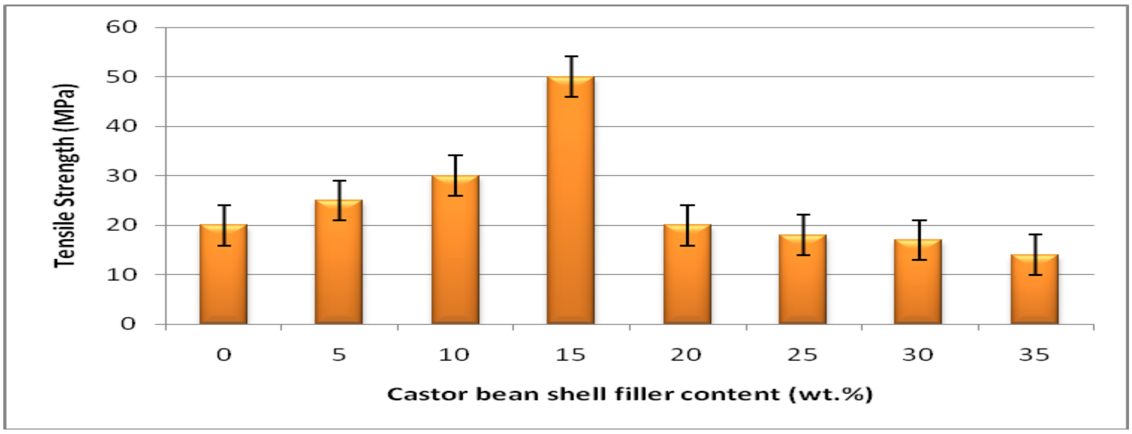

Figure 7. Tensile strength of the castor bean shell/epoxy composite

3.4.2 Tensile modulus

Figure 8 shows the tensile modulus of the castor bean shell/epoxy composites. The control sample A gave a tensile modulus of 99MPa. The 5 wt% castor bean shell filler gave a tensile modulus of 110MPa. At 15 wt% castor bean shell filler loading, the composite gave the highest tensile modulus of 175MPa indicating the optimal filler content for the composites. The increased filler loading from 15-35 wt% caused a decrease in the tensile modulus due to inefficient dispersion of the filler in the epoxy matrix [37], [38], [44]

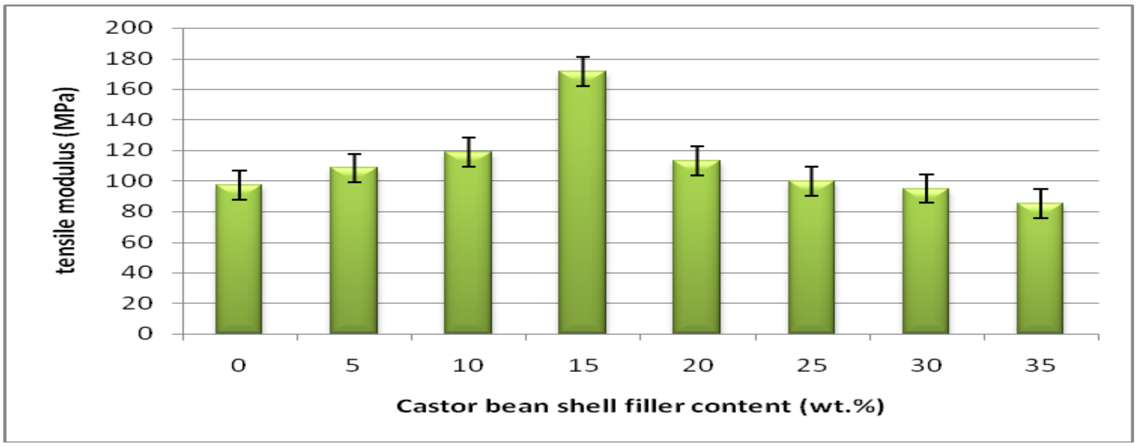

Figure 8. Tensile modulus of the castor bean shell/epoxy composite

3.4.3 Flexural strength

Figure 9 shows the variation of the flexural strength against the castor bean shell filler loading/epoxy composites. The graph showed that the flexural strength increased with the increase in the filler loading up to 15 wt% and decreased with increasing filler loading up to 35wt%. 15wt% castor bean shell filler gave the optimal flexural strength of 90MPa. The lowest flexural strength of 50MPa was recorded for the control sample A with zero castor bean shell filler loading [46], [47]. The fiber/matrix adhesion at the boundary and the composite homogeneity favor the load sharing, which is accompanied with the enhancement in the flexural strength values [45]. The variation in the mechanical behavior from the 100% epoxy is due to the fact the filler presents a stress/strain behavior similar to a brittle material and the polymer presents a behavior closer to an elastic material.

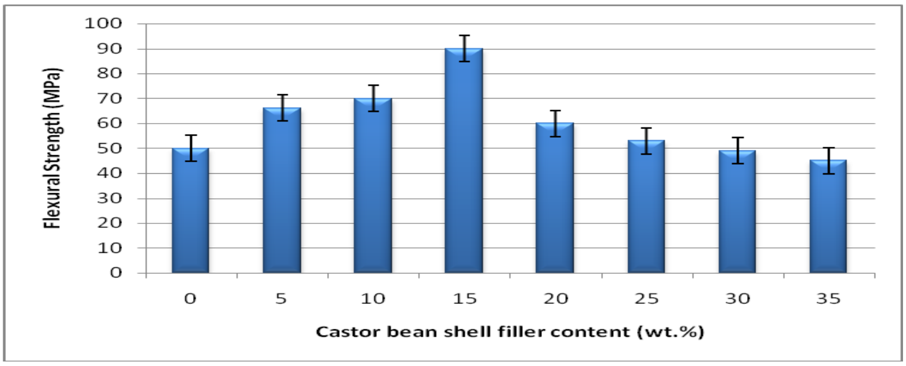

Figure 9. Flexural strength of the castor bean shell/epoxy composite

3.4.4 Impact strength

Figure 10 shows that the increase in the castor bean shell filler loading increased the impact strength. The 15wt% of castor bean shell filler loading gave the highest un-notched Izod impact strength of 70 J/m compared to the control sample A of 35J/m. However, beyond the 15 wt% filler loading, the epoxy resin could no longer wet the fillers due to filler agglomeration causing a decrease in the impact strength. The presence of filler agglomeration beyond the 15wt% caused weak interfacial adhesion between the castor bean shell filler and the epoxy matrix resulting in voids and crack propagation [38].

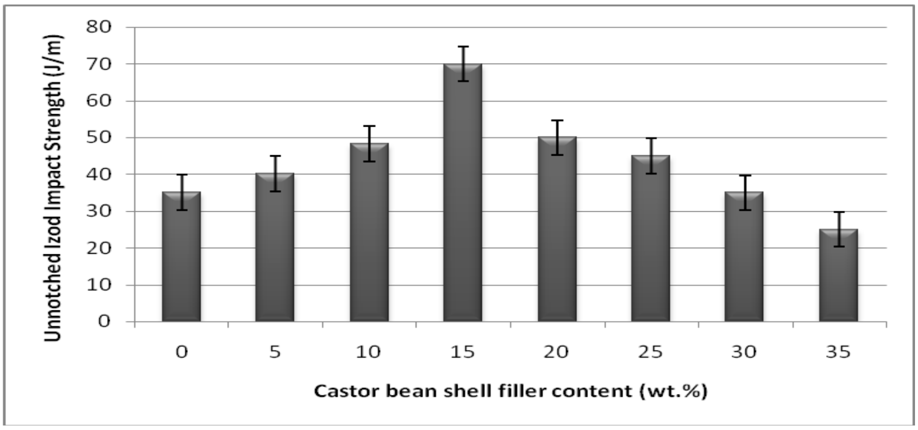

Figure10. Impact strength of the castor bean shell/epoxy composite

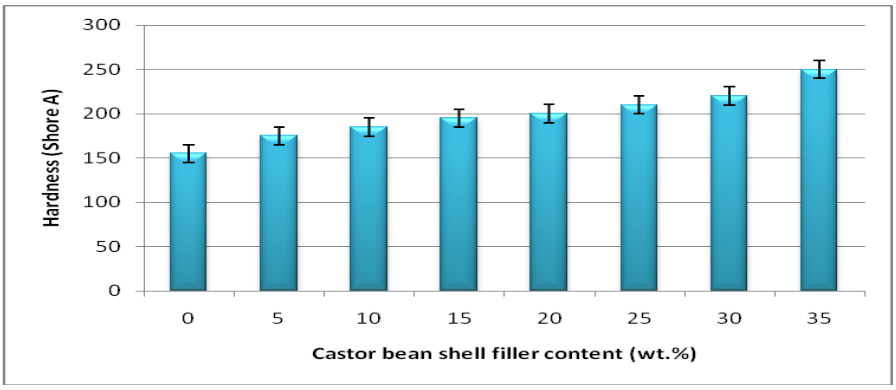

Figure 11. Hardness of the castor bean shell/epoxy composite

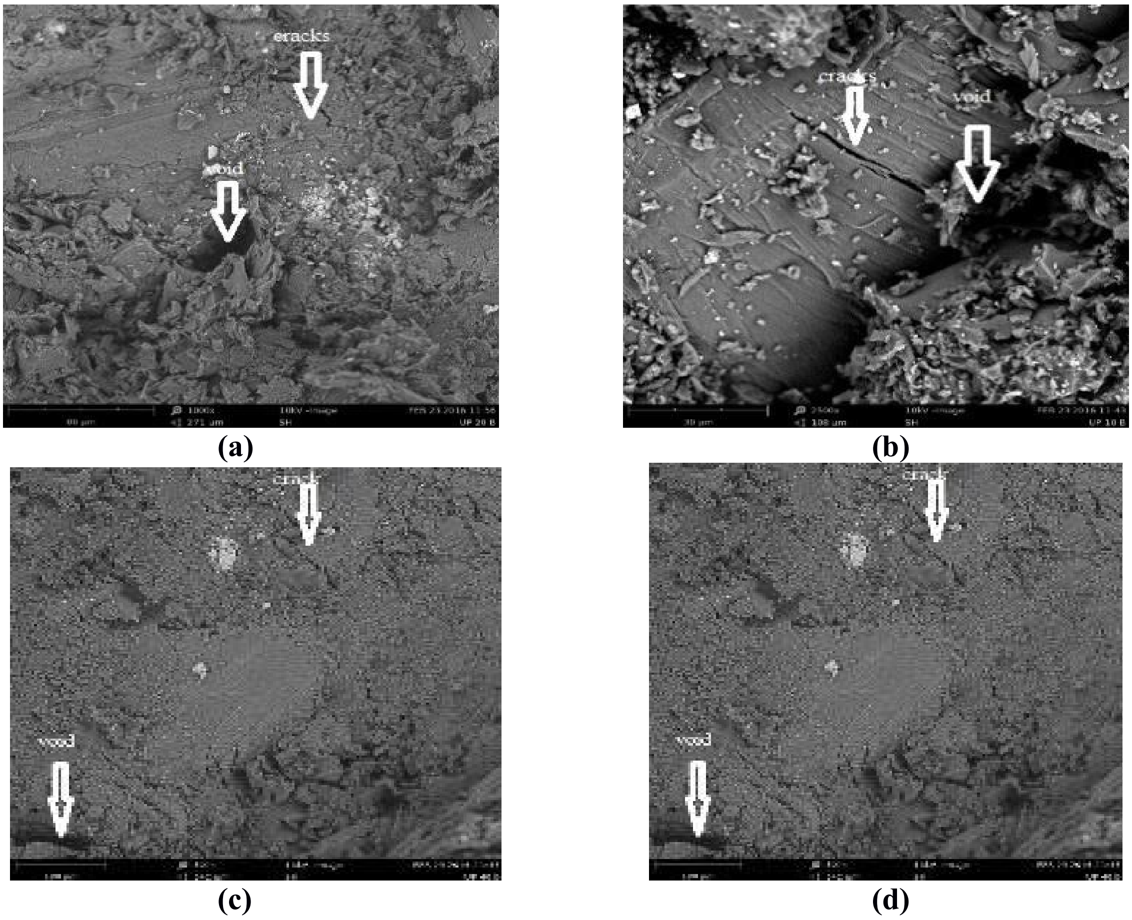

Figure 12. Fractographs of CBS/epoxy composites @ 150X for (a) 5 wt% (b) 10 wt% (c) 15 wt% and (d) 20 wt% filler content

3.4.5 Hardness

Figure 11 shows an increase in the hardness of the composites as the castor bean shell filler loading is increased from 0-35wt%. It was observed that 35 wt% filler loading exhibited the maximum hardness value of 225. This is attributable to the increase in the castor bean shell filler loading which is more resistant compared to the unfilled epoxy resin matrix [48]. Moreover, the agglomeration of filler particles at higher filler loading could withstand the hollow composites leading to the improved hardness of the composite [49]. Composite hardness increases with increasing filler percentage, but other mechanical properties only increase up to a filler percentage of 15wt percent, more than 15wt% decreases. This happened because hardness is a measure of how hard the surface of a material is. Generally, the shore a hardness of the composites increases as the filler content increased [49]. There is no relationship between hardness and other mechanical properties.

3.4.6 Morphology Study

Figure 12 shows the micrographs of the fractured surface of the composites after tensile test. In Figure 12 (c) pulling out of the filler, cracks, voids and filler agglomeration was reduced for the 15wt% castor bean shell content. The castor bean shell particles were well dispersed and adhered with the epoxy matrix resulting in higher interaction between them. These findings are evidence of the good interfacial adhesion between the filler and the matrix which leads to improved tensile, flexural, impact and hardness properties.

3. Conclusion

In this research, green composites were developed by combining epoxy polymer matrix/hardener with castor bean shell as a reinforcing filler to investigate the physical, mechanical, and morphological properties of the castor bean shell/epoxy composites. The composites fabricated by varying the weight fractions of filler in the range of 5 to 35 wt%. The effects of bio-filler content of the composites on tensile, flexural, impact and hardness properties were evaluated by universal testing machine, hardness tester and scanning electron microscopy (SEM). The study revealed that the tensile strength, tensile modulus, flexural strength and impact strength increased with the increasing of the castor bean shell filler content. The highest tensile, flexural and impact properties of the castor bean shell filler epoxy composite were achieved at the castor bean shell filler content of 15wt%. The hardness of the composites increased with the increasing of the castor bean shell filler content from 5-35wt%. The water absorption and the thickness swelling increased with the increasing of the castor bean shell filler content. Generally, the water absorption and thickness swelling of the castor bean shell filler epoxy polymer matrix composites were low during the initial stages of moisture absorption due to the visco-elasticity of the epoxy polymer matrix. The density decreased with the increasing of the castor bean shell filler content. SEM showed improved interfacial adhesion of the castor bean shell filler with epoxy resin matrix at 15wt% CBS content. The study has demonstrated that the castor bean shell is a viable reinforcement for manufacturing green composites from the viewpoint of their physical and mechanical properties.

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