

The Mechanical Properties of Castor Seed Shell-polyester Matrix Composites

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Abstract: A composite with a polyester matrix reinforced with chemically modified shells of castor seed (*Ricinus communis*) was produced. The effect of the shell (filler) on the mechanical properties of the composite was experimentally quantified. A preliminary study was earlier carried out the shell in terms of their chemical constituents, functional group and mechanical strength. The shell was ground and chemically treated to enhance good bonding and adhesion to the matrix. Composites were fabricated using a hand lay-up or contact mould method for different percentage compositions of the filler. Tests, with respect to the mechanical properties (i.e., tensile, flexural and creep response) were carried out. The result obtained was compared with the unreinforced polyester plate. It was observed that the inclusion of the filler (shell) added strength to the composite. Scanning Electron Microscopy (SEM) was taken on the composite samples to study the morphology of the composites.

Keywords: Creep, filler, mechanical strength, polyester, ricinus cumminus

INTRODUCTION

Since the discovery of composites, it has opened new areas of study which include micro and macro-scale reinforcements in composites in order to improve their mechanical, electrical, thermal, tensile, chemical resistance, solvent permeability.

Properties: Composites materials consisting of polymeric matrix and fibres/ particles have attracted scientific and industrial interest due to their improved properties. They exhibit superior property enhancement at low filler contents. Different types of polymer and fillers have been used so far to prepare polymer matrix composites. In structural and strength of material analysis the quest and demand for the substitution and replacement of heavy-weight material with lighter-weight and tougher materials which possess the same mechanical strength and properties has become a major concern. Polymer when mixed with some other materials exhibits improved mechanical, thermal and tensile strength. Polymer matrix composites have found a wide application in mechanical and structural engineering and the development of this technology has been of important research interest in the international literature. Choi *et al.* (2006) developed a rice-husks-plastics composite for building materials. Maheswari *et al.* (2008) considered the tensile and thermal degradation parameters of Tamarind fruit fibres. Oil palm fruit bunches has been successfully used to reinforce in new unsaturated Polyester composite (Khali *et al.*, 2008). Several studies have been carried

out on other fibres as reinforcement in polymeric composite production. Uma *et al.* (1997) studied the Mechanical Properties of Pineapple Leaf Fibre Reinforced Polyester composites. The Effect of filler content on the mechanical Properties of Polypropylene/oil Palm wood composite was also studied (Zaini *et al.*, 1996). The creep properties of some composite materials were studied at room temperature (Ericksen *et al.*, 1976).

In this study, castor seed (*Ricinus communis*) was used as reinforcement in a polymer (polyester) matrix composite to improve toughness strength and mechanical properties. In its nature as a plant, Ricinus is termed as the most poisonous plant. due to the presence of ricin in the crop (<http://www.wikipedia.org/wiki/Castor-oil-plant>, 2012).

Surprisingly, this compound is not found in the shell of the plant's seed (Soto *et al.*, 2002).

Of the ten top castor oil seed producers in the world, India has the highest production capacity of 830000 tonnes. Followed by China at 210000 tonnes. These are good figures when compared with the total world's production of 1209756 tonnes. (Source: Food and Agricultural Organization of the United Nations: Economic and Social Department: Statistical Division). Modern technology has also found the product good enough for biodiesel production (<http://www.wikipedia.org/wiki/Castor-oil-plant>, 2012).

In Nigeria, when processed, it serves as food and spice. The seed of the plant used amounts to tonnes of solid waste as they are not always properly utilised. This formed one of the major motivations to this study.

Table 1: XRF result of chemical compositions of the shells

Element	Si (%)	Ca (%)	Fe (%)	K(%)	Mn (Ppm)	Cr (Ppm)	Cu (Ppm)	V (Ppm)	P (Ppm)	Co (Ppm)
Conc.	2.69	8.67	0.22	5.49	150	75	11	4.64	933	3.16

Table 2: Apparent porosity and bulk density values

Specimen/samples	D (g)	W (g)	S (g)	P (app) (%)	Bd (g/cm ³)
<i>Ricinus communis</i>	0.65	0.70	0.11	8.48	1.10

Table 3: Hardness test values

Specimen/sample	1 st reading (HRA)	2 nd reading (HRA)	3 rd reading (HRA)	Average (HRA)
<i>Ricinus communis</i>	20.40	19.60	20.70	20.20



Fig. 1: Castor oil seed shell



Fig. 2: Ground castor oil seed shell

Preliminary studies ricinus communis shell: Castor oil seed is a seed of a castor oil plant which belongs to the family of “plantae” known as shell shown in Fig. 1. The shell when ground shows a brownish dust impigmented with some *Euphorbiaceae*. Its botanical or biological name is *Ricinus communis*. The seed is covered by black patches as in Fig. 2.

In this research study, the shell of the seed was used as a filler material in polyester matrix composites. The chemical composition of the shell investigated by X-ray fluorescent spectrometer XRF analysis is presented in Table 1 and the mechanical properties (hardness, bulk density, and apparent porosity) determined is presented in Table 2 and 3.

Chemical composition of the shell: The chemical composition of the shell material was done on a mini pal compact energy dispersive x-ray spectrometer. The sample (s) for the analysis was weighed and grounded in an agate mortar and pressed in a hydraulic press into pellet (s). The pellet (s) loaded in the sample chamber of the spectrometer and a voltage of 30KV and current of 1mA was applied to the X-rays to excite the sample for 10 min. The spectrums from the sample (s) were analyzed to determine the concentrations of the elements in the sample (s). The result is shown in Table 1.

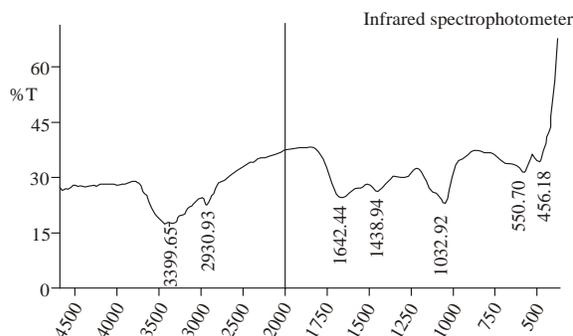


Fig. 3: FTIR diagram of ricinus communis

Mechanical property of castor oil bean shell: Some of the mechanical properties of the shells such as apparent porosity, bulk density and hardness values were determined. The values apparent porosity and bulk density are as in Table 2. The apparent porosity is then calculated as:

$$P (app) = \frac{W-D}{W-S} \times \frac{100}{1}$$

$$P (app) = \frac{\text{actual volume of open pores of specimen}}{\text{external volume of the specimen}} \times \frac{100}{1}$$

where,

W-D = Actual volume of open pores of the specimen

W-S = External volume of the specimen

$$\text{Bulk density (Bd)} = \frac{D}{W-S} (\text{g/cm}^3)$$

where,

D = Dried weight

W = soaked weight

S = suspended weight

HARDNESS VALUES

The hardness values of the sample were determined according to the provisions in ASTM E 18-79 using the Rockwell hardness testers on “A” scale (Frank Welltest Rockwell Hardness Tester) (model 38506) with 1.56mm steel ball indenter, minor load of 10kg, major load of 60kg and hardness values of 819 HRA as the standard block. The results are shown in Table 3.

The values in major peaks of the spectrum in Fig. 3 which was considered at 1002.92 cm showed a single bond anti symmetrically coupled carbon to oxygen

stretch (C-O to C-C). Apart from these absorption band, there is also recorded, the presence of hydroxyl stretch at 3399.05 cm.

MATERIALS AND METHODS

The unsaturated polyester resin used was supplied by JUNENG Nigeria Limited Enugu with specifications; NYCIL 6043 and modulus of 3.25GM and density of 1350 kg/m³

Treatment of the seed shell powder: The grounded samples of the shells were chemically treated for property enhancement. The treatment was done in two stages:

Firstly with 5% sodium hydroxide (NaOH) Solution and second with a mixture of alcohol (ethanol) and water in the ratio of 60:40. The grounded shells were each mixed with the solution, stirred and kept for 6 hours at room temperature and later decanted and the deproteinised precipitate was washed with distilled water. Again, the samples were then washed with a dilute solution of acetic acid (i.e., glycerol) and oven dried at 70-80°C. This was done in order to remove the alkaline effect of the sodium hydroxide (NaOH) solution

Moulding of composites: The composite slabs were prepared by hand lay-up method. A metallic mould 40cm x 15cm and a depth of 0.3cm was prepared and used for the production of the specimens. A release agent was applied on the mould surface to avoid sticking of the composites in the mould. Polyester resin (NYCIL 6043) belonging to ester family was used as the matrix material, Methylethylketone Peroxide (MEKP) introduced as the catalyst and cobalt derivative accelerator (CDA) was used as the hardner. The polyester resin (modules of 3.25GP and density 1350kg/m³) MEKP and Cobalt Derivative Accelerator (CDA) were supplied by Juneng Nig Ltd Enugu. Polyester resin and the hardener were mixed in the ratio of 10:1 by volume and the catalyst methylethylketone peroxide added to effect the chemical reaction. Composites of three different compositions such as 10 wt%, 20 wt% and 30 wt% of filler content were produced. Also a control specimen was produced without reinforcement. The castings produced were left for 24 hours at room temperature for curing. Specimens of suitable dimension were cut using a diamond cutter and saw for property characterization.

Tensile strength test: Tensile test was carried out on each specimen. This was done according to ASTM D 638-90 with the aid of Hounsfield (Monsato) tensometer with a load cell or beam strength of 5KN. Dog- bone shaped tensile specimens were prepared with the dimensions 200 mm×20 mm×3 mm. The tests

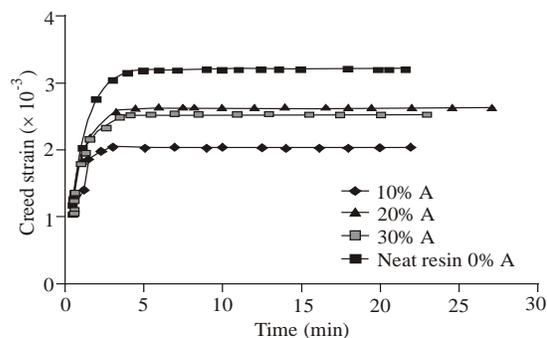


Fig. 4: Creep strain against time for different percentage particulate loading

were conducted at room temperature. Four Specimens were tested for each sample combinations. The measured stroke values versus the load data were used to calculate the tensile stress and tensile strain.

Flexural test: Flexural test was done according to ASTM D 790. Rectangular specimens were cut and loaded under three-point loading configuration. The specimen's dimensions were 40 cm x 2cm x 0.3 cm. The beam strength was 0.615 KN and the test was carried out at room temperature.

CREEP TEST

The creep test was performed according to ASTM D 2990 standard. This was done using the three-point loading system and the experiment was done at room temperature. A dog -bone shape of the produced specimens were cut from the sample composite. The dimensions of the test specimens were 150mm×20mm×3mm. During the test, a span or gauge Length (L) of 120mm was selected on the test Rigs. The sensitivity of the test rig was 0.01mm, mass of hanger = 0.3kg and the Dial Test Indicator (DTI) readings were taken. The results are shown in Fig. 4. The cross-sectional area of the specimens was given as 2.4×10³/m². The applied load/tension pull = 53.9N.

SCANNING ELECTRON MICROSCOPY (SEM)

The Scanning Electron Microscopy (SEM) was done to investigate the fracture surfaces and morphology of the composites. This was done with an EVO/MA10 microscope with a tungsten filament and an accelerating voltage of 20KV. The samples were held in the scanning chamber with double sided conductive carbon tape. The SEM micrographs were taken at the magnification level of 2.00 KX. To enhance the conductivity of the samples and flow of electrons, the scanning chamber was vacuum-evaporated before the photomicrographs were taken.

RESULTS AND DISCUSSION

Tensile stress-strain behavior for composites and the unreinforced polyester slab is shown in Table 4.

With the addition of filler content, the tensile strength increases and the composite show an improved strength compared to control counterpart. This was due to the hard and rigid fillers that restrict the elongation and hence decreases the deformation. The addition of 10wt% filler did not significantly increase the tensile strength. This may be due to of poor interfacial bonding between the filler and the matrix. With 20wt% filler, the strength showed an increment of 26%, whereas for 30wt% filler, the tensile strength was about 49% higher than the unreinforced polyester resin.

From Table 5, it can be seen that flexural strength decreases with the increase in filler loading.

Addition of the filler resulted in the stiffening of the composite and thus reduced the bending properties At 10wt% filler, the flexural strength

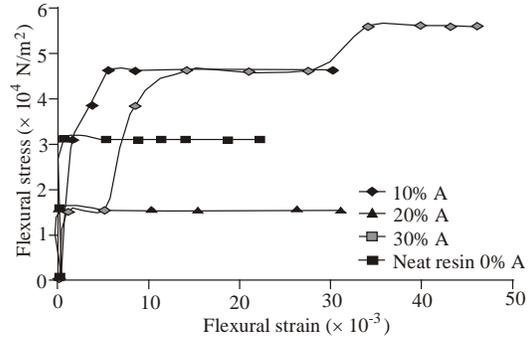


Fig. 5: Stress v/s strain curve for different percentage composition of particulate

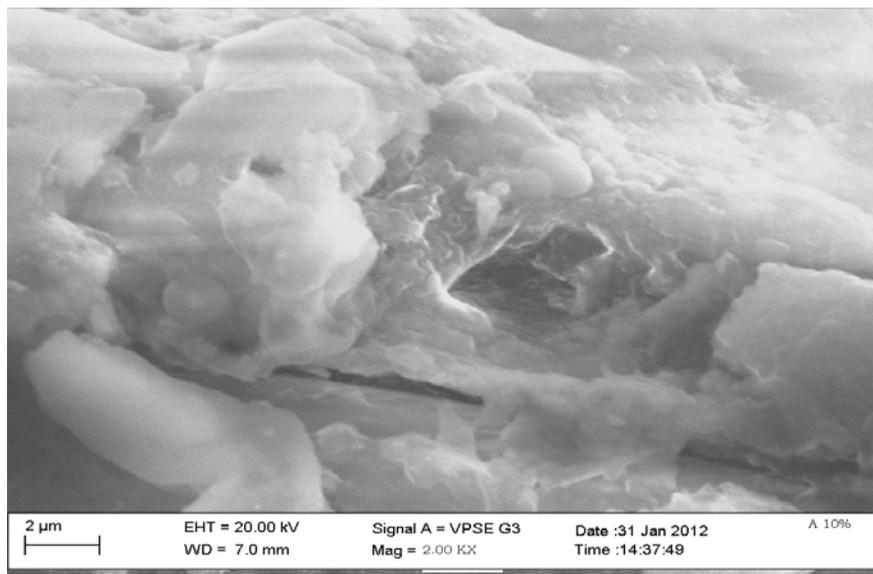
was 17.6% lower than the value of its unreinforced counterpart, at 20wt% filler; it was 44.8% lower and reached its minimum at 30wt% filler loading, which is about 72.4% lower than the unreinforced counterpart. Figure 5 also showed the stress-strain behavior of the composite under flexural load. The curves agreed with

Table 4: Tensile strength result for different % fibre composition

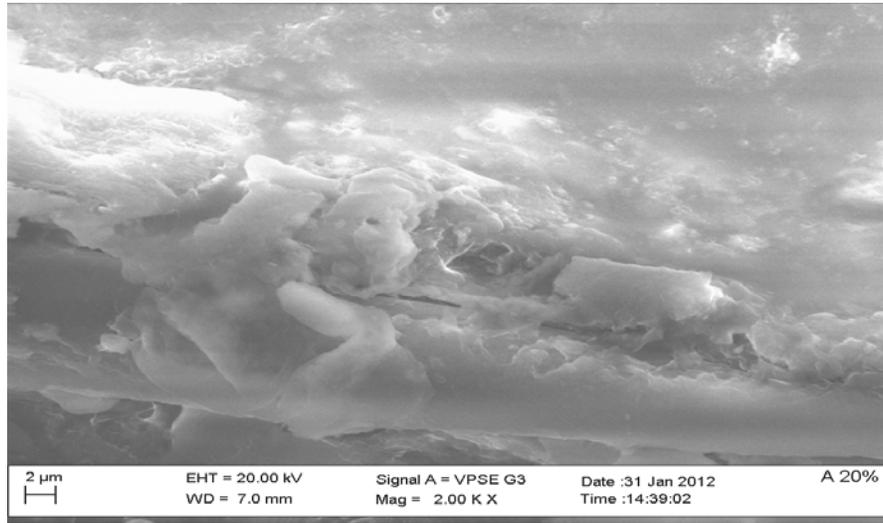
Filler content (%)	Maximum force (N)	Extension (mm)	Maximum tensile strength ($\times 10^4$) (N/m ²)	Maximum tensile strain ($\times 10^{-3}$)	Young modulus (E) ($\times 10^6$) (N/m ²)
0	475	3.375	15.83	22.50	7.04
10	275	1.25	9.17	8.33	11.01
20	600	3.375	20.00	22.50	8.89
30	750	4.375	25.00	29.20	8.56

Table 5: Flexural result for different % particulate composition

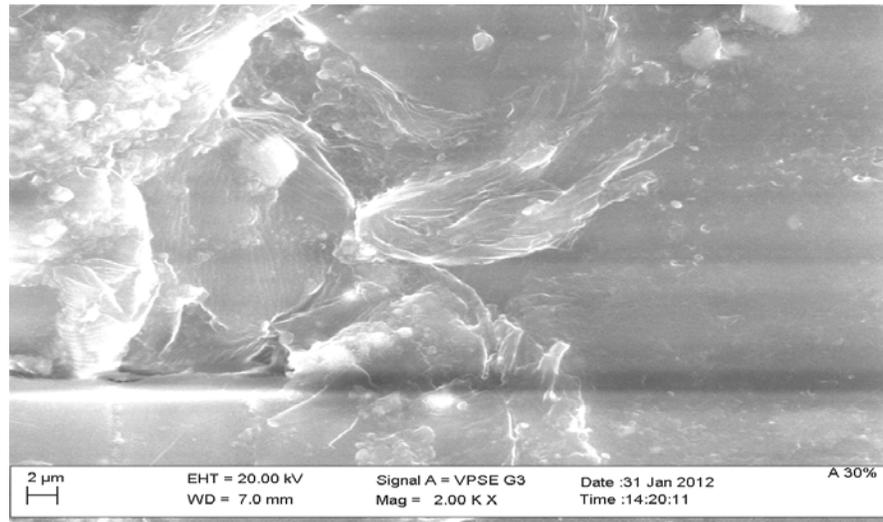
Filler content (%)	Maximum force (N)	Extension (mm)	Maximum flexural strength (10^4) (N/m ²)	Maximum flexural strain (10^{-3})	Young modulus E (10^6) (N/m ²)
0	22.30	22.30	5.58	46.00	12.13
10	18.45	15.00	4.60	30.00	15.33
20	12.30	11.00	3.08	22.00	14.00
30	6.15	15.50	1.54	31.00	4.97



(a)



(b)



(c)

Fig. 6: (a) SEM micrograph of the 10% filler composite, (b) SEM micrograph of the 20% filler composite, (c) SEM micrograph of 30% filler composite

the explained property of the material in Table 5. The composite with 30% filler content possessed the least value of the flexural modulus.

Figure 4 shows the creep response curve of the composites of the sample. The variation of creep strain ϵ (t) versus time (t) at different fiber concentration is plotted. It could be seen that as the fiber loading is increased, the time for the material to rupture or fail increased. Specimen loaded at 30wt% of the fiber experienced a longest period of creep. At 10wt% filler, there is 3.53% increase in the creeping time and at 30wt% fiber, the creeping time is 27% higher than that of the unreinforced counterpart. The increased creeping time of the composite was brought about by the

increase in the ultimate strength of composite which occurs as a result of the presence of the filler.

Figure 6a to c shows the morphology and fracture surfaces of back scattered images of 10wt%, 20wt% and 30wt% for both sample shells at 2.00KX magnification. It has been observed that fibre alignment factors play a crucial role on the overall properties of composites. There is always a chance of fibre entanglement with randomly orientated fiber reinforced composites.

The composites showed non uniform fractured surfaces as a result of failure. This occurred due to the branching of the cracks path as a result of the presence of filler.

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