Oil Bean Seed Shell as Reinforcement in New Polyester Composites

*Nwigbo S.C **Atuanya C.U *Okafor T.C.

*Mechanical Engineering Department, **Metallurgical and Materials Engineering, Nnamdi Azikiwe University Awka.

Received: June 26 2013
Accepted: August 16 2013

ABSTRACT
This work presents the production of composites with a polyester matrix reinforced with chemically modified shells of Oil bean seed (Penclethra macrophylla) shell. 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 (ie tensile, flexural and creep response) were carried out. The result obtained was compared with the unreinforced polyester plate at 30% filler loading, it showed a 79% improvement in tensile properties and 72.4% reduction in flexural strength. It was clearly 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.

KEY WORDS: Creep, Mechanical Strength, Pentaclethra Macrophylla, Polyester.

INTRODUCTION
Composites materials consisting of polymeric matrix materials and fibre particles have attracted scientific and industrial interest due to their improved properties. They exhibit superior property enhancement at low filler contents as compared with the counterparts, different types of polymer and fillers have been studied and used for preparation of polymeric composites: Organo clay in rubber (Gatos and Kocsis, 2005), Exopyo-clay (Park and Jana, 2003). Polypropylene have been used as polymers and reinforced by additions of silica, titanium dioxides, carbon filler particles, layered clays, fibre (Marchantk and Jayara, 2003; Choi et-al, 2006; Uma et-al, 1997). To produce more functional materials, different types of fillers can be used together to obtain diverse favourable properties from the composites.

The study on the nutritive value of the African Oil bean seed show that this is of reasonable proteenous ratio and protein calorie percent capable of satisfying human protein energy requirement (Isichei and Achinwehu, 1988; Nwamara and Madueke, 2010). Also is economic value of the seed. A 50kg bag of the seed cost approximately three thousand and sixty five naira and is yet to meet the demand in the African Local Market. In its processed form, it is used as snacks (Nwamara and Madueke, 2010).

In this work, Oil bean seed (Pentaclethra macrophylla) shell will be used as filler in a polyester matrix to improve the material’s inherent characteristics, especially; mechanical properties.

MATERIALS AND METHOD

Pentaclethra Macrophylla Shell
The oil seed plant is a plant that belongs to a family of plantae popularly known as Leguminosae and specie of Mimo soideae. Its botanical name is Pentaclethra macrophylla. The seed is covered by a pericarp or shell as shown in Figure 1. The shell when ground gives a dark brownish dust impigmented with black patches as in Figure 2. The chemical characterization was carried out by x-ray fluorescent spectrometer (XFR analysis) and the results shown in Table 1. Some other properties were also characterized and are presented in Tables 2 and 3.

Fig 1. Oil Bean Seed Shell
Table 1 XRF result of chemical compositions of the shells

<table>
<thead>
<tr>
<th>Element</th>
<th>Ca(%)</th>
<th>Fe(%)</th>
<th>K(%)</th>
<th>Mn (Ppm)</th>
<th>Cr (Ppm)</th>
<th>Cu (Ppm)</th>
<th>Co (Ppm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Concentration</td>
<td>1.58</td>
<td>0.18</td>
<td>9.46</td>
<td>112</td>
<td>53</td>
<td>6</td>
<td>1.32</td>
</tr>
</tbody>
</table>

Fig 2. Particulates of the Oil Bean Seed Shell.

Table 2: The Real Values of Apparent Porosity and Bulk Density

<table>
<thead>
<tr>
<th>Properties</th>
<th>D(g)</th>
<th>W(g)</th>
<th>S(g)</th>
<th>P_{app} (%)</th>
<th>Bd(g/cm^3)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Value</td>
<td>2.80</td>
<td>0.60</td>
<td>3.10</td>
<td>12.00</td>
<td>1.12</td>
</tr>
</tbody>
</table>

Table 3; Functional Groups of the Shell at Major Peaks

<table>
<thead>
<tr>
<th>Peaks (cm⁻¹)</th>
<th>Functional Group</th>
<th>% Transmittance</th>
</tr>
</thead>
<tbody>
<tr>
<td>3378.43</td>
<td>OH</td>
<td>13.48</td>
</tr>
<tr>
<td>1615.44</td>
<td>NH2</td>
<td>15.89</td>
</tr>
<tr>
<td>1447.62</td>
<td>COOH</td>
<td>17.36</td>
</tr>
<tr>
<td>1274.99</td>
<td>COOH</td>
<td>17.32</td>
</tr>
<tr>
<td>1057.99</td>
<td>C-O to C-C</td>
<td>16.84</td>
</tr>
<tr>
<td>488.01</td>
<td>Atomic (weak)</td>
<td>18.72</td>
</tr>
</tbody>
</table>

Determination of Apparent Porosity

The apparent porosity is then calculated as

\[
P_{\text{app}} = \frac{W-D}{W-S} \times \frac{100}{1}\times \frac{100}{1}
\]

Where \( W-D \) = actual volume of open pores of specimen

\( W-S \) = external volume of the specimen

Bulk density (Bd)= \( \frac{D}{W-S} \left( \frac{g}{cm^3} \right) \)

Where D = dried weight, W = Soaked weight, S = Suspended weight. The values obtained from the deductions are as in Table 2.

Hardness Value and Functional Group

The hardness value of the shell sample was determined according to the provisions in American society of Testing Materials (ASTME 18-19) using the Rockwell hardness testers on “A” scale (Frank Welltest Rockwell Hardness Tester model 38506). The result show that average value of 26 (HRA) was obtained. The result of the FTIR in Figure 3 showing the functional groups at visible peaks are tabulated in Table 3.
Figure 3. The FTIR of the Shell

Treatment of the Seed Shell Powder

The grounded seed shells were chemically treated firstly, with 5% sodium hydroxide (NaOH) solution and then a mixture of alcohol (ethanol) and water in the ratio of 60:40. The shell dust was deproteinised with the solution and washed with distilled water. The samples were then washed with a dilute solution of acetic acid (ie glycerol) and oven at 70-80°C.

Moulding of Composite

The composites slabs were prepared by hand lay up method or contact mould method.

A metallic mould was prepared and the dimension of the mould was 40cm x 15cm and a depth or thickness of 0.3 Polyester resin (NYCIL 6043) belonging to ester family was used as the matrix material, met hylethylketone peroxide (MEKP) introduced as the catalyst and combalt derivative accelerator (CDA) was used as the hardener. Polyester resin and the hardener were mixed in the ratio of 100:1 by volume and the catalyst methylethylketone peroxide added to effect the chemical reaction. Composites of three different fibre/filler compositions such as 10wt%, 20wt% and 30wt% and also a neat counterpart produced as control. The casting were left for 24 hours for proper curing at room temperature. Specimens of suitable dimension were cut for test.

Tensile, Flexural and Creep Test

Tensile test were carried out on each sample specimen. This was done according to ASTM D 638-90 with the aid of Hounsfield (Monsato) tensometer. Flexural strength was done according ASTMD 790. While the creep test was performed according to ASTM 2990 standard.

RESULTS

The results of the mechanical tensile test and flexural strength tests are presented in table 4 and figure 4, 5.

<table>
<thead>
<tr>
<th>Filler content (%)</th>
<th>Maximum Force (N)</th>
<th>Extension (mm)</th>
<th>Maximum Tensile Strength (x10^4) (N/m²)</th>
<th>Maximum Tensile Strain (x10^-3)</th>
<th>Young modulus (E) (x10^9) (N/m²)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>475</td>
<td>3.75</td>
<td>15.83</td>
<td>22.50</td>
<td>7.04</td>
</tr>
<tr>
<td>10</td>
<td>350</td>
<td>2.50</td>
<td>11.67</td>
<td>16.70</td>
<td>6.99</td>
</tr>
<tr>
<td>20</td>
<td>475</td>
<td>4.50</td>
<td>15.83</td>
<td>30.00</td>
<td>5.28</td>
</tr>
<tr>
<td>30</td>
<td>850</td>
<td>5.375</td>
<td>28.30</td>
<td>35.00</td>
<td>8.09</td>
</tr>
</tbody>
</table>
Figure 4: Flexural Stress vs Flexural Strain for Sample Shell

Figure 5: Creep Strain vs Time for Sample Shell
DISCUSSION

From Table 4, it could be seen that tensile strength increases with an increase in filler content. At 10wt% fiber loading, the tensile strength was lower to about 25% of the ultimate strength of the neat counterpart. With 20wt% fiber addition, the strength slowly increased, whereby at 30wt% fiber loading the tensile strength showed an increment of 79% higher than the neat counterpart. From observation, the optimum filler loading which yielded the highest tensile stress (strength) was at 30wt%.

These increase in tensile strength showed that the polyester composite with reinforcement were becoming stiffer and could withstand higher stress at the same strain portion. The Young modulus (E) of the composites was also increased by the addition of filler to the composite.

However, the tensile strain decreased following increased filler content. These improvements might be brought about by the chemical treatment of the shells which enhanced the fibre particle-matrix adhesion and good stress transfer was initiated.

The flexural strength of the composite also decreased with an increase in filler content as shown in Figure 4 and Table 5. It showed a minimum at 30wt% fibril content (72.4% lower than the unreinforced counterpart). The Young modulus or elastic modulus (E) was shown to be increased of the sample at low filler content (10wt%), but is reduced as the filler content is increased. This is due to high volume fraction of filler.

The time for the material to fail (creeping time) is also increased. This was seen to be maximum at 30wt% fibre content (ie 20% increment of its unreinforced counterpart). This is represented in figure 5. Thus, the reinforced composite can withstand stress concentrations for a longer time that the unreinforced composite (Ericksen, 1976). Figure 6(a-c) shows the morphology of 10wt%, 20wt%, and 30wt% of the sample shell at 2.00KX magnification. It was observed that figure alignment factor play a crucial role on the overall properties of the composite. There is also a chance of fibre entanglement with randomly orientated fiber reinforcement within the composites. The morphology of the composites has a well consolidated rich layer of filler particle. Though not explicitly marked out but some regions in fig 6c proved proper agglomeration to a large extent.

Figure 6a. Scanning Electron Micrograph for 10% Filler Content

![Figure 6a](image)

Figure 6b. Scanning Electron Micrograph for 20% Filler Content

![Figure 6b](image)
Conclusion
From the results obtained in the tests, it was established that the tensile properties of the composites produced with filler showed an appreciable improvement. When 30% filler content was studied, it was noted that up to 79% on strength when compared with the neat resin. There was also an increased Creep time with addition of filler in the composite.

Acknowledgment:
The authors declare that they have no conflicts of interest in this research.

REFERENCES