Investigation of the Impact, Hardness, Density and Water absorption of Polypropylene Filled Doum Palm Shell Particles Composite

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Abstract
Particulate fillers are used to improve the physical and mechanical properties of polymers to make it more suitable for engineering application. This study determined the impact, hardness and physical properties of polypropylene reinforced Doum palm shell particles composite; the effect of particles loading and particle size on the impact, hardness, density and water absorption properties of the composite were studied. The composites were prepared by compounding polypropylene and Doum palm shell particles using compression moulding method. The particles loading in the matrix (polypropylene) were varied from 0 - 40 wt. % at 5 wt. % intervals, after which the composites were characterized. SEM analysis was also conducted on the composites. The results showed that the addition of Doum palm shell particles stiffened the flexibility of the polymer and improved its ability to absorb and dissipate energy. Composite of 150 µm has the maximum impact strength of 4.1 kJ/m² at 35 wt. % particles loading and 3.9 kJ/m² at 30 wt. % particles loading for 300 µm. The hardness of the composite was improved with increase in particle loading of the composites. The hardness increases from 6.53 HRF to 9.1HRF at 35 wt. % for 150 µm particle size and 8.6 HRF for 300 µm. Density and water uptake of the composite increases with increase in particles loading and size in the composites. The SEM images of the composites reveal a good interfacial bonding between Doum palm shell particles and the matrix. This account for the good impact and hardness properties of the composites obtained.

Keywords: Impact strength, Hardness, polypropylene, Doum palm shell particles, Microstructural analysis, water absorption

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1. Introduction
Natural fibres/fillers composites are considered to have potential use as reinforcing material in polymer matrix composites because of their good strength, stiffness, low cost, environmental friendly and are biodegradable (Tisserat et al., 2014, Seth et al., 2018). The increasing demand for eco-friendly materials, soaring prices of petroleum based plastics and pressing environmental regulations have all triggered the growing interest in the field of composites (Srinivasa et al., 2011, O’zturk, 2011). Polymer composites consist of polymers as the matrix and one or more fillers or fibres are added to serve specific objectives or requirements to improve its mechanical and physical properties, wear resistance, reduce weight and give good thermal properties. For example, composites for aerospace and sports applications require high mechanical and thermal properties. Traditionally, synthetic fibres such as carbon or glass fibres have been used as reinforcement in composites and are able to produce better properties. However, with the growing global environmental concerns, their slow biodegradability is a disadvantage. Therefore, researchers are finding other viable approaches to enhance the biodegradability of polymeric composites. For this reason, natural fibres/fillers provide good prospective as reinforcements in polymers. The main advantages of using natural fibres/fillers in composites are low cost, sustainability, light weight, and being nonabrasive and non-hazardous and more importantly they can accelerate biodegradability of the polymeric composites (Biswa s et al., 2011, AL-Hassani et al., 2010, Seth et al., 2018 and Aji et al., 2012)

Reinforcement of polymers by particulates plays an important role in improving the mechanical and physical properties such as resistance to abrasive wear, hardness, impact strength etc. to make it more suitable for engineering application. Mechanical properties of a polymer can be controlled by incorporation of particles in polymer matrix (Clark 2002). The presence of fillers in matrix also impact on numerous properties of composite material, such as solidity, density and reduces cost. The effect of particles/filler loading, fibre length, fibre and
orientation has significant influence on mechanical behaviour of polymer composites and is studied by many investigators (Biswas et al., 2011, Clark 2002). Also, the influence of parameters such as concentration, impact velocity, fibre geometry, and shapes of the particles is very significant, and any changes in them can considerably affect the material properties. Unlike metals, fibre/particle reinforced composite materials which do not undergo plastic deformations after the impact. Elastic deformations may appear near the impact area in the case of a low intensity impact or deteriorations of the material that separates the fibres from the matrix, matrix cracking, and fibre breaking. The absorbed energy consequent to the impact depends on other parameters. If the bonding between the matrix-fibre/filler is strong, a continuous crack may spread along the material (Sanchez et al., 2005). In the case of a weak bonding, the generated crack may have an irregular form, leading to a rapid separation of the fibres from the matrix and to considerable absorption energy. When designing composite materials, it is necessary to make sure that the bonding between the fibres/fillers and the matrixes are not too weak. Poor bonding of the matrix-fillers/fibres has negative influences on the hardness and impact behaviour of the composites.

2. Material and Method

2.1 Material

The raw materials used in this work were Doum palm shell and Polypropylene with Density of 0.905 g/cm³, Melt flow index of 12 g/10 min and melting temperature of 135 – 171 °C.

2.2 Equipment

The equipment used in this work are:

- Two roll mill, Allen-Bradley with model 802T-WS1P for compounding the composite.
- Electronic hydraulic press with model No. 3851-0 for composite pressing.
- Cat NV412 Charpy Impact Testing Machine.
- Rockwell Universal Testing Machine, Type 8187.5 LKV Model B.
- Phenom pro X Scanning Electron Microscopy. Model No. 8000733.
- Standard set of sieves 150 μm and 300 μm size

2.3 Methodology

2.3.1 Doum Palm Shell Particles Preparation

The Doum palm fruit were collected from Ashaka Gari, Gombe state, Nigeria. The edible part and the kernel were removed and the shell dried in an oven for 24 hrs. It was then crushed together with the short fibres on it to smaller pieces, grind to powder and sieved using 150 and 300 μm set of standard sieves. Figure 1 show the Doum palm fruits, shell and particles prepared.

![Figure 1: Doum palm shell particles preparation from Doum palm fruits.](image)

2.3.2 Composite preparation

The composite were prepared by blending together the Doum palm shell particles and polypropylene in a two-roll mill at 180 °C and rotational speed of 45 rpm. Polypropylene was first melted to allow for adequate flow of the molten Polymer before pouring the Doum palm shell particles used as reinforcement in the composite were added. Cross mixing of the molten polypropylene and the Doum palm shell particles
was done until uniformity was obtained. The composite blend was removed from the mixer and compressed by an electrically heated hydraulic press in 100 x 100 x 10 mm mould and allowed to cool to room temperature at 8 MPa pressure before the sample were removed from the press. The composite test specimens were sliced for impact, density and water absorption.

2.3.3 Determination of Impact Strength properties
The impact tests on the formulated composite samples were carried out using Cat. NV. 412. Standard impact test samples measuring 80 x 10 x 10 mm with notch depth of 2 mm was prepared according to (ASTM, D2000), and at angle of 45° as shown in figure 2. Three specimens for each of the composition was used and the average value was recorded in Joules (J). The impact strength of the composite was calculated using equation (1).

$$E = \frac{e}{bh}$$  \hspace{1cm} (1)

where:
- \(E\) – impact strength required to shift the specimen, kJ/m²;
- \(e\) – impact energy required to shift the specimen, kJ;
- \(b\) – width of the test specimen, m;
- \(h\) – thickness of the test specimen, m

Figure 2: Sample specimens for impact test.

2.3.4 Determination of Hardness Properties
The hardness test was conducted according to ASTM E18 using the Indented universal hardness testing machine as shown in figure 3. Three different readings were taken on each of the samples and the average value is used.

Figure 3: Universal hardness testing machine

2.3.5 Determination of Water Absorption Properties
The water absorption test was conducted according to ISO 62 and ASTM D570 standard. Specimen with dimension 20 x 20 x 5 mm were prepared, dried in an oven at 35°C for 8 hours and cooled to room temperature in a desiccator. Afterwards, the weights of the dry specimen were determined using an electronic scale accurate to 0.001 g. The test specimens were immersed in distilled water as shown in figure 4. The specimens were removed after every 24 hours, dried with tissue paper and weighed to determine the change in mass and is continued until no change in mass of the specimens is observed. The moisture content was calculated using equation 2.

$$M_t = \frac{W_t - W_0}{W_0} \times 100$$  \hspace{1cm} (2)
Where: $W_t$ is the weight of the samples after immersion in water (g)

$W_o$ is the weight of the samples before immersion in water (g)

Figure 4: Water absorption procedure

### 2.3.6 Determination of Density

The density of the composite samples was obtained by measuring the mass and volume of the sample used. The sample was weighed in air using an electronic laboratory balance (new classic MF with model no. MS801S/01) and then suspended in water. The weight of the sample when suspended in water was determined, the volume of the sample was determined from the volume of water displaced by it (Archimedean principle). The density of the sample was estimated from equation 3:

$$\text{Density}, \rho = \frac{\text{Mass}, m}{\text{Volume}, V}$$

(3)

The theoretical density $\rho_t$ of composite materials in terms of weight fractions of different constituents can easily be obtained as for equation 4 (Agarwal et al., 1990).

$$\rho_{ct} = \frac{1}{\left(\frac{W_f}{\rho_f} \right) + \left(\frac{W_m}{\rho_m} \right)}$$

(4)

Where, $W$ and $\rho$ represent the weight fraction and density respectively. The suffixes $f$ and $m$ stand for the fibre and matrix respectively. The voids content ($V_v$) in the composites is calculated using the following equation 5:

$$V_v = \frac{\rho_{ct} - \rho_{ce}}{\rho_{ct}}$$

(5)

### 2.3.7 Micro-structural Analysis

The internal morphology of the produced composites was studied by microscopic analysis using the phenom pro X model Scanning Electron Microscopy (SEM). SEM is a surface analytical technique used to generate magnified topographical images of a material surface 20 x to over 100,000 x. In this research, the captures were done to a magnification of 1000 x.

### 3.0 Results and discussion

The investigation of mechanical and physical properties of composites is one of the most important techniques in studying the behavior of composite materials. The properties of particles-reinforced composites depend on the nature of matrix material and the distribution of the reinforcing particles, the nature of the particles-matrix interfaces. The result of the impact, hardness, density and water absorption of polypropylene reinforced Doum palm shell particles composite is discussed in below

#### 3.1 Effect of Doum palm shell Particles Loading on Impact Strength

When a composite is subjected to impact, rapid crack propagation is initiated through the material. When this type of crack propagation encounters filler particles in the filled composites, the filler particles can absorb the
energy and stop the propagation, if filler matrix interaction is strong (Ramakrishna et al., 2006). The impact test result is graphically illustrated in Figure 5. The addition of Doum palm shell particles increased the impact strength of the polypropylene composite for all the particle sizes investigated. This suggests that the introduction of the particles stiffened the flexibility of the polymer and improves its ability to absorb and dissipate energy. Composite with 150 µm has the highest impact strength of 4.1 kJ/m$^2$ at 35 wt. % particles loading and 3.9 kJ/m$^2$ at 30 wt. % particles loading for 300 µm. Smaller particle size provided greater amount of compaction and therefore strength to the composites. when the quantity of the particles reached a threshold, there is a possibility of the matrix not to fully flow through the particles which can reduce the capacity of the particles to share the load with it. This experimental result is in agreement with the work of (Guo et al., 2005) who investigated polypropylene/carbonate system and found that the impact strength of the composites increased at first with increase in filler content, and later, decreased with further addition of fillers. It also agreed with the work of (Hassan et al., 2012) who studied polyester/eggshell particulate composites and reported that the impact strength increases with increase in filler loading.

![Figure 5: Effect of Doum palm shell particle loading on Impact test.](image)

### 3.2 Effect of Doum Palm Shell Particles Loading on Hardness

Figure 6, shows the influence of the particle loading on the hardness property of polypropylene reinforced with Doum palm shell particles composites. It shows a progressive increase in hardness as the particle loading in the matrix increases. The introduction of Doum palm shell particle in the matrix increases the hardness property from 6.53 HRF to 9.1 HRF at 35 wt. % for 150 µm particle size and 8.6 HRF for 300 µm Doum palm sell particles size. This may be attributed to the fact that the addition of Doum palm shell particles in polypropylene stiffened the elasticity and improved the matrix surface resistance to the indentation. The result also reveals that the smaller the particles, better matrix-particles interaction the better the hardness property of the composites.
3.3 Effect of Doum Palm Shell Particles Loading on Water Absorption

Figure 7 and 8 shows the interpretation of water absorption properties of polypropylene reinforced Doum palm shell particles composite for 150 and 300 µm particles size. The water absorption property of the composites increases with exposure time and increase in particles content in the matrix for all the particles sizes investigated. This is because Doum Palm shell particles contains polar hydroxyl group which result in water absorption of the composite. The hydrophilic nature of particles and the greater interfacial area between the particles and the matrix result to an increase in water uptake as the particles content increases in the matrix (Dhakal, et al., 2007). The composites with particles size of 300 µm have higher percentage water absorption than the composites with 150 µm particles. This is because smaller particles have greater amount of compaction and less porosity than larger ones which therefore reduce water intake in the composite.

Figure 7: Effect of Doum Palm Shell Particle loading on water absorption for 150 µm
3.4 Effect of doum palm shell Particles Loading on Density

Figure 9 shows the graphical interpretation for the variation of density with doum palm shell particle loading. The density of both set of the composite increases with increase in weight content of the doum palm shell particles. This shows that the particle is denser than the matrix. It was seen that there are some differences between the experimental and the theoretical densities of fabricated composites. This variation is due to the presence of void content and pores in the composites. Figure 10 shows the void content for both sets of the composites for all the samples. With increases of Doum palm shell particle loading, the void content in the composites also increases. These voids content significantly affect the mechanical properties of the composites. Similar result was reported by (Hassan et al., 2012), and (Shakuntala et al., 2014).
3.5 Micro-structural Analysis

Microstructure studies were made on the composites developed from Doum palm shell particles and polypropylene. The SEM images of the composites are shown in Figure 11. From the photographs, Figure 11a shows a good interfacial bonding between Doum palm shell particles and the matrix. This is what account for the high mechanical properties. Figure 11b shows a poor bonding between the matrix and the particles. Aggregation of the particles is seen at some point due to poor mixing of the composites. Figure 11(c) to (e) reveals inadequate mixing of the composites. The particles are seen detaching themselves from the matrix (Polypropylene) surface leading to poor interfacial bonding. Figure 11(g) shows good bonding. There is good dispersion between the particles and the matrix. Figure 11(h) to (i) shows poor mixing of the particles and the matrix. Some clusters of particles are distributed at some point in the matrix leading to poor interfacial bonding. The presence of uneven particles distribution in the matrix is due to the random particles orientation adopted during the compounding and pressing stage and is probably the cause of the reduced impact strength with higher particle compositions.

Figure 10: Variation of Voids with doum palm shell Particle loading

(a) 10 wt. % for 150 µm    (b) 15 wt. % for 150 µm    (c) 20 wt. % for 150 µm
(d) 25 wt. % for 150 µm    (e) 30 wt. % for 150 µm    (f) 35 wt. % for 150 µm

Particles concentration
4.0 Conclusion

The study shows that the addition of Doum palm particles in polypropylene matrix improve the impact strength of the composite by stiffening the flexibility of the polymer and improved its ability to absorb and dissipate energy. It also showed that composite with 150 µm has better impact strength. The study also reveals that the hardness property of the composite was improved with increase in particle loading of the composites and has better hardness property at 150 µm particles size. The density and water absorption properties of the composites increase with increase in particle loading and increases particles sizes. The SEM images of the composites showed a good interfacial bonding between Doum palm shell particles and the matrix. These account for the good impact and hardness properties of the composites. From the impact and hardness result of the composites, the composite has potential in automobile interior and other engineering application.

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