Physical and mechanical properties of flamboyant (Delonix Regia) pod filled polyester composites

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ABSTRACT
The purpose of this research is to develop new polymeric composite materials from the flamboyant pod (Delonix Regia), an agricultural waste, with polyester as a matrix and to investigate their properties and application areas. The flamboyant pod particles of 75 μm were incorporated into the polyester resin with different loadings of 10, 20, 30, 40, and 50 wt%. The influence of the pod particles on mechanical and morphological properties was determined and investigated. The results showed that 10 wt.% gave the best results for tensile, impact, and flexural properties with values of 40.6 MPa, 4.6 kJ/mm², and 86.82 MPa respectively, the values are however lower than the unfilled. The hardness properties increased with increasing filler loadings with values from 23.8 HV – 32.7 HV for 10 wt% - 50 wt% respectively. The micrographs of the fractured impact samples confirmed the 10 wt% filler loading having the best properties with homogenous dispersion of the flamboyant pod particles (FPP) within the polyester resin. It can be concluded that lightweight composites with reasonable properties have been developed at 10 wt % loading which is suitable for non-load bearing and indoor applications in the automotive and building industries as partition tops, walls, and boards owing to their saturation in water after 30 days of immersion. The flamboyant pod material can be further explored for added values with tougher polymer matrices.

1. Introduction
Polymeric composites have found various applications in different engineering fields ranging from automobiles, marines, domestics, and aerospace (Kumar et al., 2019). These composites made from natural or synthetic fibres are built using reinforcements and polymeric matrices. However, the challenges of anisotropic properties which are connected to composites reinforced with fibres have made researchers focus on particulate reinforced composites to substitute the fibre reinforced components (Jiang et al., 2010).

Plant fibres from various resources are extensively used by industries for various applications (Saleh et al., 2021; Gholampour and Ozbakaloglu, 2019; Aisyah et al., 2019; Bello et al., 2015). The middle of the 20th Century recorded a drastic rise in the use of synthetic fibres leading to the collapse of the natural fibre industry’s market stakes (Jawaid and Abdul Khalil, 2011). But, the significant property of these natural fibres which are their non-carcinogenicity and biodegradability brought back their relevance with an added benefit of being economical (Bodros et al., 2007). This has expanded its utilization for railway coaches, automobiles, construction materials, furniture, and cabinets. Also, agricultural materials and products from forestry have largely yielded 30–40% unused resources, which can find applications for further processing with great value.

Flamboyant (Delonix Regia) pods are obtained from the Delonix Regia tree (flamboyant flame tree). It is a popular tree (Figure 1) commonly found in Nigeria and Africa (Agunsoye et al., 2017). The dark brown pods with dimensions of 5 cm in width and 60 cm in length frequently fall from the tree at dry seasons (Krauchenco et al., 2003). The problem is that the Delonix Regia pods litter the environment especially during the dry season and have not been fully explored and applied in composite materials. The pods have previously been burnt, but toxic greenhouse gases (GHGs) such as nitrous oxide, carbon dioxide and carbon monoxide produced are injurious to human health (Ezeokpube et al., 2014).

Research works on Delonix Regia (DR) pods, a natural waste, have been scarcely reported. The few available ones include its use as a precursor for activated carbon development, with significant surface

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properties suitable to serve as a catalyst in biodiesel production (Narowksa et al., 2020). The seed has found application in animal diets as a source of protein (Maradini et al., 2021; Costa et al., 2021), to improve the weights in animals such as tilapia, rabbits, and broilers (Bake et al., 2014; Kaga, 2013; Krishnan et al., 2015). Dhawane et al. (2015) carried out a study on the transesterification of *Hevea brasiliensist* oil as a heterogeneous catalyst using the approach of central composite design on flamboyant pods obtained steam activated carbon. Despite the laudable researches mentioned above, the fabrication of novel polymeric composites using seeds of *Delonix Regia* (DR) as filler with reprocessed low-density polyethylene (RLDPE) (Agunsoye et al., 2017) is the only researches mentioned above, the fabrication of novel polymeric compositions have not been reported in the literature to the best of our knowledge.

The driving force for this research is the abundant nature of *Delonix Regia* trees at senior staff club, Ahmadu Bello University, Zaria, Nigeria was the source of the flamboyant pods. The unsaturated polyester resin (UPR), cobalt accelerator, and Methyl-Ethyl-Ketone-Peroxide catalyst used for the experiment were provided by Olasco Chemical Company, Zaria, Kaduna State, Nigeria. Other chemicals include Acetic acid (Sigma-Aldrich, Germany), Sodium hydroxide (Sigma-Aldrich, Germany).

2. Materials and methods

2.1. Materials

*Delonix Regia* trees at senior staff club, Ahmadu Bello University, Zaria, Nigeria was the source of the flamboyant pods. The unsaturated polyester resin (UPR), cobalt accelerator, and Methyl-Ethyl-Ketone-Peroxide catalyst used for the experiment were provided by Olasco Chemical Company, Zaria, Kaduna State, Nigeria. Other chemicals include Acetic acid (Sigma-Aldrich, Germany), Sodium hydroxide (Sigma-Aldrich, Germany).

2.2. Methods

2.2.1. Extraction and treatment of flamboyant

The seeds of the Flamboyant pod were extracted from the hard pod manually. This involves the process of breaking the pod to remove the seed; the pods were then a crushed using crusher and then ground and sieved into a smaller particle size of 75 μm. The ground pods were treated with a 3 % solution of sodium hydroxide (NaOH) for 1 h followed by neutralization with 1 % acetic acid. Finally, it was washed with fresh distilled water and dried for 24 h in an oven at 30 °C. The formulation used is shown in Table 1.

2.2.2. Fabrication of UPR/Flamboyant pod composites

Flamboyant pod (75 μm particle size), unsaturated polyester, UPR, accelerator, and MEKP catalyst were fabricated by simple mixing and casting technique, using a glass mould with five different filler loadings based on the design in Table 1. A mould with a dimension of 120 mm × 120 mm × 3 mm was used for casting the composite slabs in which foil paper served as mould release agent. The cast composite samples were allowed to cure for about 24 h at 27 °C ambient temperature before they were cut according to ASTM standards for the physical, mechanical and morphological analysis.

2.2.3. Characterization of the composites

The characterizations were performed in line with ASTM standards for testing materials. The composite samples were conditioned for 24 h at 27 °C room temperature, after which they were subjected to various tests.

2.2.3.1. Physical properties

2.2.3.1.1. Density. The composite samples were cut into specific sizes according to ASTM D4052 standard and were weighed and their masses recorded. A known volume of water was obtained in a measuring cylinder (250 ml) and one piece of the sample is immersed in the measuring cylinder and the increase of the water is recorded. The difference between the initial known volume water and the volume of water with a sample in it gives the volume of the composite. The mass of that piece of composite is then divided by the volume of the piece to get the density of that particular piece of composite (Equation 1). The same procedure was carried out for all the other composites to obtain their densities and an average of five (5) specimens were used per sample.

\[
p = \frac{m}{v}
\]

where:

\( p \) = density (g/cm³)
\( m \) = mass of sample (g)
\( v \) = volume of sample (cm³)

2.2.3.1.2. Water Absorption. The test for water absorption was done according to ASTM D570 standard procedure. The flamboyant pod-filled polyester composites were dried in an oven for 30 min at 60 °C to eliminate residual wetness from the composite before cooling. Immediately upon cooling, the composite specimens were weighed and submerged in distilled water. The test was carried out at room temperature (25 °C). The composite samples were removed from the water and reweighed after 24 h intervals. Water absorption is expressed as an increase in weight percentage (Equation 2). An average of five (5) specimens were used per sample.

\[
\text{Water absorption (\%)} = \frac{\text{final wt} - \text{initial wt}}{\text{initial wt}} \times 100
\]

Table 1. Formulation for UPR/Flamboyant pod particles composites.

| S/No | FPP Fillers (wt%) | UPR matrix (wt%) |
|------|-------------------|------------------|
| 1    | 0 (control)       | 100 (control)    |
| 2    | 10                | 90               |
| 3    | 20                | 80               |
| 4    | 30                | 70               |
| 5    | 40                | 60               |
| 6    | 50                | 50               |

FPP = flamboyant pod particles; UPR = unsaturated polyester resin.
2.2.3.2. Mechanical properties. The composite samples were conditioned at 27 °C ambient temperature for 24 h, after which the following mechanical tests were carried out:

2.2.3.2.1. Tensile properties. The test was carried out using the Monsanto Tensometer (type w serial No 9875). This test was used to obtain the tensile strength, elongation at break, and modulus of the material. The specimen was cut into dumbbell shape (Figure 2) according to the ASTM D638-98 standards. The required dimension of each sample used was 120 mm \( \times 20 \) mm \( \times 3 \) mm at a crosshead speed of 2 mm/min and 40 mm gauge length for the tensile testing. An average of five (5) specimens were used per sample for the test.

Equations 3–5 were applied for the composites tensile property determinations:

\[
\text{Elongation at break (Eb)} = \frac{\text{extended length}}{\text{gauge length}} \times 100 \tag{3}
\]

\[
\text{Stress} = \frac{P}{A} \left( \frac{N}{mm^2} \right) \tag{4}
\]

where, \( P = \) load (N), \( A = \) area (mm\(^2\))

\[
\text{Strain} = \frac{\text{elongation}}{\text{gauge length}} \tag{5}
\]

2.2.3.2.2. Flexural properties. A universal material testing machine (cat. Nr. 261) was used to conduct the flexural test. The behaviour of materials, when exposed to 3-point beam loading, is what the flexure test determines. This procedure was used to get the flexural strength, modulus, and strain at break for the flamboyant pod-filled polyester composites. A flexural test was conducted using a 2 mm/min crosshead speed based on ASTM D790-98 standard procedure with a sample of 100 mm \( \times 30 \) mm \( \times 3 \) mm dimension. The gauge/span length of 80 mm was used for an average of five (5) specimens per sample. The test setup with samples is shown in Figure 3.

The bending properties of the samples were obtained using the expressions in Eqs. (6) and (7):

\[
\text{MOR} = \frac{3Fl}{2bd^2} \tag{6}
\]

where:
- MOR = modulus of rupture (flexural strength) in N/mm\(^2\)
- \( F = \) force (N).
- \( l = \) span/gauge length distance (mm).
- \( b = \) sample width (mm).
- \( d = \) sample thickness (mm)

\[
\text{MOE} = \frac{Fl^3}{4bd^2D} \tag{7}
\]

where:
- MOE = modulus of elasticity (Flexural Modulus) in N/mm\(^2\)
- \( F = \) force (N).
- \( l = \) span/gauge length (mm).
- \( b = \) sample width (mm).
- \( d = \) sample thickness (mm).
- \( D = \) deflection (mm).

2.2.3.2.3. Impact strength. Using the 412-07-15269C Charpy Impact Testing Machine (15 and 25 J capacity), the tests were done following ASTM D-256 standard procedure. Composite samples were cut into 100 mm \( \times 13 \) mm \( \times 3 \) mm dimensions with a notch of 0.5 mm (Figure 4) and positioned on the machine using knots to tightly hold them with equal lengths at both ends. A hammer of 15 J energy capacity was used to impact the sample leading to its breakage and the energy absorbed recorded. The test was performed for the remaining samples and the average values of five (5) specimens were recorded accordingly.

2.2.3.2.4. Hardness. The Vickers hardness tester of the model (MV1-PC) (Figure 5) was used to decide the hardness of the composite samples based on ASTM D2240 standard procedures. The sample with the dimension of 30 mm \( \times 30 \) mm with a thickness of 3 mm was placed between the upper and lower indenter of the machine and the upper indenter was made to be in contact with the sample which gives the reading of the hardness of that particular point of contact. The hardness near the two edges and the centre of each sample was taken and the mean value was recorded.

2.2.3.3. Morphological property

2.2.3.3.1. Scanning electron microscopy. To view the composites’ surface morphology, a scanning electron microscope (SEM, Joel TM, model JSM-7600F) was used to study the impact fractured specimens to determine the interfacial bond and cross-section of the fabricated and fractured specimens. During the analysis, 16 kV voltage and operational space of 4–6 mm were employed.

3. Results and discussion

3.1. Physical properties

3.1.1. Density test result

The densities of composite materials are very fundamental, and it is the very essence of choosing them in place of metals. Materials of high specific strength are often preferred by engineers as opposed to those with lower mass density. Figure 6 shows the effect of density on UPR/FPP composites. The control sample had a density value of 1.15 g/cm\(^3\) which increased to 1.27 g/cm\(^3\) at 50 wt%. The density of the composites increased with increasing filler loadings with marginal values. This increase might be a result of the density of the pod particles (0.93 g/cm\(^3\)).

The fabricated composite materials gave moderate density which is capable of giving high specific strength.

3.1.2. Water absorption

Figure 7 illustrates the percentage moisture uptake of UPR/FPP composite at different filler loadings for 1–30 days. The general trend was that an additional increase in filler loading resulted in the water absorption property increasing for the composites as well; the sample exhibited higher water absorption. This may be due to the fact that the flamboyant pod is hydrophilic (having –OH groups). It is a well-known fact that fibres from the natural origin are generally hydrophilic, containing several hydroxyl groups (–OH) in the structure of the fibre leading to the formation of many hydrogen bonds, whereas, polymer molecules are hydrophobic i.e. they do not contain any polar group as such, the polymer does not easily bond to water molecules explaining its ability to stay dry (Kabir et al., 2011). However, as the UPR content decreases there
is also an increase in filler content resulting in a larger surface area of fillers exposed to water; the fillers are not fully surrounded by the matrix. This explained why the samples with higher filler loading absorbed more water as a result of the increase in the available –OH groups which formed linkages of hydrogen bonding. It was observed that the control sample absorbed less water which might be due to the hydrophobic nature of UPR resin.

The behaviour of the developed material as displayed in the curve obeyed Fickian Law as they became saturated after 30 days of immersion showing linearity. The little water ingress by the control sample might be due to micropores and void spaces developed during fabrication as a result of air bubbles because polymeric resins are hydrophobic. The 50 wt% absorbed more water than other samples which is of great disadvantage to the composites as this ultimately drops the composite’s longevity probably due to the micro-organisms naturally contained in water which attacks the cellulosic flamboyant pod particles thereby degrading it.

3.2. Mechanical properties

3.2.1. Tensile strength

The tensile strength of a material is the maximum stress a material can attain before failure. The average tensile strength of five (5) specimens examined for each loading of reinforcement is shown in Figure 8.

The outcome of filler loadings on the composites’ tensile properties manufactured from the flamboyant pod is shown in Figure 6. The UPR which is the control sample had higher tensile strength (41.24 MPa) than the UPR/FPP composites. The reduction in the tensile strength between the UPR/FPP and UPR might be due to the weak filler and matrix interface resulting from the presence of impurities, lignin, and waxes on the filler. However, 10 wt % UPR/FPP had the maximum tensile strength (40.62 MPa) with respect to the other UPR/FPP, which decreased with increasing filler loadings up to 50 wt%. The decrease could be as a result of filler-filler interaction at higher filler content whereby load was not effectively transferred through the matrix to the filler. Sun et al. (2006) stated that there is usually a reduction in the particulate composite’s tensile strength with increasing filler content which obeys a power law.

![Figure 3](image1.png)

Figure 3. (a) A universal material testing machine and (b) flexural test samples.

![Figure 4](image2.png)

Figure 4. Notched Charpy impact test samples.

![Figure 5](image3.png)

Figure 5. (a) Vickers hardness tester (model MV1-PC) and (b) test samples.

![Figure 6](image4.png)

Figure 6. Density result for Flamboyant pod Filled polyester composite.
due to matrix-filler poor bonding relationship. At higher filler content, the poor bonding strength between the matrix and the filler may occur because of excess fillers.

The tensile modulus can be defined as tensile strength per strain of the material and it is a measure of the stiffness of the materials. It is observed that the tensile modulus improved with a rise in filler loading for the UPR/FPP composites because of the nature of the material. The unfilled composites gave the lowest modulus of 188.27 MPa and the highest filler loading of 50 wt% gave the highest modulus value of 254 MPa. It can be deduced that the higher the tensile modulus, the stiffer the composite becomes.

3.2.2. Elongation at break

The consequence of filler loadings on the breaking elongation of the composites as presented in Figure 9 indicated that the elongation of the UPR was higher than any of the UPR/FPP composites. The breaking elongation drops with additional pod particle loading, with the elongation at break of 10 wt% UPR/FPP composite slightly having a value of 18% which is higher than other reinforced filler loadings. Increase in filler content could be responsible for this behaviour as the filler is stiffer than the matrix hence, the polymer-polymer chain is broken by the filler, hence the reduction in elongation.

3.2.3. Flexural properties

The bending or fracture strength of composite samples is the sample’s resistance to deformation under an applied load. From Figure 10, the maximum flexural strength of the URP/FPP composite was at 10 wt% with a value of 86.82 MPa; this shows the rigidity of the composite. The lowest flexural strength was at 50 wt% filler with the value of 48.98 MPa which could be as a result of poor interaction and wetting of the flamboyant pod filler and polyester resin. This also implies that flexural strength decreased almost linearly with an increase in filler composition.

The flexural modulus (Figure 10) improved as the pod filler loading increased. The maximum flexural modulus was found to be at 50 wt% URP/FPP with a value of 2382 MPa and the lowest was found to be with the unfilled composite sample with a value of 1852 MPa. The Flamboyant pod particles improved the bending rigidity of the composites materials to a great extent; this could be due to the hard nature of the flamboyant particles.

3.2.4. Impact strength

The capacity of composite materials to resist sudden shock loading during deformation depicts a very good impact strength. The developed composites have moderate impact strength after the addition of the FPPs (Figure 11). The maximum impact strength was found to be at 10 wt% filler loading with a value of 4.6 kj/mm².

Figure 7. Water Absorption result for flamboyant pod-filled polyester composite.

Figure 8. Tensile strength and modulus of flamboyant (Delonix Regia) pod-filled polyester composites.

Figure 9. Elongation at break of flamboyant pod filled polyester composites.

Figure 10. Flexural strength and modulus of flamboyant (Delonix Regia) pod-filled polyester composites.

Figure 11. Impact strength of Flamboyant (Delonix Regia) pod filled polyester composites.
Higher filler loadings reduced the composite's materials capacity to take in additional energy. The resin without any filler (control) has the highest impact strength value of 5.2 kJ/mm², which is just an 11.35% drop compared with the 10 wt% loadings, showing the maintenance of structural integrity of the resin. It is thus, established that the impact property of materials has a direct relationship with toughness which is extremely dependent on the form of the constituent material as also reported by Achukwu et al. (2014).

3.2.5. Hardness test

The comparative resistance of composite materials surface under specified load by an indenter is shown in Figure 12. It shows an improvement of the composite's hardness value and the sample's resistance to localized deformation by the inclusion of the FPP fillers as expected because additional fillers decrease the polymer brittleness.

There was a 45% increment in hardness values of the developed composites (32.7 HV, when loaded up to 50 wt%) to the unfilled polyester resin (18 HV). This improvement agrees with the hardness values obtained by Agunsoye et al. (2014) in their particulate coconut shell and recycled polypropylene matrix.

3.3. Morphological result

3.3.1. Scanning electron microscopy

The micrograph reveals a uniform spread of the FPP fillers in the polyester resin for the 10 wt% (Figure 13a). This shows the maintenance of good chemical interaction that still exists between the matrix and the filler particles. This factor gave a positive influence on the mechanical properties for the 10 wt% loadings.

There is a poor and irregular cross-section for the 50 wt% of FPP loading (Figure 13b) at 500 magnifications, showing signs of particle agglomeration within the matrix. Figure 14 showed a better view of the wetting and adhesion abilities for the two extreme loadings under consideration.

![Figure 12. Hardness Test for Flamboyant pod Filled unsaturated polyester composite.](image)

![Figure 13. The fractured surface of UPR/flamboyant pod composite for (a) 10 wt% and (b) 50 wt% filler loadings at a magnification of 500x.](image)

![Figure 14. The fractured surface of UPR/flamboyant pod composite for (a) 10 wt% and (b) 50 wt% filler loadings at a magnification of 1000x.](image)
The 50 wt% (Figure 14b) showed very poor wet ability and adhesion which supports the poor results gotten from the previous analysis. The impact fractured micrographs samples confirmed the 10 wt% filler loading (Figure 14a) having the best physical and mechanical properties with smoother cross-section and stronger adhesion between the UPR matrix and flambayan pod particles. However, structures seen in the micrograph (Figure 14b) might be due to intrinsic flaws obtained during the composites processing. The availability of these blank areas within the composite may lead to disjoinedness which will likely weaken the composite’s mechanical properties.

4. Conclusion

The development and study of the mechanical and physical properties of flambayan (Delonix Regia) pod-filled polyester composites have been successfully achieved with a very fine filler particle size (75 μm). It can thus be concluded that:

i. Composites with good mechanical properties have been developed. Tensile, impact, and flexural properties were best at 10 wt% suggesting that the loading of Flambayan (Delonix Regia) pod as a filler should not be beyond 10 wt% for the attainment of best physico-mechanical property. The composite’s hardness increased even up to 50 wt% loading with a marginal value indicating a factor for consideration in its areas of application.

ii. The research has encouraged the possibility of using Delonix Regia pod particles for new polymeric composites. Lightweight composites materials with reasonable properties have been developed at ≤ 10 wt% loading which is suitable for non-load bearing and indoor applications in the automotive and building industries as partition tops, walls, and boards only, owing to their saturation in water after 30 days of immersion. The flambayan pod material can be further explored for added values with tougher polymer matrices and improved treatments.

Declarations

Author contribution statement

E.O. Achukwu: Conceived and designed the experiments.
J.O. Odey, M.M. Owen: Analyzed and interpreted the data; Wrote the paper.
N. Lawal, G. A. Oyilagu, A. I. Adamu: Performed the experiments.

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Data availability statement

Data included in article/supplementary material/referenced in article.

Declaration of interests statement

The authors declare no conflict of interest.

Additional information

No additional information is available for this paper.

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