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Effect of Particle Size on the Mechanical, Water Absorption and Thermal Properties of Flamboyant Seed Particle-filled Polyester Composites for Tile Applications

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The study examined the effect of particle size on the mechanical, water absorption, and thermal properties of polyester composites filled with flamboyant seed particles for tile applications. The composites were made using polyester filled with 10 wt % flamboyant seeds of various particle sizes. The results showed that the composites' bending and impact strengths decreased with particle size, with 100 µm particles having the highest values of 46.99 MPa and 1.86 J/m², respectively, while the flexural modulus, hardness, and water absorption increased with particle size. The micrographs of fractured samples showed that particles of 100 µm had better interfaces and a homogenous dispersion within the polyester matrix, resulting in improved properties. The thermograph showed that 100 µm particles have better thermal stability with an onset decomposition temperature of 318 °C and maximum weight loss at 373 °C. The study found that the size of the seed particles significantly influences the properties of the composites, with particle sizes of 100 µm showing superior performance. The study recommends the use of 100 µm flamboyant seed particles for low-load bearing applications like wall tiles due to their low water absorption and superior mechanical and thermal properties.

Keywords: Flamboyant seeds, Composites, Mechanical properties, Particle size, Thermal stability.

1. Introduction

The study of naturally reinforced composites, made from renewable resources like plants (cellulose), is on the increase (Shesan et al., 2019; Dong, 2017; Haruna et al., 2014), and their properties are based on reinforcement type and structure. Cellulosic reinforcement offers low weight, low cost, and good mechanical properties compared to synthetic fibres. They are renewable, abundant (Okeola et al., 2018; Jusoh et al., 2016; Ten et al., 2012), flexible, biodegradable, and pose minimal health hazards (Lotfi et al., 2019; Mochane et al., 2019; Jawaid and Abdul Khalil, 2011). They are sustainable, ecofriendly, and affordable, reducing waste and pollution in bio-composites (Karimah et al., 2021; Mohammed and Attallah, 2020; Al-Ogla. and Omari, 2017). It offers economic feasibility, reduced production energy by 80%, weight by 10%, and cost by 5% compared to glass-reinforced composites (Akash et al., 2016). They serve as an alternative to carbon, glass, and man-made fibres in composite production and are useful for structural engineering applications due to their non-corrosive nature (Kuan

et al., 2021; Karakoti *et al.*, 2019; Lotfi *et al.*, 2019; Safri *et al.*, 2017; Akash *et al.*, 2016; Haghdan and Smith, 2015; Pai, and Jagtap, 2015; Jawaid and Abdul Khalil, 2011). Composites made of synthetic materials like glass, carbon, and other components are expensive, non-biodegradable and have undesirable environmental effects. Researchers are exploring the use of natural reinforced composites to reduce production costs and encourage waste biomass recycling (Kuan *et al.*, 2021). Biodegradable and bio-based sources are being explored to preserve the environment (Chabros *et al.*, 2019), however, little attention is given to flamboyant seeds.

Agricultural waste produces biomass, with only 10% used for biocomposites (Dungani *et al.*, 2016), which offers a sustainable and eco-friendly alternative for filling polymeric materials. Common waste disposal practices, such as incineration, dumping, and indiscriminate disposal, pose health risks and contribute to environmental issues like greenhouse

gas generation, degradation, and air pollution. This is a crucial issue that needs to be addressed to protect the global environment (Dungani *et al.*, 2016). These wastes can be harmful or employed to generate bio-economy (Adejumo and Adebiyi, 2020). It offers low density, mechanical resistance, cost, biodegradability, and availability for reinforced plastic composites, promoting innovative solutions for green markets and job creation (Dong 2017; Adejumo and Adebiyi, 2020).

Flamboyant plants, commonly found in our environment, produce pods with seed chambers, which are often discarded as waste, posing environmental and health risks. The reduction of environmental effects is essential, and this can be achieved by adding value to waste products for industrial applications. Flamboyant pod seeds, an agricultural waste, are being explored for oil extraction (Adejumo et al., 2019), biochemical profile (Abulude et al., 2018), chemical and nutritional compositions (Ovedeji et al., 2017), and physicochemical properties of carboxymethylated seeds (Pacheco-Aguirre et al., 2010). Studies have demonstrated its use in the transesterification of Hevea brasiliensis oil (Dhawane et al., 2015) and as a protein source in animal feeds (Olugbenga et al., 2020; Supapong et al., 2017; Bake et al., 2014; Kaga, 2013; Alamede et al., 2010; Egene et al., 2008). The physical and mechanical properties of polyester composites filled with flamboyant pod particles were studied (Achukwu et al., 2022). Another study reported the use of flamboyant seed particles in recycled low-density polyethylene (Agunsoye et al., 2017). Despite numerous studies, there is limited exploration into using flamboyant seed particles as filler for creating new polymeric composites. Flamboyant pod waste, abundant in society, can be utilised in polymeric composites as low-density, cost-effective, biodegradable, and nontoxic materials, reducing waste disposal. environmental pollution, and contributing to green markets. This promotes waste biomass recycling, and reduces production costs and greenhouse gas emissions (Bhardwaj et al., 2021; Karimah et al., 2021; Bolcu and St'anescu, 2020). This study explores the influence of particle size on the properties of flamboyant seed particle-filled polyester composites for tile applications.

2. Materials and Methods

2.1. Materials

The raw materials used for the composite fabrication include polyester resin, a catalyst (methyl ethyl ketone peroxide), a promoter (cobalt naphthenate), and flamboyant seed particles. Equipment used includes glass moulds, a Retsch Masch 70992 jaw crusher, a ball mill grinding machine (Kera b.v. 057748), and a standard sieve (Seive-Tronic ISO 3310-1:2000, BS 410-1:2000). Others are the flexural strength tester (universal material testing machine, Cat Nr. No. Model: 26; impact testing machines; Resil Impactor (model number Ceast 363); hardness testing machine (Vickers Hardness Tester MV1-PC, Mh-v CM. 07/2012-1329, China); scanning electron microscope (Joel TM, model JSM-7600F, Japan) and thermogravimetric analyzer (Perkin Elmer TGA4000, Perkin Elmer Inc., USA).

2.2. Methods

2.2.1. Sample Collection and Preparation

The dried flamboyant pods were collected from the flamboyant trees at Ahmadu Bello University, Zaria. They were split open, the seeds collected, crushed using a jaw crusher (Retsch mesh Nr. 70992), and the hard seed coat (testa) separated from the soft inner part (endosperm/cotyledon). The coats collected were washed with clean water to remove the endosperm/cotyledon particles, air-dried for 48 hours, and then oven-dried at 60 °C for 48 hours to ensure proper drying. The dried crushed seeds were ground with a ball mill grinding machine (Kera b.v. 057748) and then sieved to various particle sizes of 100, 200, 300, 400, and 500 μ m using a standard sieve (Sieve-Tronic ISO 3310-1:2000, BS 410-1:2000).

2.2.2. Composites Preparation

Composites were prepared with 10 wt% using particle sizes of 100, 200, 300, 400, and 500 μ m via hand layup method. The polyester matrix and flamboyant seed particles were mixed, and the catalyst (1%) and promoter (1%) were added. The mixture was poured into a mould, covered with a glass sheet, pressed, and cured for 24 hours at ambient temperature.

2.2.3. Composites Characterisation

2.2.3.1. Flexural Test:

The test was carried out according to ASTM D790. The 3-point bending test was carried out on a universal material testing machine (Model: Cat Nr. 26) with a maximum load of 100 kN and a cross-head speed of 5 mm/min. The dimensions of the samples were $100 \times 30 \times 3$ mm in length, width, and thickness, respectively. The flexural strength and modulus were determined from the data obtained using Equations 1 and 2 and recorded.

$$\sigma_{\rm f} = \frac{3FL}{2bd^2} \tag{1}$$

$$\mathcal{E}_f = \frac{FL^3}{4bd^2\Delta L} \tag{2}$$

Where σ_f is flexural strength in Mega Pascal, F is the force (load) applied at the fracture point (Newton), L is the length of the support span (sample original length) (80 mm), b is sample width (mm), and d is sample thickness (mm). \mathcal{E}_f is the flexural modulus;

 ΔL is the amount by which the length of the sample changes (deflection).

2.2.3.2. Impact Test:

This was conducted using a Resil Impactor (model no.: Ceast 363) with a maximum capacity of 2 joules, according to the ASTM D256 ISO180 standard. The specimen with a notch of 2 mm at 45° radii was placed on the V-notch across the parallel jaw in the machine. The sample fractured at the V-notch due to the impact force applied by the hammer, and the impact energy absorbed by the specimen was then observed and recorded in joules per square metre.

2.2.3.3. Hardness Test:

The test was conducted according to ASTM D2240 using the Vickers Hardness Tester (MV1-PC, Mh-v CM. 07/2012-1329) with a maximum load capacity of 0.3 Kgf (100 HV). The test was carried out at a constant temperature of 23 ± 2 °C. Then, each of the specimens was mounted on a compartment, indented, and the hardness values were recorded.

2.2.3.4 Water Absorption:

This was carried out according to ASTM D570. The samples were conditioned in an oven at 40°C for 24 hours. Then, they are placed inside desiccators for 24 hours and weighed (Wi) using an analytical balance. The weighed samples were then placed inside a plastic container containing water. The samples were then repeatedly removed, dripped and weighed (Wf) at specified times for four (4) weeks. The percentage of water absorbed by the composites was determined and recorded using Equation 3 (Kumar and Singh, 2014);

Water absorption (%) =
$$\frac{Wf-Wi}{Wi} \times 100$$
(3)

2.2.3.5. Morphological Characterisation:

The surface morphology of the composites was studied using a Scanning Electron Microscope (SEM, Joel TM, model JSM-7600F) at 20 KV and 8000X magnification on the tensile fractured specimens.

2.2.3.6. Thermal Characterisation:

The thermal behaviour of the sample composites was studied with a Perkin Elmer TGA 4000-type thermal analyzer (Perkin Elmer Inc., USA). Samples with an average weight of 14.47 mg were heated under a nitrogen atmosphere at a rate of 10°C/min over a temperature range of 30 to 950°C. The

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samples' weight loss, temperature, derivative, and heat flow were recorded.

3. Results and Discussion

3.1. Flexural Properties

Flexural strength measures a material's ability to withstand bending forces. Figure 1 reveals that as particle size increases, the bending strength of the composites decreases, with the highest value of 46.99 MPa observed for particles of 100 µm and the lowest value of 36.27 MPa for particles of 500 µm. The increased particle size leads to reduced bending strength due to poor filler distribution, resulting in poor contact area and increased unevenness, causing voids, which increase with larger particle sizes. The composites' flexural strength may decrease due to random orientation or uneven filler dispersion in the polymer matrix (Okey-Onochie et al., 2020). The polyester matrix's inconsistency in flamboyant seed particles leads to defects that restrict stress, potentially causing confined weakness which leads to composite fracture. The composites' bending ability decreases as the particle size increases (Okey-Onochie et al., 2020; Lawal et al., 2019; Onuoha et al., 2017; Nwanonenyi et al., 2013; Onuegbu et al., 2011). The study is consistent with previous findings [Zakari et al., 2020; Daramola et al., 2017; Abdulrahman and Haruna, 2015].



Figure 1: Effect of Particle Size on Flexural Properties of the Composites

The figure also displays the flexural modulus, a measure of a material's resistance to bending and its stiffness when flexed. It shows that increasing the particle size of fillers (100-500 μ m) increases the flexural modulus of composites considered. A rapid increase from 100 μ m to 200 μ m followed by gradual increases from 200 μ m to 500 μ m. The improvement in modulus depends on composite morphology (Daramola *et al.*, 2017). Studies have shown a consistent rise in bending rigidity with an increase in particle size (Hanana and Rodrigue, 2021; Lawal *et al.*, 2019; Rahman *et al.*, 2010).

3.2. Impact Strength

Impact strength measures a material's ability to absorb energy before fracture, indicating its

tolerance to sudden shocks causing crack propagation. The balance between material strength and ductility is vital for product performance, service life, safety, and liability (Shesan *et al.*, 2019). Composites with brittle materials have low toughness due to minimal elastic deformation.



Figure 2: Effect of Particle Size on Impact Strength of the Composites

Figure 2 illustrates the impact strength of the composites based on particle size, revealing a decrease with increasing size. This may be attributed to weaker bonds between the flamboyant seed particles and the polyester matrix, resulting in a decreased surface area. The impact values for composites filled with particle sizes of 100 to 500 µm were 1.86, 1.76, 1.66, 1.51, and 1.30 $J/m^2,\,$ respectively. The composite's increased interface area leads to stronger mechanical bonding between the matrix and filler, resulting in higher impact strength with smaller particle sizes (Nasution et al., 2016). The lower impact strength values for larger particle sizes may be due to poor surface area and weak interaction within the polyester matrix due to irregular filler distribution, leading to void creation. The impact strength of fibre-reinforced polymer composites is influenced by the filler, polymer matrix, and filler-matrix interfacial wetting (Rahman et al., 2010). Kuan et al. (2021) found that a smaller particle size with a higher aspect ratio than the critical value enhances mechanical properties. The size of filler particles likely increased stress concentration in composites, leading to a decrease in impact strength (Onuegbu et al., 2011). The study agrees with the reported findings (Lawal et al., 2019, Manjunatha and Ahmed, 2017; Nasution et al., 2016; Onuegbu et al., 2011; Saini et al., 2010).

3.3. Hardness

Hardness measures a material's resistance to surface indentation. It is essential in engineering as it enhances wear resistance due to erosion by water, oil, steam, or friction in structural materials (Shesan *et al.*, 2019). Figure 3 shows that the hardness of the composites increases gradually with the size of flamboyant seed fillers, with values ranging from 32.80 to 41.10 HV for particles 100-500 μ m. This increase may be attributed to the orientation of the fillers and the polyester matrix. The hardness of a composite is influenced by the filler distribution within the matrix (Rahman *et al.*, 2010). The size of

filler particles is found to increase the hardness of composites, which agrees with some work findings (Akaluzia *et al.*, 2020; Lawal *et al.*, 2019; Onuoha *et al.*, 2017; Nwanonenyi *et al.*, 2013, Saini *et al.*, 2010).



Figure 3: Effect of Particle Size on Hardness of the Composites

3.4. Water Absorption

Figure 4 shows the impact of particle size on composites' water absorption over four weeks. The composites showed rapid water absorption in the first two days, increasing with immersion time and stabilising after two weeks in distilled water, then remaining constant. Numerous scientific studies have consistently reported similar findings (Achukwu *et al.*, 2022; Reddy *et al.*, 2018; Nasution, *et al.*, 2016; Abdulrahman and Haruna, 2015; Ameh *et al.*, 2015; Nohammed, 2015; Nwanonenyi *et al.*, 2013).



Figure 4: Effect of Particle Size on Water Absorption of the Composites

Moreover, the graph reveals that composites with larger particles absorb more water due to poor particle dispersion within the matrix, resulting in increased voids and water absorption. The highest value is observed in composites with particle sizes of 500 µm. Poor bonding in composites results in increased micro-voids, leading to increased water absorption (Yang et al., 2006). Larger particles in a matrix increase porosity and voids, promoting water absorption, while smaller particles create compact materials with lower absorption. This is influenced by factors such as filler-matrix orientation, voids, crosslinking degree, and interfacial bonding between fillers and polymers [Sahu. and Gupta, 2020]. Studies have shown that composites with smaller particle sizes absorb less water [Nasution et al., 2016; Ameh et al., 2015; Saini et al., 2010]. The studies indicate that smaller particle sizes enhance filler-matrix dispersion, bond stability, and reduce water absorption in composites.

3.5. Morphological Analysis

The fracture surface of composites allows for analysis of fibre-matrix adhesion. The micrographs (Plate I) show a regular dispersion and bonding between 100 µm particles and the polyester matrix. Plate I (a) indicates a fairly good interfacial interaction between the flamboyant seed particles and polyester matrix, contributing to improved mechanical properties. Good interfacial interaction enhances fibre matrix interface strength, distributes load evenly, and facilitates efficient stress transfer from matrix to filler (Asim et al., 2017). However, the interfacial bond is unstable, with micro-cracks on the defect surfaces (Supian et al., 2021). Plate I (b) shows tensile fractured surfaces of a 500 µm particle, revealing a rough surface with numerous voids and well-defined spacing due to filler pulled out, indicating poor interfacial adhesion at the interface. The poor adhesion indicates a low affinity between the polymer matrix and the filler (Hossain et al., 2017). Poor interfacial bonding causes fillers to detach from resin surfaces (Das and Biswas, 2016). Supian et al. (2021) found that the bonding between fibre and matrix is unstable when the defect surface has voids. Kolawole et al. (2015) reported that the presence of voids in a composite can lead to the propagation of fractures, thereby reducing its functional capacity. This might be attributed to the poor mechanical properties of the composite of particles of 500 µm.



(a). 100 µm



(a). 500 µm

Plate I: Effect of Particle Size on Morphology of the Composites

3.6. Thermogravimetric analysis

Thermogravimetric analysis (TGA) is a method used to measure the change in mass of a sample during heating, thereby assessing the thermal stability of the materials. It observes changes in a material's properties as temperature and time increase (Mohammed, 2015). This provides information on a material's thermal stability and decomposition behaviour, which determines its suitability for a specific application. The method also evaluates the weight of a sample in gaseous atmospheres (Neto et al., 2022). Lignocellulose, a blend of cellulose, hemicellulose, lignin, and other volatile components, is sensitive to temperature and is likely to undergo complete thermal degradation above 400 °C. Thermal degradation of cellulose occurs between 300 and 325 °C, with hemicellulose preceding it (250 to 300 °C), while lignin occurs in a broader range that initiates earlier but extends to higher temperatures; however, the effect of the two latter is proportional to fibre content (Monteiro et al., 2012).





Figure 5: Effect of Particle Size on Thermal Properties of the Composites

Figure 5 displays thermographs of composites with particle sizes of 100, 300, and 500 µm that reveal a multi-step decomposition pattern. The initial °C, degradation occurs between 40 and 70 with weight loss due to filler moisture evaporation, and lignocellulose decomposition around 300 °C. degradation of naturally Thermal reinforced materials occurs between 40 and 300 °C, starting with water evaporation below 150 °C, followed by lignocellulose breakdown and matrix decomposition at 250-300 °C (Mohammed, 2015). The second composite decomposition process results in weight loss due to linear chain fragmentation, with weak link scission causing losses at 35-445 °C and random scission in smaller chains at 445-575 °C (Wilson García et al., 2021). The final stage of coal formation leads to active residue formation, with a stability plateau (Mohammed, 2015), and the broken matrix component may result in intermediate species formation (Chu et al., 2018).

The thermograph shows that the smallest particle size of 100 µm exhibits better thermal stability compared to larger particle sizes of 300 and 500 µm. The composite with 100 µm shows rapid weight loss in the second stage, indicating homogeneous seed particle dispersion in the matrix, as shown in the SEM micrograph (Plate I). A swift decrease in weight loss signifies the presence of a specific component in a substance (Monteiro et al., 2012). As the size increases, the onset degradation temperatures shift to lower temperatures. The smallest particle size of 100 µm onset decomposes at 318 °C with a maximum weight loss at 373 °C, while the largest 500 µm particle has an onset decomposition temperature of 314 °C with a maximum weight loss at 400 °C. The thermograph showed a slow weight loss in the second step at around 370 and 372 °C for the corresponding 300 and 500 µm due to uneven

filler-matrix dispersion. The observed slow decomposition pattern may be due to uneven fillermatrix dispersion, which may slow down the degradation process. The region's slow weight loss decline suggests the presence of multiple volatile components (Monteiro *et al.*, 2012). This could be ascribed to the particles' tendency to agglomerate, resulting in heterogeneities and defects within the matrix, as well as the lignocellulosic particle's lower thermal stability (Mohammed, 2015).

4. Conclusion

Polyester composites filled with flamboyant seed particles with a 10% weight fraction and varying particle sizes of 100-500 µm have been successfully studied for their mechanical, water absorption, morphological and thermal properties. The findings demonstrate that particle size significantly affects the properties of the samples considered. Composite with 100 µm particle size displayed superior mechanical properties, with higher bending and impact strength values of 46.99 MPa and 1.86 J/m², respectively. The composite exhibited flexural modulus, hardness, and water absorption values of 4.64 GPa, 32.8 Hv, and 2.43%, respectively. The study suggests the use of 100 µm flamboyant seed particle materials for low-load bearing applications such as wall tiles and table tops due to their low water absorption, good mechanical properties, and better thermal stability.

Conflict of interest

The authors declare no conflict of interest.

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