

EFFECT OF PARTICLE SIZE ON TENSILE PROPERTIES AND DENSITY OF *DELONIX REGIA* SEED PARTICLES FILLED UNSATURATED POLYESTER RESIN COMPOSITES

H.I. Umaru¹, U.S. Ishiaku¹, M.K. Yakubu² and A.A. Kogo¹

¹Department of Polymer and Textile Engineering, Ahmadu Bello University, Zaria, Nigeria

²Nigerian Institute of Leather and Science Technology, Zaria, Nigeria

*Corresponding Author Email Address: hiumaru@abu.edu.ng or htrk227@gmail.com

Phone: +2348032060944

ABSTRACT

This study was aimed at examining the effect of particle size on the tensile properties and density of *Delonix regia* seed particles (DSP) filled unsaturated polyester resin (UPR) composites. The composites were fabricated using a glass mould via hand mixing, and the average DSP particle sizes of 100, 200, 300, 400, and 500 μm were used at 12 % filler loading. The effect of particle size on the tensile properties and density of DSP-filled unsaturated polyester resin was investigated. The results showed a decrease in tensile strength, tensile modulus, elongation at break, and density as the filler particle size increased. The smallest particle size (100 μm) has the highest values of tensile strength, tensile modulus, elongation at break (%) and density with the corresponding values of 35.43 MPa, 0.47 GPa, 7.09% and 1.22 g/cm³ and the largest particle size (500 μm) with values of 15.25 MPa, 0.28 GPa, 5.17% and 1.12 g/cm³ respectively.

Keywords: Density, DSP, Particle size, Tensile, and UPR

INTRODUCTION

Globally, environmental awareness and government regulatory policies have brought a fundamental change in designing composites that conform to the environment (Mohammed *et al.*, 2015; Al-Oqla and Omari, 2017; Shireesha *et al.*, 2019; Karimah *et al.*, 2021). Bio-fillers in the form of agricultural waste such as sugarcane bagasse, corn stalk, rice husk, etc. offer viable and naturally benign filling resources for polymeric materials (Adejumo and Adebisi, 2020). Plant-based natural fibres can be used in polymer composites, replacing more expensive and non-renewable synthetic fibres like glass (Swenck *et al.*, 2013) and making lightweight composites with lower density (1.2 – 1.6 g/cm³) than glass fibre (2.4 – 2.5 g/cm³) (Jawaid and Abdul Khalil, 2011; Kabir *et al.*, 2011; Mohammed *et al.*, 2015).

The use of natural fibres would decrease waste disposal problems, and ease environmental pollution (Al-Oqla and Omari, 2017; Mohammed and Attallah, 2020; Karimah *et al.*, 2021). They have high strength that can be used for many load-bearing applications. There are many potential natural resources in abundance in our environment, mostly from waste bio-mass. Recycling waste bio-mass reduces greenhouse gas emissions (Bhardwaj *et al.*, 2021; Karimah *et al.*, 2021; Bolcu and Stănescu, 2020) while also assisting in the development of new green markets, job creation, bio-energy production (Adejumo and Adebisi, 2020), and bio-composites (Bhardwaj *et al.*, 2021). Waste natural fibre recycling is a viable option for the environment (Bhardwaj *et al.*, 2021) and provides a solution to the reuse of agricultural wastes (Dong,

2017). This led to the idea of exploiting *Delonix regia* seed particles from its waste pods (bio-mass), thereby producing value-added products that are lightweight, non-toxic, low cost, and have good mechanical and biodegradable properties.

The *Delonix regia* plant is commonly found in our environment and when matured, the pods are mainly considered waste; a waste bio-mass disposed into the environment. Abulude *et al.* (2018), reported that *Delonix regia* is widely cultivated and found in parks and estates in tropical cities throughout the world. This study was aimed at the effective exploitation of waste *Delonix regia* seed particles as fillers in the modification of unsaturated polyester resin to improve its strength, and dimensional firmness and serve as an alternative and better way of disposing of waste *Delonix regia* pod.

MATERIALS AND METHODS

Materials

The raw materials used for the fabrication of the composites were Unsaturated Polyester Resin (UPR), cobalt naphthenate (promoter), Methyl Ethyl Ketone Peroxide (MEKP), and *Delonix regia* Seeds Particles (DSP).

For Sample Preparation

The *Delonix regia* pods were slit-opened and the seeds collected, crushed using a Jaw Crusher (Retsch mesh Nr.70992) and cleaned; this separated the hard seed coat (testa) from the soft inner part (endosperm/cotyledon). The seed coats were further washed with clean water to remove all the endosperm/cotyledon particles, air-dried for 48 hours, and then oven-dried at 60 °C for 48 h to ensure proper drying. The dried crushed seeds were ground with a ball mill grinding machine (Kera b.v. 057748) and then sieved to different particle sizes; 100, 200, 300, 400, and 500 (μm) using a standard sieve (Sieve-Tronic ISO 3310-1:2000, BS 410-1:2000). The microstructure observation of the seed particle sizes was studied using digital Dino – lite microscopy at 75X magnification.

Composites Preparation

Composites were prepared using different filler sizes of DSP (100 – 500 μm). The mould for each casting was properly scraped, cleaned, and well lubricated with wax to ease removal, and then foil paper was placed on. The quantity of UPR required for each sample was measured and poured into a mug, and 12 % DSP was then added and mixed. 1 wt. % of the catalyst (MEKP) was added and stirred for 2 minutes, followed by 1 wt. % of the promoter (cobalt naphthenate) being added and stirred for an additional 3 minutes. The mixture was then carefully poured into the mould (100 mm x 100 mm x 3 mm), covered with another glass sheet of the

same size, and then pressed for the proper spread of resin. The samples were cured under pressure using a load at ambient temperature for 24 hours. The composites were then removed from the mould and then cut into dumbbell dimensions according to ASTM 638.

The Tensile Test (ASTM D638)

The tensile test of the sample composites was carried out according to ASTM D638 using a tensile strength testing machine (model: TM 2101 – T7) with a maximum force of 10 KN. The dimensions of the dumbbell-shaped samples were cut from the moulded samples, and the dimensions were ascertained using the vernier callipers. The test was performed at a cross-head speed of 2 mm/min at 25 ± 3 °C. The test specimens were held in the grips of the testing machine and tightened evenly and firmly to prevent any slippage as the test commenced. The uni-axial load was applied to each end of the respective samples until they failed. The resistance and elongation of the specimens were detected and recorded by the load cell until a failure or rupture occurred (Ameh *et al.*, 2015). Stress-strain curves were plotted from the force-extension data obtained on a special graph paper during the tests, and the required mechanical properties were obtained. From the tensile test, tensile properties (tensile strength, tensile modulus, and elongation at break) were determined and recorded.

Density Measurement (ASTM D792)

The densities of the composites were determined according to ASTM D792. The mass of each composite sample was determined using an analytical weighing balance, and the volume obtained via the dimensions of each side (length x breadth x width), was accurately measured using a digital vernier calliper. The density of the samples was computed as the ratio of mass to volume (g/cm^3) (Lawal, 2019).

RESULTS AND DISCUSSION

The microscopic images of *Delonix regia* Seed Particles (DSP) used is presented in Figure 1 and all the samples show a collection of circular-flat shaped materials. This agrees with Olugbenga *et al.* (2020) who reported that the *Delonix regia* seed particles appear to look like an aggregate of roundish ball-like and flat-shaped materials.

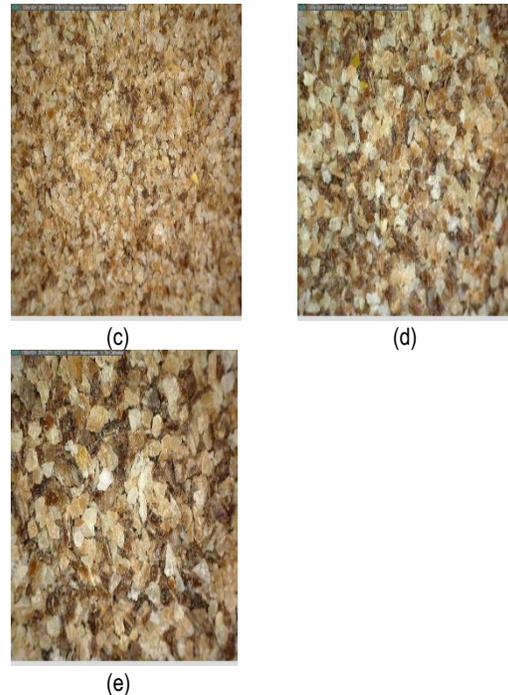
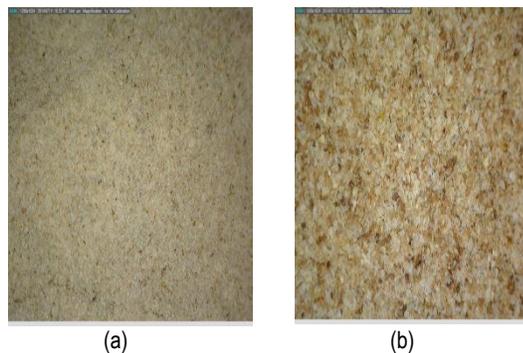


Figure 1: Microstructure images of DSP (x75 magnification): **Key:** (a) 100 μm (b) 200 μm (c) 300 μm (d) 400 μm (e) 500 μm, respectively

The Effect of Particle Size on Tensile Properties

The effect of particle size on the tensile strength of DSP-UPR composites was presented in Figure 2. The results from the graph revealed that the tensile strength decreased gradually with the increased particle size of the filler. The reasons for this could be attributed to reducing interfacial interaction between the hydrophilic nature of the fillers and the hydrophobic UPR matrix as the particle size increased. Poor adhesion of the filler matrix and the agglomeration of the filler particles at higher particle sizes may also cause a decrease in tensile strength. Okey-Onochie *et al.* (2020) reported in their findings that the decrease in tensile strength of composites is due to poor interfacial adhesion of the filler-matrix of the polymeric material. Alternatively, it may be due to interfacial voids being created when composites are subjected to tension, and these spaces act as stress concentrators and dissipating stress more rapidly, leading to deformation at reduced tensile stress. Many studies found that fillers with larger particle sizes had lower tensile strength than fillers with smaller particle sizes (Saini *et al.*, 2010; Njoku *et al.*, 2011; Onuegbu and Igwe, 2011; Nwanonenyi and Chike-Onyegbula, 2013; Nwanonenyi *et al.*, 2013; Ameh *et al.*, 2015; Kucukdogan *et al.*, 2016; Manjunatha and Ahmed, 2017; Onuoha *et al.*, 2017; Lawal *et al.*, 2019; Okey-Onochie *et al.*, 2020). However, a marginal decrease having almost similar tensile strength values was observed for 300 and 400 μm. The insignificant decrease in tensile strength values for these composites may be attributed to the improved surface area of the particles in the matrix of the latter rather than the former, which favours tensile strength. The increased interfacial area gives rise to good interfacial bonding between the hydrophilic particles and hydrophobic matrix and hence improves tensile strength by impeding a considerable decrease in tensile strength as particle size increases. The factors responsible for better mechanical

properties include the bond between the fibre and the matrix (Shireesha *et al.*, 2019). Poor bonding considerably affects the mechanical properties of natural fibre reinforced polymer composites (Al-Oqila and Omari, 2017). Literature reports the distinctive features responsible for the mechanical properties of composites, which include the physical properties of the filler (Kabir *et al.*, 2011; Al-Oqila and Omari, 2017) and its interfacial adhesion property (Mohammed *et al.*, 2015; Mochane *et al.*, 2019; Shesan *et al.*, 2019).

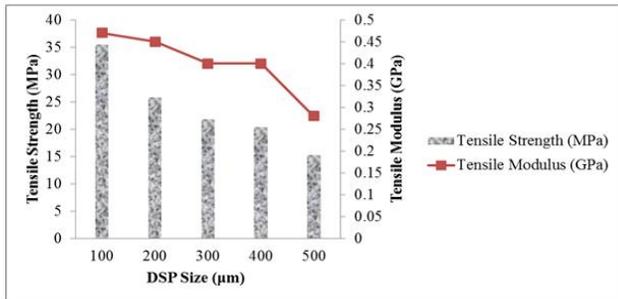


Figure 2: Effect of Particle Size on Tensile Strength and Modulus of DSP-UPR Composites

Results for the effect of particle size on the tensile modulus of DSP-UPR composites presented in Figure 2 have shown a decrease in tensile modulus as the DSP size increased. The higher tensile modulus with the smaller particles can be ascribed to the good interfacial dispersion of the fillers within the UPR matrix, resulting in better interfacial bonding/adhesion of smaller DSP within the UPR matrix. The bond between the fibre and the matrix is responsible for better mechanical properties (Shireesha *et al.*, 2019). However, the decrease in modulus with increased DSP may be due to the absence of pressure to acquire good interfacial contact and possible fibre agglomeration. Previous studies have shown that the decrease in tensile modulus with particle size is due to the creation of interfacial voids that act as stress concentrators (Okey-Onochie *et al.*, 2020). Ekwueme and Igwe (2018) reported a similar trend in which the tensile modulus of palm fibre-filled natural rubber decreases linearly with increases in filler particle size. The results obtained by Manjunatha and Ahmed (2017) on coconut shell particle-filled epoxy composites also showed a similar decline in modulus with the increase in filler particle size. Nevertheless, a similar tensile modulus of 0.40 GPa was obtained for both 300 µm and 400 µm. At this point, the results showed possible improved interfacial adhesion for the composite with a larger particle size (400 µm) when compared to a smaller particle size (300 µm). There was also a drastic drop in the tensile modulus of DSP 500 µm composites when compared to other composites studied. This may be attributed to poor mixing or uneven dispersion of the filler particles in the polymer matrix (Okey-Onochie *et al.*, 2020).

The Effect of Particle Size on Break Elongation

The effect of particle size on the percentage (%) elongation at break of DSP-UPR composites was presented in Figure 3. Results from the graph showed decreased % elongation at break of the composites with increased DSP. There was a sharp decrease in the % elongation at break from 100 µm to 200 µm and thereafter decreased marginally with further increased particle sizes (300 µm to 500 µm). A higher percentage of elongation at break was

obtained with the smallest filler particle (100 µm) and a lower value for the largest filler particle size (500 µm). The decrease in % elongation at break with an increase in particle size of DSP may be attributed to poor distribution of the particles as the size increased and hence poor bonding between the fillers and the UPR matrix. Poor bonding will considerably affect the mechanical properties of natural fibre reinforced polymer composites (Al-Oqila and Omari, 2017). Literature also reported that inconsistent distribution of the filler led to weak interfacial bonding between hydrophilic filler/fibre and polymer matrices due to the hydroxyl group in filler/fibres, thus resulting in weak mechanical and physical properties of the composites (Karakoti *et al.*, 2019; Dong, 2017). This behaviour is also similar to the trend reported by Lawal *et al.*, (2019); Ekwueme and Igwe, (2018); and Manjunatha and Ahmed, (2017).

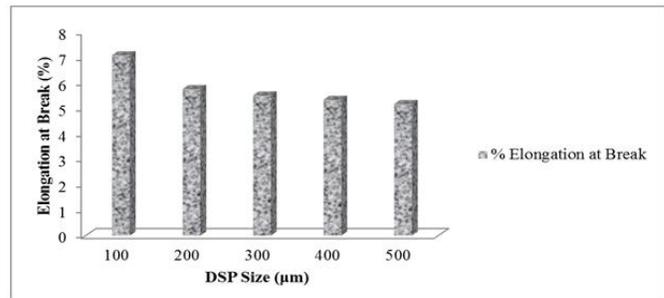


Figure 3: Effect of Particle Size on Elongation at Break of DSP-UPR Composites

Effect of Particle Size on Densities

The effect of particle size on densities of the DSP-UPR composites at 12 % filler loading was presented in Figure 4. It was observed from the graph that the density of DSP-UPR composites decreased with an increase in particle size of the filler. The decrease in density of the composites with larger particles may be attributed to the poor contact area between the filler and the matrix, which caused poor interaction of the fillers within the polymer matrix, which led to more porosity, causing increased volume and, accordingly, decreased density. Therefore, a further increase in the particle size of the filler reduces its contact area within the UPR matrix, which increases porosity and therefore decreases the density of the composites. Mehdi *et al.* (2009) posit that density is related to porosity, and thus larger particles abridge the contact area between the particles, therefore leading to porosity. Several researchers reported similar trends of decreased density with increased particle size (Eichie and Kudelinbu, 2009; Mehdi *et al.*, 2009; Tercia *et al.*, 2012; Faruh Nordyana *et al.*, 2013; Lawal, 2019).

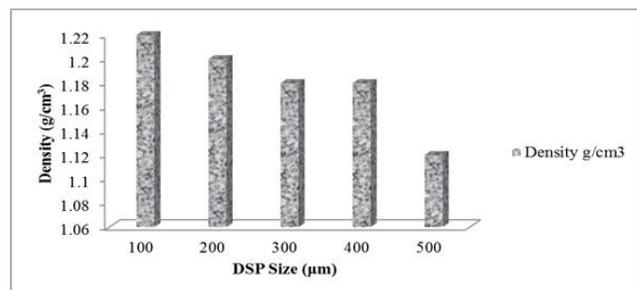


Figure 4: Effects of Particle Size on Densities of UPR-DSP Composites

Nevertheless, the particle sizes of 300 μm and 400 μm showed no difference in densities with increased filler size, both having identical densities of 1.18 g/cm^3 respectively. This may be attributed to better interaction between the fillers of the larger particles within the matrix, thus increasing its contact area (lower surface area), thereby reducing voids and agglomeration that may likely occur due to larger particle size affecting porosity. Subsequently, a decrease in volume favours density. Ameh *et al.* (2015) also reported a similar trend.

Conclusion

The *Delonix regia* seed particles filled with unsaturated polyester resin composites were fabricated, and the effect of particle size on tensile properties and density of the composites was studied. The results showed that the tensile strength, tensile modulus, elongation at break, and density of the composites decreased with increased particle size (100 – 500 μm) of the fillers. From the results obtained, the composites have shown relatively good tensile properties and low weight. This study can be considered as a feasible source of effectively exploiting *Delonix regia* by-products as a suitable resource for the fabrication of composite materials which can be used for particle board, partition board, automobile interior parts, and interior wall tiles.

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