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# Original article

# Experimental investigation and theoretical prediction of tensile properties of *Delonix regia* seed particle reinforced polymeric composites



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#### ABSTRACT

Possibility of using *Delonix regia* (*Dr*) seed particles as reinforcement for production of new polymeric composites has been studied. Dried *Dr* seeds were processed using a disc grinder and then classified. *Dr* particles of 105  $\mu$ m average size were incorporated into recycled low density polyethylene (RLDPE) up to 10 wt% and RLDPE/*Dr* particle composites were produced through compounding and compressive moulding technique. Effects of *Dr* particle addition on morphology and the tensile properties of the produced composites were investigated. The composite tensile properties were confirmed and validated using mono-variate regression model involving full factorial design of one factor-five levels. Results obtained indicated presence of void within the composite grain which impair the tensile strength and strain of the RLDPE composite at 10 wt% of *Dr* seed particle addition. About 300% and 41% maximum increments in tensile strength and strain were noticed at 4 wt%. The developed models show that wt% of *Dr* particles has significantly statistical influences on both tensile strength (*P* = 0.00827) and strain (*P* = 0.01756). About 93% and 89% response prediction by the models shows that the models exhibit fit goodness and they are appropriate for confirmation of experimental tensile properties. Therefore, this study has given birth to a new polymeric composite for engineering applications.

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#### 1. Introduction

Delonix regia seed particles have been synthesized to serve as reinforcement for polymer in an attempt to produce composites for engineering structures. They are renewable sources for carbon which are the alternative to conventional carbon, glass and aramid fillers. Particles have been sourced from forestry, agro and industrial wastes (Agunsoye and Aigbodion, 2013; Bello et al., 2015, 2017). Currently, sourcing of particulate filler from forestry is highly discouraging because of the consequence of deforestation, leading to the global warming. However, agricultural products release a huge amount of potential materials for particle production. Those materials can be accessed at a very low cost. They are renewable and can be processed easily with less expensive equip-

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ment and their usage as a filler does not affect food supply for human consumption. Lightness of their particles make them ideal fillers in a polymer or metal for composite fabrication. Although many works have been reported on particles synthesized from the agro materials for composite production (Bello et al., 2015, 2017), the Delonix regia (Dr) seed is very rare. Dr seeds are environmental littering wastes which are obtainable from the burst Dr pods. They usually fall from the trees especially during the dry season. The Dr trees are short, stout, smooth, gray-coloured trunk trees with characteristics saint scent. They bear broad umbrella feathery leaves with moderate lighting shades (Brown, n.d.). They belong to Plantae kingdom, Fables order and Fabaceae family. It is a very common tree that grows across the globe. Processing of Dr seeds for particle or fibre production for polymer matrix composite fabrication will not only remove wastes from tree harbouring places; create wealth from wastes but also gives birth to new reinforcing materials.

Applications of polymeric composites have been emerged in different engineering sectors which include marines, automobiles, aerospace and domestics. The composites have been built using a polymeric matrix and a reinforcement from either synthetic and natural fibre. However, because of anisotropy properties associated with fibre reinforced composites and difficulty in their fabrications,

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researches have been focusing on reinforcements of polymers with the particles to develop different forms of isotropic particulate reinforced composites which can replace their fibre reinforced composite counterparts (Jiang et al., 2010; Kim et al., 2012; Mohd Hirmizi et al., 2012; Paul et al., 2009; Senthilkumar et al., 2012; Shashikanta et al., 2015; Srivastava and Verma, 2015; Danasabe, 2018).

Coconut shell particle reinforced polypropylene composites were developed through softening/compaction techniques. Both alkaline treated and untreated coconut shell particles each at 80 and 150  $\mu$ m were used as reinforcements (Agunsoye et al., 2014). Results of their experimental investigations reveal greater mechanical properties in respect of alkaline treated coconut shell reinforced composites.

Atuanya et al. (2011) developed recycled low density polyethylene (RLDPE)/wood composite boards via compression technique at varied temperatures from 140 to 180 °C and pressures of 30– 40 kg/cm<sup>2</sup> for duration between 7 and 13 min. Morphological examination and investigation of mechanical properties show homogenous distribution of wood particles within the RLDPE which is attributed to enhancement in mechanical properties of the composites (Atuanya et al., 2011).

Mechanical properties of SiC-treated recycled high density polyethylene (RHPE) was studied. Results indicated that the addition of SiC to RHPE gave rise to improved tensile and impact strength, greater modulus of elasticity and increased stiffness. However, there is a reduction in the ductility of the composites (lbitoye et al., 2013).

A systematic approach to evaluate the physio-mechanical properties of bean pod ash particles reinforced recycled polyethylene (RLDPE) polymer based composites was presented (Atuanya et al., 2014a,b). RLDPEs were filled with 75  $\mu$ m sized bean pod ash particles up to 30 wt% at 5% interval. The results indicated the fair distribution of the bean pod ash particles within the RLDPE matrix and improvement in the mechanical properties.

Barley husk, coconut shell and soft wood reinforced polypropylene composites were produced at 40 wt% of particle addition using a high speed mixer/injection moulding technique. Results depicts that thermal stabilities of barley husk and coconut shell are 235 and 195 °C respectively and they are comparable with soft wood whose thermal stability is 245 °C. Cellulose and starch contents of barley and coconut shell are 50% and 34%, respectively. Tensile strength of barley reinforced composites is 10% greater than that of soft wood. Charpy impact strengths in respect of coconut shell and barley husk are 25% and 35% greater than that of the soft wood reinforced composites (Bledzki et al., 2010).

Electrical properties of aluminium powder reinforced epoxy composites were studied by (Jafar et al., 2011). Epoxy resin EP 10 cured with HY 931 hardener in ratio 3:1 used as a matrix was filled with 10–70  $\mu$ m sized aluminium powders from 20 to 50 wt%. Thereafter, measurement of samples' conductance and capacitance was made at room temperature and frequencies between 10<sup>2</sup> and 10<sup>6</sup> Hz using samples which have been sandwiched between two gold electrodes and a programmable automatic LRC bridge (PM60304 Philips). It was observed that alternating current conductivity increased with an increment in wt% of aluminium powder additions (Jafar et al., 2011).

Wear behaviour of propylene matrix composites was studied under different applied loads and wt% of carbonised bone particles. The composites were produced by varying contents of carbonised bone ash particles from 5 to 20 wt% through compounding and compressive moulding process. It was observed from the experimental investigation a decrease in the wear rate with an increase in wt% of carbonised bone particle additions (Asuke et al., 2014).

In this current study, inner surface of the *Dr* seed has been examined and the particles obtained from the processing of the *Dr* seed have been incorporated into RLDPE matrix for development of polymeric composites. The aim is to study effects of the *Dr* particles on the morphological and tensile properties of the RLDPE. Also, experimental tensile properties are confirmed using nonlinear regression model involving full factorial design of one factor-five levels. Polymeric composites fabricated from polyproethylene and agro-filler such as *Dr* particles will be isotropic in nature if an even distribution of the *Dr* particles within the polyethylene matrix is achieved. Such composites can be used in home furniture, composite tiles, interior, front and rear parts of automobiles.

# 2. Materials and methods

Delonix regia seed and RLDPE are the major materials used in this work. Dr seeds were obtained from Dr trees within University of Lagos. RLDPE were packed from Waste Management Centre, University of Lagos Nigeria. Dried Dr pods (see Plate 1) were split into two to release the Dr seeds. Then, the seeds were rinsed in water and dried in a Gen lab oven at 150 °C for 6 h. Moreover, the dried seeds were pulverized using a disc grinder. The Dr particles obtained were sized using a set of sieve arranged in descending order of grain fineness in accordance with (Bello et al., 2015, 2017; Hassan et al., 2015). The Dr particles collected in 105 µm sieve were used to fill RLDPE from 4 to 10 wt% at 2% interval. Inner surface of Dr seed was examined using scanning electron microscope (SEM, ASPEX 3020) and the elemental composition of the Dr sample was determined using energy dispersive X-ray spectroscope (EDX) attached with the SEM. RLDPE/Dr particulate composites used in this work were produced in accordance with (Agunsoye and Aigbodion, 2013). In the typical production, mixture of RLDPE and Dr particles were compounded using corotating twin extruder (APV Baker, model: MP19PC). The rotational segmented screw speed of 60 rpm and temperature of 120 °C were applied for the processing of RLDPE/Dr particle mixture. The RLDPE/Dr particles blend was compressed/compacted in a metal die mould at 1200 N/mm<sup>2</sup> to form a composite sample. Percentage wt. of Dr particle addition was increasing from 4 up to 10 wt% at 2% interval. The mixing speed and processing temperature were maintained constant throughout the experimental exercises. Morphology of the  $10 \times 10 \times 4 \text{ mm}^3$  representative samples of the produced composite materials was examined using the SEM. A dog bone shape tensile samples with gauge length of 250 mm, width of 6 mm and 6 mm thickness were cut from each sample and their tensile properties were examined using Instron universal testing machine, model 3369 at cross head speed of 0.05 mm/s equivalent to a strain rate of 0.0002 1/s, in accordance with ASTM D 638 (Ashori and Nourbakhsh, 2010). Plate 2 presents the photographs of a set of tensile samples at one level of reinforcement before, during and after tensile investigation.

#### 3. Results and discussion

#### 3.1. SEM micrographs

Morphology and EDX elemental composition of the *Dr* seed is presented in Plate 3. It is observed that the seed has three layers: outer, middle and inner layers. The outer layer is a continuous thick region with alternate white and black lamellar. The middle layer is dark and appears goose and dimple with discontinuous polygonal shape. The EDX chemical analysis of the middle layer presents two highest peaks indicating the presence of carbon and oxygen as the major elements. The remaining short peaks indicate other elements (metals) present in the analysed region as shown in the column chart inserted in Plate 3. The inner layer has an imper-



Plate 1. Delox regia: (a) tree (b) split pods (c) seed (d) pulverized seed.



Plate 2. RLDPE/4 wt% Dr composite tensile sample(s), (a) before tensile investigation, (b) during investigation, (c) after investigation.



Plate 3. SEM/EDX of Delox regia.

fect rectangular/tubular structure with alternate white and black colour. The elemental composition of the inner layer is similar to that of the middle layer except that metals present have higher count scores. Generally, findings obtained from chemical analysis of the *Dr* seed is that the seed contains mainly of organic compounds due to presence of carbon and oxygen with little inorganic compounds.

Micrographs in Plate 4a–c present the morphology of the unfilled RLDPE and RLDPE/*Dr* particulate composites at 4 and 10 wt% and their elemental compositions, respectively. It is evident from Plate 4a–c, differences in the geometry of the RLDPE/*Dr* composite micrographs and that of unfilled RLDPE polymer. This difference could be ascribed to *Dr* particle additions. Morphologies of RLDPE/*Dr* composite in Plate 4b and c, reveal homogenous dispersion of the *Dr* particles within the RLDPE matrix without a sign of any particle agglomeration within the matrix or particle detachment or pullout from the RLDPE matrix. This indicates a good chemical interaction between the filler and the reinforcement leading to occurrence of chemical bonding/interface between the *Dr* filler and the RLDPE matrix rather than Van dal Waal's attraction. Since the strength of interfacial bonding is one of the signifi-

cant factor that influences the mechanical properties of the particle reinforced composites, joining of *Dr* particles with RLDPE polymer molecules (good interfacial bond) is evidence supporting good tensile performance of the RLDPE/*Dr* composites under tensile investigation. However, impressions noticed in the micrograph in Plate 4c may represent some defects inherent in the composite molecules during processing. Presence of these empty regions within the polymer/composite may create region of discontinuity which can impair the mechanical properties of the polymer/composite. EDX spectrographs indicates presence of C and O as shown in Plate 4b and c. Presence of O may be linked with *Dr* particles added to the polyethylene which contains O as one of its elemental composition.

#### 3.2. Tensile properties

Average tensile strength and elongation of four different samples analysed at each level of reinforcement is presented in Fig. 1. The inserted error bars indicate the standard deviation of the tensile properties. Observation in Fig. 1 is an enhancement in



Plate 4. SEM/EDX: (a) unfilled RLDPE, (b) RLDPE/4Dr composites (c) RLDPE/10Dr composites.



Fig. 1. Tensile properties of RLDPE/Dr particulate composite.

the tensile properties of the RLDPE/Dr particulate composites. Tensile strength increases from 5.327 N/mm<sup>2</sup> of the RLDPE polymer to 21.424 N/mm<sup>2</sup> of RLDPE composite at 4 wt% of Dr seed particle addition. The maximum enhancement in the tensile strength is equivalent to about 300% increase. The increment in the tensile strength is continuous up to 8 wt% but the degree of enhancement is lower than that at 4 wt% of Dr particle addition. Similar observations have been made by (Ashori and Nourbakhsh, 2010) who filled polypropylene with oak and pine fibers in ratio 60:40 (w:w), in each case. About 14% and 15% improvement in tensile strength has been reported, respectively by (Ashori and Nourbakhsh, 2010). Similarly, (Agunsoye and Aigbodion, 2013) unveiled 14% and 40% enhancement in the tensile strength at 20 and 30 wt% additions of uncarbonised and carbonised bone particles to polyethylene. Close findings have also been reported in literature by (Asuke et al., 2012; Atuanya et al., 2011, 2014a,b; Dhaliwal et al., 2013; Rajeev et al., 1997). Strength of particle reinforced composites depend on size of the particles, homogenous dispersion of the particle within the matrix, particle wt% additions, interfacial adhesion between the matrix and reinforcement and stress transfer at the interface between the matrix and reinforcements. Since average Dr particle size of 105 µm is constant throughout the experimentation, the possible proposition to the strength enhancement is the homogenous dispersion of the Dr particles within RLDPE matrix which leads to their fortified bonding with the matrix molecules. The presence of strong bond/interface between the matrix molecules and reinforcement particle causes effective stress transfer from the matrix to the reinforcements which stiffens the matrix and enhance the load carrying ability of the entire RLDPE/Dr composites. Even distribution of Dr particles throughout the entirety of the matrix enhances their capabilities to delays the deformations of the composites which can lead to particle detachment from the matrix molecules through cavitation process. The good compatibility between the Dr particles and RLDPE matrix increases their interaction causing strong adhesion at interface between the matrix and the reinforcements which is a prerequisite to better performance of the composite under tensile loading. The lower enhancement in tensile strength above 4 wt% can be explained by the fractographies of the fractured surfaces of the tensile samples (4, 8 and 10 wt% Dr additions) presented in Plate 5a-c. Each micrograph in Plate 5a-c is characterised with composite strands which are produced due to composite elongation resulting from tensile straining experienced by each sample during the tensile analysis. Also, voids of different magnitudes are very apparent in Plate 5b and c while Plate 5a shows tomography of RLDPE/4% Dr composite fractured surface with highest structural integrity

among others. RLDPE/4% Dr composite's pull out or fracture occurred during tensile investigation without much pronounced cavitation. Presence of voids in Plate 5b and c may be connected with defects inherent in the composite during compaction process. These defects aided the cavitation process during the tensile analysis leading to formation of voids. These voids create empty spaces within the composites which reduce composite resistance to yielding to deformation from rapidly increasing external pressure/stress constraint resulted from the rate of applied uniaxial tensile load to the cross sectional area of the examined RLDPE/Dr composite samples. The voids act as stress raisers through a reduction in the cross sectional areas of the tested composites. This causes composite necking to occur in a rapid manner which is followed by fracture of the composites at strength level which is much lower than the normality/expectation. Moreover, a progressive enhancement in tensile strain is noticed up to 8 wt%. About 41% increment is noticed at 4 wt% Dr particle filling level where maximum tensile strength enhancement is found, although about 145% greatest tensile strain increase is observed at 8 wt% Dr particle addition. This improvement can be associable with fair interfacial adhesion between the matrix and filler which gave room for prolonged elongation at reduced stress before the occurrence of the composite pull out. When the filling level of Dr particles increases to 10%, the improvement in tensile elongation drops to 95%. The decrease in the tensile improvement may be linked with the approach of matrix saturation which implies that there are freely existing Dr particles which are not well bonded with the matrix. During stretching, those particles interfere with composite flow through mobility from their respective locations within the matrix. This creates empty micro spaces/voids leading to cavity formation within the composites (see Plate 5c). The cavitation process aids the detachment of polymer grain at a shorter elongation than expectation. Similar observation has been made in literature (Akram et al., 2016). The general inference is that incorporation of Dr seed particles to RLDPE matrix improves both tensile strength and strain which is very uncommon with many agro particles that have been reported in the literatures. The exceptional behaviour of the Dr particles may be linked with balances between hard/stiff elements such as C, Zr, Ti, Fe and soft/ductile elements such as Al, K, Ba and Ca characterising the *Dr* particles (see Plate 3).

# 3.3. Fitting of tensile properties

Results of experimental investigations on tensile strength and strain were confirmed and then validated using statistical modelling. Both tensile strength and strain were fitted through nonlinear mono-variate regression models using full factorial design of experiment which involves one dependent variable/factor (response) and independent variable (predictor) at five different levels. This is equivalent to 5<sup>1</sup> (5) designs of experiment in accordance with (Pallant, 2005; William, 1998). Each of the model was built at  $\alpha$  = 0.05, meaning 95% confidence level, significance and goodness of fit were diagnosed using analysis of variance (ANOVA) and least square criteria. Matching of experimental and theoretical tensile strength on Fig. 2a shows a fair model prediction of the experimental data. ANOVA on Table 1 shows that are 5 degrees of freedom (DF) characterising this model, meaning that five different matrices were solved in the prediction of response and its characteristic standard errors (William, 1998). F and P values are 35.17 and 0.00827 respectively. With this high F value, there are very low chances that noise could occur in the response prediction (Atuanya et al., 2014a, 2014b). P value (0.00827) < 0.05 reveals that wt% of Dr particle addition is a statistical significant factor that influences the tensile strength of the RLDPE composite while keeping other factors constant. In Eq. (1) revealing the model function, it is observed that both wt% of Dr particle additions and its interaction



Plate 5. Fractographies of RLDPE/Dr composites at (a) 4 wt% (b) 8 wt% (c) 10 wt%.



Fig. 2. (a) Matching of experimental and theoretical tensile strength (b) residual of tensile strength.

# Table 1ANOVA/statistics of tensile strength.

		DF	Sum of squares	Mean square	F value	Prob>F	Statistics
Tensile strength	Model Error Total Adj. <i>R</i> -square	2 3 5	1085.53 46.29 1131.82	542.76 15.43	35.17	0.00827	0.93183

with each other in the second degree  $(wt%^2)$  play vital role in the prediction of the response (tensile strength). However, wt% significantly enhances the tensile strength of the composite and its  $\beta$  value of 8.01659 indicates strong bonding between the *Dr* particles and the RLDPE matrix. However, presence of  $(wt%)^2$  degrades the tensile strength because of negative value of its  $\beta$  coefficient. At 4 wt% filler level,  $(wt%)^2$  will be equivalent to 16 wt% addition, meaning that at this level, the model predicts poor adhesion between the matrix and the reinforcement. Possible explanation for this is that particle packing density is very high and very close distance between them creates inability of the RLDPE matrix to join all of them together. This results in domains of many freely existing particles that mobile freely when the composite is under tensile investigation. These particles create vacancies or empty

spaces on leaving their location. Presence of these micro defects within the RLDPE accounts for reduction in the tensile strength as the *Dr* particle addition rises above 4 wt%. Similar explanation has been offered in literature (Agunsoye and Aigbodion, 2013). However, the regression coefficient of determination for this model is 0.93183 meaning that this model can confidently explain 93.183% of the response. The remnant accounts for residuals (differences between the experimental and predicted values) as shown by the scatter plot in Fig. 2b. This is a fair prediction comparing with what has been reported in the literatures (Pallant, 2005). Therefore, this model exhibit good predictability of the experimental tensile strength of RLDPE/*Dr* composites. The limitation of the developed model is that it cannot be used to predict the tensile strength of the unfilled polymer.



Fig. 3. Confirmation and validation of experimental tensile strain using second degree polynomial.

Table 2ANOVA/statistics of tensile strain.

		DF	Sum of squares	Mean square	F value	Prob>F	Statistics
Tensile strain	Model Error	2 3	37.70509 2.73171 40.4268	18.85255 0.91057	20.70412	0.01756	
	Adj. <i>R</i> -square	5	40.4368				0.88741

Tensile strength  $(T_s) = 8.01659 (wt\%) - 0.75964 (wt\%)^2$  (1)

In addition, results of theoretical predictions of tensile strain of RLDPE/Dr composites are presented in Fig. 3. P value for this current model is 0.01756 (see Table 2). Since the model was built at  $\alpha$  = 0.05 that is 95% confidence level; *P* value, 0.01756 indicates that wt% of Dr particles is significantly influencing the tensile strain of the RLDPE/Dr composite. The same explanation offered in respect of  $\beta$  values in Eq. (1) also affirms the behaviour of wt% and its interaction in the second degree in the prediction of the tensile strain as shown in Eq. (2). Passage of the modelled curve through the origin declares the limitation of this model in the RLDPE polymer tensile strain prediction and its maximum point just beyond 8 wt% reveals decline in the tensile strain. This shows good agreement with the experimental results. Therefore, the model offers excellent estimation of the composite tensile strain theoretically (see Fig. 3a). The adjusted R-square for this model is 0.88741 implying that the ratio of the observed to the expected cumulative probabilities approaches a unity. The slight deviation/ variance from the unit value explains the residuals depicted in the scatter plot in Fig. 3b. Explanation of about 89% response in the null hypothesis by this model is a fact supporting the appropriateness of this model for confirming and validating the tensile strain of the RLDPE/Dr polymeric composites.

Tensile strain 
$$(T_{sn}) = 0.79778 (wt\%) - 0.04697 (wt\%)^2$$
 (2)

#### 4. Conclusion

Finally, new polymer composites through incorporation of *Dr* particle into RLDPE have been developed using compounding and compressive technique. Maximum enhancement in tensile strength has been recorded at 4 wt% of *Dr* seed particle addition through experimental investigation. The statistical model exhibits a good predictability of the tensile properties. Hence, a novel approach to wealth creation through waste recycling has been disclosed.

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