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Influence of particle size and loading variations on the tensile properties of *Newbouldia laevis* fibre particle reinforced polyester composite

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ABSTRACT

This study investigated the tensile properties of *Newbouldia laevis* fibre particle-reinforced polyester composites as potential sustainable eco-friendly replacement materials for synthetic fibres in lightweight polymer composite applications. The bulk density of the fibre was found to be 0.098 ± 0.005 g/cm³. The tensile properties of fibre particle polyester composites fabricated on the basis of varying fibre particle sizes and fibre particle loading were examined. The results revealed an increase in the tensile and yield strengths increased as the particle size decreased. Additionally, the tensile strength, yield strength, and elastic modulus decreased as the fibre particle loading increased. Furthermore, the maximum tensile and yield strengths obtained from the 10 wt% fibre particle-loaded composite were 31.35 ± 1.613 N/mm² and 31.277 ± 1.621 N/mm², respectively. The results obtained from this study reveal that *Newbouldia laevis* fibre particles are suitable reinforcement biomaterials for lightweight composite applications.

1. Introduction

At present, there is an increase in global awareness of the need to develop eco-friendly composite materials for lightweight engineering applications. This has led to increased research in the field of natural fibre sources as an alternative to synthetics fibre for the reinforcement of polymer composites; this is due to the harmful effects of the use of synthetic fibres on human health and the environment [1,2]. Natural products from plants such as groundnut shells, wheat husks and coconut husks, which are regarded as natural fibres or biowaste materials, are now shredded to particle sizes (natural particulates) and used as reinforcing fillers for polymer composites [3,4]. These natural plant products are renewable and biodegradable, which makes them safe materials for use in our environment. In addition, composites of these natural fibres are widely acceptable because of their strength, light weight and cost of production compared with the use of synthetic fibres such as fibreglass and carbon fibres, which are petroleum-based materials [5,6]. Many studies have reported improvements in the tensile properties of natural fibre or fibre particle reinforced composites by varying factors such as fibre particle size and percentage content. Bhaskar and Singh (2013) investigated the effects of the percentage weight content on the physical and tensile properties of coconut shell particle-reinforced epoxy composites. The results revealed that the density, ultimate strength, modulus of elasticity and percentage elongation decrease as the percentage weight of the shell particle increases [7].

Seth *et al.* (2018) examined the effects of particle size and loading on the tensile and flexural properties of a Doum palm shell reinforced composite and reported that better properties of the composite were obtained for smaller particles of the fibre [8]. Umaru *et al*. (2022) examined the effect of particle size on the tensile properties and density of Delonix Regia seed particle-filled polyester composites and reported that the tensile strength, tensile modulus, elongation at break and density decrease as the filler particle size increases from 100 µm to 500 µm [9]. Rashed *et al. (*2008) investigated the impact of the fibre size and percentage on the tensile strength of jute fibre-reinforced composites. The results revealed an increase in tensile strength as the fibre size and

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percentage increased and then decreased [10]. Wong *et al.(*2022) investigated the impact of the percentage content of oil palm empty fruit bunch fibres (OPEB) on the mechanical properties of reinforced composites and reported that the tensile strength increased by 17.4% as the content increased to 2.5 wt% [11]. Hanana and Rodrigue (2020) investigated the effect of particle size on composites of wood fibres and polyethene and reported that the tensile modulus increased as the particle size increased from 250 µm to 500 µm and that the tensile strength increased as the particle size increased from 250 µm to 355 µm [12]. Pandya *et al*. (2019) studied the effect of the percentage loading of waste rubber particles on the tensile and flexural strengths of sisal fibre-reinforced epoxy composites and reported that the maximum tensile strength and flexural strength were achieved at 5 wt% waste rubber particle loading but decreased beyond the 5 wt% waste rubber particle loading [13].

Vignesh *et al*. (2021) studied the effects of the fibre content on the tensile properties of Indian mallow fibre/polyester composites (MFPs) by varying the fibre content from 10 to 50% and reported optimum tensile strengths and moduli of 46 MPa and 3.56 GPa, respectively, at a 50 wt% fibre content, whereas the elongation at break was 1.39% [14]. Ismail *et al. (*1997) studied the effects of filler content and size on the tensile properties of oil palm wood flour-reinforced epoxide natural rubber composites and reported that the tensile strength and tensile modulus are high at small filler sizes, and that the tensile strength and elongation at break decrease as the filler content increases [15]. Oghenerukevwe and Uguru (2018) investigated the effect of the hardwood sawdust/oil bean pod shell filler % content on the mechanical properties of reinforced epoxy hybrid composites. The results revealed that as the filler loading increased to 50%, the tensile strength increased, and the elongation increased to 40% and then started to decrease [16]. However, the use of *Newbouldia laevis* fibre particles as reinforcements for resin matrices in composite fabrication is less known or explored. The *Newbouldia laevis* plant, a plant well known as Ogirisi in the Eastern Region of Nigeria, is an important plant native to tropical Africa. It has a high crude fibre content and is widely planted and researched in the field of medicine because of its health benefits [17,18]. In this study, fibres from *Newbouldia laevis* plants were used as a novel material; they were extracted, treated, ground, sieved to particle sizes and used as reinforcements for the fabrication of the polymer composite. Finally, the tensile properties were determined on the basis of varying particle sizes and loading of the fibre particle-polymer composite.

2. Materials and methods

2.1 Fibre Extraction and Processing

The fibre was obtained from the *Newbouldia laevis* plant (Figure 1(a)) in Enugu State of Nigeria. The sodium hydroxide (NaOH) pellets, polyester resin (matrix), methyl ethyl ketone peroxide (MEKP) catalyst, cobalt naphthenate accelerator and wax release agent were purchased from Enugu State in Nigeria.

The plant stem was first harvested and soaked in water (retting) for 28 days (Figure 1(b-c)). This was followed by manual extraction of the fibres from the outer cuticle and the epidermal layer of the stem. The surface modification of the fibre was performed by soaking the fibres in sodium hydroxide solution (3 w/v%) for 1 hour [19]. The fibres were then rinsed in water to remove excess NaOH from the fibre surface before drying in sunlight for 48 hours, as shown in Figure 1(d).

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2.2 Fibre Particle Preparation

During tensile testing of a single fibre, the assumption of circularity of the fibre is fairly true for synthetic fibres but incorrect for natural fibres [20]. Natural fibres are made up of smaller networks of fibres, which this results in irregular shapes and non-uniform cross-sectional areas due to varying fibre diameters along the fibre length. Hence, during the testing of these fibres, failure may occur at any weak section along the fibre length. This makes accurate determination of the actual value from mechanical tests quite difficult because of large discrepancies in the values obtained.

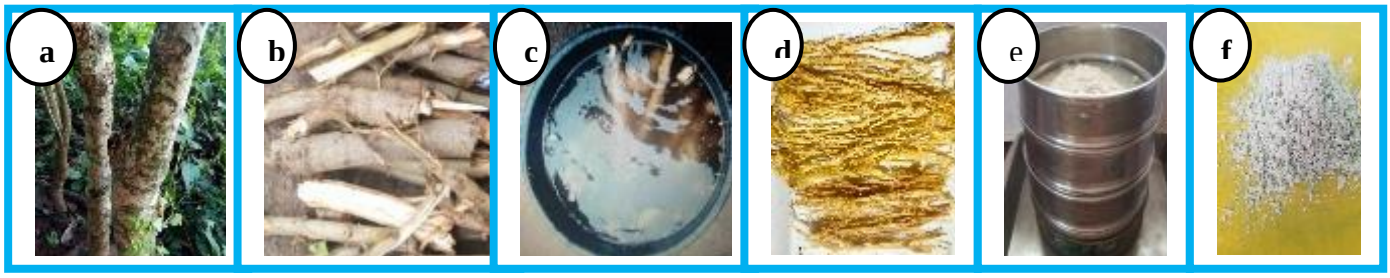

Figure 1:(a) *Newbouldia laevis* plant; (b) harvested stem; (c) water retting;(d) treated fibres; (e) sieving process; (f) fibre particle

These large discrepancies in values obtained from tests results in large standard deviations of the fibre properties which affects the effective use of these composite materials. As part of the solution to this drawback, reducing the fibre to particle sizes (fillers) increases its surface area and ensures uniform dispersion during mixing with the resin matrix, which results in equal strength in all directions of the manufactured composite, unlike fibres, which offer unidirectional reinforcement in a composite. Research has revealed that natural fibre particle-reinforced composites have greater tensile, compressive and flexural strengths than short- and long-fibre-reinforced composites do [21, 22]. The use of fillers as reinforcements reduces the cost of composite fabrication and increases the stiffness of the composite material [23]. The fibre particles were prepared by grinding the sun-dried fibres to powder

via a laboratory grinding machine and then sieved through standard sieves with sizes of 180 µm (BSS 85), 250, 355 and 500 µm, as shown in Figure 1(e-f).

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2.3 Polyester and fibres particle densities

A manufacturer-specified polyester matrix with a density of 1.114 g/cm^3 was used for composite fabrication. The bulk density was determined according to the ASTM D7481-09 standard method by weighing the fibre particle sample to a marked level in a measuring cylinder. This process was repeated eight times, and the average value was taken. The bulk density was determined via Eq. (1):

$$\partial_f = \frac{W_2 - W_1}{V_f} \quad (1)$$

where ∂_f is the bulk density of the *Newbouldia laevis* fibre particle, W_1 is the weight of the empty measuring cylinder (g), W_2 is the weight of the measuring cylinder and fibre (g), and V_f is the volume of fibre (ml) [24].

2.4 Composite fabrication

The hand lay-up method was used for this fabrication process. This was done in two stages; First, the composite was fabricated on the basis of varying fibre particle sizes and subjected to a tensile test. Then, composites based on varying fibre loadings (resin-fibre weight ratios) were fabricated using the particle size that gave the maximum value of tensile strength from the former tensile test.

For the first stage, appropriate quantities of each of the sieved *Newbouldia laevis* fibre particle sizes (180 µm, 250 µm, 350 µm and 500 µm) were mixed with polyester resin at a weight ratio of 30:70 and stirred using a mixer machine at an Rpm of 1400 for 10 minutes. Appropriate amounts of the cobalt naphthenate accelerator and MEKP catalyst were added, and the mixture was further stirred. The mixture was then applied to the mould and allowed to cure at room temperature for 72 hr. It was demoulded and trimmed to the sample size according to ASTM standards (Figure 2). The samples were then subjected to a tensile test.

Furthermore, the fibre particle size from the composite, which provided the maximum tensile strength, was used for fabricating composites by mixing with polyester resins at weight ratios of 10:90, 20:80, 30:70 and 40:60. The mixtures were stirred and appropriate quantities of cobalt naphthenate accelerator and MEKP catalyst were added and further stirred before being applied to the mould. The composite samples were then allowed to cure at room temperature for 72 hrs, trimmed to size according to ASTM standards and subjected to tensile testing.

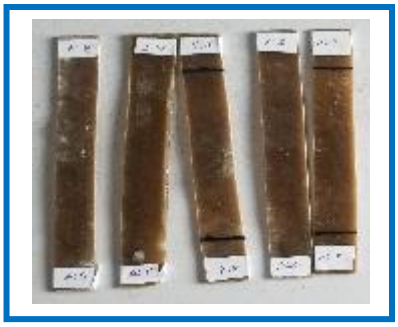

Figure 2: Composite samples

2.5 Tensile tests

The test was performed according to the ASTM D3039 standard method on a universal tensile testing machine (Testometric) of 50 kN. For the test, the samples of the *Newbouldia laevis* fibre particle composites with dimensions of 150 × 25× 4 mm were clamped on the jaws of the machine with a gauge length of 120 mm and tensioned at a crosshead speed of 10 mm/min. The test values of the force and extension were recorded and used to calculate the ultimate tensile strength (σ_T), yield strength (σ_y), strain (ε) and elastic modulus (E) via Eqs. (2), (3), (4) and (5). Additionally, the elongation at break was determined from the test.

$$\sigma_T = \frac{F_T}{W_C t_C} \quad (2)$$

$$\sigma_y = \frac{F_y}{W_C t_C} \quad (3)$$

$$\varepsilon = \frac{L_1 - L_0}{L_0} \quad (4)$$

$$E = \frac{\Delta\sigma}{\Delta\varepsilon} \quad (5)$$

where F_T is the maximum load (N), W_C is the width of the composite sample, t_C is the thickness of the composite sample, F_y is the yield force, L_1 is the new length of the composite sample, L_0 is the gauge length of the sample, $\Delta\sigma$ is the gradient stress in the elastic region (MPa) and $\Delta\varepsilon$ is the gradient strain in the elastic region.

The composites with varying particle sizes and particle loadings were subjected to tensile tests; in each case, five samples were tested, and the average values were determined [25, 26].

The results from the experiment were statistically analysed. The standard deviation (S) and 95% confidence interval (C) were calculated via Eqs. (6) and (7).

$$S = \sqrt{\frac{\sum_{i=1}^{n}(x_i - \ddot{X})^2}{n-1}} \quad (6)$$

$$C = \ddot{X} \pm 1.96 * \frac{S}{\sqrt{n}} \quad (7)$$

where x_i represents the sample data and $\ddot{X}$ represents the sample mean.

Furthermore, regression analysis via Pearson product-moment correlation using Minitab 16 software was performed to determine the relationship from the analysis [27].

A summary of the procedures for fibre extraction, composite fabrication and tensile testing is shown in Figure 3.

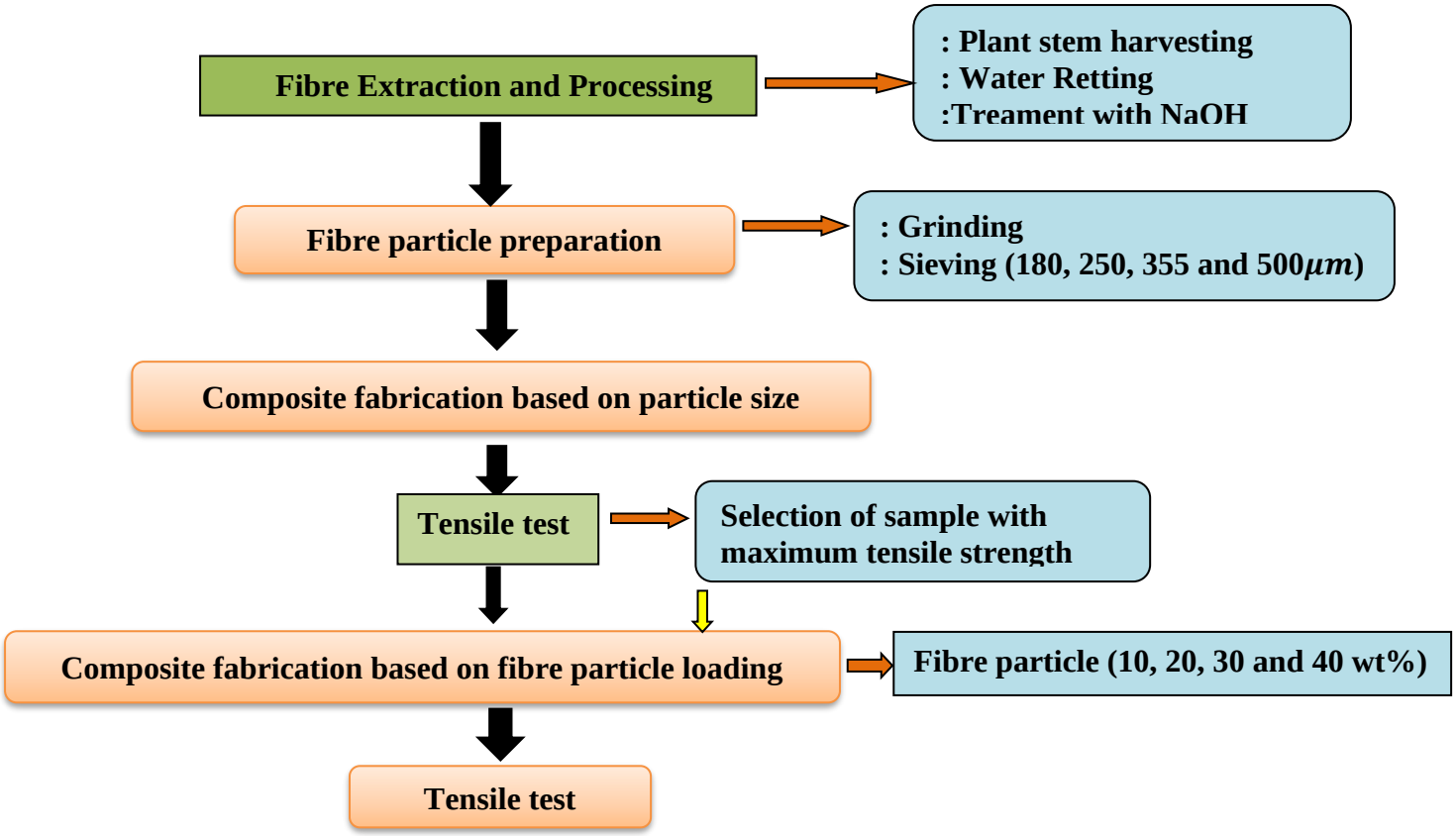

Figure 3: Schematic diagram of the steps for extraction, fabrication and tensile testing

3. Results and discussion

3.1 Bulk density of *Newbouldia laevis* fibres

The average values obtained from the measurements are shown in Table 1

Table 1: Measurement from bulk density test

Description	Value
W_1	104.636 g
W_2	103.652 g
V_F	10 mL

The value of the bulk density of the *Newbouldia laevis* fibres was found to be 0.098±0.005 g/cm^3, which is much lower than that of synthetic fibres, such as glass fibres, whose density is 2.4 g/cm^3. With a density of 0.098 g/cm^3 for this fibre, the weight of the composite will be significantly lower than that of the same fibre for the same composite applications; thus, a lighter weight is derived from the use of this natural fibre particle [28].

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3.2 Tensile

The tensile tests results (Figures 4 and 5) revealed that the maximum values of the tensile strength and yield strength were 24.766±2.65 N/mm^2 and 24.714±2.65 N/mm^2, respectively, for the composite with a fibre particle size of 180 µm, whereas the minimum values of the tensile strength and yield strength were 11.447±1.13 N/mm^2 and 9.73±3.08 N/mm^2, respectively, for the composite with a particle size of 500 µm. The results indicate that the tensile and yield strengths decrease as the fibre particle size increases from 180 µm to 500 µm. This agrees with the research on sisal fibre particles that revealed an increase in tensile strength as the particle size was decreased from 300 mm to 150 mm [29]. In addition,there have been similar reports from researchers on

wood, palm seed and bamboo fibre composites, which revealed that tinier particles had higher strength values than did large-size fibre particles [30,31,32]. The reason for these trends is that the transfer of stress from the matrix to the fibre depends on the interfacial region. An effective stress transfer occurs with a strong interface, resulting in increased strength and stiffness [33]. As the particle size increases, the particle surface area significantly decreases, which leads to poor interfacial interactions between the fibre particles and the matrix and weak bonding of the constituents of the composites [34].

Furthermore, the regression model (Appendix A) obtained for tensile strength and particle size is shown in Eq. (8) and the analysis revealed an R-sq. (adjusted) of 72.38%, a negative correlation (r = -0.86) and a statistically significant (P<0.05) relationship between the tensile strength and the particle size of the composite.

$$Tensile\ strength = 29.03 - 0.03736 * Particle\ size \quad (8)$$

The regression model (Appendix B) obtained for yield strength and particle size is shown in Eq. (9) and the analysis revealed an R-sq. (adjusted) of 69.24%, a negative correlation (r = -0.84) and a statistically significant (P<0.05) relationship between the yield strength and the particle size of the composite.

$$Yield\ strength = 30.13 - 0.04327 * Particle\ size \quad (9)$$

The elastic modulus result (Figure 6) for the particle size composites shows a maximum average value of 1888 MPa for a particle size of 180 µm, with a sharp decrease for the 250 µm particle size, which may be due to the poor distribution of the fibre particles within the matrix, resulting in poor alignment of the particles within the composite [35]. The regression model (Appendix C) obtained for elastic modulus and particle size is shown in Eq. (10) and the analysis revealed an R-sq (adjusted) of 0.00%, weak negative correlation and the relationship between the elastic modulus and the particle size of the composite is not statistically significant ($P > 0.05$)

$$Elastic\ modulus = 1825 - 0.718 * Particle\ size \quad (10)$$

This result reveals that the range in the particle size of *Newbouldia laevis* in this study has a negligible effect on the elastic modulus of the composite. This agrees with the findings of the work on periwinkle shell-reinforced polyester composite [36], epoxy/silica composite [37] and epoxy/alumina trihydrate composite [38].

The result (Figure 7) for the percentage elongation at break of the particle size composite shows a maximum value of $8.299 \pm 0.925\%$ for the 250 µm particle size fibre composite and a minimum value of $7.052 \pm 1.00\%$ for the 500 µm particle size composite. The regression model (Appendix D) obtained is shown in Eq. (11) and the analysis revealed an R-sq (adjusted) of 0.00%, weak negative correlation and the relationship between the percentage elongation at break and the particle size of the composite is not statistically significant ($P > 0.05$)

$$Elongation\ at\ break = 8.250 - 0.002257 * Particle\ size \quad (11)$$

The result reveals that the particle size of *Newbouldia laevis* has a negligible effect on the percentage elongation at break of the composite. This finding agrees with the work on PP-wood flour composite [39].

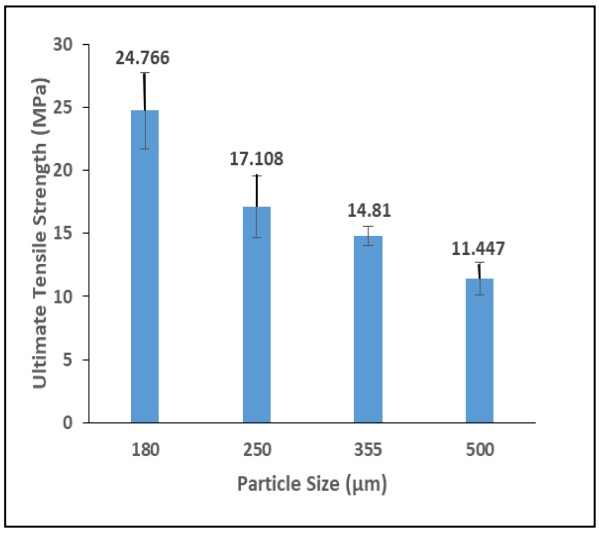

Figure 4: Ultimate tensile strength and particle size

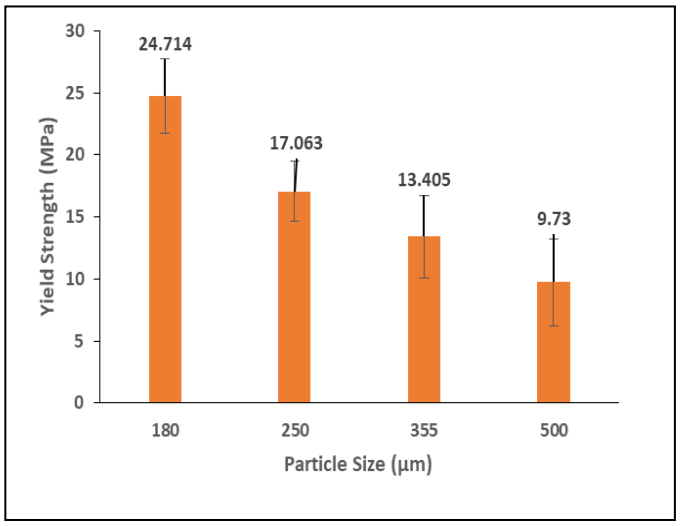

Figure 5: Yield strength and particle size

For tensile results (Figures 8 and 9) based on fibre particle loading, the maximum values of the tensile and yield strengths were found to be 31.35 ± 1.613 N/mm^2 and 31.277 ± 1.621 N/mm^2, respectively, and were obtained from the 10% fibre particle-loaded composite, whereas the minimum average values of the tensile and yield strengths were found to be 23.984 ± 3.373 N/mm^2 and 23.947 ± 3.365 N/mm^2, respectively, and were obtained from the 40% fibre particle-loaded composite. This signifies that the tensile and yield strengths decrease as the fibre loading increases from 10% to 40%. This findings agrees with the report on Abaca fibre which revealed that tensile strength decreased by 23.9% as the fibre loading increased from 30% to 40% [40]. The reason for

this trend is that as the fibre particle loading increases, the issue of clumping arises, which often leads to agglomeration defects [34]. These defects lead to poor interfacial adhesion between the fibre particles and the polyester matrix and ultimately result in weak bonding of the constituents of the composites. Furthermore, the regression model (Appendix E) obtained for tensile strength and particle loading is shown in Eq. (12) and the analysis revealed an R-sq (adjusted) of 48.6%, a negative correlation ($r = -0.72$) and a statistically significant ($P<0.05$) relationship between the tensile strength and the particle loading of the composite.

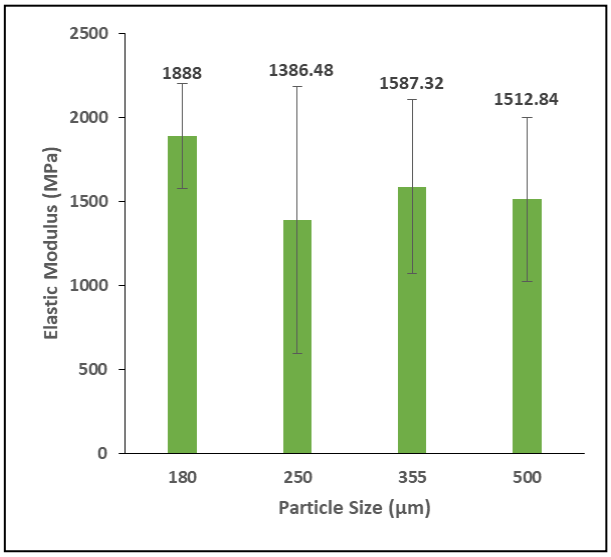

Figure 6: Elastic modulus and particle size

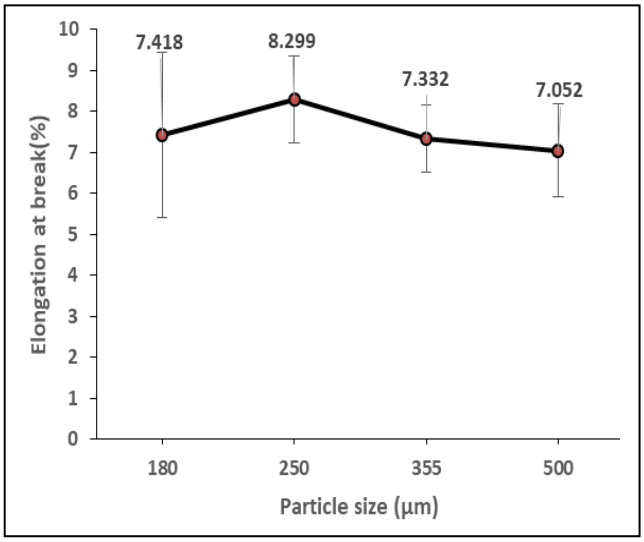

Figure 7: Elongation at break and particle size

$$Tensile\ strength = 33.07 - 0.2454 * Particle\ loading \quad (12)$$

The regression model (Appendix F) obtained for yield strength and particle loading is shown in Eq. (13) and the analysis revealed an R-sq (adjusted) of 48.48%, a negative correlation ($r = -0.72$) and a statistically significant ($P<0.05$) relationship between the yield strength and the particle loading of the composite.

$$Yield\ strength = 32.07 - 0.2454 * Particle\ loading \quad (13)$$

Additionally, the result (Figure 10) for the elastic modulus based on fibre particle loading shows a minimum value of 1404 MPa at 40% fibre particle loading and a maximum value of 2590 MPa at 10% fibre particle loading. The results show that the elastic modulus decreases as the fibre particle loading increases. This finding agrees with the findings of Rimdusit *et al.* (2011), who studied rubber wood flour polymers and reported that the particle size has an inverse relationship with the tensile modulus [41]. A similar report from research on nanocomposites reveals that the elastic modulus decreased as the amount of fibre filler increased; owing to the accumulation of local fibres within the composite [42]. The regression model (Appendix G) obtained for elastic modulus and particle loading is shown in Eq. (14) and the analysis revealed an R-sq (adjusted) of 49.48%, a negative correlation ($r = -0.72$) and a statistically significant ($P<0.05$) relationship between the elastic modulus and the particle loading of the composite.

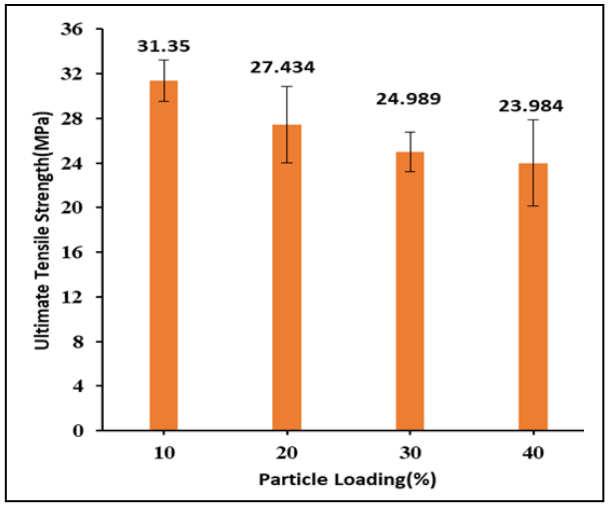

Figure 8: Ultimate tensile strength and particle loading

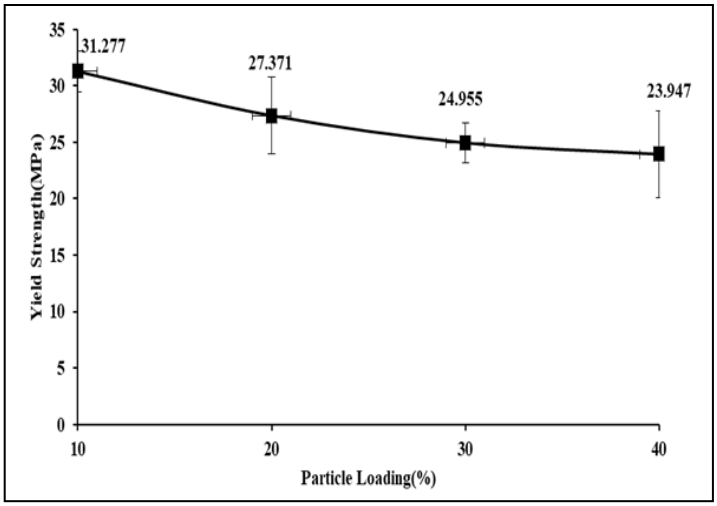

Figure 9: Yield strength and particle loading

$$Elastic\ modulus = 2815 - 37.07 * Particle\ loading \quad (14)$$

The result (Figure 11) of the percentage elongation at break for the fibre particle loading shows a maximum value of 7.142±0.82% for 40% loading and a minimum value of 76.560±0.38% for 10% fibre particle loading.
The regression model (Appendix H) obtained is shown in Eq. (15) and the analysis revealed an R-sq (adjusted) of 11.32%, weak positive correlation (r = 0.40) and the relationship between the percentage elongation at break and the particle loading of the composite is not statistically significant (P > 0.05)

$$Elongation\ at\ break = 6.278 - 0.02258 * Particle\ loading \quad (15)$$

This reveals that the particle loading has a little or negligible effect on the percentage elongation at break of the composite. Thus, the density of this natural fibre is lower than that of synthetic fibre as well as its strength compared to that of some natural fibre composites, such as coir/epoxy composites (17.86 MPa) and coconut shell powder composites (24.36 MPa), indicating that *Newbouldia laevis* fibres can be used in lightweight polymer composite applications, such as automobile bumpers and parts, particle boards and some industrial machine casings [43, 44].

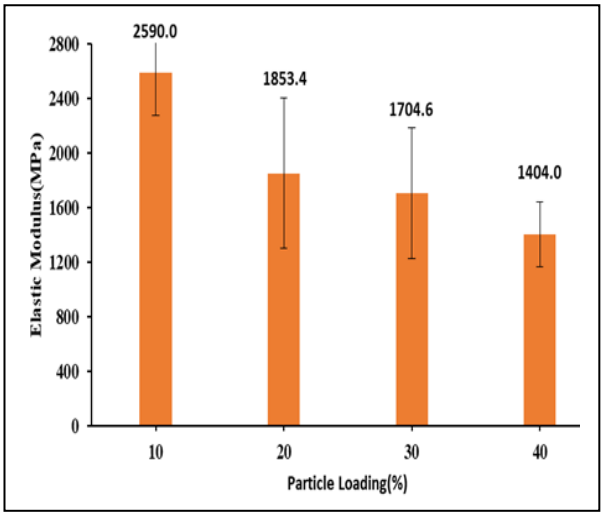

Figure 10: Elastic modulus and particle loading

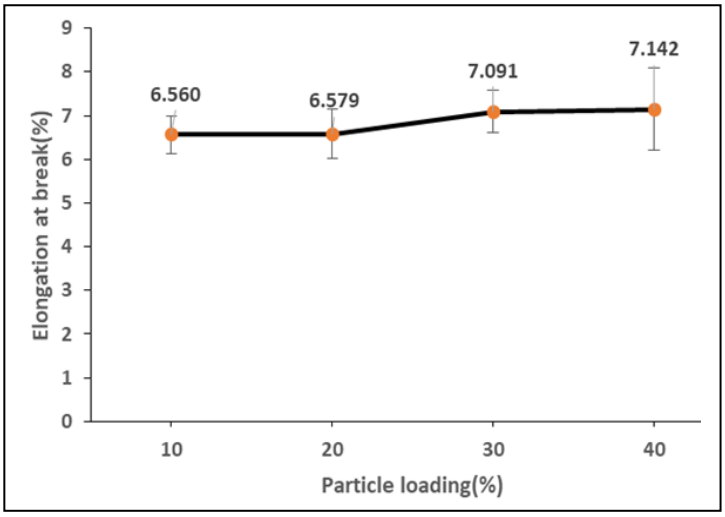

Figure 11: Elongation at break and particle loading

4. Conclusion

A study on a *Newbouldia laevis* fibre particle-reinforced composite revealed that the particle size and fibre loading affect the tensile properties of the composite. The tensile and yield strengths increased significantly as the particle size decreased from 500 µm to 180 µm. Additionally, the tensile strength, yield strength and elastic modulus decreased as the fibre particle loading increased from 10 wt% to 40 wt%. The particle size and loading have a minimal or negligible effect on the percentage elongation at break of the particulate composite. The maximum values of 31.35±1.613 N/mm^2 and 31.277±1.621 N/mm^2 were obtained from the 10 wt% fibre loading for tensile and yield respectively.
Hence, this study's results reveal that *Newbouldia laevis* fibre particles are suitable reinforcement biomaterials for lightweight composite applications.

Disclosure statement

No potential conflicts of interest were reported by the author (s)

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Data availability statement

All data relevant to the study are included in the article. In addition, the sample datasets are available from the corresponding author upon reasonable request.

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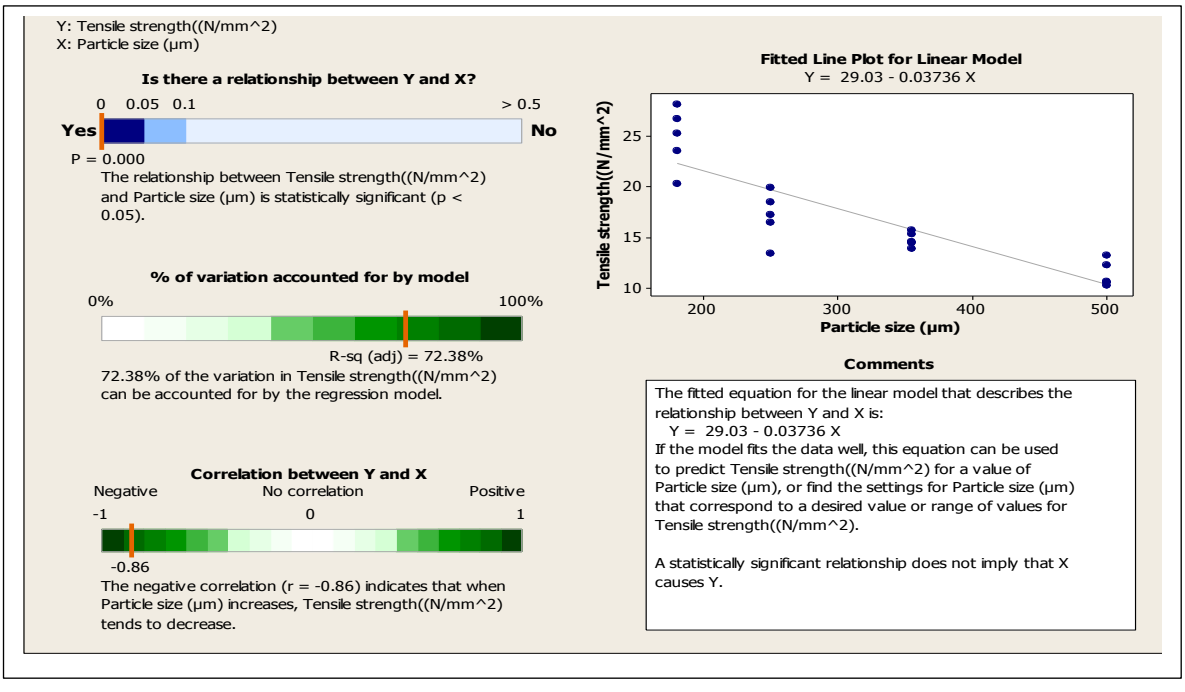
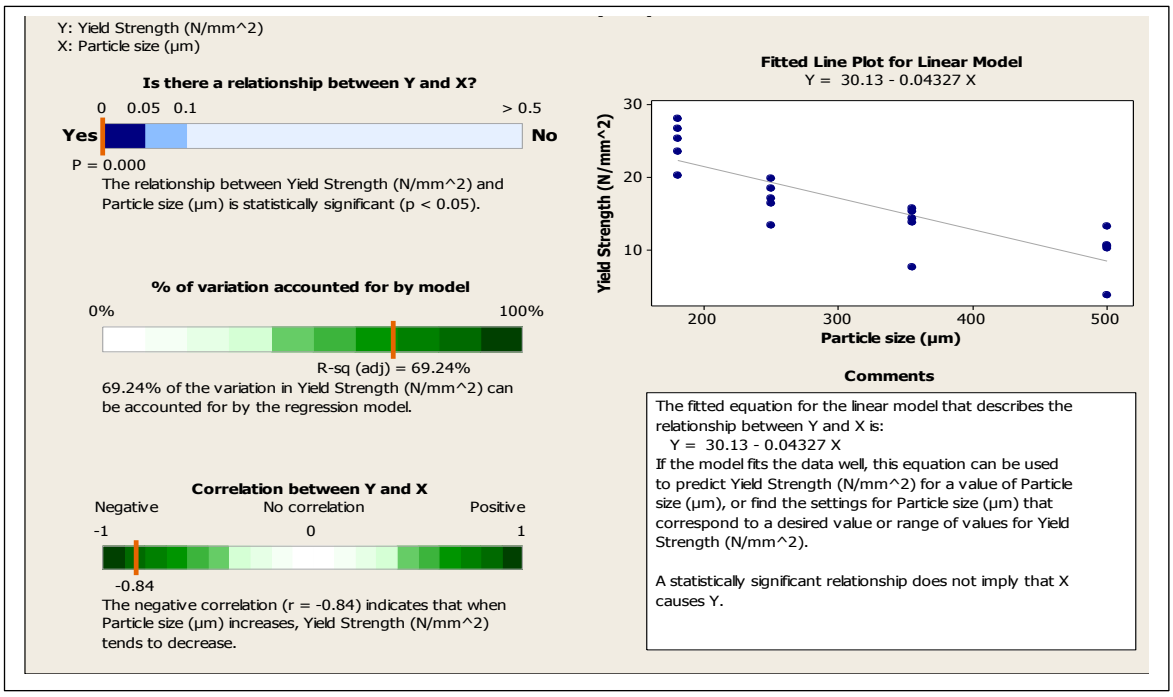
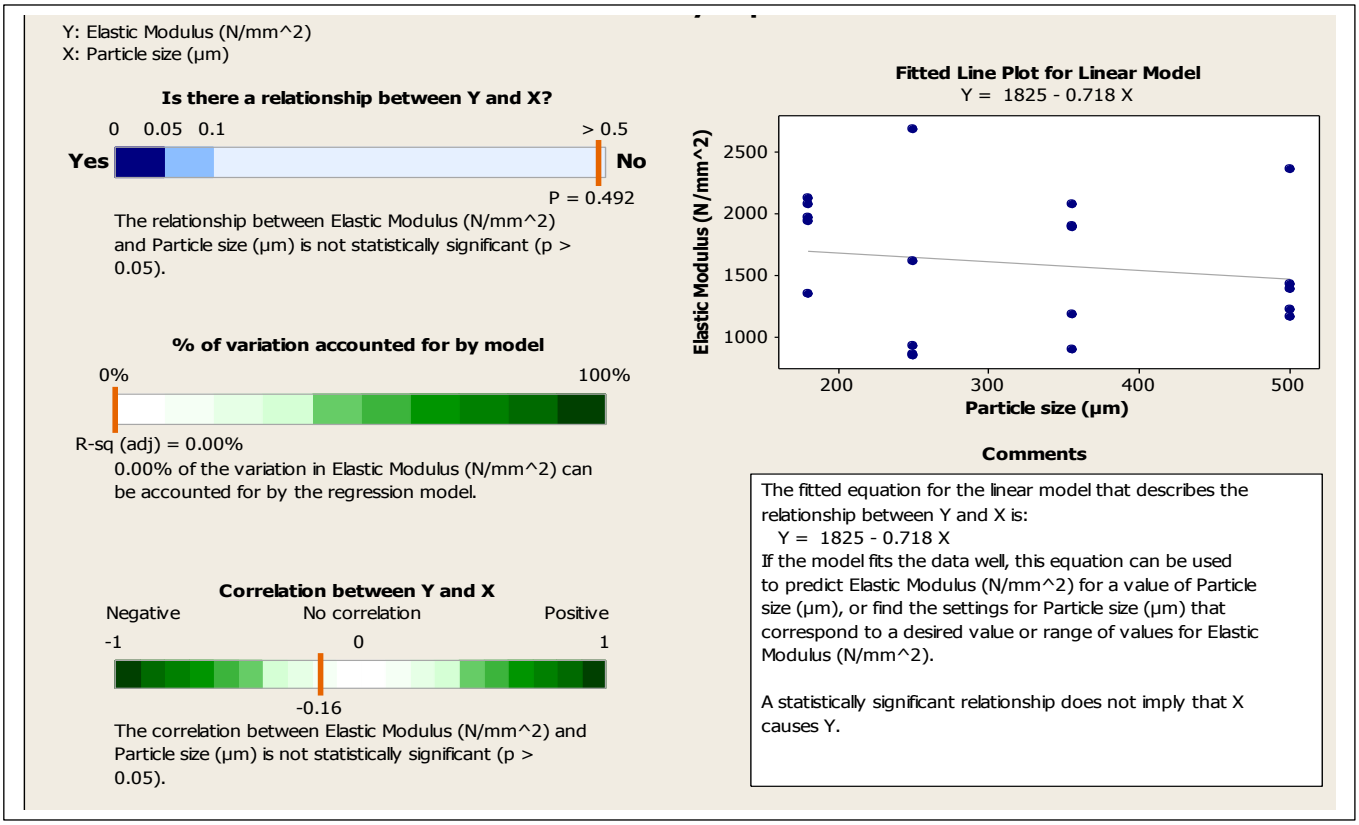
Appendix A: Regression for tensile strength vs particle size



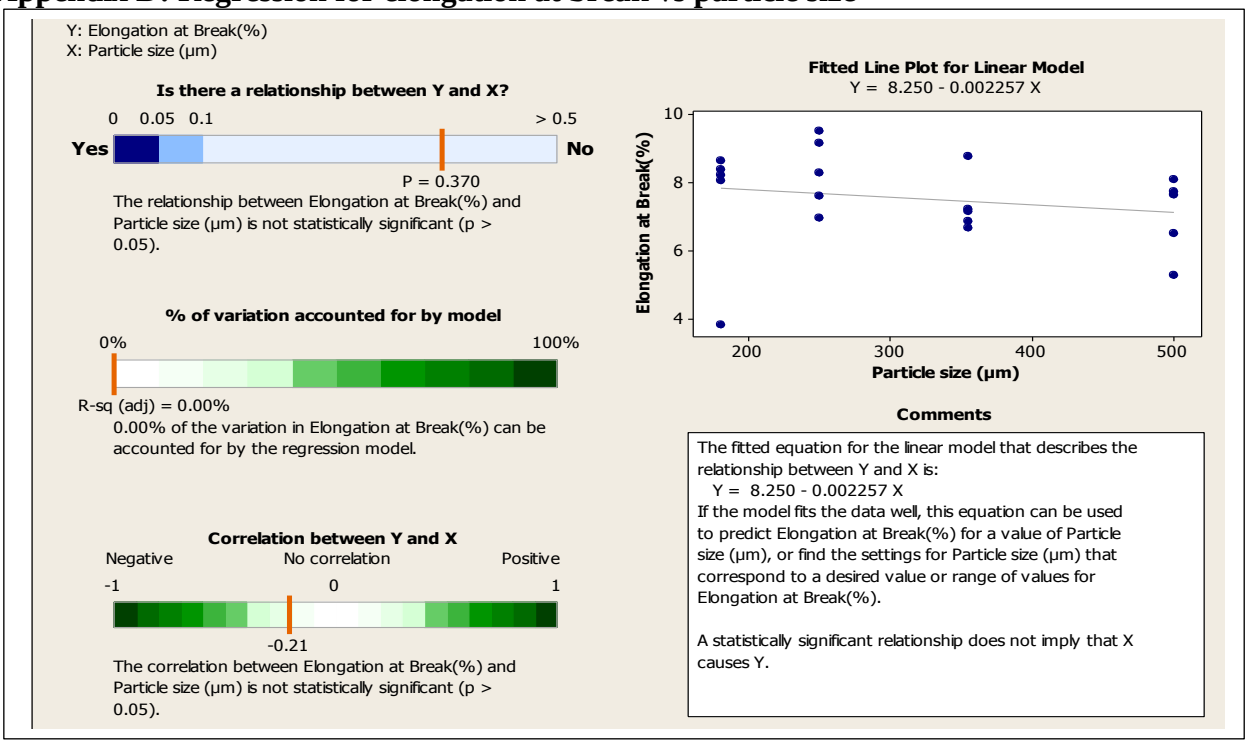
Appendix B: Regression for yield strength vs particle size



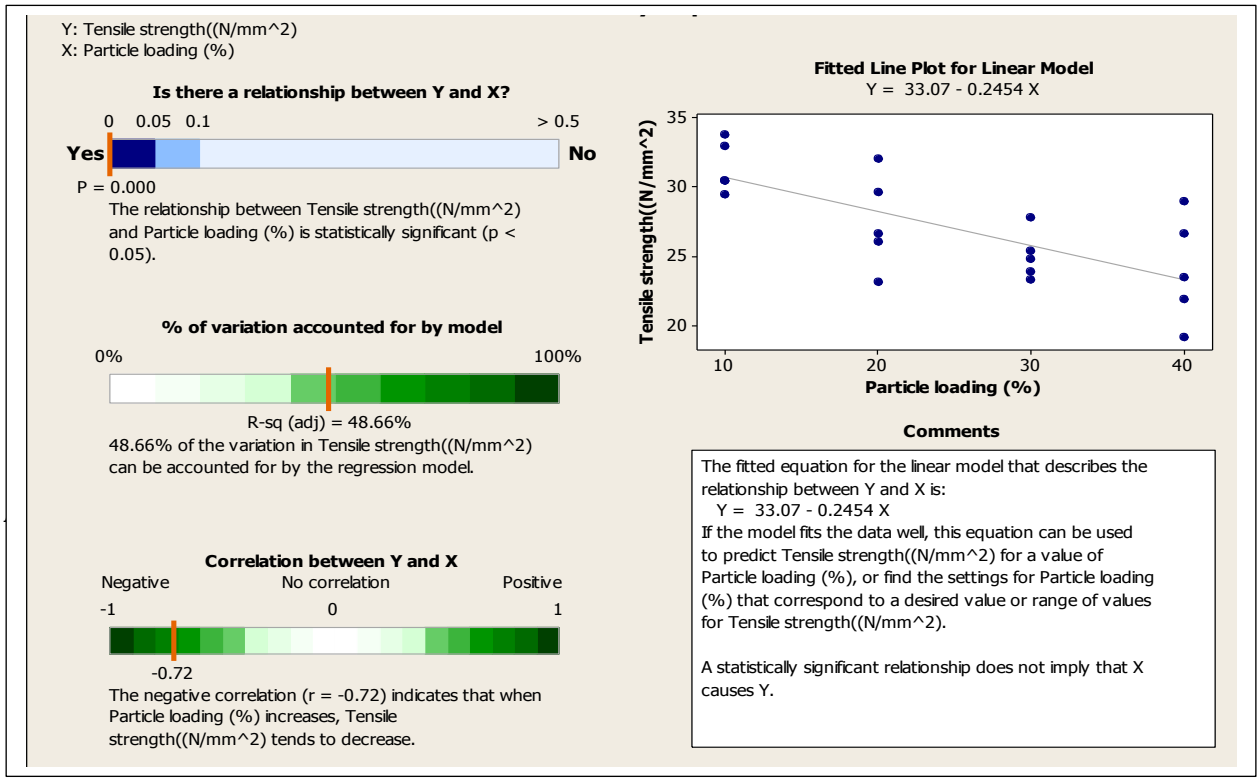
Appendix C: Regression for elastic modulus vs particle size



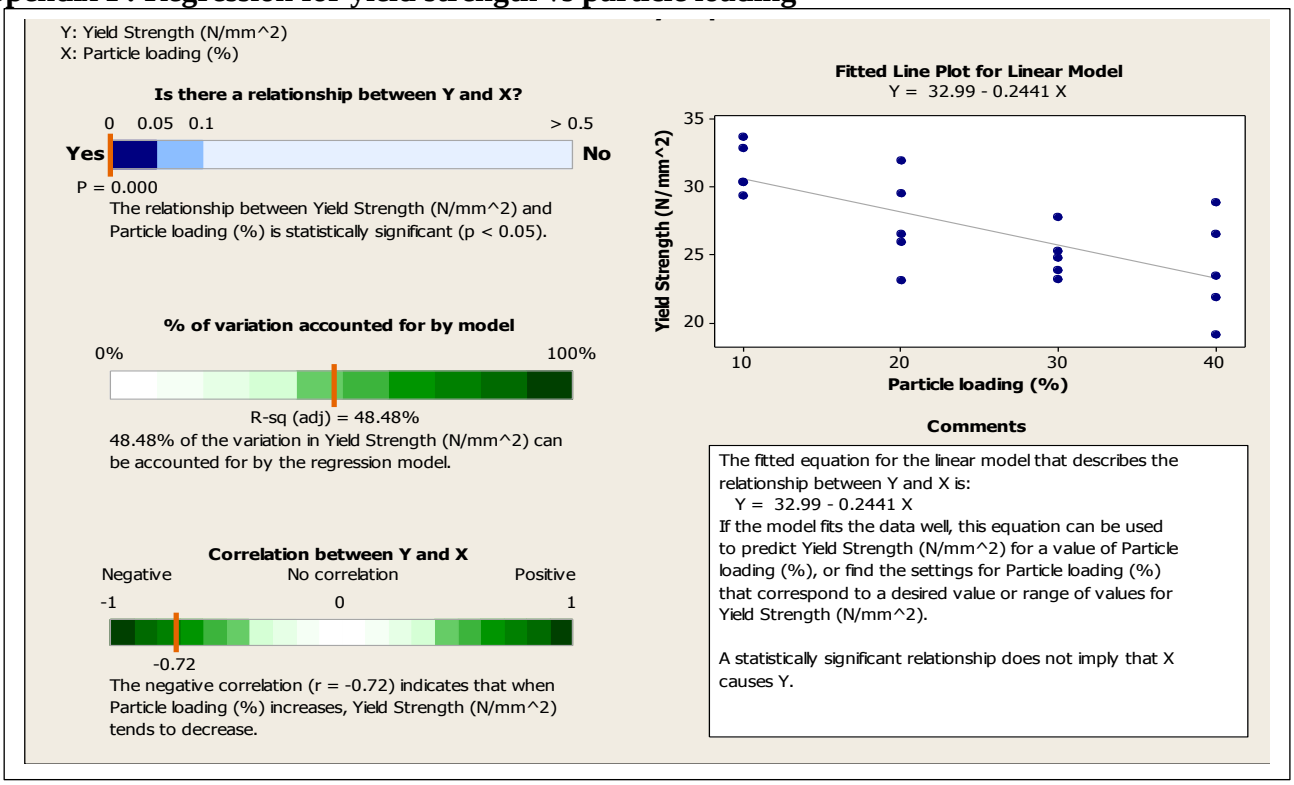
Appendix D: Regression for elongation at break vs particle size



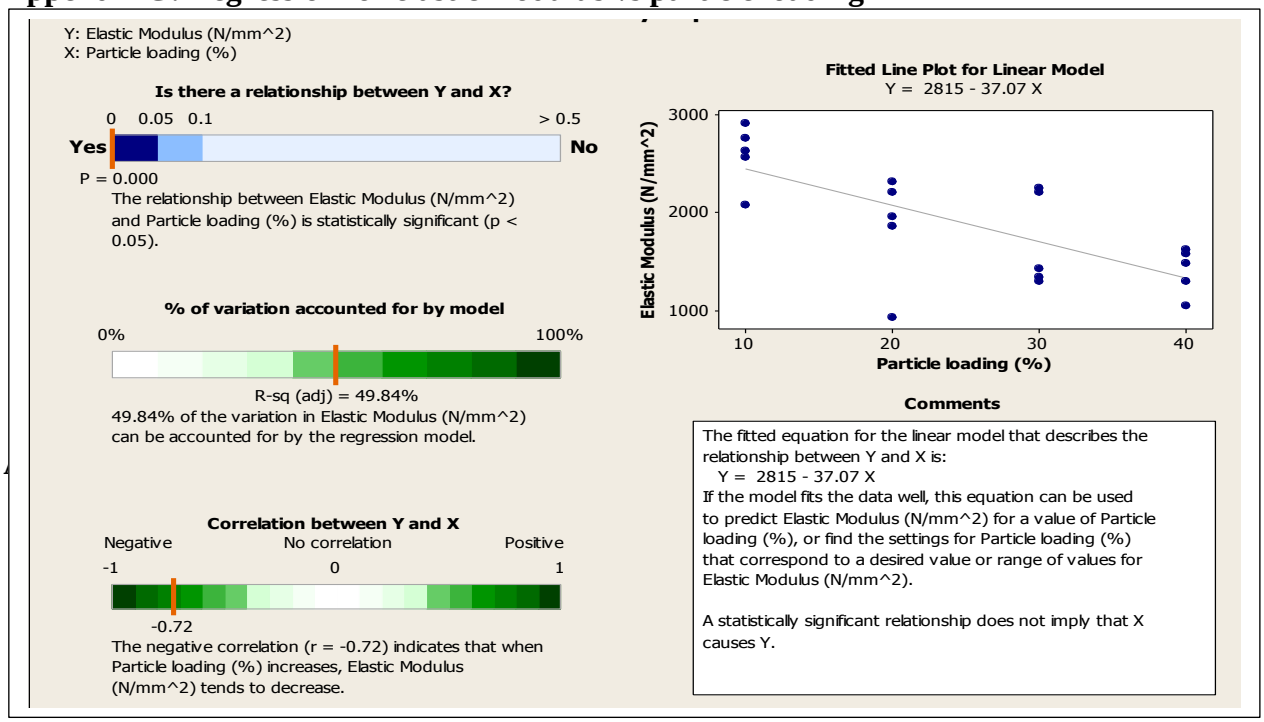
Appendix E: Regression for tensile strength vs particle loading



Appendix F: Regression for yield strength vs particle loading



Appendix G: Regression for elastic modulus vs particle loading



Appendix H: Regression for elongation at break vs particle loading

