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Effect of Particle Size and Filler Content on Mechanical Properties of Avocado Wood Flour-Low Density Polyethylene Composite

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ABSTRACT: The application of natural material excavates from wood source in composite production has increased taking the merit of low cost, readily available and light weight. The objective of this study was to evaluate the effects of particle size and filler content on the mechanical properties of avocado wood flour-low density polyethylene composite (AWF-LDPE) modified with NaOH/CH_3C00H using Response Surface Methodology. The influence of process parameters (size and weight of avocado wood flour (AWF)) on the properties (elongation, flexural strength, flexural modulus and impact strength) of the produced composite was evaluated. The AWF was injected at the size of particles from 20-100 mesh (850-150 µm) and flour content of 5-25 % wt into low-density polyethylene (LDPE) matrix to produce the composite of AWF-LDPE by the process of the injection molding machine. The optimization for the process parameters and mechanical properties of the composites was done using central composite design (CCD) of response surface methodology. The optimum condition for the factors and the properties of the composite were forecast with regression models of CCD. The ultimate process conditions for the process variables were 100 mesh size and 25 % wood flour content, while the properties were 4.8768 % elongation, 41.2921 MPa flexural strength, 0.6614 GPa flexural modulus and 83.384 KJ/m^2 impact strength. The coefficient of determinations (R^2) was close to unity with the maximum error of 0.09139 %. The values obtained from the AWF-LDPE composite at optimum condition indicated that it is recommended for suitable material for particle board in interior part of appliances and automobile application.

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In this modern time, there is an increase in application of additive made from wood source as filler in polymer industry for composite production (Supri and Lim, 2009; Obasi, 2012; Dungani *et al.*, 2016; Obasi, 2015; Government, 2019(a); Government *et al.*, 2019(b-e); Government *et al.*, 2018(a-b); Government *et al.*, 2021(a-b)). The simple reason is due to the advantages of low cost, light weight, readily availability, high specific properties and easy to process (Blezki and Gassan, 1999; Ochi, 2008; Kannappan and Dhuri, 2012; Atuanya et al., 2014; Obasi, 2015; Government, 2019(a); Government *et al.*, 2019(b-e); Yakubu *et al.*, 2013). Therefore, the need to hunt more filler in composite application is paramount for new engineering materials (Bawon et al., 2019; Yakubu *et al.*, 2013; Orji and McDonald, 2020; Rajak *et al.*, 2019; Sood and Dwivedi, 2018). Plant filler made from avocado wood flour has given less attention with more emphasis on its edible content. Reports have shown that the wood flour has enormous cellulose content comparable to previous fillers (Government and Onukwuli, 2016a; Government *et al*, 2016(b); Government, 2019(b-d); Dungani *et al*, 2016; Obasi, 2015; Government, 2019(a); Government *et al*, 2018(a-b): Government *et al.*, 2021(a-b)). In another word, the AWF is a suitable natural fiber for composite

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utilization (Government and Onukwuli, 2016a; Government *et al.*, 2016b; Government *et al.*, 2018(a-b)). One of the major problem of wood source-polymer composite is the compatibility between the flour and polymer matrix (Government *et al.*, 2019 (b-e); Obasi, 2015; André *et al.*, 2018; Verma *et al.*, 2019; Zakaria *et al.*, 2018)). The problem results to low interaction of wood flour and the matrix, high water absorption and reduced stress transfer from the filler to the matrix (Haristov and Vasileva, 2003; Nunes *et al.*, 2002; André *et al.*, 2018; Fortunati *et al.*., 2019; Obasi and Onuegbu, 2013; Reddy *et al.*, 2013; Azeez *et al.*, 2019; Azeez *et al.*, 2013; Azeez *et al.*,, 2018; Government *et al.*., 2019(b-d); Government *et al.*, 2022; Homkhiew, Ratanawilai *et al.*, 2014; Laadila *et al.*, 2017). This can be reduced to minimum by the introduction of chemical modifying agents (sodium hydroxide, acetic acid, maleated polyethylene, etc) (Nachtigall *et al.*, 2007; Aimi *et al.*, 2014; Rashid *et al.*, 2016 Government *et al.*, 2019(b-d); Homkhiew *et al.*, 2014; Feldmann *et al.*, 2016; Government *et al.*, 2022; Hakeem *et al.*., 2015; Zhang *et al.*, 2020).Previous work demonstrated that numerous researches have been performed on the experimentation of the mechanical properties of various wood filler polymer composite (Supri and Lim, 2009; Obasi, 2012; Netra *et al.*, 2012; Salmal *et al.*, 2013; Blezki and Gassan, 1999; Ochi, 2008; Kannappan and Dhuri, 2012; Laadila *et al.*, 2017; Turku *et al.*., 2018). Notwithstanding, few concern is attributed to the main component factors binding the properties and compositions of the composite using optimization process (Chanda, 2015; Harun and Geok, 2016; Laadila *et al.*, 2017; Najafi *et al.*., 2013; Turku *et al.*, 2018; Government *et al.*, 2021(a); Government *et al.*, 2022). Therefore, the need for consideration of these factors to minimize material and processing cost, improve the properties by obtaining optimal values for the composite production (Chanda, 2015; Harun and Geok, 2016; Laadila *et al.*, 2017; Najafi *et al.*, 2013; Turku *et al.*, 2018; Government *et al.*, 2021(a); Government *et al.*, 2022).

However, this work utilized novel filler AWF and the effect on the process variables for the optimization of mechanical properties of avocado wood flour-low density polyethylene for the usage in particulate board in appliances and automobile parts.

MATERIAL AND METHODS

Preparation of the wood filler: The avocado wood was extracted in Eastern part of Nigeria, Federal Housing Estate, Trans Ekulu, Enugu State. After the removal of the bark, the wood was subjected to 14 days drying in the sun for 8 hours. The wood was cut to small size by cutlass and ground using grounding machine. The AWF was later manually sieved from100 to 20 mesh sizes (150-850 µm).

Purchasing of low density polyethylene: The LDPE was obtained at Awada Market, Onitsha Anambra State, a product of Exxon Mobile Limited in Kingdom of Saudi Arabia.

Purchasing of sodium hydroxide and acetic acid: The sodium hydroxide pellets of 98 % extra pure w.w 40.0 was a product of LOBA Chemie Laboratory reagents and fine chemicals Pvt. Limited, Mumbai, India. The acetic acid is manufactured by BDH chemicals Limited, Poole, England with brand name Anala R* analytical reagent acetic acid glacial 60.05 with acidimetric pure 99.5 % wt. per mt at 20^0c of 1.047 to 1.052 g. These reagents were bought in Enugu State at Ogbete Main Market.

Purchasing of maleated polyethylene: The maleated polyethylene (MAPE) was bought in Southern part of Nigeria in Benin, Edo State produced Sigma-Aldrich chemical corporation, USA.

Surface modification of AWF: The AWF was immersed in a 6wt% solution of sodium hydroxide for 16 hrs and 4 vol% acetic acid for 1 h. The filler was rinsed completely with distilled water, filtered and sun-dried for 10 hrs and mixed with 5 wt% maleated polyethylene.

Production of thermoplastic avocado pear wood filler composites: The treated AWF was injected into LDPE. The treated was filled at 5, 10, 15, 20, and 25% by weight. The mixture was injection moulded through the application of injection molding machine with trade name HUICHON/5SON10/500×1000 no.6241 1990/6 manufactured by PO Yuen (TO'S) Machine FTY limited in Democratic People Republic of Korea for making the test specimen at Ekenedilichukwu workshop, Onitsha, Anambra State. The composite products were subject to mechanical test for the analysis of elongation, flexural strength, flexural modulus and impact strength.

Analysis of tensile elongation of composites: This analysis was carried out at the University of Nigeria Civil Engineering Workshop, Nsukka Enugu State of Nigeria. The universal tensometer BSS1610 model no. 8889 produced by Hounsfield tensometer limited was used for the analysis. This test was done on ASTM D638 (ASTM, 1990). The equipment has a cross-head speed between 10-100 cm/s. The sample test dimension was 3.2mm x 19mm x 160mm. The sample was firmly fixed into chucks of the equipment. A constant load was applied to the sample till it breaks.

Elongation was calculated using the relationship in Eq. (1).

$$F = \Delta E/E \qquad (1)$$

Where ΔE is the change in length and E is the original length of the sample.

Analysis of flexural properties of composites: The analysis was carried out using equipment in the tensile analysis. The dimension of this test was ASTM D790 3.2mm x 19mm x 300mm (ASTM, 1990). The sample was fixed on a 3-point support span. A specific load was applied in the centre of the sample until fracture and constant deflection occurred. It was evaluated using Eq.(2).

$$F_s = 1.5N\iota_s/wv^2 \qquad (2)$$

Where F_s is the flexural strength, N is the applied force, ι_s is the length of flexural sample, w is the width of the sample, v is the thickness of the sample.

The flexural modulus was determined using Eq. (3).

$$F_E = \iota_s^2 \frac{S}{2wv^2} \qquad (3)$$

Where F_E is the flexural modulus, S is the tangent of the force-deflection plot.

Analysis of impact strength: The impact strength was obtained through Charpy impact test using impact testing machine with trade name LOS LOSENHAUSENWERK DUSSELDORFER MASCHINENBAU AG. DUSSELLDORF, model no.17562/1963 produced in Germany located at the Mechanical Engineering Workshop, University of Nigeria, Nsukka, Enugu State. The specimen was cut using the dimension 3.2 mm × 19 mm×80mm. The composites sample was placed in the machine and pendulum hammer was released to strike the sample. The impact strength was recorded through equipment.

Experimental design and optimization: The experimental design and optimization used was response surface methodology (RSM) which is component of CCD. The software applied was design expert 7.0. version. The two input factors were involved and four responses were inserted in the CCD. The face centered design option was adopted to suit the conversion of particle size in mesh size to microns. The input factors were filler content (A) and particle size (B). The design comprised of 13 runs. This process is made up of five levels (lowest, low, medium and highest). The coding of the level is -β, 0, +1 and +.β. The input factors are filler content ranges from 5-25% and particle size 100-20 mesh. The levels, coding and input factor was captured in Table 1. The response surface functions were used to forecast the elongation, flexural strength, flexural modulus and impact strength. The response surface function was stated in eq. (4).

$$V_i = C_O + \sum_{i=1}^{n} C_i\, A + \sum_{i=0}^{n} A_{ii}\, A_i^2 + \sum_{i=1}^{n}\sum_{i=i+1}^{n} C_{ij} A\, B_j + \varepsilon \qquad (4)$$

Where V_i is the predicted response, C_o is the constant coefficient, C_i the linear coefficients, C_{ii} is the quadratic coefficients, Cij is the interactive coefficients, A_i and B_j are the coded values of the variables, n is the number of independent test variables and ε is the random error.

Table 1: level and code of input factors for two-factor central composite design

Input factors		Coded level			
	- β	-1	0	+1	+β
Particle size (mesh) (A)	20	20	60	100	100
Particle size (µm}	850	850	250	150	150
Wood filler content (%) (B)	5	5	15	25	25

RESULTS AND DISCUSSION

Table 2 shows design matrix of AWF-LDPE composite. The experimental results from design matrix were inserted in RSM of CCD for prediction of mechanical properties and optimization of AWF-LDPE composite. Table 3(a-d) shows ANOVA for AWF-LDPE composite for $V_{E,}$ $V_{FS,}$ and V_{FM} and $V_{IM,}$ respectively. As shown in Table 3(a-d), the F-values gave good account of the models described $V_{E,}$ $V_{FS,}$ and V_{FM} and V_{IM}, respectively. The probability values for the responses were less than 0.05. The values of determination coefficient (R^2) were used to estimate the significance of the model. It was observed that the values of R^2, adjusted R^2 and predicted R^2 were nearer to each but close to unity. The percentage of all the values of R^2 was not enumerate by the models with both adjusted and predicted values of R^2 been in agreement. This is an indication of reasonable significance of the models. These values portray better precision and consistency of the experimental results (Khuri *et al.*, 1987; Government *et al.*, 2019(c-d); Kandar & Akil, 2016; Peng *et al.*, 2015; Rostamiyan *et al.*, 2014; Rostamiyan *et al.*., 2015(a); Rostamiyan *et al.*., 2015; Government *et al.*, 2018(a-b). The trend has been reported previously (Soury *et al.*, 2009; Hadi, 2011; Patpen *et al.*, 2015; Government *et al.*,, 2021(a); Government *et al.*, 2022; Homkhiew *et al.*, 2014; Government, 2019(a); Government *et al.*, 2019(b-e); Peng *et al.*, 2015; Rostamiyan, *et al.* 2015(b)).

Table 2: Design matrix of AWF-LDPE composite in coded and actual form

	Factors				Responses			
	Coded		Actual					
Run	A	B	A (mesh)	B (%)	V_E (%)	V_{FS} (MPa)	V_{FM} (GPa)	V_{IM} (KJ/m^2)
1	-1	1	20	25	4.43	35.12	0.617	63.76
2	0	1	60	25	4.69	39.71	0.66	79.4
3	1	-1	100	5	5.61	38.21	0.549	81.25
4	0	0	60	15	4.89	38.31	0.613	81.81
5	0	0	60	15	4.89	38.31	0.613	81.81
6	0	-1	60	5	5.72	35.29	0.51	78.5
7	0	0	60	15	4.89	38.31	0.613	81.81
8	-1	0	20	15	4.64	32.59	0.554	60.7
9	1	0	100	15	5.1	40.4	0.615	82.56
10	1	1	100	25	4.88	41.39	0.662	83.33
11	0	0	60	15	4.89	38.31	0.613	81.81
12	0	0	60	15	4.89	38.31	0.613	81.81
13	-1	-1	20	5	5.27	30.78	0.5	65

Where A, B, V_E,$V_{FS,}$ V_{FM} and V_{IM} are particle size, filler content, elongation, flexural strength flexural modulus and impact strength, respectively.

The Eq.6-9 stated the models for prediction of V_E, $V_{FS,}$ V_{FM} and V_{IM} using RSM and the results of the ANOVA presented in Tables a to 3d.:

V_E = +5.63896+5.34243E-003 * Particle Size-0.10013 * Filler Content+6.87500E-005 * Particle Size * Filler Content-1.34510E-005 * Particle Size^2+1.87130E-003 * Filler Content^2 (6)

V_{FS} = +25.89752+0.18524 * Particle Size+0.40790 * Filler Content-7.25000E-004 * Particle Size * Filler Content-7.71322E-004 * Particle Size^2-6.08163E-003 * Filler Content^2 (7)

V_{FM}=+0.41476+1.85409E-003 * Particle Size+0.010773 * Filler Content-1.01895E-005 * Particle Size^2-1.59918E-004 * Filler Content^2 (8)

V_{IM} = +51.25634+0.67378 * Particle Size+0.47396 * Filler Content+2.07500E-003 * Particle Size * Filler Content-4.03908E-003 * Particle Size^2-0.019033 * Filler Content^2 (9)

Table 3a: ANOVA of AWF-LDPE composite for V_E

V_E	Source	Sum of Squares	Df	Mean Square	F Value	p-value Prob > F	
	Model	1.593647	5	0.318729	1199.383	< 0.0001	Significant
	A-Particle Size	0.26137	1	0.26137	983.5382	< 0.0001	
	B-Filler Content	1.146329	1	1.146329	4313.649	< 0.0001	
	AB	0.003025	1	0.003025	11.38311	0.0119	
	A^2	0.002388	1	0.002388	8.98589	0.0200	
	B^2	0.180536	1	0.180536	679.3578	< 0.0001	
	Residual	0.00186	7	0.000266			
	Lack of Fit	0.00186	3	0.00062			
	Pure Error	0	4	0			
	Cor Total	1.595508	12				
	R-Squared	0.998834					
	Adj R-Squared	0.998001					
	Pred R-Squared	0.991561					

Table 3b: ANOVA of AWF-LDPE composite for V_{FS}

V_{FS}	Source	Sum of Squares	Df	Mean Square	F Value	p-value Prob > F	
	Model	111.1791	5	22.23583	408.3751	< 0.0001	Significant
	A-Particle Size	77.21079	1	77.21079	1418.025	< 0.0001	
	B-Filler Content	23.87292	1	23.87292	438.4413	< 0.0001	
	AB	0.3364	1	0.3364	6.178199	0.0419	
	A^2	7.852181	1	7.852181	144.2103	< 0.0001	
	B^2	1.906856	1	1.906856	35.02062	0.0006	
	Residual	0.381147	7	0.05445			
	Lack of Fit	0.381147	3	0.127049			
	Pure Error	0	4	0			
	Cor Total	111.5603	12				
	R-Squared	0.996583					
	Adj R-Squared	0.994143					
	Pred R-Squared	0.975034					

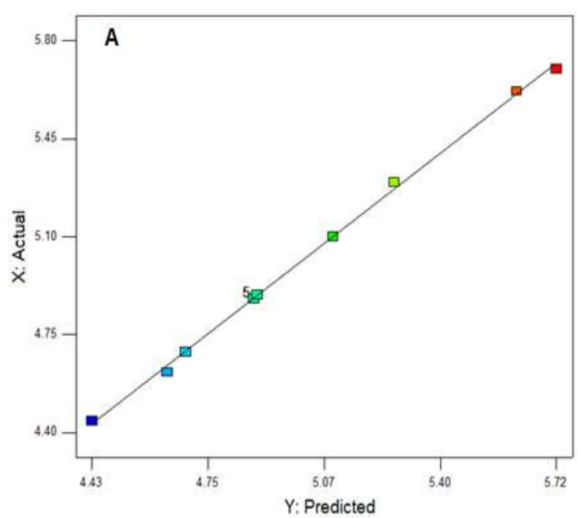
Table 3c: ANOVA of AWF-LDPE composite for V_{FM}

V_{FM}	Source	Sum of Squares	Df	Mean Square	F Value	p-value Prob > F	
	Model	0.031232	5	0.006246	1337.846	< 0.0001	Significant
	A-Particle Size	0.004069	1	0.004069	871.4863	< 0.0001	
	B-Filler Content	0.02447	1	0.02447	5240.996	< 0.0001	
	A^2	0.00137	1	0.00137	293.4973	< 0.0001	
	B^2	0.001318	1	0.001318	282.3917	< 0.0001	
	Residual	3.27E-05	7	4.67E-06			
	Lack of Fit	3.27E-05	3	1.09E-05			
	Pure Error	0	4	0			
	Cor Total	0.031264	12				
	R-Squared	0.998955					
	Adj R-Squared	0.998208					
	Pred R-Squared	0.992524					

Table 3d: ANOVA of AWF-LDPE composite for V_{IM}

V_{IM}	Source	Sum of Squares	Df	Mean Square	F Value	p-value Prob > F
	Model	796.8007	5	159.3601	2641.824	< 0.0001
	A-Particle Size	559.5051	1	559.5051	9275.306	< 0.0001
	B-Filler Content	0.543878	1	0.543878	9.016238	0.0199
	AB	2.7556	1	2.7556	45.6815	0.0003
	A^2	215.319	1	215.319	3569.493	< 0.0001
	B^2	18.67715	1	18.67715	309.6242	< 0.0001
	Residual	0.422254	7	0.060322		
	Lack of Fit	0.422254	3	0.140751		
	Pure Error	0	4	0		
	Cor Total	797.2229	12			
	R-Squared	0.99947				
	Adj R-Squared	0.999092				
	Pred R-Squared	0.996146				

Analysis of predicted and actual plots: Figure 1 presents actual and predicted plots of the mechanical properties of AWF-LDPE composite. The predicted versus actual indicates the connections between the experimental and predicted result of RSM model. The plots indicated fitness of the models predicted by the RSM. It was recorded from the Figure 1 that the plotted point were diagonally aligned. This is a confirmation of the model fitness. The results were previously reported (Myers *et al.*, 2002; Myers, 2004; Montgomery, 2001; Government *et al.*, 2018(a); Government *et al.*, 2019(b); Kandar & Akil, 2016; Rostamiyan *et al.*, 2014: Government *et al.*, 2022). This is a clear evidence for the models prediction of the properties of the composite. *Analysis of 3D surface plots:* The`3D surface plots show the interaction and effect of the response and factors for the AWF-LDPE composite. The responses were elongation, flexural strength, flexural modulus and impact strength and the interaction with the factors (particle size and weight of filler) were captured in Fig.2 (a-d). The increase in the filler content decreased the elongation of the AWF-LDPE composite as it was viewed on Fig. 2(a). It was concluded that chemical treatment of the filler does not affect the elongation of the composites. This result was in agreement with previous authors (Thakore *et al.*, 1999; Ahmed *et al.*, 1996; Obasi, 2012; Supri and Lim, 2009; Government. *et al.*, 2022; Homkhiew. *et al.*, 2014; Government. *et al.*, 2019(d): Government *et al.*,2018(a-b)). Small sizes of filler displayed higher elongation. This is due to high degree of dispersion of AWF particle in LDPE matrix. Similar observation was observed in previous researcher (Lee *et al.*, 2009; Brent *et al.*, 2014. Fig. 2(b) shows the increase in the wood filler improved the flexural strength of the AWF-LDPE composites. This result was related to other studies (Kord, 2011; Salmah, *et al.* 2013). The observation in Fig. 2(c) indicates that the flexural modulus of the composites increased as the wood filler is added.



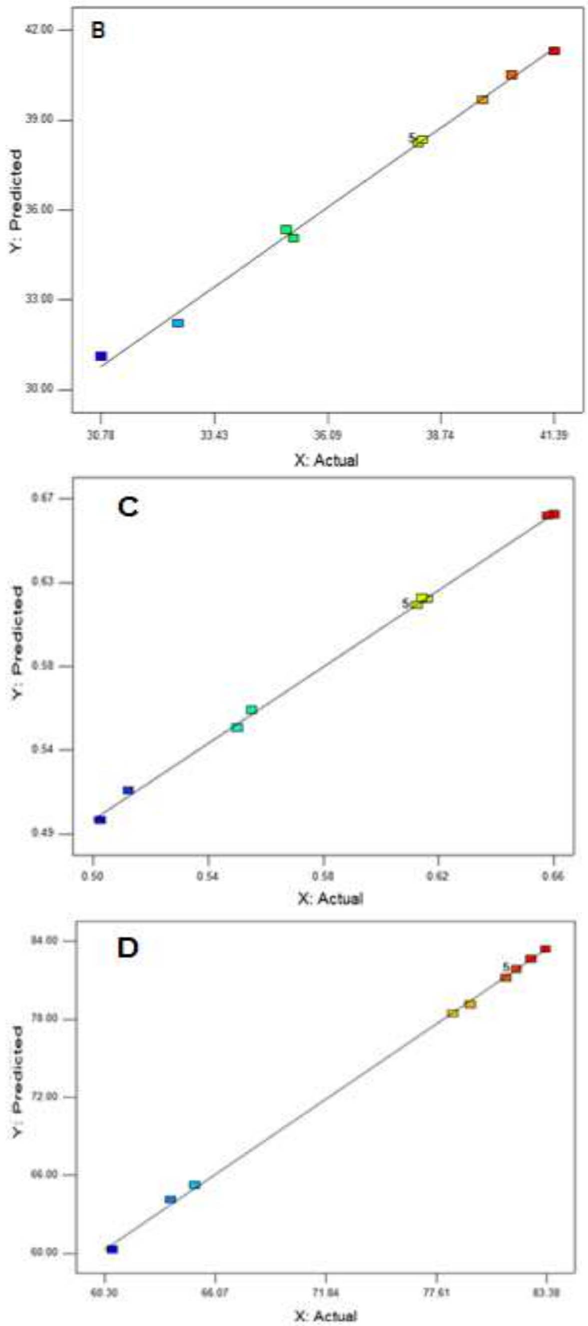
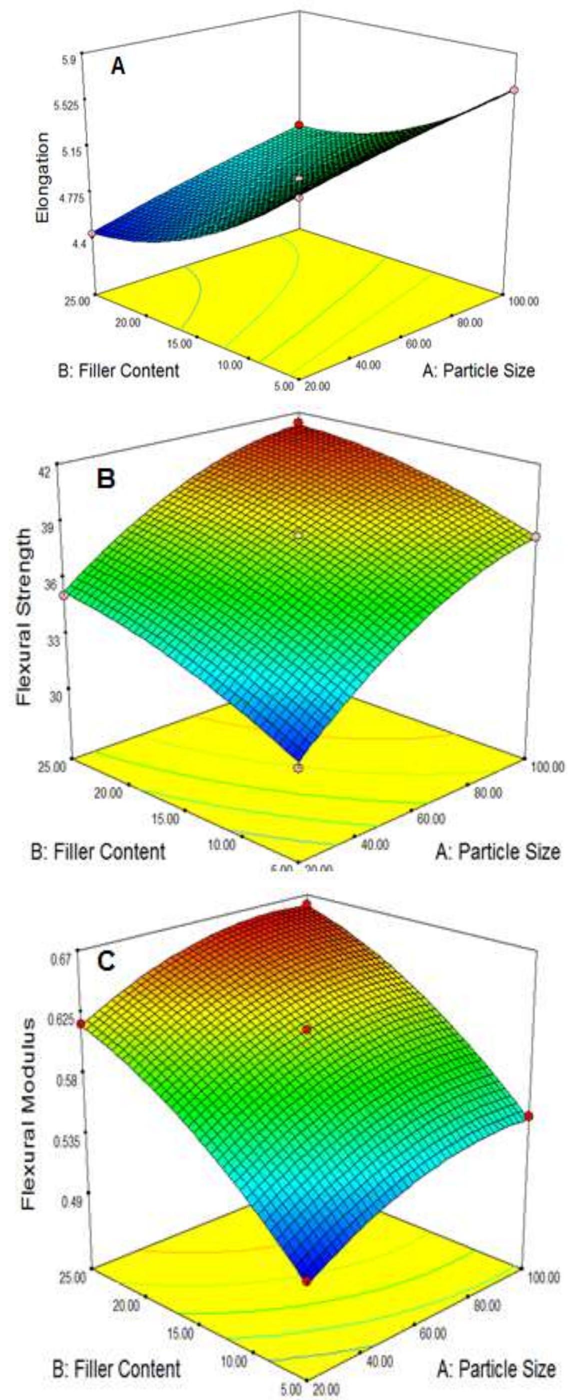


Fig. 1: Actual versus predicted plots for the properties of AWF-LDPE (a) V_E (b) V_{FS} (e) V_{FM} (g) V_{IM}

The ultimate flexural strength and modulus occurred at minimum sized particle. This is due to increase in homogenization of LDPE and AWF at lower size particle which prevents bending. These are shown in Fig. 2(b) and Fig. 2(c), respectively. These discussions have been displayed by previous works (Obasi, 2015; Brent *et al.*, 2014; Nwanenyi, 2013; Government *et al.*, 2022: Homkhiew *et al.*, 2014: Government *et al.*, 2019(b-d)). It can be shown in Fig. 2(d) that the impact strength improved to a maximum filler content. This is related to the work of some authors (Joseph *et al.*, 2002; Lou *et al.*, 2007). The impact strength observed maximum growth at lowest particle. This trend could be due to high dispersion of AWF in the LDPE matrix that needs higher energy for impact failure. Similar results were previously examined by earlier researchers (Yang, *et al.* 2004; Rahman *et al.*, .2010; Government *et al.*, 2022; Homkhiew *et al.*, 2014; Government *et al.*,2019 (a-e)).



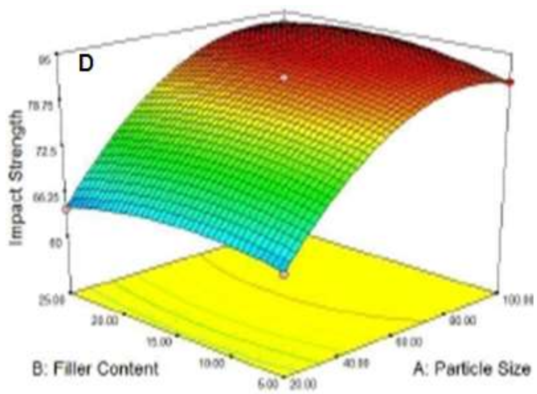


Fig. 2: 3D Surface plots for the mechanical properties of AWF-LDPE composite (a) V_E (b) V_{FS} (c) V_{FM} (d) V_{IM} as connecting factors of particle size and filler weight.

Analysis of the optimization: From Table 4, it was recorded from CCD using RSM that the optimal conditions for the properties of AWF-LDPE composites was observed at 100 mesh particle size and 25 % filler weight which corresponded to the factors at this condition. In the same vein, the responses which were equivalent to the mechanical properties of AWF-LDPE composite at these conditions viz: 4.876767 % elongation, 41.29209 MPa flexural strength, 0.661395 GPa flexural modulus and 83.38396 KJ/m^2 impact strength, respectively. Similar situation was predicted by later researchers (Government *et al.*, 2022; Homkhiew *et al.*, 2014; Government, *et al.* 2019 (b-d)).

Table 4: Optimization of AWF-LDPE composite

Properties	Particle Size (mesh)	Filler Content (%)	Predicted Value of properties	Experimental Value of properties	Error (%)
V_E (%)	100	25	4.876767	4.88	0.06626
V_{FS} (MPa)	100	25	41.29209	41.3	0.01916
V_{FM} (GPa)	100	25	0.661395	0.662	0.09139
V_{IM} (KJ/m^2)	100	25	83.38396	83.33	0.064756

Validation of the model: The results from the experimental evaluation for the properties of the composite were compared with that generated from RSM as illustrated in Table 4. It can be observed that the following percentage difference at optimum process parameters with regards to elongation, flexural strength, flexural modulus and impact strength between the experimental and predicted values using RSM model were less than 0.1%. This negligible errors represented better validation of mechanical properties as regarded for the RSM models.

Conclusion: The AWF as filler through surface treatment as led to the improvement of AWF-LDPE composite properties. The prediction of mechanical properties for AWF-LDPE composite using CCD of RSM gave mathematical expressions that described the connecting factors of the experimental results. The experimental results were totally related to the prediction of the properties of the composite using RSM at optimum conditions. The introduction of novel filler (AWF) in the LDPE matrix for the production of AWF-LDPE composites has been established for composite application.

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