ISSN NO. 2320-5407

*Journal homepage: http://www.journalijar.com*
*Journal DOI: 10.21474/IJAR01*

*INTERNATIONAL JOURNAL OF ADVANCED RESEARCH*

**RESEARCH ARTICLE**

**OPTIMISATION STUDIES ON THE MECHANICAL PROPERTIES OF NEEM BARK FLOUR-FILLED HIGH DENSITY POLYETHYLENE COMPOSITES USING CENTRAL COMPOSITE DESIGN.**

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## Manuscript Info

***Manuscript History:***

Received: 18 March 2016
Final Accepted: 22 April 2016
Published Online: May 2016

***Key words:***
Mechanical properties, Optimisation, Response surface methodology, Neem bark flour, High density polyethylene, Central design composites.

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## Abstract

This research is carried out to study the effect of filler content and the particle size on the mechanical properties of neem bark flour-filled high density polyethylene composites. The fiber was characterized. Response surface methodology was used to optimize the preparation of the composite. A second order polynomial model was developed to predict the mechanical properties based on central composite design. The results show that the neem bark used for this study contains 9.04% lignin, 65.43% cellulose, 23.55% hemicellulose. It was found that the composite design was best fit for a quadratic regression model. The selected optimum condition for this composite is 21.63 mesh particle size and 40wt% filler content at 77.7% desirability. The mechanical properties at optimum are 14.51MPa, 5.28%, 0.45GPa, 92.31KJ/m$^2$, and 242.14Pa for tensile strength, percentage elongation, tensile modulus, impact strength and hardness respectively. The optimum conditions were validated with little error less than 0.2%.



## Introduction:-

Customarily, laboratory trial has been used to determine the mechanical properties of polymer composites, as well as other engineering materials. However, experimental determination of mechanical properties of materials is expensive and consumes time [1]. Finding a cost effective way of forecasting the mechanical properties of polymer composite materials would help in solving the problems associated with laboratory trial. One way of achieving this is through development of a model which could be used to forecast the mechanical properties of polymer composites [1]. Statistically based design of experiments (DOE) helps to improve the development cycle time, improve reliability, reduce process variability, and increase overall product quality [2]. In this study, Neem Bark based composite was developed and a mathematical model was also developed, using central composite design, to predict the mechanical properties of the developed composite.

## Experimental:-

### Materials:-

High density polyethylene, supplied by Indorama Petrochemical Company Limited, Port Harcourt, Nigeria, was used as the polymer matrix. The melt flow index (MFI) of the Polyethylene was 2.16 g/min, density 0.946g/cm3. Neem bark obtained from Enugu State, Nigeria, was used as reinforcement fillers.

### Fiber Characterization:-

Fiber characterization was carried out to determine the cellulose, hemicellulose and lignin contents of the forest flame pod. The cellulose content was determined according to kurschner and hoffer [3]**.** Firstly, the fiber was treated with n-hexane and methanol. Then, 0.7g of the treated fiber was added in a 95% solution of nitric acid and ethanol mixture. The mixture was filtered and the residue washed first with hot water, then with ethanol to completely remove the residual acid. The residue was oven dried at $100^0$C to a constant weight. The cellulose corresponds to the insoluble fraction of the sample. The hemicellulose content of the plant fiber was determined by neutral detergent fiber method, [4]. Neutral detergent fiber was prepared by refluxing 0.7g of the fiber sample with sodium lauryl sulphate solution (neutralized to pH 7.0) for 1 hour. The solution was filtered and the residue washed with hot distilled water and ethanol. The residue was subsequently oven dried to constant weight at $100^0$C for 8 hours. The weight obtained is the neutral detergent fiber weight. Hemicellulose content was calculated as the difference in weight of neutral detergent fiber and the acid detergent fiber prepared from acid hydrolysis of the same mass of the sample. The lignin content was estimated according to Ververis et al. [5]. In determination of the lignin content, 0.7g of the fibers were heated with 5ml of 72% w/w $H_2SO_4$ solution for 4.5 hours in order to hydrolyze the cellulose and hemicellulose. After the heating, the solution was filtered and the residue thoroughly washed with hot distilled water and absolute ethanol to completely remove any acid present. The solid residue was dried at $105^0$C for 24hrs and weighed. This residue is known as the acid detergent fiber with weight, $w_1$. The residue was then transferred to a pre-weighed dry porcelain crucible and heated at $600^0$C for 5hrs. After cooling, its weight $w_2$ was determined. Acid insoluble lignin was then calculated by the difference ($W_1$-$W_2$). Each test was carried out three times.

**Preparation of bio composite:-**

Neem bark was ground and sieved to a particle size of 10mesh-40mesh . Each particle size of the neem bark flour and the HDPE were mixed using an internal mixer at 50 rpm screw speed. This process was done at a room temperature until a homogeneous mixture was obtained. The mixture was poured into an injection molding machine at temperature of $180^0$C and the composite was injection molded. The injection molded biocomposites are shown in Fig 1. After cooling, the solidified samples were prepared for the test.

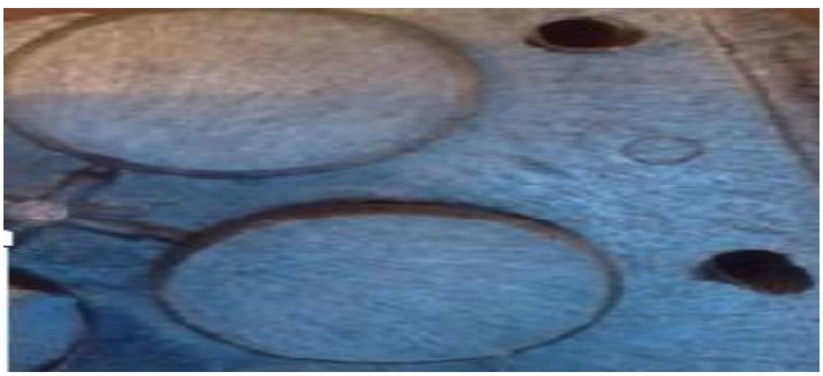

Fig 1**:** Injection Moulded Neem Bark Flour-Filled High Density Polyethylene Composites.

**Mechanical Test:-**

All mechanical tests were carried out at the temperature of $23^0$C ± 2 and relative humidity of 50 ± 5%. The tensile strength test was carried out in a universal testing machine (Hounsfield Tensometer, model number 8889, with an accuracy of BSS 1610) according to ASTM D638 at a crosshead speed of 5mm/min. The impact test was performed using an impact pendulum tester on notched rectangular specimens according to ASTM D256. The specimen was loaded on the testing machine and the pendulum from the impact tester was allowed to strike the specimen. The Izod notched impact energy absorbed was determined. Brinell hardness test was conducted in accordance with ASTM E103. Each test was carried out three times. The average result was then calculated and presented.

**Experimental design and statistical analysis:-**

Response surface methodology (RSM) was used to optimize the conditions for the fiber preparation to obtain optimum mechanical properties. The design of experiments was done using Minitab17.0. The screening experiment shows that only particle size and filler content were significant factors. Thus, the two independent variables were employed by central composite design (CCD). The experiments were randomized to protect against unknown bias distorting the experimental result. The design matrix for the experiment is presented in Table 1. The response functions measured were tensile strength, impact strength, percentage elongation, Brinell hardness and tensile modulus. A second order polynomial equation was fitted for each factor as follows:

$$Y = \beta_0 + \beta_1 x_1 + \beta_2 x_1 + \beta_{11} x_1^2 + \beta_{22} x_2^2 + \beta_{12} x_1 x_2$$

where y is the estimated response; $\beta_0$, $\beta_1$, $\beta_2$, $\beta_{11}$, $\beta_{22}$ and $\beta_{12}$ are constant parameters; $x_1$ and $x_2$ are the values of the independent variables of particle size and filler contents respectively. Table 1 shows the design matrix with the uncoded values of the process variables and their corresponding responses. The variance of each factor was partitioned into linear, quadratic and interactive terms.

Table 1**:** Design matrix for Neem Bark Flour-Filled High Density Polyethylene Composites.

| Std Order | Run order | Blocks | Particle Size (mesh) | Filler Content (%) | Tensile Strength (MPa) | Elongation (%) | Tensile Modulus (GPa) | Impact Strength (KJ/m$^2$) | Brinell Hardness (Pa) |
|---|---|---|---|---|---|---|---|---|---|
| 11 | 1 | Block1 | 3.78 | 30.00 | 14.26 | 4.55 | 0.35 | 79.6 | 378.2 |
| 5 | 2 | Block1 | 25.00 | 15.85 | 17.88 | 6.74 | 0.41 | 75.6 | 130.5 |
| 6 | 3 | Block1 | 25.00 | 30.00 | 15.45 | 5.88 | 0.43 | 87.9 | 137 |
| 12 | 4 | Block1 | 25.00 | 30.00 | 15.4 | 6 | 0.45 | 88 | 136.3 |
| 14 | 5 | Block1 | 25.00 | 44.14 | 14.02 | 4.91 | 0.47 | 95.8 | 211.5 |
| 9 | 6 | Block1 | 46.21 | 30.00 | 10.43 | 3.97 | 0.48 | 59.6 | 139.8 |
| 8 | 7 | Block1 | 25.00 | 30.00 | 15.45 | 5.98 | 0.43 | 87.9 | 136.3 |
| 2 | 8 | Block1 | 40.00 | 20.00 | 11.17 | 4.2 | 0.49 | 57.3 | 135.7 |
| 7 | 9 | Block1 | 25.00 | 30.00 | 15.45 | 5.88 | 0.43 | 87.9 | 136.3 |
| 3 | 10 | Block1 | 10.00 | 20.00 | 15.59 | 5.47 | 0.32 | 75.3 | 199.8 |
| 4 | 11 | Block1 | 40.00 | 40.00 | 9.86 | 3.81 | 0.53 | 60.1 | 144.6 |
| 10 | 12 | Block1 | 10.00 | 40.00 | 13.84 | 4.11 | 0.4 | 83.7 | 452.2 |
| 1 | 13 | Block1 | 25.00 | 30.00 | 15.4 | 5.98 | 0.43 | 88 | 137 |
| 13 | 14 | Block1 | 25.00 | 30.00 | 15.45 | 5.88 | 0.45 | 87.9 | 136.3 |

## Results and discussion:-

### Model Selection and Verification of the Mechanical Properties:-

The experimental data were analyzed using Minitab 17.0 software. Analysis of variance (ANOVA) and regression analysis were used to analyze all the responses in order to evaluate the coefficient terms and for model fitting. The results are tabulated in Table 2-6. The ANOVA demonstrated that the quadratic regression model for all the mechanical properties were highly significant. The test shows very low P-value; $P < 0.001$ for models of all responses. This probability value means that there is less than 0.1% chance that a model value of this magnitude could occur due to noise. However, the lack- of- fit for all mechanical properties were significant. This implies that the model requires further analysis. Many insignificant model terms were also observed. In order to remove the insignificant terms and improve the model, backward elimination regression with alpha to exit = 0.10, was used to modify the original quadratic model. The goodness-of-fit of the modified models were further inspected using the $R^2$ values. The $R^2$ values are shown in Table 7. The values show that 94% for tensile strength, 95% for percentage elongation, 93% for tensile modules, 94% for impact strength and 97% for Brinell hardness of the total variability of the response data around its mean was explained by the model. The difference between the predicted and adjusted R-square was also considered. A rule of thumb is that the adjusted and predicted R-square should be within 0.2 of each other [6]. Table 7 Shows reasonable agreement between adjusted R-square and predicted R-square (within 0.2 each of the others). Adequate precision was used to measure the signal to noise ratio. A ratio greater than 4 indicates an adequate signal and shows that the model can be used to navigate the design space [7]. The adequate precisions in Table 7 indicate that the model can be used to navigate the design space.

Table 2**:** Analysis of Variance for Quadratic Model of Tensile strength.

| Source | Sum of Squares | df | Mean Square | F Value | p-value Prob > F |
|---|---|---|---|---|---|
| Model | 60.42 | 5 | 12.08 | 14.81 | 0.0007 |
| A-particle size | 16.95 | 1 | 16.95 | 20.77 | 0.0019 |
| B-filler content | 3.40 | 1 | 3.40 | 4.17 | 0.0756 |
| AB | 0.048 | 1 | 0.048 | 0.059 | 0.8137 |
| $A^2$ | 27.44 | 1 | 27.44 | 33.62 | 0.0004 |
| $B^2$ | 0.11 | 1 | 0.11 | 0.14 | 0.7174 |
| Residual | 6.53 | 8 | 0.82 | | |
| Lack of Fit | 6.52 | 3 | 2.17 | 3262.22 | < 0.0001 |
| Pure Error | 3.333E-003 | 5 | 6.667E-004 | | |
| Cor Total | 66.95 | 13 | | | |

Table 3**:** Analysis of Variance for Quadratic Model of Percentage Elongation.

| Source | Sum of Squares | df | Mean Square | F Value | p-value Prob > F |
|---|---|---|---|---|---|
| Model | 10.80 | 5 | 2.16 | 17.15 | 0.0004 |
| A-particle size | 0.94 | 1 | 0.94 | 7.49 | 0.0256 |
| B-filler content | 0.41 | 1 | 0.41 | 3.26 | 0.1084 |
| AB | 0.24 | 1 | 0.24 | 1.87 | 0.2088 |
| $A^2$ | 7.35 | 1 | 7.35 | 58.41 | < 0.0001 |
| $B^2$ | 0.34 | 1 | 0.34 | 2.72 | 0.1376 |
| Residual | 1.01 | 8 | 0.13 | | |
| Lack of Fit | 0.99 | 3 | 0.33 | 95.18 | < 0.0001 |
| Pure Error | 0.017 | 5 | 3.467E-003 | | |
| Cor Total | 11.81 | 13 | | | |

Table 4**:** Analysis of Variance for Quadratic Model of Tensile Modulus.

| Source | Sum of Squares | df | Mean Square | F Value | p-value Prob > F |
|---|---|---|---|---|---|
| Model | 0.036 | 5 | 7.127E-003 | 22.92 | 0.0002 |
| A-particle size | 0.023 | 1 | 0.023 | 73.54 | < 0.0001 |
| B-filler content | 1.871E-003 | 1 | 1.871E-003 | 6.02 | 0.0397 |
| AB | 4.000E-004 | 1 | 4.000E-004 | 1.29 | 0.2895 |
| $A^2$ | 5.911E-004 | 1 | 5.911E-004 | 1.90 | 0.2053 |
| $B^2$ | 9.283E-005 | 1 | 9.283E-005 | 0.30 | 0.5997 |
| Residual | 2.488E-003 | 8 | 3.109E-004 | | |
| Lack of Fit | 1.954E-003 | 3 | 6.514E-004 | 6.11 | 0.0399 |
| Pure Error | 5.333E-004 | 5 | 1.067E-004 | | |
| Cor Total | 0.038 | 13 | | | |

Table 5**:** Analysis of Variance for Quadratic Model of Impact Strength.

| Source | Sum of Squares | df | Mean Square | F Value | p-value Prob > F |
|---|---|---|---|---|---|
| Model | 1801.93 | 5 | 360.39 | 13.99 | 0.0009 |
| A-particle size | 344.71 | 1 | 344.71 | 13.39 | 0.0064 |
| B-filler content | 259.82 | 1 | 259.82 | 10.09 | 0.0131 |
| AB | 7.84 | 1 | 7.84 | 0.30 | 0.5962 |
| $A^2$ | 943.95 | 1 | 943.95 | 36.65 | 0.0003 |
| $B^2$ | 78.17 | 1 | 78.17 | 3.04 | 0.1196 |
| Residual | 206.02 | 8 | 25.75 | | |
| Lack of Fit | 206.01 | 3 | 68.67 | 25751.20 | < 0.0001 |
| Pure Error | 0.013 | 5 | 2.667E-003 | | |
| Cor Total | 2007.96 | 13 | | | |

Table 6**:** Analysis of Variance for Quadratic Model of Brinnel Hardness.

| Source | Sum of Squares | Df | Mean Square | F Value | p-value Prob > F |
|---|---|---|---|---|---|
| Model | 1.293E+005 | 5 | 25856.84 | 59.15 | < 0.0001 |
| A-particle size | 18030.28 | 1 | 18030.28 | 41.25 | 0.0002 |
| B-filler content | 2444.81 | 1 | 2444.81 | 5.59 | 0.0456 |
| AB | 14823.06 | 1 | 14823.06 | 33.91 | 0.0004 |
| $A^2$ | 31912.48 | 1 | 31912.48 | 73.01 | < 0.0001 |
| $B^2$ | 3501.44 | 1 | 3501.44 | 8.01 | 0.0221 |
| Residual | 3496.83 | 8 | 437.10 | | |
| Lack of Fit | 3496.18 | 3 | 1165.39 | 8918.82 | < 0.0001 |
| Pure Error | 0.65 | 5 | 0.13 | | |
| Cor Total | 1.328E+005 | 13 | | | |

Table 7**:** Model Summary.

| Response | R-Squared | Adj.R-Squared | Pred.R-Squared | Adeq Precision |
|---|---|---|---|---|
| Tensile strength | 0.94 | 0.89 | 0.69 | 17.80 |
| Percentage Elongation | 0.95 | 0.92 | 0.70 | 14.81 |
| Tensile Modulus | 0.93 | 0.88 | 0.81 | 20.53 |
| Impact Strength | 0.94 | 0.89 | 0.50 | 14.36 |
| Brinnel Hardness Value | 0.97 | 0.95 | 0.81 | 22.42 |

From Table 2-6, it was found that the factors in the model had significant effects on the model. For the tensile strength and percentage elongation of the composite, the effects of filler content (B), particle size and filler content interaction (AB) and the square terms of filler content ($B^2$) were not significant; all other model terms were found to be significant. For tensile modulus and impact strength, the square terms of filler content ($B^2$) and the particle size and filler content interaction (AB) were not significant, all other model terms were significant ($\alpha < 0.05$). All model terms for Brinell hardness was significant ($\alpha < 0.05$).

The model equations for the developed composites are shown in equations 2, 3, 4,5and 6 for tensile strength, percentage elongation, tensile modulus, impact strength and Brinell hardness number respectively.

Tensile Stregth = $+16.10368 +0.31096\, x_1 - 0.10649\, x_2 - 8.52285E\text{-}003 x_1^2$ (2)
Elongation = $+5.20028+0.19810x_1 - 0.054221x_2 - 4.36093E\text{-}003x_1^2$ (3)
Tensile Modulus = $+0.25597 +4.03196E\text{-}003\, x_1+2.56028E\text{-}003\, x_2$ (4)
Impact Strength =$+54.87936+1.87382x_1+0.49706x_2-0.049129x_1^2$ (5)
Brinnel Hardness = $+217.32725-8.33700x_1+1.78341\, x_2-0.40583\, x_1x_2+0.29213\, x_1^2+0.21769x^2$ (6)
Where; $x_1$ is the particle size

$X_2$ is the filler content

**Residual analysis:-**

Residuals play important role in judging model adequacy [6]. Residuals are the difference between the actual and predicted values. Table 8-12 contains actual values, predicted values and residuals for the mechanical properties of the developed composites. To check whether the residuals followed a normal distribution, a normal probability curve of the residuals was constructed. If the residual plots approximately along a straight line, then the normality assumption is satisfied. Figures 2,3,4,5 and 6 show a normal plot of residuals for the responses. These figures show that there is no apparent problem with normality as the residuals plot approximately follows along a straight line.

Table 8**:** Actual values, Predicted Values and Residuals for tensile strength.

| Standard Order | Actual Value | Predicted Value | Residual |
|---|---|---|---|
| 1 | 15.40 | 15.36 | 0.044 |
| 2 | 11.17 | 12.78 | -1.61 |
| 3 | 15.59 | 16.23 | -0.64 |
| 4 | 9.86 | 10.65 | -0.79 |
| 5 | 17.88 | 16.86 | 1.02 |
| 6 | 15.45 | 15.36 | 0.094 |
| 7 | 15.45 | 15.36 | 0.094 |
| 8 | 15.45 | 15.36 | 0.094 |
| 9 | 10.43 | 9.08 | 1.35 |
| 10 | 13.84 | 14.10 | -0.26 |
| 11 | 14.26 | 13.96 | 0.30 |
| 12 | 15.40 | 15.36 | 0.044 |
| 13 | 15.45 | 15.36 | 0.094 |
| 14 | 14.02 | 13.85 | 0.17 |

Table 9**:** Actual Values, Predicted Values and Residuals for Percentage Elongation

| Standard Order | Actual Value | Predicted Value | Residual |
|---|---|---|---|
| 1 | 5.98 | 5.80 | 0.18 |
| 2 | 4.20 | 5.06 | -0.86 |
| 3 | 5.47 | 5.66 | -0.19 |
| 4 | 3.81 | 3.98 | -0.17 |
| 5 | 6.74 | 6.57 | 0.17 |
| 6 | 5.88 | 5.80 | 0.079 |
| 7 | 5.88 | 5.80 | 0.079 |
| 8 | 5.98 | 5.80 | 0.18 |
| 9 | 3.97 | 3.42 | 0.55 |
| 10 | 4.11 | 4.58 | -0.47 |
| 11 | 4.55 | 4.26 | 0.29 |
| 12 | 6.00 | 5.80 | 0.20 |
| 13 | 5.88 | 5.80 | 0.079 |
| 14 | 4.91 | 5.03 | -0.12 |

Table 10**:** Actual values, Predicted Values and Residuals for Tensile Modulus.

| Standard Order | Actual Value | Predicted Value | Residual |
|---|---|---|---|
| 1 | 0.43 | 0.43 | -3.576E-003 |
| 2 | 0.49 | 0.47 | 0.022 |
| 3 | 0.32 | 0.35 | -0.027 |
| 4 | 0.53 | 0.52 | 0.010 |
| 5 | 0.41 | 0.40 | 0.013 |
| 6 | 0.43 | 0.43 | -3.576E-003 |
| 7 | 0.43 | 0.43 | -3.576E-003 |
| 8 | 0.43 | 0.43 | -3.576E-003 |
| 9 | 0.48 | 0.52 | -0.039 |
| 10 | 0.40 | 0.40 | 1.300E-003 |
| 11 | 0.35 | 0.35 | 1.982E-003 |
| 12 | 0.45 | 0.43 | 0.016 |
| 13 | 0.45 | 0.43 | 0.016 |
| 14 | 0.47 | 0.47 | 2.216E-004 |

Table 11**:** Actual values, Predicted Values and Residuals for Impact Strength.

| Standard Order | Actual Value | Predicted Value | Residual |
|---|---|---|---|
| 1 | 88.00 | 85.93 | 2.07 |
| 2 | 57.30 | 61.17 | -3.87 |
| 3 | 75.30 | 78.65 | -3.35 |
| 4 | 60.10 | 71.11 | -11.01 |
| 5 | 75.60 | 78.90 | -3.30 |
| 6 | 87.90 | 85.93 | 1.97 |
| 7 | 87.90 | 85.93 | 1.97 |
| 8 | 87.90 | 85.93 | 1.97 |
| 9 | 59.60 | 51.47 | 8.13 |
| 10 | 83.70 | 88.59 | -4.89 |
| 11 | 79.60 | 76.17 | 3.43 |
| 12 | 88.00 | 85.93 | 2.07 |
| 13 | 87.90 | 85.93 | 1.97 |
| 14 | 95.80 | 92.96 | 2.84 |

Table 11**:** Actual values, Predicted Values and Residuals for Brinell Hardness.

| Standard Order | Actual Value | Predicted Value | Residual |
|---|---|---|---|
| 1 | 137.00 | 136.53 | 0.47 |
| 2 | 135.70 | 149.34 | -13.64 |
| 3 | 199.80 | 204.75 | -4.95 |
| 4 | 144.60 | 121.56 | 23.04 |
| 5 | 130.50 | 113.63 | 16.87 |
| 6 | 137.00 | 136.53 | 0.47 |
| 7 | 136.30 | 136.53 | -0.23 |
| 8 | 136.30 | 136.53 | -0.23 |
| 9 | 139.80 | 142.70 | -2.90 |
| 10 | 452.20 | 420.47 | 31.73 |
| 11 | 378.20 | 393.38 | -15.18 |
| 12 | 136.30 | 136.53 | -0.23 |
| 13 | 136.30 | 136.53 | -0.23 |
| 14 | 211.50 | 246.49 | -34.99 |

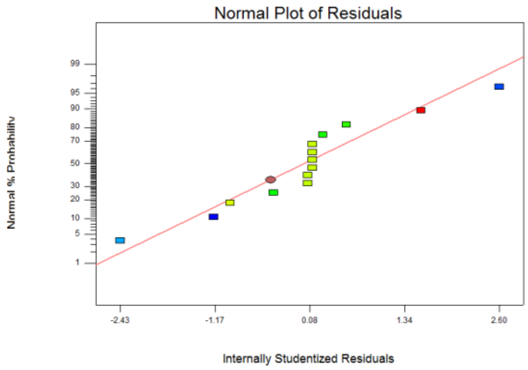


Fig 2: Normal Plot of Residuals for Tensile Strength.

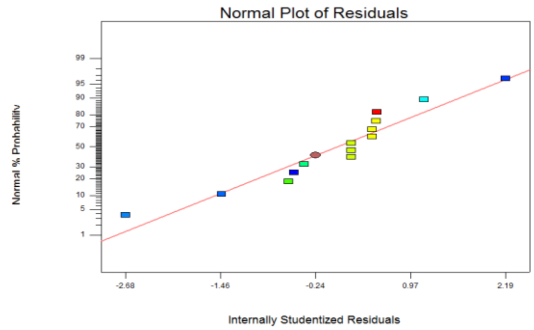


Fig 3: Normal Plot of Residuals for Percentage Elongation.

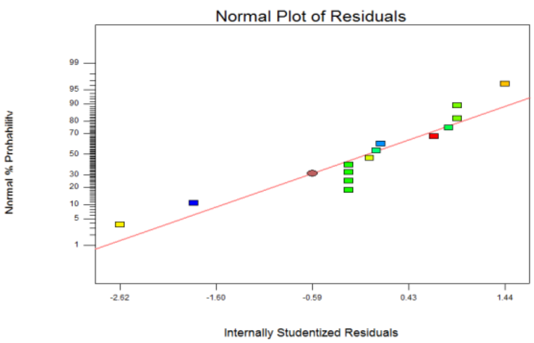


Fig 4: Normal Plot of Residuals for Tensile Modulus.

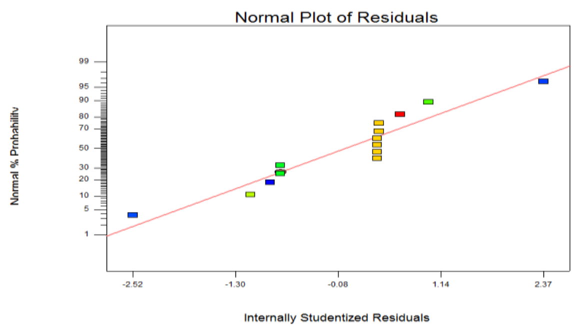


Fig 5: Normal Plot of Residuals for Impact Strength.

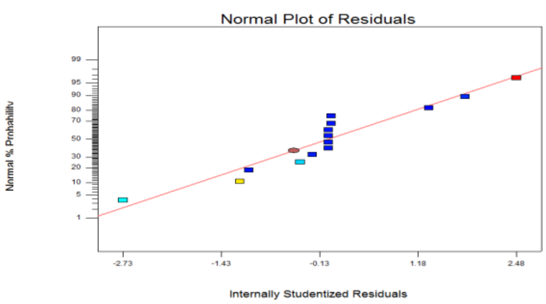


Fig 6: Normal Plot of Residuals for Hardness.

The actual response value versus the predicted response value graph was used to determine if the model is a satisfactory fit to the data. The condition is that the data point should be approximately split evenly by the 45 degree line [7]. Figure 7-11 shows the plot of predicted versus actual values for the tensile responses. The plots show that the data points were, approximately, evenly split by the 45 degree line. This show that the models are satisfactory fit to the data. All the values were well predicted by the data. From the above analysis, it can be concluded that this model is suitable for predicting the mechanical properties of forest flame pod flour-filled high density polyethylene composites within the limits of the experiment.

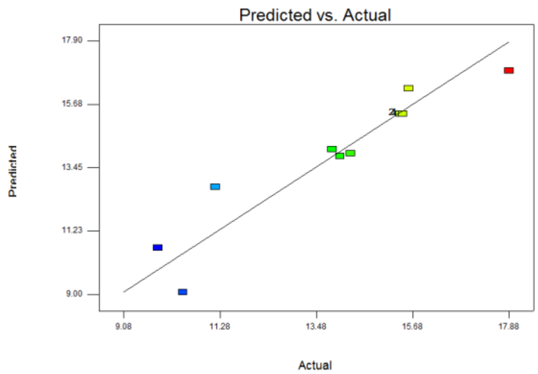


Fig 7. Plot of Predicted Versus Actual Response for Tensile. Strength

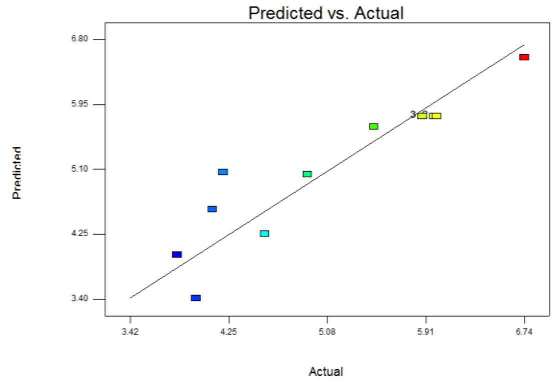


Fig 8. Plot of Predicted Versus Actual Response for Percentage Elongation.

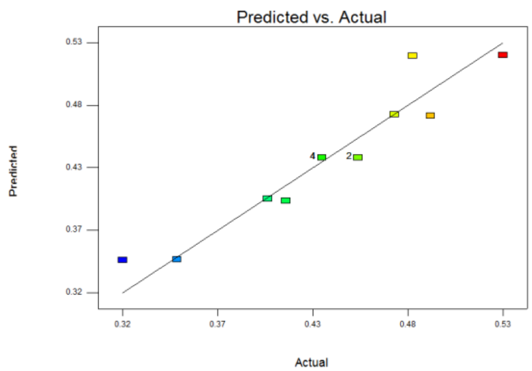


Fig 9. Plot of Predicted Versus Actual Response for Tensile Modulus.

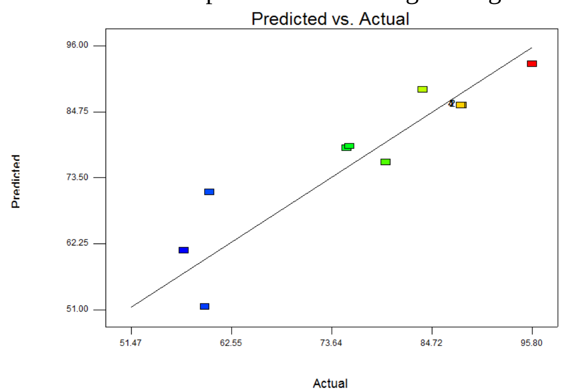


Fig 10. Plot of Predicted Versus Actual Response for Impact Strength

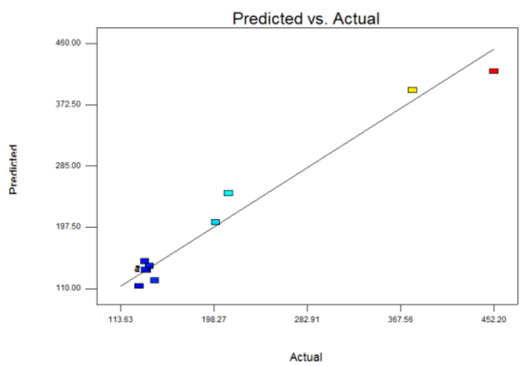


**Fig 11**. Plot of Predicted Versus Actual Response for Brinell Hardness

**Analysis of Response Surface:-**

The 3D response surface plot for the combined effect of particle size and filler content on tensile strength, percentage elongation, tensile modulus, impact strength and Brinell hardness value are shown in figures 12 through 16 respectively. The figures show that there is a quadratic effect of particle size and filler content on the responses except for tensile modulus. Figure 12 indicates that tensile strength decreases slightly with an increase in filler content. This result is similar to the one obtained by Obidiegwu et al [8] in “Walnut Shell Powder on the Properties of Polypropylene Filled Composite” and Salmah et al.[9] in “Coconut Shell Reinforced Unsaturated Polyester Composites.” According to Salmah et al [9], the decrease in tensile strength as the filler loading increases. This is due to the poor adhesion of the filler-matrix interface and the agglomeration of filler particles. From Fig 13, it can be inferred that the elongation at break of the composite decreased with an increase in the filler content. According to Ahmed [10], with growing filler content, the stiffness of the composite becomes gradually enhanced, with parallel diminution in the elongation at break. With the improvement in rigidity, the ductility of composites declines, consequently the composites break at lower elongation. The incorporation of filler that has a poor union to the matrix appears to cause disruption in the alignment of the polymer chains. The tensile modulus increases as both the filler content and size increased, as can be seen from Fig 14. This may be ascribed to the fact that the toughness of composites is improved by the addition of filler [11]. The impact strength of the composite increased with the increase in filler content as shown in Fig 15, while lower particle size favours impact strength. Such an increase in impact strength of a thermoplastic composite with an increase in filler content has been reported by Ahmed [10]; Bigg [12] in his study on "Mechanical Properties of Particulate filled Polymers and Igwe [13]. As the concentration of the filler content increased, Izod notched impact energy increased due to the more energy required for crack propagate [11]. The composite displayed the highest hardness values at higher filler contents as shown in Fig 16. This may be ascribed to the fact that the addition of fibers into plastic composite improved the matrix surface resistance to the indentation [14].

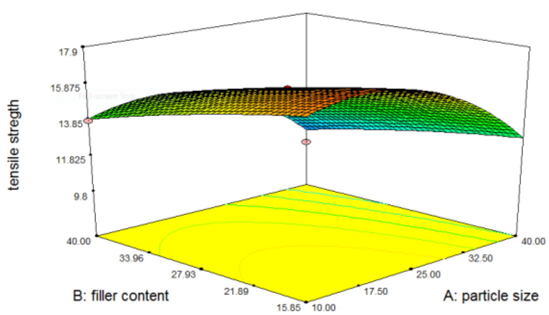


Fig 12: Response Surface Plot of the Combined Effect of Independent Particles on Tensile Strength

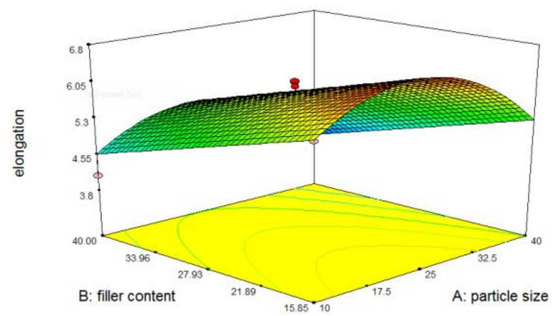


Fig 13: Response Surface Plot of the Combined Effect of Independent Particles on Percentage Elongation

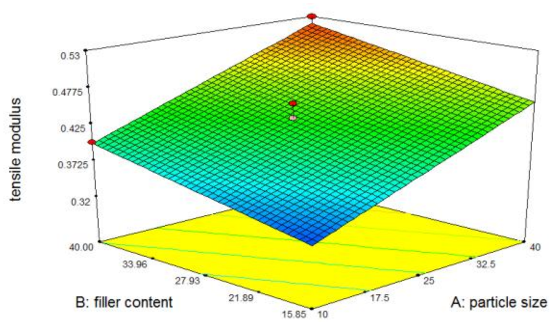


Fig 14: Response Surface Plot of the Combined Effect of Independent Particles on Tensile Modulus

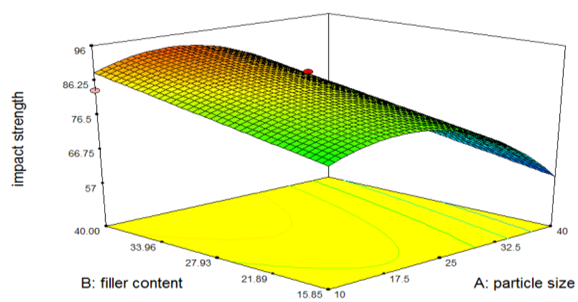


Fig 15: Response Surface Plot of the Combined Effect of Independent Particles on Impact Strength

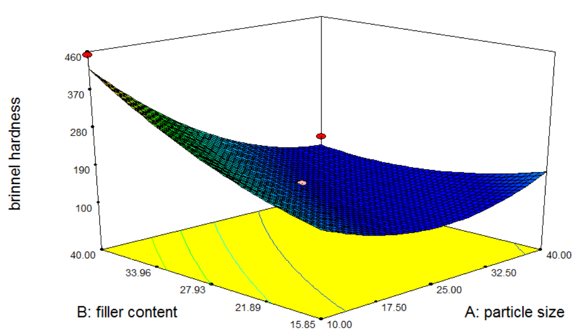


Fig 16: Response Surface Plot of the Combined Effect of Independent Particles on Brinell Hardness number.

Table 12**:** Chemical Composition of the Neem Bark.

| Fiber | cellulose (%) | Hemicellulose (%) | Lignin (%) |
|---|---|---|---|
| Neem Bark | 65.43 | 23.55 | 9.04 |

It can be viewed in Table 12, that the neem bark has very good cellulose, hemicellulose and lignin content. This result shows that bark cellulose content is close to some fibers in the literature.

**Process Optimisation:-**
Numerical optimization was used to explore the design space to determine factor settings that met the design goal. The goal is to maximize the responses. Desirability was used as the criteria for selecting factor settings used for the optimization. In this study, the factor settings that gave the combined optimum responses are 21.63 mesh size and 40% filler content at 77.7% desirability. These factors settings gave the responses of; 14.51MPa, 5.28%, 0.45GPa, 92.31KJ/m$^2$, and 242.14Pa for tensile strength, percentage elongation, tensile modulus, impact strength and hardness value respectively.

**Model Validation:-**
To validate the model, the optimum responses obtained from the model equations were compared to the experimental values obtained at the same factors setting. The_results are shown in Table 13. The closeness of the predicted values to the experimental values shows that the model can be used for reliable prediction within the experimental limit.

Table 13: Validation and confirmation of results for Neem Bark flour/HDPE.

| Factors | | Predicted Value | | | | | Experimental Value | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| Filler content (%) | Particle size (mesh size) | Tensile strength (MPa) | Elongation (%) | Tensile modulu (GPa) | Impact Strength (KJ/m$^2$) | BHV (Pa) | Tensile strength (MPa) | Elongation (%) | Tensile modulus (GPa) | Impact Strength (KJ/m$^2$) | BHV (Pa) |
| 21.63 | **40** | 14.51 | **5.28** | **0.45** | **92.31** | **242.14** | **14.40** | **5.18** | **0.52** | **92.15** | **242.94** |

## Conclusion:-
The work studied the effect of filler content and particle size on the mechanical properties of forest neem bark Flour-filled high density polyethylene composites. A quadratic regression model was developed for modeling the responses. Quadratic model was selected because of its high significance level. The model for all the responses shows high values of $R^2$ and the adequate precision for all the responses are above 4; thus the model can be used to predict the mechanical properties of neem bark flour-filled high density polyethylene composites. From the 3D response surface, it was found that increase in particle size improves all the mechanical properties within the experimental limit. Increase in filler content resulted in decrease of tensile strength and percentage elongation while tensile modulus, impact strength and hardness values improved. The optimal conditions with regards to the mechanical properties were found to be at 21.63mesh particle size and 40% filler content with a desirability of 77.7%. The corresponding responses at the optimal condition was found to be 14.51MPa, 5.28%, 0.45GPa, 92.31KJ/m$^2$and 242.14Pa for tensile strength, percentage elongation, tensile modulus, impact strength and hardness value respectively. The optimum conditions were validated with minimum error less than 2 %.

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