Nigerian Journal of Engineering, Vol. 28, No. 1, April 2021 ISSN (print): 0794 - 4756, ISSN (online): 2705-3954.

Nigerian Journal of Engineering,
Faculty of Engineering, Ahmadu Bello University, Zaria, Nigeria
journal homepage: www.njeabu.com.ng

The Effect of Process Parameters on Potash Treated Short PP/Honckenya Fibre Reinforced Composites using Response Surface Methodology (RSM)

***[1]N. I. Mbada, [1]O. Aponbiede, [1]U. Shehu and [2]M. T. Isa**

[1]Department of Metallurgical and Materials Engineering Ahmadu Bello University, Zaria, Nigeria.
[2]Department of Chemical Engineering Ahmadu Bello University, Zaria, Nigeria.
*izerk09@yahoo.com; aponbiede@yahoo.com; mrshehu53@gmail.com; mtisaz@yahoo.com

Research Article

Abstract

Natural fibre is increasingly used for polymer based composite development due to the fact that they are cheap, reneweable and eco-friendly. However, in order to boost their usage and area of application, there is need to optimize the process parameters of the end use properties of composites. In this research, Box-Behnken design based on response surface methodology was adopted to optimize the treated natural fibre as reinforcement of polypropylene matrix. Effect of fibre weight loading, compression time and compression temperature on tensile strength and modulus as responses were investigated. Honckenya fibre surface modification was done with Potash (KTN) at 4%w/w for 24hours. Short random Honckenya fibre reinforced PP composite were compounded at 200oC and subsequently compression moulded, afterwards tensile test was conducted on the developed composite. The critical fibre length of the short fibre was 5.09mm, while the fibres were chopped into 10mm length. From the analysis of variance based on Box-Behnken Analysis of Variance (ANOVA), the coefficient of determination R2 was 0.9935 and 0.9942 for tensile strength and modulus respectively. The multiple response optimization processing parameters were 48wt% fibre loading, 12.52minute and 173.26°C with tensile strength and modulus responses of 18.59MPa and 671.14MPa respectively at a desirability of 0.988.

Keywords

Honckenya, Box-Behnken, Critical Fibre Length, Polypropylene, Potash treatment, Natural fibre, Composites.

Article History

Received: – Sept., 2020
Accepted: – January, 2021
Reviewed: – Nov., 2020
Published: – March, 2021

1. Introduction

Natural fibres (NF) and their application in composite development are becoming more and more attractive to materials developers in building and construction, automobile original equipment manufacturers (OEMs), home and electronics appliances and research communities (Boland *et al.*, 2015; Fogorasi and Barbu, 2017; Peças *et al.*, 2018; Dahy, 2019). This attractiveness of natural fibre for composites material development is premised on some of the desirable attributes which natuaral fibres possessed. These include lightweight, cost effectiveness, biodegradability, eco-friendliness, renewability and ample reserve as alternative biomass source to fossil fuel sources, emerging chemical and bio-plastic resource base (Faruk *et al.*, 2013; Pereira, *et al.*, 2015).

Natural fibres have been used to reinforce different types of polymers, for instance Granda *et al.*, (2016) used a semicrystalline fibre of Leucaena *collinsii* as reinforcement for PP composites; also sisal reinforced epoxy composite sandwich structure were characterized for energy dissipation and sound wave attenuation by Zhang *et al.* (2018); Tong *et al.*, (2014) developed composites materials based on recycled HDPE and rice husk. In addition to the traditional textile fibres such as flax, hemp, jute, cotton, ramie that were first used in natural fibre reinforced composites, others such as kenaf, sisal, coir, banana and palm fibres have been used to a large degree of success (Mohammed *et al.*, 2015; Pereira *et al.*, 2015; Pickering *et al.*, 2016). Recently, researchers have also sought to add to the inventory of the existing natural fibres (Maache *et al.*, 2017; Manimaran *et al.*, 2018; Peças *et al.*, 2018). Mbada *et al.* (2019) characterized Honckenya fibre in order to establish its suitability to serve as reinforcement in polymer matrices. Among the various methods of optimization and prediction of end use properties of developed polymer composites, response surface methodology based on design of experiment is fast gaining acceptance due to its robustness, good and reliable predictive capability, statistical basis, easy to use and flexibility (Aimi *et al.*, 2014; Rasyid *et al.*, 2016). In that respect, Bar *et al.*, (2017) studied the effect of process parameters on novel thermally treated flax-Polypropylene based low-twist hybrid yarns for thermoplastic composites. Box-Behnken response surface methodology was used for the optimization of the process parameters which were feed core twist level, core-to-twist ratio (sheath ratio) and thermal treatment temperature on tensile modulus, flexural rigidity and weaveability responses. The research findings showed that hybrid yarn tenacity and hybrid yarn modulus decreased with increasing sheath ratio but increased with increase in surface treatment temperature. Also from the RSM analysis it was observed that yarn modulus increased with increase in core yarn twist.

Alzebdeh *et al.* (2016) used RSM methodology to optimize process parameters which are fibre volume fraction, NaOH concentration and treatment time on the tensile and flexural responses of chopped date palm frond reinforced with LDPE and PP composites. The response surface plot revealed that tensile strength of composites increased with increase in NaOH concentration, but decreased with fibre volume fraction, while the treatment time had little influence on the tensile strength. The study also established that the flexural strength of the composites also follow similar trend. Box-Behnken RSM method was used for optimization of parameters in compression moulding of woven flax reinforced PLA composites. The influence of three process variables-moulding temperature, time and pressure on impact strength was investigated by Kandar and Akil, (2016). The optimum process parameters for the compression moulding at a desirability value of 0.945 were 200°C, 3 min and 30 bar to give an impact strength value of 48.9KJ/m^2. The objectives of this research is to optimize process parameters such as fibre weight ratio, compression time and compression temperature of KTN treated short PP/Honckenya fibre reinforced composites (C-KS-PP/HFRCs) using Box-Behnken response surface methodology of design of experiment and to determine the tensile strength and modulus responses.

2. Materials and Method

2.1 Material Collection

Retted Honckenya fibre, Potash, Polypropylene (PP) pellets and steel mould

2.2 Methods

2.2.1 Potash (KTN) treatment

Potash salt (KTN) at concentration of 4%w/w was used to treat Honckenya fibre, the choice of 4%w/w concentration is based on initial screening test as well as on other research works (Mbada *et al.*, 2016). The pH value and conductivity of the solution at 26.0^{o}C were 10.20 and 1120µS respectively. The KTN was dissolved in water at a concentration of 4%w/w; because of the weak solubility of the potash, boiling water was used in the preparation of the 4%w/w KTN solution. The retted fibres were immersed in the KTN solution for 24h after which the solution was drained off and the fibres thoroughly washed with water to neutral pH.

2.2.2 Heat treatment of fibre

KTN treated fibres were subjected to heat treatment process using a Genlab MINO30 oven. The heat treatment temperature was set at 75^{o}C for 5h 30min. The oven temperature was then increased to 150^{o}C for 30min. The oven was switched off and the fibres were allowed to cool in the oven down to room temperature. After removal of the fibres from the oven it was stored in a plastic bag. The treatment cycle was performed in order to remove moisture from the fibre and to pre-condition the fibre to the compounding temperature, which is as high as 200^{o}C. Also, the storage of the fibre in a plastic bag is due to the fact that natural fibres are known to be hygroscopic materials; hence to forestall re-absorption of moisture by the fibre.

2.2.3 Determination of critical Fibre length

The critical fibre length for the short random Honckenya fibre reinforced PP composites was determined using the Equation 1:

$$L_c = \frac{\sigma_{Tf} D}{2\tau} \tag{1}$$

Where L_c is the critical length; σ_{Tf} is the fibre tensile fracture stress and τ is the matrix shear stress and D is the fibre diameter. Equation 2 gives the parameters for calculation of τ;

$$\tau = \frac{3P_{max}}{4bh} \tag{2}$$

Where, P_{max} is the maximum load at fracture, b is the width and h is the thickness of the specimen. From equations 1 and 2 the critical length was calculated based on the following parameters: P_{max} =105N, b = 19.5mm, h= 3.75 mm, σ_{Tf}=891.53MPa, D=12.32 μm, L_c= 5.09 mm.

2.2.4 Short fibre production and composite compounding

The Honckenya fibres were chopped into short fibres of 10mm length using strong steel scissors. Three different fibre weight ratios of the short random oriented Honckenya fibres were used for the compounding with neat PP. The PP together with the short Honckenya fibres were introduced into the two-roll mill at a temperature of 200^{o}C with the rear roll speed of 1.15rpm (revolution per minutes) and the front roll speed of 0.35rpm. The compounding process was carried out at NILEST, Zaria using two roll mill manufactured by Reliable Rubber and Plastics Machinery, U.S.A at fibre loading ratios of 40wt%, 44wt% and 48wt%.

2.2.6 Composites production

The compounded composites of the PP and the short fibres were moulded in a compression moulding machine model-Wenzhou Zhiguang. The compounded composites were placed in the steel mould with bottom and top platen, the mould was introduced into the compression moulding machine at high temperatures-varied according to the chosen factor levels and fixed pressure of 4MPa. The processing parameters- temperature and time were varied according to Box Behnken (BB) response surface methodology design of experiment. Table 1 shows the BB design of experiment (DOE) variables and the factor levels.

Table 1: Variable Levels for the BB Experimental Design

Variables	**Symbol**	**Coded Variables**		
		Low	Medium	High
	Code	-1	0	+1
Fibre Weight (%)	A	40	44	48
Time (sec)	B	10	12.5	15
Temperature (^{o}C)	C	165	170	175

Based on BB experimental design, 15 experimental runs were performed. The effect of fibre weight ratio (A), compression time (B) and temperature of forming (C) on the tensile response of the developed C-KS-PP/HFRCs were investigated at three factor levels.

2.2.7 Testing of Composites

The developed short random fibre reinforced PP composites were subjected to tensile test using specimen dimension of 100mm×12mm×4mm with a gauge length of 40mm according to ASTM (D638). The tensile test was conducted on Monsanto tensometer Type ‘W’ at room temperature at the strength of materials laboratory, Mechanical Engineering Department, A.B.U Zaria.

3. Results and Discussion

3.1 Critical Fibre Length

The critical fibre length of the Honckenya fibre was L_c= 5.09mm. The Honckenya fibres were chopped to 10mm length in order to ensure that fibre shortening below the critical fibre length as a result of the compounding process is avoided. It is believed that the fibre length in a short fibre reinforced composites should be longer than the critical length in order to ensure that the load bearing capability of the fibre would be achieved. At fibre length lower than the critical fibre length, the fibre would only exist as a particle filler in the matrix; therefore if the fibre length is lower than the critical fibre length reinforcing efficiency will not be achieved (Peltola *et al.*, 2011). Manufacturing process such as extrusion has been reported for shortening of fibre length during processing (Sanomura and Kawamura, 2003); it was also reported by Peltola *et al.*, (2011) that fibre shortening in compounding process occurred as a result of shear force acting on the fibre.

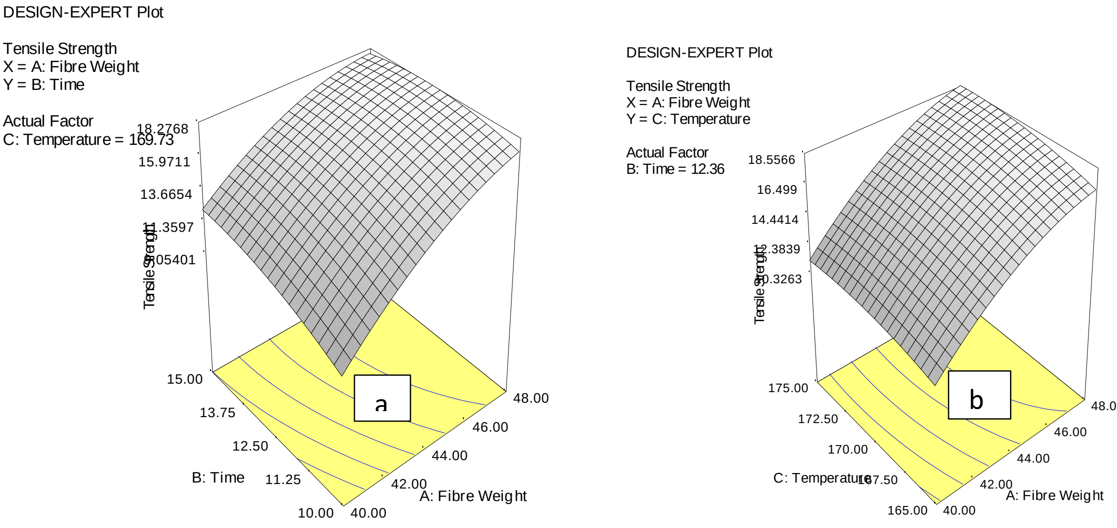
3.2 Short Random Fibre Composite

The KTN treated short PP/Honckenya fibre reinforced composites (C-KS-PP/HFRCs) tensile test responses based on BB design of experiment values is presented in Table 2 using the actual experimental values for the factor levels. The effect of fibre weight ratio (A), compression time (B) and temperature of forming (C) on the tensile strength and modulus responses of the developed C-KTN-TSHFRPC were determined for optimum processing conditions.

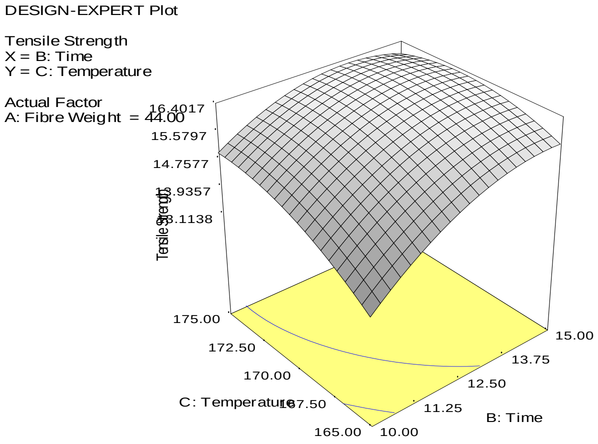
The 3-D response surface plot of the effects of the fibre weight% loading, time and temperature of compression on tensile strength and tensile modulus responses were obtained by keeping one of the factors constant at zero while varying the other two variables. The 3-D response surface plots for the parameters are shown in Figure 1(a, b and c) for the tensile strength responses.

Table 2: BB Design Matrix and Tensile Responses for (C-KTN-S-PP/HFRCs)

Run	A: Fibre Weight (wt%)	B:Time (Sec)	C:Temperature (ºC)	Tensile Strength (MPa)	Tensile Modulus (MPa)
1	44.00	10.00	165.00	13.10	422.10
2	44.00	12.50	170.00	16.05	574.59
3	44.00	15.00	165.00	16.00	472.73
4	40.00	12.50	165.00	10.14	350.24
5	40.00	10.00	170.00	9.40	337.38
6	44.00	10.00	175.00	14.53	483.72
7	48.00	10.00	170.00	17.48	626.46
8	48.00	12.50	165.00	17.02	570.38
9	40.00	12.50	175.00	11.33	407.36
10	40.00	15.00	170.00	12.04	390.90
11	44.00	15.00	175.00	16.01	526.09
12	48.00	15.00	170.00	17.56	634.27
13	48.00	12.50	175.00	18.82	647.50
14	44.00	12.50	170.00	16.37	579.78
15	44.00	12.50	170.00	16.17	570.55



(a) (b)

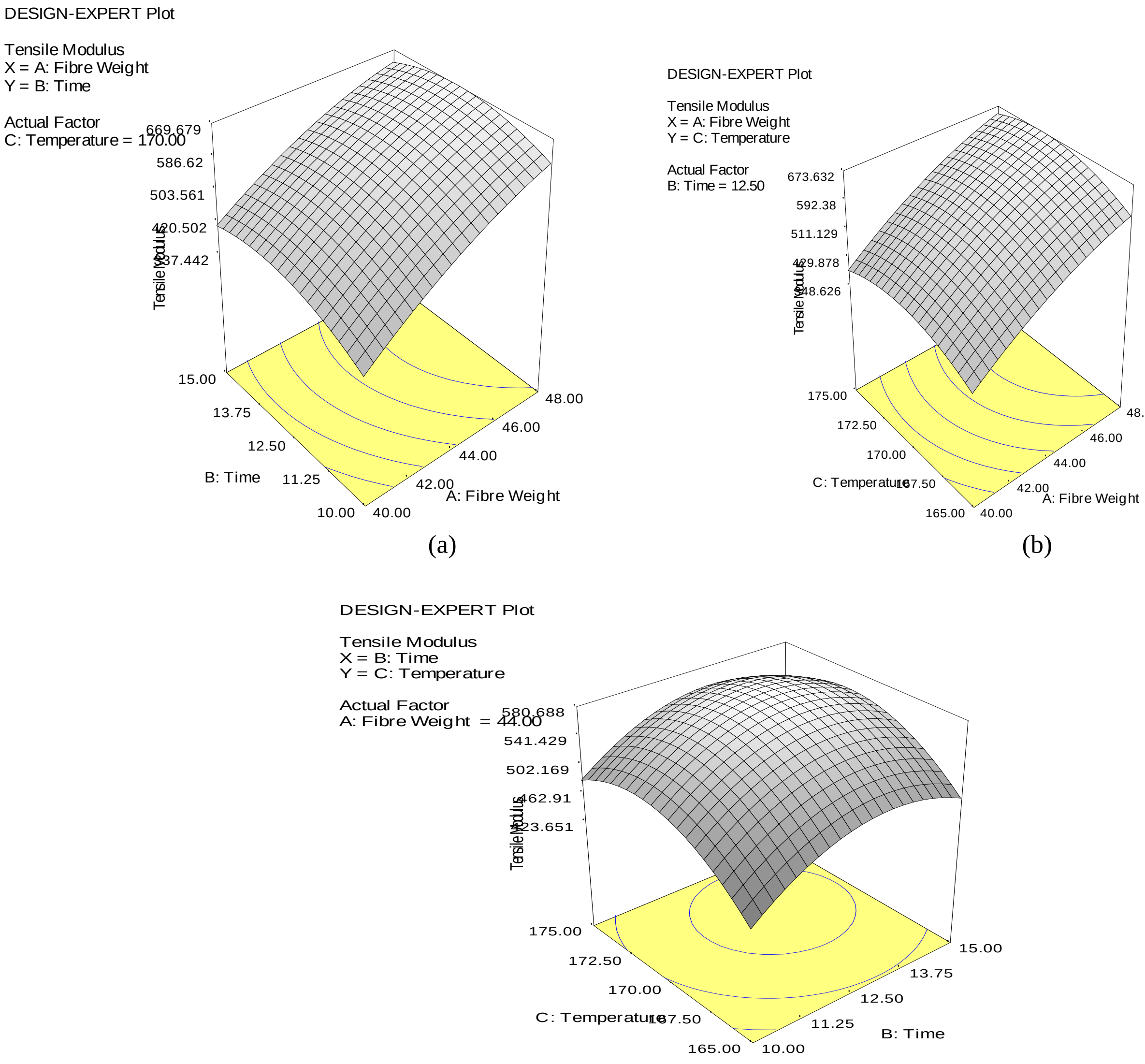


(c)

Figure 1: Response Surface Plot of a)Fibre Weight against Time, b) Fibre Weight against Temperature and c) Time against Temperature on Tensile Strength

The effect of fibre weight loading was more pronounced than that of time and temperature for the tensile strength. The fibre weight addition leads to an increase in tensile strength.

The 3-D surface plots of tensile modulus is presented in Figure 2 (a, b and c) for tensile modulus responses.



(a) (b)

(c)

Figure 2: Response Surface Plot of a) Fibre Weight against Time, b) Fibre Weight against Temperature and c) Time against Temperature on Tensile Strength

The plot of fibre weight addition against compression time and the plot of fibre weight against temperature on tensile strength and modulus show a sharp increase in tensile strength and modulus as a result of increase in fibre loading. For the 3-D surface plot of Time against temperature on tensile strength and modulus the tensile strength and modulus responses increased then dropped off toward the maximum factor levels of both time and temperature. The effect of fibre weight loading was more pronounced than that of time and temperature for tensile strength and modulus.

Numerical optimization of the response variables was performed with the target goal of maximizing the responses, while keeping the factor variables within the set range. From the optimization analysis, the best processing parameters based on desirability was chosen as the optimum processing parameters to give the optimum responses. The multiple response optimization processing parameters were 48wt% fibre loading, 12.52minute and 173.26ºC with tensile strength and modulus responses of 18.59MPa and 671.14MPa respectively at a desirability of 0.988. Therefore, the best processing parameters for the production of 4%w/w potash treated short PP/Honckenya fibre reinforced composites should be based on the optimized process parameters.

The Analysis of variance (ANOVA) tables for the BB-design are shown in Tables 3 and 4. The P-Value of the independent variables- fibre weight%, compression time and moulding temperature as well as other statistical parameters of interest are presented; similarly, linear, interaction and quadratic model terms are also shown on the ANOVA Table.

Table 3: ANOVA Response Table for Tensile Strength

Source Model	**Sum of Square**	**DF**	**Mean Square**	**F Value**	**Prob > F**	
	117.42	9	13.05	84.60	< 0.0001	significant
A	97.79	1	97.79	634.06	< 0.0001	
B	6.30	1	6.30	40.86	0.0014	
C	2.45	1	2.45	15.91	0.0104	
A^2	6.53	1	6.53	42.32	0.0013	
B^2	2.06	1	2.06	13.36	0.0147	
C^2	1.08	1	1.08	6.97	0.0460	
AB	1.64	1	1.64	10.62	0.0225	
AC	0.093	1	0.093	0.06	0.4725	
BC	0.50	1	0.05	3.27	0.1304	
Residual	0.77	5	0.15			
Lack of Fit	0.72	3	0.24	9.17	0.0999	not significant
Pure Error	0.052	2	0.026			

Table 4: ANOVA Response Table for Tensile Modulus

Source Model	**Sum of Square**	**DF**	**Mean Square**	**F Value**	**Prob > F**	
	1.535E+005	9	17052.37	96.03	< 0.0001	significant
A	1.234E+005	1	1.234E+005	694.92	< 0.0001	
B	2944.51	1	2944.51	16.58	0.0096	
C	7763.83	1	7763.83	43.72	0.0012	
A^2	3347.86	1	3347.86	18.85	0.0074	
B^2	8443.98	1	8443.98	47.55	0.0010	
C^2	9600.55	1	9600.55	54.07	0.0007	
AB	503.10	1	503.10	2.83	0.1532	
AC	100.00	1	100.00	0.56	0.4868	
BC	17.06	1	17.06	0.096	0.7691	
Residual	887.87	5	177.57			
Lack of Fit	845.05	3	281.68	13.16	0.0715	not significant
Pure Error	42.82	2	21.41			

The relationship between the dependent variables and the independent variables were modeled using BB design. The analysis of the BB-design response for the composites from ANOVA Table 3 and 4 show that the model is significant. The RSM model that best fitted the response is a quadratic model of the factors given main effects and interaction effects between the process factors. The F-Value of 84.60 and 96.03 for the tensile strength and tensile modulus respectively suggest that the model is significant and that there is only a 0.01% chance for tensile strength and modulus that large could have occurred due to noise. The lack of fit values of 0.0999 and 0.0715 is not significant for both tensile strength and modulus responses respectively. These values affirm the significance of the model. The model R^2 was 0.9935 for tensile strength, while the tensile modulus R^2 was 0.9942; the Adjusted R^2 values were 0.9817 and 0.9839 for tensile strength and modulus respectively. The signal to noise ratio as measured by adequate precision, for tensile strength and tensile modulus are 29.42 and 28.66 respectively. "Adeq Precision" with a ratio greater than 4 is desirable, and it implies that the variation in the observed response is not much affected by signal noise. The coefficient of determination R^2 of 0.9935 and 0.9942 for tensile strength and tensile modulus responses indicate that the variations observed in the response is totally accounted for by the relationship with the process variables.

4. Conclusion

Response surface methodology based on design of experiment using Box-Behnkem design was adopted for the optimization of process parameters of KTN treated short PP/Honckenya fibre reinforced composites (C-KS-PP/HFRCs). Composite compounding and compression moulding technique were adopted for the production of the composite.

With the following conclusions drawn from the study:

i. Honckenya fibre critical length was calculated to be 5.09 mm.

ii. The optimum process parameters for C-KS-PP/HFRC was 48wt% fibre loading, 12.52 minute and 173.26°C with tensile strength and modulus responses of 18.59MPa and 671.14MPa respectively at a desirability of 0.988.

iii. The F-Value of 84.60 and 96.03 for the tensile strength and tensile modulus respectively suggest that the model is significant and that there is only a 0.01% chance for tensile strength and modulus of with F-values of this magnitude could occur due to noise.

iv. The R^2 values of 0.9935 and 0.9942 for tensile strength and tensile modulus respectively indicate that there is a linear relationship between the process variables and the tensile responses.

iv. Fibre weight addition had a more pronounced effect on tensile strength and modulus responses.

5. Acknowledgment

I wish to acknowledge the contribution of my supervisors, whose depth of experience and length of years in teaching and research have impacted this work positively. I also wish to acknowledge the efforts of staff and students of the Department of Metallurgical and Materials Engineering A.B.U, Zaria.

References

Aimi, N. N., Anuar, H., Manshor, M. R., Nazri, W. B., & Sapuan, S. M. (2014). Optimizing the Parameters in Durian Skin Fiber Reinforced Polypropylene Composites by REsponse Surface Methodology. Industrial Crops and Products, 291-295.

Alzebdeh, K., Nassar, M., Al Rawahi, H., & Al–Hinai, N. (2016). Characterization of Mechanical Prtoperties of Date Palm Fronds Reinforced Composites: A Comparative Evaluation. Proceedings of the ASME 2016 International Mechanical Engineering Congress and Exposition. 14, pp. 1-10. Phoenix: ASME.

Bar, M., Das, A., & Alagirusamy, R. (2017). Studies on Flax-Polypropylene Based Low-Twist Hybrid Yarns for Thermoplastic Composite Reinforcement. Journal of Reinforced Plastics and Composites, 36(11), 1-14.

Boland, C. S., De Kleine, R., Keoleian, G. A., Lee, E. C., Kim, H. C., & Wallington, T. J. (2015). Life Cycle Impacts of Natural Fiber Composites for Automotive Applications: Effects of Renewable Energy Content and Lightweighting. Journal of Industrial Ecology, 179-189.

Dahy, H. (2019). Natural Fibre-Reinforced Polymer Composites (NFRP) Fabricated from Lignocellulosic Fibres for Future Sustainable Architectural Applications, Case Studies: Segmented-Shell Construction, Acoustic Panels, and Furniture. Sensors, 1-14.

Faruk, O., Bledzki, K. A., Fink, H.-P., & Sain, M. (2013). Progress Report on Natural Fiber Reinforced Composites. Macromelecular Materials and Engineering, 9-26.

Fogorasi, M. S., & Barbu, I. (2017). The Potential of Natural Fibres for Automotive Sector - Review . IOP Conf. Series: Materials Science and Engineering, 1-10.

Granda, L. A., Espinach, X., Méndez, J. A., Tresserras, J., Delgado-Aguilar, M., & Mutjé, P. (2016). Semichemical Fibres of Leucaena Collinsii Reinforced Polypropylene Composites: Young's Modulus Analysis and Fibre Diameter Effect on the Stiffness. Composites Part B, 1-18.

Kandar, M. M., & Akil, H. M. (2016). Application of Design of Experiment (DoE) for Parameters Optimization in Compression Moulding for Flax Reinforced Biocomposites. Procedia Chemistry, 19, 433-440.

Maache, M., Bezazi, A., Amroune, S., Scarpa, F., & Dufresne, A. (2017). Characterization of a novel natural cellulosic fiber from Juncus effusus L. Carbohydrate Polymers, 163-172.

Manimaran, P., Senthamaraikannan, P., Sanjay, M., Marichelvam, M., & Jawaid, M. (2018). Study on Characterization of Furcraea Foetida New Natural Fibre as Composite Reinforcement for Lightweight Applications. Carbohydrate Polymers, 650-658.

Mbada, N. I., Aponbiede, O., Ause, T., & Alabi, A. (2016). Effects of Mercerization Treatment on Kenaf Fibre (Hibiscus cannabinus L.). International Journal of Materials Engineering, 6(1), 8-14.

Mbada, N. I., Aponbiede, O., Shehu, U., & Isa, M. T. (2019). Characterization of Honckenya (Clappertonia ficifolia) Fibre as a Potential Natural Fibre Reinforcement for Polymeric Composites. Nigerian Research Journal of Engineering and Environmental Sciences, 4(2), 559-568.

Mohammed, L., Ansari, M. N., Pua, G., Jawaid, M., & Islam, S. M. (2015). A Review of Natural Fibre Reinforced Polymer Composite and its Applications. International Journal of Polymer science, 1-16.

Peças, P., Carvalho, H., Salman, H., & Leite, M. (2018). Natural Fibre Composites and Their Applications: A Review. Journal of Composites Science, 1-20.

Peltola, H., Madsen, B., Joffe, R., & Nattinen, K. (2011). Experimental Study of Fiber Length and Orientation in Injection Molded Natural Fibre/Starch Acetate Composites. Advances in Materials Science and Engineering, 1-7.

Pereira, P. H., Rosa, M. F., Cioffi, M. O., Benini, K. C., Milanese, A. A., Voorwald, H. J., & Mulinari, D. R. (2015). Vegetal Fibers in Polymeric Composites: A Review. Polímeros, 25(1), 9-22.

Pickering, K., Effendy, A. M., & Le, T. (2016). A Review of Recent Developments in Natural Fibre Composites and their Mechanical Performance. Composites: Part A, 98-112.

Rasyid, A. M., Salim, M. S., Akil, H. M., & Ishak, Z. A. (2016). Optimization of Processing Conditions Via Response Surface Methodology (RSM) of Nonwoven Flax Fibre Reinforced Acrodur Biocomposites. Procedia Chemistry, 19, 469-476.

Sanomura, Y., & Kawamura, M. (2003). Fiber Orientation Control of Short-Fiber Reinforced Thermoplastics by Ram Extrusion. POLYMER COMPOSITES, 24(5), 587-595.

Tong, J. Y., Royan, N. R., Ng, Y. C., Ghani, M. H., & Ahmad, S. (2014). Study of the Mechanical and Morphology Properties of Recycled HDPE Composite Using Rice Husk Filler. Advances in Materials Science and Engineering, 1-6.

Zhang, J., Shen, Y., Jiang, B., & Li, Y. (2018). Sound Absorption Characterization of Natural Materials and Sandwich Structure Composites. Aerospace, 5(75), 1-13.