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Production of Spindle Palm Petiole Fiber Reinforced High Density Polyethylene Composites

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ABSTRACT

The work is on the production of spindle palm petiole fiber reinforced high density polyethylene (HDPE) composites. The Spindle Palm Petiole Fiber (SPPF) and HDPE composites were produced using injection molding machine. SPPF were characterized to determine their chemical compositions. Central Composite Design (CCD) was applied as an optimization tool of RSM for cellulose and tensile strength. The chemical composition of the SPPF is cellulose (65%), hemicelluloses (17.1%) and lignin (14.1%). Surface modifications of the fiber enhanced the properties of the fiber. Quadratic model adequately described the relationship between percentage cellulose yield and variables: chemical concentration, mass/volume ratio and time. The cellulose content at optimal level is 60.3% at 3.5wt% concentration, 4g/l mass/volume ratio and time of 16hr. Also for the composite, the quadratic model described the relationship between tensile strength and temperature, fiber/polymer ratio and time. The optimum tensile strength of 42.0 Mpa was obtained at fiber/polymer ratio of 29wt%, temperature of 172 °C and time of 10 min. Water absorbed by the untreated fiber was high compared to the chemically treated fiber. The chemical treatment created a better interfacial bonding of SPPF/HDPE and this could be responsible for the observations.

Keywords: Spindle palm petiole fiber, Sodium hydroxide, cellulose, chemical composition, Central Composite Design, Response Surface Methodology, tensile strength, polyethylene

1. INTRODUCTION

A composite is a material made from two or more different materials which when combined are stronger and superior than those individual materials used. Composites are designed with a particular use in mind like added strength, efficiency, light weight or durability. Polymeric composites are polymers that have been filled with natural compounds or synthetic materials so as to improve their physical and chemical properties or to reduce cost (Ejikeugwu, 2021; Dayanidhi, 2019). Natural fibers composites are gaining importance due to increased knowledge in environmental safety and utilization of renewable materials towards greener society (Satyanarayana et al 2009). Natural fibers are non-carcinogenic, bio-degradable and easily obtainable from biomass, forestry and agricultural residues. Esther and Johnson (2017) opined that based on the sustainability benefits, bio-fibers such as plant fibers are substituting synthetic fibers in composites.

According to Jawaid et al (2014), some advantages of natural fibers are non hazardous to man density per unit volume, low cost, renewability and biodegradability. Thus, due to these numerous advantages, natural fibers are replacing synthetic fiber which is of high cost; they create severe ecological and health hazards to human. Although the main disadvantage of natural fiber composite is high moisture absorption which has resulted to poor fiber/matrix interaction and decrease in the properties of the composites thus obstructs their industrial utilization and production.

So for any polymeric composite to have good properties, there must be a homogeneous distribution of the reinforcing component, good orientation and high aspect ratio of the natural fiber and this could be achieved by using chemicals for the surface modification of natural fibers. Kalia et al (2009) stated various surface modification techniques that could be used to remedy the problem of low interfacial bonding between the natural fibers and polymer matrix.

Furthermore, there are several potential fibers that have not been discovered yet. Spindle palm fibers are available in huge amounts in almost every part of Nigeria, and there is no much understanding of their properties and effect of filler size reduction on the petiole of the fiber.

2. EXPERIMENTAL

2. 1. Materials and methods

Materials used for this study are fiber (SPPF) and High density polyethylene. The polyethylene was purchased from the Indorama Eleme Petrochemicals Limited, Port-Harcourt, Nigeria. The SPPF was prepared from the petiole of spindle Palm frond by mechanical method. This was done by decortications /traumatizing of the petiole as described by Deyholos and Potter (2014).

Kengkhetkit and Amornsakchai (2012) noted that fibers can be decorticated without significant damage and can be produced with high quality, even when no pretreatments are carried out. This method separated the fibers apart as it is shown in Figure 1 & 2.

The fibers extracted were cleaned, washed thoroughly with distilled water that removed most of the impurities and sun dried for two days. The clean SPPF were treated with of Sodium Hydroxide (NaOH) for varied concentration and time. Thereafter, they were washed with distilled water to remove the excess NaOH, and then dried in an oven at 70 °C for 24 hours.

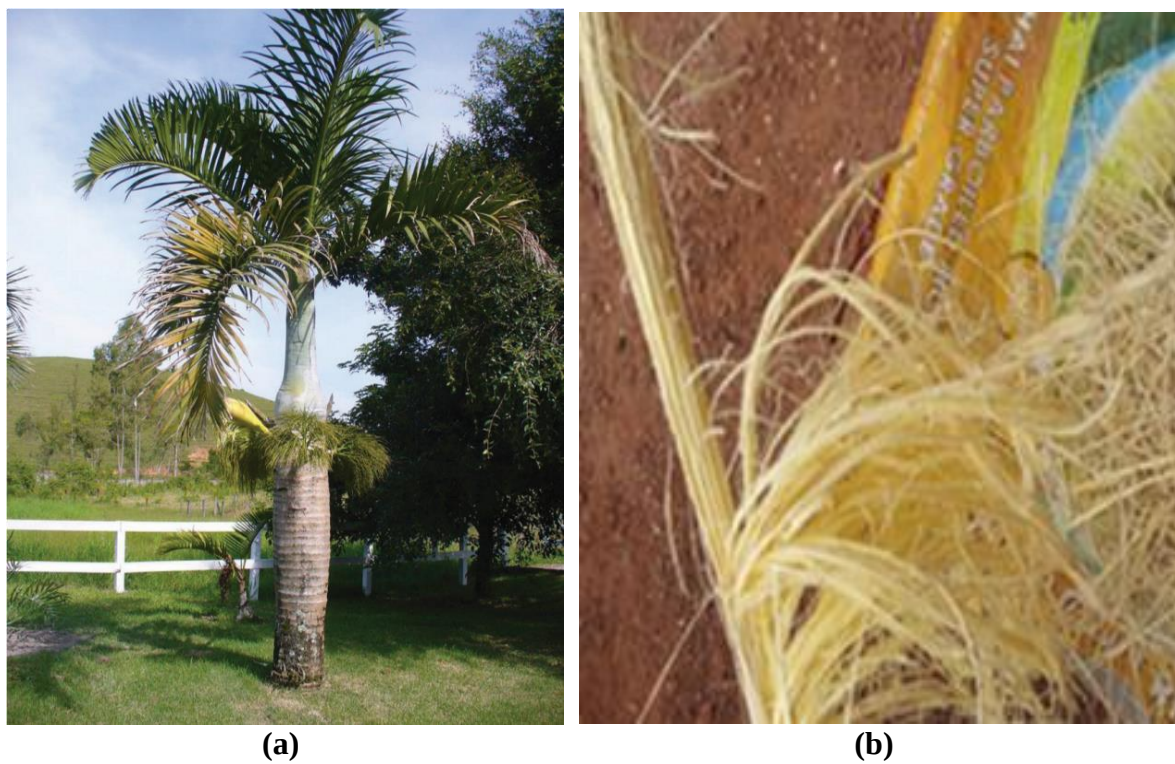


Figure 1. (a) Spindle palm tree; (b) mechanically extracted spindle palm petiole fiber (SPPF)



Figure 2. SPPF treated with NaOH

2. 2. Characterization

The chemical composition of SPPF was determined using TAPPI Standard Test Method T204 cm-17 for extractives, TAPPT Standard Test Method T203 cm-99 for α cellulose and lignin content by TAPPI Standard Test Method T222. Fourier transform infrared (FTIR) spectroscopic analysis was done with Buck scientific M530 USA. The morphology of DPPF was carried out with JEOL JSM 7500F Scanning Electron Microscope (SEM). The particle size reduction of the SPPF was carried out with a laboratory mill made by Christy and Norris Ltd, Chelmsford, England. The fiber was milled to fine powder. The same process was repeated continuously till the fiber particle size of 150 μ m was gotten.

2. 3. Determination of effects of process variables

Effects of process variables on the chemical composition of the fibers were determined considering variables: concentration of the chemicals, the mass/ volume ratio of the chemicals and time. Also the effects of process variables on the mechanical properties of the composites were determined by varying the effect of fiber/polymer ratio, temperature and time.

2. 4. Experimental Design for Optimization Study

Central Composite Design (CCD) of design expert software (version 11) was applied as optimization tool of RSM for percentage cellulose and tensile strength. The RSM experimental data was analyzed using analysis of variance (ANOVA), fit statistics, mathematical modeling and graphical plots. Optimum response was obtained was validated.

2. 5. Composites Production

In the composites production, fibers, resin and coupling agent (Maleic Anhydride Polyethylene – MAPE) were mixed physically in a bowl and then poured into injection molding machine where the proper mixing and melting occurred and mechanical test samples were formed inside the molds produced with vertical milling center CNC. Then a press was used for compression process.

2. 6. Test procedures

Universal Testing Machine (Testometric 2500kgf Rochdale England of serial no 52978) was used for mechanical properties of the composites at cross-head speed of 50 mm/min according to ASTM D638 for tensile strength, ASTM D790 for flexural and ASTM D6110 -18 for Charpy impact test at standard laboratory atmosphere.

3. RESULTS AND DISCUSSION

3. 1. Chemical composition of the fiber.

It was seen that the chemical composition of the untreated fiber is cellulose (40.65%), hemicelluloses (31.23%) and lignin (21.62%). The presence of high cellulose content is an indication that SPPF is a potential natural fiber resource, and makes it suitable for application in polymeric composites (John et al, 2008). The Chemical composition of the treated SPPF with NaOH varied significantly at different condition.

From Figure 3, 4 and 5, the percentage cellulose increased with increase in the variables. From the three factors considered, the peak was noticed at 60%, 3.5wt%, 4g/l and 16hr before it started reducing for percentage cellulose, concentration, mass/volume ratio and time respectively. This shows that NaOH has high extractive properties. This is similar to the observation of Sreekala et al (2000).

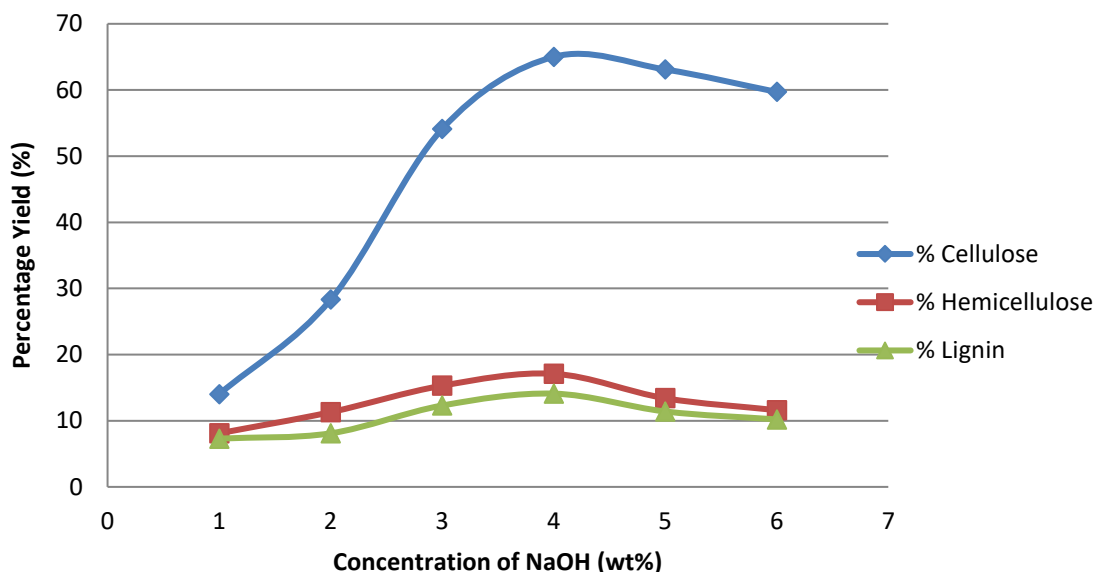


Figure 3. Yield versus concentration of SPPF treated with Sodium hydroxide.

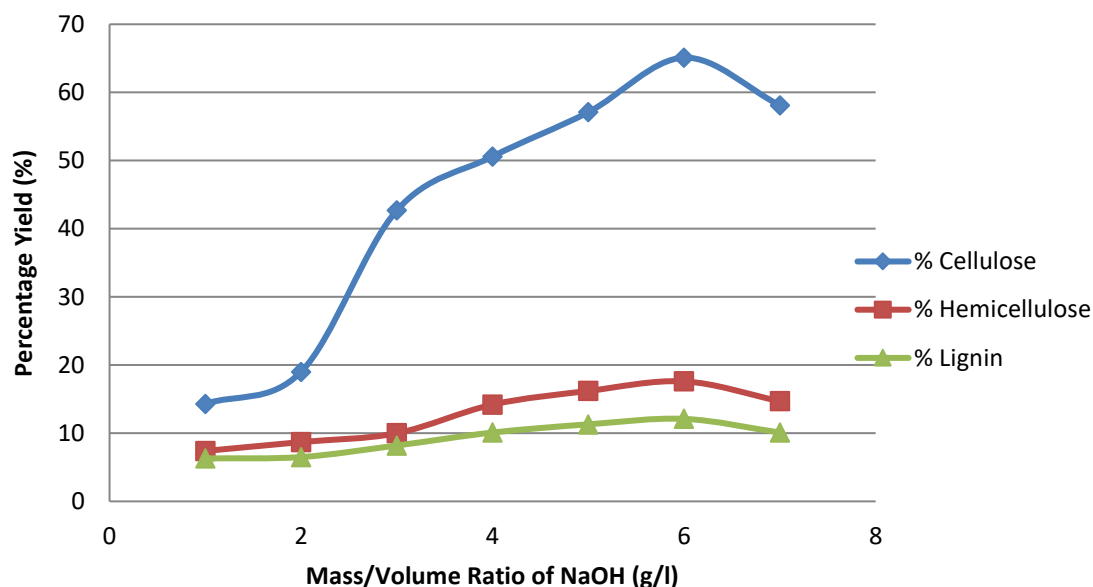


Figure 4. Yield versus Mass/Volume ratio of SPPF treated with Sodium hydroxide

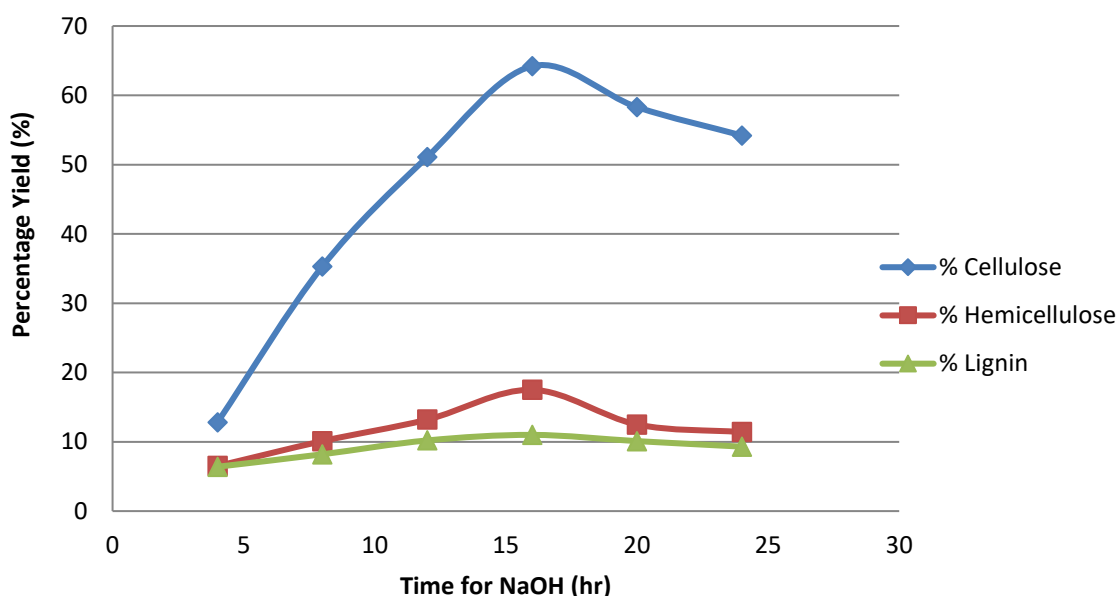


Figure 5. Yield versus Time of SPPF treated with Sodium hydroxide

3. 2. RSM Results of Percentage Cellulose as a function of the treatment factors

The RSM result of percentage cellulose of SPPF treated-NaOH is presented on Table 1. It is observed that percentage cellulose varied with interactive factors of concentration, mass/volume ratio and time. It showed trend of increased percentage cellulose with increase in considered factors till the maximum point is reached. The nature of the effect of the interactions among the factors on the cellulose yield can be seen through analysis of the experimental data like analysis of variance, fit statistics, diagnostic and 3D plots.

Table 1. RSM Results of SPPF treated with NaOH

Std	Run	A: Concentration wt%	B:Mass/Volume Ratio, g/L	C: Time Hr	Percentage Cellulose, %
5	1	2	2	24	28.6
14	2	3.5	4	24	54.3
12	3	3.5	6	16	64.7
11	4	3.5	2	16	35.0
13	5	3.5	4	8	56.6
8	6	5	6	24	44.5
20	7	3.5	4	16	60.0

2	8	5	2	8	14.0
3	9	2	6	8	51.0
1	10	2	2	8	12.6
9	11	2	4	16	52.8
7	12	2	6	24	54.0
4	13	5	6	8	45.2
10	14	5	4	16	40.7
16	15	3.5	4	16	60.0
17	16	3.5	4	16	60.0
15	17	3.5	4	16	60.0
19	18	3.5	4	16	60.0
6	19	5	2	24	15.0
18	20	3.5	4	16	60.0

3. 3. Analysis of variance (ANOVA) and fit Statistics

The ANOVA and fit statistics of % cellulose of SPPF treated - NaOH is presented on Table 2. The Model F-value of 122.47 implies the model is significant P-values less than 0.0500 indicate significant model. In this case A, B, C, AC, BC, A², B², C² are significant model terms and the three factors – concentration, mass/volume ratio and time were responsible for the quadratic nature of the model.

Table 2. ANOVA and Fit statistics of percentage cellulose in SPPF treated-NaOH.

Source	Sum of Squares	Df	Mean Square	F-value	p-value	
Model	5344.94	9	593.88	122.47	< 0.0001	Significant
A-Concentration	156.82	1	156.82	32.34	0.0002	
B-Mass/Volume Ratio	2377.76	1	2377.76	490.34	< 0.0001	
C-Time	28.90	1	28.90	5.96	0.0348	
AB	1.20	1	1.20	0.2477	0.6294	

AC	43.71	1	43.71	9.01	0.0133	
BC	27.01	1	27.01	5.57	0.0399	
A ²	455.05	1	455.05	93.84	< 0.0001	
B ²	262.15	1	262.15	54.06	< 0.0001	
C ²	47.67	1	47.67	9.83	0.0106	
Residual	48.49	10	4.85			
Lack of Fit	48.49	5	9.70			
Pure Error	0.0000	5	0.0000			
Cor Total	5393.43	19				
Std. Dev.	2.20		R ²		0.9910	
Mean	46.45		Adjusted R ²		0.9829	
C.V. %	4.74		Predicted R ²		0.8843	
			Adeq Precision		33.8058	

3. 4. Mathematical model

The mathematical model of percentage cellulose as a function of treatment factors in terms of the significant is presented in Equations 1. The percentage cellulose was dependent on concentration, mass/volume ratio and time. It is a quadratic model with the highest power of the variables being two.

$$\text{Percentage Cellulose} = +59.85 - 3.96A + 15.42B + 1.70C - 0.3875AB - 2.34AC - 1.84BC - 12.86A^2 - 9.76B^2 - 4.16C^2 \quad (1)$$

3. 5. Graphical Analysis of the Percentage Cellulose

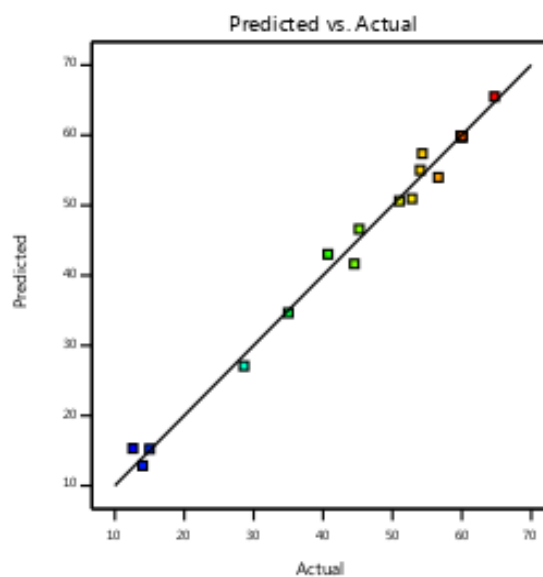
The graphical analyses of the percentage cellulose of treated SPPF are shown in Figure 6. Figure 6a is a plot of the predicted values versus actual values of the percentage cellulose in SPPF treated with NaOH. The points clustered along the line of best fit, which indicated that the generated model adequately predicted the actual yield of the cellulose. In Figures 6b, c and d, 3D plots of percentage cellulose as a function of the interactions between the factors: concentration and mass/volume ratio; concentration and time and lastly, mass/volume ratio and time respectively are observed. The percentage cellulose increased with increase in all the factors until it got to a peak which was the optimal points. Optimal percentage cellulose of 63.85% was obtained at optimal time of 16.92hr, optimal concentration of 3.75 g/l and optimal mass/volume ratio of 4.57wt%.

Design-Expert® Software
Trial Version

Percentage Cellulose

Color points by value of
Percentage Cellulose:

12.6  64.7



(a)

Design-Expert® Software
Trial Version

Factor Coding: Actual

Percentage Cellulose (%)

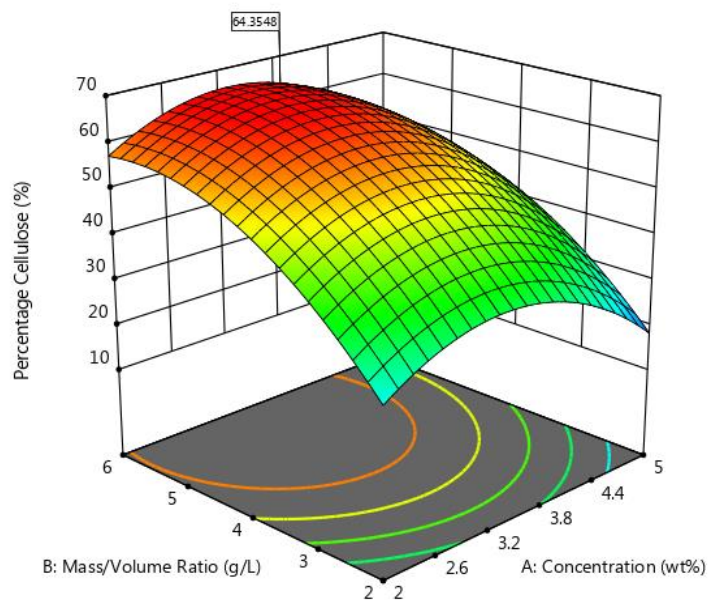
12.6  64.7

X1 = A: Concentration

X2 = B: Mass/Volume Ratio

Actual Factor

C: Time = 17.3333



(b)

Design-Expert® Software

Trial Version

Factor Coding: Actual

Percentage Cellulose (%)

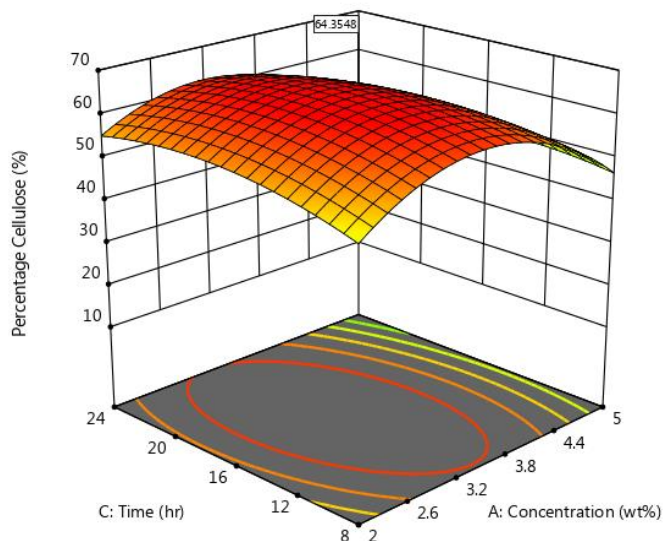
12.6 64.7

X1 = A: Concentration

X2 = C: Time

Actual Factor

B: Mass/Volume Ratio = 5.93333



(c)

Design-Expert® Software

Trial Version

Factor Coding: Actual

Percentage Cellulose (%)

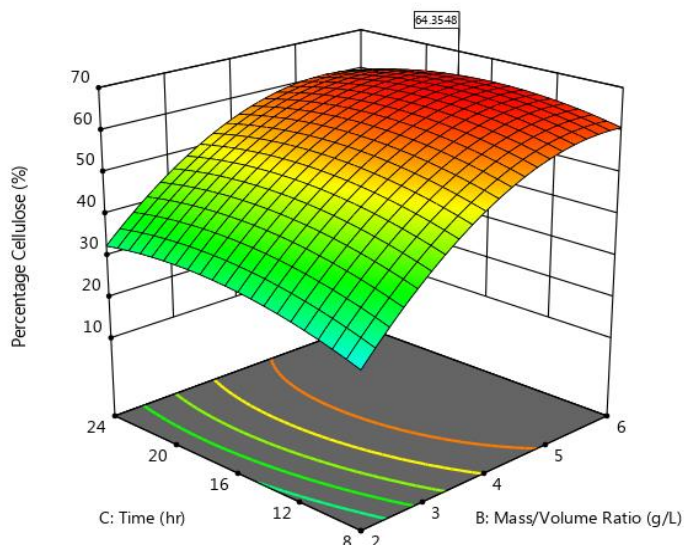
12.6 64.7

X1 = B: Mass/Volume Ratio

X2 = C: Time

Actual Factor

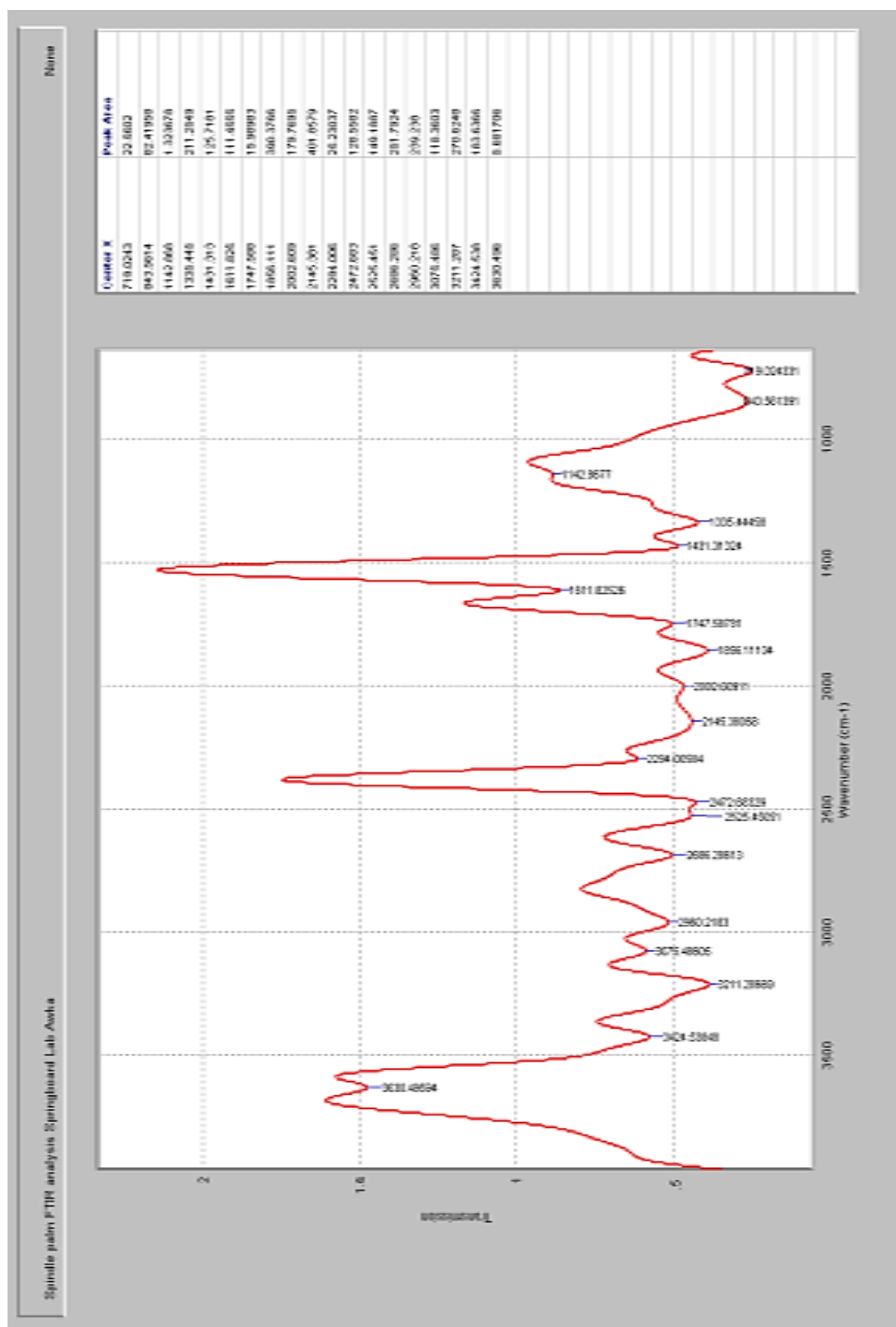
A: Concentration = 3.75



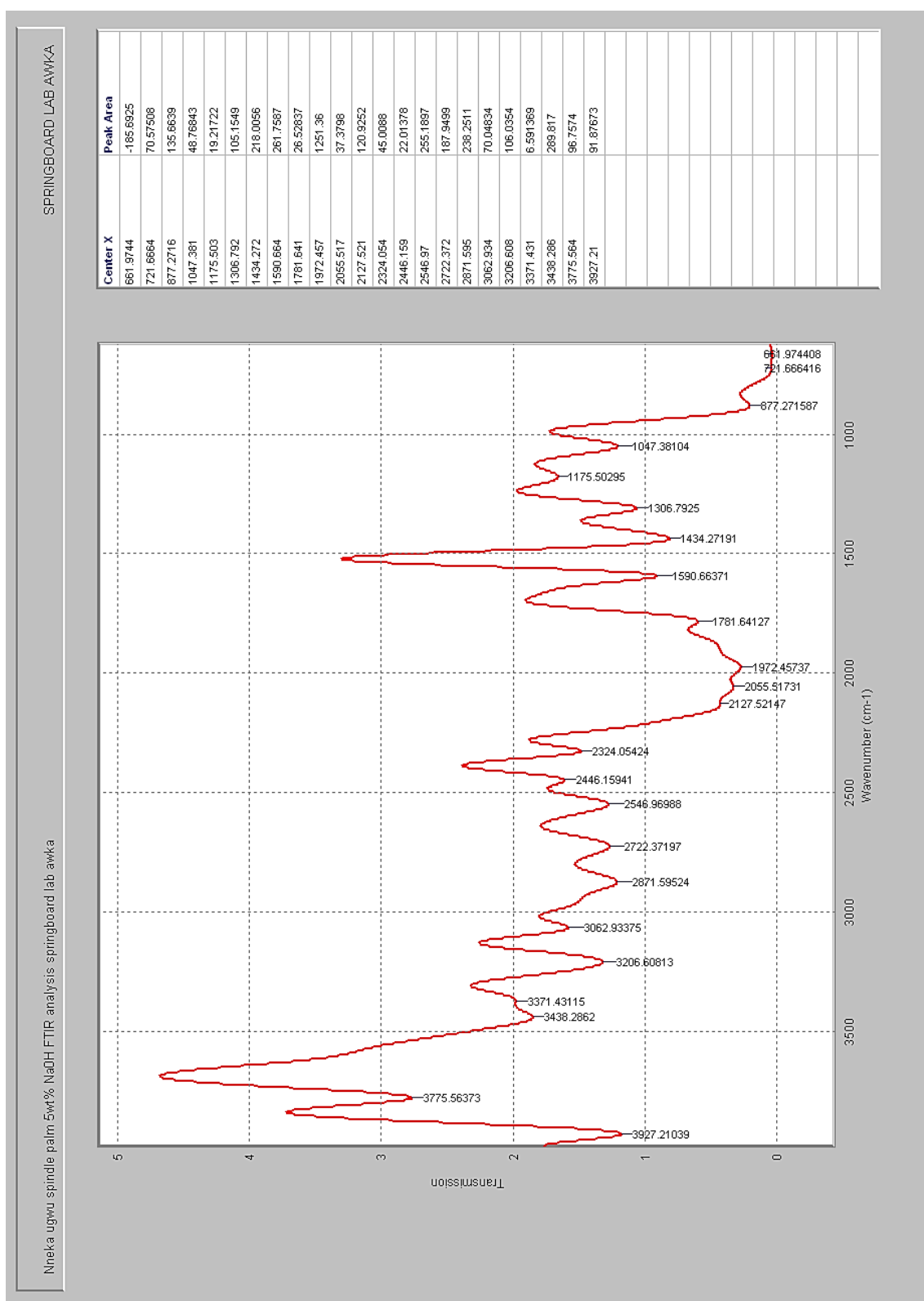
(d)

Figure 6. (a) Predicted Versus Actual percentage, (b) Percentage cellulose versus Concentration and mass/volume ratio, (c) Percentage cellulose versus Concentration and Time and (d) Percentage cellulose versus Mass/Volume ratio and Time for the SPPF treated with NaOH.

3. 6. Functional groups of untreated DPPF



(a)



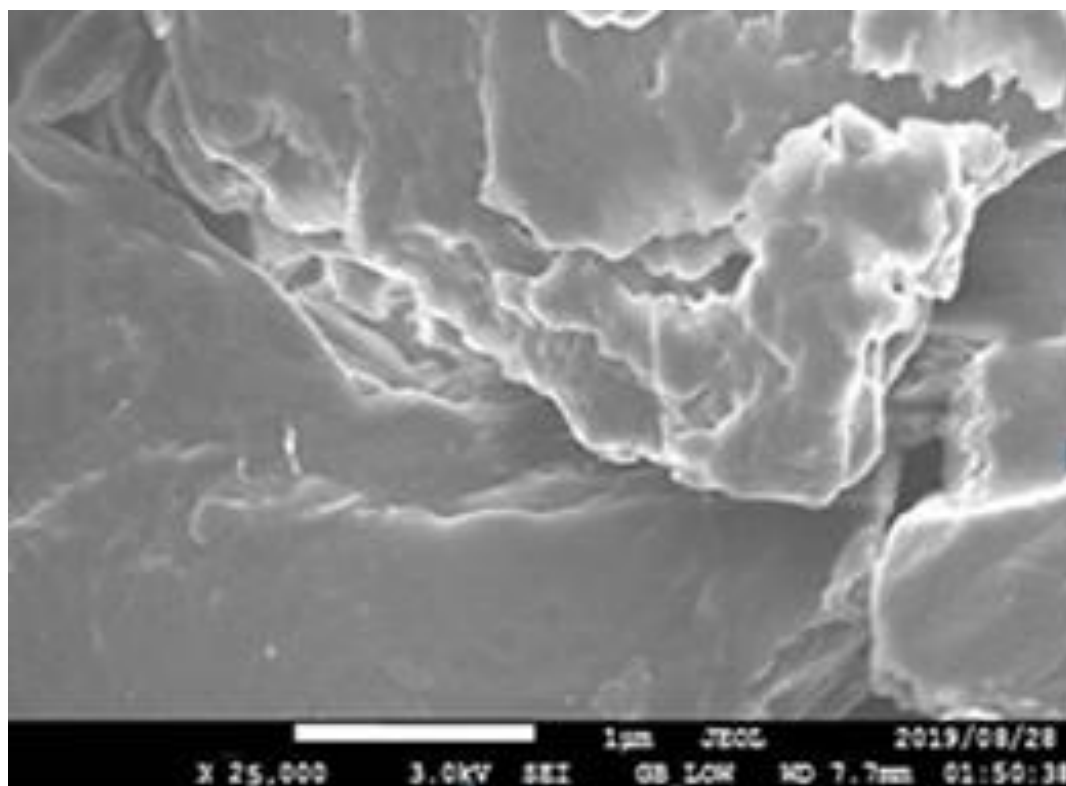
(b)

Figure 7. FTIR Spectroscopic Analysis of (a) untreated SPPF, (b) NaOH treated SPPF

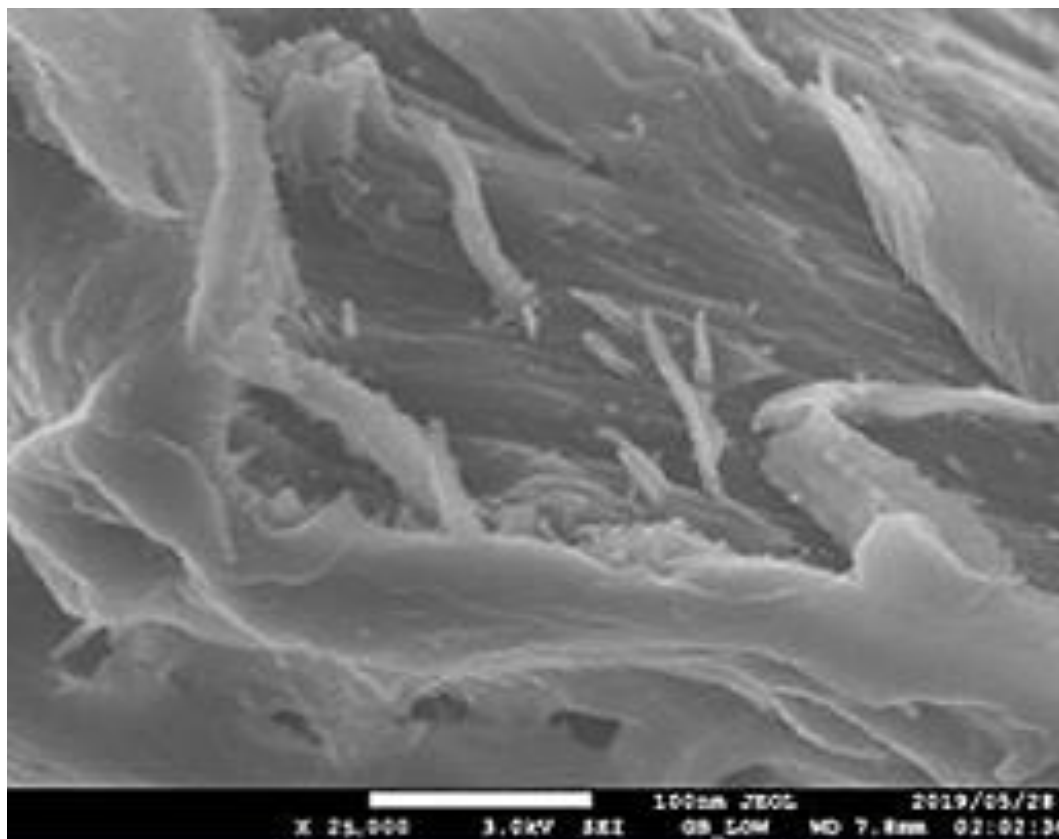
The functional group of untreated and NaOH-treated SPPF is shown in Figure 7a and b. The transmittance peaks of interest were identified. The result of untreated SPPF showed peak at 3200 cm^{-1} corresponding to hydroxyl stretching vibrations in hemicelluloses and/or cellulose. The peak at 1700 cm^{-1} corresponds to carbonyl group from either carboxylate groups or ester linkages in pectin. The peak located at 1600 cm^{-1} wave number belongs to C=C bonds or aromatic bonds from lignin. It was observed that there were structural changes and increase in the vibrations after chemical treatment. Some of the lignin and hemicelluloses were removed after the treatment thereby exposing more of the hydroxyl group in the cellulose making the active sites in the fiber ready for reactions. Also the peak at 1700 cm^{-1} disappeared after chemical treatment on the fibers. Chemical treatment also reduced hydrogen bonding due to removal of the hydroxyl groups by reacting with the chemicals. This is in agreement with the reports of (Skreekala et al 2000; Abidi and Herath, 2017). The peak located at 1600 cm^{-1} decreased in treated fibers. There is an indication of in-plane bending vibrations of the $-\text{CH}_2$ and $-\text{CH}$ groups of cellulose (Prasanna and Velmourougane, 2017) and C-H deformation in the amorphous region of the celluloses in the treated SPPF.

3. 7. Morphology

The micrograph of the SEM of NaOH treated SPPF micro cellulose is presented in plate 1. It was observed that the untreated one has presence of wax, oil, and surface impurities but that of treated micrographs shows successful removal of the surface contaminants and also have exposed surface more than the untreated one. This means that the surface of the NaOH treated SPPF will be readily available to react with the matrix (HDPE).



(a)



(b)

Plate 1. (a) SEM of untreated SPPF and (b) SEM of SPPF treated with NaOH

3. 8. Water absorption rate

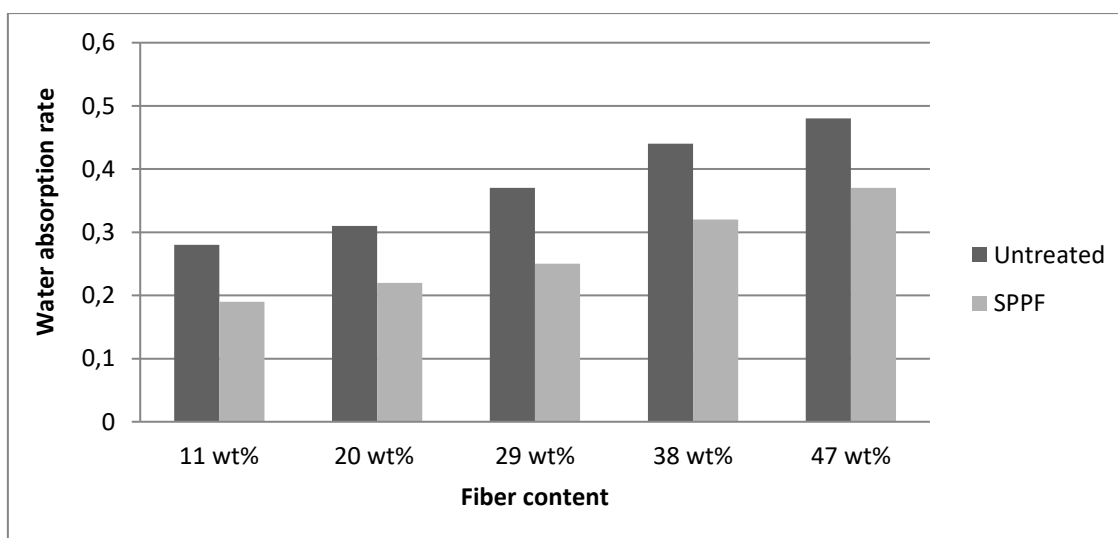


Figure 8. Effect of fiber/polymer ratio on water absorption rate of composites.

The water absorption rates of the composites made with treated and untreated SPPF is presented in Figure 8. It was observed that the water absorbed by the composites made with untreated fibers was high compared to that of NaOH - treated SPPF composites. This might be due to better interfacial bonding or possible removal of hemicelluloses during the chemical treatment resulting to a more hydrophobic fiber as hemicelluloses is the most hydrophilic component of fiber (Saheed and Jog 1999). Chemicals activate the hydroxyl groups or introduce new molecules which effectively interlock with the matrix (Ali et al 2017).

3. 9. Mechanical properties of the composites

The Effect of fiber/polymer ratio on the tensile strength of SPPF composites is presented on Figure 9. It was seen that tensile strength of the chemically treated composite is higher than untreated composites. This might be due to surface modification that resulted in more exposed surface readily available to react with another surface and enhancement in the adhesion between the fiber fibrillation. The tensile strength increased as the fiber/polymer ratio was increasing till maximum tensile strength of 48.5MPa at fiber/polymer ratio of 29/69. Then after this point, there was a decrease in tensile strength. This might be as a result of inefficient fiber/polymer bonding at a higher fiber ratio that can lead to fiber–fiber contact and thereby lowers stress transfer between fibers and the matrix (Al-Khaabi et al 2005). The tensile modulus of both treated and untreated SPPF composites increased as the fiber ratio increased

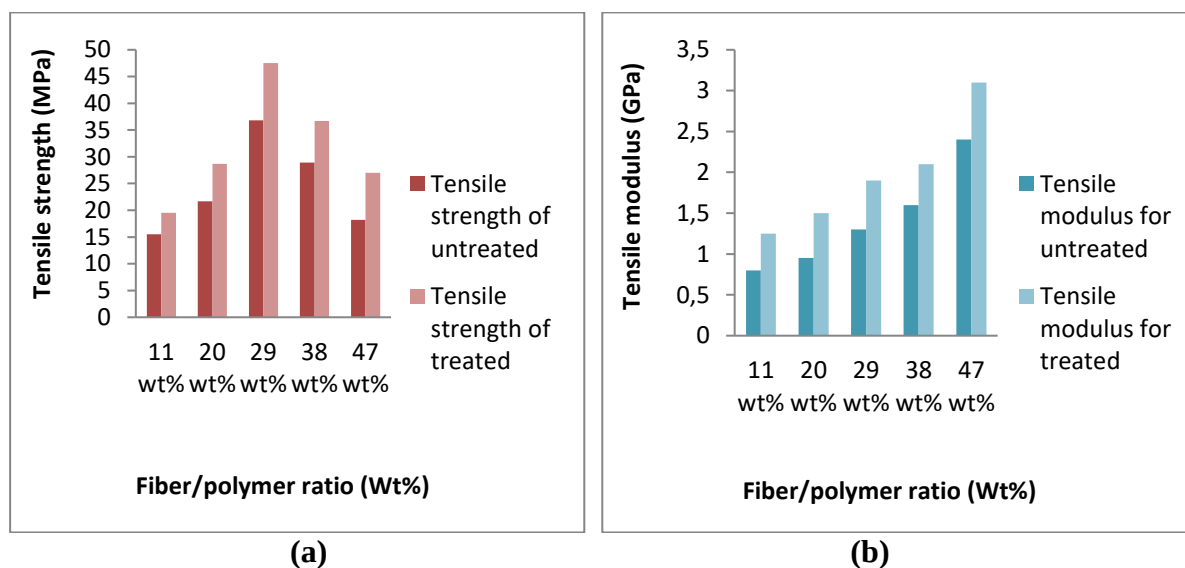


Figure 9. Tensile properties of untreated and treated SPPF/HDPE composites.

Treatment with NaOH makes the fiber surface to be rough leading to improved interfacial bonding between fiber and matrix. Yan et al (2000) stated that alkalization causes fiber bundle breakages (fibrillation) which increases the effective surface area available for wetting by the matrix, better fibre/matrix interface due to the reduction in fiber diameter and increased fiber aspect ratio hence improved mechanical properties. These findings are similar to the report of Goud and Rao, (2011).

Flexural properties of untreated and treated composites at various fiber contents for flexural strength and flexural modulus are presented in Figures 10. The flexural strength of SPPF/HDPE composite increased with increasing fiber content with maximum value of 41.1GPa at 29 Wt% fiber content, then decrease in flexural strength was seen with higher fiber contents. The reduction could be as a result of inadequate filling of HDPE resin into the SPPF during production of the composites and then fiber/fiber interaction instead of fibre/matrix interaction.

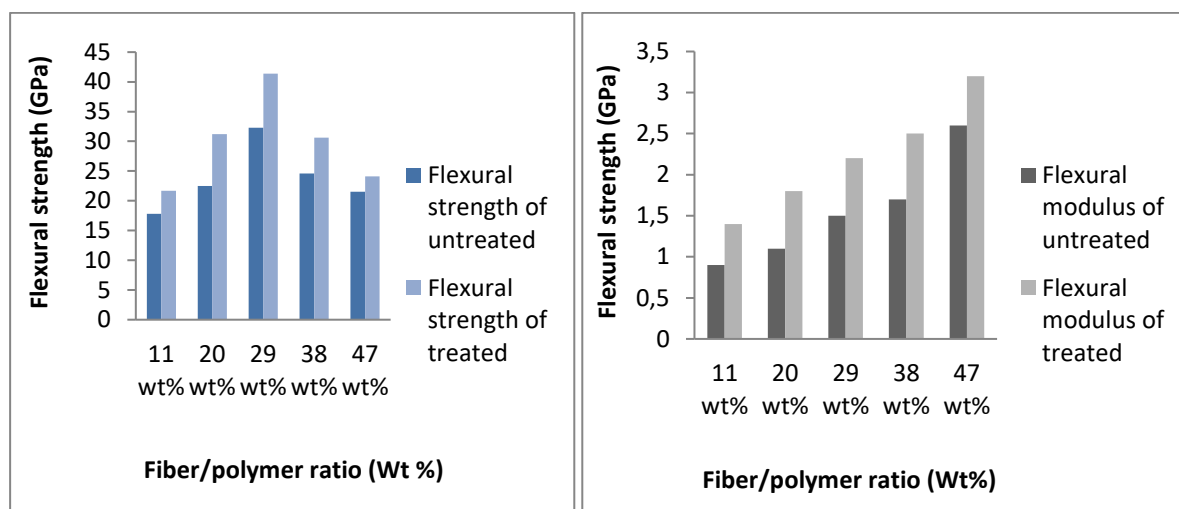


Figure 10. Flexural strength and modulus of untreated and treated SPPF/HDPE composites.

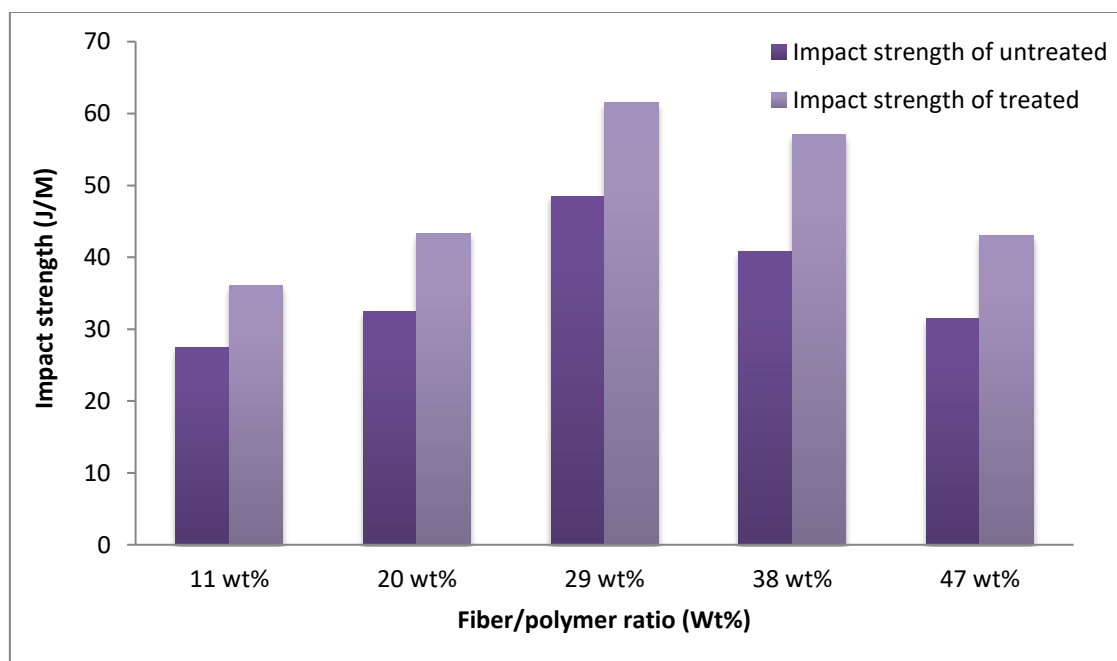


Figure 11. Impact strength of untreated and treated SPPF/HDPE composites.

But flexural modulus increased as the fiber content increased. This behavior may be attributed to the incorporation of rigid SPPF filler which offered a characteristic reduction in the ductility of HDPE matrix thereby resulting to increase in flexural modulus. This confirms that alkalization of fibers improves fibre/matrix interface and enhances fiber properties.

The impact strength of untreated and treated SPPF composite is presented in Figure 11. It is observed that the impact strength increased with increase in fiber ratio for both untreated and treated composites till it got to maximum impact strength 61.5J/M at 29wt% of fiber content, after which there is a slight reduction of the impact strength. This may be as a result of the existence of weak interfacial interaction between the filler and matrix for higher filler content beyond 29 wt%. Bengtsson et al (2007) reported that agro fibers act as stress concentrates in a polymer matrix thereby reducing the crack initiation energy. The surface modification increased the compatibility and its ability to dissipate energy during fracture.

3. 10. RSM results on the Tensile Strength of the Composites

The RSM result of Tensile strength of composites of chemically treated SPPF is presented on Tables 5. The tensile strength varied with interactive factors of temperature, fiber/polymer ratio and time and increased with increase in considered factors till the peak.

Table 3. RSM Results of SPPF composites treated with Sodium hydroxide

Std	Run	A: Fibre/Polymer Ratio wt%	B: Temperature °C	C: Time min.	Tensile strength Mpa
5	1	20	167	15	18.2
10	2	38	172	10	37.4
4	3	38	177	5	26.7
1	4	20	167	5	12.2
13	5	29	172	5	40.1
14	6	29	172	15	44.3
7	7	20	177	15	15.6
19	8	29	172	10	48.3
6	9	38	167	15	19.2
8	10	38	177	15	29.7
11	11	29	167	10	37.8
20	12	29	172	10	48.3
9	13	20	172	10	29.2

2	14	38	167	5	15.4
17	15	29	172	10	48.3
15	16	29	172	10	48.3
3	17	20	177	5	13.2
12	18	29	177	10	43.4
18	19	29	172	10	48.3
16	20	29	172	10	48.3

3. 11. Analysis of variance (ANOVA) and fit Statistics of Tensile strength of the composites

The ANOVA and fit statistics of the tensile strength of composites of SPPF treated-NaOH is presented on Table 4. The Model F-value of 191.26 implies that the model is significant. P-values less than 0.0500 indicate model terms are significant and A, B, C, AB, A², B², C² are significant model terms. Temperature, fiber/polymer ratio and time were responsible for the quadratic nature of the model.

Table 4. ANOVA and Fit statistics of Tensile strength in SPPF treated with NaOH.

Source	Sum of Squares	Df	Mean Square	F-value	p-value	
Model	3636.04	9	404.00	2846.38	< 0.0001	Significant
A-Fibre/Polymer	160.00	1	160.00	1127.27	< 0.0001	
B-Temperature	66.56	1	66.56	468.97	< 0.0001	
C-Time	37.64	1	37.64	265.16	< 0.0001	
AB	68.45	1	68.45	482.22	< 0.0001	
AC	0.3200	1	0.3200	2.25	0.1641	
BC	2.42	1	2.42	17.05	0.0020	
A ²	640.69	1	640.69	4513.93	< 0.0001	
B ²	174.40	1	174.40	1228.75	< 0.0001	
C ²	111.36	1	111.36	784.60	< 0.0001	
Residual	1.42	10	0.1419			
Lack of Fit	1.42	5	0.2839			

Pure Error	0.0000	5	0.0000			
Cor Total	3637.46	19				
Std. Dev.	0.3767	R ²	0.9996			
Mean	33.61	Adjusted R ²	0.9993			
C.V. %	1.12	Predicted R ²	0.9930			
		Adeq Precision	134.8953			

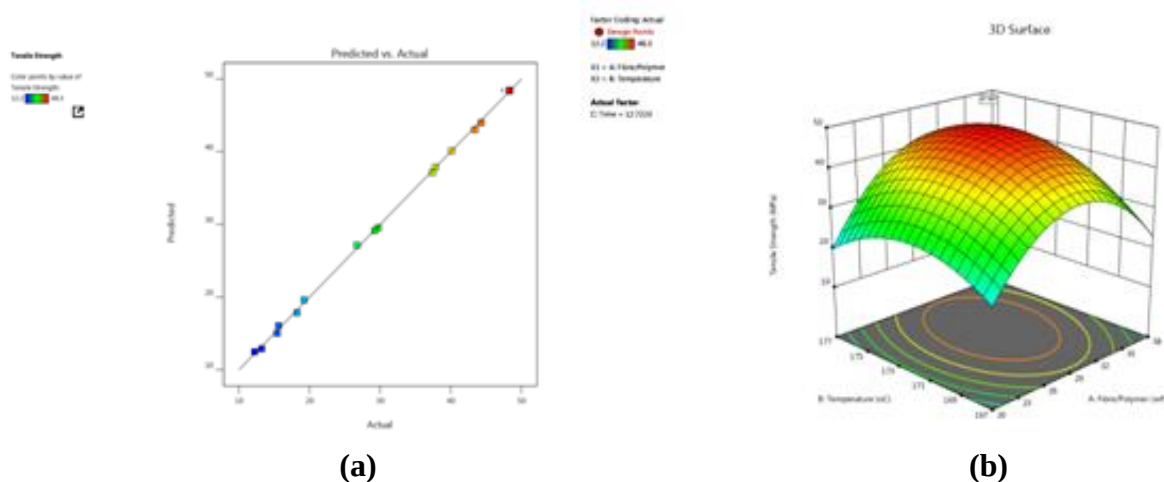
3. 12. Mathematical model for the composite

The mathematical model is presented in Equation 4. It is observed that the tensile strength was dependent on temperature, fiber/polymer ratio and time. It is a quadratic model with the highest power of the variables being two.

$$\text{Tensile strength} = +48.41 + 4.00A + 2.58B + 1.94C + 2.92AB - 0.2000AC - 0.5500BC - 15.26A^2 - 7.96B^2 - 6.36C^2 \quad (1)$$

3. 13. Graphical Analysis for the Composites

The graphical analyses of the tensile strength of composites with treated DPPF are shown in Figure 12. Figure 12a is a plot of the predicted values versus actual values of the tensile strength of the composite. The points clustered along the line of best fit, which indicated that the generated model adequately predicted the actual tensile strength. Figures 12b, c and d are 3D plots of tensile strength as a function of the interactions between temperature and fiber/polymer ratio; time and fiber/polymer ratio and time and fiber/polymer ratio respectively. The tensile strength increased with increase in all the variables until it got to a peak. An optimal tensile strength of 42.03MPa was obtained at time of 10 min, temperature of 172 °C and fiber/polymer ratio of 29wt%.



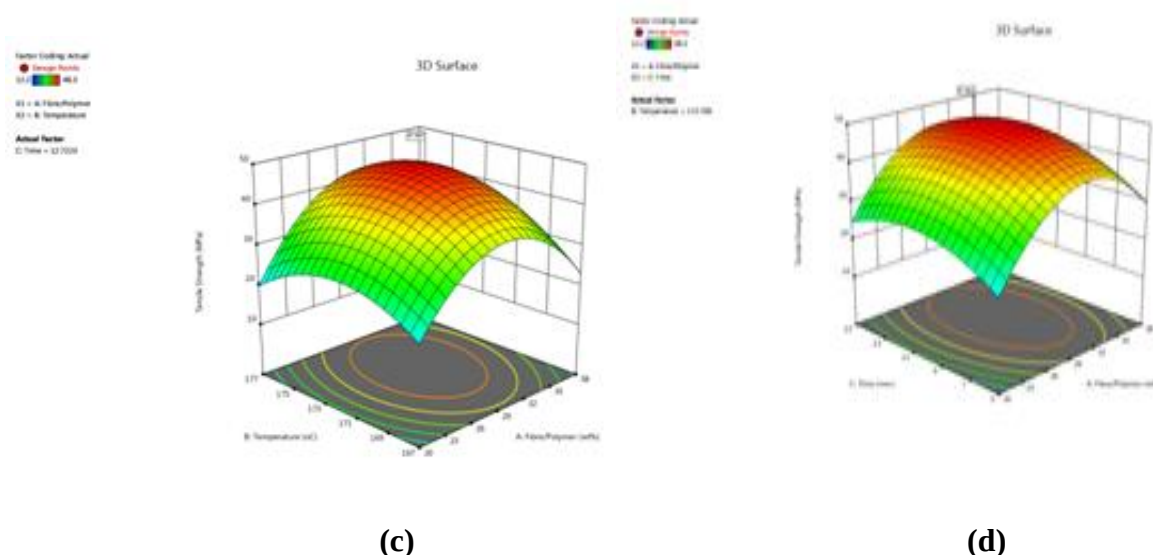


Figure 12. (a) Predicted versus Actual Tensile Strength, **(b)** Tensile strength versus Temperature and Fiber /Polymer ratio, **(c)** Tensile strength versus Time and Fiber/Polymer ratio and **(d)** Tensile strength versus Time and Temperature of SPPF treated NaOH fiber composites.

4. CONCLUSIONS

This paper has produced SPPF reinforced HDPE composite with a cellulose content of 65% and tensile strength of 48.5 MPa. The generated models can also serve as a good starting basis for further experimentation in this area.

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