

# Optimization Of Chemical Treatment Of Morinda Morindoides Fiber

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**Abstract**—The chemical treatment of morinda morindoides was studied to improve the mechanical properties of the fiber. The fiber was separately treated with four different chemicals namely, sodium hydroxide, acetic anhydride, nitric acid and zinc chloride. The chemical treatment was optimized using central composite design (CCD). Quadratic models were developed and validated with minimal errors. The tensile strengths of the morinda morindoides treated with sodium hydroxide, acetic anhydride, nitric acid and zinc chloride at the optimum conditions were found to be 1623.07, 1522.714, 1408.48 and 1277.011 Mpa respectively. Scanning electron microscopy (SEM) and Fourier transform Infra red (FTIR) spectroscopy showed that the chemical treatment was capable of removing the surface contaminants from the fiber. Sodium hydroxide showed better fiber surface modification capacity than the other chemicals. Chemical treatments can therefore be used to modify the surface of natural fibers, making the fibers more hydrophobic and consequently increasing the physio-mechanical properties of the fibers.

**Keywords**—*morinda morindoides, chemical treatment, tensile strength*

## I. INTRODUCTION

In recent time enamours interest in the development of composites with natural fibers has been shown by some industries such as automobile, construction and packaging industries. These composites have been found to exhibit properties comparable with the conventional materials used by these industries. Composites are made by a combination of two or more components to achieve the desired properties which could not be achieved with the components separately. One of the materials acts as the reinforcement while the other is the matrix. The reinforcement is embedded into the matrix to improve the mechanical and physical properties of the composites [1].

Natural fibers have attracted the interests of many researchers, material scientist and industrialists because of the specific advantages these natural fibers have over the conventional or synthetic fibers .

Because of their biodegradable nature, natural fibers have been increasingly adopted to replace synthetic fibers in many industrial applications. [2] .Some of the hazards associated with the use of synthetic fibers are non biodegradability, non-recyclability, high density and some other known health hazards [3]. However, the greatest problem of using these synthetic fibres resides with how to conveniently dispense with them once they attained their useful life span. Several materials fibers as reinforcements for composites have been studied and these include Sisal [4], bagasse [5], kenaf [6]. etc. Compared to inorganic fiber, natural fiber presents some advantages such as lower density and lower cost. They are less abrasive to the processing equipment, harmless, biodegradable, renewable and their mechanical properties can be comparable to those of inorganic fibers.[7].

In spite of the advantages mentioned above, there are some drawbacks in using natural fibers as reinforcements in thermoplastics. The main drawbacks are the difficulties of achieving good dispersion and strong interfacial adhesion between the hydrophilic natural fiber and hydrophobic polymers which leads to composites with rather poor durability and poor mechanical properties [8], [9] . Certain chemical treatments on the natural fibers are needed to enhance the performance of the fibers as reinforcement for the composites production.

In this study, four chemicals namely Sodium Hydroxide, Acetic anhydride, Nitric acid and Zinc chloride were used to treat the Morinda Morindoides (Ogbuebo) natural fiber for the natural fiber reinforced high density polyethylene composites.

## I. MATERIALS AND METHODS

Sodium hydroxide, acetic anhydride, Acetic acid, Nitric acid and Zinc chloride used for the chemical treatments were of analytical grade and were obtained from Cliff Integrated Company, Enugu, Nigeria.

### A. Extraction of Natural Fiber

The natural fiber was extracted by water retting method. The plant was obtained from Olo in Ezeagu

Local Government Area and was sufficiently traumatized and completely submerged in flowing water. It was left for two weeks after which it was brought out and thoroughly washed with clean water. The bundles were further placed inside the water and further left for another one week at room temperature. At the end of the one week the fiber was brought out and washed with excess tap water. It was sundried. The strands with some defects were sorted out and discarded.

**B. Chemical compositions of fiber**

• **Moisture Content Determination**

The fibre was weighed and dried in an oven at the temperature of 103°C for 30 minutes, followed by cooling for 30 minutes and then weighed again. This step was repeated until the weight was constant. The moisture content of the test specimen was expressed as a percentage loss in weight of the final oven-dry weight using the equation;

$$\text{Moisture content} = \frac{W_0 - W_1}{W_0} \times 100$$

(1)

Where  $W_0$  is weight of fibre before drying in the oven and  $W_1$  is weight of fibres after drying in the oven.

• **Ash content**

The ash content determination was according to the work done by Thomsen et al. [10] with modifications. The ash content was determined by burning 5g of the fiber placed in a platinum crucible in a muffle furnace at 600°C for 30 minutes. The percentage of the ash content was determined using the equation (2).

Ash content is given as

$$\text{Ash content} = \frac{W_3 - W_2}{W_1} \quad (2)$$

Where,  $w_2$  is the weight of the porcelain crucible,  $w_3$  is the weight of the porcelain crucible and fiber burnt to ashes and  $w_1$  is the initial weight of the fiber

• **Wax content**

The wax determination was done according to work done by Thomsen et al. [10] with modifications. 4g of the fiber was inserted into a thimble and put in a soxhlet reflux flask and mounted unto an oil extraction flask containing 300 millimetres of petroleum ether. The upper end of the reflux flask was connected to a condenser and was air tight. With the aid of non flame bearing heating mantle, the solvent in the flask was heated and it condensed into the reflux flask containing the thimble. The solvent was kept in contact with the fiber sample until the reflux flask was filled up and siphoned over, thus carrying extracted oil or fat down to the boiling flask. This process was repeated for about four hours with not less than fourteen refluxes. Then, the defatted fiber was carefully removed, dried in an oven at 80°C for 30 minutes, cooled in desiccators and reweighed. The weight loss due to fat extracted was obtained as

the weight of the fat or oil or wax using the equation below.

Fat/wax content (%) is given as

$$\frac{W_4 - W_5}{W_1} \times 100 \quad (3)$$

Where,  $w_4$  is the weight of thimble and fiber before defatting,  $w_5$  is the weight of thimble and fiber after defatting and  $w_1$  is the initial weight of the fiber.

• **Determination of Lignin Content by Gravimetric Method**

This procedure was in accordance with the work done by Onyeagoro [11]. 2.0g of the sample was weighed and placed inside a beaker. 72%  $H_2SO_4$  was added and allowed to stand for 2 hours. 8%  $H_2SO_4$  was later added and the solution refluxed for 3 hours. The residue was filtered with purpling cloth and washed severally with hot water. A crucible with sample on it was weighed. The sample was oven dried at 110°C for 1 hour and then cooled inside desiccators after which the weight was taken. The sample was burnt to ash in a furnace at 500°C for 3 hours. It was then cooled inside desiccators and finally weighed. The % lignin was calculated using equation (4)

$$\% \text{ Lignin} = \frac{W_2 - W_1}{W_s} \times 100 \quad (4)$$

Where,

$W_1$  = weight of ash sample + crucible

$W_2$  = weight of oven dried sample + crucible

$W_s$  = initial weight of dried sample

• **Determination of Hemicelluloses Content**

Hemicelluloses content determination was according to work done by Thomsen et al. [10] with modifications. Hemicellulose was extracted by soaking 10g of the sample in 100mls of 8.5% sodium hydroxide solution for 24 hours at room temperature and was filtered. The hemicellulose was precipitated by acidifying it with acid and ethanol. The precipitate was washed after filtration and dried over night and was weighed after cooling in a dessicator. The hemicelluloses content was calculated using the equation below.

Hemicelluloses content (%) is give as

$$\frac{W_3 - W_2}{W_3} \times 100 \quad (5)$$

Where,  $w_3$  is the initial fiber weight before extraction,  $w_2$  is the final fiber weight after extraction.

• **Determination of Cellulose Content**

The remaining residue after hemicelluloses extraction was washed, dried, cooled in a desiccator and weighed as the cellulose fraction. The cellulose fraction was expressed as the percentage of the dry matter.

**C. Chemical treatment of fiber.**

The fiber was treated with four chemicals which are sodium hydroxide, acetic anhydride, nitric acid and zinc chloride

- Alkali Treatment of the fiber

This procedure was in accordance with work done by Nural and Ishak [12]. The washed fiber was treated with 6% NaOH. The fiber was immersed in the alkali solution for 50 minutes, then neutralized with acetic acid and washed with distilled water repeatedly until all sodium hydroxide was eliminated. Finally, the fiber was washed with distilled water and dried at room temperature for 48h

- Acetic Anhydride Treatment

The acetylation process was in accordance with work done by Bledzki, et al. [13]. The fiber was soaked in distilled water for an hour, filtered and placed in a round bottom flask containing 10% acetic acid solution for 30 minutes. After which it was placed on flask containing 14% acetic anhydride solution. The process temperature of acetylation was 30°C and duration was 70 minutes. After modification, the fiber was washed periodically with distilled water until acid free. Finally, modified fiber was air dried for certain time before analysis.

- Nitric Acid Treatment

The nitric acid treatment was according to Vautard et al., [14]. The size reduced fibers were oxidized with 6% nitric acid. The prepared oxidizing solution was boiled to a temperature of 60°C and the fiber immersed in the solution at the said temperature for 50 minutes. After, it was neutralized with NaOH solution and washed with distilled water repeatedly until all the nitric acid was eliminated. Finally, the fiber was washed again with distilled water and dried to a constant weight at room temperature.

- Zinc Chloride Treatment

Zinc chloride treatment was done in accordance with the work done by Nadanthangam et al., [15]. The fiber was soaked in 3% zinc chloride solution for 70 minutes after which it was washed with distilled water until the washing solution became chloride free. The fiber was washed with distilled water and dried at room temperature for 48 hours.

#### A. Determination of tensile strength

Hounsfield universal testing machine (specifications; trade name: Tensometer limited 81 Morland Rad, Cryodon England, make: Hounsfield universal tensometer was used to determine the tensile strength, and elongation at break of the specimen according to ASTM [16]. After finding the average diameter (D) of the fibres, the cross sectional area of each individual fibre was determined using the relation

$$A = \pi r^2 \quad (6)$$

where r = radius

After this, they were gripped in between two frames to assure a good gripping and straight position in the test clamps. The opening of the frame determines the gauge length; for this experiment it was set at 50 mm. The single fibre tensile tests were performed on a mini tensile testing unit of the Hounsfield Tensometer machine with a load cell of 200N where the crosshead speed was set at 1 mm/min; the load and the displacement were registered during the complete test. A rotating drum on which special graph sheet was wrapped was fixed to the side of the equipment. A manually operated rotating handle was used for loading the specimen under test. With the help of an adjustable mercury level and a manually operated marker, the load and extension values of the specimen were recorded on the graph sheet at a regular interval until failure occurred. The tensile strength was calculated from this relation

$$\text{Tensile Strength (Mpa)} \text{ is given as } \frac{\text{Maximum load (N)}}{\text{Area(mm}^2\text{)}} \quad (7)$$

#### B. Statistical analysis and optimization

Central composite design was used to optimize the chemical treatment conditions. This involved three numeric factors and one categoric factor. The addition of categoric factor multiplied the total number of the experiments by the level of the factor; in this case we have 13x4 = 52 experiments. CCD is characterized by three operations namely; 2n axial runs, 2<sup>n</sup> factorial runs and six center runs. The total number of experiments is 2<sup>n</sup> + 2n + nc where n is the number of factors, nc is the number of center points [17]. The factors and levels used for the CCD are shown on Table 1 while the design matrix is shown on Table 3.

Table 1. Factors and levels for chemical treatments of the fibers using CCD

Factors	Units	Levels				
		-α	-1	0	+1	+α
Chemical strength	%	2	6	10	14	18
Treatment time	Mins.	30	50	70	90	110
Type of chemicals		-Sodium hydroxide -Acetic anhydride -Nitric acid -Zinc chloride				

II. RESULTS AND DISCUSSIONS

A. Chemical composition of natural fiber

Natural fibre consists of cellulose, hemicelluloses, lignin, wax, water content and water soluble substances. These compositions differed with the different species of the plant.

The chemical structure of cellulose is the same for all natural fibres, but the variations in the cellulose content was as a result of the degree of polymerization or length of the polymer chains of the cellulose. The degree of polymerization of cellulose has a significant effect on the mechanical properties of a fibre as it is directly related to the molecular weight [18]. When the cellulose has a higher degree of polymerization and molecular weight, the fibre will have greater strength properties [18].

From Table 2, it can be seen that *Morinda morindoides* had high cellulose content. This high cellulose expresses its degree of polymerization, thus explaining the fiber's effectiveness when used in composite production.

Table 2. Chemical Compositions of the fiber

A. Selection of a good predictive model for chemical treatments of the fiber

The sequential model sum of square was used to compare different models it shows the statistical significance of adding new model terms step by step in increasing order. It provided accounts of variation and associated P-values (Prob>F) so that one can see how far it is worth going in degree of polynomial. The objective was to add a higher level source of term only if it explains a significant amount of variation beyond what was already accounted for. The model was selected based on the highest order model that was significant (P-value small) and not aliased, lack of fit (P-value > 0.10) and reasonable agreement between Adjusted R-squared and predicted R-squared (within 0.2 of each other). For this fibre, the predicted R-squared was in close range with the adjusted R-squared for all the sources of the model, but the model was suggested based on the model p-value and lack of fit p-value. The suggested model for this fiber was quadratic model.

S/N	Sample fibre	Local name	Cellulose (%)	Hemi cellulose (%)	Lignin (%)	Ash content (%)	Wa x (%)	Moisture content (%)
	<i>Morinda morindoides</i>	Ogbuebo	55.76	8.621	31.22	2.457	0.5	0.48

Table 3. Design matrix with the experimental values for all the chemical treatment process

Std Order	Run Order	Chemical concentrations (%)	Time (Minutes)	Chemical types	Tensile Strength (Mpa)
11	1	10.00	70.00	NaOH	1740.88
27	2	6.00	50.00	Acetic Anhydride	1402.4
8	3	10.00	110.00	NaOH	1200
38	4	10.00	70.00	Acetic Anhydride	1200
7	5	10.00	30.00	NaOH	1720.8
32	6	18.00	70.00	Acetic Anhydride	970.48
5	7	2.00	70.00	NaOH	1113.56
35	8	10.00	70.00	Acetic Anhydride	1001.52
46	9	10.00	30.00	Zncl	1195.88
4	10	14.00	90.00	NaOH	1800
40	11	6.00	50.00	Zncl	1128

9	12	10.00	70.00	NaOH	1593.28
3	13	6.00	90.00	NaOH	1596.8
37	14	10.00	70.00	Acetic Anhydride	1120.44
28	15	14.00	50.00	Acetic Anhydride	920.8
48	16	10.00	70.00	Zncl	1048.44
36	17	10.00	70.00	Acetic Anhydride	1072.8
47	18	10.00	110.00	Zncl	488
52	19	10.00	70.00	Zncl	912
16	20	6.00	90.00	Nitric Acid	1193.2
33	21	10.00	30.00	Acetic Anhydride	1074.08
41	22	14.00	50.00	Zncl	879.92
34	23	10.00	110.00	Acetic Anhydride	801.56
17	24	14.00	90.00	Nitric Acid	958.48
6	25	18.00	70.00	NaOH	1780
49	26	10.00	70.00	Zncl	1250.12
29	27	6.00	90.00	Acetic Anhydride	1400.84
45	28	18.00	70.00	Zncl	803.16
19	29	18.00	70.00	Nitric Acid	920
30	30	14.00	90.00	Acetic Anhydride	960.44
13	31	10.00	70.00	NaOH	1040
24	32	10.00	70.00	Nitric Acid	1082
1	33	6.00	50.00	NaOH	1400.88
12	34	10.00	70.00	NaOH	1760
39	35	10.00	70.00	Acetic Anhydride	1170.4
10	36	10.00	70.00	NaOH	1600
15	37	14.00	50.00	Nitric Acid	967.12
43	38	14.00	90.00	Zncl	674.88
2	39	14.00	50.00	NaOH	1797.28
20	40	10.00	30.00	Nitric Acid	1200
14	41	6.00	50.00	Nitric Acid	1180.44
31	42	2.00	70.00	Acetic Anhydride	1600
23	43	10.00	70.00	Nitric Acid	1128
25	44	10.00	70.00	Nitric Acid	840.44
44	45	2.00	70.00	Zncl	960
42	46	6.00	90.00	Zncl	1420
18	47	2.00	70.00	Nitric Acid	1161.28
50	48	10.00	70.00	Zncl	1200
22	49	10.00	70.00	Nitric Acid	1161.28
26	50	10.00	70.00	Nitric Acid	1082.24



51	51	10.00	70.00	Zncl	1240.96
21	52	10.00	110.00	Nitric Acid	962.8

A. Analysis of variance for the chemical treatment process

ANOVA was used to interpret the relative contribution of each factor to the total variations. R-squared, predicted R-squared and adjusted R-squared values were equally used to ascertain if the model selected will produce good prediction for average outcome. Table 4 shows the analysis of variance for the chemical treatment process.

Attention was focused on predicted R-square (0.6911) and adjusted R-square (0.8494) because the regular R-square (0.9178) can be artificially inflated by simply continuing to add terms to the model, even when the terms were not statistically significant. The adjusted R-squared plateaus when insignificant terms are added to the model, and the predicted R-square will decrease when there are too many insignificant terms.[19].

F- value of 17.3 implied that the selected model was significant. There was only 0.01% chance that this large F-value can occur due to noise. Values of prob>F less than 0.0500 indicated significant model terms but values greater than 0.100 indicated insignificant model terms. ANOVA showed that the linear effect of chemical concentration, time, chemical types, quadratic effect of chemical concentration and the interaction effects of chemical concentration and chemical types were significant with their prob>F value less than 0.05. Lack of fit F-value of 1.09 implied insignificant lack of fit. There was 55.48% chance that a lack of fit F-value this large could occur due to noise. The predicted R-squared of 0.6911 was in good agreement with the adjusted R-square of 0.8494. Adequate precision of 14.938 was above four, indicating an adequate signal.

Table 4. shows the analysis of variance for the chemical treatment process.

Source	Sum of Square	df	Mean Square	F Value	P-Value Prob>F
Model	4.610E+005	9	5.122E+005	17.3	<0.0001
A-Chemical Concentration	1.288E+005	1	1.288E+005	4.35	0.0040
B-Time	2.066E+005	1	2.066 E+005	6.98	0.0045
C- Chemical Type	2.327E+005	3	7.757 E+005	26.19	<0.0001
AC	2.94E+005	3	2.64 E+005	8.91	0.0012
A <sup>2</sup>	1.154E+005	1	1.154E+005	3.90	0.0413
Residual	1.244E+006	42	29619.048		
Lack of fit	8.387E+005	26	32257.69	1.09	0.5548
Pure error	4.053E+005	16	25331.25		

Final equation in terms of coded factors:

$$\text{Tensile strength (Mpa)} = +1189.96 - 51.79A^2 - 65.60B + 359.54 C[1] - 12555 C[2] - 59.52 C[3] + 212.83AC[1] - 25.76 AC[2] - 129.96 AC [3] \quad (8)$$

Final equation in terms of actual factor

NaOH:

$$\text{Tensile strength (Mpa)} = +1376.51304 + 40.26000 (CC)^2 - 3.28021 \text{ Time} \quad (9)$$

Nitric acid :

$$\text{Tensile strength (Mpa)} = +1487.89574 - 19.38750 (CC)^2 - 3.28021 \text{ Time} \quad (10)$$

Acetic Anhydride:

$$\text{Tensile strength (Mpa)} = +1814.44099 - 45, 43833 (CC)^2 - 3.28021 \text{ Time} \quad (11)$$

Zinc chloride:

$$\text{Tensile strength (Mpa)} = +1577.37048 - 27.2267(\text{CC})^2 - 3.28021 \text{ Time} \quad (12)$$

where CC represents chemical concentration.

The equation (8) can be split into four equations each representing the categoric level;

$$\text{Tensile strength (Mpa)} = +1189.96 - 51.79A^2 - 65.60B + 359.54 C[1] + 212.83 AC[1] \quad (13)$$

$$\text{Tensile strength (Mpa)} = +1189.96 - 51.79A^2 - 65.60B - 1215.55C [2] - 25.76AC [2] \quad (14)$$

$$\text{Tensile strength (Mpa)} = +1189.96 - 51.79A^2 - 65.60B - 59.52 C[3] - 129.96 AC[3] \quad (15)$$

$$\text{Tensile strength (Mpa)} = +1189.96 - 51.79A^2 - 65.60B \quad (16)$$

A. Effect of chemical type on the tensile strength of the fiber

Chemical type being a categoric factor was used to compare the magnitude of the effects of different chemicals on the tensile strength of the fiber. The graph of the effect of chemical types on the tensile

strength of the fiber is shown in Fig. 1. From the graph, it can be seen that NaOH had highest effect on the tensile strength of the fiber, followed by acetic anhydride, nitric acid and lastly zinc chloride. This was because alkaline treatment increased the surface roughness and the amount of cellulose exposed on the fiber [20]. This resulted from the removal of cementing substances like Lignin and hemicellulose during the treatment.

The treatment of the fiber with zinc chloride also increased the tensile strength of the fiber. This was because of the fact that when fiber is treated with zinc chloride, the cellulose became partially gelatinous and binds the fiber together within and between the fibers, thereby increasing the tensile strength of the fiber [15]. Treatment of the fiber with acetic anhydride is called acetylation and it replaces the polymer hydroxyl groups with acetyl group thereby making the fiber more hydrophobic [21]. This enhances interfacial bonding and ultimately increases the tensile strength of the fiber.

The treatment of fiber with nitric acid is called oxidation and it introduces oxidizing groups to the surface of the natural cellulose fiber. This creates a rough surface and increases the specific surface area and thereby increasing the tensile strength of the fiber.

Design-Expert® Software  
 Factor Coding: Actual  
 Tensile Strength (Mpa)  
 • Design Points  
 X1 = C: Chemical types

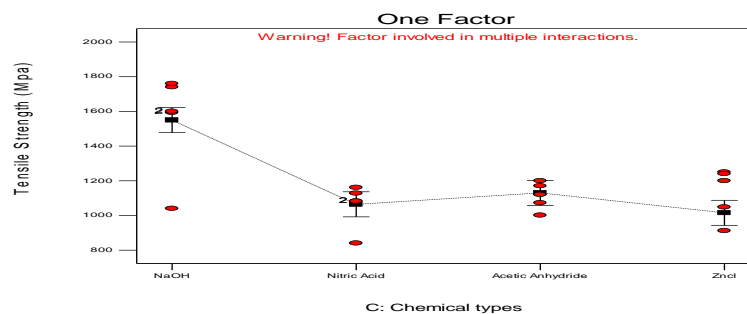


Figure1. Effect of Chemical types on the tensile strength of the treated fiber

A. Optimal conditions for the chemical treatment process

Numerical optimization was used to search the design space using the model created during analysis to find factor settings that met the defined goal. Maximization of tensile strength was set as goal to be met for the optimization. The factor setting used for the optimization was selected based on the highest desirability.

Optimum conditions based on the categoric factor for *Morinda morindoidies* fiber

(a) 14% NaOH for 50minute with predicted tensile strength of 1631.22 Mpa

(b) 6% Acetic anhydride for 70minutes with predicted tensile of 1542.2 Mpa

(c) 10% Nitric acid for 60minutes with predicted tensile strength of 1411.30 Mpa

(d) 6% zinc chloride for 30minutes with predicted tensile strength of 1289.91 Mpa.

The experiment was repeated at the optimum conditions and the experimental values obtained were compared with the predicted values. As can be seen from Table 5, the deviations obtained were minimal as recorded by the low errors.

Table 5. Validation of optimum conditions for *Morinda morindodies* fiber

Model desirability	Chemical Strength (%)	Chemical Type	Time (minutes)	Tensile Strength (Mpa)		Error (%)
				Predicted values	Experimental values	
0.982	14.0	NaOH	50.0	1631.22	1623.074	0.5
0.912	6.0	Acetic anhydride	70.0	1542.77	1522.714	1.3
0.820	10.0	Nitric acid	60.0	1411.30	1408.48	0.2
0.781	6.0	Zinc chloride	30.0	1289.91	1277.011	1.0

A. Scanning Electron Microscopy (SEM) Analysis of Untreated and Treated Fibers

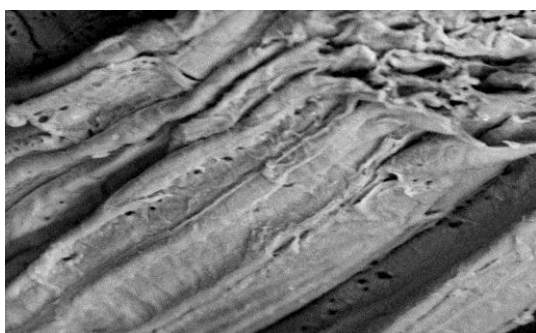
A. Fourier Transformed Infrared Spectroscopy (FTIR)

The effect of chemical treatments on the surface of the fiber is shown as SEM photomicrographs in Figure 2 for the untreated fiber and fiber treated with sodium hydroxide. The SEM micrograph of the surfaces of untreated fiber shows the presence of wax, oil, and surface impurities. Waxes and oils provide protective layers to the surface of the fibers [22]. The chemical treatments on the fiber were successful in removing the surface contaminants as evidenced in the Figure 2b. The micrographs show very clean surfaces which confirmed the removal of the wax, oil and other surface contaminants from the fiber surface.

FTIR analysis was used to characterize the natural fiber treated with various chemicals. The FTIR spectra of the untreated and treated fiber are shown on Figure 3. All the spectra revealed broad and intense peak at  $3400\text{cm}^{-1}$ . This band suggested hydrogen bonded O–H stretching vibration from the cellulose and lignin structure of the fiber [23], [24]. The peak around  $1636\text{cm}^{-1}$  present on the fiber represents the C = O of the hemicellulose [25]. The bands at  $1457\text{cm}^{-1}$ ,  $1374\text{cm}^{-1}$ ,  $1418\text{cm}^{-1}$ ,  $1269\text{cm}^{-1}$  and  $1031\text{cm}^{-1}$  were characteristics of C – H, C – O deformation, bonding or stretching vibrations of many groups in lignin and carbohydrates [26],[27], [28]. The peaks around  $668\text{cm}^{-1}$ ,  $517\text{cm}^{-1}$  and  $407\text{cm}^{-1}$  can be said to be originated from the  $\beta$  - glucosidic linkages between the sugar units in the hemicelluloses and celluloses [29]. After the treatment with sodium hydroxide (Figure 3b), the bands at  $1732\text{cm}^{-1}$  and  $1245\text{cm}^{-1}$  attributed to the stretching vibrations of C = O and C – O groups respectively disappeared. These kinds of groups are present in lignin and hemicelluloses structures. This disappearance can be attributed to the removal of lignin and hemicelluloses components of the fiber. The band at  $1318\text{cm}^{-1}$  is related to the vibration  $\nu(\text{C} - \text{O})$  of esters, ethers and phenols groups attributed mainly to the presence of waxes in the epithermal tissue [30] and the disappearance of this band on the treated fiber results from the removal of those waxes.

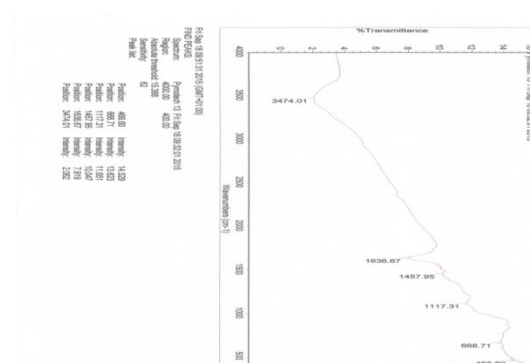


(a)



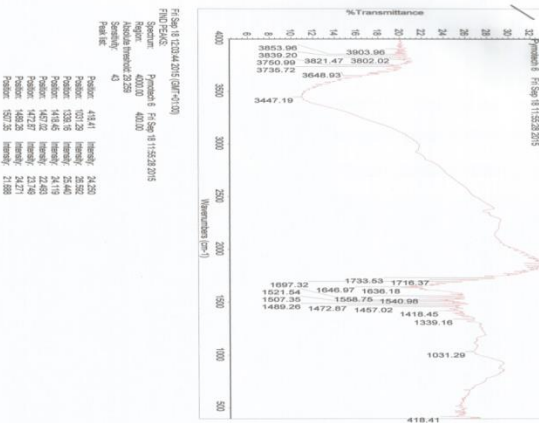
(b)

Fig. 2. SEM micrograph (a) untreated (b) treated fiber



(a)





(b)  
 Fig. 3. FTIR spectra (a) untreated (b) fiber treated with NaOH

### III. CONCLUSION

The work has succeeded in optimization of chemical treatment process using central composite design. Two numeric factors which are chemical concentration and pre treatment time and one categorical factor which is chemical types with four levels of sodium hydroxide, acetic anhydride, nitric acid and zinc chloride were optimized. Quadratic model was developed and diagnosed for the chemical treatment process. The optimum conditions developed were verified with little error of less than 2%. It was found out that chemical treatment was successful in removing the waxy encrusting substances from the surface of the fiber. Alkaline treatment was more effective in removing the surface impurities of this fiber.

### ACKNOWLEDGEMENT.

The authors wish to acknowledge the invaluable disposition of the staff and management of Pymotech Research Centre and Laboratories for the use of their facilities during the laboratory experiment.

### References.

- Irene S., Fashion, Salah Ethaggar, Hatem Elay at (2010). Experimental investigation of Natural fiber reinforced polymer. *Material Sciences and Applications*, 3, pp 59-62.
- Nurul Munirah Abdullah, Ishak Ahmed (2012). Effect of chemical treatment on Mechanical and Water sorption properties of coconut fiber unsaturated polyester from Recycled PET. *International scholarly Research Network*. Vol 272, article ID 134683, 8 pages.
- Herrera-Franco, P. J., Valadez-Gonzalez, A. (2005). Fiber-Matrix Adhesion in Natural Fiber Composites. *Natural Fibers, Biopolymers, and*

*Biocomposites*, A. K. Mohanty, M. Misra, and L. T. Drzal, eds., CRC Press, Boca Raton.

4. Chiou, B. S., Glenn, G. M., Imam, S. H., Inglesby, M. K., Wood, D. F., and Orts, W. J. (2005). *Starch Polymers: Chemistry, Engineering, and Novel Products. Natural Fibers, Biopolymers, and Biocomposites*, A. K. Mohanty, M. Misra, L. T. Drzal, eds., CRC Press, Boca Raton.

5. Cao, Y., Shibata, S., Fukumoto, I. (2006). Mechanical properties of biodegradable composites reinforced with bagasse fibre before and after alkali treatments. *Composites Part A: Applied Science and Manufacturing*, 37(3), 423.

6. El-shekeil, Y. A., Sapuan, S. M., Azaman, M. D., Janaid, M. (2013). Optimization of blending parameters and fiber size of kenaf-Base – fiber reinforced the thermoplastics polyurethane composites by Taguci method. *Advances in materials science and Engineering Vol. 2013*, article, ID 686452, 5 pages.

7. Bledzki, A. K., Reihmane, S., Gassan, J. (1996). Properties and modification methods for vegetable fibers for natural fiber composites. *Journal of Applied Polymer Science*, 59(8), 1329-1336.

8. Kokta, B.V., Maldas, D., Daneault, C., Beland, P. (1990). Composite of poly (vinyl chloride) and wood fibers. Effect of chemical treatment *Polymer Composites Vol 11, no 2*, pp. 84-89.

9. Liu, W., Mohanty, A. K., Drzal, L. T., Askel, P., Misra, M. (2004). Effects of alkali treatment on the structure, morphology and thermal properties of native grass fibers as reinforcements for polymer matrix composites. *Journal of Materials Science*, 39(3), 1051-1054.

10. Thomsen, A.B., Rasmussen, S.K., Bohn, V., Nielsen K.V., Thygesen, A (2005) Hemp raw materials. The effect of cultivar, growth conditions and pretreatment on the chemical composition of the fibers. (Denmark. Forskningscenter Risoe . Risoe –R. ,No 1507(EN))

11. Onyeagoro, G.N., Enyiegbulam, M.E., (2012). Physico-mechanical Properties of Cellulose acetate butyrate / Yellow Poplar Wood Fibre Composites as a Function of Fibre Aspect Ratio, Fiber Loading and Fibre Acetylation. *International Journal of Basic and Applied Science Vol. 1, No. 2*, pp. 1 – 13.

12. Nurul Munirah Abdullah, Ishak Ahmed .(2012). Effect of chemical treatment on Mechanical and Water sorption properties of coconut fiber unsaturated polyester from Recycled PET. International scholarly Research Network. Vol 272, article ID 134683,
13. Bledzki, A.K., Mamun, M., Luka – Gabor. Gutowski, V.S, (2008). The effect of acetylating on the properties of flax fiber and its polypropylene composites. Express polymer letters vol.2 No 6 ,pp413-422.
14. Vantard, F., Fioux, P., Vidal, L. J., Dentze, J., Schultz, M., Nerdein, B., Defort (2013). Influence of an oxidation of carbon fibre surface by boiling nitric and on the adhesion strength in carbon fibre-acrylate composite cured by electron beam. Surface and interface analysis Vol. 45, issue 3, pp 722-741.
15. Nadantangam, U., Karande, K. G. B., Hadge S.T., Mhaske, A.K., Bhari Malla (2012). Abstract No oral – 89. Enzyme/ZnCl pretreatment of short staple cotton fibers for energy reduction during non fibrillation by refining process research conference on Technologies for property.
16. ASTM D1037-12.(2012) Standard Test Methods for Evaluating Properties of Wood-Base Fiber and Particle Panel Materials. ASTM International, West Conshohocken, PA , www.astm.org
17. Ejikeme, M.E., Ejikeme, P.C.N., Abalu, B.N. (2013). RSM Optimization process for uptake of water from Ethanol- water solution using oxidized starch. Pacific Journal of Science and Technology. 14(2): 319 – 329.
18. Mohanty, A. K., Misra, N., Hinrichsen, G. (2000). Biofiber, biodegradable polymers and biocomposites. An overview. Macromolecular materials and Engineering 276-277(1), 1 – 24.
19. Mark J. Anderson, Patrick J. Whitcomb (2005). RSM Simplified optimizing processes using response surface methods for design of experiments CRC press Taylor and Francis group Boca Raton London New York.
20. Bei Wang (2004) Pretreatment of flax fibers for use in rotationally molded biocomposites. A thesis submitted to the college of graduate studies and research department of agricultural and Bioresource Engineering university of Saskatchewan Saskatoon, Saskatchewan.
21. Liu, W., Drzal, L. T., Mohanty, A. K., Misra, M. (2007). Influence of processing methods and fiber length on physical properties of kenaf fiber reinforced soy based biocomposites. Composites Part B: Engineering, 38(3), 352
22. Kumar, S., Siddaramanah, H. (2005). Studies on corn starch filled poly (Styrene – Co-Butylacrylate) latex reinforced polyester Nonwoven on composites 5(2005) (9): 227 – 331.
23. Oh, S.Y., Yoo, D.I., Shin, Y., Kim, H.C., Kim, H.Y., Chung, Y.S., Park, W.H., Youk, J.H. (2005). Crystalline structure analysis of cellulose treated with sodium hydroxide and carbon dioxide by means of X-ray diffraction and FTIR spectroscopy. Carbohydr. Res., 340, 2376–2391.
24. Kondo, T. (1997). The assignment of IR absorption bands due to free hydroxyl groups in cellulose. Cellulose, 4, 281–292.
25. Yan, T., Xu, Y., Yu, C. (2009). The Isolation and Characterization of Lignin of Kenaf Fibers, Journal of Applied Polymer Science, Vol. 114, No. 3, 2009, pp. 1896-1901.
26. Yokoi, H., Nakase, T., Goto, K.; Ishida, Y., Ohtani, H., Tsuge, S., Sonoda, T., Ona, T. (2003). Rapid characterization of wood extractives in wood by thermal desorption-gas chromatography in the presence of tetramethylammonium acetate. J. Anal. Appl. Pyrolysis, 67, 191–200.
27. Ishida, Y., Goto, K., Yokoi, H., Tsuge, S., Ohtani, H., Sonoda, T., Ona, T. (2007). Direct analysis of phenolic extractives in wood by thermochemolysis-gas chromatography in the presence of tetrabutylammonium hydroxide. J. Anal. Appl. Pyrolysis , 78, 200–206.
28. Mészáros, E., Jakab, E., Várhegyi, G. (2007). TG/MS, Py-GC/MS and THM-GC/MS study of the composition and thermal behavior of extractive components of Robinia pseudoacacia. J. Anal. Appl. Pyrolysis, 79, 61–70.
29. Bledzki, A.A., Mamun, A.A., Volk, J., (2010). Barley husk and coconut shell reinforced polypropylene composites: The effect of fiber physical, chemical and surface properties”, Composite Science and Technology, 70, pp. 840-846
30. Herrera-Franco, P. J., Valadarez-González, A. (2005). A study of the mechanical properties of short natural-fiber reinforced composites. Composites: Part B, 36, 597–608.