

# Optimum Conditions for Mercerization of Oil Palm Empty Fruit Bunch Fibre

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## Abstract -

Oil Palm Empty Fruit Bunch Fiber was treated with a solution of 2wt% - 10wt% NaOH for 30mins - 150mins and the Tensile Strength, Toughness, Modulus of Elasticity, Yield Strength and Ductility were studied using response surface methodology. The results reveal that mercerization of oil palm empty fruit bunch fiber increases the Modulus of Elasticity up to eighteen times, the yield strength about eight times, Tensile strength about six times and Toughness about six times. The optimum concentration and time for most of the mechanical properties was a NaOH concentration of 6wt% and treatment time of 90mins. Treatment time has the most significant effect on fiber mechanical properties among the two properties studied. The optimum NaOH concentration and time from this study is recommended for mercerization of Oil Palm Empty Fruit Bunch Fiber prior to use in composite manufacture.

**Index Terms**— Mercerization, Oil Palm Empty Fruit Bunch Fiber, Response Surface, Tensile Strength, Modulus.

## I. INTRODUCTION

Alkali Treatment of cellulosic fibers, also called mercerization, is the usual method to produce high quality fibers [1]. The standard definition of mercerization as proposed by ASTM D1965 is: the process of subjecting a vegetable fiber to an interaction with a fairly concentrated aqueous solution of strong base, to produce great swelling with resultant changes in the fine structure, dimension, morphology and mechanical properties [2]. Alkali treatment improves the fiber-matrix adhesion due to the removal of natural and artificial impurities [3]. It leads to fibrillation, which causes the breaking down of the composite fiber bundle into smaller fibers. In other words, alkali treatment reduces fiber diameter, thereby increasing the aspect ratio. The development of a rough surface topography and enhancement in aspect ratio offer better fiber-matrix interface adhesion and an increase in mechanical properties [4]. Alkali treatment increases surface roughness resulting in better mechanical interlocking and the amount of cellulose exposed on the fiber surface. This increases the number of possible reaction sites and allows better fiber wetting.

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The following reaction takes place as a result of alkali treatment:



Mercerization affects the chemical composition of natural fibers, degree of polymerization and molecular orientation of the cellulose crystallites due to cementing substances like lignin and hemicellulose which are removed during the mercerization process. Consequently, mercerization has a lasting effect on the mechanical behavior of natural fibers, especially on fiber strength and stiffness. The tensile strength and modulus of jute fiber were improved by 120% and 150% respectively after treatment with 25wt% NaOH for 20 minutes at 20°C [5]. It also leads to the increase in the amount of amorphous cellulose at the expense of crystalline cellulose and the removal of hydrogen bonding in the network structure [6, 4, 7, 8, 9, 10, 11].

Most parameters used in mercerization treatment were alkali concentration, fiber soaking temperature and fiber soaking duration. Though several research results have been published regarding natural fiber mercerization treatment, which is the primary fiber treatment technique, there are still scanty works conducted in dealing with interaction of factors and optimizing the mercerization treatment conditions towards enhancement of natural fiber reinforced composite mechanical properties [2]. This work presents optimum conditions for mercerization of oil palm empty fruit bunch fiber at ambient temperature.

## II. METHODOLOGY

The Equipment used for the experiment as follows: Monsanto tensometer machine; Pneumatic grips; General Laboratory glassware and consumables.

The Chemicals and reagents used for the experiment are as follows: Sodium Hydroxide; Water; Acetic acid.

### A. Sample Preparation and Treatment

The fibers used in this work were prepared at Center for Composite Research & Development (CCRD), JuNeng Nigeria Limited, Nsukka, Nigeria. Extraction of the Oil Palm Empty Fruit bunch fiber was done through water retting. The fibers were washed and conditioned at ambient conditions until constant mass. The dried fibers were chopped into 100mm lengths and used for the determination of tensile property characterization and chemical treatment of fibers.

### B. Alkali Treatment (Mercerization)

The chopped fibers were each soaked in a transparent plastic vessel containing Sodium Hydroxide at different concentrations (2wt % NaOH, 4wt % NaOH, 6wt % NaOH,

8wt % NaOH and 10wt % NaOH) and each for different soaking times (30mins, 60mins, 90mins, 120mins and 150mins). The fibers were then washed thoroughly with water to remove the excess of NaOH on the fibers. Final washing was done with water containing little acetic acid. Fibers were dried in an air oven at 70 °C for 3hours.

**C. Tensile Test for Fibers (ASTM D3822)**

Single fibers were carefully separated from the bundles manually and both fiber ends were glued on the pieces of paper for handling purposes. A masking tape was used. The tests were carried out on a Monsanto tensometer machine at the Civil engineering laboratory, University of Nigeria, Nsukka based on ASTM standards. All tests were displacement controlled with the loading rate of 0.5 mm/min.

**III. ANALYSIS OF RESULTS AND DISCUSSION**

The Tensile Strength was obtained as the highest point on the Stress-Strain curve. The Modulus of Elasticity was obtained by determining the slope of a straight line drawn as tangent to the linear-elastic region of the Stress-Strain curve. The Yield Strength was obtained as the Stress at which a line, drawn at 0.2% offset of the strain and with the Modulus of Elasticity as its slope, intersects with the Stress-Strain curve. The Toughness was obtained as the area under the Stress-Strain curve. MATLAB version 7.9 was used for all the analysis.

It can be observed from Table 1 and Table 2, that oil palm empty fruit bunch fiber treated with 2wt % NaOH had its toughness increased by 14% relative to the untreated sample, the tensile strength of the treated sample was more than 2.3 times the untreated one, yield strength was 2.7 times the untreated, modulus of elasticity increased more than 3.5 times, while there was no significant improvement in ductility (percentage elongation). Eighty percent of the mechanical properties had their maximum values after the fiber had been treated at this concentration for 120 minutes. It can be observed from Table 1 and Table 3, that oil palm empty fruit bunch fiber treated with 4wt % NaOH had its toughness increased more than 5 times relative to the untreated sample, the tensile strength of the treated sample was more than 3.5 times the untreated one, yield strength was increased nearly 5 times, modulus of elasticity increased more than 6.5 times, while there was no significant improvement in ductility (percentage elongation). Treatment at this concentration resulted in increase in all mechanical properties in comparison with that of 2 wt % NaOH. Forty percent of the mechanical properties had their maximum values after the fiber had been treated at this concentration for 60 minutes and another forty percent after treatment for 90 minutes.

It can be observed from Table 1 and Table 4, that oil palm empty fruit bunch fiber treated with 6wt % NaOH had its toughness increased by about 19% relative to the untreated sample, the tensile strength of the treated sample was more than six times the untreated one, yield strength was increased approximately seven times, modulus of elasticity increased more than eleven times, while there was no significant improvement in ductility (percentage elongation). Treatment at this concentration resulted in increase in all mechanical properties in comparison with that of 4 wt %, except for toughness and ductility. Forty

percent of the mechanical properties had their maximum values after the fiber had been treated at this concentration for 60 minutes and another forty percent after treatment for 90 minutes.

It can be observed from Table 1 and Table 5 that oil palm empty fruit bunch fiber treated with 8wt % NaOH had its toughness increased by more than 1.2 times relative to the untreated sample, the tensile strength of the treated sample was almost six times the untreated one, yield strength was increased more than 3.5 times, modulus of elasticity increased nearly 17 times, while there was no significant improvement in ductility (percentage elongation). Treatment at this concentration resulted in a slight drop in tensile strength and yield strength, while other mechanical properties increased slightly in comparison with that treated with 6 wt % NaOH. All mechanical properties had their maximum values after the fiber had been treated at this concentration for 90 minutes.

It can be observed from Table 1 and Table 6 that oil palm empty fruit bunch fiber treated with 10wt % NaOH had its toughness increased by 50% relative to the untreated sample, the tensile strength of the treated sample was more than five times the untreated one, yield strength was increased more than two times, modulus of elasticity increased 4.5 times, while there was no significant improvement in ductility (percentage elongation). Treatment at this concentration resulted in a slight drop in all mechanical properties in comparison with that of 8 wt %, except for ductility. Sixty percent of the mechanical properties had their maximum values after the fiber had been treated for 60 minutes at this concentration.

The general observation is that the best results are obtained for long-time treatments (90-120mins) with low concentration of NaOH (2wt%-8wt %) and short-time treatments (60mins) with high concentration of NaOH (10wt %), though low concentration NaOH gave best results overall. This is similar to the observation of Wlodek et al (2012) on treatment of Jute fiber.

The general Response Surface Model used is:

$$y = c_0 + c_1x_1 + c_2x_2 + c_3x_1x_2 + c_4x_1^2 + c_5x_2^2$$

where  $y$  represents the response, which in this case are the mechanical properties,  $x_1$  and  $x_2$  represent NaOH concentration and treatment time respectively and  $c_i$  are model constants.

It can be observed from Table 7 to Table 11 that NaOH concentration and treatment time contribute from 14% to 51% of the variability observed in the mechanical properties of the fibers, based on the values of the  $R^2$ . This is not out of place, considering that factors like plant variety, climate, maturity, harvesting technique, retting degree, size (fiber diameter) and other factors that affect the mechanical properties of natural fibers were not factored into our model and due to a lower hemicellulose content of oil palm empty fruit bunch fiber in comparison to other fibers like banana fiber.

The  $R^2$  values reveal that NaOH concentration and treatment time contribute to variations in mechanical properties of Oil Palm Empty Fruit Bunch Fiber in this order (from the most to the least): tensile strength (51%), yield strength (38%), modulus of elasticity (23%), toughness and percentage reduction in area (15%) and percentage elongation (14%).

The significance of each model coefficient can be judged based on the value of the t-statistics (which must have a magnitude of 2 or more to be significant) or the p-value. The coefficient of NaOH concentration is significant for tensile strength, modulus of elasticity and yield strength at 90% confidence, the coefficients of time and time squared are significant for tensile strength and yield strength at 95% confidence. The coefficients of NaOH concentration squared and the interaction between NaOH concentration and treatment time are not significant. No coefficient of the RSM model was significant for toughness and percentage elongation. The non-significant interaction between NaOH concentration and time for most mechanical properties indicate that the variables do function almost independently.

In general, a model of the reduced form:  $y = c_0 + c_1x_1 + c_2x_2 + c_3x_2^2$  may be used to effectively model the mechanical properties of mercerized oil palm empty fruit bunch fiber.

The statistical model based on tensile strength is adequate at 95% confidence bound, while that of yield strength is significant at 90% confidence bound based on the F-statistics. The statistical models for other mechanical properties are not adequate.

The response surface models were optimized using a MATLAB 7.9 code and the optimum NaOH concentrations and time obtained are presented in Table 12. It can be observed from Table 12 that NaOH concentrations of approximately 5-7wt% give the best results for times of approximately 80-90mins.

The response surface models were used to obtain the surface plot and study the interaction of the variables (NaOH concentration and time) with respect to all mechanical properties studied and the results are presented in Figure 1 to Figure 5. It can be observed from Figure 1 to Figure 5 that NaOH concentration and time have a significant quadratic relationship with most of the mechanical properties, indicating the existence of a global optimum NaOH concentration and time, except for ductility (percentage elongation), which relates linearly with NaOH concentration. The nature of the contour lines, which are parallel one to another for all mechanical properties, reveal that there is no significant interaction between the two variables (NaOH concentration and treatment time) for all mechanical properties. This corroborates the numerical values obtained from the response surface models.

The analysis of variance study on the contributions of varying NaOH concentration and time on the observed improvement and variation in mechanical properties of empty palm bunch fiber are presented in Table 13 to Table 17. It can be observed from the results of Table 13 to Table 17 that time has significant effect on the observed changes in fiber tensile strength and yield strength at 95% confidence, while the contribution of NaOH concentration is not significant for all mechanical properties of empty palm bunch fiber studied, though it plays more role in improvement of toughness and ductility than time does. Based on the above observation it will be wrong to study the mechanical properties of empty palm bunch fiber as a function of NaOH concentration alone, while keeping time constant, as some authors do with certain fibers. It will be more appropriate to keep NaOH concentration constant and

vary time in the study of the mercerization of empty palm bunch fiber as observed in some studies [1].

#### IV. CONCLUSION

Mercerization can be used to produce high strength fibers. The mercerization of oil palm empty fruit bunch fiber increases the Modulus of Elasticity up to eighteen times, the yield strength more than eight times, Tensile strength more than six times and Toughness about six times. Ductility is the only property not affected by mercerization. The optimum concentration and time may vary depending on the mechanical property of interest, but a NaOH concentration of 6wt% and treatment time of 90mins has the best improvement on all mechanical properties. This is therefore the recommended NaOH concentration and time for mercerization of oil palm empty fruit bunch fiber. Treatment time has the most significant effect on fiber mechanical properties among the two properties studied.

#### APPENDIX

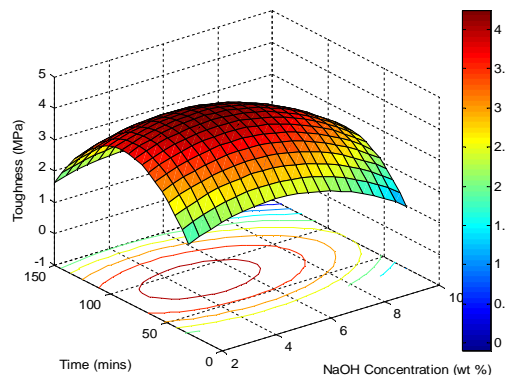


Figure 1: Surface Plot of NaOH Conc. and Time interaction on Toughness

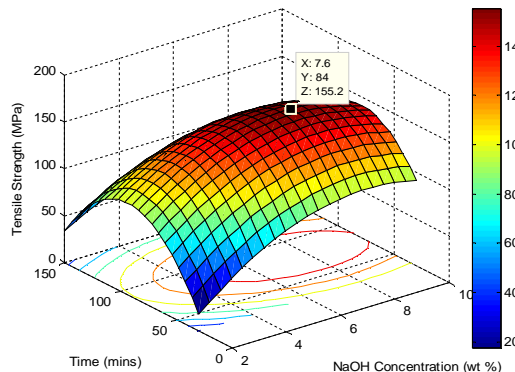


Figure 2: Surface plot of NaOH conc. and time vs. Tensile Strength

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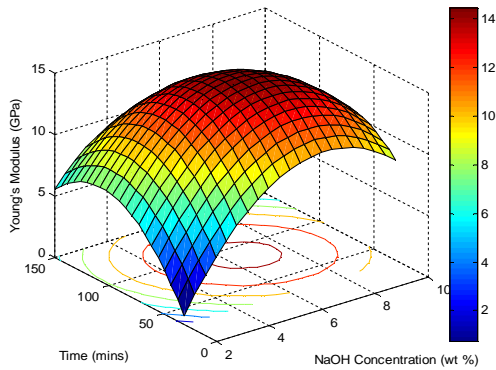


Figure 3: Surface Plot of NaOH conc. and time vs. Young's Modulus

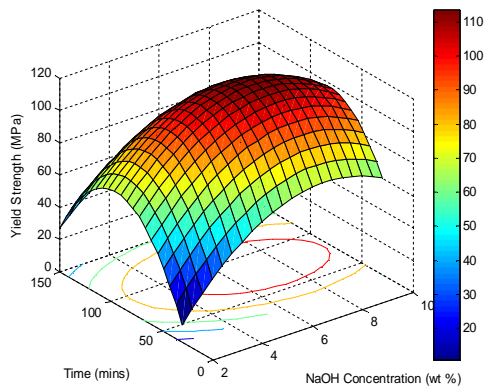


Figure 4: Surface plot of NaOH conc. and time vs. Yield Strength

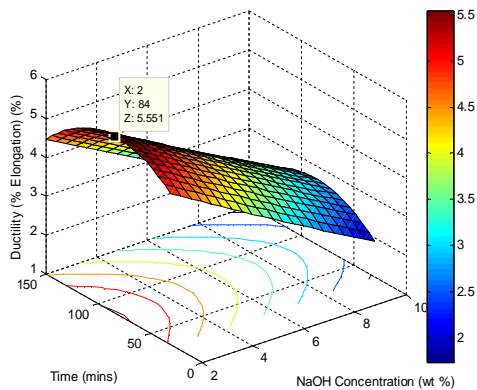


Figure 5: Surface plot of NaOH conc. and time interaction on %Elongation

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Table 1: Mechanical Properties of Untreated Empty Palm Bunch Fiber

Toughness (MPa)	%El (%RA) (%)	Tensile Strength (MPa)	Yield Strength (MPa)	Young Modulus (GPa)	Poisson Ratio	Shear Modulus (GPa)
2.3699	15.12 (13.14)	36.1078	26.5766	1.8909	0.4496	0.6522

Table 2: Mechanical Properties of Empty Palm Bunch Fiber Treated with 2 wt% NaOH

Variables (Units)	Time (mins)	30	60	90	120	150
Toughness (MPa)		2.0761	0.7917	2.2744	*2.7092	1.5499
Tensile Strength (MPa)		43.3645	49.7159	55.0786	*85.1043	36.4263
Modulus of Elasticity (GPa)		3.2450	7.1210	3.3660	*9.0780	8.3260
Yield Strength (MPa)		32.7285	34.9675	42.8189	*72.0920	23.2409
Percentage Elongation (%)		*5.7500	2.1250	5.0000	3.7500	5.0000

\*Maximum values

Table 3: Mechanical Properties of Empty Palm Bunch Fiber Treated with 4 wt% NaOH

Variables (Units)	Time (mins)	30	60	90	120	150
Toughness (MPa)		0.6843	*14.3601	2.5243	6.8328	3.0796
Tensile Strength (MPa)		50.5294	140.8620	*169.8607	137.5546	85.8809
Modulus of Elasticity (GPa)		5.8290	5.2700	12.8400	3.1810	*14.6800
Yield Strength (MPa)		32.4204	51.5168	*157.1335	82.5811	71.6270
Percentage Elongation (%)		2.0000	*15.5750	2.1875	8.0000	4.1250

\*Maximum values

Table 4: Mechanical Properties of Empty Palm Bunch Fiber Treated with 6 wt% NaOH

Variables (Units)	Time (mins)	30	60	90	120	150
Toughness (MPa)		0.9421	*2.8331	2.1540	1.0595	0.6943
Tensile Strength (MPa)		56.7366	151.2424	*221.6477	42.3879	39.5472
Modulus of Elasticity (GPa)		6.3020	*23.3800	16.5100	4.5150	7.7410
Yield Strength (MPa)		41.4708	119.0245	*216.3740	25.6929	31.0433
Percentage Elongation (%)		2.2500	2.3250	1.7000	*3.2500	2.1250

\*Maximum values

Table 5: Mechanical Properties of Empty Palm Bunch Fiber Treated with 8 wt% NaOH

Variables (Units)	Time (mins)	30	60	90	120	150
Toughness (MPa)		2.1576	1.1098	*5.3847	1.9992	1.1022
Tensile Strength (MPa)		84.1348	98.9586	*211.2172	98.9812	81.4439
Modulus of Elasticity (GPa)		8.4820	10.9000	*34.6600	5.2110	11.7800
Yield Strength (MPa)		65.1417	88.9040	*126.2990	63.9426	57.4827
Percentage Elongation (%)		3.3000	1.6250	*3.3250	3.2500	1.8750

Table 6: Mechanical Properties of Empty Palm Bunch Fiber Treated with 10 wt% NaOH

Variables (Units)	Time (mins)	30	60	90	120	150
Toughness (MPa)		1.4199	*3.5656	0.9490	2.4830	0.6604
Tensile Strength (MPa)		97.8939	*184.0318	89.1509	84.4281	76.1398
Modulus of Elasticity (GPa)		6.8400	*10.7900	7.5530	3.2560	6.5260
Yield Strength (MPa)		*86.1386	78.1704	73.4620	62.8273	72.0961
Percentage Elongation (%)		2.2750	3.0250	1.7500	*4.4500	1.5000

\*Maximum values

Table 7: Response Surface Model Based on Toughness

Variables	Coefficients	Std. Error	t-stat	P-value	F-stat
Constant	-1.7341	4.7757	-0.36311	0.72053	SSE=166.03
NaOH Conc. (wt %)	0.67362	1.1678	0.57682	0.57083	DFE=19
Time (mins)	0.10178	0.077855	1.3073	0.20672	DFR=5
NaOH Conc. * Time	-9.0302e-4	0.0049268	-0.18329	0.85651	SSR=29.46
NaOH Conc.^2	-0.063008	0.08833	-0.71332	0.48432	F=67426
Time^2	-5.6483e-4	0.00039258	-1.4388	0.16649	P-val = 0.64801
	R <sup>2</sup> = 0.1507	Adj. R <sup>2</sup> = -0.0728			

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Table 8: Response Surface Model Based on Tensile Strength

Variables	Coefficients	Std. Error	t-stat	P-value	F-stat
Constant	-140.2432	68.855	-2.0368	0.05585	SSE=34513
NaOH Conc. (wt %)	36.0216	16.838	2.1394	0.045603	DFE=19
Time (mins)	3.8753	1.1225	3.4524	0.0026684	DFR=5
NaOH Conc. * Time	-0.0670	0.071034	-0.94322	0.35741	SSR=35313
NaOH Conc.^2	-2.0710	1.2735	-1.6262	0.12038	F=3.888
Time^2	-0.020047	0.0056601	-3.5419	0.0021781	P-val =0.013511
	R <sup>2</sup> = 0.5057	Adj. R <sup>2</sup> = 0.3757			

Table 9: Response Surface Model Based on Modulus of Elasticity

Variables	Coefficients	Std. Error	t-stat	P-value	F-stat
Constant	-15.0202	11.26	-1.334	0.19798	SSE=922.9
NaOH Conc. (wt %)	5.3777	2.7534	1.9531	0.065692	DFE=19
Time (mins)	0.2670	0.18356	1.4546	0.16211	DFR=5
NaOH Conc. * Time	-0.0092113	0.011616	-0.793	0.43757	SSR=270.3
NaOH Conc.^2	-0.34831	0.20825	-1.6725	0.11081	F=1.1129
Time^2	-0.0011597	0.00092557	-1.2529	0.22544	P-val =0.38653
	R <sup>2</sup> = 0.2265	Adj. R <sup>2</sup> = 0.0230			

Table 10: Response Surface Model Based on Yield Strength

Variables	Coefficients	Std. Error	t-stat	P-value	F-stat
Constant	-107.6181	63.28	-1.7007	0.10532	SSE=29151
NaOH Conc. (wt %)	29.2316	15.474	1.889	0.074247	DFE=19
Time (mins)	2.7550	1.0316	2.6705	0.015119	DFR=5
NaOH Conc. * Time	-0.045485	0.065282	-0.69675	0.4944	SSR=18025
NaOH Conc.^2	-1.8113	1.1704	-1.5476	0.13821	F=2.3496
Time^2	-0.014049	0.0052018	-2.7009	0.014163	P-val =0.080527
	R <sup>2</sup> = 0.3821	Adj. R <sup>2</sup> = 0.2195			

Table 11: Response Surface Model Based on Ductility (% Elongation)

Variables	Coefficients	Std. Error	t-stat	P-value	F-stat
Constant	4.7625	4.9214	0.96771	0.34535	SSE=176.32
NaOH Conc. (wt %)	-0.411109	1.2035	-0.34159	0.73641	DFE=19
Time (mins)	0.038388	0.08023	0.47847	0.63777	DFR=5
NaOH Conc. * Time	2.6667e-4	0.0050771	0.052523	0.95866	SSR=29.083
NaOH Conc.^2	0.0024554	0.091025	0.026975	0.97876	F=0.62681
Time^2	-2.3651e-4	0.00040455	-0.58461	0.56569	P-val =0.68142
	R <sup>2</sup> = 0.1416	Adj. R <sup>2</sup> = -0.0843			

Table 12: Optimum NaOH concentration and time predicted with response surface model

	Toughness	Tensile Strength	Young Modulus	Yield Strength	%El
NaOH Conc. (wt %)	4.7263	7.3314	6.5411	6.9800	2
Time (mins)	86.3195	84.4037	89.1388	86.7504	82.2827

Table 13: ANOVA for Empty Palm Bunch Fiber Based on Tensile Strength

Source	Sum Sq.	Df	Mean Sq.	F	Prob>F
NaOH Concentration (wt %)	13356.4198	4	3339.105	1.8723	0.16456
Time (mins)	27934.7207	4	6983.6802	3.9158	0.021075
Error	28535.0281	16	1783.4393		
Total	69826.1686	24			

Table 14: ANOVA for Empty Palm Bunch Fiber Based on Toughness

Source	Sum Sq.	Df	Mean Sq.	F	Prob>F
NaOH Concentration (wt %)	53.5657	4	13.3914	1.9685	0.14807
Time (mins)	33.0806	4	8.2701	1.2157	0.34281
Error	108.8433	16	6.8027		
Total	195.4896	24			

Table 15: ANOVA for Empty Palm Bunch Fiber Based on Modulus of Elasticity

Source	Sum Sq.	Df	Mean Sq.	F	Prob>F
NaOH Concentration (wt %)	226.2109	4	56.5527	1.4124	0.27478
Time (mins)	326.3502	4	81.5875	2.0377	0.13732
Error	640.6368	16	40.0398		
Total	1193.1979	24			

Table 16: ANOVA for Empty Palm Bunch Fiber Based on Yield strength

Source	Sum Sq.	Df	Mean Sq.	F	Prob>F
NaOH Concentration (wt %)	6462.8459	4	1615.7115	1.1368	0.37451
Time (mins)	17972.7083	4	4493.1771	3.1615	0.042926
Error	22739.7141	16	1421.2321		
Total	47175.2683	24			

Table 17: ANOVA for Empty Palm Bunch Fiber Based on Ductility (%El)

Source	Sum Sq.	Df	Mean Sq.	F	Prob>F
NaOH Concentration (wt %)	58.4487	4	14.6122	1.8409	0.17036
Time (mins)	58.4487	4	4.9873	0.62831	0.64926
Error	127.0015	16	7.9376		
Total	205.3994	24			