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Optimum Conditions for Mercerization of Plantain Empty Fruit Bunch Fiber

Osoka E. C.[1, *], Onukwuli O. D.[2]

[1]Department of Chemical Engineering, Federal University of Technology, Owerri, Nigeria
[2]Department of Chemical Engineering, Nnamdi Azikiwe University, Awka, Nigeria

Abstract

Empty Plantain 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 empty plantain bunch fiber increases the Modulus of Elasticity up to fifty times, the yield strength more than thirty times, Tensile strength more than fifteen times and Toughness about thirteen times. The optimum concentration and time for most of the mechanical properties was a NaOH concentration of 4wt% and treatment time of 120mins. NaOH concentration has the most significant effect on fiber mechanical properties among the two properties studied, though there is significant interaction between the two variables. The optimum NaOH concentration of 4wt% and treatment time of 120mins from this study is recommended for mercerization of Empty Plantain Bunch Fiber prior to use in composite manufacture.

Keywords

Mercerization, Empty Plantain Bunch Fiber, Response Surface, Tensile Strength, Modulus

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1. Introduction

Alkali Treatment of cellulosic fibers, also called mercerization, is the typical 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 thus increasing the number of possible reaction sites and allowing for better fiber wetting.

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

$$\text{Fiber} - \text{OH} + \text{NaOH} \rightarrow \text{Fiber} - \text{O} - \text{Na} + + \text{H}_2\text{O}$$

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,

* Corresponding author
E-mail address: emmaosoka@yahoo.com (Osoka E. C.), onukwuliod@yahoo.com (Onukwuli O. D.)

especially on fiber strength and stiffness. The tensile strength and modulus of jute fiber were improved by 120% and 150% respectivelyafter 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]. Some authors have studied the effect of mercerization on empty plantain bunch fiber and observed that it improved fiber and composite mechanical properties, but their treatment was done at a single NaOH concentration and treatment time and may not have given us fibers with optimum mechanical properties [12, 13]. This work presents optimum conditions for mercerization of empty plantain fruit bunch fiber at ambient temperature. NaOH solution with concentration of 2-10wt% will be used for mercerization the fiber at treatment times of 30-150mins to determine optimum mercerization conditions that will consider NaOH concentration and treatment time interactions.

2. 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.

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 Empty Plantain 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.

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.

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.

3. 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 Tables 1 and6, that Empty Plantain Bunch Fiber treated with 2wt % NaOH had its Toughness increased 3.4 times relative to the untreated sample, the Tensile Strength of the treated sample was more than 12.5 times the untreated one, Yield Strength was increased almost 8 times, Modulus of Elasticity increased 20 times, while there was no significant improvement in ductility. 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 Tables 2 and 6, that Empty Plantain Bunch Fiber treated with 4wt % NaOH had its Toughness increased more than 13 times relative to the untreated sample, the Tensile Strength of the treated sample was more than 16.5 times the untreated one, Yield Strength was increased more than 32 times, Modulus of Elasticity more than 50 times, while there was no significant improvement in ductility. Treatment at this concentration resulted in increase in all mechanical properties in comparison with that of 2 wt % NaOH. Eighty percent of the mechanical properties also had their maximum values after the fiber had been treated at this concentration for 120 minutes. This is most probably because mercerization alters fiber composition by removing cementing substances (lignin and hemicellulose) and

increases its aspect ratio through fibrillation, thus increasing fiber strength, but as the process continues cellulose dissolution sets in, thus weakening the fiber. This can be observed for treatment times above 120mins.

It can also be observed from Tables 3 and 6, that Empty Plantain Bunch Fiber treated with 6wt % NaOH had its Toughness increased about 13 times relative to the untreated sample, the Tensile Strength of the treated sample was more than 16.5 times the untreated one, Yield Strength was increased almost 24 times, Modulus of Elasticity more than 40 times, while there was no significant improvement in ductility. Treatment at this concentration resulted in a slight drop in all mechanical properties in comparison with that of 4 wt %, indicating that the optimum concentration of NaOH for mercerization of Empty Plantain Bunch Fiber would lie between 4wt% and 6wt %. Eighty percent of the mechanical properties also had their maximum values after the fiber had been treated at this concentration for 120 minutes. This is due to removal of cementing substances (lignin and hemicellulose) and increase in fiber aspect ratio through fibrillation at the onset of mercerization, thus increasing fiber strength, and subsequent cellulose dissolution which weakens fiber for treatment times beyond 120mins. The change in NaOH concentration from 4wt% to 6wt% did not significantly change treatment time for onset of cellulose dissolution

Observations from Tables 4 and 6 show that Empty Plantain Bunch Fiber treated with 8wt % NaOH had its Toughness increased almost 9 times relative to the untreated sample, the Tensile Strength of the treated sample was more than 15 times the untreated one, Yield Strength was increased almost 30 times, Modulus of Elasticity increased more than 20 times, while there was no significant improvement in ductility. Treatment at this concentration resulted in a slight drop in almost all mechanical properties in comparison with that of 6 wt %. Forty percent of the mechanical properties had their maximum values after the fiber had been treated at this concentration for 30 minutes. Treatment using 8wt% NaOH significantly reduced time for onset of cellulose dissolution, thus lowering optimum treatment time at this concentration.

Observations from Tables 5 and 6 reveal that Empty Plantain Bunch Fiber treated with 10wt % NaOH had its Toughness increased 9.5 times relative to the untreated sample, the Tensile Strength of the treated sample was more than 11 times the untreated one, Yield Strength was increased more than 19 times, Modulus of Elasticity increased 17 times, while there was no significant improvement in ductility. Treatment at this concentration resulted in a slight drop in all mechanical properties in comparison with that of 8 wt %. Eighty percent of the mechanical properties had their maximum values after the fiber had been treated at this concentration for 30 minutes. Generally a slight decrease in ductility was observed after treatment.

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.

Tables 7 to 12 reveal that NaOH concentration and treatment time contribute from 38% to 73% 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.

It can be observed based on the R^2, NaOH concentration and treatment time contribute to variations in mechanical properties of Empty Plantain Bunch Fiber in this order (from the most to the least): Tensile Strength, Yield Strength, Modulus of elasticity, Toughness and Ductility.

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 all mechanical properties, the coefficient of the interaction between NaOH concentration and treatment time is significant for Toughness, Tensile Strength, Percentage Elongation and Percentage Reduction in Area while the coefficient of NaOH concentration squared is significant for Toughness, Tensile Strength, Modulus of Elasticity and Yield Strength. Only for Tensile Strength did the coefficient of treatment time show some significance. The significant interaction between NaOH concentration and time for most mechanical properties indicate that the variables do not function independently; rather the choice of NaOH Concentration could determine whether or not a mechanical property will increase or reduce with treatment time and vice versa.

In general, a model of the reduced form: $y = c_0 + c_1x_1 + c_2x_1x_2 + c_3x_1^2$ may be used to effectively model the mechanical properties of mercerized Empty Plantain Bunch Fiber.

The statistical model based on Tensile Strength and Yield Strength are adequate at 95% confidence bound, while the other models are adequate at 90% confidence bound based on the F-statistics. Figures 1 to 5 reveal the effect of interaction of the two variables on the mechanical properties of Empty Plantain Bunch fiber. It can be observed that there is a

significant linear relationship between time and all the mechanical properties, while a quadratic relationship exists for NaOH concentration, indicating an optimum concentration. The nature of the contour lines - not being parallel one to another - except for Yield Strength and Young's Modulus, reveal a high level of interaction between the two variables for these mechanical properties. This corroborates the numerical values obtained from the Response Surface Models.

Tables 13 to 17 show results from Analysis of Variance study on the contributions of varying NaOH concentration and time on the observed improvement and variation in mechanical properties of Empty Plantain Bunch Fiber. The Tables reveal that NaOH concentration has significant effect on the observed changes in Fiber Toughness and Ductility at 90% confidence, it also has significant effect on observed changes in fiber Tensile strength, Modulus of elasticity and Yield strength at 95% confidence, while the contribution of time is not significant for all mechanical properties of Empty Plantain Bunch Fiber studied.

The above observation explains why several authors studied the mechanical properties of some natural fibers as functions of NaOH concentration alone, while keeping time constant.

4. Conclusion

Mercerization can be used to produce high strength fibers. The mercerization of empty plantain bunch fiber increases the Modulus of Elasticity up to fifty times, the yield strength more than thirty times, Tensile strength more than fifteen times and Toughness about thirteen 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 4wt% and treatment time of 120mins has the best improvement on all mechanical properties. This is therefore the recommended NaOH concentration and time for mercerization of empty plantain bunch fiber. NaOH concentration has the most significant effect on fiber mechanical properties among the two properties studied, though there is significant interaction between the two variables.

Table 1. Mechanical Properties of Empty Plantain Bunch Fiber Treated with 2 wt% NaOH.

Time (mins) Variables (Units)	30	60	90	120	150
Toughness (MPa)	3.6740	1.8963	1.6332	*15.7895	11.2058
Tensile Strength (MPa)	144.0551	149.8745	189.5307	383.8447	*647.1856
Modulus of Elasticity (GPa)	24.6400	26.3800	19.9000	*27.4700	25.5600
Yield Strength (MPa)	92.5748	99.9599	150.3280	*172.9783	72.6703
Percentage Elongation (%)	3.1250	1.7500	1.4500	*5.7000	3.2500

*Maximum values.

Table 2. Mechanical Properties of Empty Plantain Bunch Fiber Treated with 4 wt% NaOH.

Time (mins) Variables (Units)	30	60	90	120	150
Toughness (MPa)	13.5841	24.8134	35.3623	*51.9357	40.0158
Tensile Strength (MPa)	288.1098	576.4851	615.0219	*860.5139	718.4211
Modulus of Elasticity (GPa)	28.0100	27.7800	*70.3500	48.1400	27.5100
Yield Strength (MPa)	194.2555	354.6910	308.4525	*661.5980	465.2110
Percentage Elongation (%)	5.7500	5.7500	6.9500	*7.2750	7.2500

*Maximum values.

Table 3. Mechanical Properties of Empty Plantain Bunch Fiber Treated with 6 wt% NaOH.

Time (mins) Variables (Units)	30	60	90	120	150
Toughness (MPa)	9.0455	9.7172	16.1488	*50.9526	9.6217
Tensile Strength (MPa)	379.2531	481.8816	588.1484	*855.6013	484.8420
Modulus of Elasticity (GPa)	53.0300	22.2200	38.9300	40.1300	*58.2800
Yield Strength (MPa)	311.9990	301.5460	394.3195	*487.3150	369.0740
Percentage Elongation (%)	2.8750	3.3750	3.8000	*7.5250	2.5000

*Maximum values.

Table 4. Mechanical Properties of Empty Plantain Bunch Fiber Treated with 8 wt% NaOH.

Time (mins) Variables (Units)	30	60	90	120	150
Toughness (MPa)	*35.2483	19.1183	17.6685	5.6792	10.8406
Tensile Strength (MPa)	665.1185	*783.1146	703.5303	529.9170	491.8404
Modulus of Elasticity (GPa)	24.6500	21.1600	23.9700	25.8100	*29.9200
Yield Strength (MPa)	425.2360	327.1095	*611.1310	529.9170	459.4035
Percentage Elongation (%)	*7.1250	4.6250	4.0500	2.0000	3.0000

*Maximum values.

Table 5. Mechanical Properties of Empty Plantain Bunch Fiber Treated with 10 wt% NaOH.

Time (mins) Variables (Units)	30	60	90	120	150
Toughness (MPa)	*37.3020	5.3062	7.3692	5.1919	0.6821
Tensile Strength (MPa)	*614.2335	353.2127	267.1565	262.9740	177.0635
Modulus of Elasticity (GPa)	14.6400	21.6100	*23.6000	21.8300	17.5600
Yield Strength (MPa)	*404.1075	250.8445	144.2045	210.5690	177.0635
Percentage Elongation (%)	*8.5000	2.5000	3.6250	2.7500	1.0000

*Maximum values.

Table 6. Mechanical Properties of Untreated Empty Plantain Bunch Fiber.

Toughness (MPa)	%El (%)	Tensile Strength (MPa)	Yield Strength (MPa)	Young Modulus (GPa)
3.5669	9.25	51.6471	19.7363	1.3070 (1.0162)

Table 7. Response surface model based on toughness.

Variables	Coefficients	Std. Error	t-stat	P-value	F-stat
Constant	-39.4839	21.8280	-1.8089	0.08632	SSE = 3468.5
Conc. (wt %)	16.2275	5.3376	3.0402	0.006734	DFE =19
Time (mins)	0.3697	0.35584	1.0389	0.31187	DFR =5
Conc. * Time	-0.0578	0.022518	-2.5673	0.018855	SSR =2235.5
Conc.^2	-0.9469	0.40372	-2.3455	0.030009	F =2.4493
Time^2	-6.8642e-5	0.0017943	-0.038256	0.96988	
	R^2 = 0.3919	Adj. R^2 = 0.2319			P-VALUE =0.071205

Table 8. Response surface model based on tensile strength.

Variables	Coefficients	Std. Error	t-stat	P-value	F-stat
Constant	-793.5894	208.33	-3.8093	0.0011844	SSE = 3.1595e5
Conc. (wt %)	329.5172	50.944	6.4682	3.3705e-6	DFE =19
Time (mins)	9.2166	3.3963	2.7137	0.013775	DFR =5
Conc. * Time	-1.0257	0.21492	-4.7723	0.00013247	SSR =8.5164e5
Conc.^2	-19.4045	3.8532	-5.0359	7.3374e-5	F =10.243
Time^2	-0.0118	0.017125	-0.68955	0.49882	
	R^2= 0.7294	Adj. R^2= 0.6542			P-VALUE = 7.039e-5

Table 9. Response surface model based on modulus of elasticity.

Variables	Coefficients	Std. Error	t-stat	P-value	F-stat
Constant	483.30	19145	0.025244	0.98012	SSE = 2.6682e9
Conc. (wt %)	1.1830e4	4681.6	2.527	0.020538	DFE = 19
Time (mins)	127.1052	312.11	0.40725	0.68838	DFR = 5
Conc. * Time	0.3483	19.751	0.017636	0.98611	SSR = 1.7697e9
Conc.^2	-1.0932e3	354.1	-3.0873	0.0060642	F = 2.5203
Time^2	-0.4513	1.5738	-0.28674	0.77741	
	R^2=0.3988	Adj. R^2=0.2405			P-VALUE = 0.065262

Table 10. Response surface model based on yield strength.

Variables	Coefficients	Std. Error	t-stat	P-value	F-stat
Constant	-497.8359	183.76612	-2.70907	0.01391	SSE = 2.46e5
Conc. (wt %)	233.6400	44.93729	5.1992	5.1044e-5	DFE = 19
Time (mins)	4.1869	2.99582	1.39757	0.17835	DFR = 5
Conc. * Time	-0.2721	0.18958	-1.43547	0.16741	SSR = 3.9e5
NaOH Conc.^2	-16.1247	3.39888	-4.74411	0.00014	F = 6.0541
Time^2	-0.0106	0.01571	-0.70423	0.48983	
	R^2=0.6144	Adj. R^2=0.5129			P-VALUE = 0.0016318

Table 11. Response surface model based on ductility (% elongation).

Variables	Coefficients	Std. Error	t-stat	P-value	F-stat
Constant	-1.4160	3.1233	-0.45336	0.65542	SSE = 71.015
Conc. (wt %)	1.8808	0.76377	2.4626	0.023521	DFE = 19
Time (mins)	0.0279	0.050918	0.54738	0.59049	DFR = 5
Conc. * Time	-0.0089	0.0032222	-2.7569	0.012545	SSR = 43.367
NaOH Conc.^2	-0.0951	0.057768	-1.646	0.1162	F = 2.3206
Time^2	9.1270e-5	0.00025675	0.35548	0.72614	P-VALUE = 0.083484
	R^2= 0.3791	Adj. R^2 = 0.2158			

Table 12. Optimum NaOH Concentration and Time from Response Surface Models.

	Toughness	Tensile Strength	Young Modulus	Yield Strength	%El
NaOH Conc. (wt %)	3.9907	4.5263	5.4335	6.2559	8.4848
Time (mins)	150	150	142.9179	117.2018	30

Table 13. Anova for Empty Plantain Bunch Fiber Based on Toughness.

SOURCE	SUM SQ.	DF	MEAN SQ.	F	PROB>F
NaOH Concentration (wt %)	2004.7	4	501.175	2.57	0.0776
Time (mins)	584.39	4	146.099	0.75	0.5720
Error	3114.81	16	194.676		
Total	5703.9	24			

Table 14. Anova for Empty Plantain Bunch Fiber Based on Tensile Strength.

SOURCE	SUM SQ.	DF	MEAN SQ.	F	PROB>F
NaOH Concentration (wt %)	497057.4	4	124264.4	3.31	0.0372
Time (mins)	68657	4	17414.3	0.46	0.7614
Error	600875.8	16	37554.7		
Total	1167590.2	24			

Table 15. Anova for Empty Plantain Bunch Fiber Based on Modulus of Elasticity.

SOURCE	SUM SQ.	DF	MEAN SQ.	F	PROB>F
NaOH Concentration (wt %)	2.08409e9	4	5.21022e8	4.23	0.0159
Time (mins)	3.83072e9	4	9.57681e7	0.78	0.5558
Error	1.97073e9	16	1.23171e8		
Total	4.43789e9	24			

Table 16. Anova for Empty Plantain Bunch Fiber Based on Yield strength.

SOURCE	SUM SQ.	DF	MEAN SQ.	F	PROB>F
NaOH Concentration (wt %)	395578.4	4	98894.6	8.87	0.0006
Time (mins)	63431.1	4	15857.8	1.42	0.2720
Error	178486.3	16	11155.4		
Total	637495.8	24			

Table 17. ANOVA for Empty Plantain Bunch Fiber Based on Ductility (%El).

SOURCE	SUM SQ.	DF	MEAN SQ.	F	PROB>F
NaOH Concentration (wt %)	36.542	4	9.13562	2.39	0.0939
Time (mins)	16.744	4	4.18594	1.10	0.3919
Error	61.096	16	3.81852		
Total	114.383	24			

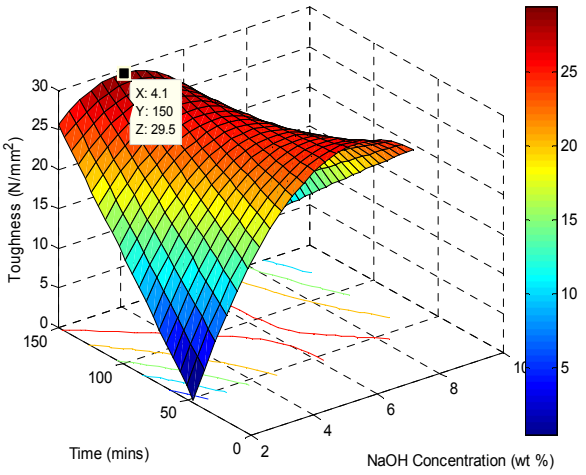

Figure 1. Surface Plot of Effect of NaOH Conc. and Time interaction on Toughness.

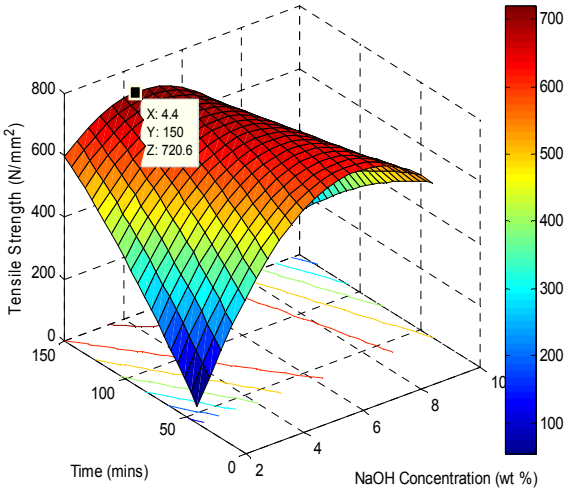

Figure 2. Surface Plot of Effect of NaOH Conc. and Time interaction on Tensile Strength.

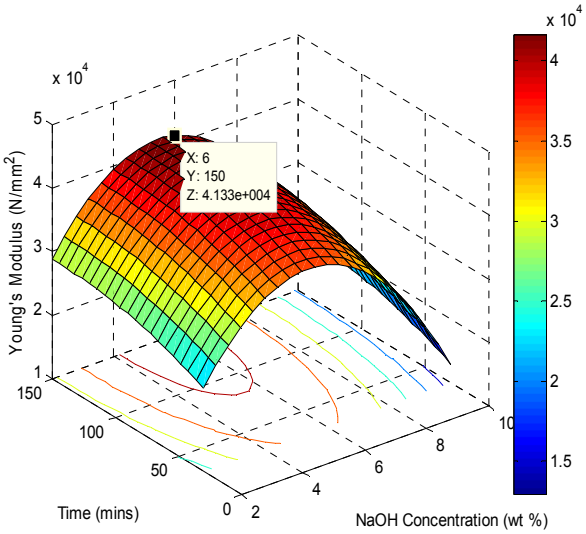

Figure 3. Surface Plot of Effect of NaOH Conc. and Time interaction on Young's Modulus.

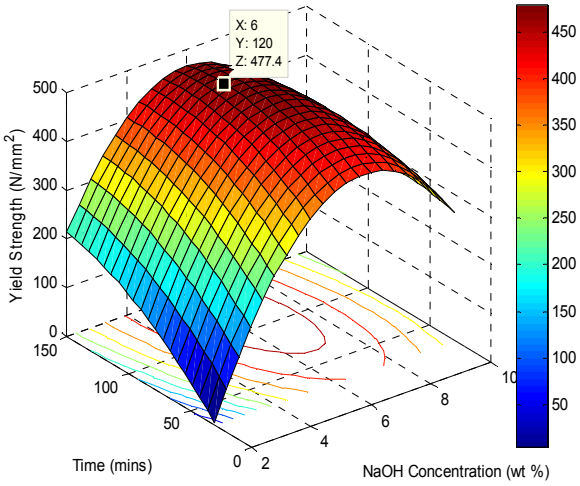

Figure 4. Surface Plot of Effect of NaOH Conc. and Time interaction on Yield Strength.

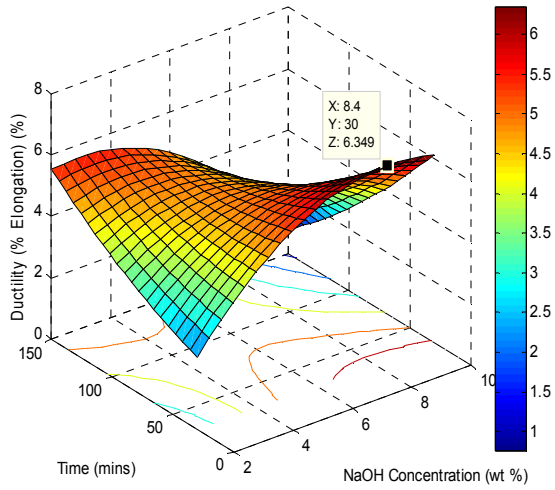

Figure 5. Surface Plot of Effect of NaOH Conc. and Time interaction on Tensile Ductili.

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