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Optimal Parameter Setting for Mercerization of Bamboo Fibres

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ABSTRACT

A Study on the main and interaction effect of mercerization, using 4,6 and 8wt% concentration of NaOH at 30, 60 and 90 minutes soaked time in alkali, was carried out on bamboo fibre to observe the effect of the pre-treatment on the tensile properties of fibre and the optimal parameter setting for mercerization of bamboo fibre. The result of the investigation indicated that retting bamboo fibre for too long using NaOH could have negative effect on the fibre. The optimal parameter setting, measured as a function of the tensile strength, was obtained at 8wt% NaOH concentration and 60 minutes soaked time in NaOH.

1. Introduction

Hydrophilic fibres and hydrophobic matrix are not very compatible this result in poor adhesion at the fibre–matrix interface and hinders effective load transfer from matrix to the fibre [1, 2]. Hence, it is important to alter the natural state of natural fibres in order to enhance the interfacial adhesion of fibre and matrix and to improve the resulting composite. Previous researchers have attempted to enhance the interfacial adhesion of natural fibres with matrix through alkali treatment [3, 4, 5, 6, 7, 8]. Alkaline treatment is carried out to increase the interfacial adhesion between fibres and their matrix. During alkaline treatment, hemicelluloses, lignins and pectins are removed from natural fibre and the fibre surface becomes rougher [9]. Mercerization treatment is represented as follows;

$$\text{Fibre-OH} + \text{NaOH} \rightarrow \text{Fibre - O}^{+}\,\text{Na}^{+} + H_2O \quad (1)$$

The effectiveness of mercerization depends on the type and concentration of the alkali solution, temperature and time of treatment. Optimal condition of mercerization ensures improvement of tensile strength and absorption characteristics [10]. However, some researchers have reported decrease in tensile properties of fibre and fibre composites when treated with high alkali concentration and extended period of treatment due to damage of the main structures of the fibre [11, 5, 12]. Some researchers had used different concentration of NaOH and soaking time to mercerize various natural fibres. Kushwaha & Kumar [9], immersed bamboo fibres in 5wt% NaOH concentration for 30 minutes at room temperature. They reported increased in tensile strength and elastic modulus of the produced bamboo fibre-epoxy composite by 40% and 35% respectively. El-

Shekeil et al. [13], used 0.5M NaOH for 1 hour in alkaline treatment of palm fibre. In mercerization of oil palm fruit fibre and rice husk, Hassan et al. [14] used 6 wt % NaOH and soaking time of 2hours. Lina et al. [15], used the same parameter setting for mercerization of banana fibre, they all reported improvement in the tensile properties of the composites produced with these fibres. Flax fibre was mercerized with 5wt% NaOH for 30 min by Yan et al [16], they observed 16.1% improvement in tensile strength of flax/epoxy composite produced with the treated fibre. Sawpan et al [17], used 5%wt NaOH for 30 minutes on Hemp Fibre. They observed higher tensile strength and young modulus for the hemp PLA composite produced with the treated fibre. A research was conducted by Shukor et al. [18] to study the effect of NaOH concentration (3%, 6% and 9%) on kenaf fibre reinforced PLA bio-composite. They obtained the maximum flexural modulus and impact strength of the composite at 6wt% NaOH concentration at room temperature. Roy et al. [19], found the maximum tensile strength of alkaline treated jute fibre to be at 0.5% NaOH for 24hours.
Mishraet al [20], used 5 and 10 wt% concentration NaOH at 1hour for pineapple and sisal fibre. They observed that that at 10wt%, the polyester composites produced with the fibres became weaker. John et al. [21], used 0.5,1,2 and 4%wt NaOH for 1hour on sisal and oil palm fibre. Shehu et al [4], used 2,4,6,8 and 10 wt% NaOH for soaking time of 1200seconds (20minutes) on baobab pod fibres; they found that 10wt% NaOH treatment gave the highest tensile strength.
Symington et al. [11], carried mercerization using 3%wt NaOH at 10,20 and 30 minutes for mercerization of lax, abaca, kenef and sisal fibre. They found that alkaline treatment has a negative effect on all the fibres and suggest that a treatment time of 10 minutes or less with 3wt% NaOH be used for the fibres. Wong et al. [5], used 1, 3 and 5wt% NaOH for 24hours at room temperature for bamboo fibre mercerization. They observed that untreated fibre has higher strength than all the treated fibres. Viel et al. [12], used 5%wt NaOH for 24hours during mercerization of bamboo fibre. They found reduction in interfacial shear strength from 7.0MPa in untreated bamboo fibre – Poly lactic acid (PLA) to 5.3MPa in treated fibers. Prasad et al [22], reported a decrease in tensile strength of sisal fibres after 90hours of soaking at 5% aqueous NaOH. Zhu et al. [23], reported decrease in tensile strength of flax fibre when mercerized with 5% NaOH for 2hours. Herrea-Franco & Valadez-Gonzalez [2], used 5, 10, and 15wt% NaOH for 1and 2hours on Curaua fibres. He observed decrease in tensile strength with increasing NaOH content. Munawar et al. [24], used 2% wt/v NaOH for 2hours on pineapple fibre. They observed decrease in tensile strength and young modulus of the produced pineapple-HDPE composite of mercerized fibre.

Due to variation in chemical constituents of different types of fibre, the optimal mercerization conditions such as NaOH concentration and soaking time varies with fibre. However, different researchers had used different parameter settings in mercerization of the same type of fibre as shown in the preceding review. Thus, there is no standard parameter setting for bamboo fibre mercerization. Hence, there is need to optimize the mercerization parameter settings for each natural fibre in order to obtain an acceptable standard for that particular fibre. It is also noted from literature that many researchers worked on optimum NaOH concentration, little have been done on main effect and interaction effect with soaking time.
In this study, attempt is made to obtain optimal parameter setting for mercerization of bamboo fibre at room temperature. The main and interaction effect of NaOH concentration and soaking time was studied.

2. Materials and Method

2.1. Materials

Bamboo fibres extracted from *bambusa vulgaris,* was used in this research. Sodium Hydroxide was obtained from local chemical shop in Aba, Nigeria. Figure 1 shows the extracted bamboo fibres used for the mercerization studies.

Figure 1: Extracted bamboo fibres used for mercerization

2.2. Experimental design

Taguchi orthogonal array was used in the optimization of the mercerization parameter settings. Taguchi technique of design of experiment (DOE) attempts to obtain maximum information with minimum number of experiments. This method discovers significant factors quickly and reduces both experimental cost and time [25, 26].

The following optimization steps were followed in this study:

- Select control factors
- Select Taguchi orthogonal array
- Conduct experiment
- Measure the responses
- Analyze result
- Predict optimum parameter setting.

2.2.1 Factors and factor level selection

The effectiveness of mercerization depends on the concentration of the alkali solution, temperature and time of treatment. However, in order to reduce cost, some researchers have found mercerization treatment to be effective at room temperature [9, 17]. Previous researchers used different concentration of alkali and soaking time for mercerization. Thus there is no agreement on parameter settings for mercerization of natural fibres in general and bamboo fibres in particular. In this study, factor levels were selected from the range of levels used by previous researchers and optimized to achieve the best mercerization parameter settings within the feasible design space. The selected factors and their levels are listed in Table 1.

Table 1: Factors and their level in the experimental design

	Factors	
Levels	Soaked time (minutes)	NaOH concentration (wt %)
1	30	4
2	60	6
3	90	8

2.2.2 Taguchi orthogonal array

In this study, Taguchi orthogonal design was used to determine the optimal parameter settings for optimization of mercerization parameter settings using tensile properties as the response. Based on Taguchi's Orthogonal Array design, the experimental data needs to be transformed into a signal-to-noise (S/N) ratio for analysis. The quality characteristic that higher value represents desired response, 'higher-the-better', is used. The S/N ratio was calculated as a logarithmic transformation of the loss function.

In this study, the effect of the variables, time soaked in alkali and alkali concentration and their interactions on tensile properties was designed on a *L*9 orthogonal array. The design matrix for 0.25mm and 0.30mm average fibre diameter, produced using MNITAB 17.0 software, and the corresponding responses are presented in Table 2 and Table 3 respectively.

Table 2: Design matrix and the responses for mercerization (0.25mm Diameter)

Soaked Time (Min)	NaOH Concentration	Signal-to-Noise Ratio	MEAN
30	4	59.2645	925.9255
30	6	61.89804	1244.699
30	8	62.44637	1440.412
60	4	62.0025	1260.1
60	6	65.01386	1790.75
60	8	65.24262	1961.496
90	4	63.10542	1434.304
90	6	60.77777	1099.162
90	8	60.31446	1076.949

Table 3: Design matrix and the responses for mercerization (0.3mm Diameter)

Soaked Time (Min)	NaOH Concentration	Signal-to-Noise Ratio	MEAN
30	4	57.89004	789.6622
30	6	60.96123	1206.411
30	8	61.99306	1274.141
60	4	57.96523	799.0634
60	6	62.50641	1356.203
60	8	63.81298	1646.216
90	4	62.0325	1273.113
90	6	59.1073	904.1525
90	8	58.49041	903.1499

2.3. Mercerization of bamboo fibre

The fibres were soaked in various concentration of NaOH at different soaking times in line with the design matrix (Table 2 and Table 3). After soaking, the fibres were washed with running water and immersed in distilled water for one hour in order to remove any residual NaOH. Finally, they were dried in an oven at 60^0C until a constant weight was achieved. The dried fibres were stored in plastic bags to prevent exposure to moisture.

2.4. Single fibre tensile test

In the single fibre tensile test (SFTT), four measurements were taken along the gauge length of a single fibre and the average was recorded as the fibre diameter. The two ends of each fibre were adhered into paper grips using adhesive glue with a gauge length of 10 mm. The test set-up is presented in Figure 2. The single fibre is elongated under tensile load until it breaks. The fibres that broke close to the grip were rejected. The tensile test was conducted at ambient conditions using the universal testing machine UTEST model UTM-7001, whereby the fibres were subjected to cross head speed of 1 mm/min until complete failure, according to ASTM D3379 (ASTM 1989). The Tensile testing machine displayed the load and maximum stress, which is the tensile strength of the fibre. A minimum of 20 fibres from each set were tested.

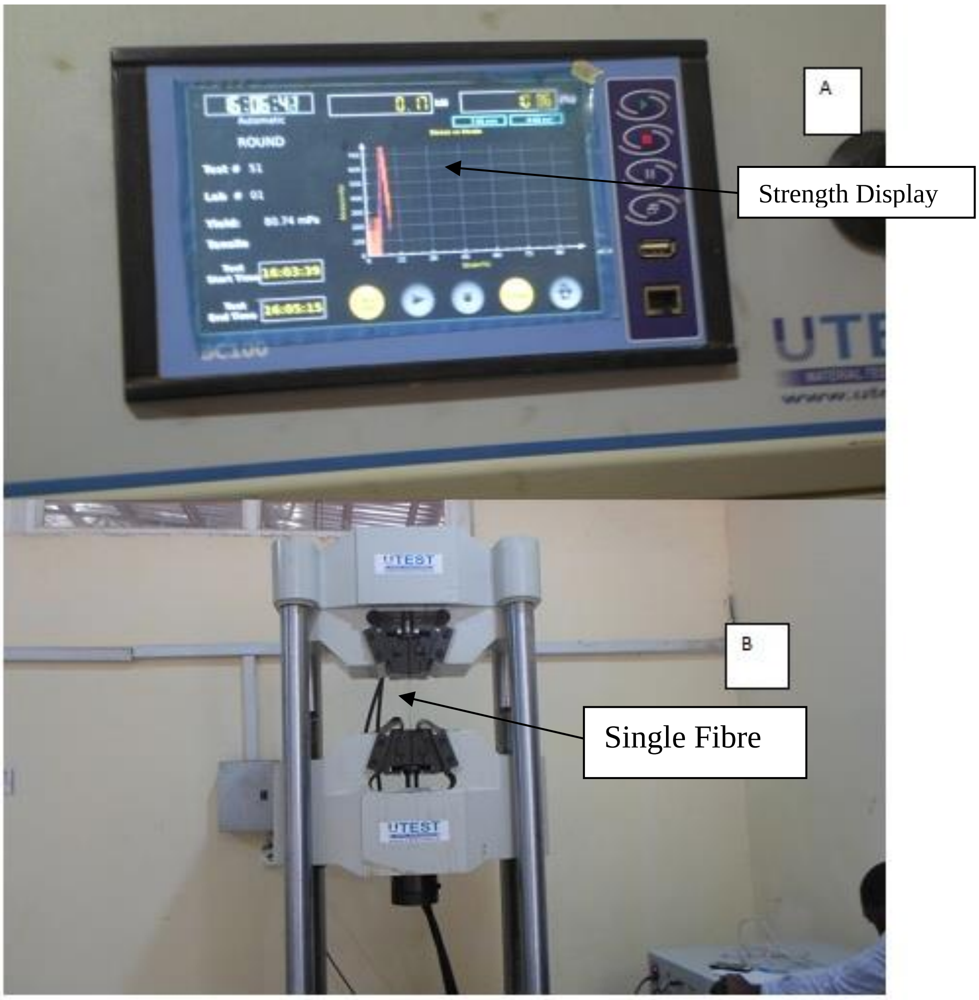

Figure 2: Universal testing machine UTEST model UTM-7001 setup for single fibre tensile test. "A" is the load and maximum strength display, "B" is the tensile test setup

3. Results and Discussion

3.1 Analysis of S/N ratio

From Taguchi analysis (Table 4 and Table 5), the optimal mercerization parameter settings - the levels with the highest signal-to-Noise ratios, measured as a function of tensile strength was obtained at level 2 for soaked time in NaOH which corresponds to 60 minutes and level 3 for NaOH concentration which corresponds to 8% for both the 0.25mm and 0.30mm diameter fibres. The "Delta" values (Table 4 and Table 5) are the defining differences between highest signal-to-noise ratio and the lowest signal to noise ratio of a factor. The ranks of the delta indicate that soaked time in NaOH has higher influence than NaOH concentration for the 0.25mm diameter fibre (Table 4). While for 0.30mm diameter fibre, the rank shows higher influence of NaOH concentration than time soaked in NaOH (Table 5).

Table 4: Response table for signal to noise ratios - larger is better (0.25mm)

Level	Soaked Time (Minutes)	NaOH Concentration (%)
1	61.2	61.46
2	**64.09**	62.56
3	61.4	**62.67**
Delta	2.88	1.21
Rank	1	2

Table 5: Response table for signal to noise ratio - larger is better (0.33mm)

Level	Soaked Time (Min)	NaOH Concentration
1	60.28	59.3
2	**61.43**	60.86
3	59.88	**61.43**
Delta	1.55	2.14
Rank	2	1

3.2. Effect of NaOH concentration and soaked time

The main effect plot for mean of 0.25mm and 0.30mm diameter fibres (Figures 3 and 4), shows that tensile strength increases with increase time soaked in NaOH from 30 minutes to 60 minutes and decreases with further increase in soaked time from 60 minutes to 90 minutes Soaking for a long time caused some deterioration to the fibre particles and reduced the tensile strength. The main effect plot for NaOH concentration shows a steady increase in tensile strength with increase in alkali concentration up the maximum concentration used in this study; that is 8% NaOH concentration.

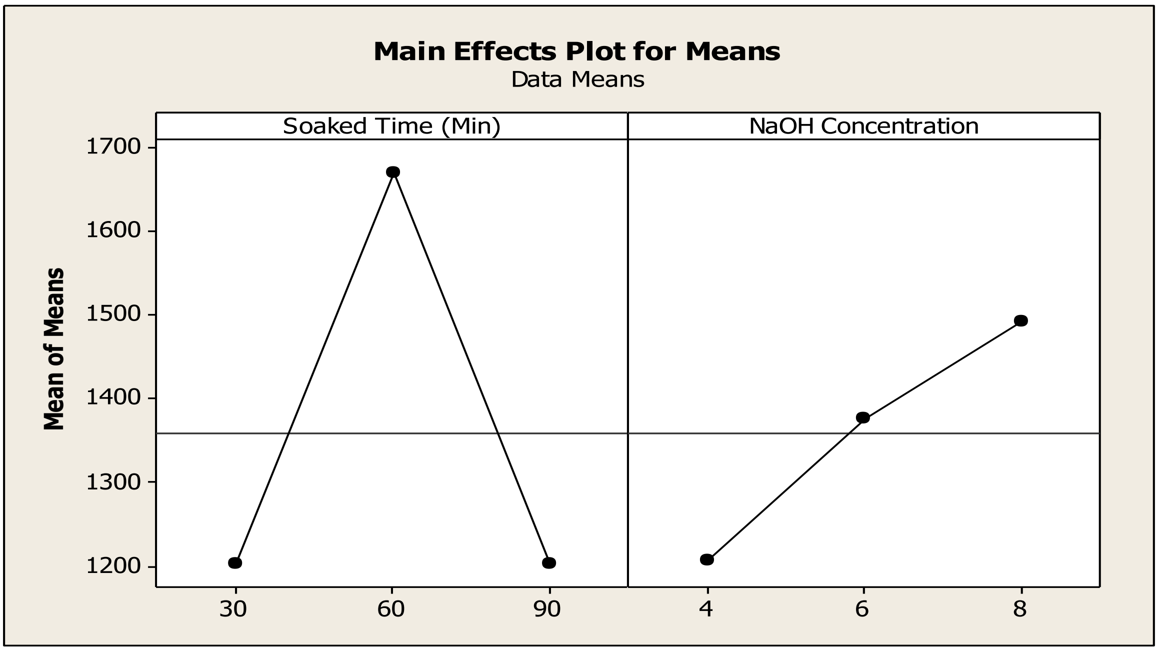

Figure 3: Main effect plot of mercerization for 0.25mm diameter fibre

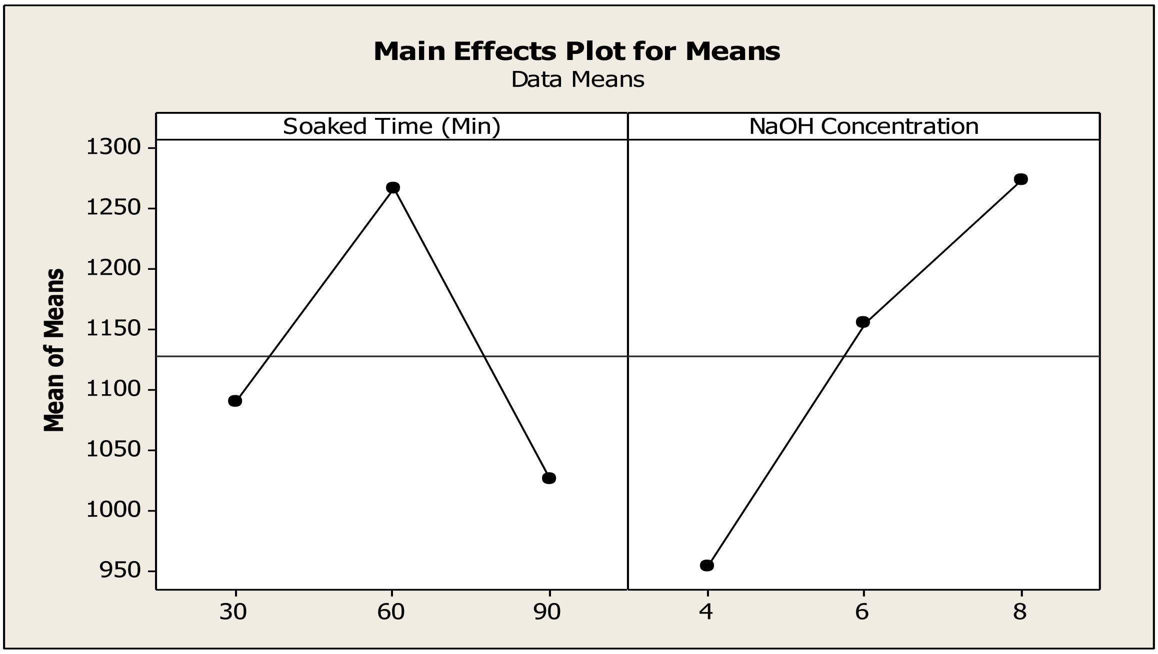

Figure 4: Main effect plot of mercerization for 0.30mm diameter fibre

The interaction plot for means of 0.25mm and 0.30mm diameter fibres are shown in Figure 5 and 6. Parallel lines indicate no interaction in an interaction plot while intersections indicate interaction. From the interaction plot, it can be seen that there non parallel line among the variables. At 4% NaOH concentration; which was the lowest concentration used, longer soaking time (i.e. 90 minutes) showed higher tensile strength while 30 minutes soaked time showed the lowest tensile strength. However, at higher NaOH concentration of 8 %, the longer soaking time of 90 minutes resulted to the lowest tensile strength while 60 minutes soaked time gave rise to the highest tensile strength. The tensile strength of the 30 minutes soaked time was between 60 minutes and 90 minutes.

At low treatment time the removal of lignin, waxes and other impurities was minimal resulting in low tensile strength. Even at high concentration of NaOH with low treatment time, the tensile strength was observed to be low. The fibres required some time to swell so that the mercerization agent will be able to enter into the fibre and decompose the impurities. Thus, enough time is required for the swelling action to take place. Vardhini *et al* [27], observed similar behaviour on decomposition of lignin in banana fibre. However, soaking for too long time (beyond 60 minutes) lead to decomposition of cellulose resulting in decreased tensile strength.

Teli & Jadhav [28], also observed similar behaviour in *Pandanus Odorifer* fibre.They observed increase in tensile properties as the concentration of NaOH is increased from 2 to 15% at 60 minutes soaked time, after which further increase in concentration resulted to decrease in tensile strength. They attributed this to the removal of amorphous hemicelluloses and other waxy substances from the fibre on alkali treatment which increases the overall crystallinity of the fibre. Removal of hemicelluloses results to fibrils rearrangement in compact manner which leads to a close packing of the cellulose chain resulting to improvement of tensile properties of the fibre.

Differences were not observed in the behaviour of 0.25mm diameter fibres and 0.30mm diameter bamboo fibres when soaked in NaOH. However, the tensile strength of 0.25mm diameter fibres was generally, higher than that of 0.30mm diameter fibres both before and after modification. This agrees with the findings of Sergio *et al* [29], on giant Bamboo fibres, where they found the fibre diameter to vary inversly with tensile stregth.

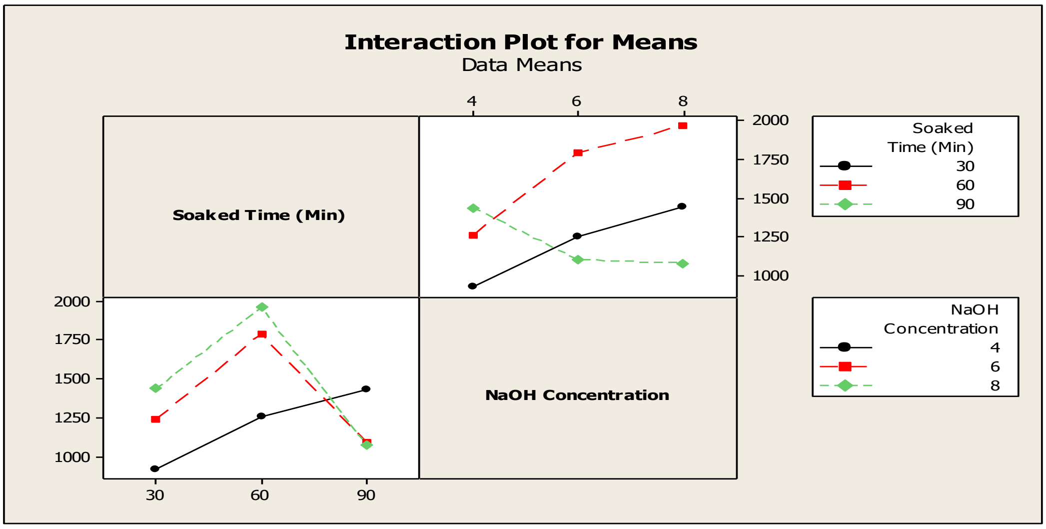

Figure 5: Interaction plot of mercerization for 0.25mm diameter fibre

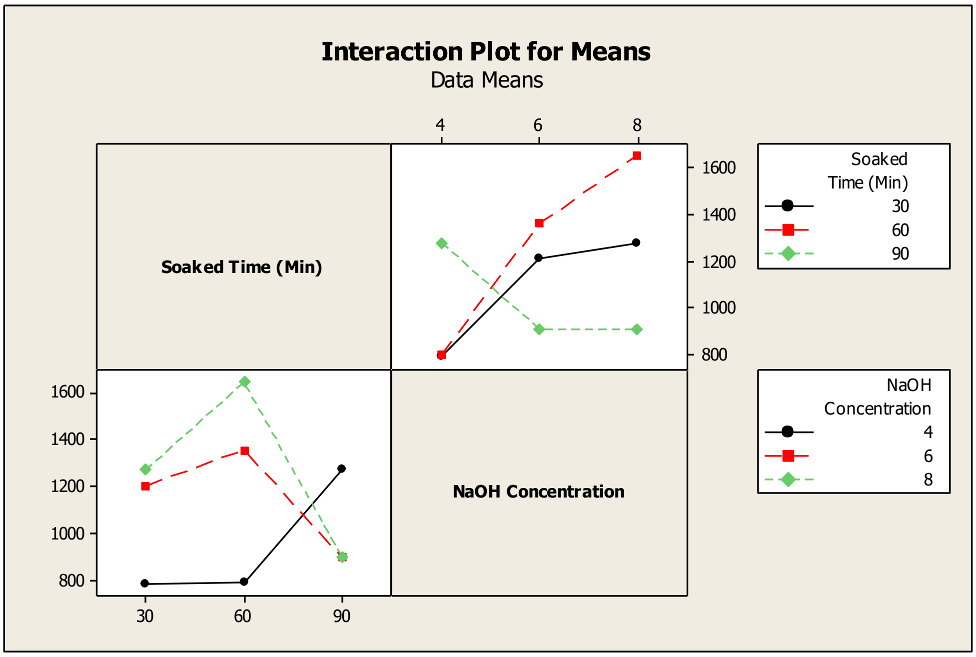

Figure 6: Interaction plot of mercerization for 0.30mm diameter fibre

4. Conclusion

Tensile testing was carried out to determine the main and interaction effect of mercerization on bamboo fibre. The result of this investigation shows that interaction between NaOH concentration and treatment time affects the tensile strength of the fibre. At low treatment time, the tensile strength was observed to be low. However, soaking for too long time (beyond 60 minutes) lead to decomposition of cellulose resulting to decreased tensile strength. At low concentration, long treatment time resulted to high tensile strength; while at high concentration, long treatment time lead to low tensile strength. The optimal parameter setting for high tensile strength was obtained at 8 wt% NaOH concentration and 60 minutes treatment time. Generally, treating bamboo fibre for too long using NaOH could have a negative effect on the fibre. It is therefore, suggested that the optimal parameter setting obtained in this investigation be used in mercerization of bamboo fibre for composite production.

5. Conflict of Interest

There is no conflict of interest associated with this work.

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