THE ALKALI TREATMENT PARAMETERS USING TAGUCHI MODEL IN ORDER TO OBTAIN THE OPTIMUM TENSILE STRENGTH OF SINGLE KENAF FIBER

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Abstract

The development of high-performance engineering products made from natural resources is increasing worldwide. Kenaf plants have been extensively exploited over the past few years. Chemical treatment is considered to modify the fiber surface properties. In this study, kenaf bast fibers were treated with various concentrations of NaOH with different immersed time, immersed temperature, and dried temperature. Fiber bundle tensile were performed to evaluate the effect of treatments on the fiber tensile strength. Taguchi Methods are used in order to obtain the optimal parameter which could affect the tensile strength of kenaf fibers. Three-Level Orthogonal array is used to design the experiment. Finally, the experimental results will be evaluated using analysis of variance (ANOVA). The analysis of variance (ANOVA) shows that the most significant alkali parameter is NaOH concentration, which accounts for 40.19 percent of the total. It is also found that the optimum treatment is kenaf immersed in 3 wt. percent NaOH solution for 1 hour at 33 degrees celcius and dried at 60 degrees celcius which is supported by the Fourier Transform Infrared Spectroscopy.

Keywords: *kenaf alkali treatment, Taguchi Method, optimization, fourier transform infrared spectroscopy*

1. Introduction

Natural fiber especially kenaf are increasingly used as an alternative instead of glass and other sintetic fibers in composite materials. It has an ability in absorbing nitrogen and phosphorus included in the soil (Abe, 1998), low density, high toughness, comparable specific strength properties, non-abrasiveness during processing, low energy consumption in fabrication, and CO_2 neutrality (Mohanty, 2000). Recently, kenaf is used in the textiles (Ramaswamy, 1995), fiber-board and automotive industries (Magurno, 1999).

Kenaf, *Hibiscus cannabinus*, is an herbaceous and common wild annual plant of tropical and also subtropical in Africa and Asia that can be grown under a wide range of weather condition (Seller, 1999). The plant which is from Malvaceae family has a unique combination of long bast and short core fibers. It also can grow in a warm season at a short amount of time.

Kenaf has been known in Indonesia since the development of a program called ISKARA (Intensification of Citizen's Fiber Bag) in 1978 when the fiber was mostly used for

sack industries. There are many potential area which kenaf could be cultivated in this country, like Lamongan, Jepara, Banten, and Klaten. Unfortunately, the high rate of kenaf grown in Indonesia is not followed by the utilization itself.

Work has been done to develop methods for processing kenaf fibers. Investigation of different treatments in fiber has been conducted. Various treatments for obtaining high strength and other mechanical properties of fibers were screened and tested to determine the best process for large-scale production. Current research on kenaf fiber is dealing with fiber characterization which used only one or two different parameters but work on optimization using Taguchi Methods with more than three parameters in increasing the tensile strength of kenaf fibers and the interface characteristics for further application in composites is limited.

Kenaf is presently being used in paper production on a very limited basis. Various uses of the bast fibers have been explored, such as in the making of industrial socks to absorb oil spills, as well as making woven and non-woven textiles. The kenaf bast fiber is known to have the potential as a reinforcing fiber in thermoplastic composites, because of its superior toughness and high aspect ratio in comparison to other fibers. A single fiber of kenaf can have a tensile strength and modulus as high as 11.9 GPa and 60 GPa, respectively (Karnani, 1997).

Rowell et al. (Rowell, 1999) studied the potential of kenaf as a reinforcing fiber in a polypropylene matrix and compared the mechanical properties with other commonly used composite systems. The results are shown in Table 1. Kenaf core fibers are also used in product applications such as animal bedding, summer forage, and potting media (Ramaswamy, 2003).

Table 1. Properties of filled/reinforced polypropylene composites (Rowell, 1999)

Filler/reinforcement in PP	Units	Neat PP	Kenaf	Glass	Talc	Mica
Filler by weight	%	0	50	40	40	40
Filler by volume	%	0	39	19	18	18
Specific gravity	-	0.9	10.7	1.23	1.27	1.26
Tensile modulus	Gpa	1.7	8.3	9	4	7.6
Specific tensile modulus	Gpa	1.9	7.8	7.3	3.1	6
Tensile strength	Mpa	33	65	110	35	39
Specific tensile strength	Mpa	37	61	89	28	31
Flexural modulus	Gpa	1.4	7.3	6.2	4.3	6.9
Specific flexural modulus	Gpa	1.6	6.8	5	3.4	5.5
Elongation at break	%	>>10	2.2	2.5	-	2.3
Notched izod impact	J/m	24	32	107	32	27
Water absorption (24h)	%	0.02	1.05	0.06	0.02	0.03

The properties of the composites depend on those of the individual components and on their interfacial compatibility. The adhesion between fiber and matrix is obtained by mechanical anchoring of the fiber ends into the matrix. In many cases, the absorption of moisture by untreated fibers, poor wettability cause insufficient interfacial adhesion to the polymer matrix. The lack of interfacial interactions leads to internal strains, porosity and environmental degradation. The wettability of the fiber depends on the polymer viscosity and the surface tension of both materials. The surface tension of the polymer must be as low as possible, at least lower than that of the fiber. Therefore, modification of the fiber and/or polymer matrix is a key area for obtaining good composite properties.

Alkali treatment (or mercerization) is one of the most widely used chemical treatments for natural fibers, especially the kenaf fiber when reinforcing thermoplastics and thermosets. This treatment involves immersing the fibers in an alkaline solution, frequently that of NaOH, for a period of time. The important modification that is done by alkali treatment is the disruption of hydrogen bonding in the network structure, thereby increasing surface roughness. Alkali treatment also leads to fibrillation of the fiber bundles into small fibers. In other words, this treatment reduces fiber diameter and thereby increases aspect ratio. The addition of aqueous sodium hydroxide (NaOH) to the natural fiber promotes the ionization of the hydroxyl group to the alkoxide (Eq. 1) (Aziz, 2003).

$$\text{Fiber} - \text{OH} + \text{NaOH} \rightarrow \text{Fiber} - \text{O} - \text{Na} + \text{H}_2\text{O} \quad (1)$$

There are several approaches to investigate the effects of different testing parameters. The most simple one is the single-parameter by single-parameter approach, i.e., only one parameter is changed for a given test run. This is of course the most time consuming and costly approach as the testing parameter number increases. To overcome this, the experimental design and dimensional analysis theory were introduced. The Taguchi Methods, by developing a set of standard Orthogonal Arrays (OA) and a methodology for the analysis of results, can extract information from experiment more precisely and more efficiently than other approaches, also fewer number of tests are needed even when the number of parameters being investigated is quite large (Taguchi, 1992). Since Taguchi Methods have been proved successful for many manufacturing circumstances, it is chosen in this study. The purpose of this study is to systematically investigate the effects of different alkali parameters and to obtain the best fiber resulting in the optimum composites.

2. Experimental Method

2.1 Materials

Kenaf raw fibers used in this work are supplied by Balittas (Balai Penelitian Tanaman Tembakau dan Serat) Malang and came in straight long fibers. The fibers have been separated from their stalks by water retting for about 20 days in Balittas. After the water retting process is completed, the fibers were then cleaned with water and dried under the sunlight before they were delivered.

2.2 Fiber Chemical Treatment

Kenaf fibers were chopped approximately up to 15 cm length in the middle and the bottom and the top part were discarded because the properties of the fiber varied from the bottom to the top. Kenaf fibers then were immersed in the NaOH solutions with different

concentrations (3, 6, and 9% NaOH) with different temperatures (33, 60, and 100°C) and different immersed time (1, 2, and 3 h). After immersion, the fibers were washed with running tap water and then immersed in distilled water containing 1% acetic acid to neutralize the remaining NaOH molecules. The fibers were then dried at different temperatures (33, 60 and 100°C) until dry.

2.3 Statistical Method

The four factors and three levels are selected, and the orthogonal array selected is L9. The Taguchi method is a powerful design of experiments tool, which provides a simple, efficient, and systematic approach to determine optimal parameters [10]. The treatment factors and their levels are listed in Table 2. The reason of using very selective range and levels of process parameters is because we would like to provide simplicity to find the basic knowledge and effect of how many percent the process parameters especially the alkali treatment affects the response variable. The experimental layout for the experiments is tabulated in Table 3. It was decided to select the trials at random and complete all of three successive replications. A number of readings at different fibers are taken to measure the respective response parameters.

Table 2. Treatment factors and their levels

Treatment parameter	Symbol	Unit	Level 1	Level 2	Level 3
NaOH	A	%	3	6	9
Immersed time	B	h	1	2	3
Immersed temperature	C	deg. C	33	60	100
Drying temperature	D	deg. C	33	60	100

Table 3. Experimental layout using L9 orthogonal array

Test Number	Treatment parameter			
	NaOH (A)	Immersed time (B)	Immersed temperature (C)	Drying temperature (D)
1	1	1	1	1
2	1	2	2	2
3	1	3	3	3
4	2	1	2	3
5	2	2	3	1
6	2	3	1	2
7	3	1	3	2
8	3	2	1	3
9	3	3	2	1

2.4 Fiber Bundle Tensile Test

Fiber bundle tensile strength tests were performed using universal tensile machine (UTM, Torsee AMU-5-DE) and the specimen was prepared by referring to ASTM D-3379. For every set of chemical treatment, 5 specimens were tested to determine the average fiber bundle strength. The tests were conducted at a standard laboratory atmosphere of 25 °C and 60% relative humidity. The tested fibers were embedded in resin and mechanically polished to facilitate the measurement of the average diameter of each fiber from its cross-sectional

area. The maximum breaking load was determined directly from the stress–strain curve and the unit break (UB) is calculated as follows (Eq.2)

$$UB = F/d \quad (2)$$

Where

F Maximum breaking load (N)

d Cross-sectional area of the fiber (mm sq.)

2.5 Infrared spectroscopy (FTIR)

Fourier Transform Infrared Spectroscopy (FTIR) is a technique that is extremely useful for the characterization of organic materials (including polymers) and certain inorganic compounds. The spectra obtained by FTIR provide information about the presence of specific molecular structures. This test works based on the absorption of infrared photons that excite vibrations of molecular bonds. This test will be performed to study the surface condition in fiber to know whether the hydrogen bonding in the network structure was distrupted or not, which is increasing the surface roughness. The FTIR analysis was performed using a FTIR spectrophotometer 8201PC Shimadzu. In order to obtain a good resolution of spectra, it was necessary to mill the kenaf fibers to an average length of 0.2 mm.

3. Results and Discussion

3.1 Alkali Treatment Optimization

Table 4 shows the experimental results for the alkali treatment. The influences of individual alkali treatment parameters on the tensile strength of kenaf fiber can be clearly seen in Fig. 1. The analysis of variance (ANOVA) showed in Table 5 presents that within the experimental level ranges, the most significant alkali parameter is NaOH concentration, which accounts for 40.19% of the total. The immersion time gave the contribution of 33.58% where the drying and immersion temperature parameters give the account of 21.39% and 4.85% respectively. It should be noted that this percentage contribution of each alkali parameter to the tensile strength is only valid within the experimental parameter amount level ranges, that is, the NaOH concentration from 3% to 9% wt., the immersion and drying temperature from room temperature to 100°C and the immersion time from 1 hour to 3 hours.

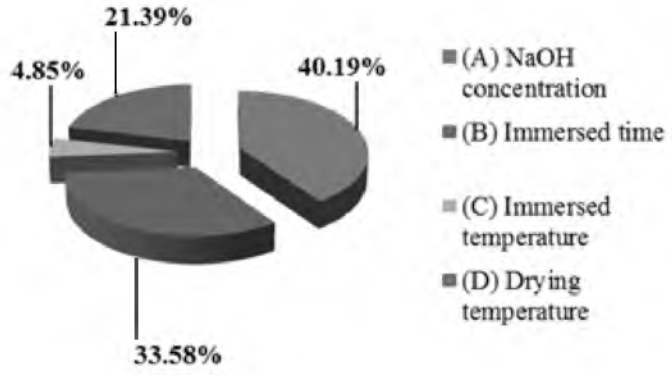

Fig. 1 The contribution in percentage of each factors that affect the single kenaf fiber tensile strength

Table 4. Experimental results (average from three replications)

Test No.	NaOH (%)	Immersed Time (h)	Immersed Temp. (deg. C)	Dried Temp. (deg. C)	Average Tensile Strength (MPa)
1	3	1	33	33	169.6445
2	3	2	60	60	176.3297
3	3	3	100	100	105.2200
4	6	1	60	100	213.9073
5	6	2	100	33	162.9590
6	6	3	33	60	254.2996
7	9	1	100	60	205.4518
8	9	2	33	100	175.7929
9	9	3	60	33	124.5595

Table 5. ANOVA of tensile strength

Factor	dof	Sum of square	Variance	contribution (%)
A	2	22.3722	11.1861	40.19
B	2	18.6941	9.3470	33.58
C	2	2.6975	1.3488	4.85
D	2	11.9064	5.9532	21.39
Error	0	0	-	-
Total	8	55.6702	27.8351	100

The optimum level for each factor summarized in Table 6. It can be seen that for factor A (NaOH concentration), the highest signal to noise is level 2, which means the optimum level for factor A is level 2 (6% wt. NaOH). It is the same with factor B, C, and D, which has the optimum condition in level 1, 1 and 2, respectively. It can be concluded that the optimum parameter for each factor which affects the single fiber tensile strength is 6% wt. NaOH concentration, 1 hour immersion at room temperature and dried at 60 deg. Celcius.

Table 6. Signal to noise ratio for each level of all factors

Level	A (NaOH)	B(Time)	C(I.Temp)	D(D.Temp)
1	41.6714	45.0817	43.9665	42.4558
2	45.3655	42.9132	42.8410	44.8176
3	42.5430	41.5849	42.7723	42.3064
ΔS/N	-3.6941	-2.1685	-1.1942	-2.3618

3.2 Fourier Transform Infrared Spectroscopy Analysis

FT-IR microscopy is a very powerful technique to characterize the chemical composition of natural and synthetic fibers, both organic and inorganic in nature. Due to its capability to measure with a high lateral resolution usually the availability of a single fiber is sufficient to perform an analysis. As the FT-IR measurement is non-destructive other analytical techniques might be applied afterwards.

Furthermore, FT-IR microscopy provides objective results and is in most cases quicker, easier, and sometimes, more selective than classical methods. Due to these multiple benefits the IR-microscopic method is described by ASTM International as standard method for forensic analysis of fibers (ASTM E2224-10) and for identification of fibers in textiles

(ASTM D276-12). Using the ATR- (Attenuated Total Reflectance) technique minimal sample preparation is required to perform an FTIR- microscopic measurement.

Just a fixation of the fiber on a flat substrate like a metal plate is required to avoid its movement during visual inspection and definition of the measurement positions. In this application note measurements of different natural and synthetic fibers using the fully automated FT-IR microscope LUMOS are presented.

Immersing kenaf in NaOH solution for an hour is the best parameter for immersion time (Table 6). Immersing fiber into an amount of time will led to the fiber damage. Natural fibres are composed of polysaccharides which correspond respectively to residual middle lamellae on their surface and components which play the role of a matrix by coating the cellulose fibrillar structure within the fibres. These include pectins and hemicelluloses that have high polar components of their surface energy, which favour the formation of hydrogen bonds with water. In addition water penetrates the amorphous regions, may diffuse along lumen paths and reduces the interactions between crystalline and amorphous zones. This induces reduction of their mechanical properties which could weaken the interlayer cohesion.

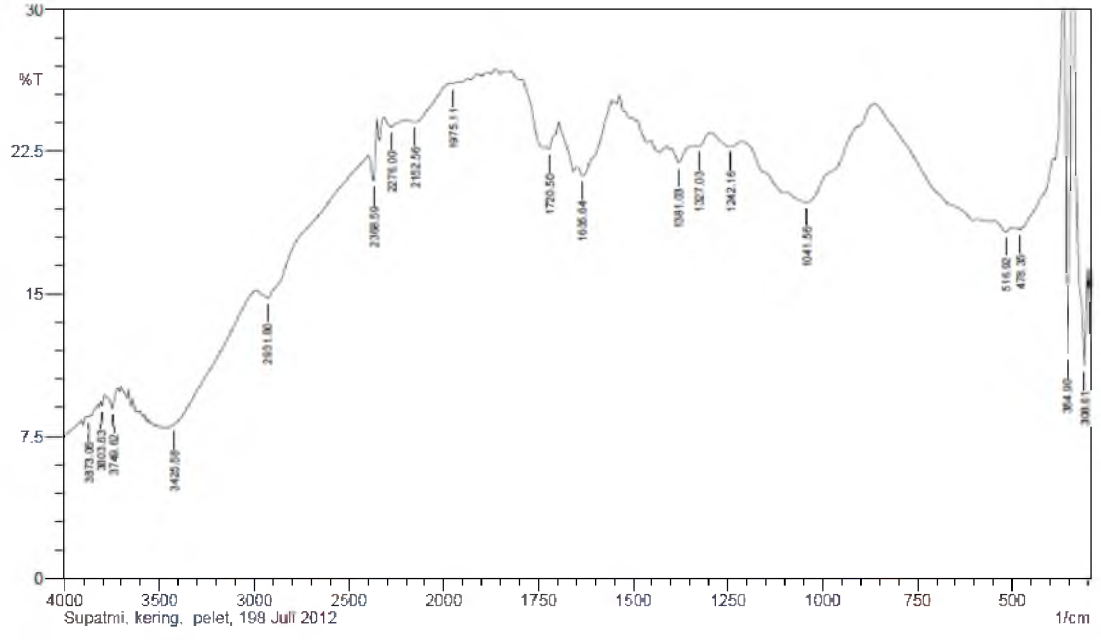

Fig.2 FTIR results of raw (untreated) kenaf fiber (Sosiati, 2013)

The most interesting results from the optimum combination parameters are the immersion temperature. It could be seen that the optimum value is immersing kenaf at a room temperature. Altough these results had many contradictions with the previous studies, but the results are valid because they are supported by the FTIR results. Figs 3 and 4 are the FTIR results of kenaf fiber both treated with 6 wt.% NaOH solution but with different immersing temperature. The cristallinity index of kenaf immersed at room temperature (3.52) is higher than that at 100^{o}C (3.08). As we know, the higher cristallinity index, the higher its tensile strength.(Ciolacu, 2011)

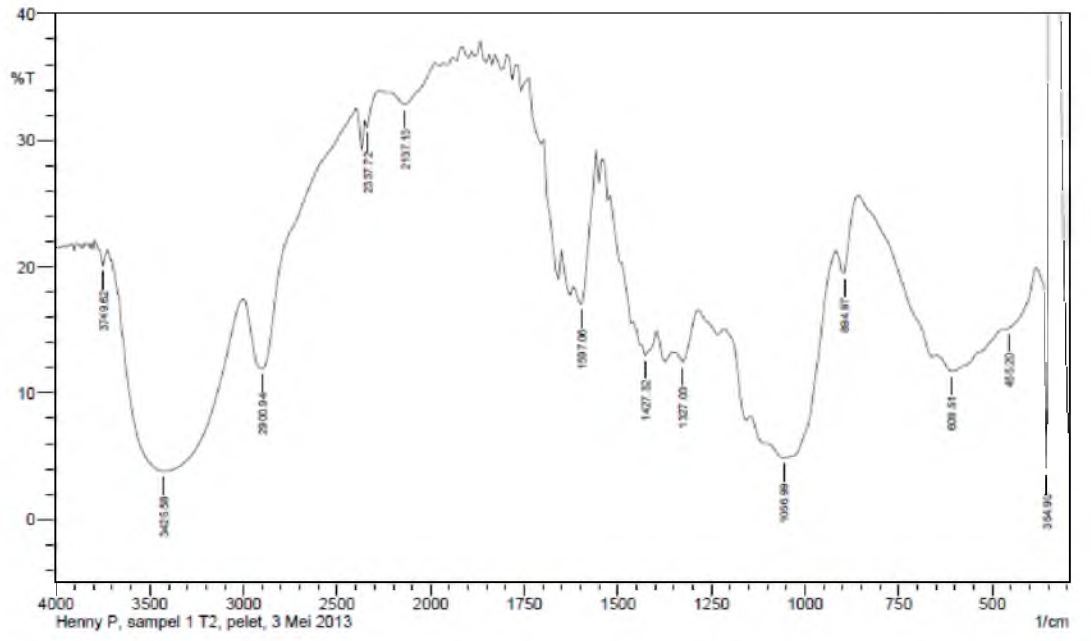

Fig. 3 FTIR results of kenaf fiber treated with 6% NaOH solution for an hour and immersed at room temperature.

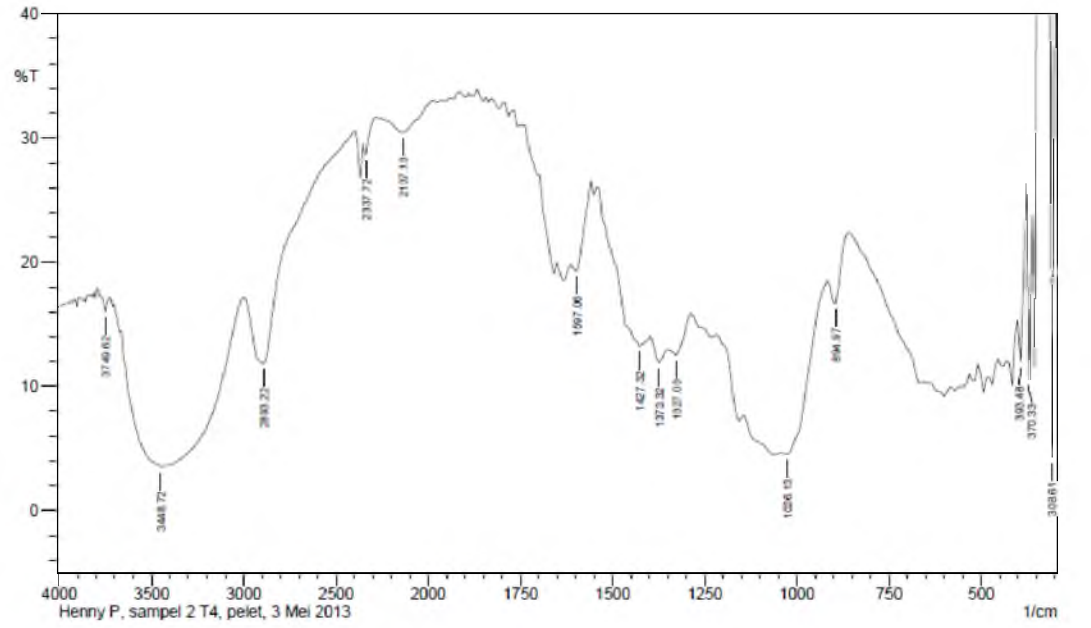

Fig. 4 FTIR results of kenaf fiber treated with 6% NaOH solution for an hour and immersed at 100°C.

If we compared the FTIR results of untreated fiber (Fig. 2) to the FTIR results of both treated kenaf fiber (Figs. 3 and 4), it could be observed that the treatments affected the presence of some functional groups in kenaf fiber. It is obvious that the broad absorption band around 3400 cm^{-1} is due to the stretching frequency of the hydroxyl group (–OH). The maximum absorption band –OH group stretching of fiber treated with NaOH 6 wt.%

immersed at 100°C is more than the other samples: kenaf immersed at room temperature and untreated kenaf.

The band peak at 2931-2893 cm^{-1} is due to the carbon-hydrogen bond (C-H) stretching vibration. For untreated kenaf, absorption peak at 1720 cm^{-1} is assigned to C=O stretching of the acetyl group in hemicelluloses and 1242 cm^{-1} is assigned to C-O stretching of the aryl group in lignin which are unable to see in FTIR for both treated kenaf fiber and the band peak at 1635 cm^{-1} is the mode of the absorbed water. The small peak at 1427 cm^{-1} in treated samples corresponds to CH_2 symmetric bending. A peak in the region of 1373-1381 cm^{-1} in all samples shows the weak C-O stretching and the absorbance at 1327 cm^{-1} is due to S ring stretching in lignin. For the treated samples, absorbance at 895 cm^{-1} has an association with C-O-C stretching of glycoside bonds.

4. Conclusion

In order to increase the tensile strength of kenaf bast fiber, the effect of alkali treatment was investigated. The results obtained from the experiments are summarized as follows:

1. The NaOH concentration of alkali treatment has a high effect in increasing the tensile strength of kenaf bast fibers compared to the other factors, which has the contribution of 40.19 %. The other factors, such as immersion time, gives an account of 33.58%, followed by drying temperature (21.39%) and immersion temperature (4.85%).
2. The optimum parameters for each factor that affecting the tensile strength are 6% wt. NaOH, 1 hour immersion, the room temperature immersion and drying temperature of 60 deg. Celcius.
3. The Fourier Transform Infrared Spectroscopy showed that the cristallinity index of kenaf immersed at room temperature (3.52) is higher than that at 100°C (3.08) which support the tensile strength result, because the higher cristallinity index, the higher its tensile strength.

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