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Two Parameters Weibull Analysis on Mechanical Properties of Kenaf Fiber under Various Conditions of Alkali Treatment

Mohd Yussni Hashim*, Azriszul Mohd Amin, Omar Mohd Faizan Marwah, Mohd Hilmi Othman, Nurnajwa Hani Hanizan, Mohd Khairul Efni Norman

Faculty of Mechanical & Manufacturing Engineering,
Universiti Tun Hussein Onn Malaysia, Batu Pahat, 86400, MALAYSIA

*Corresponding Author

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Abstract: This paper present two parameters Weibull analysis result on mechanical properties of kenaf fiber under different condition of alkali treatment. The mechanical properties of kenaf fiber that as focused in this study was fiber matrix interfacial shear strength (IFSS) and fiber tensile strength (TS). Kenaf fiber were treated under various conditions alkali concentration at 2, 6 and 10 (w/v%), immersion duration at 30, 240 and 480 minute and immersion temperature at 27, 60 and 100oC. Unsaturated polyester matrix was used in this study to determine the interfacial shear strength (IFSS). The result shows that Weibull modulus of kenaf fiber interfacial shear strength (IFSS) at 30 minutes immersion duration value was 2.59 to 3.12 with characteristic strength, σo-IFSS value range was 0.29MPa to 0.37MPa. The highest Weibull modulus was at room temperature and 6% alkali concentration. For kenaf fiber tensile strength Weibull modulus, the range was 2.40 to 3.07 with characteristic strength value range from 345MPa to 597MPa. The highest Weibull modulus also was measured at room temperature and 2% alkali concentration. The characteristic strength value shows a degrading pattern with the increment on immersion temperature and alkali solution concentration.

Keywords: Kenaf fiber, Weibull analysis, fiber matrix interfacial shear strength (IFSS), fiber tensile strength (TS).

1. Introduction

In the recent years, triggered by the environmental consciousness increased, there were actively attempt to reduce the dependence on petroleum based fuels and products in industries[1-2]. This leads to the need to investigate environmentally friendly and sustainable materials to replace the existing synthetic fibers One of the challenges in exploring natural fiber application in polymer matrix composite is the variability of fiber strength. The scatter of the tensile strength of brittle fibers is caused primarily by the presence of mechanical defects of random size and by the geometrical variability of their transverse dimensions. Unlike synthetic fiber, natural fiber exhibit large variation between fiber and within fiber diameter. Differences in natural fiber tensile strength character could be generated from fiber geometrical shape, morphological structure and fiber fracture mechanism[3]. According to Yuping and Xungai, fiber flaws (morphological defects) and fiber diameter variations affect the tensile behaviour, particularly for non-uniform fibers. The weakest point in a fiber could be where there is an internal flaw or where fiber diameter is small or a combination of both. If this weakest point reaches its breaking limit, then the whole fiber breaks [4]. It also found that that the strength of fiber is mainly dominated by the elemental composition of material and sizing on fiber surface, which functioning as "backbone" and " clothing " for the filament, respectively [5].

Application of Weibull statistic on the tensile strength of treated flax fiber was studied by Zafeiropoulos *et al.* [6]. Tensile test was conducted at three different gauge lengths with 1.0 mm/min cross head speed. Diameters of the fiber were measured with transmission light optical microscope and were considered as perfectly round for simplicity

*Corresponding author: yusni@uthm.edu.my

reasons. The probability of failure was estimated using four different probability index or estimators as presented in Table 1. Two methods were used to estimate the parameter of Weibull distribution, which are linear regression method and maximum likelihood method. It was found that the maximum likelihood and the linear regression methods delivered similar estimations for Weibull modulus, $\boldsymbol{m}$ and characteristic strength, $\boldsymbol{\sigma_o}$ values. Defoirdt *et al.* [7] examined the tensile properties of coir, bamboo and jute fiber following the normal and Weibull distribution. They found that the calculated Weibull modulus was differing significantly for the various test length. Man-made fibers usually have Weibull modulus between 5 and 15, while for natural fiber that have a larger variation in their properties the values vary mostly between 1 and 6.

Table 1 - Probability index or estimators, P_f [6]

Probability index or estimators, P_f	Estimators name
$P_{fi} = \frac{i-0.5}{n}$	-
$P_{fi} = \frac{i}{n+1}$	Mean ranks
$P_{fi} = \frac{i-0.3}{n+0.4}$	Median ranks
$P_{fi} = \frac{i-3/8}{n+0.25}$	-

Another studied about effect of fiber morphology on the tensile strength of natural fiber was performed by Fidelis *et al.* [3]. Jute, sisal, curaua, coir and piassava fibers were tested under direct tension in a universal testing machine. Weibull statistical analysis was used to quantify the variability of fiber strength. The mean rank estimator was used to calculate probability index. Experiment results shows that sisal fibers presented the highest Weibull modulus at 3.70, whereas the curaua fiber presented the lowest one at 2.22. This indicated that the sisal fiber had the lowest variability and curaua fiber is the highest. Guo *et al.* [8] investigated the tensile behavior characteristics of palm fiber through tensile experiments. Two parameter, three parameter Weibull models and modified model were employed to perform statistical analysis on fiber strength. Tensile test was conducted at four different gauge length at fixed cross head setting value. The result shows that Weibull modulus decreased as the testing length increased in accordance with the gradual increase in the scatter of fiber strength because of the discrete distribution of fiber flaws. The tensile strength of palm fiber can be predicted by modified model accurately. Table 2 showed the Weibull modulus value that was found by others researchers previously. The Weibull modulus range was found to be 1.96 to 8.56. It was slightly bigger than Weibull modulus range value for natural fiber that stated by Defoirdt *et al.* [7] which are 1 to 6.

The tensile strength of aloevera fibers was increased after the alkali treatment as reported by Balaji [9]. The mechanical properties are improved, due to partial removal of hemicellulose and formation of new hydrogen bonds between the cellulose fibrils along the fiber. The range of Weibull modulus tensile strength range was 3.22-3.33. Recent study by Senthamaraikannan and Kathiresan [10] shows the Weibull distribution plot for tensile strength of untreated and alkali treated *Coccinia Grandis .L Fibers*. The results indicate that Weibull modulus tensile strength range was 10.18 to 13.07. Furthermore, Tarress *et al.*[11] found that Henequen strands show higher cellulose content and intrinsic tensile strength in the range of 500 MPa. They create a relationship between the length of the reinforcements and its characteristic strength. Henequen strands, with a mean length of 485.04 µm, showed a theoretical Weibull characteristic strength of 450 MPa.

The interface bonding from the chemical treatment of the reinforced fibers can be seen as a great factor that affecting the mechanical properties of fiber composite. The alkali treatment serves as a method to increase the surface roughness that are resulting in better mechanical interlocking and increases the amount of cellulose exposed on the fiber surface[12]. In the present work, alkali treatment was selected as a kenaf fiber treatment method and the experimental work set up was continued from previous study reported by Yussni *et. al* [13]. This study aims to conduct two parameters Weibull analysis on mechanical properties of kenaf fiber under various conditions of alkali treatment. The selected mechanical properties were kenaf fiber matrix interfacial shear strength (IFSS) and kenaf fiber tensile strength (TS).

2. Experimental work

2.1 Kenaf fiber & alkali treatment

Bast type kenaf fibers was supplied from Kenaf Natural Fiber Industries (Malaysia) Sdn.Bhd. Sodium hydroxide (NaOH) used in this studied was supply by BDH Prolabo (UK) in 1 kg container at small pallet shape. Alkali solution concentration was prepared using weight per volume percentage (w/v %). Alkali treatment setting is according to three factors at three levels full factorial design matrix as shown in Table 3. Once treatment process is finished, kenaf fiber was taken out from container and washed in running tab water. A drop of acetic acid was dropped into a beaker that contained washed kenaf fiber to remove any excessive sodium hydroxide. Alkali treated kenaf fiber were conditioned at 100oC ~105oC for one hour in high temperature steamer for fiber drying process.

Table 2 - Weibull modulus range for natural fiber tensile strength variability

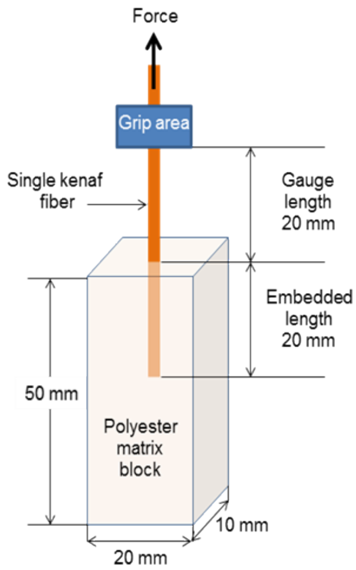
Fiber	Weibull modulus (m)	Finding by researchers	Year/ References
Flax	1.96 - 4.23	Calculated Weibull modulus and characteristic strength values are different for different gauge lengths used of the same fiber grade	2007[6]
Coir Bamboo Jute	5.8 - 6.0 3.5- 9.3 2.7 - 4.6	Strength decreases as the test length increases due to more flaws in the fiber that make the chance of failure larger	2010[7]
Caraua Jute Coir Piassava Sisal	2.22 2.74 2.74 3.68 3.70	- Sisal fiber show the lowest variability whereas the curaua presented the highest one variability - Thus, the curaua fiber has a larger probability of failure when submitted to similar tensile stresses	2013[3]
Palm	5.42 - 8.56	- Weibull modulus decreases as the gauge length increases - This demonstrates that longer gauge length could result in greater strength scatter	2014[8]
Saharan aloe vera	3.22-3.33	Partial removal of hemicellulose and formation of new hydrogen bonds between the cellulose fibrils increase fiber tensile strength	2017[9]
Coccinia grandis	10.18 to 13.07	Tensile test results of all trials were situated inside the line and fit perfectly the Weibull distribution	2018[10]
Henequen	2.60 - 2.88	Henequen fibers show a high cellulose content that allows obtaining strong interfaces when a coupling agent is added to the composite formulation	2019[11]

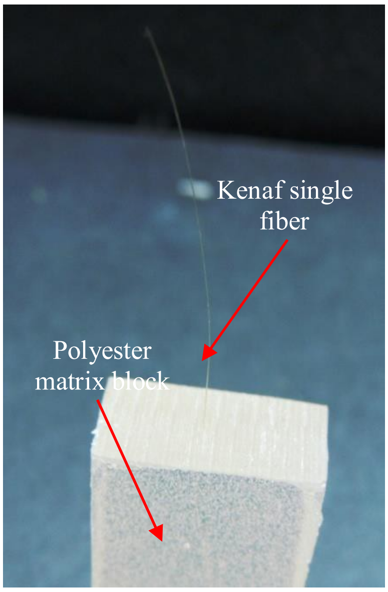
Table 3 - Independent variables matrix of alkali treatment conditions for kenaf fiber physical properties evaluation

Independent variables	**Actual variables value**		
	Low	**Center**	**High**
X_1: Alkali concentration (w/v%)	2	6	10
X_2: Immersion time (min)	30	240	480
X_3: Immersion temperature (±1°C)	27	60	100

2.2 Pull out specimen preparation

Fiber pull out test was used to identify kenaf fiber-polyester matrix interfacial shear strength [14]. Silicon was used to fabricate pull out test specimen mould. Razor blade was used to slice silicon mould at the ending area of each pattern for placing the single kenaf fiber. Silicon release agent was spray all over the silicon mould before single kenaf fiber was carefully inserted into sliced area until 20 mm length of the fiber are embedded into the mould. Needle was used to ensure embedded kenaf fiber is at straight position during curing process. Polyester matrix is prepared after all kenaf fiber was successfully inserted into silicon mould. Unsaturated polyester (UP) resin was added with 1% Methyl Ethyl Ketone Peroxide (MEKP) under constant mixing duration and speed in all pull out test specimen preparation. Then resin mixture was cast into the mould. After casting process was done, the mould was left over night to be cured at room temperature. The schematic diagram and actual kenaf fiber polyester matrix pull out specimen is show in Figure 1.





(a) Schematic diagram (b) Pull out test specimen

Fig. 1 - Schematic diagram and kenaf fiber polyester matrix pull out test specimen

2.3 Pull out test

Lloyd Universal Testing machine with 10N load cell was use for kenaf fiber polyester matrix pull out test to determine the interfacial shear strength. The free end of kenaf fiber gripped and the load was applied to pull the fiber out of matrix. The corresponding interfacial shear strength can be calculated from the force at which debonding occurs using the following Kelly/Tyson Equation 4.2 [15-16]:

$$\tau = \frac{\boldsymbol{F}}{\pi D l} \tag{2.1}$$

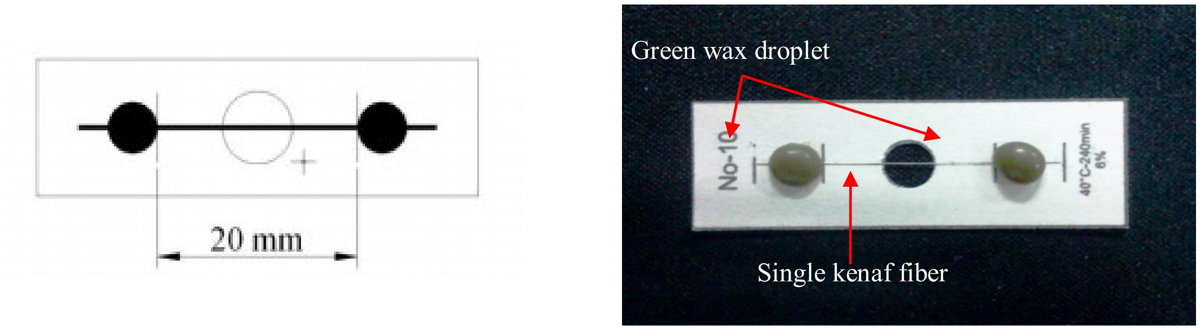
where $\boldsymbol{\tau}$ is interfacial shear strength, $\boldsymbol{F}$ is force required to pull out or break the fiber, D is kenaf fiber average diameter and l is embedded kenaf fiber length in polyester matrix. In this test, a crosshead speed of 1.0 mm/min was set as a default speed. Gauge length was set at 20 mm. Kenaf fiber diameter average value for each alkali treatment conditions was used as kenaf diameter value with assumption the fibers were considered perfectly round. Seven specimens were tested at each treatment conditions and average value was measured.

2.4 Fiber cross-sectional area

Kenaf fiber tensile test specimen mounting tab was prepared according to ASTM C1557-03 for each treatment conditions [17]. This test method was selected instead of ASTM D3379-75 because it was more suitable single fiber and ASTM D3379-75 was designed for high modulus single filament materials. Kenaf fibers were made into specimens by bonding them to paper tabs, which acted as frames to hold fibers, using a droplet of green wax. The nominal gauge length, which is the length between two wax droplets, was 20 mm. Figures 2 show a schematic of test specimen (a), and a specimen mounted on a paper tab as a frame for the grip and test assembly (b).

2.5 Fiber tensile test

The test specimens mounted on paper tab were installed in Lloyd Universal Testing machine with 10N load cell. Once the paper tab was clamp properly, it was cut away by a scissors at both sides leaving the fiber as an only source of tensile load resistance. The average value of kenaf fiber cross sectional area for each alkali treatment conditions was used as kenaf cross sectional area value with assumption the fibers were considered in ellipse shape. Testing was conducted at a constant crosshead speed at a rate of 1.0 mm/min and the gauge length was 20 mm. For every alkali treatment conditions, tensile test was repeated for seven times and its average value was measured. Samples that broke near the edge of the clamps were excluded from the analysis and the test was repeated again to obtain the specimen that only broke at the center area.



(a) Schematic tensile test specimen (b) Specimen mounted on paper tab

Fig. 2 - Tensile test specimen preparation

2.6 Weibull analysis calculation

In this study, the following Weibull distribution function was used to determine the variability of fiber strength resulting from a distribution of flaw size along the fiber. The Weibull distribution can be written as;

$$(\sigma)_f = - \boldsymbol{exp}\left\{-\frac{L}{L_0}\left(\frac{\sigma-\sigma_u}{\sigma_o}\right)^m\right\} \quad (2.1)$$

where $P_f(\sigma)$ is the probability of failure of a fiber of length L at an applied stress σ, L_o is the length of elementary unit of the fiber, σ_u is the lowest value of strength normally set to zero; σ_o is the characteristic strength and m is the shape parameter called Weibull modulus. If values of m are high, it shows less variability in strengths[18]. Assuming L_o is set to unity and $\sigma_u = 0$, the equation 3.15 can be simplified to a two parameter Weibull equation where L becomes dimensionless as equation 3.16. This equation can be transform into linear regression for solving the Weibull modulus, m value.

$$P_f(\sigma) = 1 - \boldsymbol{exp}\left\{-L\left(\frac{\sigma}{\sigma_o}\right)\right\} \quad (2.2)$$

$$\ln\left[\ln\left(\frac{1}{1-P_f(\sigma)}\right)\right] = m\ln(\sigma) - m\ln(\sigma_o) + (L) \quad (2.3)$$

Setting $\ln\left[\ln\left(\frac{1}{1-P_f(\sigma)}\right)\right]$ against $\ln(\sigma)$ gives a straight line with a slope m. σ_o is deduced from the y-intercept; $ln(L) - mln(\sigma_o)$. σ_n values are the experimental values which are ordered as $\sigma_1 < \sigma_2 < ... < \sigma_i ... < \sigma_n$, where n is the size of the sample and i is the rank of each data point. σ_i is assigned probability of P_{f1} such that $P_{f1} < P_{f2} ... < P_{fi} ... < P_{f1}$, where $0 < P_{fi} < 1$. Since the true value of P_{fi} is unknown, estimator is used so that on average, the errors arising from estimation compensate each other. In this study median rank estimator as shown in Table 1 was selected as an estimator [19].

3. Results and discussion

The result of Weibull plot for kenaf fiber interfacial shear strength (IFSS) and kenaf fiber tensile strength (TS) after various alkali treatment conditions was presented in Table 4. The mechanical properties of kenaf fiber are analyzed with a two-parameter Weibull distribution tool on Minitab17 software. The probability distribution plot of data collected from 30 trials of kenaf fiber interfacial shear strength (IFSS) and kenaf fiber tensile strength (TS) after various alkali treatment conditions was presented in Table 4. The plot helps to estimates the statistical parameters like shape and scale factors of distribution and also supports to confirm the integrity of acquired data with Weibull distribution[20]. The shape factor verifies consistency of measured data and its intensity is proportional to the ratio of mean value to standard deviation; comparatively less degree of deviation indicates quite appreciable distribution. At the first column of Table 1, it shows the Weibull analyses, which are Weibull modulus, m and characteristic strength, σ_o. The rest of the columns present the alkali treatment conditions and the measured responses value. Weibull modulus and characteristic strength for untreated kenaf fiber interfacial shear strength (IFSS) and tensile strength (TS) was $m_{IFSS} = 2.85$, $\sigma_{o\text{-}IFSS} = 0.36$ MPa and $m_{TS} = 3.11$, $\sigma_{o\text{-}TS} = 434.69$ (MPa) respectively. The Weibull modulus and characteristic strength for untreated and alkali treated kenaf fiber was difficult to compared due to fluctuation in experimental results.

From Table 4, for kenaf fiber interfacial shear strength at 30 minutes immersion duration, the range of Weibull modulus value was 2.59 to 3.12 with characteristic strength value range from 0.29MPa to 0.37MPa. The highest Weibull modulus was at room temperature and 6% alkali concentration. For kenaf fiber tensile strength Weibull modulus, the range was 2.40 to 3.07 with characteristic strength value range from 345MPa to 597MPa. The highest Weibull modulus also was measured at room temperature and 2% alkali concentration. The characteristic strength value shows a degrading pattern with the increment on immersion temperature and alkali solution concentration. At 240 minutes immersion time, Weibull modulus of kenaf fiber interfacial strength range was 2.73 to 2.86 with characteristic strength range at 0.32 to 0.36MPa. On the other hand, kenaf fiber tensile strength Weibull modulus range was 2.55 to 3.11 with characteristic strength range from 314MPa to 548MPa. Similar degrading pattern was observed for characteristic strength behavior changes of kenaf fiber tensile strength. At longer immersion time, the range of kenaf fiber interfacial shear strength Weibull modulus was 2.73 to 2.86 with characteristic strength range from 0.25MPa to 0.35MPa.

Table 4 - Weibull analysis of kenaf fiber IFSS and tensile strength at various alkali treatment conditions

Weibull analysis	Time (min)	Temperature Level (ºC)								
		Room temperature (±27)			60			100		
		Alkali Concentration (w/v %)								
		2	6	10	2	6	10	2	6	10
m_{IFSS}	30	2.59	3.12	2.89	2.62	2.79	2.77	3.02	3.10	2.95
$\sigma_{o\text{-}IFSS}$		0.35	0.37	0.33	0.33	0.36	0.35	0.32	0.29	0.31
m_{TS}		3.07	2.86	2.76	2.64	2.56	2.52	2.53	2.45	2.40
$\sigma_{o\text{-}TS}$		597	489	364	578	413	351	546	462	345
m_{IFSS}	240	2.77	2.80	2.86	2.75	2.73	2.76	2.81	2.86	2.74
$\sigma_{o\text{-}IFSS}$		0.34	0.35	0.33	0.35	0.36	0.34	0.34	0.32	0.33
m_{TS}		3.11	2.89	2.82	2.73	2.63	2.68	2.69	2.61	2.55
$\sigma_{o\text{-}TS}$		548	447	386	539	409	340	404	370	314
m_{IFSS}	480	2.73	2.79	2.81	2.82	2.74	2.81	2.87	2.86	2.74
$\sigma_{o\text{-}IFSS}$		0.31	0.28	0.29	0.32	0.31	0.32	0.27	0.28	0.25
m_{TS}		3.02	2.85	2.87	2.78	2.73	2.54	2.66	2.63	2.52
$\sigma_{o\text{-}TS}$		428	382	353	379	358	342	412	326	312

Notes :

m_{IFSS} : Weibull shape parameter @ Weibull modulus for interfacial shear strength (IFSS)

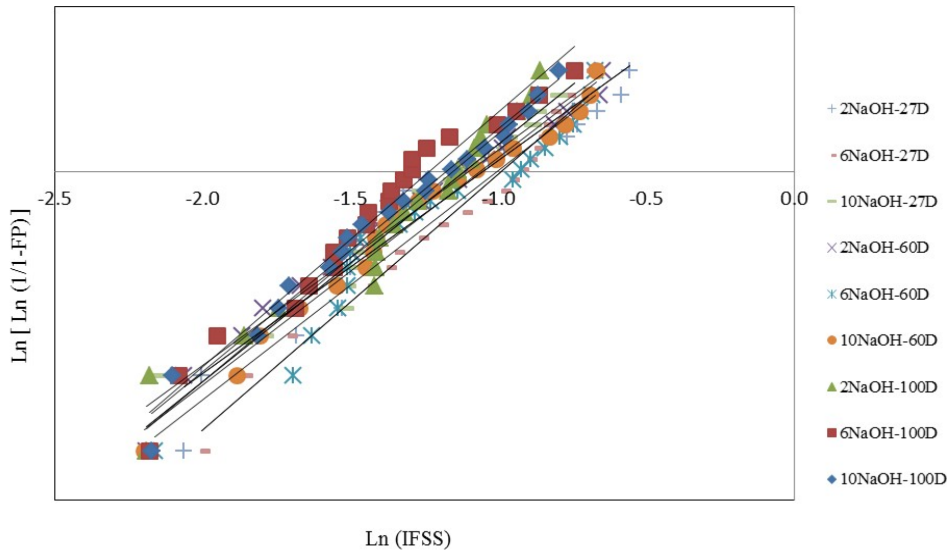
$\sigma_{o\text{-}IFSS}$: Weibull scale parameter @ characteristic strength for interfacial shear strength (IFSS)

m_{TS} : Weibull shape parameter @ Weibull modulus for tensile strength (TS)

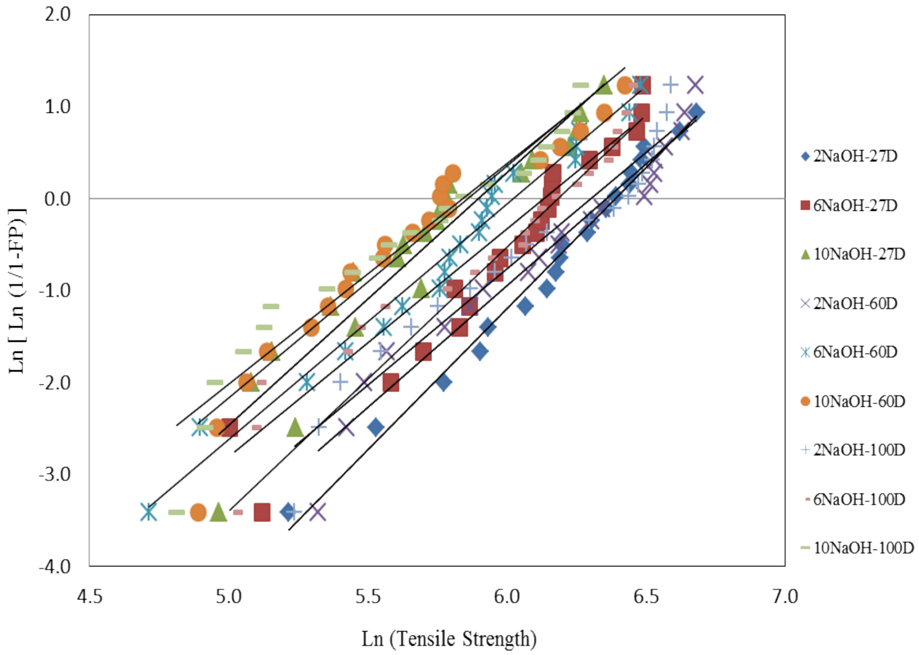
$\sigma_{o\text{-}TS}$: Weibull scale parameter @ characteristic strength for tensile strength (TS)

For kenaf fiber tensile strength Weibull modulus, the range was 2.52 to 3.02 with characteristic strength value range from 312MPa to 428MPa. The characteristic strength value also shows a degrading pattern with the increment on immersion temperature and alkali solution concentration.

Figure 3 shows an example of Weibull plot for kenaf fiber interfacial shear strength and kenaf fiber tensile strength treated with alkali at constant time (30minute) over different NaOH concentration and immersion temperature. Weibull modulus of interfacial shear strength was close to each other at all treatment conditions compared to fiber tensile strength. This probably due to the less variant measured in interfacial shear strength data. From Weibull plot in Figure 3, all the coefficient of determination or R^2 value was above 90%. The studied result shows that kenaf fiber with various alkali treatment process exhibit different Weibull modulus and characteristic strength at each condition. The Weibull modulus range for all treatment process was 2.40 to 3.12. The low Weibull modulus values indicate a high scattering in fiber strength, which probably due to the fiber non-homogeneous. A fiber with a large cross-sectional area has a higher potential of having much flaw than a fiber with a small cross-sectional area [21]. Similar results regarding Weibull modulus and characteristic strength range was presented by Du *et al.* [22].



(a) Ln [ln(1/1-FP)] vs Ln (IFSS)



(b) Ln [ln(1/1-FP)] vs Ln (IFSS)

Fig. 3 - The example of Weibull plot for (a) kenaf fiber interfacial shear strength (b) kenaf fiber tensile strength treated with alkali at constant time (30 minute) over different NaOH concentration and immersion temperature

4. Conclusion

Two parameters Weibull analysis on mechanical properties of kenaf fiber under various conditions of alkali treatment were investigated. Kenaf fiber interfacial shear strength and kenaf fiber tensile strength Weibull modulus range for all treatment conditions was found at 2.40 to 3.12 ranges. The highest Weibull modulus was measured at room temperature and 2% alkali concentration. The characteristic strength value shows a degrading pattern with the increment on immersion temperature and alkali solution concentration.

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