



Study on the strength of oil palm fiber reinforced composite material

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Chapter 1

Introduction

1.1 Background of study

A fiber reinforced polymer (FRP) is a composite material made by combining a polymer matrix (either thermoplastics or thermosettings) together with fibers such as glass, carbon, basalt and aramid. The most common thermoplastic materials used for natural fiber composites are poly (lactic acid) (PLA), polypropylene (PP), nylon, polyvinyl chloride (PCV) and polyethylene. While thermosetting matrices such as polyester epoxy, phenolic and polyurethane are also widely used to fabricate natural composite [1].

In the recent years, the concept of “all green composite” has attracted the attention of many scientists and engineers due to that composite give a combination of superior mechanical performance and environmental advantages such as renewability, bio-degradability and recyclability [2]. All green composite or natural bio-composite is a composite material consists of polymer and reinforcements from renewable resources. One of the renewable polymer is poly(lactic acid) (PLA). In general, the composition and structures of polymer chains of PLA (mainly refer to the ratio of L-isomer and D-isomer of lactic acid) can affect the crystallization and degradation behaviour of PLA (Figure 1.1) [3-4]. PLA is a biodegradable polymer with mass capacity up to 140,000 tons per year [5]. Despite to the advantages, PLA has low mechanical performances, and this has limited PLA usage on other applications instead of structural components and packaging. Therefore, the research interest on producing PLA-based materials will be more focused on improving PLA toughness by incorporated with natural fibers [6-15].

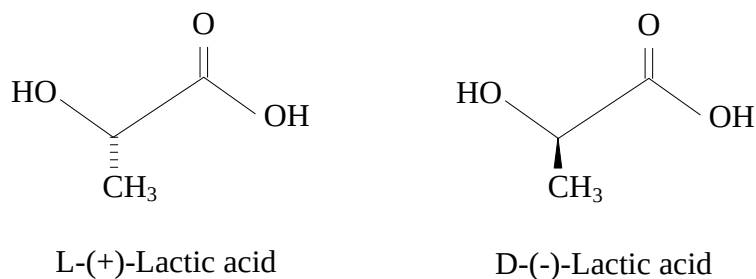


Figure 1.1 Optical isomers of lactic acid.

On the other hand, the mechanical strengths and modulus, low density, low cost, non-irritation to skin, less health risk, biodegradability and renewability of the natural fibers over the conventional fibers (glass and carbon fibers) lead them as attractive candidates for reinforcements in natural fiber composites. The mentioned natural fibers include flax [7-8, 16-17], hemp [11, 18-20], jute [14, 21], oil palm [12, 22-25], sisal [26-28], ramie [29, 30], coir [31-33], bamboo [34-36] and many others. These composite materials are potentially used in packaging, leisure, sport and electronics industries. However, the hydrophilic nature of the natural fibers resulted by the presence of hemicellulose and lignin in natural fibers can weaken the bonding between fibers and matrix. Hence, this may reduce the efficiency of the stress transfer from matrix to the fibers [37].

1.2 Oil palm fiber

Oil palm (*Elaeis guineensis*) is the most efficient oilseed crop and it cultivated on approximately 15 million ha across the world [38]. Malaysia and Indonesia are the major oil palm cultivating countries, where produce 85% of the world's oil palm. According to Hassan and Soom [39] report, 55 ton of fibrous biomass can be produced annually in 1 ha of oil palm plantation while yielding 5.5 ton of palm oil and palm kernel oil. Lignocellulosic fibers can be retting from the frond, trunk, fruit mesocarp and empty fruit bunch [40]. The empty fruit bunches are more preferred among all the fiber sources because empty fruit bunches are able to yield up to 73% of the fibers [41-42].

Oil palm empty fruit bunch fiber, in short also known as oil palm fiber (OPF), is one of the natural fibers which obtained from the empty fruit bunch (EFB) (Figure 2). Some researchers reported that the OPF is tough and has some similarity in structures compared with coir fibers [40, 43-44]. Table 1.1 listed the chemical composition of OPF and the schematic illustration for the structure of hemicellulose, cellulose and lignin is presented in Figure 1.3 [45]. As mentioned above, these non-cellulosic materials on the surface of OPF are weakening the adhesion with polymer during the fabrication process. Therefore, surface treatment on the OPF is necessary to enhance the interfacial bonding between fiber and matrix.

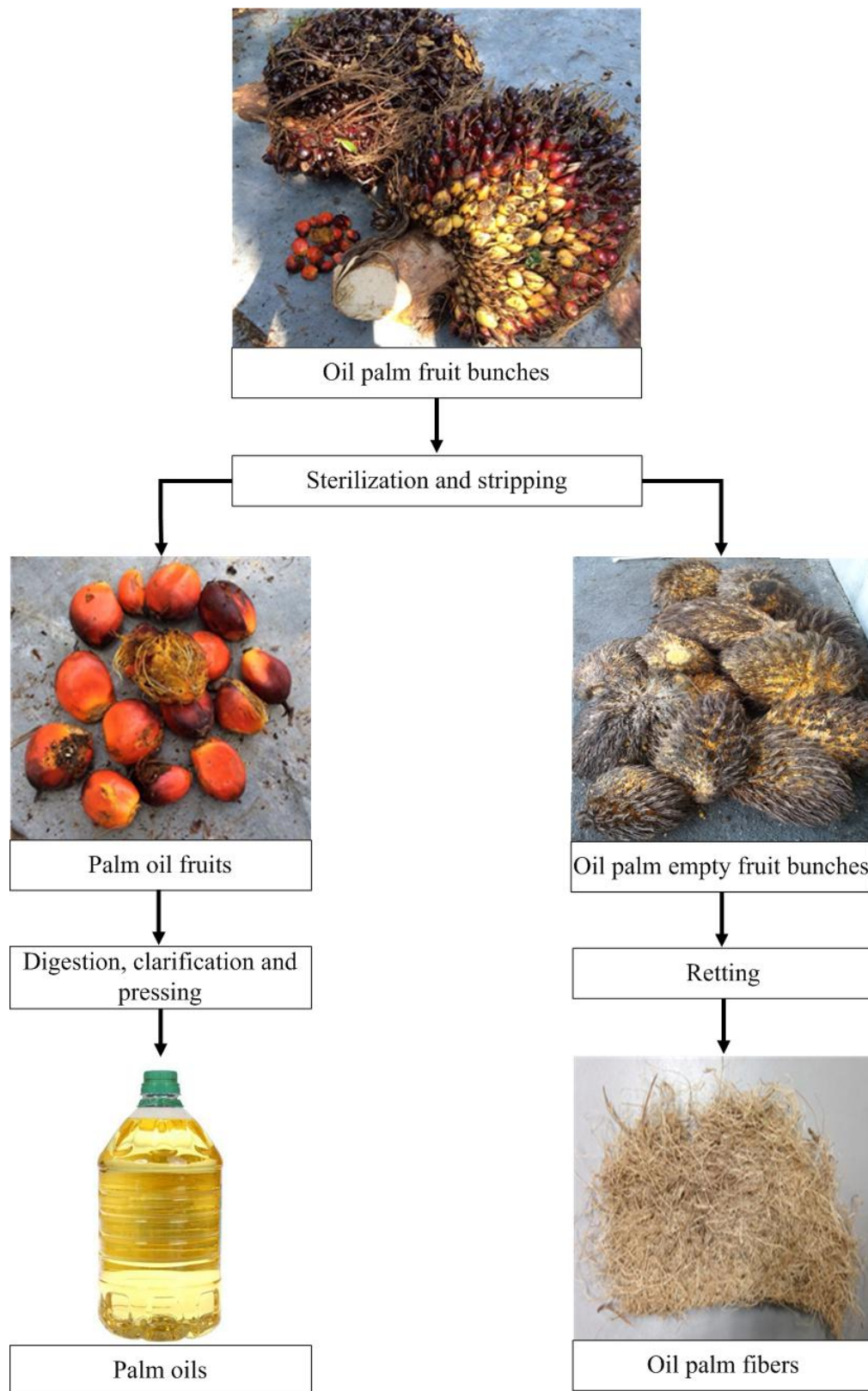
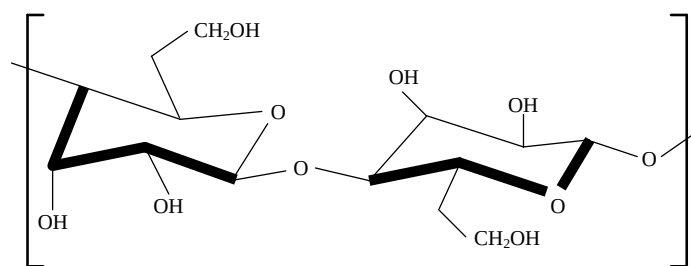
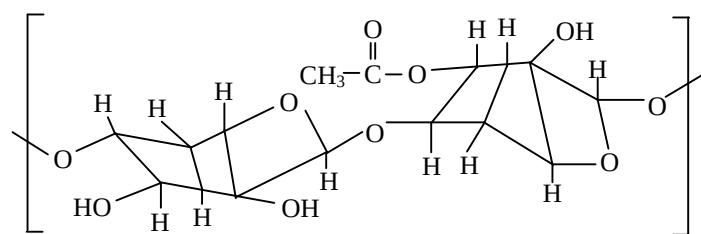


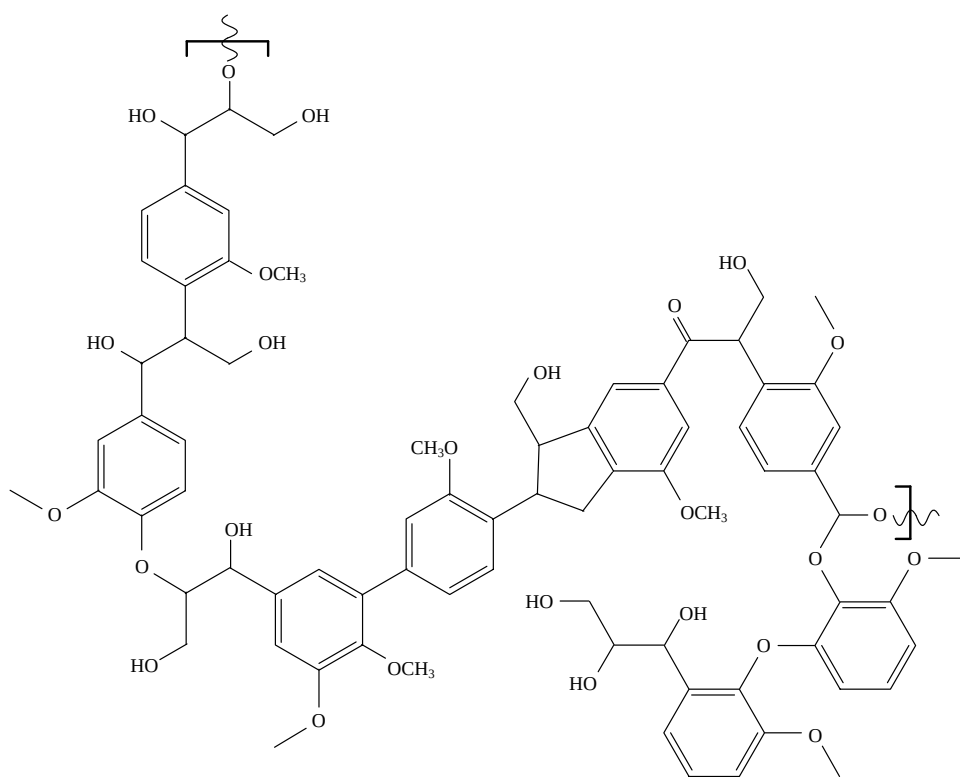
Figure 1.2 Simplified process diagram of oil palm.



(a) Cellulose



(b) Hemicellulose



(c) Lignin

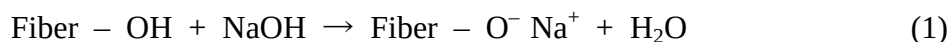
Figure 1.3 Schematics illustration for structural representations of cellulose, hemicellulose and lignin [45].

Table 1.1 Chemical composition of OPF [44].

Composition	Chemical composition (%)
Hemicellulose	17-33
Cellulose	43-65
Lignin	13-37

1.3 Alkali Treatment

Alkali treatment is one of the surface treatments on natural fiber when the fibers were used to fabricate natural fiber composites. Alkali treatment can disrupt hydrogen bonding in the chemical structures of natural fiber and increasing the roughness of the fiber surface as well. Through the NaOH treatment, the lignin, wax, pectin, oils and hemicellulose on the fiber surface can be removed and the amount of -OH groups exposed will be increased [46-48]:



Alkali treatment has been used on different types of natural fibers such as oil palm, jute, hemp, sisal, bamboo, flax, banana, coir and kenaf. Gassan and Bledzki [48] was immersed the jute fiber with up to 28% alkali solution and they have reported that adhesion between the natural fibers and matrix does affect the tensile performance of the composite. They also observed that the tensile strength and tensile modulus were increased by 120% and 150% respectively. Moshiul Alam *et al.* [12] reported that the NaOH treatment on the fiber was gave up to 7% and the increment in tensile properties of the OPF reinforced PLA composites were found in their study. These researchers concluded that the NaOH treatment on the fibers can enhances the roughness of the fiber surface and provide a larger contact area to interact with matrix in fiber reinforced polymer. As a result, it improves the mechanical interlocking between fiber and matrix.

Shukor *et al.* [13] conducted a research to investigate the effect of NaOH concentration (3%, 6% and 9%) for treating kenaf fiber reinforced PLA biocomposites. They found that 6% alkali treatment at ambient temperature is the optimum condition to obtain maximum flexural modulus and impact strength of the composite. Roy *et al.* [49] also treated jute fibers with 0.25% to 1.0% alkali solution for 30 minutes up to 48 hours at room temperature and reported that the ultimate tensile strength can be obtained from soaking fibers with 0.5% NaOH

treatment for 24 hours. Many researchers also reported that the tensile properties of the fiber and its composite decreased when the fiber treated with very high alkali concentration and prolonged treatment period [50-51]. This may due the damage of the main structures of the fibers.

1.4 Weibull distribution

Weibull distribution [52], named after Waloddi Weibull, is a continuous probability distribution which can be used to solve broad range of problems. Based on weakest link theory, Weibull suggested a simple distribution of material strength x :

$$F = 1 - \exp \left[-n \left(\frac{x}{x_0} \right)^m \right] = 1 - \exp \left[-\frac{V}{V_0} \left(\frac{x}{x_0} \right)^m \right] \quad (2)$$

where F is a failure probability of a long fiber connected by n independent segments, x is the fiber strength, V is the fiber volume, V_0 is a reference volume, m is Weibull modulus and x_0 is scale parameter.

Recently, Weibull distribution was used to analyse the single fiber test results of natural fiber due to high disparity of fiber strengths. This statistical approach has been used to analysis the strength of jute [48], sisal [53], flax [54], okra [55], and abaca [56] but there is no related statistical analysis applied on OPF. Therefore, alkali treatment and the gauge length effect on the OPF by utilization of the Weibull distribution model will be carried out and explained in Chapter 2 and 3 respectively.

1.5 Fracture behaviour in FRP composites

FRP composites have played important role in various applications for their high specific strength and modulus. Recently, renewal of interest in the research of fibers derived from naturally sustainable sources as potential reinforcement for high performance composites has been growing. However, many studies reported that the weak interfacial bonding ability between fiber and matrix can decrease the mechanical performances of natural fibers reinforced composites [11-12, 57]. Therefore, there is an interest in developing test methods to study the fracture behaviour of the composite, especially on the interfacial fracture

toughness and interfacial fracture toughness, which influence the macroscopic properties of the composite materials.

The recent evaluation on crack propagation behaviour of FRP was based on meso-mechanical analysis. The common test methods used to quantify the interfacial fracture between fiber and matrix are Microbond test [58-60], Broutman test [61], fiber push-out [62] and fiber pull-out test [63]. The crack propagation in FRP was evaluated from fracture process like matrix fracture and fiber/matrix interfacial cracking [64-66]. Kotaki *et al.* [67] and Hojo *et al.* [68] reported that the crack propagation behavior in FRP is strongly influenced by the interfacial bonding between fiber and matrix. Koiwa *et al.* [69] introduced the measurement on the interfacial fracture toughness of mode I and mode II crack growth by using real size model composite. They reported that a significant increase in fracture toughness with increasing bonding length. However, the results of fracture toughness were evaluated at very short range of the matrix lengths.

1.6 Aims and objectives of the research

This work presents contributions towards the improved mechanical performance, statistical analysis and interfacial fracture evaluation on OPF and its composite.

The aims of this research thesis are:

1. To determine the optimum surface treatment process of the oil palm fiber.
2. To investigate the statistical analysis on fiber strength measurement.
3. To improve the accuracy of the predicted fiber strength on the gauge length effect by modifying the Weibull model.
4. To evaluate the interfacial fracture toughness on the oil palm fiber/ poly (L-lactic) based on real model composites.
5. To investigate the alkali treatment effects on the interfacial fracture toughness and interfacial shear stress of the model composites.

1.7 Scope and originality of the research

As mentioned the above objectives, the scope of the present research is drawn as below:

1. Study on the alkali effect on the mechanical properties of the OPF

This experimental work used different alkali treatment conditions on the OPF. The optimum alkali treatment parameters are determined.

2. Study on the gauge length effect on the OPF by utilisation of Weibull model.

This study focuses on the fiber strength on different gauge lengths. Statistical approaches are utilised to predict the fiber strength. A new conical frustum volume variation has been proposed by this study and has been confirmed to improve the accuracy of the fiber predicted strength at different gauge lengths.

3. Study on the interfacial fracture behaviour of the OPF/ PLLA model composites.

This study is works on the interfacial fracture toughness and interfacial shear stress evaluation on the OPF/ PLLA model composites. This study is carried out since the adhesion between natural fiber and matrix play an important role for the mechanical performance of the natural fiber biocomposites.

The present works were simplified into a flowchart which illustrated in Figure 1.4. Furthermore, referring to the works from the present works and the literature information, there are three originalities can found in the present study. These originalities are listed as follow:

1. Limited previous works on the surface treatment of OPF. The various alkali treatment concentrations and treatment times on OPF tensile strength is the first work that carried out in the present study.
2. The modified Weibull model by integrating with conical frustum volume variation is an improved Weibull model done in the present study.
3. This present work also the first study on the interfacial fracture toughness and interfacial shear stress of the OPF/ PLLA model composites.

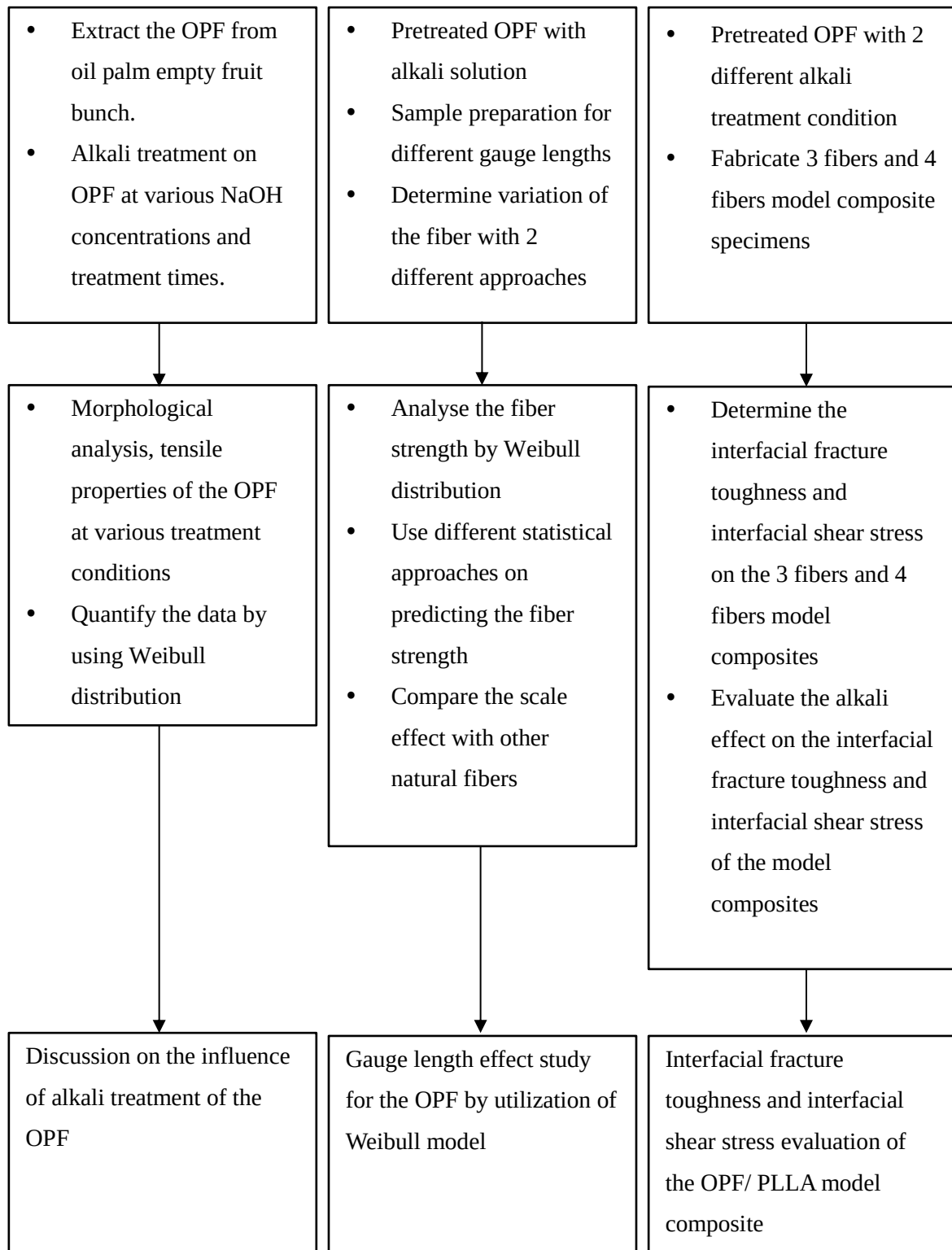


Figure 1.4 Flowchart of the present research.

1.8 Thesis Summary

Chapter 1: Introduction

A general review of the composite materials and the oil palm fiber are presented. The common surface treatment on natural fibers is also described. This chapter also included an introduction to the Weibull distribution analysis and the methods used to evaluate the fracture toughness of the composite materials.

Chapter 2: Influence of alkali treatment on the mechanical properties of oil palm fibers

The results of the surface morphology and mechanical characteristics of untreated and alkali treated fibers are presented and discussed.

Chapter 3: Statistical analysis of the tensile strength of treated oil palm fiber by utilisation of Weibull distribution model

In this chapter, an improved Weibull model is developed to predict the fiber the gauge length effect on the fiber strength. The experimental results of the fiber strength are compared with the conventional Weibull model, Zhang *et al.* [70] modified Weibull model and present improved Weibull model.

Chapter 4: Interfacial fracture toughness evaluation of poly(L-lactic acid)/ oil palm fiber composite by using double shear test method.

This chapter presents the results from the double shear test of three fibers model composite and four fibers model composite. In addition, this chapter includes an investigation of alkali effect on the interfacial fracture toughness and interfacial shear stress of the model composites.

Chapter 5: Conclusions

This chapter presents the conclusions of this thesis and summaries the contribution produced by this work.

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Chapter 2

Influence of alkali treatment on the mechanical properties of oil palm fibers

2.1 Introduction

As described in Chapter 1, alkali treatment is one of the surface treatments on natural fiber when the fibers were used to fabricate natural fiber composites [1-3]. The alkali treatment on fibers can remove the non-cellulosic materials from the fibers and compact the cellulose chains, resulting better mechanical properties of the fiber [4].

Alkali treatment effects on different lignocellulosic fibers properties such as jute [4], kenaf [5], banana [6], and coir [7] has been investigated. Gassan and Bledzki [4] reported that the optimum alkali treatment condition for jute fiber is 28% NaOH, 30 minutes soaking time. On the other hand, Mohd Edeerozey et al. [5] observed that 6% NaOH, 2 hours soaking time is the optimum treatment condition for kenaf fiber. This can be concluded that the optimum NaOH concentration and treatment time might be varying for the different types of natural fibers. Therefore, it is important for the present study to identify the optimum alkali treatment conditions for OPF.

The tensile strengths of natural fibers are highly variable. As a result, many researchers are focusing to model the large scattered tensile strengths of the natural fibers by using statistical approaches [7-10]. These researchers found that the utilisation of Weibull analysis on the tensile strength of natural fibers increase the predictive accuracy of the fiber strengths. Therefore, Weibull distribution should be considered when the strength of fiber is studied.

In this chapter, the diameter distributions along the fiber and cross-sectional surface of the fiber have been analysed. The effect of the alkali treatment on the mechanical properties of the oil palm fiber was also investigated. In addition, the Weibull distribution model was used to quantify the strengths of OPFs.

2.2 Experimental

2.2.1 Materials

Oil palm fibers were purchased from Ecofibre Technology Shn. Bhd, Malaysia. These untreated fibers were approximately 150–550 μ m in diameter and 40–150mm in length. Sodium Hydroxide (NaOH) was purchased in Hyogo, Japan.

2.2.2 Diameter distributions along fiber length

In general, natural fibers have non-uniform thickness. In order to measure the OPF diameters along its fiber length at different view angles, the specimen shown in Figure 2.1 was prepared. The micrograph of the fiber at every 1mm interval was captured by using a Nikon Eclipse ME600 microscope. When rotating both nuts, the micrograph of the fiber at the angles of 0°, 60° and 120° can be obtained. The diameters of the fibers were then measured by using an ImageJ software version 1.38x (National Institutes of Health, Maryland).

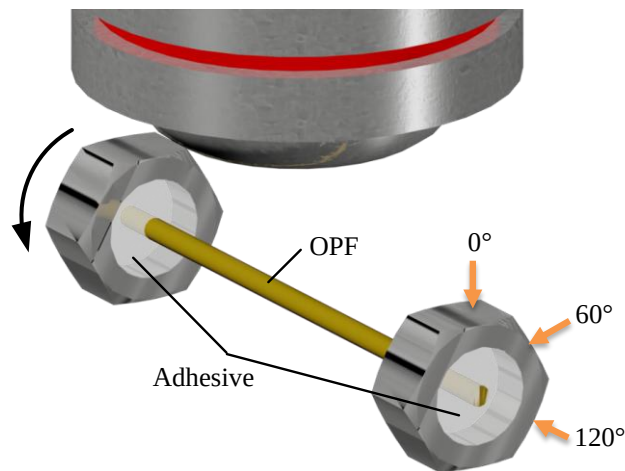


Figure 2.1 Schematic of the fiber diameter scanning at different view angles.

2.2.3 Analysis on the cross-sectional of the oil palm fibers

In order to measure the lumen area with-in fiber, the cross-sectional micrograph of the fiber was captured using a Hitachi TM3000 Scanning Electron Microscope. After that, the SEM image was converted to 8-bit grayscale and adjusted the brightness and contrast setting of the image until the lumens shown in Figure 2.2(a) is clearly seen. An ImageJ software version 1.38x was used to measure the total lumens area (Figure 2.2(a)) and the cross sectional area (Figure 2.2 (c)) of the fiber.

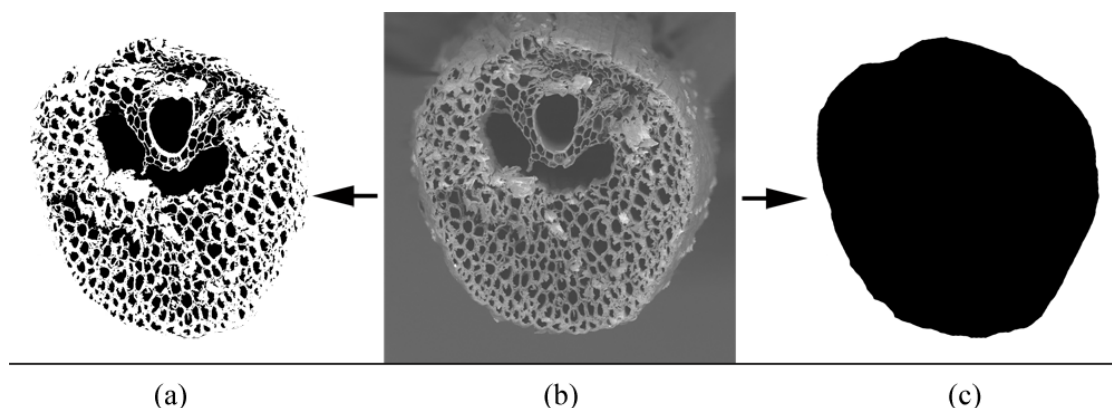


Figure 2.2 Fiber porosities measurements.

2.2.4 Alkali treatment

Oil palm fibers were kept in the oven at $21^{\circ}\text{C} \pm 1^{\circ}\text{C}$ for 24 hours for conditioning which accordance to ASTM D1776–04. The oil palm fibers were then immersed into 0.5, 1.0 and 1.5 M (mol/liter) sodium hydroxide for 0.5, 1, 2, 4, 8, 12, 24, and 48 hour(s). These treated fibers were then washed and rinsed with water for several times until the pH of the water is remaining at 7.0. After that, the fibers were dried at room temperature for 48 hours.

2.2.5 Fourier transformed infrared spectroscopy (FTIR)

Figure 2.3 shows the simple diagram of the FTIR test. The FTIR spectra of untreated and alkali treated fibers were analysed with a scanning range from 4000 cm^{-1} to 500 cm^{-1} by using a spectrometer (Jasco FT/IR-600 Plus) and the scan resolution was set to 32 cm^{-1} .

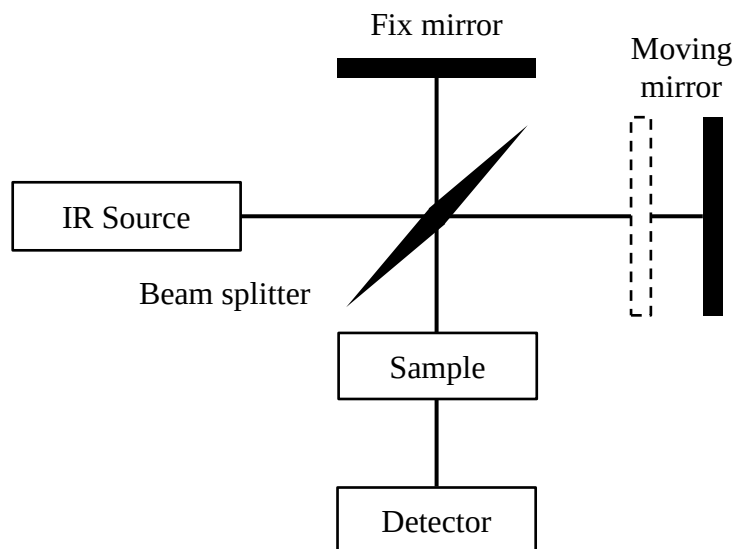


Figure 2.3 Simple diagram of the FTIR test.

2.2.6 Tensile test

The specimens were prepared according to the ASTM C1557-03. A fiber was mounted on a slotted cardboard tab as shown in Figure 2.4. Extreme care was taken to ensure that the fiber was aligned axially with the tensile direction. Cemedine EP001N adhesive was used to attach the fiber at opposite ends of the slot. The gauge length was determined by the fiber length between the adhesive spots. Once the glue spots were cured, the fibers were placed under a Nikon Eclipse ME600 microscope for diameter scanning. The fibers were considered perfectly round, and the fiber diameters were measured at 1mm intervals along its length. The uniaxial tensile strength tests were carried out by using a Tohei MT201 tensile test machine with the load capacity of 50N and the strain rate of 9mm/min. The values for the fiber fractures at or near the adhesive spots were discarded. Thirty sets of successful data were obtained for the data analysis.

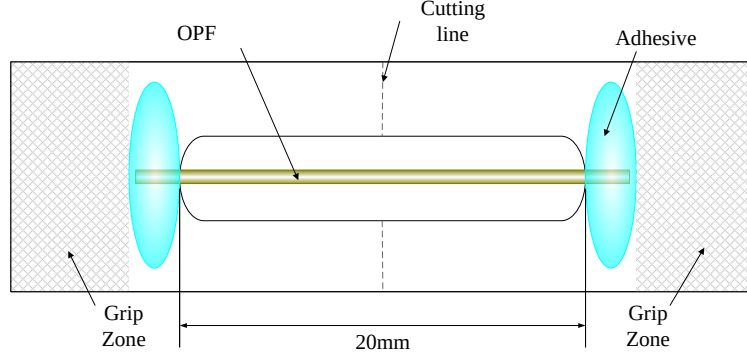


Figure 2.4 Schematic of the test specimen.

The experimental data obtained was analysed by using conventional Weibull distribution below:

$$F = 1 - \exp \left[-\frac{V}{V_0} \left(\frac{\sigma}{\sigma_0} \right)^m \right] \quad (1)$$

where F is the failure probability, V is the fiber volume, V_0 is the standard volume, σ is failure strength, σ_0 is scale parameter and m is Weibull modulus.

Survival probability (S), the specimen's fraction which will survive under the stress level, can be obtained from:

$$S = -\exp \left[-\frac{V}{V_0} \left(\frac{\sigma}{\sigma_0} \right)^m \right] \quad (2)$$

$$-\ln \ln \left(\frac{1}{S} \right) = -m \ln \sigma + m \ln \sigma_0 + \ln V - \ln V_0 \quad (3)$$

Plotting $-\ln \ln(1/S)$ versus $\ln \sigma$ is a straight line with slope where $-m$ can be obtained and σ_0 can be calculated from the value of intercept- y .

2.3 Results and discussion

2.3.1 Diameter and cross-sectional area analysis

Figure 2.5 illustrates the diameter distribution along fiber length at different view angles. The OPF diameters are varying approximately from $250\mu\text{m}$ to $510\mu\text{m}$. In order to simplify the fiber strength analysis, each fiber was considered as perfectly cylindrical. However, in

Chapter 3, the variation with-in fiber diameters will take into consideration to analyse the fiber strength distributions. As seen in Figure 2.6, the lumen area increases as increases in cross-sectional area of the fiber. The total lumens area, A_L is occupied in between 37.35 to 69.47% of the cross-sectional area of the OPF, A_f .

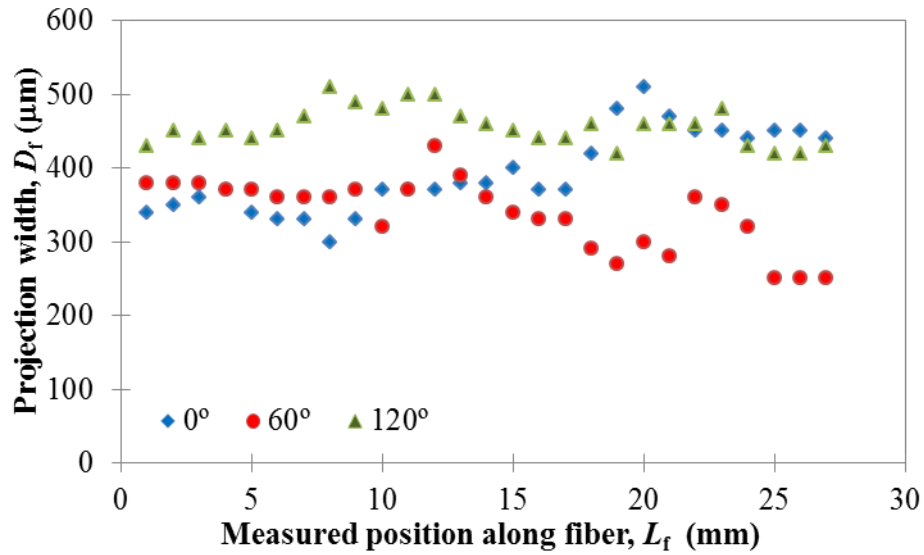


Figure 2.5 Diameter distribution of oil palm fiber.

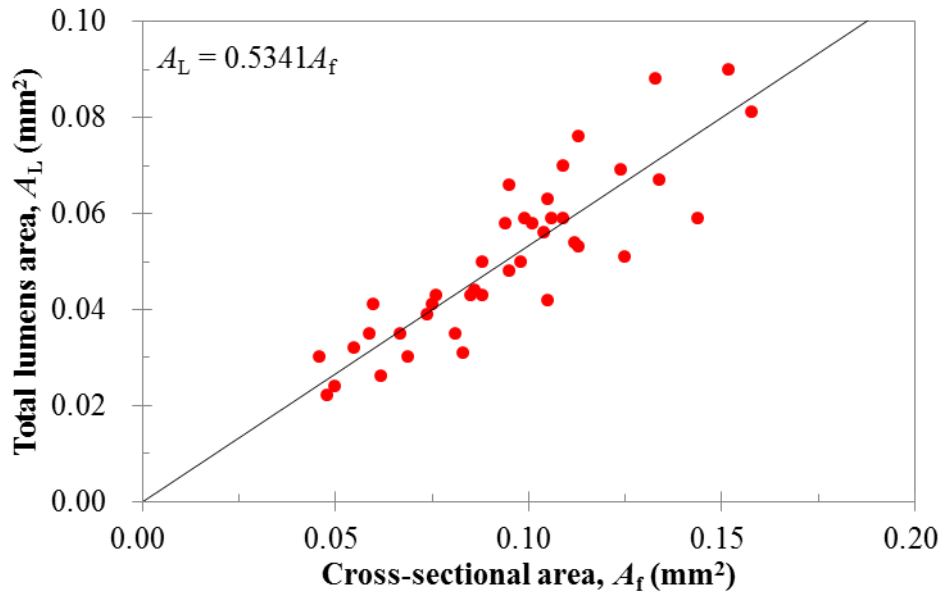


Figure 2.6 Relation between total lumens area and cross-sectional area.

2.3.2 Fourier transform infrared spectroscopy

A FTIR spectra of OPF at various treatment times is shown in Figure 2.7. The FTIR spectra analysis can be divided into 2 regions, which are functional group region (1500 to 3600 cm^{-1}) and fingerprint group region (less than 1500 cm^{-1}). In this study, the discussion on the FTIR spectra will focus on the function group region.

Table 2.1 shows the characteristic IR absorption frequencies of functional groups. The broad band range between 3200 to 3600 cm^{-1} , which indicated by region A in Figure 2.7, are corresponds to the stretching of –OH (hydroxyl group).

The most important absorbance peak in Figure 2.7 is at 1734 cm^{-1} , which indicated by arrow B. The absorbance peak at 1734 cm^{-1} points out the C=O stretching of carbonyl groups of hemicellulose and lignin [11]. As seen in Figure 2.7, the absorption intensity at 1734 cm^{-1} is progressively decreased when the treatment duration is increased. Therefore, this indicates the alkali treatment can partially removes the hemicellulose and lignin from OPF by increasing the treatment time.

Referring to the literature information, the removal of hemicellulose and lignin give two effects on OPF: (1) better load sharing as closer packing between cellulose chains [12-13]; (2) increases the possibility of the reaction sites from the fiber surface as the numbers of the cellulose revealed on the fiber surface are increased [11,14].

Table 2.1 Characteristic IR absorption frequencies of functional groups.

Functional group	Type of vibration	Wavenumber range (cm^{-1})	Intensity
O-H	Stretch	3200-3600	Strong, Broad
C=O	Stretch	1670-1820	Strong

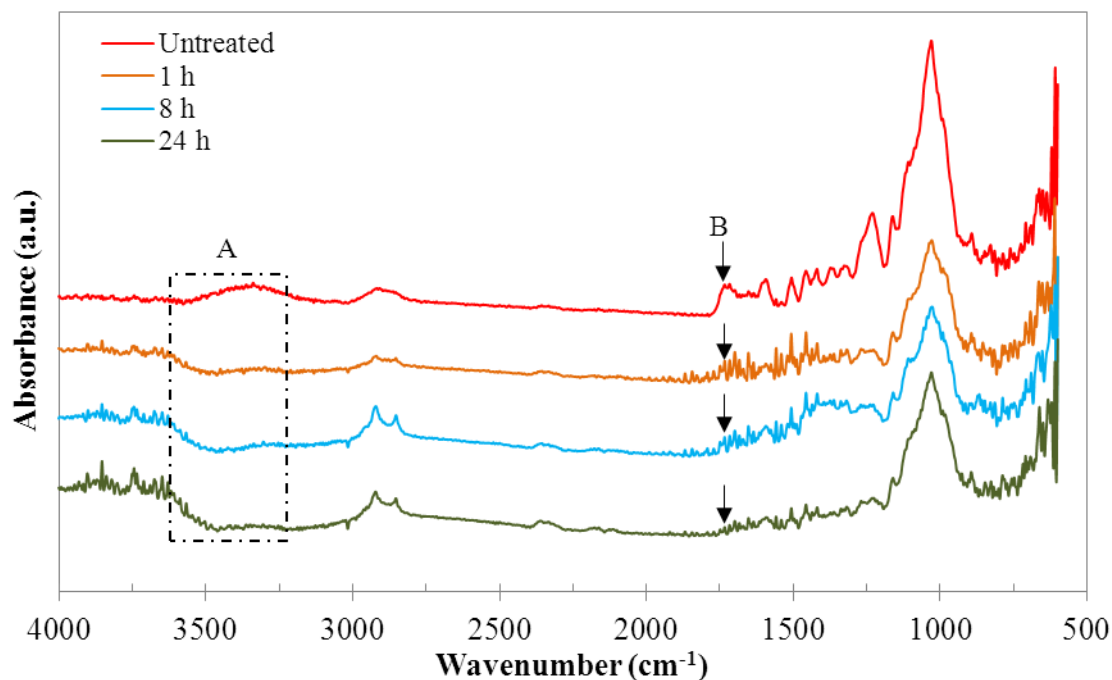


Figure 2.7 FTIR spectra of OPF.

2.3.3 Tensile properties

The relation between average tensile strength and mean fiber diameter is illustrated by the plot in Figure 2.8. The average tensile strength is the average tensile strength of 10 fibers. The mean fiber diameter is also obtained from the average diameter of 10 fibers, where the diameter of each fiber was rounded off to the nearest 10 μm . From the plot in Figure 2.8, the average tensile strength of OPF increases as the fiber diameter decreases. Similar reductions in tensile strength with increases in fiber diameter have been observed in both organic fibers [15] and inorganic fibers [16]. In addition, the tensile modulus of OPF was also increased with decreases in fiber diameter, which shown in Figure 2.9. The general explanation for these phenomena is that there are fewer flaws occurred as the fiber diameter reduced.

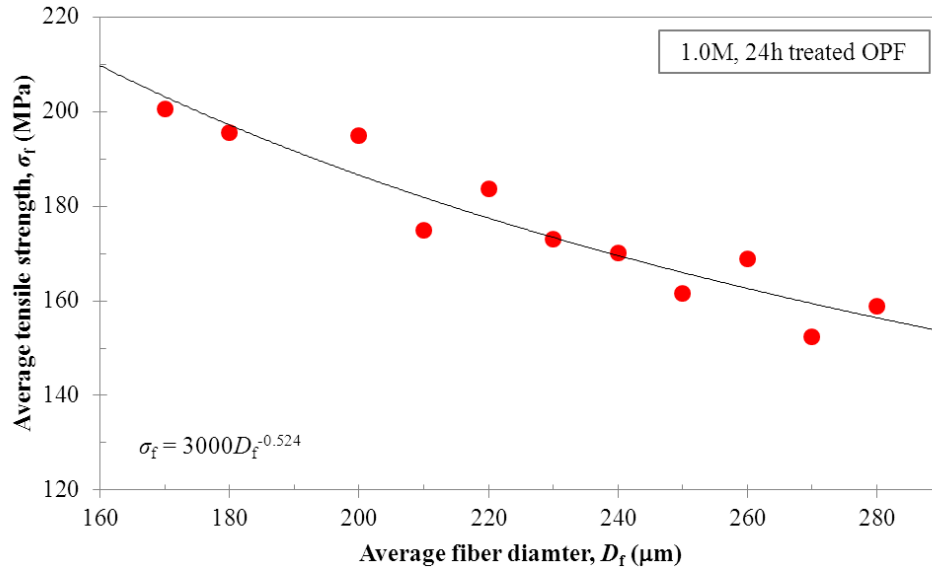


Figure 2.8 Relation between average tensile strength of OPF and mean fiber diameter.

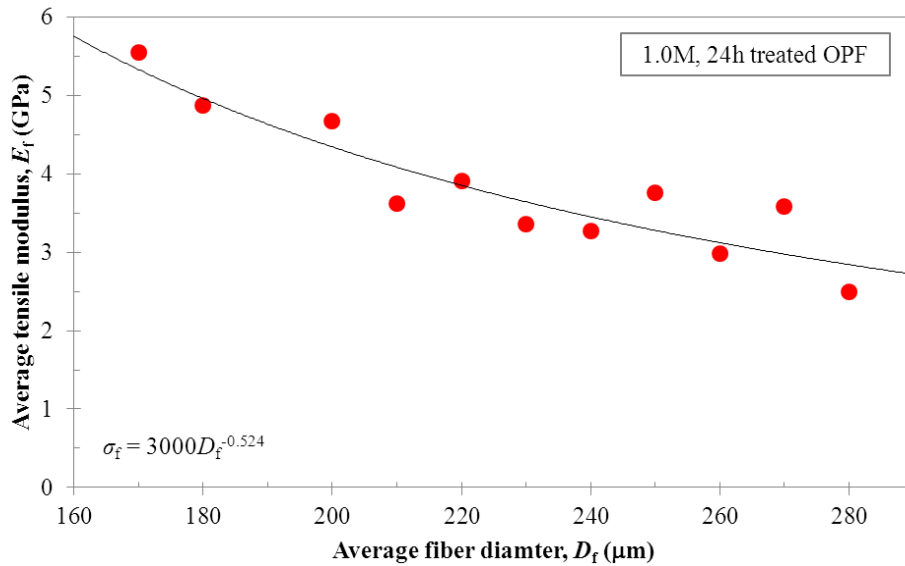


Figure 2.9 Relation between average tensile modulus of OPF and mean fiber diameter.

Tensile test of the single natural fibers is difficult to analyse due to the widely scattered value. The probabilistic approaches are essential to analyse the tensile strength data. Hence the tensile strengths of the oil palm fiber were analysed with the statistical approach in order to achieve a result with 95% confidence intervals. The experimental data have a good agreement with Weibull distribution, where the $R^2 \geq 96.98\%$ (Figure 2.10).

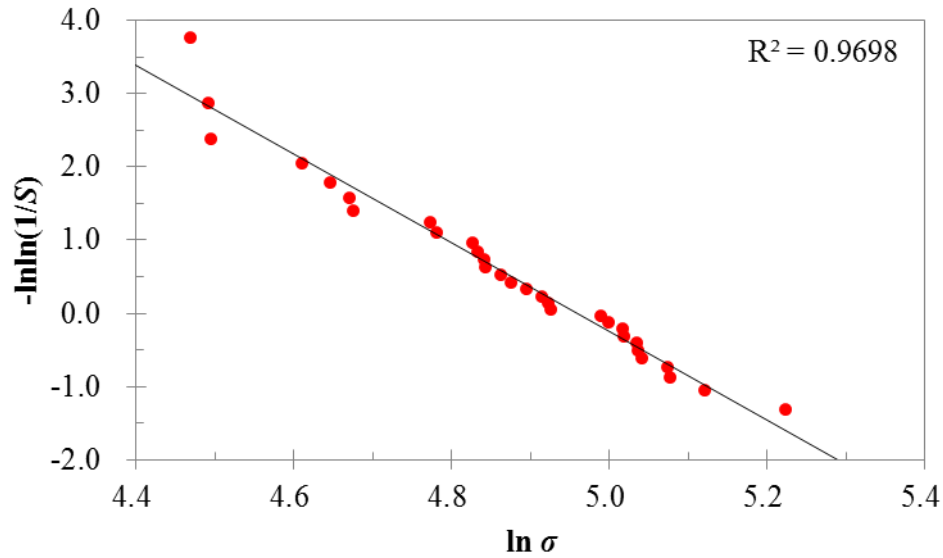


Figure 2.10 Weibull plot of the tensile strength of the specimen.

Typical stress-strain plots of the untreated and alkali treated fiber are shown in Figure 2.11. The tensile strength of the alkali treated OPF is higher than the untreated OPF at nearly the same fiber diameter. However, at the same treatment condition, the tensile strength of the fiber with smaller diameter is greater than the tensile strength of the fiber with larger diameter. As listed in Table 2.2, the tensile strength and tensile modulus of the oil palm fiber have improved by alkali treatment. From the range of treatment conditions, the maximum tensile strength and tensile modulus of the OPF was achieved at 24 hours of 1.0M alkali treatment. The tensile strength of the OPF was increased by 126.93MPa compared to untreated oil palm fiber (72.17MPa), and tensile modulus was also increased by 3.52GPa compared to tensile modulus of the untreated oil palm fiber (1.48GPa). The overall values of the elongation at break were also increased by 10% to 47% respect to elongation at break of the untreated oil palm fibers (Table 2.2). The improvement on tensile strength and tensile modulus can be explained as follows: (1) change in dimensions of the fiber where the fibres were finer after the alkali treatments; (2) closer packing between cellulose by partially removing the hemicellulose and lignin, hereby improve the load sharing between fibrils [4,12,13].

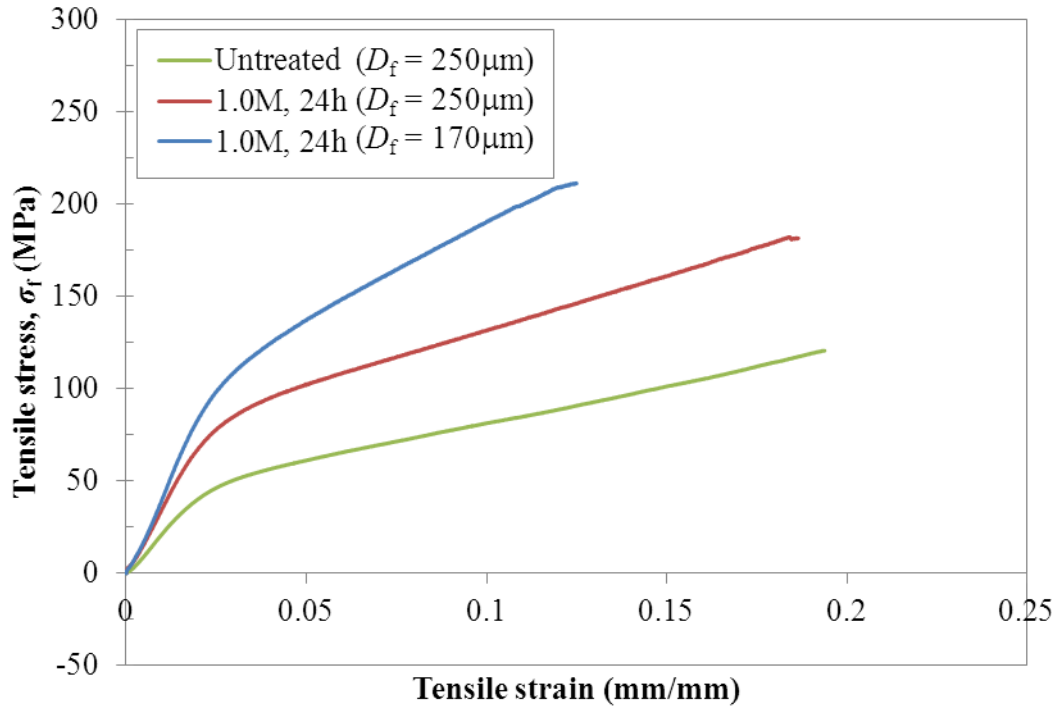


Figure 2.11 Tensile stress – strain behaviour of a single oil palm fiber.

Both tensile strength and tensile modulus of the fibers were decreased with immerse the fibers into high concentration of alkali solution or prolonged treatment time. This may due to the main structures of the fiber were attacked, resulting in weakening or damaging fiber. From the experimental data, the average diameter obtained from the untreated fiber and the 1.0M, 24h alkali treated fiber is $299 \pm 43.4\mu\text{m}$ and $205 \pm 39.4\mu\text{m}$. The probability of the critical flaws occurred is higher in the fiber with larger cross-sectional area. Therefore, tensile strength of an alkali treated fiber (smaller diameter) is expected higher than the tensile strength of untreated fiber with large diameter.

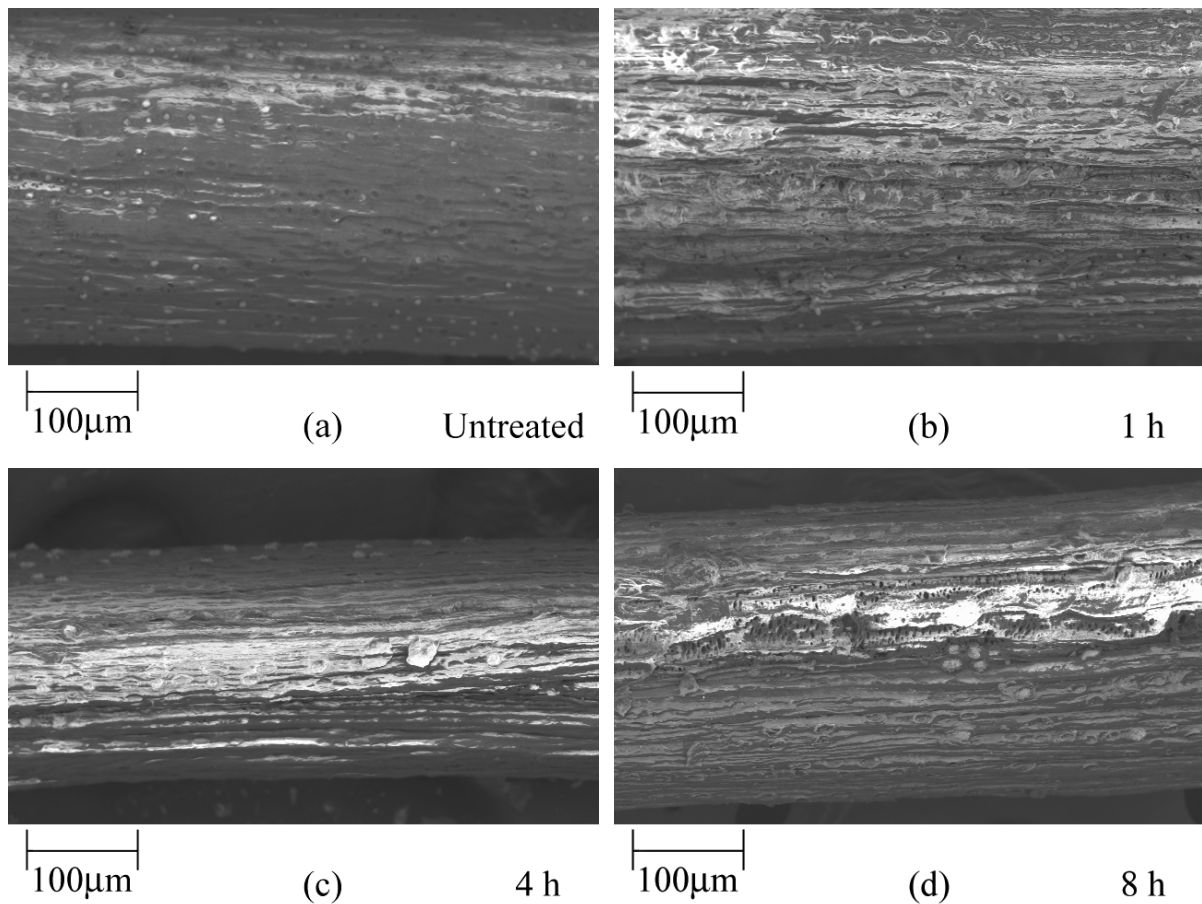
Table 2.2 Mechanical properties of OPF fiber at various concentrations and soaking times of alkali treatment.

NaOH concentration (mol/liter)	Treatment time, t (h)	Elongation at break ^a , ε_f (%)	Tensile Strength ^a , σ_f (MPa)	Tensile Modulus ^a , E_f (GPa)
0	0	12.26	72.17	1.48
0.5	0.5	13.05	116.36	2.98
0.5	1	14.31	133.63	3.70
0.5	2	13.79	141.43	3.26
0.5	4	17.96	153.62	3.47
0.5	8	15.64	144.03	3.29
0.5	12	15.90	152.23	3.47
0.5	24	17.15	154.05	3.66
0.5	48	12.87	135.90	3.49
1.0	0.5	15.39	115.86	2.45
1.0	1	14.46	132.23	3.80
1.0	2	13.92	140.94	3.95
1.0	4	16.27	159.01	3.75
1.0	8	15.11	158.42	3.88
1.0	12	14.14	166.53	4.37
1.0	24	13.76	199.10	4.95
1.0	48	14.83	170.04	3.40
1.5	0.5	15.97	128.86	3.15
1.5	1	13.10	164.93	4.53
1.5	2	14.04	145.43	4.07
1.5	4	15.10	142.01	3.52
1.5	8	15.66	137.15	3.43
1.5	12	18.53	140.76	2.82
1.5	24	21.73	94.56	2.02
1.5	48	17.04	95.85	2.57

^a Average value of thirty samples.

2.3.4 Surface morphology

Figure 2.12 shows the various SEM micrograph of the alkali-treated oil palm fibers. The surface of the oil palm fiber become rougher and finer after alkali treatment due to the leaching out of the impurities from fiber surface [17]. From the morphological observation in Figure 2.12, the surface roughness increases as increasing the alkali treatment time. The roughness development on the fiber surface gives a better interface adhesion between fiber and matrix.



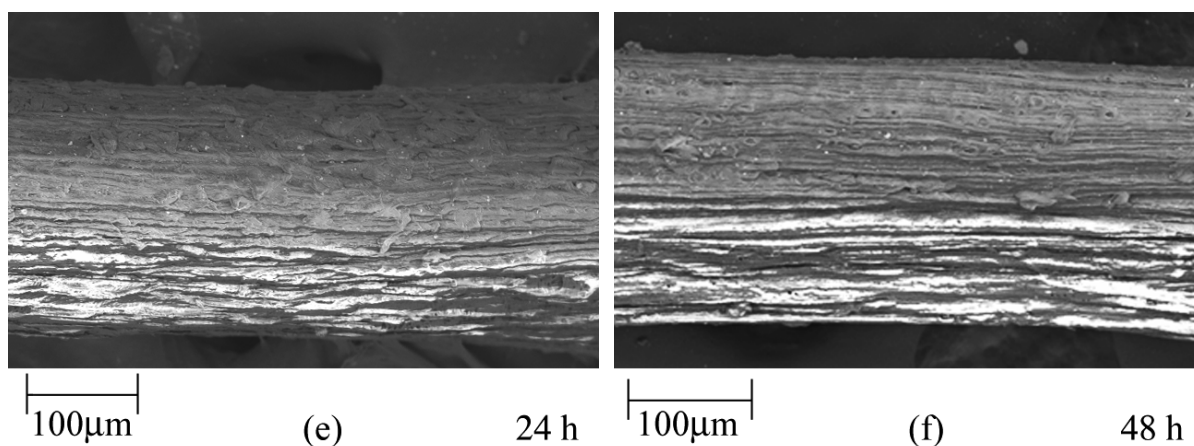


Figure 2.12 SEM micrographs of the untreated and 1.0M NaOH treated OPF at various treatment durations.

2.4 Conclusion

This chapter studied the surface treatment, morphology and mechanical properties of the OPF. The following conclusions can be listed from this research:

1. The fiber become finer and rougher after the alkali treatment due to the removal of non-cellulosic materials from the fiber as demonstrated by FTIR and morphological studies.
2. Weibull distribution function reveals a good fit with the experimental data and it is useful in scaling the fiber strengths.
3. The tensile strength of the OPF has been significantly improved by alkali treatment. In the range of treatment conditions, 1.0M, 24 h treatments of OPF give the best result in mechanical strength.
4. Higher NaOH concentration or prolonged treatment time reduce tensile performance of the fiber. This may due to the main components of the OPF were attacked.

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Chapter 3

Statistical analysis of the tensile strength of treated oil palm fiber by utilisation of Weibull distribution model

3.1 Introduction

The mechanical properties of OPFs are highly variable [1, 4-6]. The previous chapter also reported the diameters of natural fibers vary greatly not only among fibers but also along the fiber length. Therefore, statistical approaches applied on the mechanical properties are essential to analyse the high disparity of the mechanical properties of OPFs.

The Weibull/weakest link distribution of strength of a fiber can be easily derived from the strength distribution of many independent unit links of the fiber [7] and the Weibull distribution has been widely applied on the statistical analysis of mechanical strengths of natural fiber. In Chapter 2, the strength of OPFs was observed to be decreased with increasing fiber diameter. Therefore, Weibull/weakest link distribution becomes a useful tool to predict the scale effect of natural fibers.

3.2 Theoretical background

The common two-parameter Weibull distribution applied in the mechanical study is given by:

$$F = 1 - \exp \left[-\frac{V}{V_0} \left(\frac{\sigma}{\sigma_0} \right)^m \right] \quad (1)$$

where F is the failure probability of the fiber, V is fiber volume, σ is failure strength, V_0 is standard volume, m is Weibull modulus and σ_0 is scale parameter.

The average value of σ can be predicted from the equation below once the Weibull distribution parameters (m, σ_0) are obtained:

$$\bar{\sigma} = \sigma_0 \left(\frac{V}{V_0} \right)^{1/m} \Gamma \left(1 + \frac{1}{m} \right) \quad (2)$$

For a constant fiber diameter, when the average value of σ_1 at gauge length L_1 and m are obtained, the average value of σ_2 at the gauge length L_2 can be calculated:

$$\bar{\sigma}_2 = \bar{\sigma}_1 \left(\frac{L_2}{L_1} \right)^{-1/m} \quad (3)$$

where the values of σ_1 and σ_2 are the fiber stress at gauge length L_1 and gauge length L_2 respectively.

However, great discrepancies were found between conventional Weibull predicted strength and experimental data [8-9]. Therefore, Watson and Smith [10] as well as Gatans and Tamuzs [11] have suggested that the exponent $1/m$ in Equation (3) be modified to α/m :

$$\bar{\sigma}_2 = \bar{\sigma}_1 \left(\frac{L_2}{L_1} \right)^{-\alpha/m} \quad (4)$$

and the corresponding Weibull distribution (named as WSGT function [12]) becomes:

$$F = 1 - \exp \left[- \left(\frac{V}{V_0} \right)^\alpha \left(\frac{\sigma}{\sigma_0} \right)^m \right] \quad (5)$$

Manipulation above expression gives the following relation:

$$\ln(-\ln(1 - F)) - \alpha \ln \frac{V}{V_0} = m \ln \sigma - m \ln \sigma_0 \quad (6)$$

Hence, a plot of $\ln(-\ln(1 - F)) - \alpha \ln \frac{V}{V_0}$ versus $\ln \sigma_f$ which should produce a straight line, give rise to the gradient, m and intercept σ_0 at $\ln(-\ln(1 - F)) - \alpha \ln \frac{V}{V_0} = 0$. Median rank method is usually used as an estimator for probability of fiber failure.

$$F = \frac{i - 0.3}{N + 0.4} \quad (7)$$

where i is the rank of the each data point and N is the total number of the samples.

The parameter of α was found to vary in previous researches. Phoenix *et al.* [13] suggested that $\alpha = 0.60$ for Kevlar-49 fibers, and Wu and Netravali [14] obtained $\alpha = 0.77$ for Nicalon SiC fibers. However, Zhang *et al.* [15] suggested that the value of α in Equation (4) is related to the within-fiber diameter variation:

$$\ln(CV) = \alpha \ln(L) + A + \varepsilon \quad (8)$$

where CV is the average coefficient of variation, L is the gauge length, A is a constant, ε is a random error.

Considering all the above issues, it is important to determine a suitable Weibull model to predict the tensile strength of OPFs and other natural fibers which have the similar structures. In this chapter, the comparison of the statistical analysis by using the conventional Weibull distribution and the modified Weibull model are also described. The new conical frustum model on the Weibull analysis also compared with the modified Weibull model proposed by Zhang *et al.* [15].

3.3 Experimental

3.3.1 Materials

In order to study the size effects on the fiber length, fiber diameters ranged from 200 μm to 300 μm were used to analysis the fiber strength at various gauge lengths. Before fabrication process, the fibers were pretreated with 1.0M NaOH solution for 24 hours. The procedure of the alkali treatment was mentioned in Chapter 2.

3.3.2 Fiber diameter measurement

The fabrication of the specimens was described in section 2.2.6. The specimens shown in Figure 3.1 were prepared for the tensile test at four gauge lengths (10mm, 20mm, 30mm and 40mm). In order to simplify the analysis, the fibers were considered perfectly round and the micrographs of the fiber at 1mm intervals were captured by using a Nikon Eclipse ME600 microscope. The fiber diameters were then measured using an ImageJ software version 1.38x.

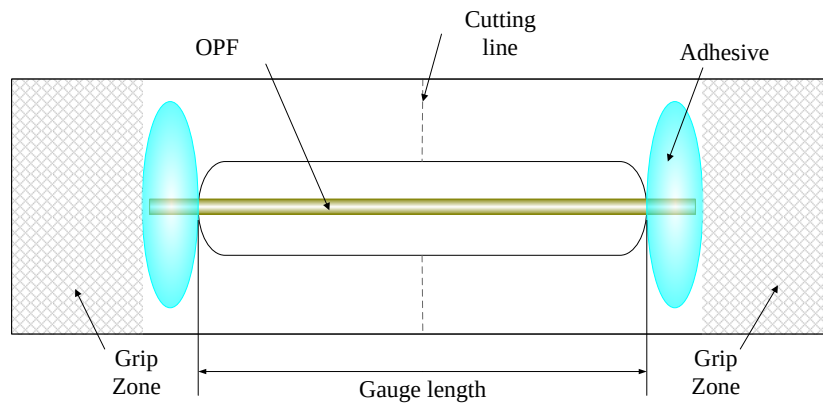


Figure 3.1 Schematic of the single fiber test specimen.

3.3.3 Single fiber tensile test

The uniaxial tensile strength tests were carried out by using a Tohei MT201 tensile test machine with the load capacity of 50N and the strain rate of 9mm/min. The tensile strengths of the OPFs were evaluated at 10mm, 20mm, 30mm and 40mm gauge lengths. The values for the fiber fractures at or near the adhesive spots were discarded. Fifty sets of successful data at each of these gauge lengths were obtained for the statistical analysis.

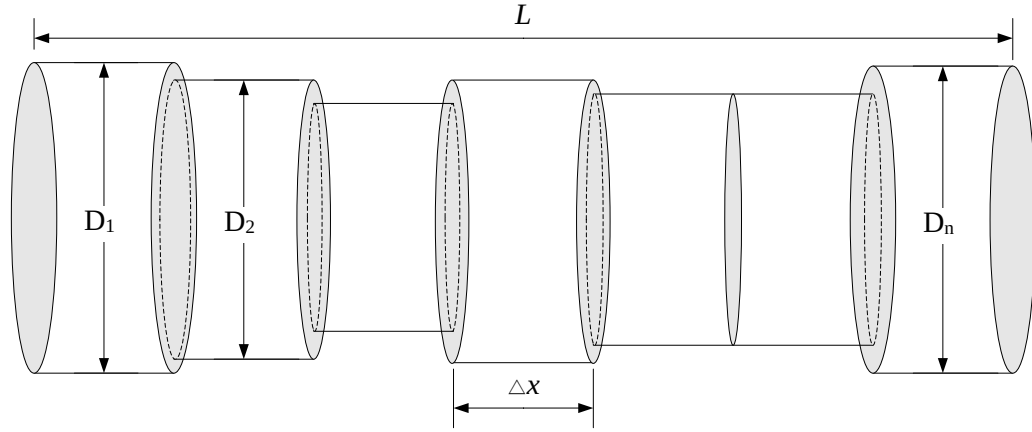
3.4 Results and discussion

3.4.1 Relationship between within-fiber and gauge length

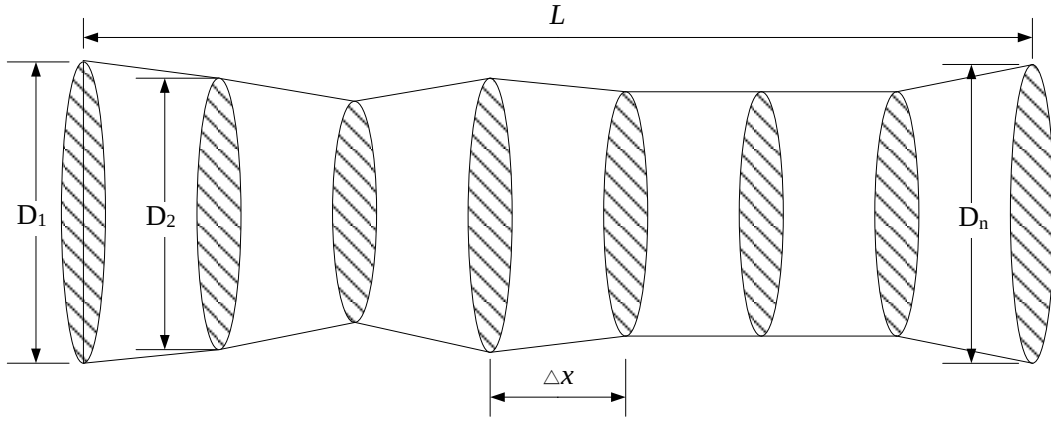
Cylinder model as illustrated in Figure 3.2(a) is a common model used for Weibull analysis of variable within-fiber diameters [16-19], where the fiber length, L had been divided into small cylindrical sections with the length of Δx . In Zhang *et al.* [15] research, they also divided the fiber into small cylindrical sections and used the diameter of the sections on each fiber to obtain the within diameter variation. Then the coefficient of variation of fiber diameter can be obtained and used to predict the strength of wool fibers. In their report, the results of the predicted strength were significantly improved by using the modified Weibull distribution. Figure 3.2(b) is the conical frustum model which used in this research to replace the Zhang *et al.*[15] model to obtain the value of coefficient of variation. The fiber was divided into n conical frustum segments where the value of Δx in this study is 1mm. the volume of the fiber used for the Weibull distribution can be obtained as follow:

$$V = \frac{\pi \Delta x}{3} [(D_1^2 + D_1 D_2 + D_2^2) + (D_2^2 + D_2 D_3 + D_3^2) + \cdots + (D_{n-1}^2 + D_{n-1} D_n + D_n^2)] \quad (9)$$

where V is the fiber volume, Δx is the fiber length and D is the fiber diameter at each section.



(a) Cylinder Model



(b) Conical Frustum Model

Figure 3.2 Schematics of measurement for volume along fiber axis.

As listed in Table 3.1, mean diameter is the average diameter of 50 tested fibers. The CV_{FD} is and CV_{CF} are the average value of the within-fiber diameter variation coefficient and within-fiber conical frustum volume variation coefficient of 50 fibers respectively. The average value of failure strength was observed to be decreased with increasing in gauge length is mainly due to the fact of the increment in flaw sizes in larger fiber volume. Similar behaviour is observed in wool [20], banana [23] and coir [24] fibers. The relation between CV within-fiber and the gauge length of the test specimens is illustrated in Figure 3.3. The average value of coefficient of variation for both fiber diameter and the conical frustum volume increases exponentially with the increasing gauge length. The correlation of the logarithm in Figure 3.3 also shows a very high value ($R^2 = 0.9829$ for Zhang *et al.* model, $R^2 = 0.9749$ for present model). As reported by Zhang *et al.*, the value $\alpha = 0.2133$ can be

obtained from the slope value of $\ln(CV)$ versus $\ln(L)$. For conical frustum model, the value of α is 0.2906, which is slightly higher than the value α obtained from Zhang *et al.* modified model.

Table 3.1 Mean fiber diameter, coefficient of variation and fiber strength.

Gauge length (mm)	Mean diameter ^a (μm)	CV within-fiber (%)		Fiber strength ^a (MPa)
		Diameter (CV _{FD})	Conical frustum (CV _{CF})	
10	245.30	11.90	19.61	177.68 ± 38.41
20	240.08	13.55	23.40	170.89 ± 35.78
30	245.60	14.98	27.96	161.48 ± 37.71
40	247.80	16.36	28.68	160.28 ± 37.66

^aStatistically analysed value of fifty samples.

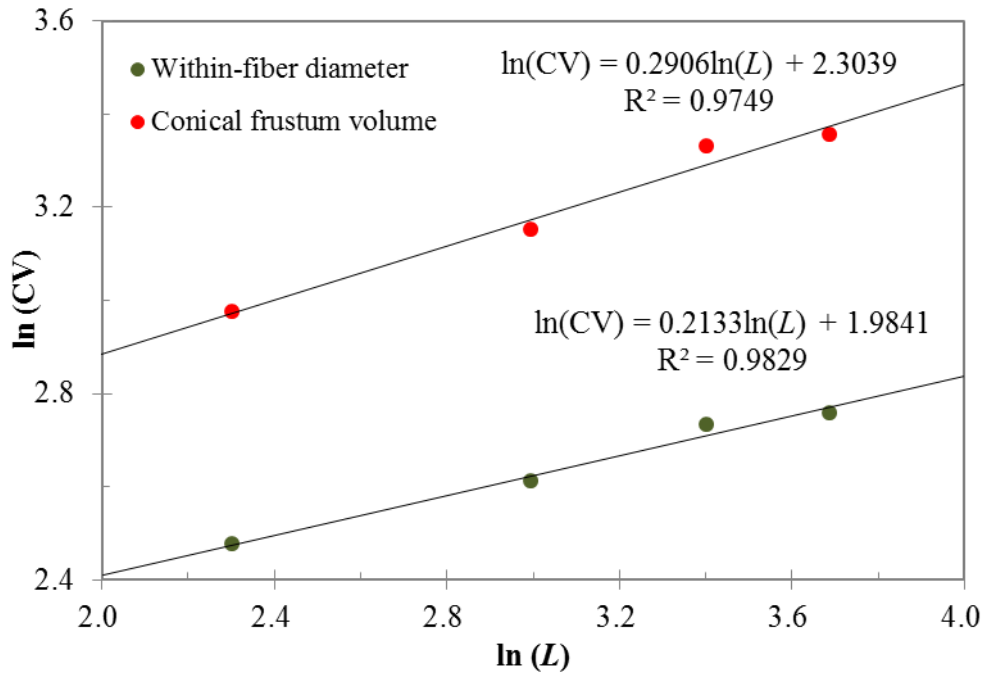


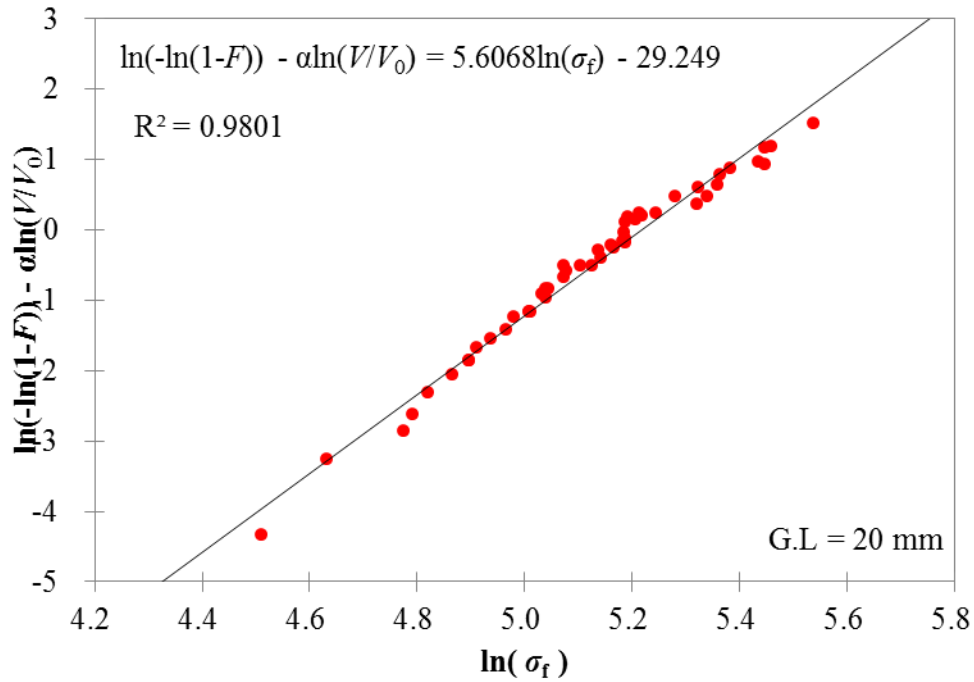
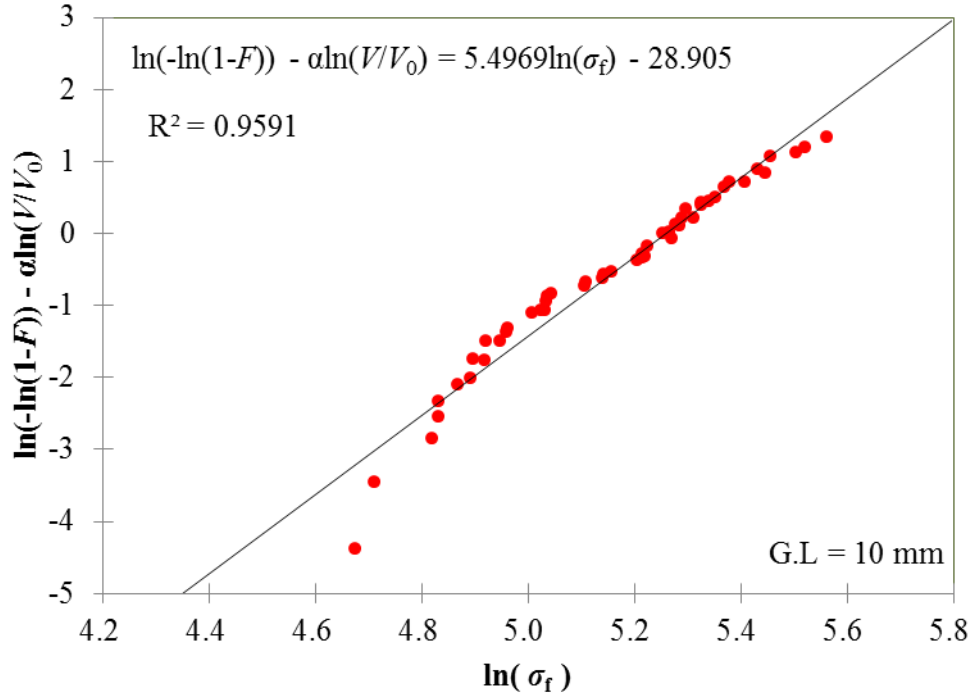
Figure 3.3 Relation between CV within-fiber and gauge length.

3.4.2 Weibull distribution and strength prediction

The Weibull plots based on volume of conical frustums for the OPF tensile strength at 10mm, 20mm, 30mm and 40mm gauge lengths are illustrated in Figure 3.4. For all the gauge lengths tested, the regression of the failure strength has a good agreement with experimental data where the value of R^2 is from 95.91% to 98.79%. The Weibull modulus, m and the scale parameter, σ_0 obtained from the Weibull plots at gauge lengths of 10mm, 20mm, 30mm and 40mm are listed in Table 3.2.

Table 3.2 The Weibull parameters.

Gauge length (mm)	Weibull modulus m	Scale parameter σ_0
10	5.50	192.19
20	5.61	184.33
30	4.80	176.36
40	4.98	174.71



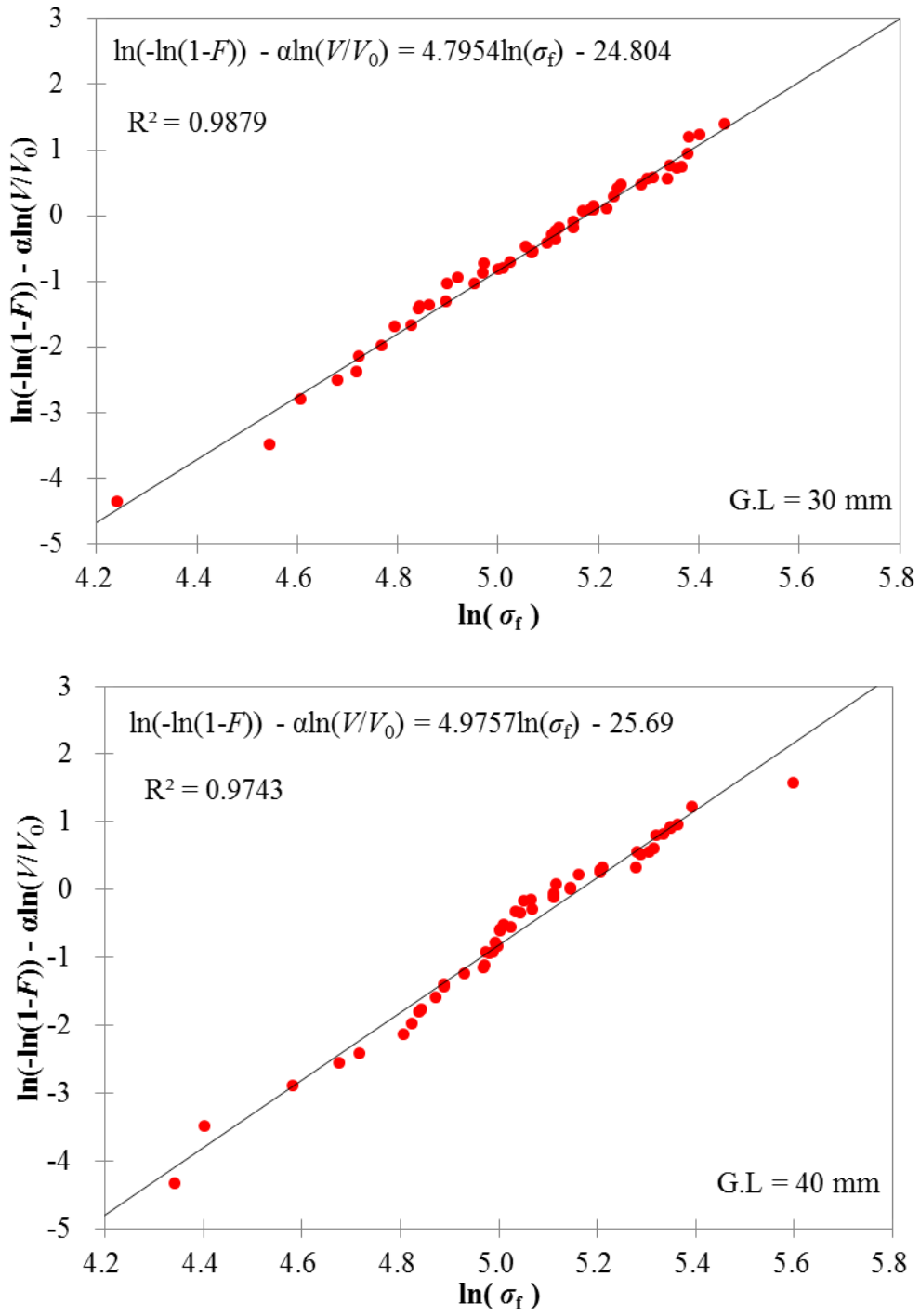
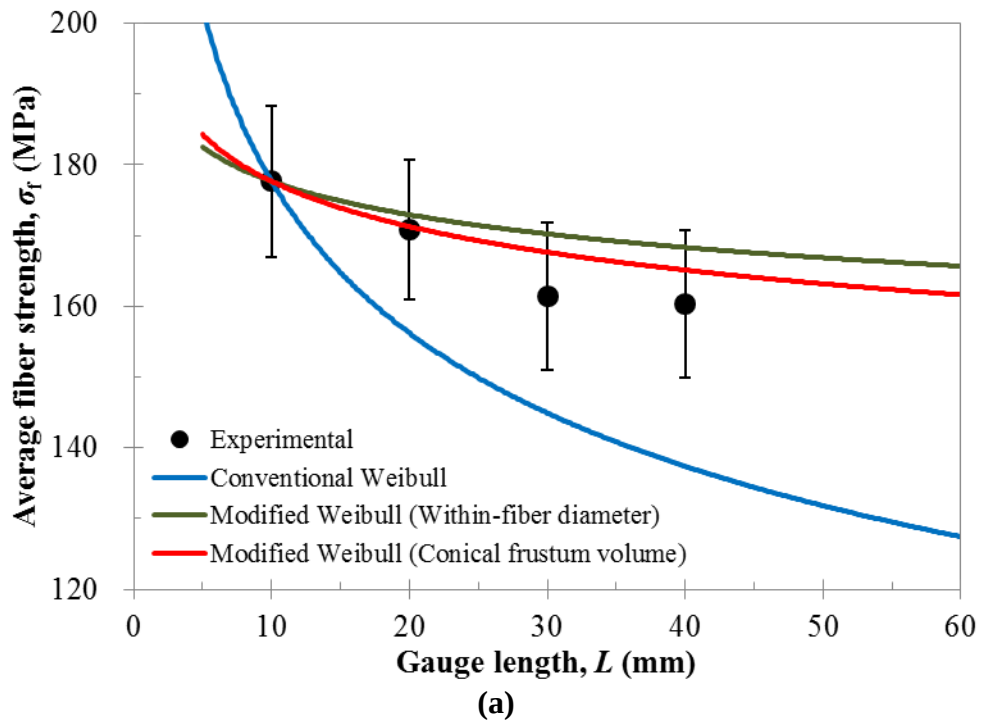


Figure 3.4 The Weibull plot of the fiber strength based on volume (conical frustum) variation at four different gauge lengths.

The Weibull model can predict the gauge length effect on the fiber strength if the data well fit on the distribution. The predicted failure strength from the conventional Weibull distribution and the modified Weibull distribution, by incorporating with within-fiber diameter and conical frustum volume, which based on the experimental value at 10mm and 40mm are shown in Figure 3.5(a) and (b) respectively. The disparity between the experimental data and the conventional Weibull predicted failure strength was observed in Figure 3.5(a) and (b). As expected, the modified Weibull models can predict the fiber strength more accurately compared with the conventional Weibull distribution, where the modified Weibull predicted strengths are within 95% confidence intervals of the experimental results. Figure 3.5(a) and (b) clearly show that the modified Weibull model by incorporating with the conical frustum volume variation can predict the gauge length effect more accurately than the Zhang *et al.* modified model. Therefore, the fiber strength at other gauge length can be estimated if the fiber strength at one of the gauge lengths is obtained.



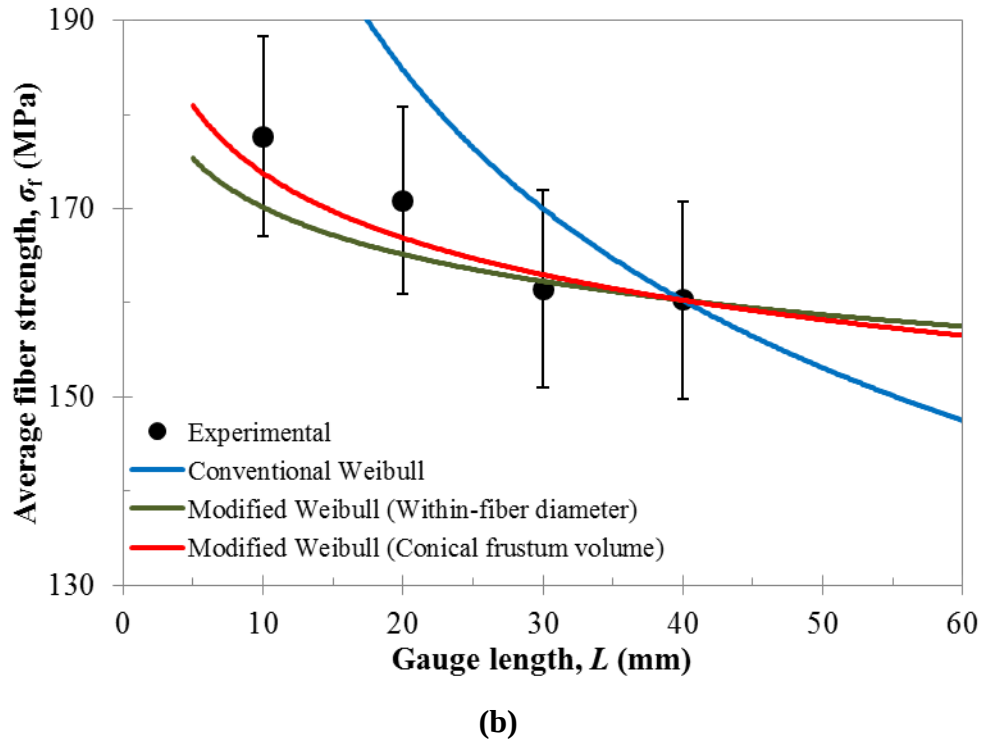


Figure 3.5 Comparison of average fiber failure strength between experimental and predicted values (Eq. (3) and (4)) based on the experiment data at (a) 10mm and (b) 40mm gauge length.

3.4.3 Parameter α

A comparison between the experimental data and predicted values from Equation (4) at different value of parameter α ($0 < \alpha < 1$, [15]) is shown in Figure 3.6. The predicted curve with the parameter $\alpha = 0.4$ was found to have a good agreement with experimental values. However, the parameter $\alpha = 0.4$ is specifically significant at very short gauge length. Hence, applying the parameter $\alpha = 0.4$ at long gauge length may decrease the predictive accuracy.

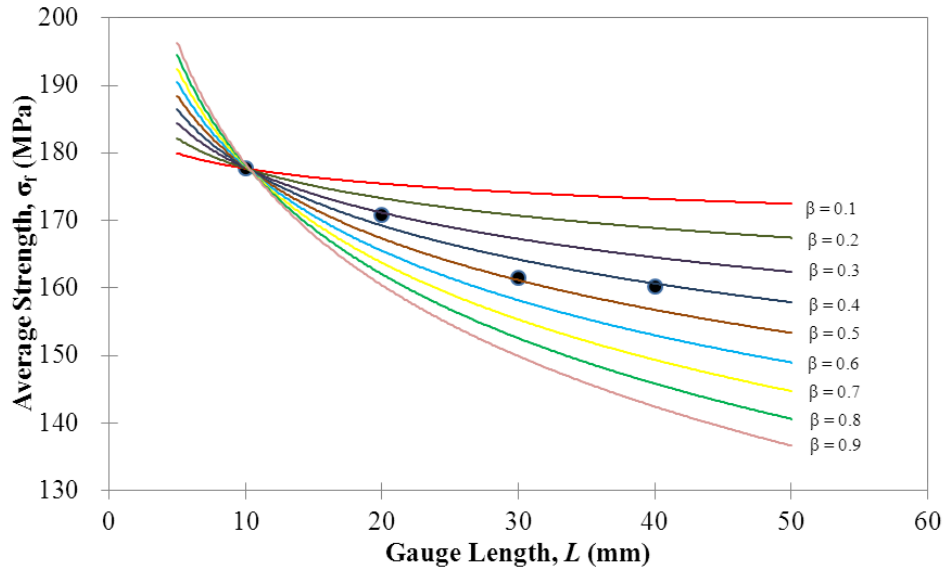


Figure 3.6 Comparison between the experimental and the predicted values of average failure strength of oil palm fibers at different value of parameter α (prediction based on the result at 10mm gauge length).

3.4.4 Scale effect

The failure strength of banana [20], coir [21], ramie [22] and kenaf [22] fibers at various gauge lengths are provided in Table 3.3. As expected, the failure strength of banana, coir, ramie and kenaf fibers decrease with increasing in gauge length.

The plot of normalised failure strength at different gauge lengths is illustrated in Figure 3.7. The strength values of oil palm, banana, coir, ramie and kenaf fibers are normalised by using each respective strength value at 10mm. Clearly, the OPF and ramie fiber are less sensitive to its gauge length compared to kenaf, banana and coir fibers. The scale effect of the fiber strength depends on the growth rate of the flaw sizes in the fiber when the gauge length is increased.

Table 3.3 Tensile strength of natural fibers at various gauge lengths.

Gauge length, L (mm)	Fiber failure strength, σ_f (MPa)			
	Banana [20] ^a	Coir [21] ^a	Ramie [22] ^a	Kenaf [22] ^a
10	1055.516	279.17	2517	1137
20	930.734	265.98	2380	1010
35	891.706	211.15	-	-
50	711.661	175.62	2172	888

^a References.

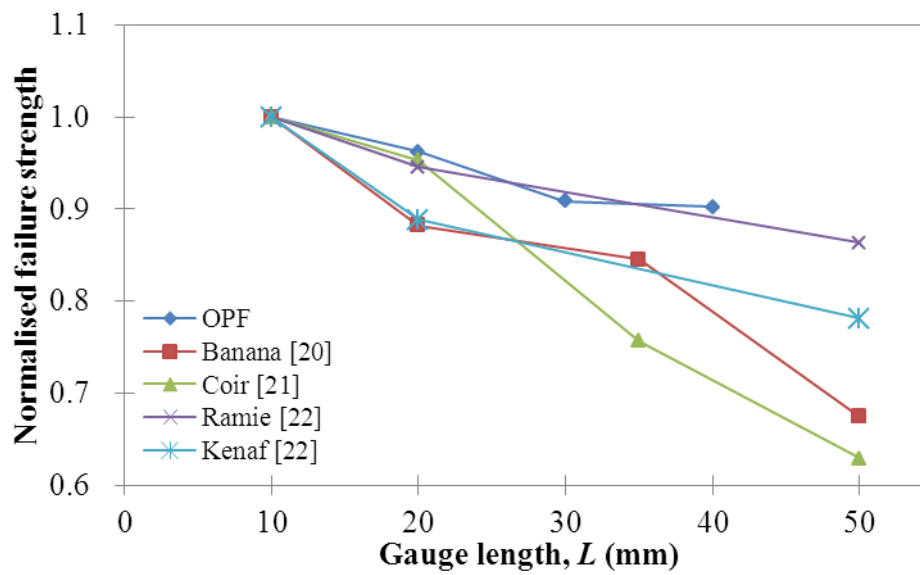


Figure 3.7 Normalised failure strength of natural fibers at various gauge lengths.

As seen from Figure 3.7, the failure strength of the OPF has less scale effect on its gauge length compared to coir fiber although Hassan *et al.* [23] have reported that the OPF has a similarity in structures to coir fiber. This could be due to the growth rate of the flaw sizes and the flaw distributions differ between OPF and coir fiber.

3.5 Conclusion

This chapter evaluated the failure strength of the OPFs at different gauge lengths. A new modified Weibull by cooperating with conical frustum volume has been studied. The following conclusions can be highlighted from this chapter:

1. The average failure strength of the OPF increases with decreasing in gauge length because the flaw sizes increase in larger fiber volume.
2. The predicted fiber strength by the modified Weibull models is more accurate than the conventional Weibull distribution. However, the predicted strength of OPF is more accurate by using the variation of conical frustum volume compared with variation of within-fiber diameter. The statistical analysis of OPF strength by incorporating the within-fiber conical frustum volume variation should be considered.
3. The optimum value of the parameter $\alpha = 0.4$ was obtained in this study. However, the value of the parameter α is specifically significant at very short gauge length.
4. The scale effect may vary although the fibers have a similarity in structures. This may be due to the growth rate of the flaw sizes and the flaw distributions differing from fiber to fiber.

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Chapter 4

Interfacial fracture toughness evaluation of poly(L-lactic acid)/ natural fiber composite by using double shear test method

4.1 Introduction

As described in Chapter 1, the low mechanical strength of natural fibers reinforced composites can be due to the weak interfacial adhesion between fiber and matrix. The existence of stress concentrations in the bond joints along the bond length transfer the load gradually between adherends. Many studies also revealed that the bonding length can affect the adhesive of joints [1-3].

In previous research on the tensile performance of hybrid polyester-based composite reinforced with glass and oil palm fiber in aluminium laminates, the poor adhesive between OPF fibers were identified [4]. Therefore, current works were carried out to investigate the interfacial bonding mechanism of the OPF and matrix.

In this chapter, Mode II interfacial fracture toughness between treated oil palm fiber and poly (L-lactic acid) (PLLA) composite was evaluated by using the double shear test method, which consists of 3 fibers model composite and 4 fibers model composite. The fibers used in both model composites were pretreated 1.0M alkali solution for 24 hours. In addition, 0.5M, 2 hours alkali treated fibers were also conducted in the present study in order to compare the alkali treatment effects on interfacial fracture toughness and the critical interfacial shear stress of the model composites. Scanning electron microscopy (SEM) was utilised to observe the crack propagation between fiber/matrix interfaces.

4.2 Experimental

4.2.1 Materials

The average fiber diameters used in this experiment is in between 190 μ m to 340 μ m. Before fabrication process, the OPFs were pretreated with mild alkali solution (0.5M NaOH for 2 hours) and strong alkali solution (1.0M NaOH for 24 hours), respectively. The mild alkali treated fibers were used instead of untreated fibers due to the weak adhesion fiber/matrix interface, resulting in difficulty to fabricate the model composite for the double shear test.

4.2.2 Sample preparation

Three fiber model composite and four fibers model composite were prepared for the Mode II interfacial test. Prior to preparing the specimens, oil palm fibers were aligned closely in parallel on a paper tab with a small tension force applied on the fibers to avoid loosening of the fibers. Both end points of the fibers were then glued by using epoxy resin adhesive. After the fibers were aligned, a small amount of melted PLLA obtained from a PLLA fiber was placed at the mid-point in between 2 oil palm fibers. The final matrix length of the specimen was measured after the melted PLLA was completely cured. The schematics and SEM micrographs of the 3 fibers model specimen and 4 fibers model specimen were shown in Figures 4.1 and 4.2 respectively.

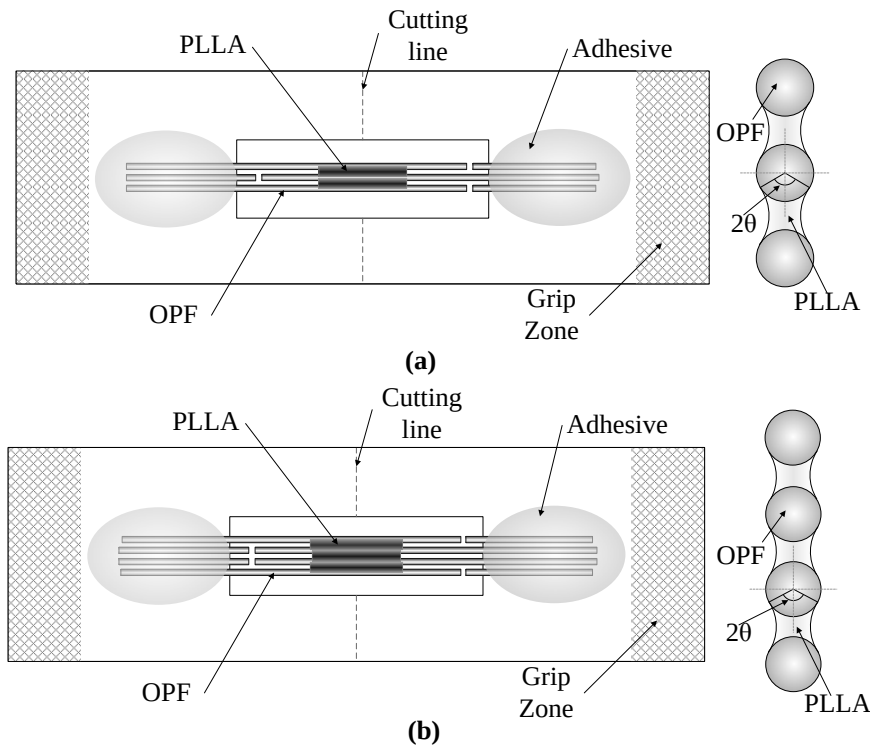


Figure 4.1. Schematics of the model composites. (a) 3 fibers model composite (b) 4 fibers model composite.

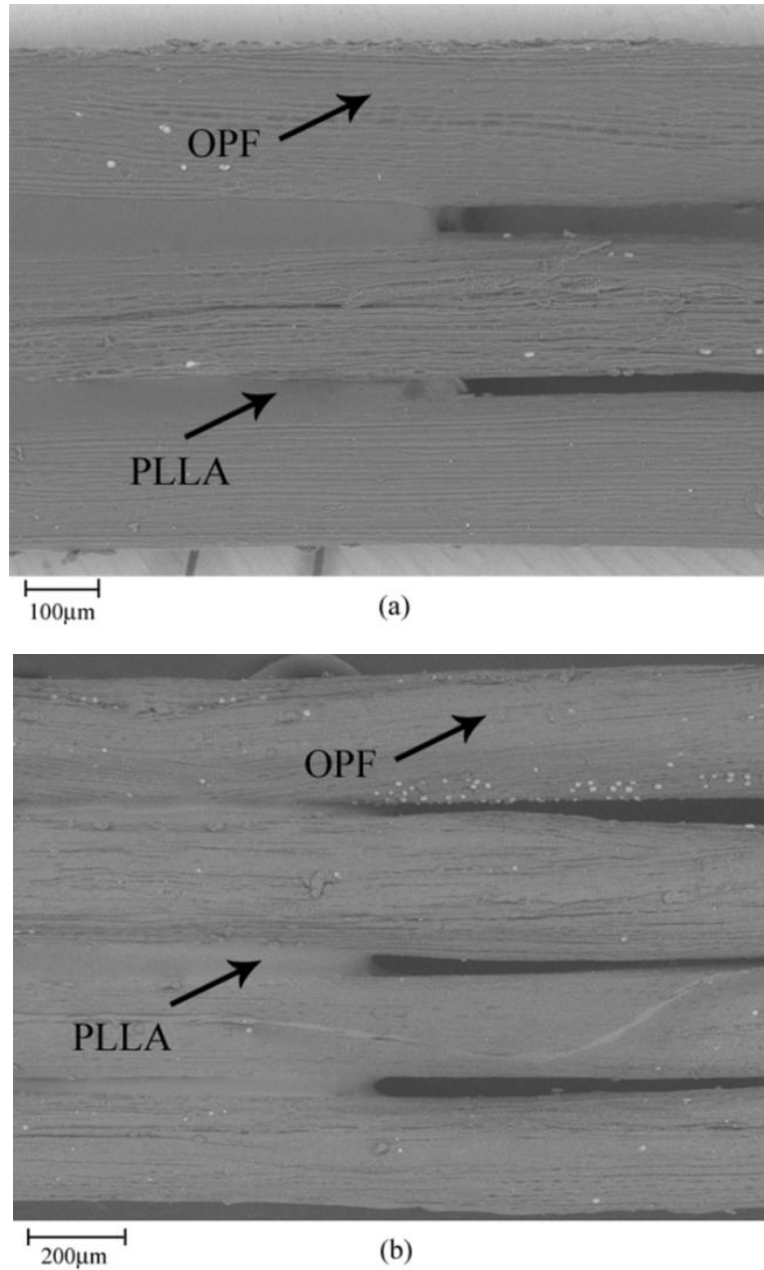


Figure 4.2 SEM Micrographs of the model composites. (a) 3 fibers model composite.
(b) 4 fibers model composite.

Figure 4.3(a) and (b) show the dimensions of the 3 fibers and 4 fibers model composites respectively. In the fabrication process, the matrix lengths on the both side of the specimen are very difficult to achieve with the same matrix length. Therefore, the matrix length, L is the average value of both side of the matrix length. The value of fiber lengths, m and n were also obtained from the average value of both sides of the specimen. The average value of spacing between fibers, t was calculated from the mean of ten random measurements of the spacing

between fibers of the specimen. Ten diameter measurements on each fiber were also performed to obtain the average value of the fiber diameter as listed in Table 4.1.

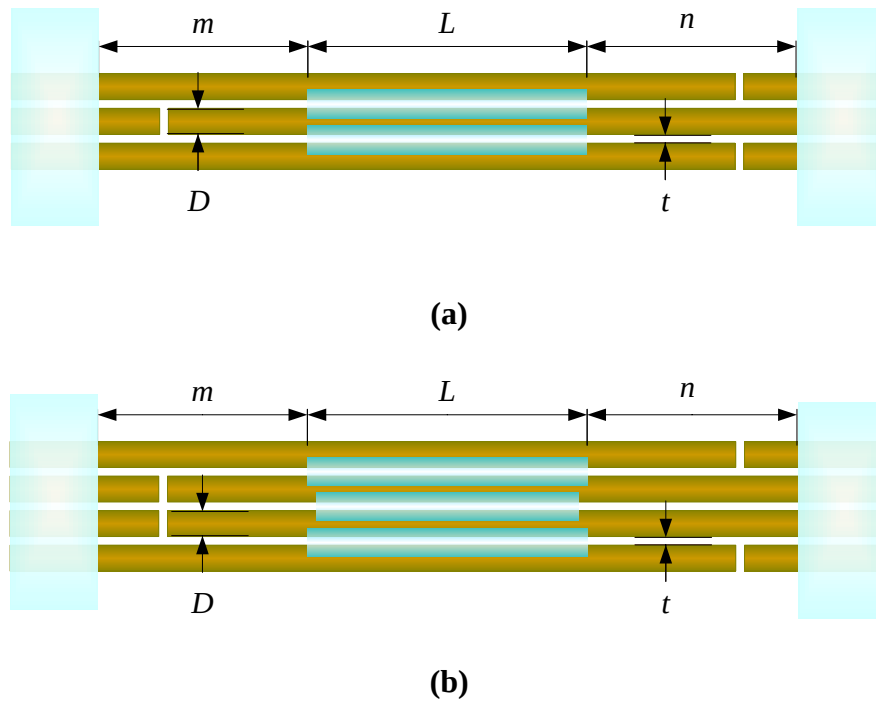


Figure 4.3 Definitions of the dimension of (a) 3 fibers model composite, and (b) 4 fiber model composite.

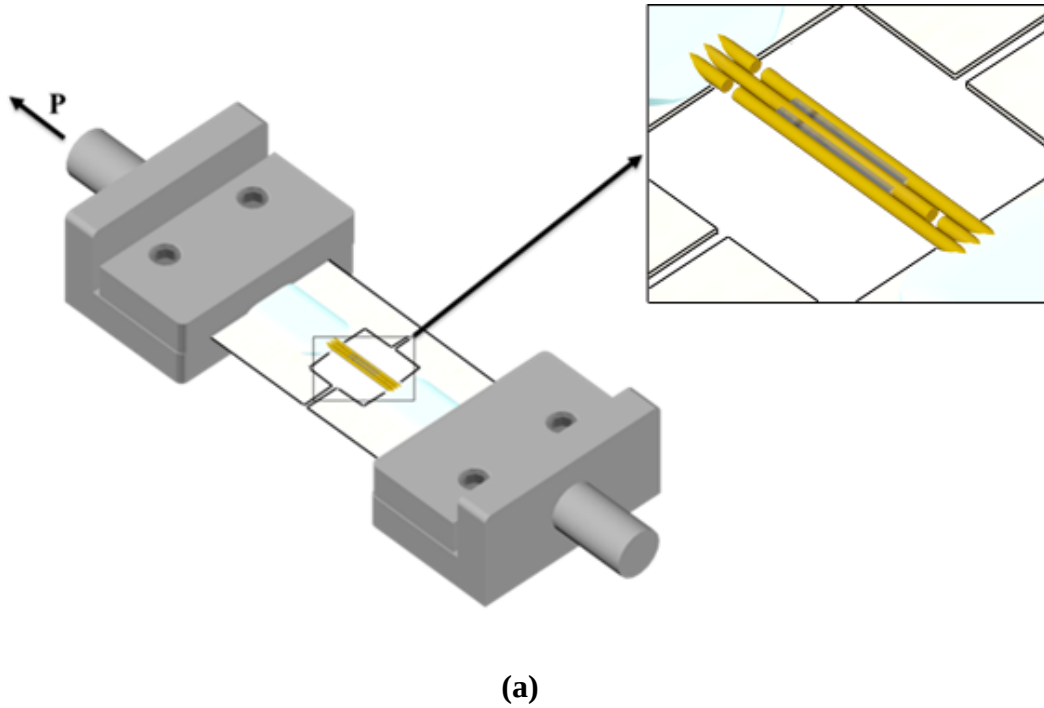
Table 4.1 Dimensions of the model composite specimens.

Dimensions	3 fibers model composite		4 fibers model composite
	0.5M, 2h	1.0M, 24h	1.0M, 24h
Fiber length, m	1510-3640	1370-3770	1190-4310
Fiber length, n	1490-3300	1240-3460	1210-3390
Fiber diameter, D	213-330	204-295	140-330
Fiber spacing, t	15-58	24-96	9-62
Fiber length, L	636-5814	237-5623	740-5250

* All units are measured in μm

4.2.3 Mode II test

Figure 4.4(a) and (b) respectively shows the schematics of the double shear test of the 3 fibers and 4 fibers model composites. Double shear test of the specimens were carried out by using a Tohei MT201 tensile test machine with a strain rate of 9 mm/min and load capacity of 50 N. The model composite mounted on the paper tab was carefully placed in the middle of 2 clamps where the center fiber is on the load pulling direction. The both sides of the marking line were carefully cut after tightening the clamps to prevent fracture before the test started. The values for the fiber fractures or the crack propagated at one side of the matrix length will be discarded.



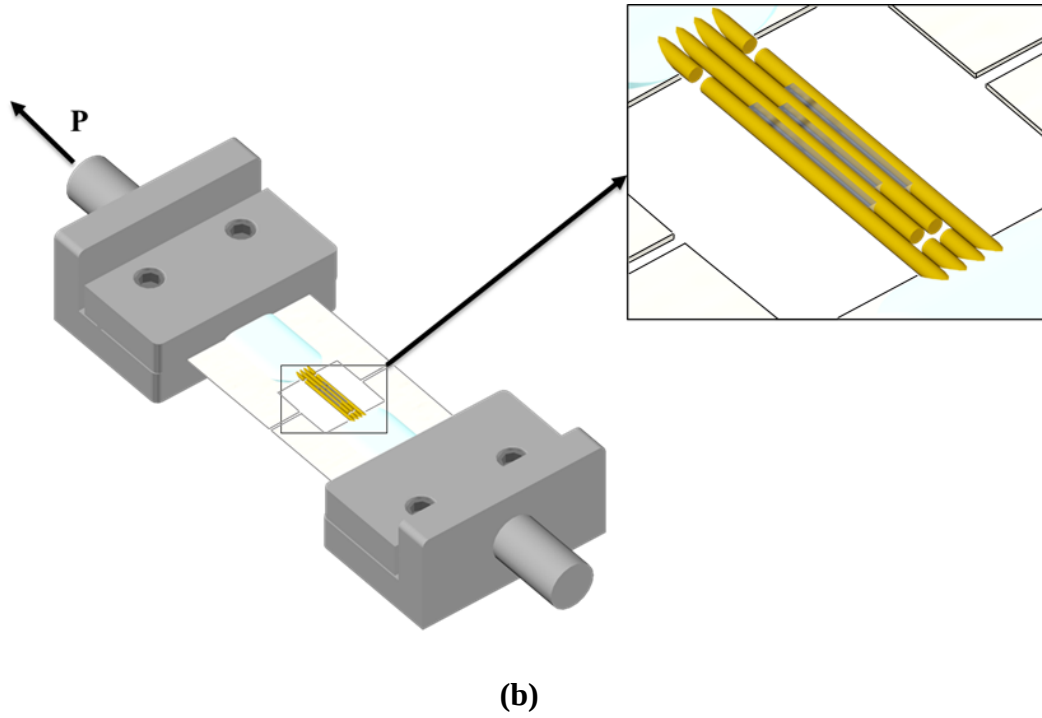


Figure 4.4 Schematic of double shear method for (a) 3 fibers model composite specimen and (b) 4 fibers model composite specimen.

4.2.4 Energy release rate

Figures 4.5(a) and (b) illustrates the schematics of the three fibers double shear model composite and the four fibers double shear model composite respectively. By simplifying the model composites into section (i), (ii) and (iii), the strain energy of the model composites can be obtained as below.

$$U = U_i + U_{ii} + U_{iii} \quad (1)$$

where U_i , U_{ii} and U_{iii} are the strain energy of the section (i), (ii) and (iii) in Figures 4.5(a) and (b). The equations of U_i , U_{ii} and U_{iii} are stated below.

Three fibers model:

$$U_i = \frac{2P^2n}{\pi ED^2} ; U_{ii} = \frac{2P^2L}{3\pi ED^2} ; U_{iii} = \frac{P^2m}{\pi ED^2} \quad (2)$$

Four fibers model:

$$U_i = \frac{P^2 n}{\pi E D^2} ; U_{ii} = \frac{P^2 L}{2\pi E D^2} ; U_{iii} = \frac{P^2 m}{\pi E D^2} \quad (3)$$

where $E = 4.95\text{GPa}$ [5], D is diameter of oil palm fiber, P is the applied force, L , m and n are the length of the section in Figures 4.5(a) and (b).

The energy release rate at crack tip A and B are given by the following equations.

Three fibers model:

$$G_A = \frac{1}{2\theta D} \cdot \frac{d}{dm} (U_i + U_{ii} + U_{iii}) = \frac{1}{2\theta D} \left(\frac{dU_{iii}}{dm} - \frac{dU_{ii}}{dL} \right) = \frac{P^2}{6\pi E D^3 \theta} \quad (4)$$

$$G_B = \frac{1}{2\theta D} \cdot \frac{d}{dn} (U_i + U_{ii} + U_{iii}) = \frac{1}{2\theta D} \left(\frac{dU_i}{dn} - \frac{dU_{ii}}{dL} \right) = \frac{2P^2}{3\pi E D^3 \theta} \quad (5)$$

Four fibers model:

$$G_A = \frac{1}{2\theta D} \cdot \frac{d}{dm} (U_i + U_{ii} + U_{iii}) = \frac{1}{2\theta D} \left(\frac{dU_{iii}}{dm} - \frac{dU_{ii}}{dL} \right) = \frac{P^2}{4\pi E D^3 \theta} \quad (6)$$

$$G_B = \frac{1}{2\theta D} \cdot \frac{d}{dn} (U_i + U_{ii} + U_{iii}) = \frac{1}{2\theta D} \left(\frac{dU_i}{dn} - \frac{dU_{ii}}{dL} \right) = \frac{P^2}{4\pi E D^3 \theta} \quad (7)$$

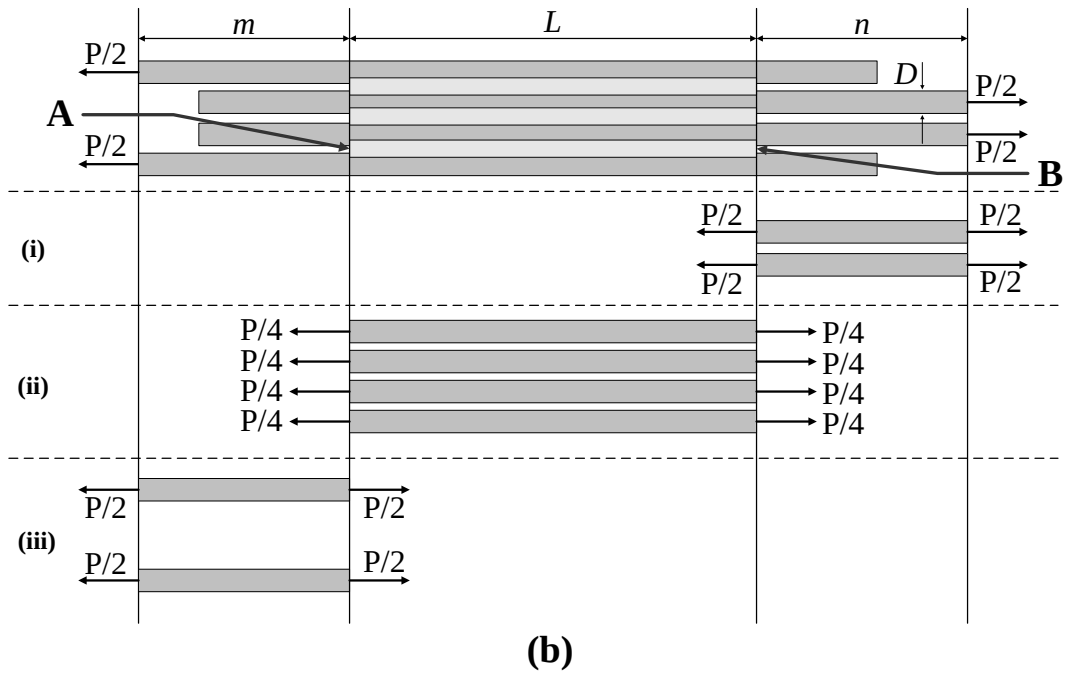
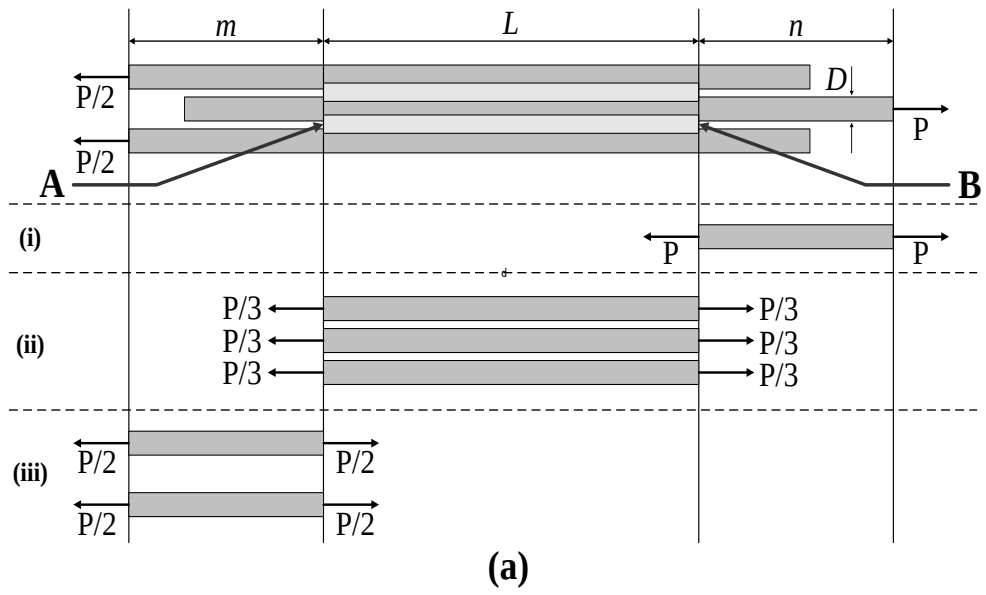


Figure 4.5 Schematics of double shear model composites. (a) 3 fibers model composite.
(b) 4 fibers model composite.

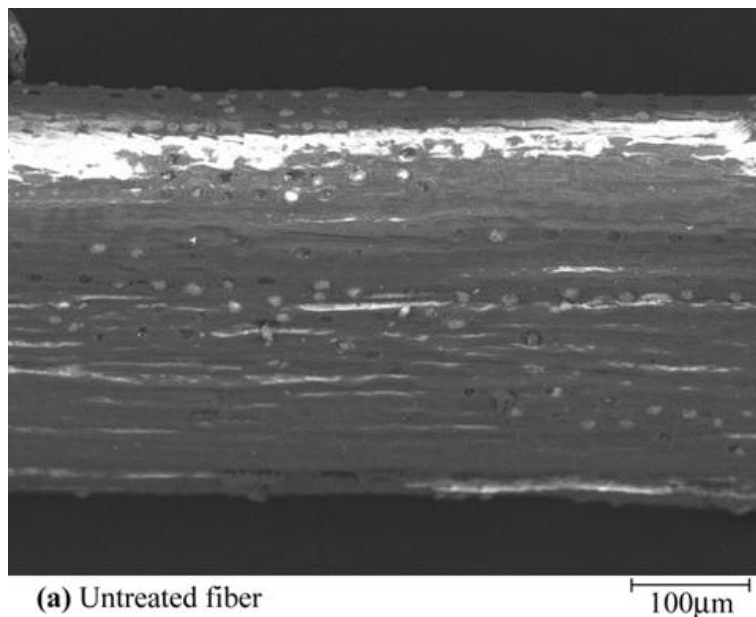
Since the matrix length is measured after the resin is cured, the both sides of the center fiber may have different matrix lengths. By considering one outer fiber bonded with minimum matrix length, $L_{\min}$ in section (iii) from the Figure 4.5(a) or (b) have the equilibrium of forces in longitudinal direction, the mean interfacial shear stress at the shortest interface of fiber/matrix with bonding area, $A = D\theta L_{\min}$ can be defined as:

$$\tau_i = \frac{P}{2D\theta L_{\min}} \quad (8)$$

4.3 Results and discussion

4.3.1 Surface morphology

Alkali treatment of oil palm fibers disrupts the chemical structures of OPF. This treatment also removes certain amount of impurities that cover on the external surface layer of OPF. As a result, the surface roughness of the fiber is improved [6-9]. Figures 4.6(a), (b) and (c) show the SEM micrographs of the untreated fiber, mild treated fiber and strong treated fiber respectively. As seen in Figure 4.6(a), the untreated fiber has smooth surface which covered by impurities materials. Hence, the specimens with very small bonding area were hardly fabricated. As mentioned in section 4.2.1, the untreated fibers were replaced with mild treated fibers to observe the alkali effect on the interfacial fracture toughness and critical shear stress of the model composites. As illustrated in Figures 4.6(b) and (c), the roughness of fiber surface increased when the NaOH concentration and the alkali treatment period were increased.



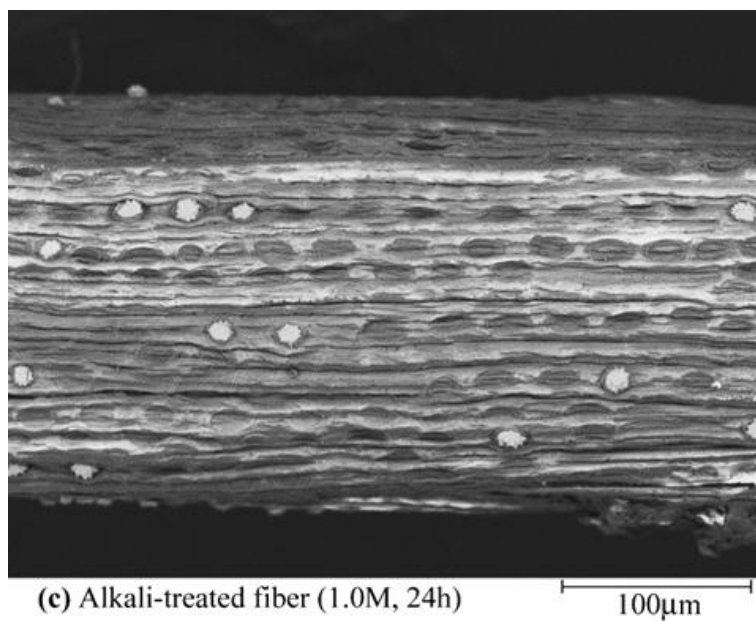
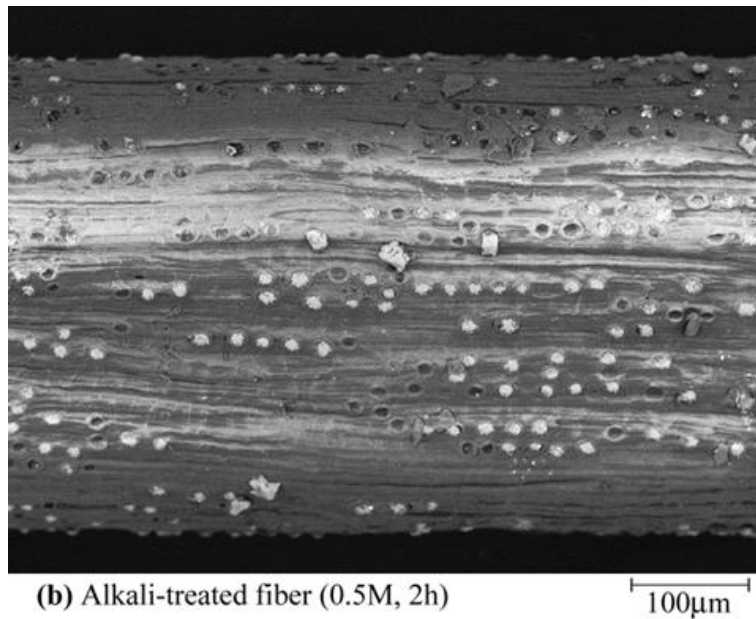


Figure 4.6 Scanning electron micrographs of oil palm fiber surface. (a) untreated fiber, (b) mild treated fiber (c) strong treated fiber.

Figures 4.7 and 4.8 show the micrographs of the fracture surface at the initial crack tips of the treated fiber/PLLA models. The surface of the fiber can be clearly observed from Figure 4.7(a) and the matrix was also found on the surface of the outer fiber (Figure 4.7(b)). Therefore, the crack was propagated along the interface between center fiber and matrix. From the experimental data, 31 out of 34 tested 3 strong treated fibers model composites and 18 out of 30 tested 4 fibers model composites were identified where the crack was propagated along the interface between center fiber and matrix.

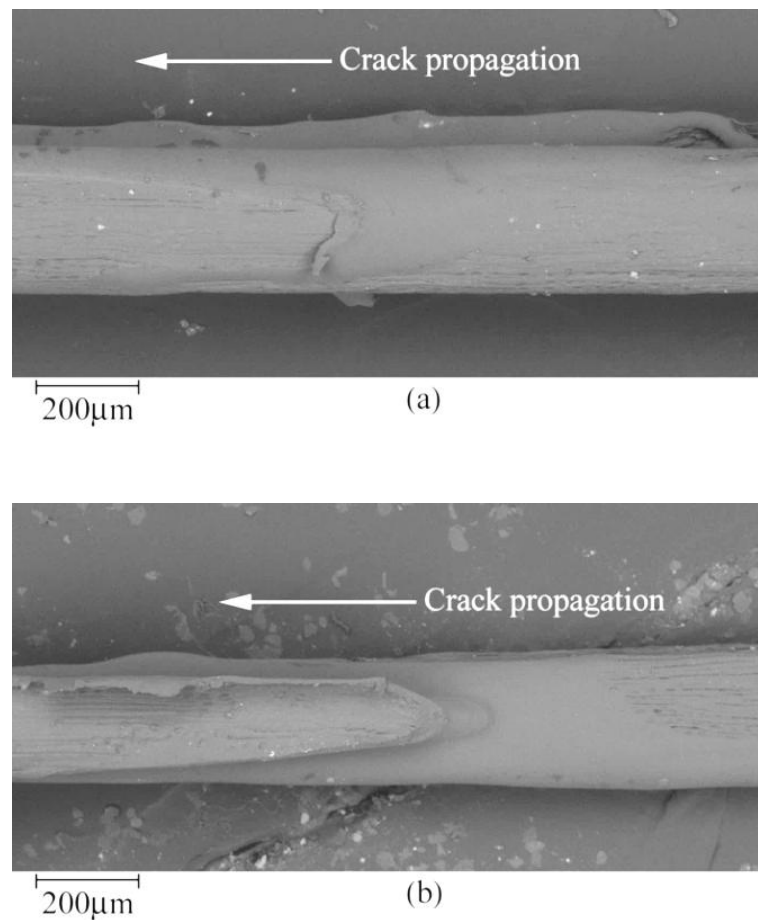


Figure 4.7 Fracture surface of the specimen (center fiber/ matrix interface). (a) Center fiber.
(b) Outer fiber.

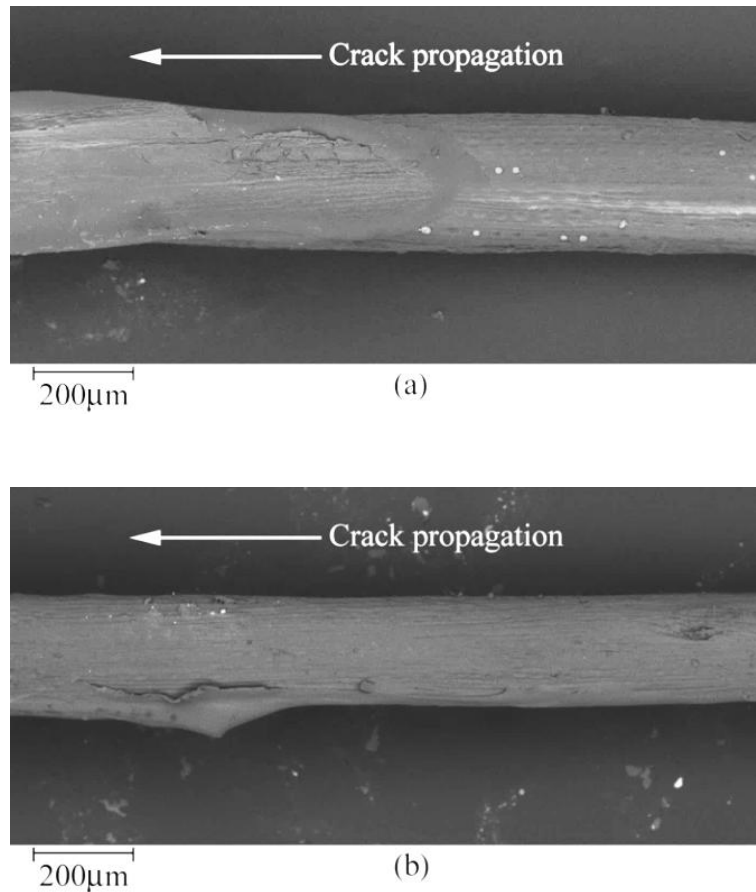


Figure 4.8 Fracture surface of the specimen (outer fiber/ matrix interface). (a) Center fiber.
(b) Outer fiber.

4.3.2 Interfacial fracture toughness of 3 fibers model composite

The plots of the interfacial fracture toughness of 3 mild treated and strong treated fibers model composite versus average fiber diameter, angle of bonding interface, average spacing between fibers and matrix length were illustrated in Figure 4.9, 4.10, 4.11 and 4.12, respectively. As seen from Figure 4.9, 4.10 and 4.11, the data points are randomly scattered. This indicates that the interfacial fracture toughness of the model composites does not affected by the average fiber diameter, angle of bonding interface, average spacing between fibers.

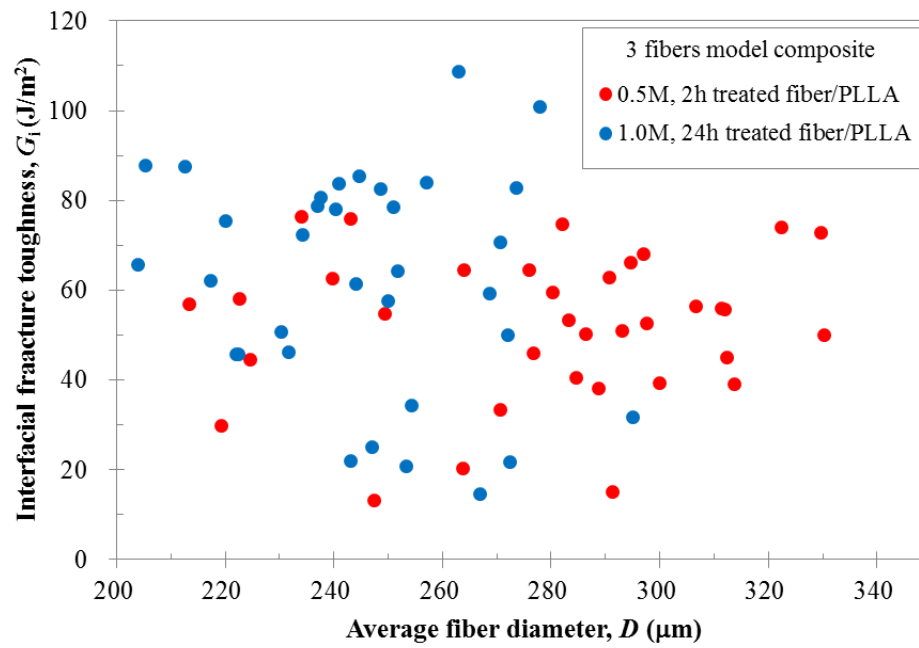


Figure 4.9 Plot of interfacial fracture toughness versus average fiber diameter.

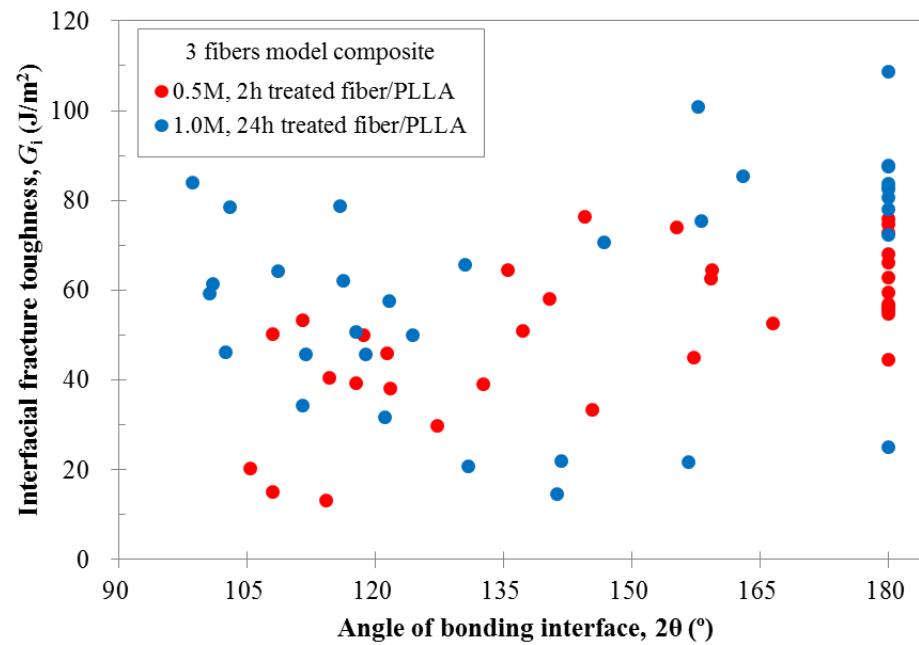


Figure 4.10 Plot of interfacial fracture toughness versus angle of bonding interface.

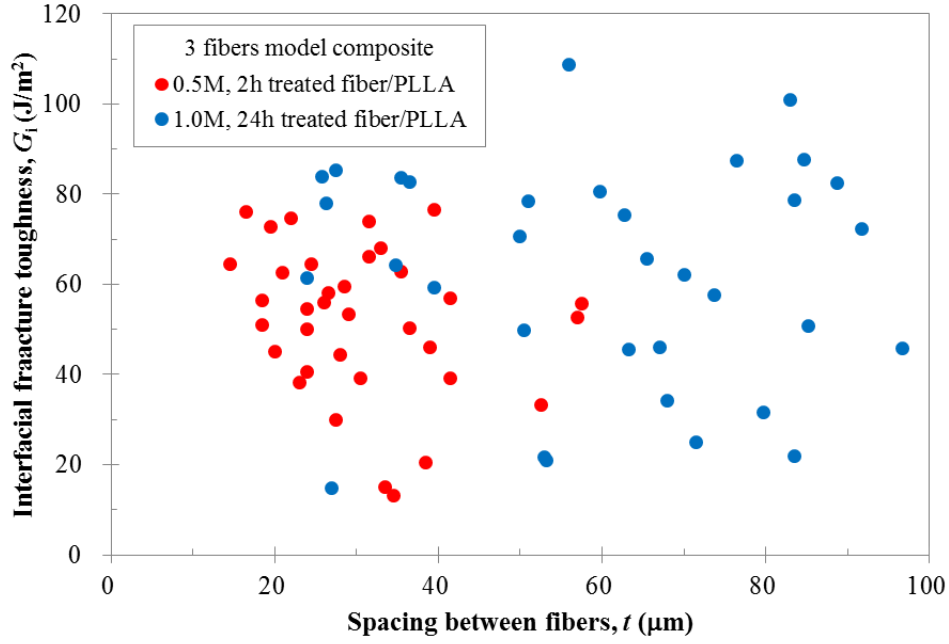


Figure 4.11 Plot of interfacial fracture toughness versus spacing between fibers.

In order to simplify the data analysis, the plot of interfacial fracture toughness versus matrix length as shown in Figure 4.12 is on a logarithmic scale with base 10 on y-axis. The large scatter in G_i in Figure 4.12 can be explained by following reasons: (1) The uneven distribution of the fiber strength, (2) The actual bonding area might be vary from the calculated bonding area, as the fiber was considered as a perfect round in data analysis. As seen in Figure 4.12, when $L < 2.5\text{mm}$, the G_i value of the 3 fibers model composite with mild treated fibers increases from 13.24J/m^2 to 75.98J/m^2 as the L increased. The G_i value is stable at $L > 2.5\text{mm}$. Hence, the data points corresponding to the matrix lengths ranging more than 2.5mm were used to calculate the average G_i value ($57.1 \pm 10.4\text{J/m}^2$).

The increase of interfacial fracture toughness, G_i with matrix length, L at small matrix lengths was attributed to the increase in volume of matrix for plastic deformation in response to shear. However, the interfacial fracture toughness leveling off when the plastic deformation at the crack tip no longer occupies uniformly the entire matrix length, but constraint at the vicinity at the crack tips with further increase in matrix length, L [10-12].

Same observation was found in 3 fibers model composite with strong alkali treated fibers. The G_i value of the 3 fibers model composite with strong alkali treated fibers generally increased with matrix length until a stable value was reach at matrix length about 2.35mm . The average G_i value obtained from the data points at $L > 2.35\text{mm}$ is $70.8 \pm 13.7\text{J/m}^2$, which is

13.7J/m² greater than the average G_i value of the 3 fibers model composite with mild alkali treated fibers.

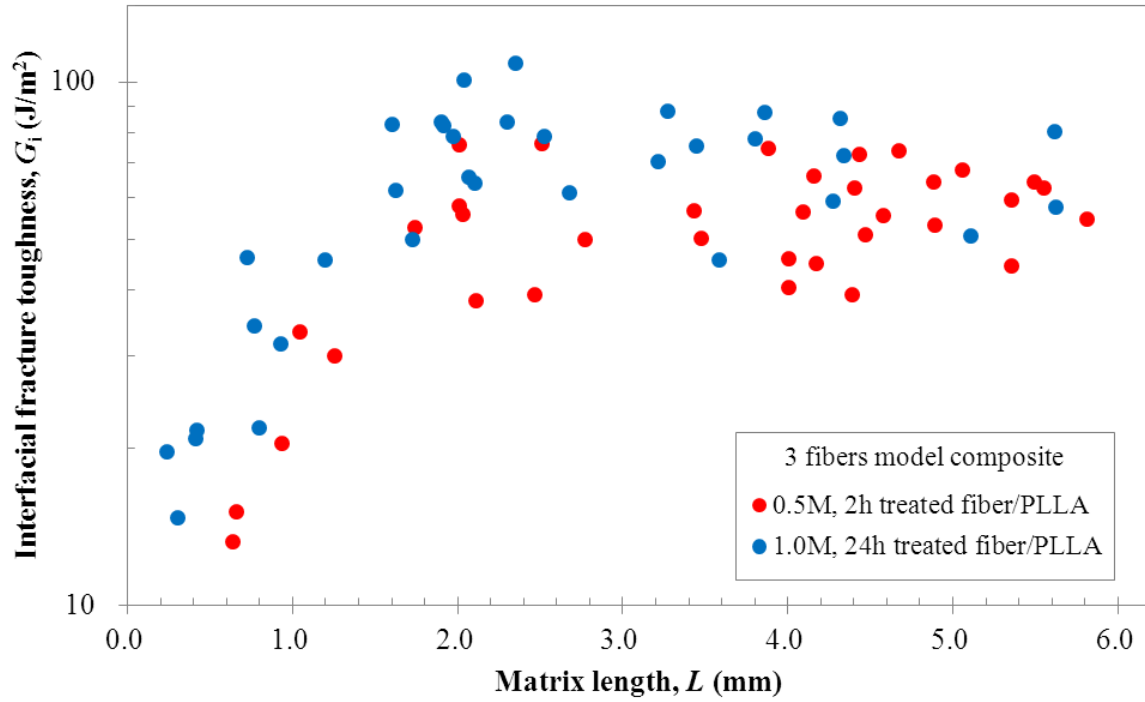


Figure 4.12 Plot of interfacial fracture toughness versus matrix length.

4.3.3 Interfacial fracture toughness of 4 fibers model composite

For 4 fibers model composite, the interfacial fracture toughness was also not affected by the average fiber diameter (Figure 4.13), angle of bonding interface (Figure 4.14), average spacing between fibers (Figure 4.15). Refer to Figure 4.16, for $0.66 < L < 2.29$ mm, G_i value of the 4 fibers model composite increases from 21.4J/m² to 102.4J/m² as the L increased. After that, the G_i value is stable with further increase in matrix length. For $L > 3$ mm, the average G_i value obtained from the data points is 73.6 ± 15.7 J/m². Figure 4.17 shows the comparison of the interfacial fracture toughness between 3 fibers and 4 fibers model composites. A slight difference between the average G_i value of the 3 fibers and 4 fibers model composites was observed.

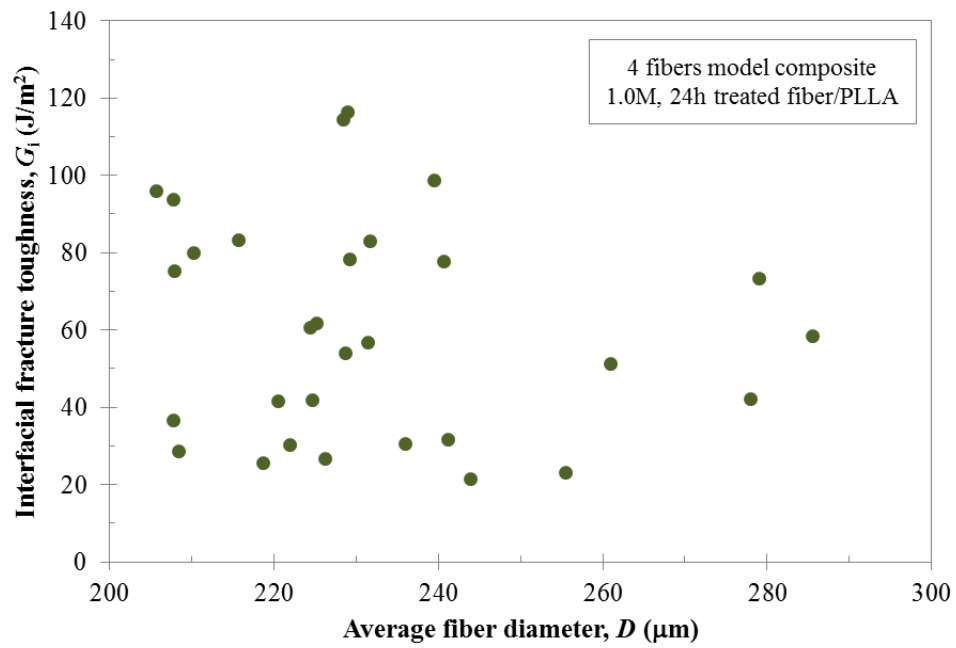


Figure 4.13 Plot of interfacial fracture toughness versus average fiber diameter.

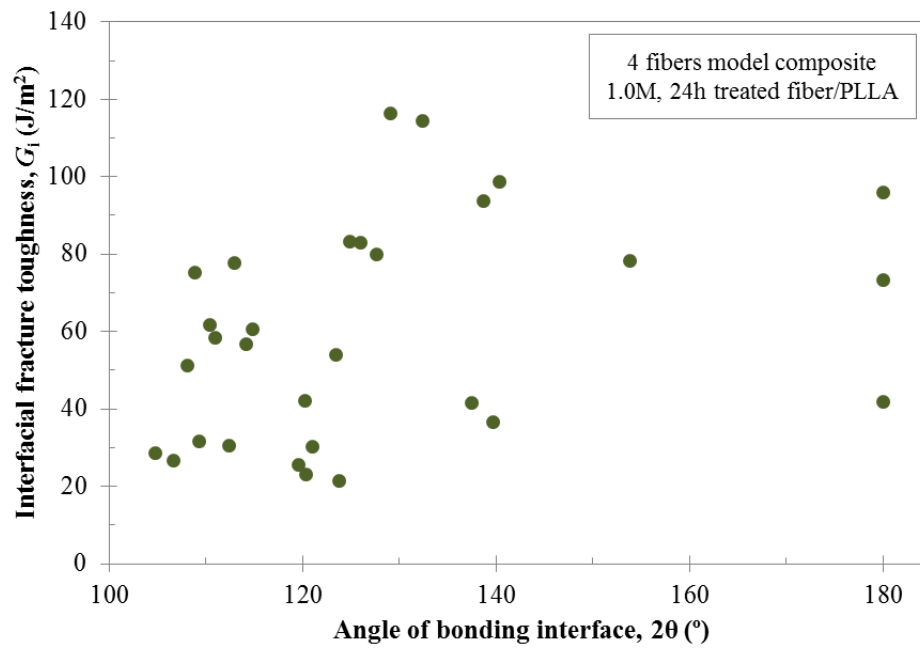


Figure 4.14 Plot of interfacial fracture toughness versus angle of bonding interface.

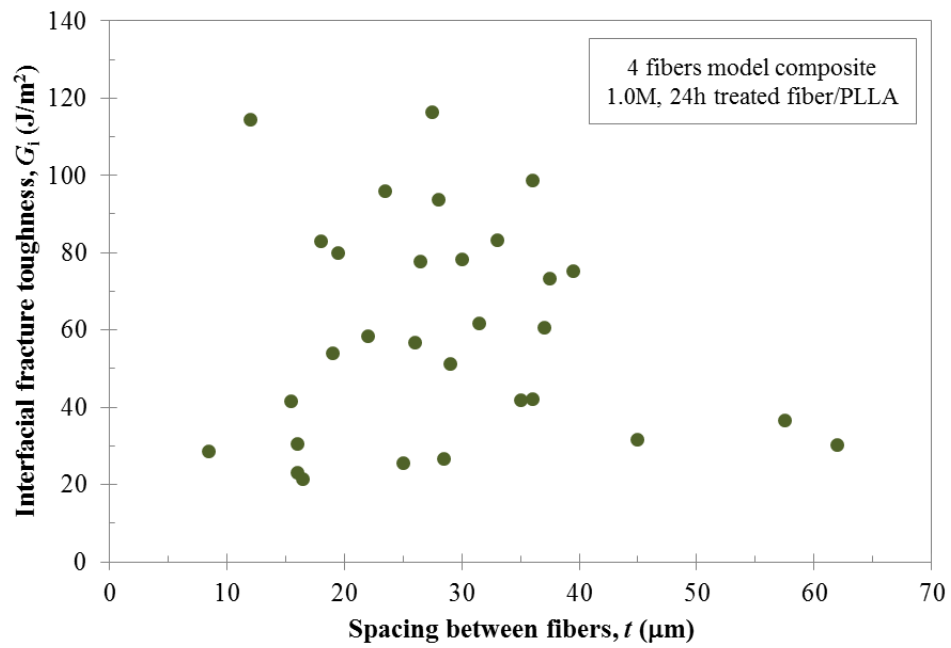


Figure 4.15 Plot of interfacial fracture toughness versus spacing between fibers.

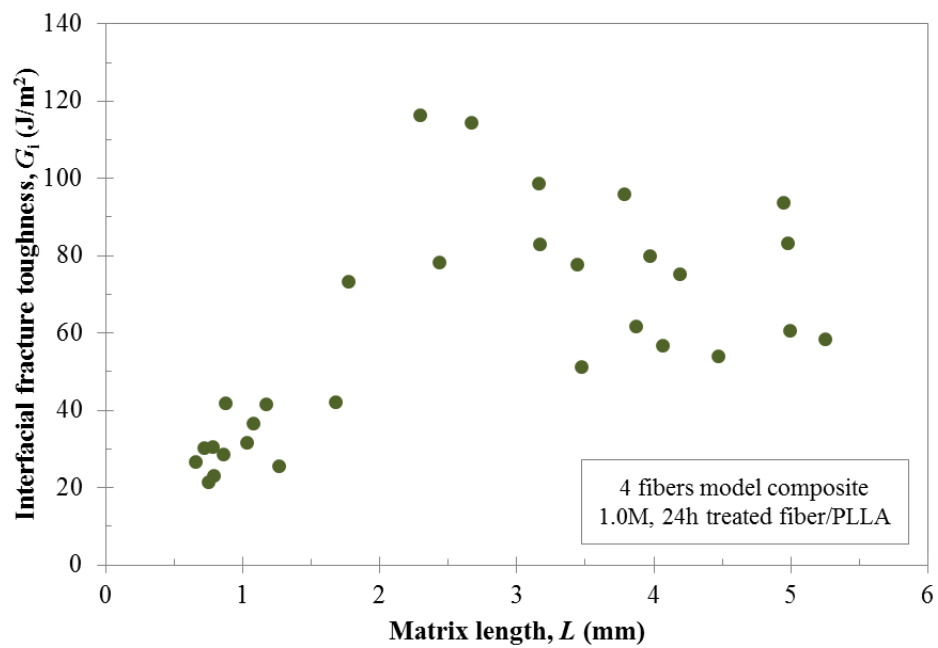


Figure 4.16 Plot of interfacial fracture toughness versus matrix length.

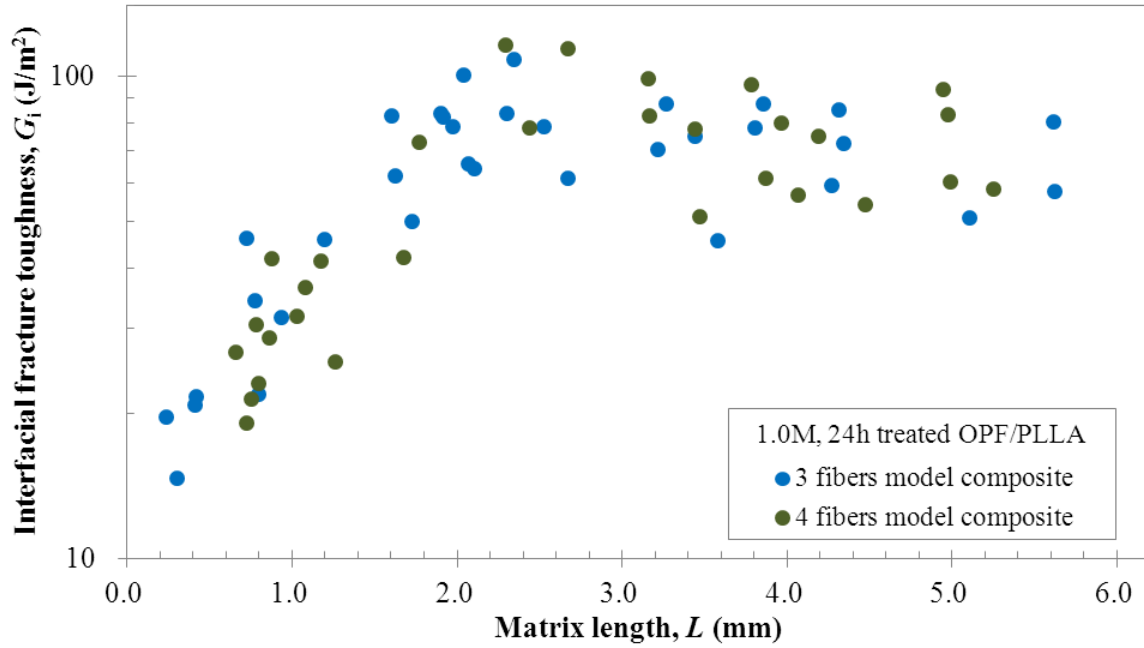


Figure 4.17 Interfacial fracture toughness of the 3 fibers and 4 fibers model composites at various matrix lengths.

4.3.4 Interfacial shear stress

Figures 4.18 and 4.19 present the relations between mean interfacial shear stress, τ_i and minimum bonding length, L_{min} . As illustrated in Figures 4.18 and 4.19, the mean interfacial shear stress, τ_i of the model composite decreased with increasing in minimum matrix length, L_{min} . Similar observations were found in other studies for kevlar fiber [13], glass fiber [14], carbon fiber [15], and steel wire [16] which bonded with either thermoplastic or thermosetting adhesives.

As seen in Figure 4.18, the τ_i values of the 3 fibers model composite have some slight differences with the τ_i values of the 4 fibers model composite, but in general data plots are in rather good agreement with the data plots obtained from 4 fibers model composite. Figure 4.19 compares the $\tau_i - L_{min}$ relation of the model composites with different alkali treatment conditions. The overall τ_i value of the model composite with mild alkali treated fibers is slightly lower than the τ_i value of the model composite with stronger treated fibers. As explanation above, the improvement of the mean interfacial shear stress is believed to be due to surface roughening by the alkali treatment. This promotes better interlocking between fiber and matrix and improves the interfacial adhesion.

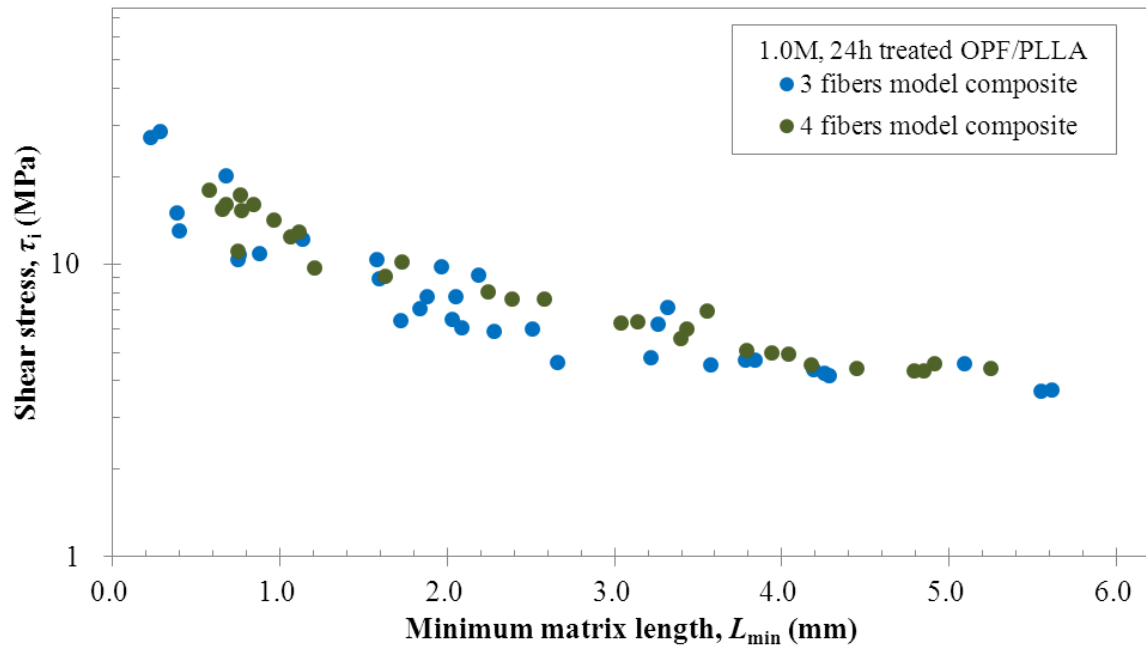


Figure 4.18 Mean interfacial shear stress versus minimum matrix length of the 3 fibers model composite and the 4 fibers model composite.

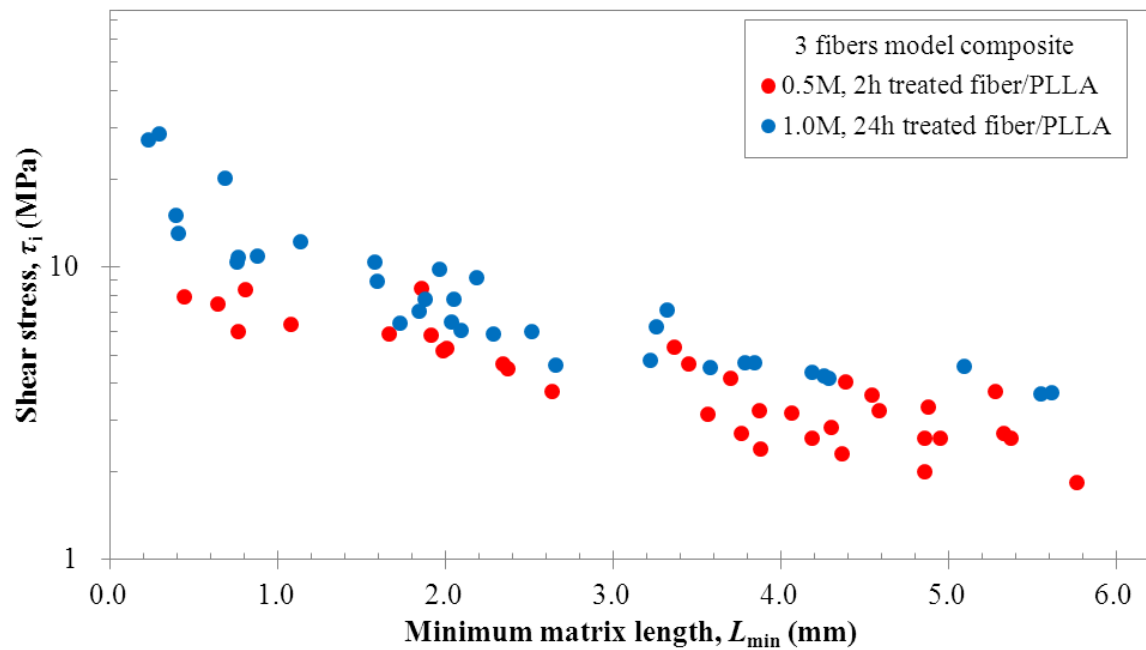


Figure 4.19 Mean interfacial shear stress versus minimum matrix length of the 3 fibers model composite with different fiber treatment conditions.

Considering the energy release rate and the interfacial shear stress of the model composites, the relation between G_A , τ_i and $L_{\min}$ can be expressed as below by substituting the Eq. (8) into Eq. (4):

$$G_A = \frac{2\theta}{3\pi ED} \tau_i^2 L_{\min}^2 \quad (4)$$

Therefore, from the above equation, $G_i \propto \tau_i^2 L^2$ and $\tau_i \propto \sqrt{G_i}/L$. As seen in Figure 4.20, the ideal relation between G_i , τ_i and $L_{\min}$ can be explained as follows. At very short matrix length, the stress is almost uniformly distributed at the whole matrix length, where the τ_i is constant. Therefore, G_i is proportional with L^2 ($G_i \propto L^2$). When the matrix length is long enough to obtain the constant value of G_i , the τ_i is inversely proportional to $L_{\min}$ at that stage. However, the cross point indicated by arrow A is difficult to identify from the experimental data due to the large scatter plots of experimental data.

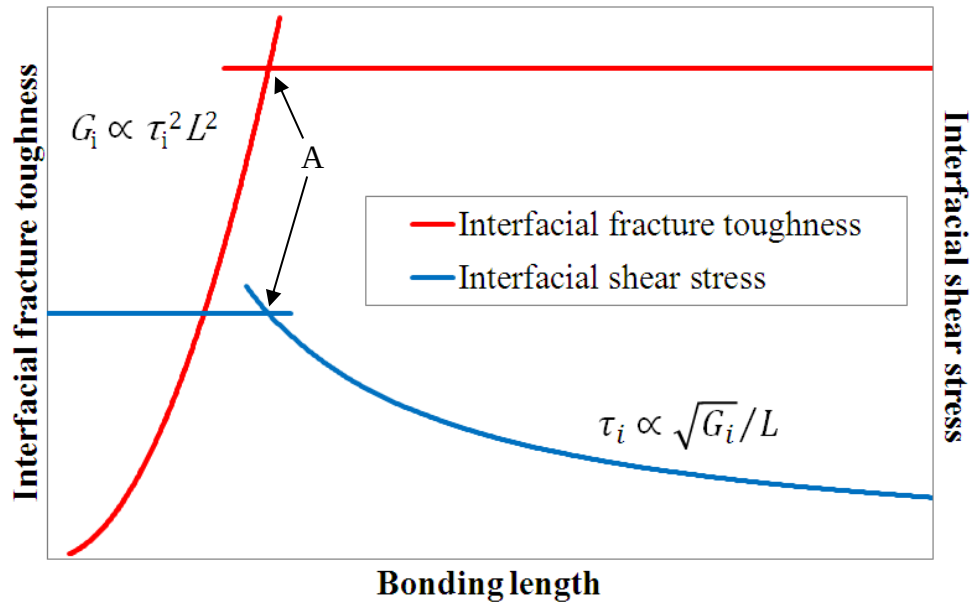


Figure 4.20 Relation between interfacial fracture toughness and interfacial shear stress.

4.4 Conclusion

The present work evaluated the interfacial fracture toughness and the interfacial shear stress of the treated oil palm fiber/ PLLA model composites. The following conclusions can be listed from this study:

1. The interfacial toughness of the model composites is independent on the fiber diameter, fiber spacing and angle of bonding. However, the interfacial fracture toughness of the model composites is dependent on matrix length.
2. For all tested model composites, the interfacial fracture toughness was increased with small matrix length and leveling off with further increase in matrix length.
3. The interfacial fracture toughness can be determined when the matrix length is more than 2.5mm and 2.35mm for 3 mild treated fibers model composite and 3 strong treated fibers model composite, respectively. Furthermore, the interfacial fracture toughness of the 4 fibers model composite can be obtained when the matrix length is more than 3mm.
4. The average interfacial fracture toughness obtained from the 3 fibers and 4 fibers model composite are 70.8 J/m^2 and 73.6 J/m^2 , which only show a slight difference. On the other hand, the average interfacial fracture toughness of the 3 mild treated fibers is 57.1 J/m^2 , which is lower than the average interfacial fracture toughness of the 3 strong treated fibers. This indicates that the interfacial fracture toughness of the model composites increase with increasing the concentration of alkali solution and treatment times.
5. The mean interfacial shear stress of the 3 strong treated fibers model composite is higher than the mean interfacial shear stress of the 3 mild treated fibers model composite at the same level of matrix length. This may due to the increase in surface roughness with increasing the alkali concentration and treatment times.

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Chapter 5

Conclusions

5.1 Conclusions

In this work, the mechanical performances of the oil palm fiber and their composites have been investigated. The tensile properties of the single oil palm fiber with various alkali treatment conditions were identified. The modified Weibull distribution was applied to predict the strength of the alkali treated fiber at various gauge lengths. The interfacial fracture toughness and the interfacial shear stress of the oil palm fiber/ poly (L-lactic acid) were also evaluated in the present work.

In the first study, an examination of the effect of NaOH concentrations (0.5, 1.0 and 1.5M) and treatment times (0.5, 1, 2, 4, 8, 12, 24 and 48 hours) for treating oil palm fiber has been conducted. The tensile strength and modulus of the oil palm fibers have been significantly improved by alkali treatment. Among the alkali treatment conditions tested, the maximum fiber strength and modulus can be obtained from the 1.0M alkali treatment for 24 hours at room temperature, which enhanced the fiber strength by 170% and fiber modulus by 240%. Clearing off the impurities and better packing of the cellulose chain were the main factors to improve the fiber tensile properties, as observed in FTIR and morphological studies. However, the tensile strength and modulus were decreased after reaching the optimum amount of alkali concentration and treatment time. This is because higher alkali concentration and treatment period can attack the main components of the fiber, resulting in weakening and damaging the fiber. The micrographs of the oil palm fiber at different treatment times were observed. The roughness of the fiber surface increases as the treatment time increases.

The second study conducted on this present work was to investigate the gauge length effect of the treated oil palm fiber. The tensile strength of the oil palm fibers at 10mm, 20mm, 30mm and 40mm gauge lengths was tested. The present modified Weibull model with incorporating conical frustum volume variation was used to compare with the conventional Weibull model and the modified model with integrating diameter variation. General observation, the fiber strength decreases with increasing the gauge length and the experimental data fit the Weibull plots very well. In addition, the conical frustum volume variation is more sensitive to gauge lengths compared with within diameter variation. The modified Weibull model with incorporating conical frustum volume variation can predict the gauge length effect more accurately compared with the conventional Weibull model and

modified Weibull model with incorporating the within-fiber diameter variation. In comparison of the scale effect of various natural fibers, the oil palm fiber was found to be less sensitive to gauge length than coir, banana and kenaf fibers.

The third study was to evaluate the interfacial fracture toughness and the interfacial shear stress of the oil palm fibers/poly (L-lactide acid) model composites. The oil palm fiber ranged from 190 μ m to 330 μ m was used as reinforcement and poly(L-lactide acid) was used as matrix to fabricate the model composite. The model composite specimens consists of three and four parallel treated fibers which bonded by melted poly(L-lactide acid). These composite models exhibited the fiber/matrix interfacial crack propagated with fracture in matrix. Thereby, the fracture characteristics of the fiber/matrix bonding interface can be successfully evaluated. In order to observe the alkali treatment effects on the interfacial toughness and the interfacial shear stress of the composite models, 3 mild treated (0.5M, 2h) fibers model specimens and 3 strong treated (1.0M, 24h) fibers model specimens were prepared. The mild treated oil palm fibers were used to replace the untreated oil palm fibers due to the difficulty of preparing the specimen, where the weak adhesive was found between untreated fiber and matrix. The matrix tends to separate from the fibers when the model composite was attached on the testing machine. From the experimental data, the interfacial fracture toughness does not affected by the fiber diameter, fiber spacing, and angle of bonding. However, the interfacial fracture toughness was depends on the matrix length. The increment of interfacial fracture toughness at very small bonding length was attributed to the increase in volume of matrix for plastic deformation in response to shear. The interfacial fracture toughness value becomes constant with further increase in matrix length when the plastic deformation at the crack tip no longer occupies uniformly the entire matrix length, but constraint at the vicinity at the crack tips. The interfacial fracture toughness of the 3 mild treated fibers and 3 strong treated fibers model composites can be determined respectively when the matrix length is more than 2.5mm and 2.35mm. For 4 fibers model composite, the interfacial toughness can be evaluated when the matrix length is more than 3.0mm. As expected, the mild alkali treated composite model has lower interfacial fracture toughness compared to the strong alkali treated composite model. On the other hand, the interfacial shear stress of the model composites was also studied. The interfacial shear stress values of the 3 fibers and 4 fibers model composites have some slight differences, but in general results are in rather good agreement for both model composites. Furthermore, the overall interfacial shear stress value of the mild alkali treated model composite is slightly lower than the interfacial shear stress value of the strong alkali treated model composite. This may due to the surface roughening by the alkali treatment.

As described above, the optimum chemical treatments condition of the oil palm fiber, Weibull analysis on gauge length effect of oil palm fiber and the interfacial fracture toughness of the oil palm fiber/poly(L-lactic acid) model composites was investigated. The optimum chemical treatment condition can be considered for industrial usage. The modified Weibull model with incorporating conical frustum volume variation can be used as a tool to predict the blast fiber strengths such as oil palm fiber. On the other hand, the interfacial fracture toughness and interfacial shear stress of the oil palm fibers can be evaluated by using double shear test method. This approach can be used to evaluate the adhesive behaviour between natural fibers and matrix since the bonding behaviour of natural fiber/matrix composites were often discussed in natural composites researches.

List of Publications

Refereed Journal:

1. M.S. Osman, C.V. Sia, “Tensile performance of hybrid polyester-based composite reinforced with glass and oil palm fiber in aluminium laminates,” AES Technical Reviews Part B: International Journal of Advances in Mechanics and Applications of Industrial Materials, Vol. 1, 2009.. pp. 77-82.
2. C. V. Sia, Y. Nakai, H. Tanaka, D. Shiozawa, “Evaluation on fracture mechanics of mode II treated fiber/PLLA based on real size model composite,” International Journal of Mechanical Engineering and Technology, Vol. 4(2), 2013, pp453-460.
3. C. V. Sia, Y. Nakai, D. Shiozawa, H. Ohtani, “Statistical analysis of the tensile strength of treated oil palm fiber by utilisation of Weibull distribution model,” Open Journal of Composite Materials, Vol.4, 2014, pp.72-77.
4. C. V. Sia, Y. Nakai, H. Tanaka, and D. Shiozawa, “Interfacial fracture toughness evaluation of poly(L-lactide acid)/ natural fiber composite by using double shear test method,” Open Journal of Composite Materials, Vol. 4, 2014, pp. 97-105.

Conference Proceeding:

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