ISSN 1996-3394

Asian Journal of Materials Science

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Asian Journal of Materials Sciences 1 (2): 45-52, 2009
ISSN 1996-3394

Effect of Surface Treatment and Gauge Length of Empty Fruit Bunch on its Morphological and Characteristic Strength Properties

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Abstract: The morphological, physical and mechanical properties of Empty Fruit Bunch (EFB) fiber modified by chemical treatments were studied. EFB fibers were treated with alkali (NaOH, 2%), alcohol (C_2H_5OH, 95%), silane (SiH_4) and natural rubber. The results showed that chemical treatment and gauge length affected fiber strength and fiber strength homogeneity. Treatment with alcohol showed the highest strength value of EFB fiber (88.9 MPa) and treatment with natural rubber provided highest homogeneity of fiber by the highest Weibull modulus value compared with other chemical treatments. The shorter the fiber gauge length, the higher the EFB fiber strength. The high Weibull modulus of 2.52 was the indication of the highly homogeneity of the 5 cm gauge length EFB fiber. However, highly characteristic strength of the untreated EFB fiber was obtained from 1 cm of gauge length. EFB fiber showed higher characteristic strength than polyester, indicating that EFB fiber is able as a good filler or reinforcement.

Key words: Empty fruit bunch, oil palm, waste, composites, Weibull modulus

INTRODUCTION

There is considerable worldwide interest in the potential for natural fiber in composite materials. The use of natural fibers especially in plastic composites is of particular interest because such fibers can serve as a good reinforce and/or filler for synthetic polymers to enhance certain properties while reducing material cost low density, high specific strength and modulus, relative non-abrasiveness, ease of fiber surface modification and wide availability. Among the natural fibers available, oil palm Empty Fruit Bunch (EFB) offers an interest possible utilization. Oil palm (*Elaeis guineensis*) is one of the major plantation commodities in Indonesia, which has a great contribution for national income from Crude Palm Oil (CPO) as the main product of oil palm fruit. In the end of 2007, the oil palm area in Indonesia is around 6 million hectares (Fazli, 2008) in which the total oil palm production will be estimated to be 18 million tons. If the palm oil industry dispose about 1.1 ton of EFB for every ton of CPO production, about 18 millions ton of EFB as one of the solid waste produced by oil palms mill are readily available in the end of the 2007 year. These EFB consist of high cellulose content and is of a potential natural fiber resources but its applications account for a small percentage of the total potential production. Many attempts have been done to use EFB for pulp and paper production, cellulose preparation, mushroom cultivation, compost and mulch. This utilization of oil palm EFB fiber is therefore an ecological and

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economical answer to the problem of waste disposal. Several studies showed that EFB of oil palm with the average of cellulose content of 14-20% (Ramli *et al*., 2002) has the possibility to be an effective reinforcement in thermoplastics and thermosetting materials (Karina *et al*., 2008) but due to the different properties between EFB fiber and resin, it leads to composites with improper final properties. EFB is hydrophilic, which lowers their compatibility with relatively hydrophobic polymer matrices (Saheb, 1999). Therefore, chemical treatments are considered in modifying the fiber surface properties to optimize the interface of fibers. Ramie and sanseveiria have been treated with alkali (Munawar *et al*., 2007). Pineapple fibers were modified with benzoyl peroxide (Lopattananon *et al*., 2006). In addition, the strength of composites is critically dependent on the fiber strength. Thus, the understanding of fiber mechanical properties is of importance. There is none of information available on the morphological, physical and mechanical properties of EFB fiber in different gauge length with chemical treatment. In this study, the EFB fibers were treated with alkali, alcohol, silane and natural rubber to evaluate their morphological, physical and mechanical properties so that the most suitable treatment for composite products can be predicted.

MATERIALS AND METHODS

Chemicals

Sodium hydroxide (Merck, pro analysis), alcohol 95% (purchased from BRATACO, domestic chemical shop), toluene di-isocyanate 98% (Merck) and silane (Dow Corning-Z-6020) were used as received. Natural rubber was of commercial grade with dry rubber content of 60%.

Fiber Preparation

Empty Fruit Bunch (EFB) of oil palm (*Elaeis guineensis*), was kindly supplied by PT Condong Garut Estate Crop, Garut, West Java. All EFBs were manually dismantled into virgin fiber then cut into 8-10 cm of length using a carding. The fibers were washed with water, dried at 70°C for 16 h.

Surface Treatment

Alkali treatment of 5 g EFB fibers was done in a boiling 2% of sodium hydroxide solution for 30 min, further washed with water and oven dried in an oven at 60°C for 24 h.

Five gram of dried EFB fibers were immersed with a mixture of 1% natural rubber solution and 1% toluene di-isocyanate for 5 h followed by decantation of rubber solution then oven dried at 60°C then dried.

Commercially silaneZ-6020 N-(β-aminoethyl)-γ-aminopropyl-trimethoxy-silane) solution was prepared by mixing silane 0.75% (active compound of 0.3%) with 60% ethanol. EFB fibers were immersed in this solution and were allowed to stand for 30 min. The ethanol/water mixture was then drained out and fibers were air dried then dried in an oven at 60°C until the fibers were completely dry.

EFB fibers were boiled in alcohol (95%)-water (1:1) solution of for 2 h and oven dried at 60°C.

Cross-Sectional Area Observation of EFB Fiber

The fiber cross-sectional area is an important parameter to determine the fiber tensile strength. The area for the fiber tensile strength is in practice calculated from fiber diameter measurements, recorded by optical microscopy (Inverted Optical Microscope Epiphot-TME, software: Omnimet) by the following equation:

$$A = \frac{\pi \bar{d}^2}{4} \tag{1}$$

Where:
d = Diameter
A = Cross section area

The fiber cross-sectional area determined by the above equation is especially for the circular shape of fiber cross section. Due to the cross sectional shape of EFB fibers was not completely circular, then the average of fiber diameters at several locations was made to reduce the errors in the mechanical strength evaluation.

Morphological Observation of Fiber

The morphology of the untreated and treated fibers was observed by using a Scanning Electron Microscope (SEM), JEOL T-330A at an accelerating voltage of 15 kv. Prior to examination, a surface of the specimen was mounted on metal stubs by double-faced tape and coated with a thin layer of gold (Au).

Specimen for Tensile Strength Test

Prior to tensile test, samples were conditioned at 23°C, RH 50% for a minimum 40 h before testing. Tensile test was performed on a Universal Testing Machine (Orientec UCT-5T) using 30 specimens at room temperature (23°C, 50%) at tensile speed of 3 mm min^{-1} with gauge length of 1, 3 and 5 cm each.

The statistical variability of the tensile strength of EFB fiber is described by the Weibull modulus. The Weibull modulus is a parameter used to describe the distribution of strength in materials which break at defects according to weakest link statistics. The tensile test gives us a load as a function of extension curve up to failure. Tensile strength was calculated as follows:

$$\sigma_{max} = F/A \tag{2}$$

Where:
F = Maximum tensile force to failure (N)
A = Cross section area of fiber before loading (m^2)

The probability of failure of EFB fiber is given by the following equation:

$$P = 1\text{-exp } [-V/V_0 (\sigma_0/\sigma)m] \tag{3}$$

Where:
σ = Failure strength
m = Weibull modulus
V = Tested material volume
V_0, σ_0 = Scaling constants (characteristic constants)
P = Probability

For Weibull plots, P = (I-0.5)/N was used to estimate fracture probability,

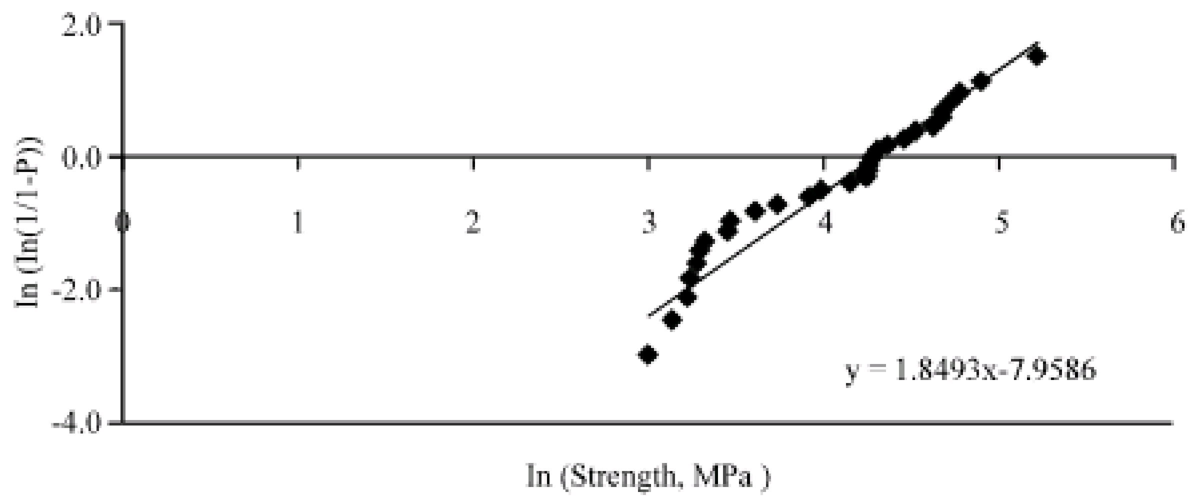

Fig. 1: Weibull plots for EFB Fiber tested at 1 cm gauge lengths

Table 1: Weibull modulus of EFB fiber at different gauge lengths and various chemical treatments

EFB-chemical treatment	Average of diameter (mm)	CV of diameter among specimens (%)	Average CV of diameter within specimens (%)	Average cross sectional area (mm^2)	Characteristic strength (MPa)	Weibull modulus (m)
Silane	0.494	22	12.8	0.200	81.15	2.84
Alcohol	0.466	20	10.8	0.177	88.90	2.92
NaOH	0.432	17	10.4	0.151	58.55	1.95
Natural rubber	0.429	17	12.1	0.149	68.95	3.56
Untreated 1 cm	0.425	18	11.5	0.148	73.92	1.85
Untreated 3 cm	0.448	21	11.5	0.162	58.68	2.22
Untreated 5 cm	0.444	21	14.8	0.161	59.01	2.52
Polyester					26.07	

Coefficient of variance: CV = (standard of deviation×100)/average

Where:

N = Number of fibers tested

I = Rank of strength for each fiber (1,2,3,..N)

By rearranging and taking the natural logarithm of both sides of the equation, the following Eq. 4 is obtained:

$$\ln (\ln(1/1-P))-\ln V/V_0 = m \ln \sigma - m \ln \sigma_0 \quad (4)$$

For a constant tested volume (gauge length for fibers), Eq. 4 is simplified to:

$$\ln(\ln(1/1-P)) = m \ln \sigma + k \quad (5)$$

Where:

$$k : -m \ln \sigma_o$$

The Weibull modulus can be determined by plotting ln (ln(1/1-P)) against ln σ as described in Fig. 1. With that method of determination, the Weibull modulus of the EFB fibers is summarized in Table 1.

This study was conducted in Polymer Test Laboratory, Division of New Material and Research Center for Physics, Indonesian Institute of Sciences, Bandung, Indonesia from April 2007 to March 2008.

RESULTS AND DISCUSSION

Micro-Structural Characteristic

Figure 2a showed the typical shape of EFB fiber cross-section taken by optical microscope. It clearly showed that cross-sectional shape of EFB fiber is non-circular. Based on these images, then, a consideration of circular shape must account for the cross sectional area calculation (Fig. 2b). Measurement of cross-sectional area was done from the average of diameter value. Each chemical treated fiber had an average co-efficient of variance for the diameter within the fiber varied between 10.8-14.8% which may show more or less rather uniform of diameter. The most uniform EFB fiber observed from alcohol treated fiber.

Morphological Observation of EFB Fiber

Surface morphology of EFB fiber was observed using SEM. Figure 3a showed SEM micrographs of the surface of an untreated EFB fiber while chemicals treated fibers can be seen in Fig. 3b-e. The observation of the fiber surface revealed that the EFB fibers were of aggregated micro-fibril. From the SEM micrographs (Fig. 3b) it showed that sodium hydroxide washed out lignin and makes micro-fibrils are visibly clear. By this alkali treatment, EFB fibers surface becoming rough. On the other hand, Fig. 3c and d showed fibers after treatment with silane and alcohol have not drastically changed compared with untreated fiber. Nevertheless, the surface of silane and alcohol treated fiber were slightly coarser than untreated fiber, indicating that some cementing materials that may present was mighty removed. As shown in the Fig. 3, micro-fibril of the fiber treated with silane and alcohol was slightly visible. Treatment with natural rubber (Fig. 3e) revealed thick coverage on the fiber surface. These

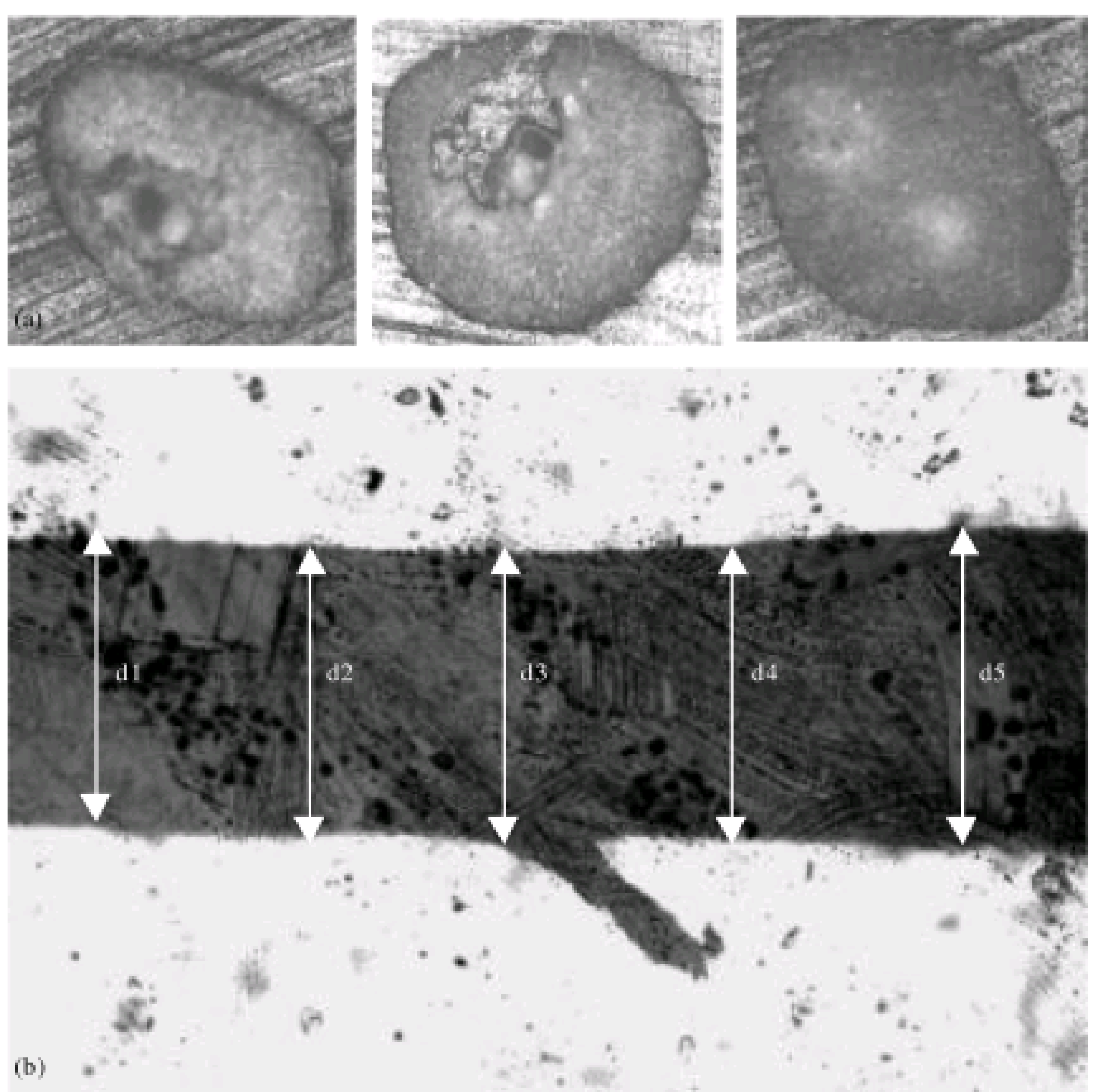

Fig. 2: (a) Typical shape of EFB fiber cross-section, (b) Cross section area calculation of EFB

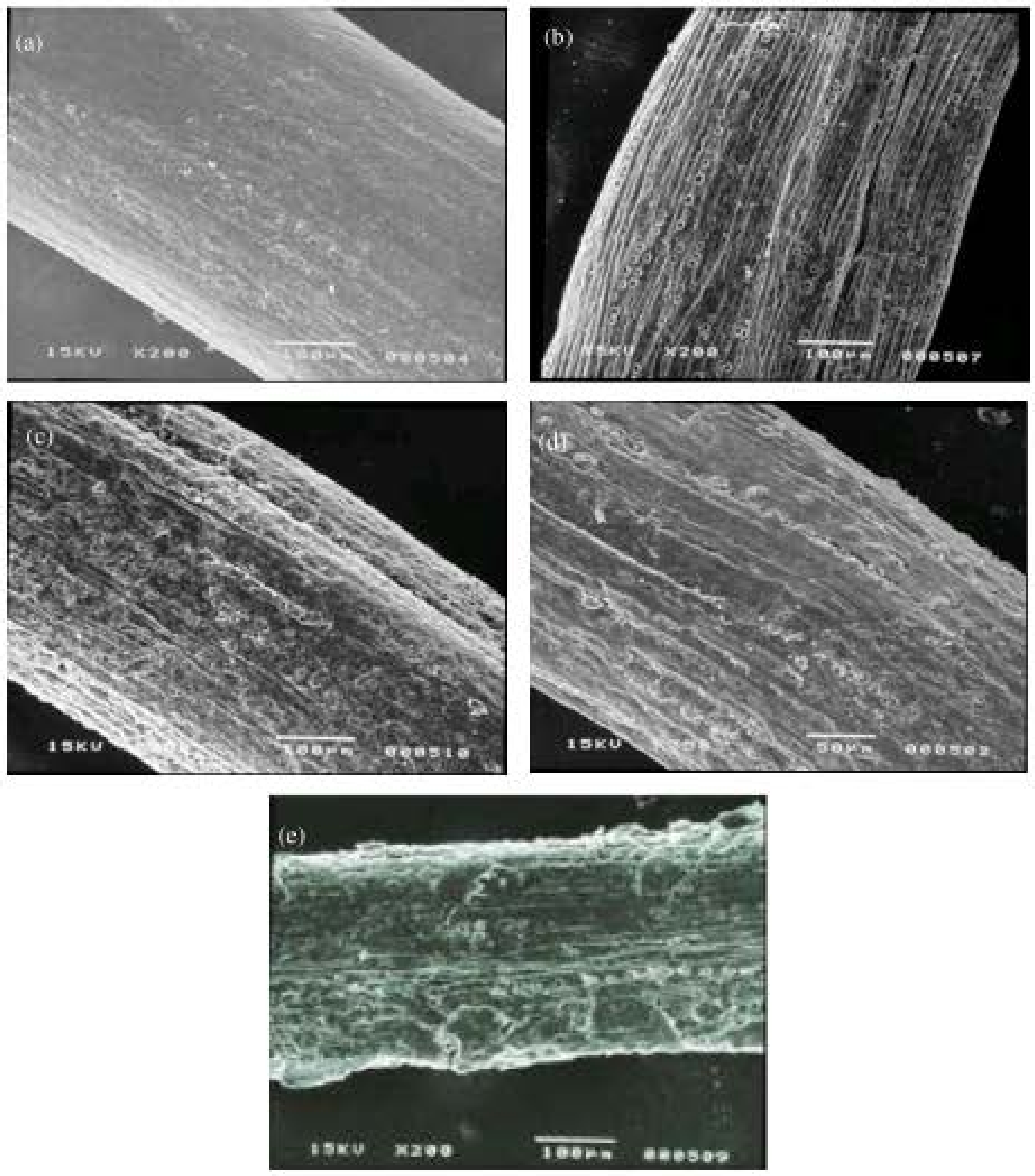

Fig. 3: (a) Untreated EFB, (b) EFB-2% NaOH, (c) EFB-Silane, (d) EFB-95% Alcohol, (e) EFB-natural rubber

rough and thin or thick layers on the EFB fibers are expected to support the mechanical interlocking when used in composites. On the other hand, the clean surfaces due to the alkali treatment is possibly provide direct bonding between the polymer or any of coupling agent and the cellulose micro-fibril.

Effect of Gauge Lengths on Fiber Strength

Table 1 showed the Weibull modulus and characteristic strength of EFB fiber at different gauge lengths and with various chemical treatments. From Table 1, it clearly showed that the longer the gauge length, the higher the Weibull modulus and the lower the characteristic strength. The lowest characteristic strength of EFB fiber was obtained at 5 cm gauge length. There is an increased probability of encountering more failure along the fiber length. Hence, the longer fiber, as indicated here, the lower the characteristic strength than shorter fiber. This result fit with the Weibull theory that the longer the gauge length, the higher volume of the fiber flaw which then caused the decreasing of its strength/characteristic strength. However, even though EFB at 5 cm gauge length gives the

Table 2: Lignin content after chemical treatment

Lignin Content after Chemical Treatment (%)	Values
Untreated EFB	25.5
Alkali 2%	21.2
Silane	19.3
Alcohol 95%	16.0

lowest value of its strength, this fiber had the most homogeneity among the other gauge lengths. The gauge length would affect the characteristic strength of EFB fiber. Table 1 also showed that characteristic strength of EFB fiber showed higher than that of polyester, indicating EFB fiber is able as a good filler or reinforcement.

Effect of Chemical Treatment on Fiber Strength

From Table 1, it can be seen that the chemical treatment increased characteristic strength of EFB fiber. Alcohol 95% produced the strongest fiber among the other chemical treatments with 88.9 MPa. On the other hand, alkali treatment resulted in the weakest strength of characteristic (58.5 MPa). The lowest fiber strength by the low concentration of alkali treatment can be probably attributed to the remaining residual lignin in the fiber. The cementing material such as lignin, pectin and hemi-cellulose removal can increase fiber strength. The dilute concentration of alkali was not able to remove lignin completely. Therefore, some amount of lignin remains in the EFB fiber and causes the low of fiber strength. On the other hand, alcohol treatment gives highest strength of EFB fiber, indicating the highest lignin removal compared with other chemical treatments (Table 2). Silane found to be effective in modifying natural fiber-polymer matrix interface and increasing the strength. From the results shown in Table 1, the silane treatment gives higher strength than that of alkali treatment. These results similar with that of earlier study (Valadez-Gunzales *et al*., 1999) that verified the interaction between silane modified fiber and the matrix was much stronger than that of alkaline treatment, which led to composites with higher tensile strength from silane treated than alkaline-treated fiber. From current results it showed that EFB fiber strength was higher than pinewood fiber, 40 MPa and rubber wood fiber, 15 MPa (Munawar *et al*., 2007) and higher than polyester strength, 29.7 MPa indicating that EFB fiber was able to use as filler or reinforcement in polyester composite. Natural rubber treatment showed the highest Weibull modulus, indicating the homogeneity of fiber strength. Covering fiber with natural rubber, empty fruit bunch fibers becoming ductile materials. Then, it fit to the theory that the more ductile the material, the higher the Weibull modulus and the more homogeneity the fiber strength.

CONCLUSION

The fiber strength was improved by chemical treatment and gauge length of the fiber. Treatment with 95% alcohol showed the strongest fibers. However, treatment with natural rubber provided the most uniform fiber than other chemical treatments. The shorter the fiber gauge length, the stronger the fiber strength. However, the longer the fiber gauge length, the more uniform the fiber. EFB fiber showed higher characteristic strength than polyester, indicating that EFB fiber is able as a good filler or reinforcement in polyester composite.

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