Mechanical Properties of Polypropylene Composite Reinforced with Oil Palm Empty Fruit Bunch Pulp

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ABSTRACT: In this study, oil palm empty fruit bunch (EFB) pulp was used as reinforcing agent in polypropylene composite. EFB pulp was prepared using soda pulping with different concentrations of sodium hydroxide (NaOH) solution. Overall, the tensile and flexural properties specifically the strength and the toughness showed improvement as the NaOH content in the treatment was increased. This was attributed to lower probability for EFB pulp to agglomerate and the production of higher aspect ratio pulp fibers. Scanning electron microscopy analysis showed evidence of the reduction in EFB bundles diameter after NaOH treatment. © 2010 Wiley Periodicals, Inc. J Appl Polym Sci 116: 1867–1872, 2010

Key words: oil palm empty fruit bunch; polypropylene; soda pulping; pulp

INTRODUCTION

The utilization of lignocellulosic material, such as wood or nonwood as a reinforcing component in polymer composites (thermoplastic or thermoset), has received considerable attention particularly for price-driven/high-volume application.[1–6] This development has been brought about by several advantages offered by lignocellulosic materials, such as low density, greater deformability, less abrasiveness to the equipment, and lower cost. Moreover, lignocellulosic-based fillers are derived from renewable resources. As can be seen by the recent trends, lignocellulosic materials have been the subject of intensive studies in producing fiber-reinforced plastic.[1–12] The increasing trend in using nonwood materials has been induced by the greater demand for light weight, high-performance materials coupled with the abundant supply of lignocellulosic materials, and escalating costs of raw materials and energy. As better understanding of the lignocellulosic-plastic interaction has been gained, the move to find substitutes for wood has been stimulated. With the increasing pressure on the forest industries, the necessity for the efficient use of the resources is vital, especially in wood composite industries. Hence, this would mean better conversion, that is, more cost-effective utilization, which is environmentally friendly, new technologies, and even the need to look for a substitute for wood.

One of the materials for this category, which is of great relevance to Malaysian scenario, is the large quantity of biomass generated from palm oil industry. Malaysia is one of the main palm oil producers in the world. The total land area of oil palm plantation is about 12% of the total land of Malaysia, which is about 3.87 million hectares. However, the oil consists of 10% of the total biomass produced in the oil palm industry. The remaining materials consist of abundance of lignocellulosic materials, such as empty fruit bunch (EFB), oil palm frond, and oil palm trunk. It is estimated that about 90 million tonnes of biomass are produced as a by-product from palm oil mill yearly.

However, the main obstacle to be resolved in producing a good lignocellulosic-thermoplastic composite, in terms of mechanical and physical properties, is the compatibility between the reinforcement material and polymer matrix. Many attempts have been carried out on using various types of chemical treatment to improve the interfacial properties.[1–4,7–13] One of the most popular treatments is alkaline treatment.[7–13] According to Bledzki and Gassan,[14] alkaline treatment of natural fiber would make the fibrils more capable to rearrange themselves along the tensile deformation. Such rearrangements among the fibrils would result in a better load sharing by them when the natural fibers are stretched and subsequently result in higher stress development in the fibers. As for lignin, when it is removed gradually, the middle lamella joining the ultimate cell is expected to be more plastic and homogenous because of the gradual elimination of microvoids. Mwaikambo and Ansell

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found that alkaline treatment produced natural fiber with rougher surface, which enhances the mechanical interlocking with polymer matrix. In addition, this treatment also produced natural fiber with higher crystallinity index, which results in stiff and strong fiber of the interest in the preparation of fiber-reinforced plastic.[15] Sreekala and Thomas had studied the effect of alkaline treatment on water sorption of oil palm fibers. They noticed that alkaline treatment had leached out the amorphous waxy cuticle layer of oil palm fibers.[16] According to Pothan and Thomas, natural fiber with higher crystallinity index was obtained when the alkali concentration was increased. This subsequently reduced the water sorption behavior of natural fiber.[17] Although alkaline treatment of lignocellulosic material enhances the properties of the thermoplastic-lignocellulosic composites, the study on soda pulping technique (a type of alkaline treatment) of lignocellulosic material in the preparation of composite is very much lacking. Hence, the aim of this study was to investigate the mechanical properties of thermoplastic lignocellulosic composites prepared from soda pulp. A lignocellulosic material from palm oil industry, that is, oil palm EFB, which consists of 80% of holocellulose and 17% of lignin, was treated with soda pulping technique, before being used as a reinforcing component in polypropylene (PP) matrix.

EXPERIMENTAL

Materials

Oil palm empty fruit bunch (EFB) was obtained from Sabutek (M) Sdn. Bdn., Teluk Intan, Malaysia. Polypropylene impact copolymer Titanpro SM950 was purchased from Titan Petchem (M) Sdn. Bhd and used as a matrix. Its density was 0.9 g/cm^3, and melt flow index was 60 g/10 min at 230°C. Sodium hydroxide (NaOH) was obtained from Systerm ChemAR, Malaysia.

Soda pulping technique

The pulping process was carried out using a digester with model IBSUTEK ZAT 92. EFB was cooked at different levels of NaOH concentration levels varied from 5 to 30%, with EFB/liquor ratio of 1 : 10 and 0.1% anthraquinone at 170°C for 2 h. The pulp was then refined using hydropulper, washed with water, and dried before it was used for compounding process.

Fiber length determination

The pulp obtained from the soda pulping process was subjected to fiber length measurement using FAS-3000 Optical Fiber Analyzer. A small amount of the pulp (~ 0.02 g) was immersed in water before being injected to the instrument for fiber length measurement.

Kappa number measurement

The kappa number of the pulp produced was conducted according to TAPPI standard T 246. An amount of 25 mL of 0.1N $KMnO_4$ was premixed with 25 mL of 4.0N sulfuric acid before being mixed with the pulp, which had already been disintegrated in water. Subsequently, the mixture was stirred using a magnetic bar for 5 min before 5 mL of 1.0N potassium iodide was added into the mixture. The mixture was then titrated with 0.1N sodium thiosulfate ($Na_2S_2O_3$) solution. A few drops of starch indicator (0.2%) were added to the mixture when the color turned light yellow. The titration was stopped when the mixture color became white. The volume of $Na_2S_2O_3$ solution used for the titration was measured. A blank test was carried out with the same procedure without pulp, and the kappa number can be obtained using equation below:

$$K = \frac{p \times f}{w},$$

where $p = (b - a)N/0.1$ (weight of 0.1N permanganate in specimen test), b = volume of $Na_2S_2O_3$ in blank test, a = volume of $Na_2S_2O_3$ in specimen, N = normality of $Na_2S_2O_3$, K = kappa number, w = oven dry weight of the specimen, and f = correction factor of different percentage of permanganate consumed.

Compounding and processing

The proportion of EFB pulp and PP was varied by weight basis (EFB/PP = 40/60, 50/50, and 60/40). Compounding of the materials was conducted with Haake Rheodrive 5000 (drive unit) and Haake Rheomix 600 with a roller blade (mixer). The mixing was carried out at 180°C for 20 min with a rotor speed of 25 rpm. The compound was then placed into a mold with a dimension of 170 mm × 170 mm × 3 mm (length × width × thickness). The mixture was preheated at a temperature of 180°C for 10 min, followed by hot pressing at the same temperature for 10 min at a pressure of 8 kgf/cm^2. Then, it was cooled for 5 min under the same pressure.

Mechanical test

Tensile test was conducted on samples with dimensions of 150 mm × 19 mm × 3 mm (length × width × thickness) according to ASTM D 638 using Instron Testing Machine model 5582, with a cross head

TABLE I
Average of Fiber Length of Pulp Prepared from Different Concentration of NaOH

% NaOH	Average fiber length (mm)
0	0.53 (±0.05)
5	0.50 (±0.03)
10	0.51 (±0.03)
15	0.48 (±0.05)
20	0.48 (±0.04)

speed of 5 mm/min. Flexural test was also conducted according to ASTM 790 with a sample dimension of 150 mm × 15 mm × 3 mm (length × width × thickness) using Instron Testing Machine model 5582 with a cross head speed of 2 mm/min. The properties were reported based on six measurements for each composite system for each test.

Scanning electron microscopy

The tensile fracture surfaces of the composite samples were studied with scanning electron microscopy (SEM, Leica Cambridge S-360, UK). The samples were mounted on an aluminum stub with double-sided tape and then gold-coated with a Polaron SEM coating unit (UK) to prevent electrical charging during the examination.

RESULTS AND DISCUSSION

Table I shows the average fiber length of pulp prepared after being treated with different concentrations of white liquor. It is noticed that no significant difference in fiber length was evident. Most of the fiber length is about 0.50 mm. From the kappa number tabulated in Table II, the lignin content in the pulp is found to decrease when the percentage of NaOH in white liquor is increased. This phenomenon is anticipated as NaOH can remove lignin from lignocellulosic material.

Figure 1 depicts the effect of percentage of NaOH content in white liquor and pulp loadings on tensile strength of the EFB pulp-PP composites. Generally, tensile strength increases as NaOH content is increased. In this study, the lignin content in EFB pulp was found to reduce with increasing NaOH

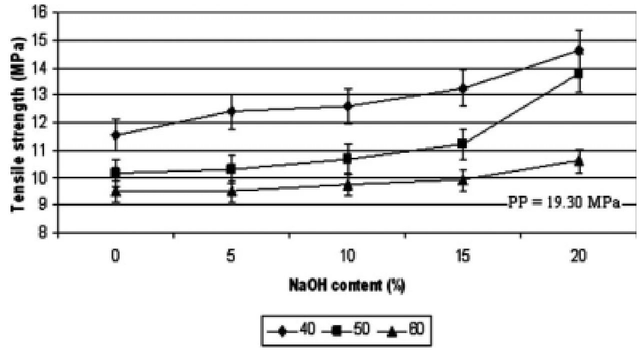

Figure 1 The effect of white liquor NaOH content and EFB pulp content on tensile strength of EFB pulp-PP composites.

content. As lignin acts as a natural adhesive binding together microfibrils in the cell wall, hence, the dissolution of lignin as a result of NaOH treatment could have loosen the microfibrils and producing high aspect ratio fibers. This would lead to better dispersion and enhanced stress distribution in the matrix.[7] Obviously, Figures 2–4 show that the NaOH treatment has reduced the diameter of the fibers. This supports the statement given above where the dissolution of lignin has led to split the fiber bundles into smaller size fibers. It can be seen that the diameter of the fibers is reduced as the NaOH concentration is increased. At 20% (Fig. 4), there are cases where fibers are in platy form because of the collapse of cell wall structure as a result of lignin dissolution. The aspect ratio of pulp is expected to increase as the percentage of NaOH in white liquor is increased because of two main reasons: (i) no significant difference in the length of the fiber prepared from different concentrations of NaOH, as observed in Table I, and (ii) diameter of the fiber decreased as the percentage of NaOH is increased. These explain

TABLE II
Kappa of Pulp Prepared from Different Concentration of NaOH

% NaOH	Kappa number
0	52.0 (±2.7)
5	46.3 (±3.4)
10	18.2 (±1.6)
15	13.5 (±2.3)
20	11.2 (±2.2)

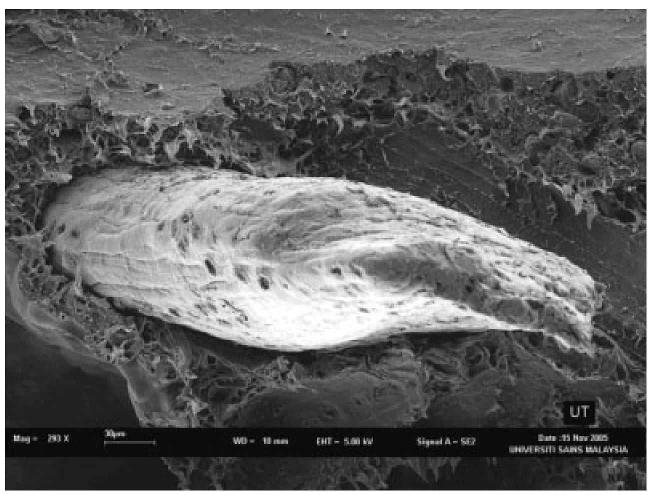

Figure 2 Fracture surface of composite prepared from untreated EFB.

Figure 3 Fracture surface of composite with EFB pulp prepared from 10% NaOH soda pulping.

the trend observed where a higher aspect ratio of fiber produced composite with a higher tensile strength.

As for the effect of EFB pulp loading on tensile strength of the composites (Fig. 1), it can be seen that the strength decreases as the pulp loading is increased. This observation is expected as the pulp content increases, the amount of PP matrix decreases resulting in insufficient of polymer matrix to hold the pulp. Because of the bulky and fluffy characteristics, the pulp is not well embedded in the matrix. This could have affected the efficiency of stress transfer from the matrix to the pulp. This result is inline with previous studies on other lignocellulosic-thermoplastic composites,[7–10] where the composites strength decreases as the overall fiber content is increased. This could be due to the fact that the lignocellulosic material used in this study is in the form of pulp, where aspect ratio of the pulp may play a major role. Natural fiber with higher lignin content on its surface may have the tendency to agglomerate through the formation of hydrogen bonding.[7] The agglomeration may then produce inhomogeneity in the matrix, which subsequently creates stress concentration points in the composite.

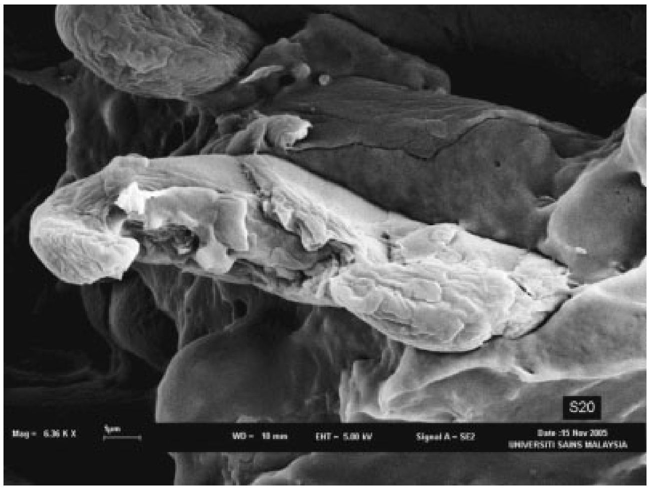

Figure 4 Fracture surface of composite with EFB pulp prepared from 20% NaOH soda pulping.

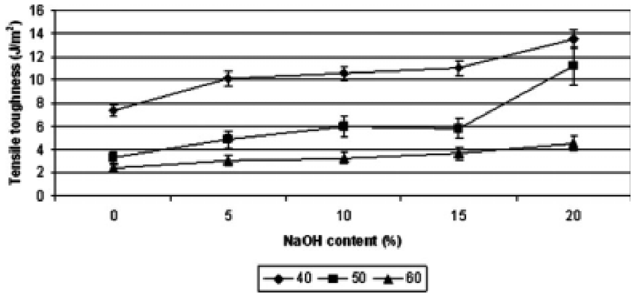

Figure 5 The effect of white liquor NaOH content and EFB pulp content on tensile toughness of EFB pulp-PP composites.

Figure 5 shows the effect of white liquor NaOH content and EFB pulp content on tensile toughness of EFB pulp-PP composites. It can be seen that the toughness of the composite increases as the NaOH content is increased. These results are similar to those of tensile strength. As the toughness of a sample indicates energy required to break the sample, the result shows that more energy is needed to break the composite with EFB pulp produced with higher percentage of NaOH. This may be attributed to: (i) lower possibility of EFB pulp to agglomerate as more lignin has been removed when higher percentage of NaOH was used during pulping process and (ii) the production of higher aspect ratio fibers as a result of lignin dissolution. However, tensile toughness of the composites decreases as the percentage of EFB pulp is increased. This relates to stress concentration point created by loosely embedded EFB pulp in the matrix because of the insufficient polymer matrix to hold the pulp.

Figure 6 depicts tensile modulus of EFB pulp-PP composites prepared from different EFB pulp content and pulp prepared from different percentage of

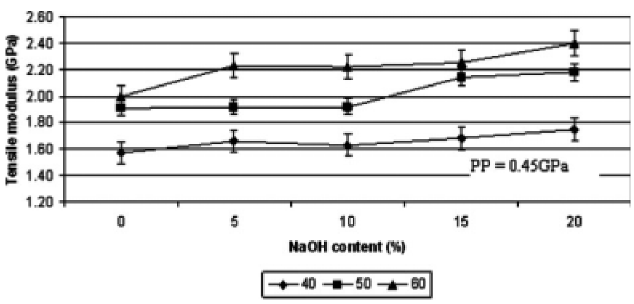

Figure 6 The effect of white liquor NaOH content and EFB pulp content on tensile modulus of EFB pulp-PP composites.

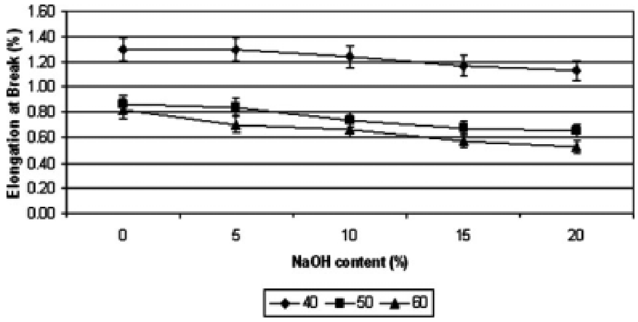

Figure 7 The effect of white liquor NaOH content and EFB pulp content on elongation at break of EFB pulp-PP composites.

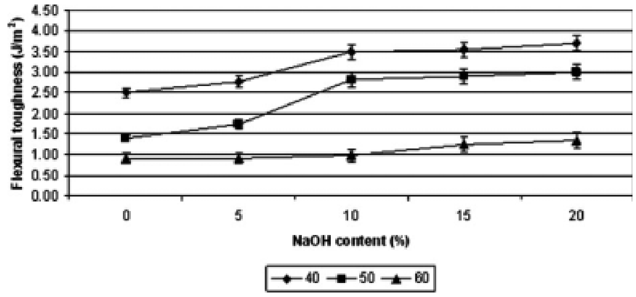

Figure 9 The effect of white liquor NaOH content and EFB pulp content on flexural toughness of EFB pulp-PP composites.

NaOH in white liquor. The stiffness of the composites increases as the white liquor NaOH content is increased. According to Bledzki and Gassan,[14] natural fiber treated with NaOH will lead to a decrease in spiral angle and increase its molecular orientation. Hence, the stiffness of the natural fiber is expected to increase with increasing natural fiber molecular orientation degree. Native EFB consists of around 30% of crystallinity, which is believed to be contributed by cellulose in native EFB.[18] Thus, higher percentage of NaOH in white liquor produces pulp with higher percentage of crystallinity and hence increases the rigidness of the pulp produced. As for the effect of EFB pulp content, the result is contrast to those observed in tensile strength and tensile toughness results. Tensile modulus of the composites increases as the EFB pulp content is increased (Fig. 6). It is expected because lignocellulosic in general has its inherent stiffness, which is higher than the matrix. This inherent stiffness adds up with the stiffness of the matrix, resulting in higher modulus. This is in agreement with the result shown in Figure 7, which depicts the effect of white liquor NaOH percentage and EFB pulp content on the elongation at break (EB) of the EFB pulp-PP composite. The incorporation of EFB pulp into polymer matrix drastically reduces the EB. Reduction in EB is due to the decreased deformability of a rigid interface between EFB pulp and PP matrix. As the elongation is reciprocal to the stiffness of a material, thus, the results show that higher percentage of EFB pulp and EFB pulp produced from white liquor with higher percentage of NaOH impart a greater stiffening effect when compared with those produced from lower percentage of EFB pulp content and EFB pulp prepared from white liquor with lower percentage of NaOH. This is in agreement with the trend observed in other studies on lignocellulosic-filled thermoplastic composites.[7]

Figures 8–10 show flexural results of the composite prepared from different EFB pulp content and pulp produced from white liquor with different percentage of NaOH content. From the results obtained, it can be clearly seen that the trend of the results is similar to those observed in tensile test. Hence, the same explanations presented in tensile test apply to the flexural test.

CONCLUSIONS

Incorporation of EFB pulp into PP matrix had resulted in the increment of tensile and flexural properties. This had been attributed to the lower probability of EFB pulp to agglomerate and the production of higher aspect ratio fibers as the percentage of NaOH in white liquor was increased. From

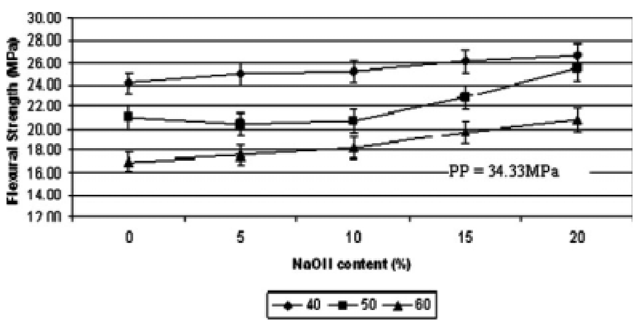

Figure 8 The effect of white liquor NaOH content and EFB pulp content on flexural strength of EFB pulp-PP composites.

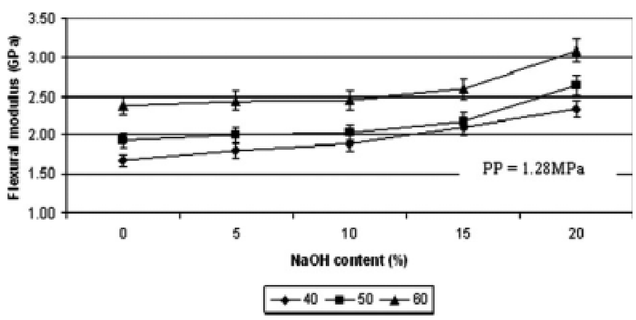

Figure 10 The effect of white liquor NaOH content and EFB pulp content on flexural modulus of EFB pulp-PP composites.

SEM studies, it was obvious that the size of pulp reduced as the percentage of NaOH in white liquor was increased. This produced better distribution of EFB pulp in the PP matrix, which subsequently enhanced the mechanical properties of the EFB pulp-PP composites. In addition, it was believed that the crystallinity of the EFB pulp as the result of NaOH treatment had contributed to the strength of the composites.

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