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EFFECT OF ALKALI TREATMENT ON MECHANICAL AND PHYSICAL PROPERTIES OF OIL PALM FROND-POLYPROPYLENE MATRIX

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***Abstract-* The main purpose of modifying the oil palm frond surfaces is to remove the cementing materials that tied up the crystallite rigid cellulose fibrils. This intention is to overcome the drawbacks of poor particle dispersion and to reduce the particle agglomeration in the polypropylene matrix. This paper investigates the effects of alkalization treatment on oil palm frond and its performance on the flexural, tensile and impact properties and water absorption of the composites. The experimental procedure involved four concentrations of Sodium Hydroxide on two different oil palm frond particle sizes. The findings obtained in this study show better properties in the treated samples and the interpretation is supported by the chemical analysis conducted.**

Keywords – Oil Palm Frond; Polypropylene Matrix; Flexural; Tensile; Impact; Water absorption; Sodium Hydroxide

I. INTRODUCTION

Five types of oil palm biomass are produced by oil palm plantation. They are oil palm fronds (OPF), oil palm trunks (OPT), empty fruit bunches (EFB), mesocarp fibres and palm kernel shell. In 2010, Malaysia's palm oil industry has generated about 80 million dry tonnes of solid biomass. This number is forecasted to increase to 85-110 million dry tonnes by the year 2020. From the statistics, 46-49% of the solid biomass is reported as oil palm frond [1]. This percentage may result in Malaysia producing about 55 million dry tonnes of oil palm frond by 2020. The massive amount created by the oil palm fronds is because they can be obtained each time of harvesting the fresh fruit bunches which is twice a month, during the tree pruning and tree replanting.

In supporting the zero burning system implied today, the cut fronds are left at the plantation to be natural fertilizers. However the residues may take a long time to be biodegraded and this will cause a disturbance to the oil palm plantation activities. To concern about this matter, this study investigates the opportunity of OPF to be filler in polypropylene matrix to be promoted in plastic industry. Two main problems may have the tendencies to be reduced which are 1) to wisely utilize the leftover OPF at the plantation and 2) to reduce the consumption of petroleum based plastic that later contributing in reduction of plastic products price.

II. LIGNOCELULOSIC PLASTIC COMPOSITE

The term of lignocellulosic is used to distinguish this composite from wood plastic composite (WPC). This is because OPF is categorized as a non-woody material, having slightly different properties and thus cannot be called as wood. Lignocellulosic plastic composite can be defined as a material made by a combination of petroleum derived polymers and bioresource [2]. According to [3], the composite consists of a matrix, which is continuous and surrounds the filler, provides the reinforcement and resulting in good performance properties of both matrix and filler. Of all the thermoplastic matrices available, polypropylene shows the most potential benefits when combined with natural fibres in making composite for industrial applications [4]. Thus, polypropylene matrix is chosen to be filled in with the oil palm frond.

III.ALKALIZATION TREATMENT

Wood and lignocellulosic fibre consists of cellulose, hemicellulose and lignin as the main components. The amorphous hemicellulose and lignin tied up together to encapsulate the cellulose so that the cellulose will be crystallized rigid and packed together [6]. They are generally called as cementing materials and often highly cross-linked to each other that act as a structural rigidity to the fibre cell wall itself [7].

However, to fabricate a thermoplastic composite, the presence of the cementing materials becomes a cause of a blockage for a good interfacial adhesion between the filler and polymer matrix. Fibre agglomeration that happened with traces of micro voids and large fibre pull-outs from the composite is believed affected by the blockage [8][9].

Sodium hydroxide (NaOH) is a suitable alkali to be used in order to eliminate those blockages to provide more hydroxyl groups on the fibre surface. This is because NaOH has a great tendency to degrade the cementing materials and thus will

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improve the particle dispersion [10]. During the alkali treatment, hydroxyl groups of the cementing materials may interact with the NaOH approached to them [5]. The interaction results in the cellular structure to be destructed and split from each other into filament forms.

$$\text{Bagasse-OH} + \text{NaOH} \leftrightarrow \text{Bagase-O}^{-}\text{Na}^{+} + H_2O \quad (1)$$

The separated fibrils then caused the interfibrillar region to become less dense and less rigid. This situation may contribute in letting the fibrils to be capable enough in rearranging themselves and thereby resulting in better load sharing [11]. As the hemicellulose and lignin are attached with the NaOH, the filaments left have more exposed surface to facilitate the chemical reaction between the cellulose and phenolic resin that later contributing in better stress transference at the interface [12]. In general, dissolving a part of cementing materials may result in possibility of interfacial interaction between fibre and matrix, increment of the effective surface contact with the matrix and also to make the load transfer between the fibre and the matrix to be possible [6].

IV. MATERIALS AND METHOD

The oil palm fronds were obtained from a local oil palm tree plantation at Universiti Teknologi MARA, Pahang. The matrix polymer is homopolymer polypropylene matrix in the form of pellets with a melt flow index of 33g/10min at 230^{o}C and melting temperature of 163^{o}C were supplied by local manufacturer.

Alkalization treatment involved a procedure of immersing the oil palm frond particles in NaOH solution with concentration of 1, 2 and 4% at ambient temperature of 30^{o}C for 30 minutes. Immersion procedure was conducted in mechanical machinery called as rotary digester for a good alkali distribution towards the particles. Particles were put turn by turn with NaOH solution into the chamber of the digester to ensure the whole particles got immersed in the alkali solution. The stainless steel caps were tightened up to secure the temperature inside the chamber and to avoid the mixture from leaking. Once the immersion procedure was done, the treated particles were immediately washed thoroughly with water until the colored residue disappeared and pH number turned to 7. After neutralized the treated particles, they were oven dried at a temperature of 80^{o}C until the particles completely dried prior to compounding process.

The compounding process was done at 10wt% OPF content at 180^{o}C in a dispersion mixer. The composite panel was hot pressed at 1000psi in 360 seconds with temperature of 180^{o}C followed by cold press at 1000psi in 60 seconds. The composite was then conditioned in a room with relative humidity of 50±5 % for at least 24 hours. Preparation of specimens tested was done according to ASTM D 790-92 for flexural test, ASTM D 638M-91a for tensile test, and ASTM D 570-81 for water absorption.

V. RESULTS AND DISCUSSION

VI. CHEMICAL COMPOSITION

Table 1 shows the effect of alkali treatment resulted in a decline trend of all the chemical components analysis which are extractive, lignin and cellulosic components.

Table 1 Effects of alkali treatment on chemical properties

Alkali	Extractive	Lignin	Holocellulose	Hemicellulose
%	%	%	%	%
0	5.50	23.86	75.65	26.24
1	2.22	21.56	69.68	19.53
2	1.91	21.44	68.96	18.13
4	1.53	20.09	68.68	14.09

Extractives tend to give a distraction to fabricate the lignocellulosic thermoplastic composite. The removal of extractives is very necessary in order to give spaces for the polymer matrix to interact with the natural filler. Compatibility between lignocellulosic material and polymer matrix in thermoplastic composite will be better with the removal of extractives which by means of better wettability [13]. This is because the extractive can block the cells in fiber when they diffused to the surface. As a result, the contact between matrix and the fiber will be reduced and thus poor adhesion happened at the interface [14]. Alkali treatment in this study seems to give an apparent decrement of extractive content which is approximately 72% at 4%NaOH to be compared with untreated oil palm frond.

Beneficial behaviors of lignin towards the fiber itself do impede fiber orientation in composite which will affect the load transfer and mechanical strength of engineered product [15]. Thus, in order to apply lignocellulosic as a raw material in thermoplastic product, lignin is necessary to be removed. In this study, lignin tends to dissolve with the treatment approached but not as great as the other chemical components reduction. This is because lignin has a characteristic of less soluble in solvents as to be compared with hemicellulose and pectin [16].

Holocellulose consists of cellulose and hemicellulose. As the cellulose increases affected by the alkali treatment, the hemicellulose then will be decreased. Hemicellulose has amorphous and hydrophilic characteristics which tend to absorb water [17]. This nature makes the hemicellulose to be the most desirable to be reduced to minimize the water absorption to the final product of filled in plastic. In this study, alkali treatment shows a positive impact on the chemical components prior to composite fabrication. Almost 50% reduction of hemicellulose can be observed on the 4%NaOH treated oil palm frond.

VII. EFFECTS OF PARTICLE SIZE

Table 2 shows the Duncan Multiple Range Test (DMRT) analysis on the effects of particle size.

Table 2 Effects of particle size

PS	FMOR	FMOE	TMOR	TMOE	Elong	Impact	WA
250 µm	40.95 b	2271 a	20.70 b	2616 a	6.40 a	3.51 b	0.23 b
425 µm	43.53 a	2251 a	22.14 a	2472 a	6.42 a	4.11 a	0.34 a

Note. PS, particle size

Note. FMOR, modulus of rupture; FMOE, modulus of elasticity; TMOR, tensile strength; TMOE, tensile modulus; Elong, elongation; WA, water absorption

Effects of particle size on the alkali treated is observed to have significant difference at its flexural strength, tensile strength, impact strength and water absorption with better result by 425µm particle size. The particle size is also observed to be significantly positive correlated with the tensile strength at r=.194 as shown in Table 3. This findings may be attributed to positive effect played by large particle size which by means of high aspect ratio. Aspect ratio in this case can be interpreted as a ratio of fiber length to fiber thickness. Long fibers generally have oriented flow along the fiber which then contribute in improving the mechanical properties of the fiber itself compared to the short ones [18]. Similar trend of result demonstrated by [19], the author evaluated his result as a consequence of large particle size having high aspect ratio that later contribute in better tensile strength.

Due to the alkali treatment, the filler may experience the fibrillation process that tends to increase the aspect ratio. This phenomenon would greatly increase the interface between the polymer matrix and filler [20]. This assumption is indeed in agreement with [5], saying that better adhesion may occur when hemicellulose is dissolved as a result of high aspect ratio. Other than that, we assume that alkali tends to be absorbed more efficiently by the larger particle size. It is may be due to better fibrillation happened on its surface compared to the smaller one.

VIII. EFFECTS OF ALKALI TREATMENT

Figure 1 shows the mean values of flexural properties.

Increment of NaOH concentration from 0 to 1% is insufficient to improve the flexural strength. When the concentration increases up to 4%, the flexural MOR of the composite enhanced significantly. It can be seen that 4%NaOH is the starting point of improving the MOR compared to the control sample. Meanwhile, an apparent increment can be observed in flexural modulus affected by the alkali treatment. Alkali treatment seems to be the factor of providing a chemical strength between filler and matrix [21]. The strength is then enhancing the interfacial adhesion hence resulting in positive trend of alkali treatment in flexural properties.

At 1%NaOH, the composite tensile strength reaches its maximum reading and latter consistently maintains with increasing of alkali concentrations as can be observed in Figure 2. Table 3 shows that the alkali treatment has a significantly positive correlated with the tensile strength and modulus at r=.473. Similar result of highest tensile modulus observed at 1%NaOH treated sample. The treatment has promoted an efficient stress transfer between the matrix and the natural fiber that indicates better tensile properties [9]. The elongation at break shows a different trend of result compared to the tensile strength and modulus but similar to flexural strength.

The composite improves its elongation at break behavior reaching the control sample reading when the alkali concentrations increase up to 4% NaOH. Filler addition is obviously can cause a steep decline in elongation. This is because the polymer molecules flowing are restricted to pass to one another as the wood fiber itself having low elongation properties [22]. In this study, the least concentration of alkali seems to not improve the elongation. As observed by 9, treatments seem to not greatly affect the elongation to be better than the untreated fibers. The value of elongation decreases attributed to rigid nature of the fiber.

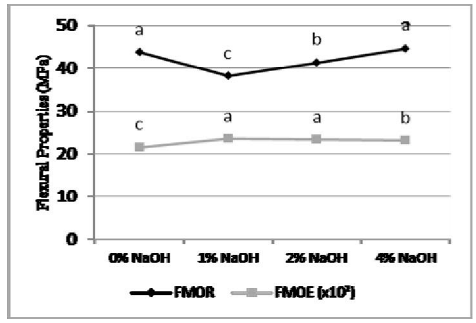

Fig. 1 Flexural properties

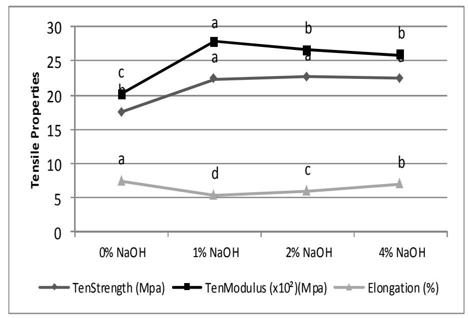

Fig. 2 Tensile properties

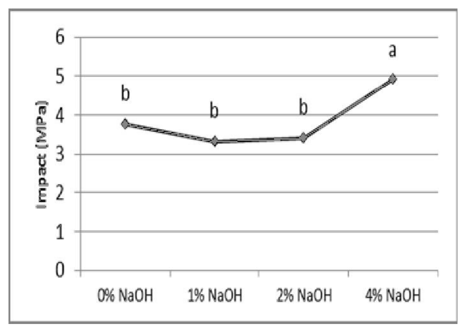

Fig. 3 Impact strength

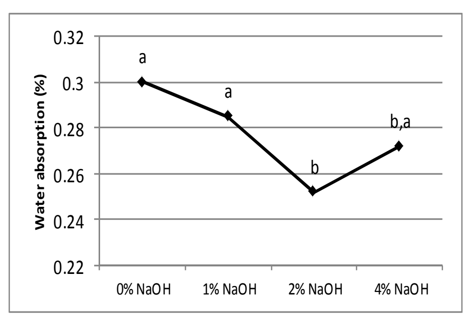

Fig. 4 Water absorption

Effect of alkalization treatment on impact strength of the OPF-PP composite shows a positive improvement as seen in Figure 3. Impact strength is normal to decrease once filler is added to the matrix. This is because filler loading may stiffen up the composite and result in high stress transferred from the matrix to the filler getting higher [23]. Due to the treatment effects, the result shows an increment reading of impact strength with increases of alkali concentrations at 95% confident level intervals. The composite shows a modest decline result in impact strength when immersed in 1%NaOH but obtain better result when approaching the 4%NaOH. This can be an interpretation of the positive effect by alkali treatment on the composite impact strength. It is because alkali treatment contributes a lot in improving the particle dispersion which later solving the agglomeration problems and increase the effective surface area for filler-matrix bonding [7].

Wood fiber has a hydrophilic polar which tends to absorb water and resulting the composite to have higher moisture content compared with the 100% plastic matrices [24]. In this study, alkali treatment shows a significant positive impact on the water absorption behavior of the composite as shown in Figure 4. The first attempt of alkali treatment on the oil palm frond-polypropylene composite at 1%NaOH shows a slight decrease in water absorption behavior. However, an apparent decrement of water absorption is observed when the alkali concentration increases up to 2%.

Dissolving the hemicellulose and volatile extractives has contributed in reducing the numerous hydroxyl groups that are available on the filler surfaces [28]. This is then resulting in less interaction of the filler and water molecules as more rigid and crystalline packed of cellulose is left. On the other hand, accumulated of the alkali concentrations towards 4% resulted in slightly increment of water absorption behavior on the composite. But somehow, the increment has no significant difference with the 2%NaOH. In general, 2%NaOH seems to be the optimal concentration in gaining a good water absorption behavior for the OPF-PP composite.

Table 3 Correlation analysis of particle size and alkalization

	MOR	MOE	TMOR	TMOE	Elon	Impact	WA
PS	.154	-.059	.194*	-.146	.009	.200**	.529**
N	-.029	.411**	.473**	.384**	-.028	.250*	-.067

Note. PS, particle size; N, NaOH treatment

Note. MOR, modulus of rupture; MOE, modulus of elasticity; Tstrength, tensile strength; Tmodulus, tensile modulus; Elon, elongation; WA, water absorption; TS, thickness swelling.

Table 4 Summaries of ANOVA of the Particle Size and Alkalization Effects

Source of variation	MOR	MOE	TMOR	TMOE	Elong	Impact	WA
Particle Size	NS	NS	**	NS	NS	**	**
NaOH	**	**	**	**	**	**	**
Particle size x NaOH	**	**	NS	**	**	**	NS

Note. **, significant difference at the 5% level

Note. NS, not significant at 0.05; MOR, modulus of rupture; MOE, modulus of elasticity; Tstrength, tensile strength; Tmodulus, tensile modulus; Elong, elongation; WA, water absorption; TS, thickness swelling.

Two-way analysis of variance was conducted to determine the significance of particle size and alkali treatment effects on OPF-polypropylene composite as shown in Table 3. Particle size variable shows significant effects on tensile strength, impact strength and water absorption at p=0.000, p=0.035 and p=0.000 respectively. On the other hand, alkali treatment variable shows significant effects on the entire composite without an exception at p=0.000. Interaction of the two variables shows significant effect on all the composite properties except for the tensile strength and water absorption.

IX. CONCLUSION

The findings of this study showed that oil palm frond is feasible to be filled in the polypropylene matrix with good mechanical and physical properties. The contribution of alkali treatment has changed the oil palm frond chemical composition by removing cementing material which consists of extractive, lignin and hemicellulose. This removal has led the composite to have better strength and modulus as the interfacial site for chemical bonding between filler and matrix is improved. Effective NaOH concentration is exhibited by 4% as most of the properties showed apparent increment at this concentration. However, to not neglect the slight higher water absorption by 4%NaOH, the optimal concentration could be lower which is at 2%. This study suggests that oil palm frond has a tendency to be the alternative natural filler in plastic products. The properties of both treated and untreated particles are comparable to other wood fibers used in wood plastic composite products.

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