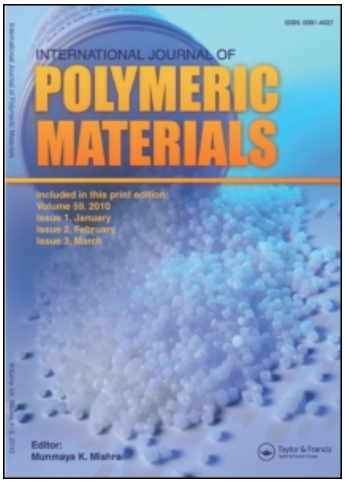

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The Effect of Oil Extraction of the Oil Palm Empty Fruit Bunch on the Processability, Impact, and Flexural Properties of PVC-U Composites

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The use of oil palm empty fruit bunch fiber (EFB) as reinforcement in the unplasticized poly(vinyl chloride) (PVC-U) is a new attraction in the thermoplastic composite technology. The objectives of this study are to investigate the effects of extracted EFB on processability, impact, and flexural properties of PVC-U composites. A soxhlet extraction was used to extract the extractives from the EFB fibers. The identification of the related functional groups present in the concentrated extract was analyzed using FTIR. To produce composites, PVC resin, EFB fiber, and other additives were first dry-blended using a heavy-duty laboratory mixer before being milled into sheets on a two-roll mill. Test specimens were then hot pressed after which the impact and flexural properties were determined. The processability studies of dry blends were carried out using a Brabender Torque Rheometer model PL2200. The FTIR analysis showed that the oil residue was successfully extracted from EFB fibers. Both the extracted and unextracted fibers decreased the fusion time and melt viscosity of PVC-U. However, the extracted fiber was found to increase the fusion time of PVC as the fiber content increased from 10 to 40 phr. The impact and flexural properties of composites were not significantly affected by the fiber extraction.

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Keywords: extraction, fusion characteristics, mechanical properties, oil palm empty fruit bunch, unplasticized poly(vinyl chloride)

INTRODUCTION

Oil palm production is a major agricultural industry in Malaysia. Currently, there are more than three million hectares of oil palm plantations in Malaysia [1]. In total, about 90 million metric tons of renewable biomass (trunks, fronds, shell, palm press fiber, and the empty fruit bunch) are produced each year. The empty fruit bunches represent about 9% of this total [1], as they are left unutilized after the fruit bunches are pressed at oil mills and the oil extracted. With the rising environmental issues in the limelight and the need for new applications of empty fruit bunch fibers (EFB) in order to minimize the abundance of this industrial waste, EFB fibers seem attractive enough to be used as a reinforcement fiber for fiber-filled thermoplastics composites.

Although the use of EFB fiber is not as popular as the use of inorganic or mineral fibers, awareness of its advantages (similar to other natural fibers) such as low density, less abrasiveness to processing equipment, lower cost, reduced health hazard, abundance, and sustainability have gradually increased its attractiveness for use in polymer composites [2].

The potential utilization of EFB for the production of polypropylene [3–5], polystyrene [6], polyesters [7], and phenol-formaldehyde resin [8]–filled composites has been reported by several researchers. They were focused on how fiber loading, fiber size distribution, and fiber surface treatment affected the mechanical properties of the composites. As with other natural fibers, many issues have been identified in the processing of the composites. These include the low compatibility between EFB fibers and the polymer matrix, high moisture intake, and poor dispersion of the fibers in the matrix. Compatibility plays a crucial role in determining the properties of a composite because of the hydrophilic nature of natural fibers (contributed by hydroxyl groups in cellulose, lignin, and hemicelluloses) and the hydrophobic thermoplastics matrix. This compatibility is made worse in the case of EFB due to the presence of oil residues on the fiber [5]. It is reported that the oil residues on the EFB fibers are still present even after the extraction process in the factory. Although the researchers [5] have concluded that the extraction of the EFB fibers has resulted in significant improvement in flexural and tensile strength, no confirmation test was done to determine the exact composition of

the extracted fibers. Thus, in this study, the chemical composition of the extract fibers was determined using FTIR analysis.

Although a study on the effect of extracted EFB fibers as reinforcement in polypropylene [5] has been reported, similar study on PVC has not been reported. In the study reported in this article, besides the effect on the mechanical properties, the processability of PVC-U compound was also investigated.

The properties of PVC products depend on the morphology and the degree of fusion, and therefore, additives that control the rate of fusion within the processing equipment become important ingredients in a successful formulation. Many studies have shown that external lubricants prolong the fusion time of PVC compound and internal lubricants shorten the fusion time [9–11]. However, the synergistic reaction between them has shortened the fusion time of PVC compound significantly. Meanwhile, processing aids used to improve the processing properties of PVC have been recognized as a fusion promoter, although the mechanism by which this processing aid accelerates the fusion of PVC has not yet been fully elucidated. According to Cogswell et al., processing aids have increased the rate of fusion by means of increasing the friction between the particles [12]. This mechanism has also been proposed by Krzewki and Collins [13]. Several workers have studied the effects of clay and rice husk ash filler loading on the processability of PVC-U dry blends [14–15]. They found that the clay and rice husk ash filler had decreased the fusion time of PVC-U dry blends as the filler content increased.

EXPERIMENTAL

Materials

The suspension PVC resin used in this study, with solution viscosity K-value 66, and trade name MH-66 was supplied by Industrial Resin Malaysia (IRM) Sdn. Bhd. Tampoi, Johor, Malaysia. Its specifications are summarized in Table 1. The additives used in the PVC formulations (shown in Table 2) were also supplied by IRM Sdn. Bhd. The EFB fibers were purchased from Sabutek Sdn. Bhd., Teluk Intan, Perak, Malaysia. The properties of EFB fibers are shown in Table 3.

Fiber Preparation

A Restsch shaker was used to separate the EFB fibers into different sizes. The shaking time was 10 min. The length of the EFB fibers used in this study was less than 75 μm. The fibers were dried to constant

TABLE 1 Specifications of PVC Suspension Resin MH-66

Appearance	White powder
Degree of polymerization	1000 ± 50
K-value	66
Specific gravity	1.4
Bulk densiy (g/cm^3)	0.50 ± 0.05
Volatile matter (max) (%)	0.5
Foreign matter (grain/100 g)	15
Particle size (retained on 250?) (max) (%)	0.3

TABLE 2 Blend Formulations

	Formulations (phr)				
	Unfilled compound	Filled compounds			
Ingredients	S_0	S_1	S_2	S_3	S_4
Poly(vinyl chloride) resin	100	100	100	100	100
Tin stabilizer (Heat stabilizer)	2.0	2.0	2.0	2.0	2.0
Calcium stearate (Internal lubricant)	0.5	0.5	0.5	0.5	0.5
Stearic acid (External lubricant)	0.6	0.6	0.6	0.6	0.6
Acrylic polymer (Processing aid)	1.5	1.5	1.5	1.5	1.5
Titanium oxide (Pigment)	4.0	4.0	4.0	4.0	4.0
Oil palm empty fruit bunch (EFB)	0	10	20	30	40

TABLE 3 Properties of EFB Fiber

Holocellulose (%)	82.5
α-cellulose (%)	60.6
Lignin (%)	17.2
Extractives (%)	2.3
Ash content (%)	5.4
Tensile strength (MPa)	130
Tensile modulus (GPa)	3.58
Elongation (%)	9.70

weight in an oven at 105°C for about 24 h, the weight loss then being calculated. The original moisture content of the EFB was found to be around 5%.

EFB Oil Extraction

A soxhlet extraction unit as described by Han and Rowell was used in this study [16]. Approximately 32 g of dried EFB fibers was

weighed and placed into an extraction thimble for each extraction process. The chemical reagent used consisted of three types of solvents, which were ethanol (99.7% purity), toluene (99.5% purity), and acetone (99.8% purity) with a volume proportion of 4:1:1, respectively. These solvents were employed to extract the oil residues of EFB fibers. The extraction was carried out at 120°C (higher than the boiling point of the three solvents) for 24 h. The extracted EFB fibers were washed with ethanol and placed in a vacuum oven at 45°C for 24 h to allow complete evaporation of the ethanol. When dried, the extracted fibers were removed to desiccators before being used in a dry blending process. The percentage of oil removal was calculated using the following equation:

$$\text{Oil Removal}\,(\%) = \frac{\text{Unextracted Fiber} - \text{Extracted Fiber}}{\text{Unextracted Fiber}}\,(100\%)$$

The collected extract consists of concentrated extract (which maybe oil) and the solvents mixture was separated using a condensation process. The concentrated extract and the condensate (solvents mixture) were analyzed using a Fourier Transform-Infrared (FTIR) analysis.

FTIR Analysis

This analysis was used to identify the related functional groups present in the unextracted and extracted EFB fibers, concentrated extract, and condensate. A small quantity of fibers was dispersed in dry potassium bromide (KBr). Each mixture was thoroughly mixed in a mortar and then pressed at pressure of 6 bars within 2 min to form a moisture-free KBr thin disc. The thin disc was placed in a sample cup of a diffuse reflectance accessory. Meanwhile, for the concentrated extract and condensate sample, each sample was injected in between two sodium chloride plates and fitted in the cell unit. The samples were scanned 16 times from 4000 cm^{-1} to 370 cm^{-1} to reduce the noise to signal ratio using a Perkins Elmer 2000 Infrared Spectrometer.

Dry Blending

The blend formulations of the unfilled compound (S_0) and filled compounds (S_1–S_4) are shown in Table 2. The fiber content of the unextracted and extracted fiber in the compounds was varied from 10 to 40 parts per hundred of PVC resin (phr). The dry blending process for each blend formulation was first done using a laboratory high-speed mixer for 10 min to homogenize the unfilled and filled

compounds. All the dry-blended compounds were then used for processability study.

Processability Study

A Brabender Torque Rheometer model PL2200 was used to study the processability of the dry-blended samples. A 54 g of dry-blended sample was placed into the mixing chamber through a loading chute. Immediately after the dry-blended sample has been loaded, a piston with 5 kg weight was inserted in place. It was then pressed gently to force all the dry-blended sample completely into the mixing chamber as quickly as possible for best reproducibility and comparability of test results. All samples were run at a constant rotor speed of 50 rpm and at mixer temperature of 180°C. The fusion characteristics of each dry-blended sample were interpreted by observing the changes of its torque curve.

Mechanical Testing

The fiber content in the composite specimens was fixed at 30 phr for the mechanical testing. Each of the dry-blended samples was first melt-blended and sheeted using a laboratory two-roll mill at a temperature of 165°C for 10 min. The milled sheets were then placed into mold with five cavities and hot pressed at temperature of 180°C and pressure of 12 MPa, for 5 min. It was cooled for 5 min before being removed from the mold cavities.

The Izod impact strength assessment was carried out on notched samples of dimensions $62.5 \times 13 \times 3$ mm at room temperature using an IMPats pendulum impact tester at an impact velocity of 3.0 m/s and 90° swing angle. The specimens were notched with a notch cutting apparatus. The notch depth was fixed at 2.6 ± 0.02 mm with angle 45°. Flexural tests were conducted in an Instron Machine Model 5567 according to ASTM D790. The samples, with dimensions $125 \times 13 \times 3$ mm, were tested at crosshead speed of 3 mm/min at room temperature. The support span for the flexural test was 51 mm. All the reported values for the tests were the average of seven specimens.

Scanning Electron Microscopy

Studies of the morphology of EFB fiber surface were performed with a JEOL Model JSM-6301 Scanning Electron Microscope (SEM). A small portion of sample was mounted on the copper stub and sputter-coated

with a thin layer of gold to avoid electrostatic charging during examination.

RESULTS AND DISCUSSION

Oil Extracted Fiber Analysis

The percentage of oil removed from the EFB fibers was approximately 4.5%. The effect of extraction on the appearance of the solvent mixture and fibers was also studied by visual inspection. The solvents mixture changed color from colorless to dark brown, whereas the extracted fibers were found to be brighter in color than the unextracted fibers. This visual inspection indicates that the extraction process has effectively extracted the extractives, which include oil residues of fibers. The analysis was carried out on the extractives and extracted fibers in order to verify that the extraction was successfully achieved.

FTIR analysis was done on the fibers, concentrated extract, and condensate. Figure 1 shows the FTIR spectrum of the extracted and the unextracted fiber. Both spectra are found to be very similar. It shows that the functional groups of the unextracted and extracted fiber cannot be distinguished using this analysis. Both spectra show a broad peak at 3393 cm^{-1} and 3391 cm^{-1} due to the absorption vibration of the hydroxyl (OH) group [6]. The peaks at 2919 cm^{-1} and 2916 cm^{-1} are due to the

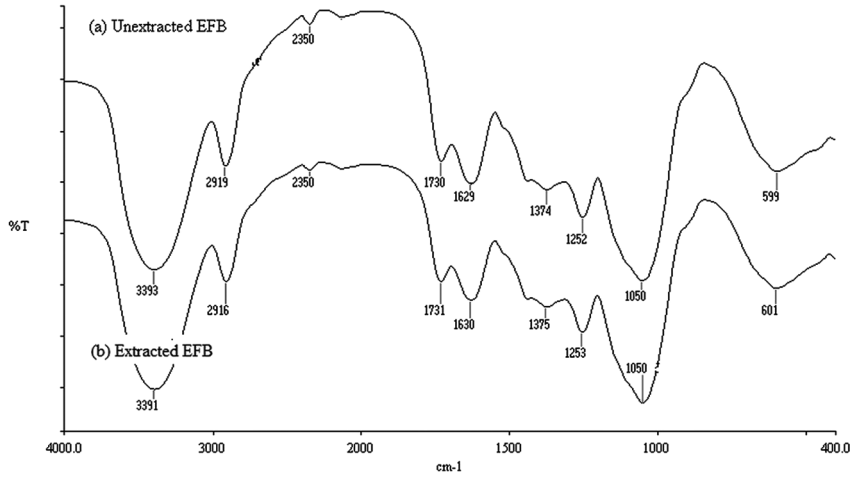

FIGURE 1 FTIR spectra: (a) unextracted EFB fiber and (b) extracted EFB fiber.

stretching of C−H groups. The presence of cellulose is obviously seen from the sharp peak at 1730 cm^{-1} and 1731 cm^{-1}, due to the stretching vibration of carbonyl group (C=O). Peaks at 1629 cm^{-1} and 1630 cm^{-1} are indicative of the presence of lignin, and attributed to the C=C vibration. The broad peaks at 1050 cm^{-1} are assigned to ether linkage (C−O−C) from lignin or hemicellulose [17].

In the analysis of the concentrated extract, ester-containing compound was detected in the spectrum as shown in Figure 2. The sharp absorption bands at 1712 cm^{-1} and 1739 cm^{-1} are attributed to the stretching of carbonyl groups, whereas the sharp absorption band at 1261 cm^{-1} is due to the asymmetric stretching of C−O−C from the oil [18]. Because the oil chemical structure contains long chains of hydrocarbon, the sharp and strong absorption band at 2925 cm^{-1} and 1458 cm^{-1} due to the stretching of CH and CH_2 groups, respectively, are clearly observed [19]. This spectrum definitely verified the existence of oil residues in the concentrated extract.

Figures 3 and 4 show the SEM micrographs of the unextracted and extracted fiber surface, respectively. The surface morphology of the extracted fiber was coarser than the unextracted fiber. Many pits on the extracted fiber surface were also observed. The difference is caused by the removal of oil layer leaving the extracted fiber surface exposed.

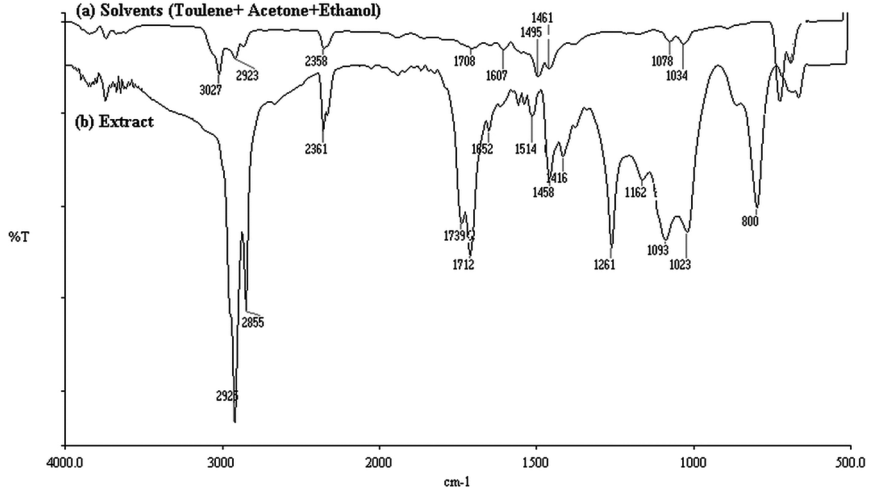

FIGURE 2 FTIR spectra: (a) solvents mixture condensate and (b) concentrated extract.

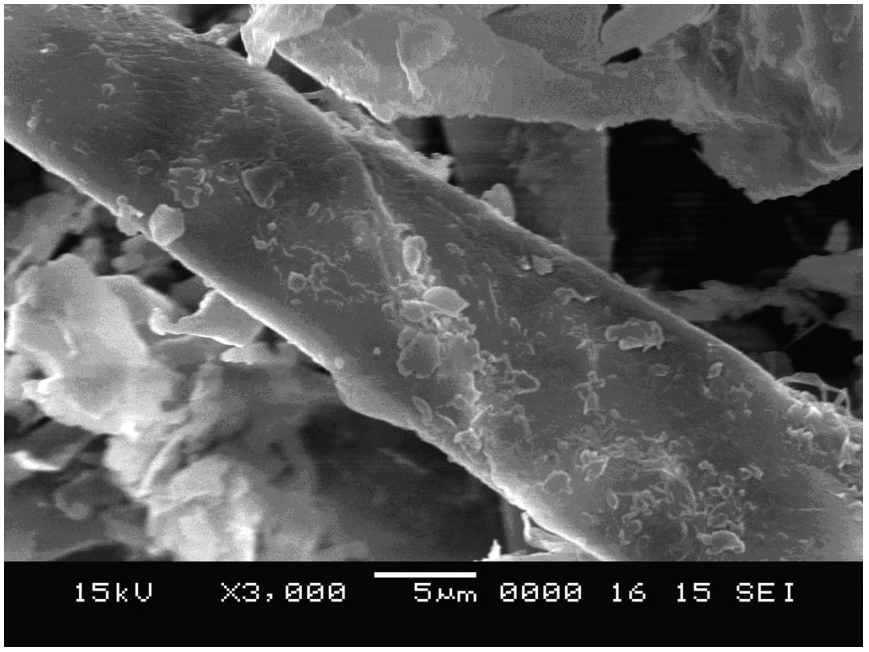

FIGURE 3 SEM micrograph for unextracted EFB fibers (magnification 3000×).

Processability Analysis

Fusion characteristics such as fusion time, torque at the equilibrium stage (end torque), and fusion temperature, which are obtained from the fusion curve, are used to elucidate the processability of the studied compound. Figure 5 shows a typical fusion curve of unfilled composite dry blend (S_0) melted in the Brabender Torque Rheometer at a starting temperature of 180°C, a rotor speed of 50 rpm, and discharged when the stabilized end torque was reached. This curve illustrates the changes of melt viscosity related to torque and temperature versus time. The temperature at point F is defined as a fusion temperature. The time taken between the point A and fusion point F is defined as a fusion time, t. The dry blend is in equilibrium state as the torque curve and stock temperature curve being smooth and stable with time. At this state, the melt is homogeneous and the torque is defined as an end torque. The stock temperature curve shows the generation of frictional heat with time during the mixing process. Due to the influence of the surface temperature in the mixing chamber and on the rotor

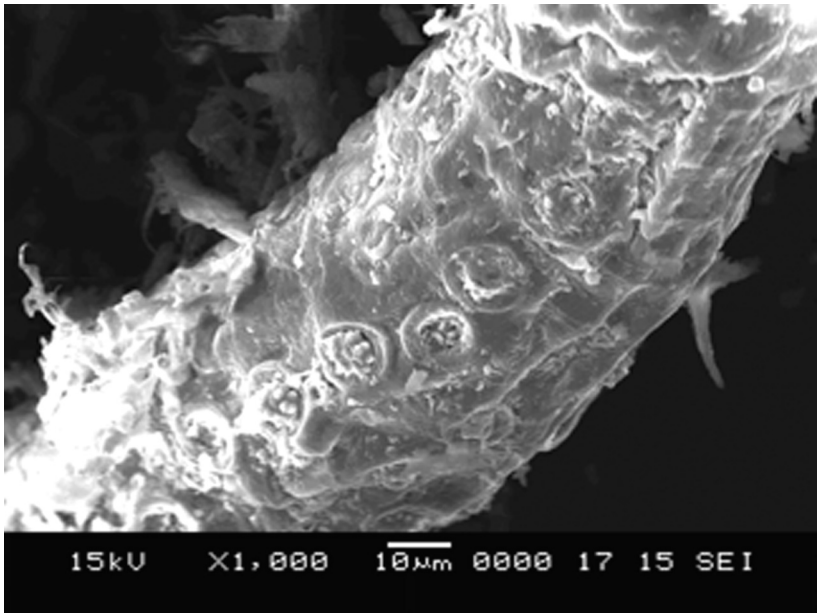

FIGURE 4 SEM micrograph for extracted EFB fibers (magnification 1000×).

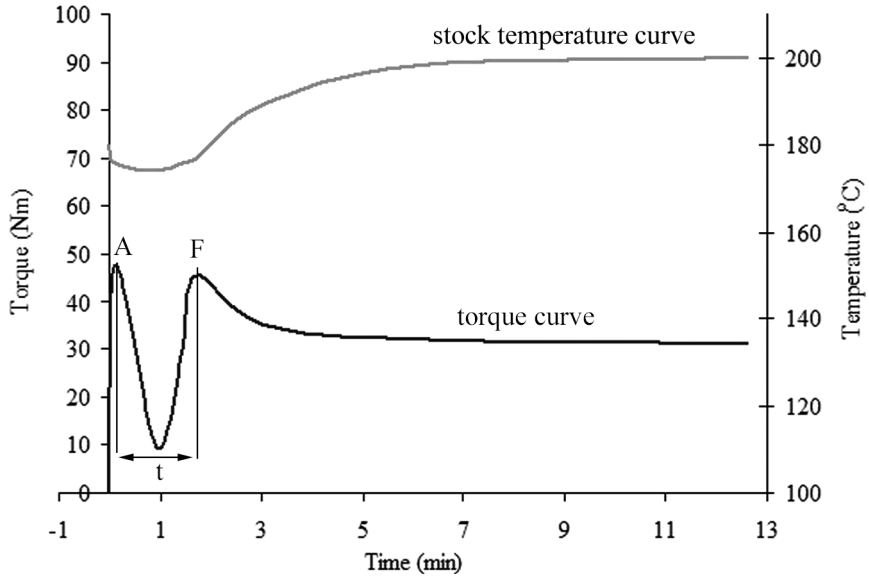

FIGURE 5 A typical fusion curve of the unfilled composite.

blades, and the shearing forces of the mixing action, the PVC resin particles start to soften at the surface. Thus, the coefficient of friction among particles when passing each other in the mixing processing is increased. This resulted in the increase of frictional heat in the compound.

Table 4 shows that the unextracted and extracted fiber decreased the fusion time of PVC-U. The fusion time decreased drastically upon the incorporation of 10 phr unextracted fiber and remained relatively constant with further increase to 40 phr. However, for the extracted fiber, an increase from 10 to 40 phr caused an increase in fusion time. This result is in agreement with the results obtained by other researchers working on other fillers such as clay and rice husk ash [14–15].

The presence of oil residues on the EFB fiber has been reported by Rozman et al. [5]. Oil residues on the fibers are able to migrate on the fiber surface due to the shearing actions of the mixer blades and frictional heat during mixing process. This oil residue forms a layer on the outer surface of EFB fiber and tends to be an internal lubricant in the filled compounds. As an internal lubricant, calcium stearate interacts with the ester components of the oil residue to form a better adhesion between the PVC resin particles. As a result, the PVC resin particles of filled compounds fused at a shorter time. Furthermore, the oil layer on the EFB fibers surface may also obstruct the thermal heat from being absorbed by EFB fibers. Consequently, most of the heat is transferred to the PVC resin particles during the mixing process. Another reason of the fusion time of filled compounds being shorter than unfilled compound is the frictional heat generated by the friction of fiber-fiber and fiber-PVC resin particles. The generated frictional heat increased the total heat of the filled compounds and allowed the PVC resin particles to fuse easily.

As mentioned earlier, the fusion time of unextracted fiber-filled compounds remained relatively constant as the fiber content increased. This may be due to the increase of frictional heat of filled compounds and adhesion property of calcium stearate probably being nullified by the increase in separation and slipping among PVC resin particles as the fiber content increased. This reason can also be cited to explain the end torque and fusion temperature of PVC-U, which decreased at 10 phr fiber content and remain relatively constant as the fiber content increased from 10 to 40 phr. This effect is attributed to the relative constant fusion time, melt viscosity, and fusion temperature as the fiber content increased from 10 to 40 phr.

For extracted fibers, the coarser surface of fibers (Figure 4) is more effective in enhancing the frictional heat of filled compounds,

TABLE 4 Fusion Characteristics of Filled Compounds

Fiber content (phr)	Unextracted EFB			Extracted EFB		
	Fusion time (s)	End torque (Nm)	Fusion temperature (°C)	Fusion time (s)	End torque (Nm)	Fusion temperature (°C)
0	96	31.1	177	96	31.1	177
10	60	28.2	175	44	30.8	176
20	68	27.3	178	58	27.5	177
30	60	27.5	177	66	27.7	178
40	58	26.1	175	78	27.0	180

compared to the unextracted fibers. Therefore, PVC resin particles filled with the extracted fibers, particularly at 10 and 20 phr, took a shortened time to fuse compared to the unextracted fibers (Table 4). As the extracted fiber content increased from 30 to 40 phr, the separation and slipping among PVC resin particles are also increased. Furthermore, the lack of oil residues resulted in the adhesion contribution of calcium stearate to become weaker as the fiber content increased. Therefore, calcium stearate took a longer time to bind PVC resin particles together. Consequently, the fusion time of PVC resin particles

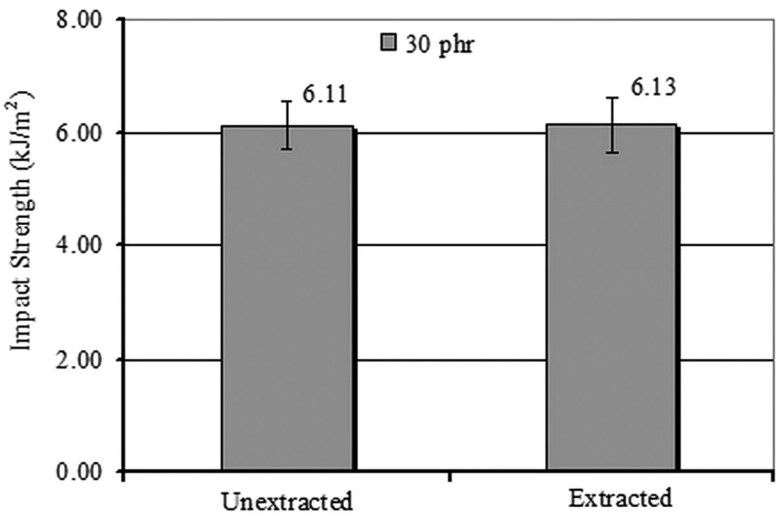

FIGURE 6 Impact strength of unextracted and extracted EFB-filled composites at fiber content of 30 phr.

increased with increasing fiber content. It can be reasoned that more thermal energy is required for the filled compounds in order for PVC resin particles to fuse together. As a result, the fusion temperature is also increased. The increase of fusion temperature has decreased the melt viscosity of the filled compound. The slightly higher melt viscosity of the extracted fiber-filled compound compared to unextracted is due to the extracted fibers not being coated by oil residues.

Mechanical Properties

Figure 6 shows the effect of extracted fiber on the impact strength of filled composites. It shows that the impact strength of the composite filled with 30 phr extracted fiber is marginally higher than the composites with unextracted fiber. The degree of enhancement is only about 0.3%. This indicates that the amount of oil residues present in the EFB fibers is not a significant factor in influencing the impact strength of the filled PVC-U composites. The impact of oil extraction in the present study is less significant compared to a previous study on polypropylene composite [5], which may be due to the polarity of PVC molecules, which enable the polymer to have a good interaction even when oil residues are present in the fibers.

Figure 7 depicts the results of flexural strength of filled composites. It shows that there is no significant improvement (only about 3%) in

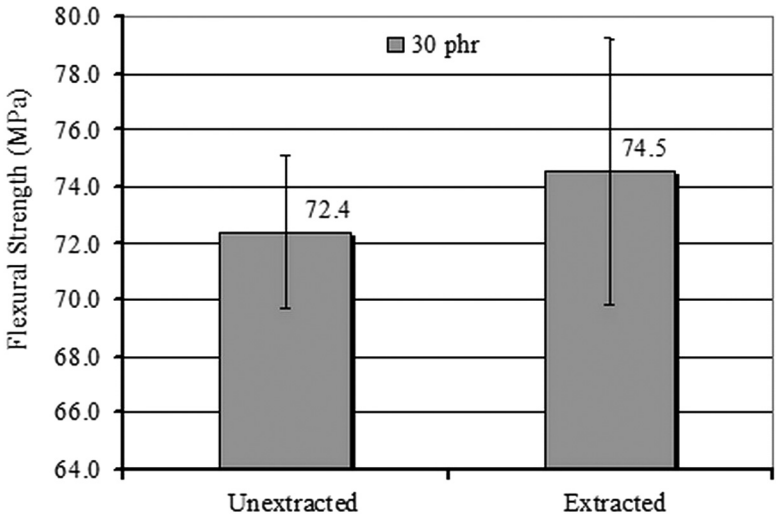

FIGURE 7 Flexural strength of unextracted and extracted EFB-filled composites at fiber content of 30 phr.

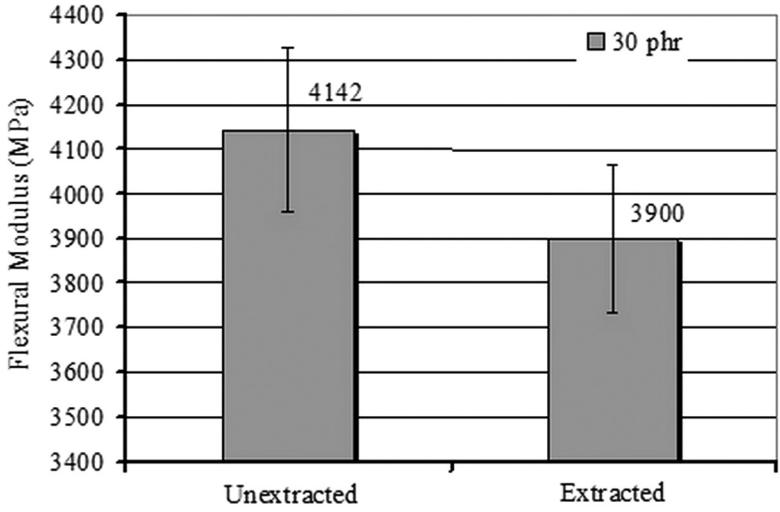

FIGURE 8 Flexural modulus of unextracted and extracted EFB-filled composites at fiber content of 30 phr.

flexural strength of the composite filled with 30 phr extracted fiber, compared to the unextracted. Figure 8 shows that the flexural modulus of extracted fiber-filled composite is slightly lower compared to unextracted fiber. The reason for this is not understood by us at present.

CONCLUSIONS

The study demonstrates the effect of oil extraction of EFB fibers on the processability, impact, and flexural properties of composites. Based on the FTIR analysis of the concentrated extract and surface morphology of the extracted fiber, the oil residues were successfully extracted from the EFB fibers by the Soxhlet extraction. The extraction of the EFB fibers has resulted in the fusion time and the melt viscosity of PVC to decrease. However, a significant increase in the fusion time was observed as the extracted fiber content was increased from 10 to 40 phr. Marginal improvement in impact and flexural strength occurred because of the oil extraction.

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