

Effects of Coupling Agent and Flame Retardant on the Performances of Oil Palm Empty Fruit Bunch Fiber Reinforced Polypropylene Composites

R. Ridzuan, M. D. H. Beg, M. Y. Rosli, M. H. Rohaya, A. A. Astimar S. Samahani, and I. Zawawi

Abstract—Alkali treated oil palm empty fruit bunch (EFB) fibres (TEFBF) and untreated EFBF fibers (UEFBF) were incorporated in polypropylene (PP) with and without malic anhydride grafted PP (MAPP) and magnesium hydroxide as flame retardant (FR) to produce TEFBF-PP and UEFBF-PP composites by the melt casting method. The composites were characterized by mechanical and burning tests along with a scanning electron microscope and Fourier transform infrared spectroscopy. The significant improvement in flexural modulus (133%) and flame retardant property (60%) of TEFBF-PP composite with MAPP and FR is observed. The improved mechanical property is discussed by the development of encapsulated textures.

Keywords—Empty fruit bunch fibers, polypropylene, mechanical property, flame retardant.

I. INTRODUCTION

INCORPORATION of natural fiber (NF) into polymer as a reinforcing agent has received increased attention, because NF-based composites show remarkably outstanding performances and can be produced with a large volume at affordable costs [1, 2]. This development in composites manufacturing reflects several advantages of lignocellulosic fibres like their light-weight, low-cost, availability, reproducibility, biodegradability and environmentally friendly characteristics over synthetic counterparts [3]. Currently, many types of NFs such as flax, hemp, jute, straw, wood, rice husks, wheat, barley, oats, rye, cane (sugar and bamboo), grass, reeds, kenaf, ramie, oil palm empty fruit bunch, sisal, coir, water hyacinth, pennywort, kapok, paper-mulberry, raphia, banana, pineapple leaf fibre and papyrus are being investigated for use in plastics. Among them, oil palm empty fruit bunch (EFB) fiber is a readily obtainable NF from palm oil industries of Malaysia, yet to be fully materialized in reinforcing plastics despite its huge yield in this country [4, 5]. Due to their availability and disposable problems, EFB fibers can preferably be used as interesting renewable resources for the fabrication of new polymeric composites that can be good

competitors to other NF reinforced composites and can provide attractive value-added markets for agricultural products by displacing petrochemical-based plastics.

However, EFB fibers have a few inherent setbacks like high moisture absorption, low thermal stability and poor incompatibility with polymer matrix [6], which preclude its potential uses in composite fabrication. To overcome these drawbacks, a number of fiber treatments using alkali, heat, acetylation and coupling agent are being widely employed in order to improve the wettability of fiber with polymer matrix [7-10]. Of these techniques, a potentially useful approach is the alkali treatment, which reportedly not only leads to the increase in interactions between fibers and polymers, but also increases the number of reactive hydroxyl groups (-OH) on the fiber surface available for chemical bonding [11, 12]. As moisture uptake by fibers can inhibit compatibility between fiber-matrix and deteriorate mechanical properties of the composite, uses of different coupling agents have been reported to improve the fiber-matrix interface and enhance mechanical properties of the composites [13-15].

On the other hand, polypropylene (PP) is a versatile soft matter extensively used for manufacturing automotive components, home appliances and other industrial products, which have tremendously increased in recent years because of its balanced mechanical properties, ease of processability and low-cost [16]. This widely used engineering thermoplastic is often transformed into composites by the inclusion of fillers or reinforcements that alter its crystalline structure, morphology and underlying properties [16-18]. In this context, EFB fiber can serve as a good candidate to improve the properties of PP through suitable processing routes. Since PP is enriched with carbon and hydrogen contents, it is combustible in air with a hot and clean flame. The flammability of PP occurs as a result of its long chain saturated hydrocarbon structure, which readily fragments at high pyrolysis temperature, yielding highly volatile low molecular weight saturated and unsaturated hydrocarbons that subsequently undergo free radical and oxidation reactions in the pre-ignition zone and flame zone [19].

Since plastics have to fulfill flame retardancy requirements laid down in mandatory regulations and voluntary specifications for various applications in the building, electrical, transportation, mining, and other industries, the

R. Ridzuan, M.H. Rohaya, A.A. Astimar and I. Zawawi are with Malaysia Palm Oil Board, No 6, Persiaran Institusi, Bandar Baru Bangi, 43000, Selangor, Malaysia; (e-mail:ridz@mpob.gov.my).

M.D.H. Beg, M.Y. Rosli and S. Samahani are with the Faculty of Chemical and Natural Resources Engineering, Universiti Malaysia Pahang, Lebuhraya Tun Razak, Gambang-26300, Pahang, Malaysia; (e-mail: dnbeg@yahoo.com).

plastics based composites must be prepared taking into account of satisfying these requirements. In this perspective, flame retardants can play important roles to inhibit or even to suppress the combustion process by physical and chemical actions, depending on their nature [20]. Considering the usefulness of coupling agent and flame retardant, we are intended to use both of them to develop a series of PP based composites with untreated and treated EFB fibers for improving the interfacial adhesion between fiber and matrix and finding out a balance of their structural, mechanical, thermal and fire-retardant properties in the present study.

II. EXPERIMENTAL

A. Materials

PP of grade G452 (melt flow index 45 g/10min, density 0.90 g/cm³), and the coupling agent polybond, which was a malic anhydride grafted PP (MAPP), were supplied by MTBE, Malaysia. Sodium hydroxide (NaOH) and magnesium hydroxide (Mg(OH)₂) of purity \approx 99%, purchased from Sigma Aldrich, USA, were used as a surface modifier and a flame retardant (FR), respectively. Empty fruit bunch (EFB) fibres were obtained from Felda Palm Oil Berhad, Malaysia. The proximate chemical compositions of EFB fibre used in this study were cellulose (47.6%), hemicellulose (25.7%), alpha-cellulose (17%), lignin (14.5%) and ashes (4.4%) [21].

B. Alkaline Treatment of EFB

The EFB fibers as-received were dried under sunlight for three days, chopped using a crusher machine and then passed through a 500 μ m-sieve to obtain fibres of small size. These chopped fibers were soaked in hot distilled water at 70°C for 6 h to remove impurities from their surface and dried again to produce untreated EFB fibers (UEFBFs). After that, UEFBFs were treated with alkali by introducing about 25g fibers in 500 ml of distilled water containing 15% (w/v) NaOH solution in a water bath at 75°C and 40 rpm for 4 hours. These treated fibres were again soaked in distilled water containing 2% hydrogen peroxide (H₂O₂) at 45°C for 8 h to activate the OH group of the cellulose. After these treatments, fibres were taken out from H₂O₂ solution, washed with distilled water and then treated by 10% (v/v) acetic acid (at room temperature) for 30 min to neutralize the excess NaOH content present in the treated fibres. The fibers were repeatedly washed with distilled water until the acid content is removed and then dried in an oven at 70°C for 48 h to obtain treated EFB fibers (TEFBFs).

C. Preparation of Composites

Compounding of PP with either UEFBFs or TEFBFs using and without using MAPP and FR was carried out at 180°C by a twin-screw compounder (MODEL: LTE26-40), having L/D = 40 (length/diameter of the screw), screw speed of 400 rpm and barrel temperature range of 170–190 °C from the feeding zone to the die zone. To find a suitable blending formulation, UEFBF and TEFBF (0–50 wt%) contents with PP were

optimized first by means of mechanical characterization. Once the fiber content was optimized, the FR content was optimized, taking a constant ratio of 1:10 for EFB fibres to MAPP in the composites. Having the optimum FR content, a series of extrudates of UEFBF+PP and TEFBF+PP with MAPP and FR of different formulations, as shown in TABLE I, and UEFBF+PP and TEFBF+PP without MAPP and FR were produced. All these extrudates were molded by an injection molding machine, Toshiba (MODEL: IS 80 EPN) at 190°C, resulting in the ultimate specimen, as shown in Fig. 1, with the thickness of approximately 3-4 mm for various mechanical tests. After molding, the test specimens were conditioned at a temperature of 23 \pm 2°C and a relative humidity of 50 % for at least 40 h, according to ASTM D618-99, prior to different characterizations.

TABLE I
BLENDING FORMULATIONS OF COMPOSITES

PP (wt%)	EFB fibers (wt %)	MAPP (wt%)	FR (wt%)
100	0	0	0
79	10	1	10
68	20	2	10
57	30	3	10
46	40	4	10
35	50	5	10



Fig. 1 Photographs of composite samples prepared for tensile, flexural and impact tests

D. Scanning Electron Microscopy

The surface of UEFBF and TEFBF were observed with a field emission scanning electron microscopy (FESEM) (model: Leo Supra 55, Oxford Instruments) and the fractured surface of composites was monitored by a SEM (EVO 50, ZEISS). Prior to SEM measurements, each sample was fixed to a metal-base specimen holder using the double-sided sticky carbon tape and then coated with gold using a vacuum sputter machine to make the sample conductive electrically.

E. Fourier Transform of Infrared Spectroscopy

Chemical textures of UEFBF and TEFBF were analyzed by means of a Fourier transform infrared (FTIR) spectrophotometer (MODEL: THERMO) using the standard KBr pellet technique. Each spectrum was recorded with 32 scans in the frequency range 4000–400 cm⁻¹ with a resolution of 4 cm⁻¹.

F. Tensile Test

The tensile tests were conducted according to ASTM D638-03 using a universal testing machine (SHIMADZU, MODEL AG-1) with a load cell of 5 kN at a crosshead speed of 10 mm/min and a gauge length of 115 mm. The test was performed at 25 °C until the tensile failure occurred. Eight specimens were tested and at least five replicate specimens were presented as an average of tested specimens.

G. Flexural Test

The Flexural tests were carried out in accordance with ASTM D790-97 standard by the same apparatus as that used in the tensile test. The distance between supports span was 51 mm while the crosshead speed utilized was 1.26 mm/min. Eight specimens were tested and at least five replicate specimens were presented as an average of tested specimens.

H. Impact Test

For the impact strength measurement, the samples were cut into a rectangular shape and notched. The Izod impact tests were conducted according to ASTM D256 using an impact pendulum tester (MODEL TOYOSEIKI), with a 1 J hammer. Eight specimens were tested and at least five replicate specimens were presented as an average of tested specimens.

I. Flexural Test

Flammability test was performed according to UL 94 using an ATLAS flammability Chamber by using the horizontal burning test. For this testing, a series of composites of various FR contents but for a fixed UEFBF and MAPP content was also prepared. The burning test was done for sample dimension of 350 mm × 100 mm × 12 mm, flame height of 38 mm and burned length of 254 mm, and the burning rate was measured.

III. RESULTS AND DISCUSSIONS

A. Surface Morphology of EFB Fibres

The SEM micrographs of UEFBF and TEFBF are shown in Fig. 2 (a) and (b), respectively. The surface of UEFBF consists of noncellulosic gummy contents cementing the fiber bundles together and appears with a lot of debris attached to it. In contrast, the TEFBF surface is relatively clean and contains huge pores, representing elimination of cementing materials and other impurities from the fiber after alkaline treatment. Alkaline peroxide was reported to remove lignin along with waxy components by breaking down the fibrils, thereby leading to an increase in porosity, effective surface area and surface charge, which can expose further hydroxyl and carboxyl groups to attract each other, causing better packing in cellulose molecules [22]. The observed surface morphologies may support the reported findings.

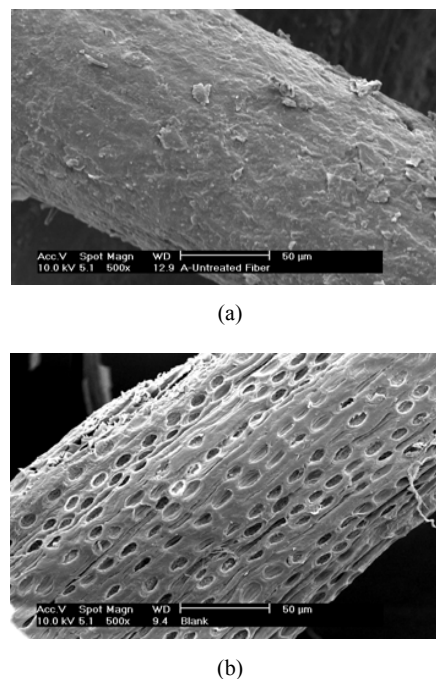


Fig. 2 SEM micrographs of (a) UEFBF and (b) TEFBF

B. FTIR Spectroscopic Analyses of EFB Fibres

Fig.3 shows the FTIR spectra of UEFBF and TEFBF. The major peaks of UEFBF appearing in the range 3500–3300 cm^{-1} is largely responsible for the O–H stretching vibration in phenolic and aliphatic structures, but most of these peaks disappear in the spectrum of TEFBF fibers with a shift of peak position [23]. Besides, the absorbance peak of UEFBF at 1740 cm^{-1} is due to ester carbonyl vibration in acetyl, feruloyl, *p*-coumaroyl, etc. groups in lignin and hemicelluloses [24]. This peak is almost vanishing in the spectrum of TEFBF, thereby strongly demonstrating a partial removal of hemicellulose and lignin by alkaline treatment. Absorption peaks at about 1654, 1647 and 1610 cm^{-1} for UEFBF originated from conjugated carboxyl–carbonyl stretching of the ester bonds between hydroxycinnamic acids and lignin are apparently cleaved, because the shape of these bands for TEFBF is found to change. A relative increase in carboxylic acid content in TEFBF is evidenced from the carbonyl group stretching at around 1256–1132 cm^{-1} and is in agreement with reported data [23]. Aromatic skeleton vibrations in lignin are assigned at 1600, 1498, 1430, 1321.37, 1256 and 1130 cm^{-1} . Further bands from lignin are located at 1461 (CH in CH_2 and CH_3), 1321, 1335 (guaiacyl ring breathing with C=O stretching), 1256 (guaiacyl ring breathing with C–O and C=O stretching) and 1132 cm^{-1} (aromatic CH inplane deformation). Changes of intensities of these bands are clearly visible in the spectrum of TEFBF, suggesting the removal of lignin and other components from fibers by treatment, which conform to the findings of SEM.

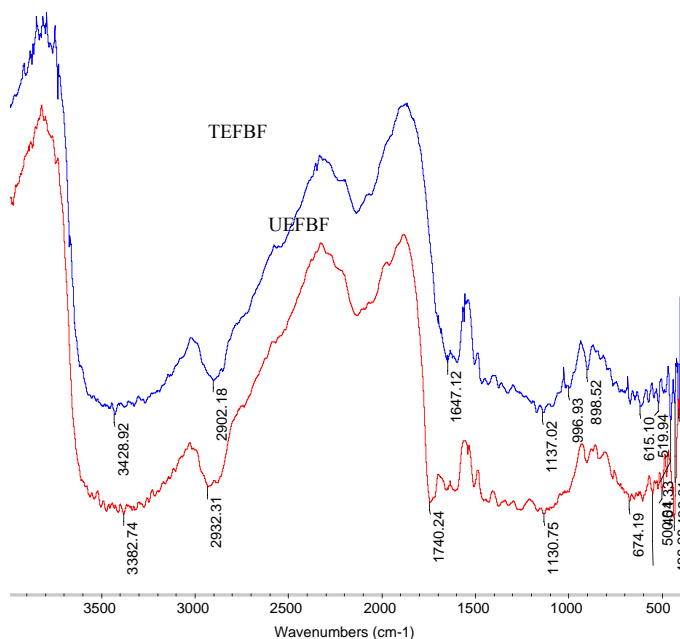


Fig. 3 The FTIR spectra of UEFB and TEFB fibers

C. Mechanical Properties

The variation in flexural modulus (FM) of composites (UEFBF+PP and TEFBF+PP) as a function of untreated and treated fibers content, without MAPP and FR is shown in Fig. 4. The maximum FM values are obtained at 20 wt% UEFBF and TEFBF contents, which is regarded as the optimum amount of fiber to produce the strong composite, as was subjected to further assessment. The variation in TS with respect to FR content for samples with MAPP is indicated in Fig. 5 (a). Notably, 10 wt% FR shows the maximum TS value of the composites and is, therefore, considered as the optimal content of FR for the strong composite. A reduction in TS with respect to the increasing FR content for the composites prepared at 20 wt% UEFBF and MAPP (Fig. 5 (a)) indicates a weak interaction between EFB fibers and PP matrix. In this connection, it should be noted down that MAPP and FR have polar groups ($-OH$) and they can compete with each other in order to encapsulate the surface of EFB fibers, thereby resulting in two different types of interaction between fibers and PP matrix. Although the possible reaction mechanism between natural fiber and polymer matrix in presence of either MAPP or MAPP+FR was described earlier [25-27], where the formation of hydrogen and ester bonds between fiber and PP and a barrier of formation of these bonds between them due to FR are shown even though FR can make both hydrogen and ester bonds only with MAPP. Clearly, the reduction in interfacial bonding between EFB fibers and PP matrix in presence of FR affects to decrease in TS . Fig. 5 (b) illustrates the variation in TS of the composites as a function of fiber content with and without MAPP and FR contents (See Table I). Both UEFBF and TEFBF-based composites without MAPP and FR show a gradual fall of TS with the increase of fiber content. In contrast, the TS gradually increase in coupled

composites without FR for cases of both TEFBF and UEFBF, with noticeable effect of treated fibers. The TEFBF and MAPP reinforced PP composites (TEFBF+MAPP+PP) at 50 wt % fibers without FR exhibit the maximum TS (46 MPa) than that (37 MPa) exhibited by the UEFBF+ MAPP+PP. Thus, the former one represents an increase in TS by 24% from the latter one.

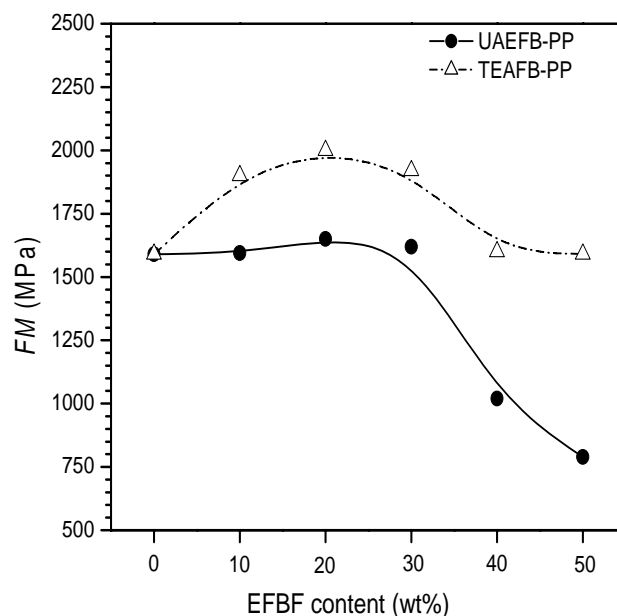
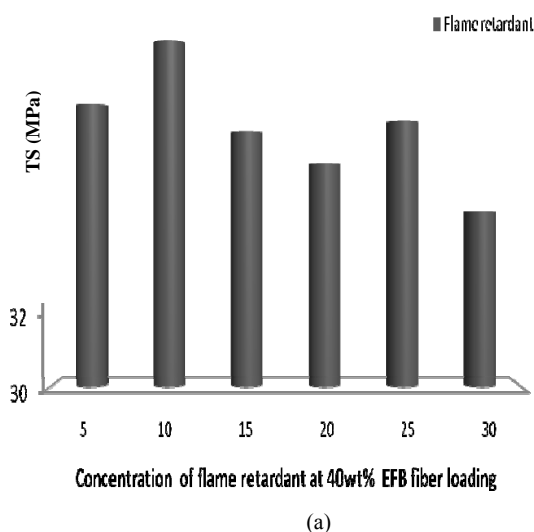


Fig. 4 The variations in FM of composites as a function of untreated and treated EFB fiber content



(a)

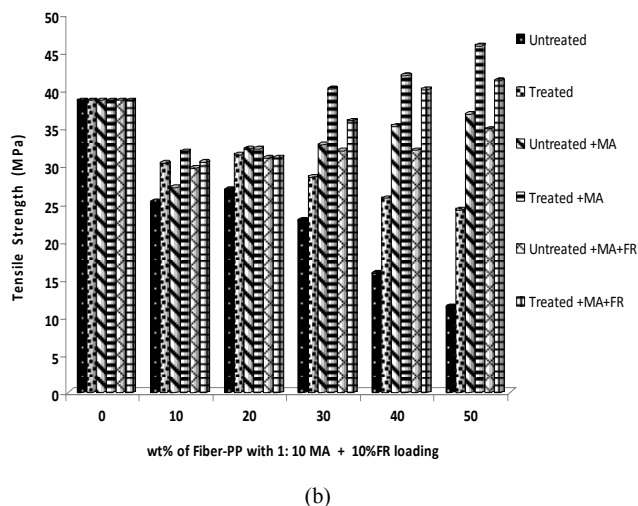


Fig. 5 (a) *TS* as a function of FR contents of composites with 20 wt% EFB fibers and (a) *TS* as a function of fiber content for UEFBF, TEFBF without and with, MAPP and FR (10wt%)

However, the lowering in *TS* for UEFBF-based composites without coupling agent are probably due to the free volume generation during fabrication, leading to a gradually weak interfacial bonding by the increase of UEFBF. On the other hand, chemical pretreatment of fibers improve the interfacial bonding between TEFBF and PP matrix [28], and this improvement is further strengthened with coupling agent in case of TEFBF reinforced composites. The only incorporation of MAPP couples the fibers with both hydrogen and ester bonds, which become enriched with the addition of more MAPP, and due to these reasons the coupled composites show superior mechanical strength. On the other hand, the addition of both FR and MAPP results in a competition of interaction PP matrix and fibers surfaces and eventually compromise to settle down the mechanical property in the composites, as discussed before.

The *FM* of various composites with the addition of MAPP and FR is shown in Fig. 6 (a). The *FM* slightly increases when 10 wt% FR is loaded in PP. As EFB fiber content rises in the composites, the *FM* increases. The maximum *FM* is achieved at the highest EFB fiber loading of 50 wt%. At this fiber loading, the values of *FM* for UEFBF+PP and TEFBF+PP are 0.75 and 1.5 GPa, respectively. TEFBF+ MAPP+ FR+PP shows the *FM* value of 3.4 GPa, followed by TEFBF+ MAPP+PP and UEFBF + MAPP +FR+PP, which show 3.5 GPa and 2.8 GPa, respectively. The overall trend of *FM* values of the composites investigated can be arranged in ascending order, as follows: UEFBF+PP < TEFBF+PP < UEFBF+MAPP+PP < UEFBF+MAPP+FR+PP < TEFBF+MAPP +PP < TEFBF+MAPP+FR+PP. The maximum increase in *FM* for coupled composites with FR from its respective uncoupled composite without FR is 133%. The *FM* of TEFBF+MAPP+PP increases by 35% from UEFBF+MAPP+PP at 50 wt% fiber loading. At high EFB fibers loading, the *FM* shows almost the same value for both

untreated and treated fiber reinforced coupled composites. This is probably due to the balanced effect of favorable entanglement between PP chain and EFB fibers, as declared elsewhere [29].

Fig. 6 (b) shows a comparison of impact strength (*IS*) for various composites, indicating a gradual increase in *IS* with the increase of fibers loading and then it shows a decrease in case of UEFBF or TEFBF with MAPP+FR+PP. Basically, the *IS* determines the absorbed energy of a material during fracture. Increased *IS* may attributed to the toughening of the composites by treated EFB fibres, whereas its decrease by the addition of high FR content is an adverse effect. The presence of high loading of solid FR particles embedded in the polymer matrix results in the reduction in absorbed impact energy.

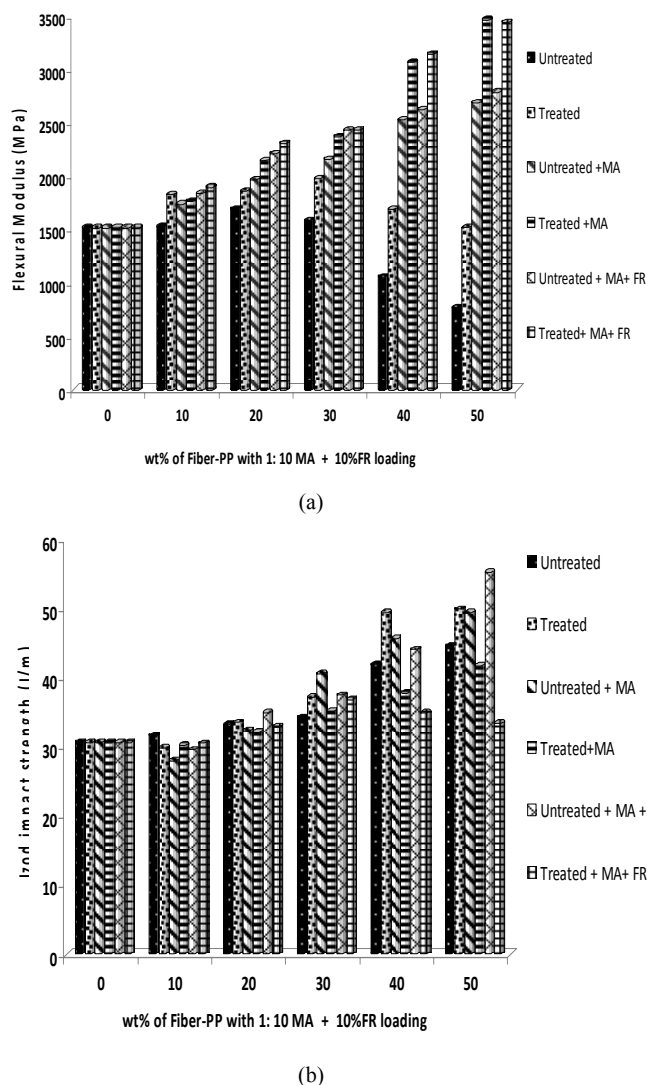
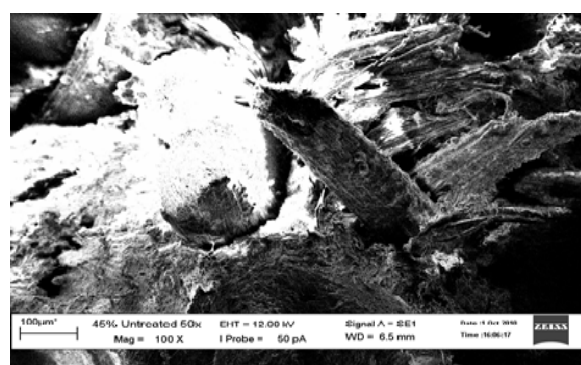


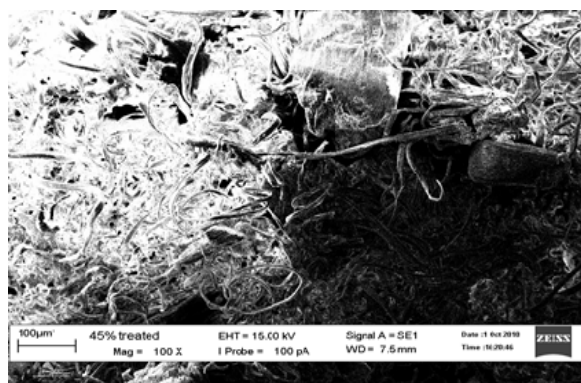
Fig. 6 The variations of (a) *FM* and (b) *IS* of the composites for increasing fiber loading with and without MAPP and FR

D. Surface Morphology

Fig. 7 illustrates the SEM images of tensile fractured surface of (a) UEFBF+PP and (b) TEFBF+PP. From the micrographs, it is obvious that huge fibers are found to be pulled-out from the matrix of both composites, where the main differences observed is that the thickness of TEFBF is smaller than UEFBF, and the surface of treated fibers reinforced composites is smoother than that of untreated ones. These are the consequences of alkali treatments that defragment the fibrils of a fiber for better mechanical interlocking [30-31]. Fig. 8 (a)-(c) shows morphologies of the tensile fractured surface of (a) UEFBF+MAPP+PP, (b) TEFBF+MAPP+PP and (c) TEFBF+MAPP+ FR+PP, showing encapsulated structures that are prevalent for the first two samples. The development of encapsulation morphology may suggest the growth of interfaces among PP, EFB, and FR. In absence of FR, fibers are encapsulated with PP through MAPP by ester bonds, and these types of textures are prominent in the surface morphologies of Fig. 8 (a) and (b). On the other hand, the polar groups of FR make ester bonds with MAPP rather than fibers and hence a less encapsulated pattern is visible in FR loaded composite surface.

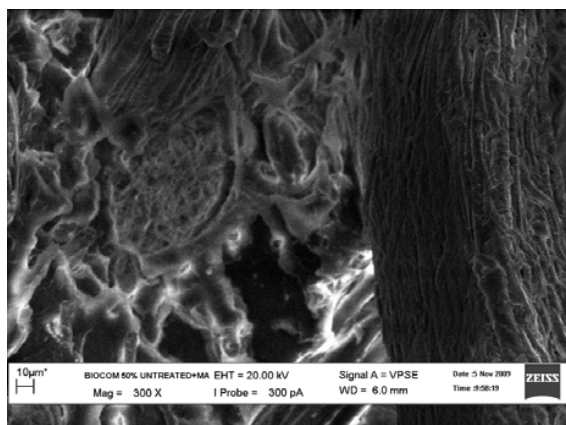


(a)

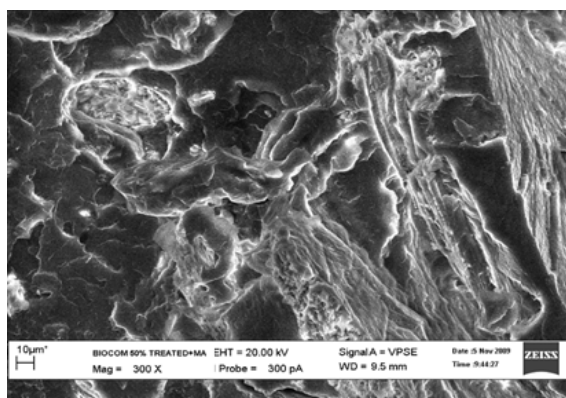


(b)

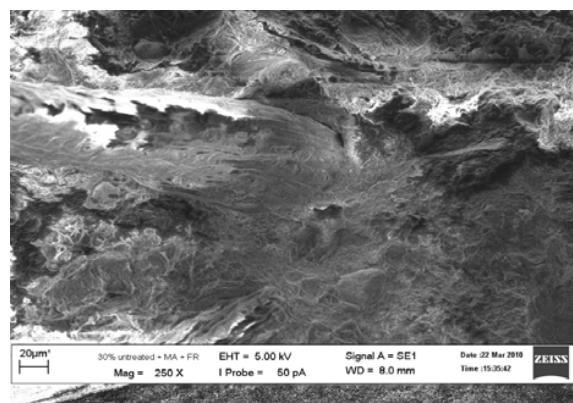
Fig. 7 SEM images of tensile fractured surface of the (a) UEFBF and (b) TEFBF reinforced composites



(a)



(b)



(c)

Fig. 8 SEM morphology of tensile fractured surface of the (a) UEFBF+MAPP+PP, (b) TEFBF+MAPP+PP and (c) TEFBF+MAPP+FR+PP

E. Flammability

The flammability of composite materials with TEFBF and MAPP, as influenced by the addition of FR of different concentrations, is demonstrated in Fig. 9. The composite without FR shows the highest burning rate of 16 mm/min, but with FR of 30 wt% it shows the value of 10 mm/min, resulting

in the burning resistance by 60%. The burning rate gradually decreases with the increase of FR contents, indicating the effectiveness of FR in the composites. On the other hand, the char residue of composites was observed to decrease with the increase of FR concentration after burning tests. Thus, the results suggest that the addition of FR improves the flame resistance property of the TEFBF+MAPP+ FR+PP.

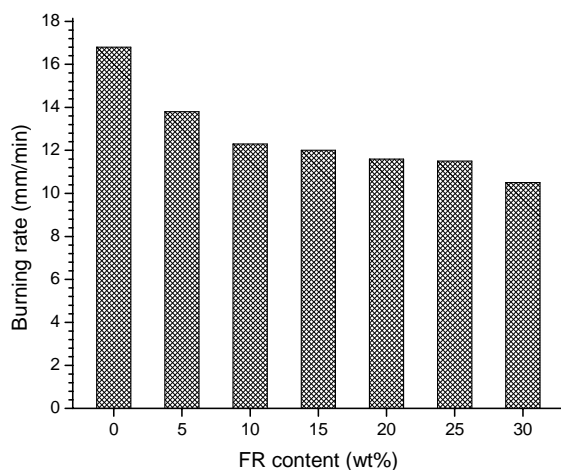


Fig. 9 Burning rate as a function of FR of the composites

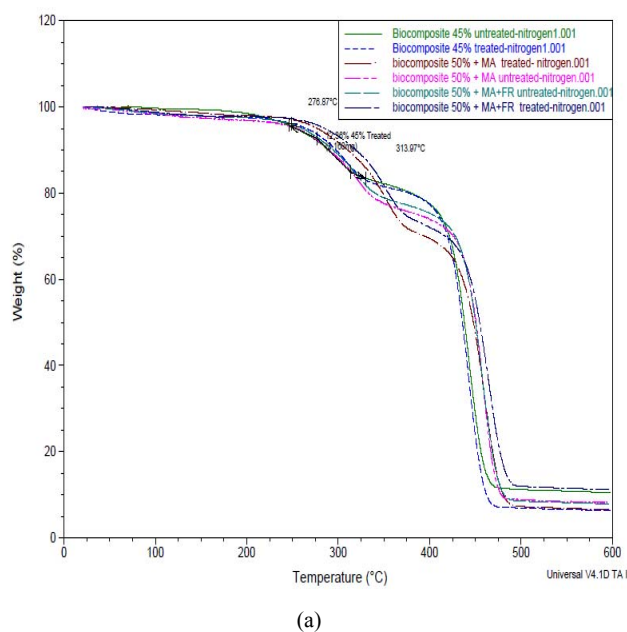
F. Thermal Properties

Fig. 10 (a) and (b) respectively demonstrates the TGA and DTG thermograms of various composites, showing two-stage degradations. The first stage degradation takes place in the temperature range of 200–380 °C, while the second stage occurs in the range of 400–500 °C. The thermal degradation histories are summarized in Table II, where the values of the temperatures of onset degradation (T_0), first-degradation (T_1), second-degradation (T_2) and the maximum-degradation (T_{max}) as well as the char residues are inserted. The data indicate that TEFBF based composites are thermally more stable than UEFBF based composite. The second stage of weight loss was attributed to the cleavage of chemical bonds of glucosidic linkage of cellulose elsewhere [32], where the thermal degradation of fiber decomposition products was claimed to lead to tar formation through levoglucosan. The EFB fiber composites with the addition of MAPP and FR show an enhancement of thermal stability of the composites because of the shifting of the sharp weight-fall of TGA curves to higher temperature (Fig.10 (b)). This high thermal stability of the samples may be an indicator of the formation of encapsulated structure by ester bonding between EFB fibers and PP, as observed in SEM images. Other reason could be due to the capability of MAPP, which can make bonds with hemicelluloses of UEFBF or TEFBF, thus stabilizing its structure and improving the thermal stability of the composites. The FR loaded composites exhibit higher thermal stability than FR unloaded composites, possibly because of the high thermal resistance of FR or of its high heat capacity.

TABLE II

A COMPARISON OF THERMAL DEGRADATION TEMPERATURES AND CHAR RESIDUES OF PURE PP, EFB FIBERS AND THEIR COMPOSITES WITH 20 WT% FIBERS

Sample	T_0 (°C)	T_1 (°C)	T_2 (°C)	T_{max} (°C)	Residue (%)
PP	206	—	—	450	0
UEFBF	196	—	—	340	26
TEFBF	215	—	—	359	17
UEFBF+PP	256	276	423	457	10
TEFBF+PP	260	282	422	458	6
UEFBF+MAPP+PP	248	295	442	467	8
TEFBF+MAPP+PP	256	323	439	474	6
UEFBF+MAPP+FR+PP	254	293	437	471	7
TEFBF+MAPP+FR+PP	264	31	443	478	11



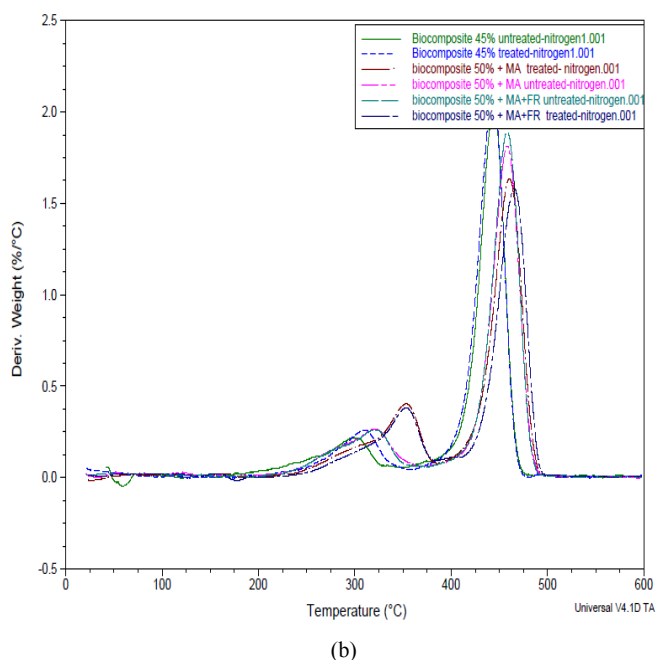


Fig. 10 (a) TGA and (b) DTG thermograms of the composites

IV. CONCLUSION

Chemical pretreatment using NaOH is applied on EFB fibers, which have been added along with MAPP and FR into PP for the production of thermally more stable, insulating composite materials. Alkaline treatment eliminates lignin, hemicelluloses etc. from fibers, resulting in an improved interfacial adhesion between EFB fiber and PP matrix. Even though alkaline treatment improves mechanical properties of the treated fiber loaded composites, higher fiber loading causes a weak attachment of EFB fibers onto PP matrix due to fiber aggregation. Then, the addition of MAPP improves the interfacial adhesion through the development of encapsulated structure and enhances mechanical properties, especially the tensile modulus by 133%. The thermal stability of the MAPP loaded composites also increase. Besides, incorporation of FR in the composite influences mechanical properties as a result of its less compatibility with fibers or PP but provides enhanced thermal stability of the composites. It is found that 10 wt% of FR shows the maximum tensile strength of composites and the flammability decreases significantly with the increasing FR concentration. Therefore, it can be concluded that addition of FR improves the flame retardancy of composites and renders good mechanical properties with synergistic performance of flammability.

ACKNOWLEDGMENTS

The authors would like to thank the Director General of MPOB for permission to publish this paper. The authors also wish to gratefully acknowledge the funding from MPOB to this study.

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