Palm-Based Bio-Composites Hybridized with Kaolinite

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ABSTRACT: This study described the mechanical and thermal properties of hybrid bio-composites from oil palm empty fruit bunch (EFB) fibers and kaolinite. The polyurethane (PU) used as matrix is formed by reacting palm kernel oil (PKO)-based polyester with crude isocyanate. The blending ratio of PU to EFB fibers was fixed at 35 : 65 and kaolinite was added at 0, 5, 10, 15, and 20% (by weight). The occurrence of chemical interactions between the hydroxyl terminals in both fillers and the PU system was determined via FTIR spectroscopy. Hybrid bio-composites showed improved stiffness, strength, and better water resistance with the addition of kaolinite to an extent. At

15% of kaolinite loading, maximum flexural and impact strengths were observed. The interaction between kaolinite with PU matrix and EFB fibers enhanced the mechanical properties of the bio-composites, which was justified from the FTIR spectrum. However, over-packing of kaolinite was observed at 20% kaolinite loading, which ruptured the cellular walls and degraded strength of the biocomposites. © 2007 Wiley Periodicals, Inc. J Appl Polym Sci 105: 2488–2496, 2007

Key words: polyurethane; hybrid bio-composites; oil palm empty fruit bunch fiber; kaolinite

INTRODUCTION

Natural fiber reinforced polymer composites or biocomposites have been emerging lately as an environmental-friendly and cost effective option compared with synthetic-filled composites such as aramide, glass fibers, or carbon. Acceptable specific strength properties, low cost, low density, nonabrasive, enhanced energy recovery, and biodegradable are factors contributing to the value added of these lignocellulosic materials.¹ Besides, they have good thermal stability² and provide excellent insulation against heat and noise, which increased the values of these bio-fibers. Ease in processing also gives an advantage of bio-fibers over synthetic fibers. Many researches have been carried out to utilize these natural fibers to produced composites especially fibers from empty fruit bunch (EFB),^{3–5} kenaf, banana fiber,⁶ jute,⁷ hemp,^{8,9} coir,^{10,11} flax,¹² kapok fabric,¹³ ramie,¹⁴ etc. Low density of the bio-composite would also contribute to its poor properties as reported by Bledzki et al.¹⁵ Because of limited function of the bio-composites produced, especially their low mechanical

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strength and stiffness, hybrid bio-composites were discovered to be an effective way to enhance mechanical and thermal properties of the bio-composites, especially for structural application. The technology in producing hybrid composite would be able to enlarge the domain of applications shown by the existing conventional composites.

Many studies have been carried out to see the improving properties of the hybrid bio-composites. Burgueno et al.¹⁶ prepared hybrid bio-composites made from flax fibers with unsaturated polyester and hybridized with woven jute, chopped glass, and unidirectional carbon fabrics. Results obtained showed an improvement in stiffness, strength, and moisturesorption stability, while flexural test demonstrated enhanced structural behavior. Rozman et al.¹⁷ studied the flexural and tensile properties of the hybrid composites of oil palm EFB fibers and glass fiber with polypropylene (PP) as the matrix. The incorporation of both fibers into PP matrix has resulted in the reduction of flexural and tensile strength. These properties were altered using coupling agents such as maleic anhydride and polymethylenepolyphenyl isocyanante. On the other hand, Mwaikambo and Bisanda¹³ prepared hybrid bio-composites with cotton-kapok fabric reinforced with unsaturated polyester. The impact strength decreased with increase in fiber volume fractions of bio-composites produced.

Kaolinite is one of the clay types in kaolin group, besides nacrite, halloysite etc.¹⁸ They have the same chemical formula, Al₂Si₂O₅ (OH)₄ but differ in layer stacking arrangement, hydroxyl (OH) positions and



Figure 1 Structure of kaolinite.¹⁸

OH bonding between interlayer. The structure consists of interconnecting layers between tetrahedral layer of SiO_4 and the octahedral layer of AlO_6 . Both layers are interconnected through bridging oxygen atoms as shown in Figure 1. The octahedral layer is covered by the hydroxyl group while the tetrahedral layer is characterized by the ditrigonal-shaped spacings. Its surface consists of oxygen shared by two silica groups at the center. Both layers are connected together by hydrogen bond between the surface hydroxyl group at the octahedral section and the oxygen atom at the tetrahedral section.¹⁸

In this study, hybrid bio-composites were prepared from oil palm EFB fibers hybridized with kaolinite with PKO polyester resin blended with isocyanate (to form polyurethane) as the matrix. The mechanical and thermal properties of the hybrid bio-composites were reported. The ratio of PU matrix to EFB fibers was fixed at 35 : 65 and the percentage of kaolinite used were 0, 5, 10, 15, and 20% (based on the overall weight of the mixture). FTIR analysis was carried out to determine the chemical interaction between PU, EFB, and kaolinite. The mechanical properties studied were flexural and impact strength, and evaluation of dynamic mechanical properties. Water absorption behavior of hybrid bio-composites was also determined.

EXPERIMENTAL PROCEDURES

Materials

The RBD palm kernel oil (PKO)-based polyester was prepared using method described by Badri et al.¹⁹ The 4,4'-diphenylmethane diisocyanate (MDI) was obtained from Cosmopolyurethane (Port Klang, Malaysia) with a NCO content of 31%. Tetramethylhexanediamine (TMHDA) was also obtained from Cosmopolyurethane (M) Sdn. Bhd. The silicone surfactant (Tegostab B8404[®]) was generously being supplied by Th. Goldschmidt Ltd., Singapore. Sabutek (M) Sdn. Bhd, Perak, Malaysia supplied the oil palm EFB fiber, while the kaolin powder was obtained from Kaolin (M) Sdn. Bhd. Malaysia.

Preparation of kaolin

The kaolin (kaolinite type) went through sieving procedure and was used without any further treatment. An Endicott sieve was used to screen the kaolinite into size $< 20 \ \mu\text{m}$. The kaolin was then analyzed with particle size analyzer type Microtrax-X100 that revealed the actual size as 17 μ m (100%).

Preparation of oil palm EFB fibers

The fiber was ground several times with a grinding machine model OM to obtain sizes ranging 250–500 μ m and then re-ground with grinding machine model FDS-IHP to get size of less than 160 μ m. The fiber was then sorted using sieves model Fritsch of sizes 160 and 100 μ m. The average size used was 150 μ m (62%) and 442 μ m (38%) obtained from particle size analyzer type Microtrax-X100. The measured density is 0.7 g ml⁻¹ by mean of overflowing method. It was then dried at 105°C for 20 h in a convection oven.

Preparation of the PKO-based resin

The PKO-based polyester (100 part by weight, pbw) is blended with 0.6 pbw TMHDA and 2 pbw silicone surfactant (Tegostab B8408[®]) for 5 min at 200 rpm using a mechanical stirrer. It is then conditioned to room temperature.

Preparation of hybrid bio-composites

Hybrid bio-composite was produced by mixing two type of fillers, kaolinite and EFB fibers with the PKObased resin mixed with isocyanate (PU matrix). The ratio of PU matrix (one to one ratio) to the EFB fibers used is 35 : 65. Kaolinite was added in the PU blend with the amount of 0, 5, 10, 15, and 20% (by weight) individually. Then, this mixture was mixed with EFB fiber and stirred in a beaker for 60 s before being poured into waxed cavity plates. It was then placed in a hot pressing machine with processing temperature of 35°C for 9 min under pressure of 760 MPa. The hybrid bio-composites were conditioned at room temperature for 24 h. They were then cut to desired size using Seppach circular saw model TS400IS for further characterizations.



Figure 2 FTIR spectra of (a) unfilled PU and (b) PU-filled with 20% kaolinite.

Characterization of the bio-composites

Fourier transform Infrared (FTIR) spectroscopy analysis

Fourier Transform Infrared (FTIR) spectroscopy analysis was carried out using Perkin–Elmer (GX spectrum) with Diamond Attenuation Total Reflectance (DATR) type and KBr method. Identification of presence of new peaks and shifting of peaks was carried out.

Flexural (three-point-bending) test

Three-point bending test was conducted according to ASTM D 790-86. The bio-composites produced were cut to test samples with dimensions of 120 mm \times 12 mm \times 3 mm (length \times width \times thickness). The test was carried out using Instron Universal Test Machine model 5525 at a cross-head speed of 3.1 mm/min. Tabulated values represented the mean of five specimens.

Impact strength test

The impact strength test was conducted according to ASTM D 256-88. The Izod method was carried out using unnotched samples with dimensions of 63 mm \times 13 mm \times 3 mm using a Zwick Impact tester model 5101 with a pendulum of 2 Joule (J) energy. The impact strength was calculated by dividing the energy (J) recorded on the tester by the cross-sectional area of the specimen. Tabulated values represented the mean of five specimens.

Dynamic mechanical analysis (DMA)

The analysis was carried out using a dynamic mechanical analyzer model TA DMA 2980. The experiment was performed in a single cantilever at a frequency of 1 Hz. The sample was cut into a 50 mm \times 10 mm \times 3 mm specimen. The testing temperature ranged from 30 to 200°C with heating rate of 5°C min⁻¹.



Figure 3 FTIR spectra of (a) bio-composite and (b) hybrid bio-composite with 20% kaolinite.

Water absorption

The measurement of water absorption was determined according to ASTM D 570-8 (Reapproved 1988). The samples were immersed in water for 7 days and the reading was taken every 24 h. The weight percentage gain (WPG) was calculated using a formula as given below:

WPG(%) =
$$\frac{(W_2 - W_1)}{W_1} \times 100$$

where W_1 and W_2 represent weight of bio-composites before and after immersion in water, respectively.

Scanning electron microscope (SEM) analysis

A Leo VP SEM-1450 SEM was used to study the adhesion and distribution of kaolinite in bio-composites using the impact fractured samples. The samples were coated with gold of thickness 20 Å with Bio-rad microscience division-SC 500 to prevent electrical charging during examination. The observation was made at 100 times magnification. Samples observed for comparison were samples of the biocomposite (0% kaolinite) and the hybrid biocomposites (5, 15, and 20%).

RESULTS AND DISCUSSION

Fourier transform infrared (FTIR) spectroscopy analysis

Figures 2 and 3 showed the present of new peaks in the FTIR spectra when 20% kaolinite (maximum loading for clearer peaks) was added into the bio-composites.

Figure 2 showed the FTIR spectra of unfilled PU and the PU-filled with 20% kaolinite. It is clearly shown that there were five differences identified in the spectrum of PU composite with the incorporation of 20% kaolinite and labeled as peak A, B, C, D, and E. Peak A indicated the O-H vibration of kaolinite. Two weak peaks appeared as shoulders near peak A, which arises from internal surface OH groups and internal OH groups of the kaolinite.²⁰ The incorporation of kaolinite also resulted in the shifting of peak 1060 cm^{-1} in the unfilled PU to 1032 cm^{-1} (peak B), referred to as the Si-O stretching bands of kaolinite. Peak C referred to the OH deformation bands of kaolinite at 938 cm⁻¹ (small peak) and 913 cm⁻¹ (sharp peak), arise from vibrations of the inner OH groups and inner surface OH groups, respectively. Peaks D and E were also referred to the bonding of Si-O and AlO deformation bands at 536 and 466 cm^{-1} ²⁰ respectively. PU composite with 20% kaolinite also showed an increased absorption in the carbonyl (C =O) regions at 1719 cm^{-1} . This sharper peak is a good evidence for the formation of ester linkages from the reaction between kaolinite and hydroxyl groups (OH) of PU matrix. CH stretching around 2850–2924 cm⁻¹ from PU matrix was detected in all bio-composites spectra.

Figure 3 showed the FTIR spectra of bio-composite (EFB fiber with PU matrix) and the hybrid bio-composite with 20% kaolinite. Both spectrum of hybrid bio-composite have five distinct peaks labeled as A, B, C, D and E. Peak A exhibited a broad O-H vibration of kaolinite without shoulders. Peak B to Peak E were the same vibrations and stretching bands observed in the PU-filled with 20% kaolinite [Fig. 1(b)]. Hybrid composites also showed a clear absorption of carbonyl (C = O) at 1719 cm⁻¹. This could be a strong indicator of the formation of ester linkages from the reaction between kaolinite and hydroxyl groups (OH) from PU matrix. CH stretching at 2850–2923 cm⁻¹ of the PU matrix was detected in both bio-

Figure 4 The flexural strength of hybrid bio-composites with 0, 5, 10, 15, and 20% of kaolinite loading.



Figure 5 The flexural modulus of hybrid bio-composites with 0, 5, 10, 15, and 20% of kaolinite loading.

composites and hybrid bio-composites spectra. In general, the compatibility between polymer matrix and filler is one of the main criteria to produce a good mechanical characteristic of a composite.²¹ Hence, the interaction occurred in hybrid bio-composites were reflected in the mechanical and thermal properties, which will be discussed later.

Mechanical properties

Figure 4 showed the trend in flexural strength of hybrid bio-composites with 0, 5, 10, 15, and 20% kaolinite. Flexural strength or modulus of rupture (MOR) depends on the stiffness of the hybrid bio-composite. The readings obtained referred to specimens when they started to fracture.⁴ In general, inclusion of kaolinite decreased the flexural strength of the bio-composites and optimized at 15% loading (11.2 MPa). Biocomposite without kaolinite (10.8 MPa) showed higher flexural strength compared to 5% (8.2 MPa), 10% (8.6 MPa), and 20% (8.6 MPa) of kaolinite. This indicated that kaolinite influenced the strength and stiffness of the hybrid bio-composites. Better adhesion between the PU matrix and EFB fibers was observed in the hybrid bio-composites. The presence of kaolinite disturbed the interfacial adhesion of PU matrix with EFB fibers except for at optimum loading of 15%. At this level, the enhanced properties of the hybrid bio-composites were contributed to the interaction of PU matrix that was sandwiched in between layers of the kaolinite. This was supported by the FTIR spectrum (Figs. 2 and 3). Inclusion of kaolinite at more than 15% caused an over packing phenomenon and resulted in deteriorating mechanical properties.

Figure 5 showed the performance of hybrid biocomposites in flexural modulus. The flexural modulus followed the same trend as the flexural strength where the maximum loading of kaloinite was observed at 15% at 11 and 2550 MPa for the flexural



Figure 6 Impact strength of hybrid bio-composites with 0, 5, 10, 15, and 20% of kaolinite loading.

strength and modulus respectively. The flexural strength dropped upon introduction of kaolinite to the bio-composites until 15% kaolinite loading. The flexural modulus increased almost linearly with increasing percentage of kaolinite up to 15% kaolinite loading (2550 MPa) and decreased thereafter. Biocomposite (0% kaolinite) showed lower flexural modulus (1340 MPa) compared to the hybrid bio-composites. Flexural modulus referred to the elasticity of the specimen.²² The absence of plasticizer in the PU blending lowered the flexural modulus especially in the bio-composites compared to the hybrid bio-composites. The presence of chemical interaction between kaolinite and PU matrix increased the modulus of hybrid bio-composites, which was supported by the FTIR spectra in Figure 3. Again, at 20% loading, over packing problem occurred, deteriorated the strength of the hybrid bio-composites due to lowering in the plasticizing effect.

Figure 6 showed the result of impact strength of the hybrid bio-composites. These results gave the same observation as the flexural strength. The bio-composite without kaolinite was stiffer (impact strength of 4000 MPa) than the hybrid bio-composites with 5%



Figure 7 Water absorption of hybrid bio-composites with 0, 5, 10, 15, and 20% kaolinite.

and 10% kaolinite. Optimum level of kaolinite was detected at 15% loading with impact strength of 4600 MPa. At this level, the strength of the hybrid bio-composites increased due to the encapsulation of the EFB fibers by the PU in combination with kaolinite filling the voidages in between EFB fibers. Again, over-packing problem resulted in reduction in strength at 20% loading of kaolinite.

Kaolinite filled the voidages in between fibers, in the PU cellular structure and the struts excessively (over-packing effect) resulted in ruptured of the cellular walls. In, addition, kaolinite, which is very common being used as nucleating agent in the formation of cellular plastic also contributes some foaming effects in the preparation of the hybrid biocomposite. As a result, to an extent (20% kaolinite loading), the foaming becomes greater, and resulted in more weak points and becoming brittle. These phenomena deteriorated the flexural and impact strength of the hybrid bio-composite without obvious effect on its hardness.

Water sorption behavior

Figure 7 showed the water sorption behavior of hybrid bio-composites with 0, 5, 10, 15, and 20% of kaolinite loading. Bio-composites without kaolinite showed the highest percentage of water absorption. Other than the hydrophilicity of the EFB fibers, it is much related to the density of the bio-composite. The density of bio-composite without kaolinite was in the range of 800–950 kg m⁻³ and the incorporation of kaolinite increased the density to 900–1050 kg m⁻³ as shown in Table I. This increment of density was contributed by the density of kaolinite itself. Hence, the hybrid bio-composites with the same thickness (3 mm) were more compact and dense and thus the water sorption of unfilled bio-composites.

On the other hands, the hybrid bio-composites showed an increasing percentage of water absorption with increasing kaolinite loading. This is due to the nature of kaolinite that was very hygroscopic. However, hybrid bio-composite with 15% of kaolinite loading obviously showed a reduction of water sorption after day 2. This indicated the presence of good interaction of PU matrix to EFB fibers and kaolinite

TABLE I Density of the Hybrid Biocomposites With the Inclusion of Kaolinite

Kaoilinite loading (%)	Density (kg m $^{-3}$)
0	925
5	984
10	1001
15	1076
20	1123



Figure 8 Storage modulus of hybrid bio-composites with 0, 5, 10, 15, and 20% kaolinite.

where the PU matrix encapsulated both the EFB fibers and the kaolinite. The presence of kaolinite introduced a well dispersed EFB fibers and kaolinite themselves in the resin, being encapsulated by the resin and finally reacted with the isocyanate to produce poyurethane composite.

In general, percentage of water absorption showed large initial uptake for each bio-composites. This is contributed by the porous structure of EFB fibers, which transports the water via capillary into the gaps and flaws at the interfaces between fibers and matrix.²³ For day three to day seven, the percentage of water absorption increased almost linearly which consisted of diffusion of water molecules inside the microgaps between matrix chains. It also contributed by the transport of water into microcracks in the matrix, formed during the blending process.

Dynamic mechanical (DMA) analysis

DMA measures the deformation of a material in response to vibrational forces. The storage modulus, E' indicates stiffness of a material and tan δ gives the amount of energy dissipated as heat during the deformation.²⁴ The investigation of dynamic storage modulus and internal friction over a wide range of temperatures and frequencies has proven to be very useful in studying the structure of polymers and the variations of properties in relation to end-use performance.²⁵ Figure 8 showed the storage modulus of hybrid bio-composites with 0, 5, 10, 15, and 20% kaolinite. DMA curves indicated that the storage modulus, E' decreased abruptly around the onset glass transition (T_q) temperature of PU matrix. Addition of kaolinite increased the storage modulus of the biocomposites to an optimum level at 15% loading (2150 MPa) indicating an increase in the ability to store energy. This is clearly shown in Figure 8. The hybrid



Figure 9 Tan δ of hybrid bio-composites with 0, 5, 10, 15, and 20% of kaolinite.

bio-composite with 20% kaolinite showed low storage modulus compared to hybrid bio-composite with 15% kaolinite. This is resulting from the amount of filler that over-packed the PU matrix and gave out poor mechanical properties. This result supported the flexural and impact strength properties, which was discussed earlier.

Values of T_g depend on the chemical structure. Flexibility of the molecular chain, steric hindrance and bulkiness of the side groups attached to the backbone chain affect the value of T_g . DMA is a sensitive technique compared to Differential Scanning Calorimetry (DSC) to determine the T_g and has been used in studying the effect of temperature on the mechanical properties of materials including polymers and composites.²⁵ The onset, loss modulus and tan δT_g obtained from DMA is shown in Figures 8, 9, 10 and Table II. The hybrid bio-composite with 15% kaolinite has the lowest onset T_g (59°C). In the glassy region, thermal energy is sufficient to surmount the potential barriers for translational and rotational motions of



Figure 10 Loss modulus of hybrid bio-composites with 0, 5, 10, 15, and 20% kaolinite.

	Loss		
	Onset	Modulus	tan δ
Specimen	$T_{g'}$ (°C)	$T_{g'}$ (°C)	$T_{g'}$ (°C)
Bio-composite (0 % kaolinite)	70	87	115
Hybrid bio-composite (5 % kaolinite)	67	91	124
Hybrid bio-composite (10% kaolinite)	70	90	129
Hybrid bio-composite (15% kaolinite)	59	84	110
Hybrid bio-composite (20% kaolinite)	68	89	122

TABLE IIThe Onset Glass Transition, T_g of the Hybrid Bio-Composites From DMA Analysis

segments of the polymer molecules. Above the T_g temperature, the amorphous polymer is softer and more flexible.

Delta (δ) in tan δ sometimes referred to as internal friction or damping such as vibration or sound damping. It is the ratio of energy dissipated per cycle to the maximum potential energy stored during a cycle. Figure 9 delineated the variation of tan δ of bio-composite and hybrid bio-composites. An improvement in interfacial bonding between matrix and filler or filler to filler adhesion was observed with lowering of tan δ values.⁶ The higher damping at the interfaces was, the poorer the interface adhesion would be. The lowest tan δ was observed on the bio-composites without kaolinite. Bio-composites with uniform distribution of EFB fibers encountered strong molecular interactions between the fibers and the polyurethane matrix. It is interesting to note that hybrid bio-composite with 15% kaolinite gave the next lowest tan δ , indicating the presence of better filler and matrix bonding. Tan δ is also used to evaluate T_g of the samples. From Table II and Figure 9, it is justified that T_g for hybrid biocomposite with 15% kaolinite has the lowest temperature. This is also in agreement with T_g obtained from storage modulus. The width of tan δ peak for 15% hybrid bio-composite was much broader compared to others. This behavior suggested that there are better molecular relaxations in 15% kaolinite hybrid biocomposite. The molecular motions at the interfacial region generally were contributed to the damping of the material apart from those of the constituents.²⁴

Scanning electron microscope (SEM) analysis

SEM micrographs was used to study the adhesion and distribution of kaolinite in bio-composites using the impact fractured samples. The observation was made at 100 times magnification on impact fractured samples of the biocomposites (0% kaolinite) and hybrid bio-composites with 15 and 20% kaolinite as shown in Figure 11. These micrographs supported the findings in the analysis of the mechanical properties. Biocomposite without kaolinite showed encapsulation of PU matrix around the EFB fibers. The fractured surface was smooth as shown in Figure 11(a) as the tendency for the fiber to loosen from the PU matrix was absent. However, when 15% kaolinite was included, the PU matrix encapsulated the individual particles of kaolinite and resulted in a uniform distribution of filler in the matrix [Fig. 11(b)]. Some of the kaolinite covered surface and lumen of the EFB fibers as well as voidages in between fibers. This produced better bonding between the PU matrix and kaolinite and reduced the intrafacial adhesion of kaolinites (kaolinite to kaolinite bonding).



Figure 11 SEM micrographs of (a) the bio-composite (0% kaolinite) and hybrid bio-composites with (b) 15% kaolinite and (c) 20% kaolinite.

It was also observed that with higher loading of kaolinite, the occurrence of nucleation increased and built up more spherical cellular structure in the hybrid bicomposites. It was suspected that the kaolinite filled these sites (spherical gaps) until it reached the saturation points (15% kaolinite), where it can no longer being hold by the PU matrix (20% kaolinite). The over-packing phenomenon occurred and created weak points of rupture.

CONCLUSIONS

Results from the study indicated that addition of kaolinite had successfully improved the mechanical and thermal properties of the bio-composites to an extent. Although at lower percentage loading, there was an obvious evidence of some deteriorating effects, the strength was built up with the addition of 15% kaolinite. Some chemical interactions existed between PU matrix and EFB and between PU matrix and kaolinite. The incorporation of kaolinite into the EFB fibers could be as high as 15%. At > 15% kaolinite loading, the hybrid biocomposites encountered over packing problem that caused weak adhesion between PU matrix and the fillers: EFB and clay.

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