Tensile Properties, Water Uptake, and Thermal Properties of Polypropylene/Waste Pulverized Tire/Kenaf (PP/WPT/KNF) Composites

Ai Ling Pang [a] and Hanafi Ismail [a,b,*]

The effects of kenaf (KNF) loading and 3-aminopropyltriethoxysilane (APTES) on the tensile properties, water uptake, and thermal properties of KNF-filled polypropylene (PP)/waste pulverized tire (WPT) composites were investigated. The composites were prepared using a Thermo Haake Polydrive internal mixer, where KNF loadings (0, 5, 10, 15, 20 phr) and constant PP/WPT (70/30) were used. The results showed that the tensile modulus and water uptake increased, but tensile strength and elongation at break decreased, with increased KNF loading. The composites with APTES exhibited a higher tensile strength and tensile modulus, but lower elongation at break and water uptake than composites without APTES. The presence of APTES enhanced the interfacial adhesion between PP/WPT matrices and KNF. Thermal stability of PP/WPT/KNF composites increased with KNF loading, and was found slightly higher, but insignificant for composites with APTES. Fourier transform infrared (FTIR) spectra analysis was performed to study the interactions among the PP, WPT, KNF, and the ethoxy functional groups in APTES.

Keywords: Polypropylene; Waste pulverized tire; Kenaf; Water Uptake; Thermal properties; FTIR

*Contact information: a: School of Materials and Mineral Resources Engineering, Universiti Sains Malaysia, Engineering Campus, 14300 Nibong Tebal, Penang, Malaysia; b: Cluster for Polymer Composites (CPC), Science and Engineering Research Centre (SERC) Universiti Sains Malaysia, Engineering Campus, 14300 Nibong Tebal, Penang, Malaysia; *Corresponding author: hanafi@eng.usm.my*

INTRODUCTION

The use of natural fillers in polymer composites has become a common practice in research and the plastics industry. The growing usage of natural fillers (such as wood fibre, bamboo fibre, rice husk powder, kenaf pulp, luffa fibre, hemp, and jute, *etc.*) in composites applications is due to the variety of benefits offered by these natural fillers. For instance, apart from the attributes of low density and cost, they are also renewable, available in abundance, biodegradable, harmless, and less abrasive to equipment during processing (Xie *et al.* 2010; Santiagoo *et al.* 2011; Ismail *et al.* 2010; Demir *et al.* 2006; Arbelaiz *et al.* 2006). Kenaf (KNF) fibre, also known as *Hibiscus cannabinus*, is one of the most well-known natural fibres utilized as fillers and/or reinforcement in polymer composites applications (Ismail *et al.* 2010a,b; Abdrahman and Zainudin 2011; Janoobi *et al.* 2009).

However, the incompatibility between hydrophobic polymer matrices and the hydrophilic nature of natural fibre has resulted in several problems that affect the performance and properties of the composites. One of the problems is the absorption of moisture by natural fibre in the polymer composites. Moisture uptake by natural fibre can lead to swelling and dimensional changes in the composites, which then leads to poor adhesion between the fibre and polymer matrices. This lack of adhesion will result in low stress transfer efficiency, and this phenomenon can lead to undesirable dimensional and mechanical properties (Zabihzadeh 2010). In order to solve this problem, coupling agents or compatibilizers have been introduced to the polymer system during the processing of composites (Xie *et al.* 2010; Santiagoo *et al.* 2011; Ismail *et al.* 2010). Coupling agents are chemicals that link the hydrophobic polymer matrices and hydrophilic fibres in order to improve the interfacial adhesion between them. Silane coupling agents have been widely used and reported by a number of researchers (Xie *et al.* 2010; Janoobi *et al.* 2009; Zainal and Ismail 2011; Ansari and Ismail 2010; Demjen *et al.* 1999).

Polypropylene (PP) is widely used as a matrix in natural fillers filled polymer composites. PP is well-known for its lower production cost, easy processing properties, low density, and recyclability. These attributes make PP a popular choice as matrix material (Santiagoo *et al.* 2011; Demjen *et al.* 1999; Awang and Ismail 2008; Awang *et al.* 2007; Supri *et al.* 2012).

The growing amount of rubber waste from tire industries and tire users has caused problems in waste disposal management. There are a number of studies on the use of waste tire dust as fillers in polymer composites systems (Awang *et al.* 2007, 2008; Supri *et al.* 2012). Thus far, to the best of our knowledge, the tensile properties, water absorption, and thermal properties of PP/WPT/KNF composites have not been studied or reported. The aim of this research is to study the effect of KNF loading and a coupling agent, 3-aminopropyltriethoxysilane (APTES), on the tensile properties, water uptake, and thermal properties of PP/WPT/KNF composites.

EXPERIMENTAL

Materials

The PP Grade 6331 was supplied by Titan Pro Polymers (M) Sdn. Bhd. (Johor, Malaysia) with a melt flow index of 14 g/10 min at 230°C and a density of 0.9 g/cm^3. WPT, a recycled product of mechanically ground scrap tire, was obtained from Mega Makmur Saintifik Sdn. Bhd. (Malaysia). The particle size of WPT ranged from 0.2 to 150 μm with a specific surface area of 0.089 m^2/g (determined from the Malvern Particle Size Analyzer). Kenaf (KNF) was obtained from Lembaga Kenaf & Tembakau Negara (Kelantan, Malaysia). KNF was ground using a mini grinder from Rong Tsong Precision Technology Co. with Product Id: RT-34. An Endecotts sieve was used to obtain the final particle size of KNF with the range of ≤ 75μm. 3-aminopropyltriethoxysilane (APTES), was supplied by Sigma-Aldrich (Malaysia). The composites were prepared according to the formulations as indicated in Table 1.

Table 1. Compositions of PP/WPT/KNF Composites with and without APTES with Different KNF Loading

Materials (phr)	PP/WPT/KNF					PP/WPT/KNF/APTES			
PP	70	70	70	70	70	70	70	70	70
WPT	30	30	30	30	30	30	30	30	30
KNF	-	5	10	15	20	5	10	15	20
APTES	-	-	-	-	-	1.5	1.5	1.5	1.5

*phr = parts per hundred parts of resin

Composites Preparation

Melt compounding of a series of PP/WPT/KNF composites with different KNF loadings [0, 5, 10, 15, 20 phr] was carried out in a Thermo Haake Polydrive internal mixer at a temperature and rotor speed of 180°C and 50 rpm, respectively, with a total mixing time of 10 minutes. PP was first charged into the internal mixer and melted for 2 minutes prior to the addition of WPT. KNF was then added to the mixture after 6 minutes of mixing. For composites with APTES, APTES was added to the mixture 2 minutes after KNF, and the mixing process was carried out for a total time of 10 minutes. Next, the PP/WPT/KNF composites were compression moulded in an electrically heated hydraulic press (GoTech Testing Machine Model KT-7014 A) to produce 1 mm moulded sheets. Compression moulding of all the compounds was done at 180’C with a preheating time of 6 minutes, followed by hot-pressing time of 4 minutes, and cool-pressed under 6.21 MPa pressure for 2 minutes. The moulded sheets of the composites were then cut into dumbbell-shaped specimens using a Wallace die cutter.

Water Uptake

The water uptake test was carried out in accordance with ASTM D570. The specimens were first dried in an oven for 24 hours at 50°C until a constant weight was obtained. Weight difference after immersion was recorded by weighing them periodically on a Sartorius balance Model: BS224S, with a precision of 1 mg. The excess water on specimen surfaces was removed with tissue paper before weighing. The water uptake test was performed for 30 days and the percentage of water uptake was calculated using Equation (1),

$$W_{\mathrm{t}}(\%) = \frac{W_2 - W_1}{W_1} \text{ x } 100 \tag{1}$$

where W_t is the total water uptake by the specimen, and W_1 and W_2 are the weights of the specimen before and after immersion in distilled water respectively.

Thermal Analysis

The thermogravimetric analysis (TGA) was performed by using a Perkin Elmer Pyris 6 TGA. TGA analysis was conducted in the ramp mode from 30 to 600°C under nitrogen at a flow rate of 50 mL/min. The heating rate was 20°C/min.

Fourier Transform Infrared (FTIR) Spectroscopy

FTIR analysis was performed using a Perkin-Elmer Spectrum GX FTIR spectrophotometer. Small pieces of composites (1 cm x 1cm) were cut from the 1-mm thick compression moulded sheet, placed on a Miracle ATR accessory (miracle base optics assembly), and the spectra were recorded in reflection. The spectrum resolution was 4 cm^{-1} and the scanning range was 4000 to 550 cm^{-1}.

RESULTS AND DISCUSSION

Tensile Properties

Table 2 shows the tensile properties of PP/WPT/KNF composites with and without the 3-aminopropyl-triethoxysilane (APTES) coupling agent. It can be clearly seen that the tensile strength of PP/WPT/KNF composites decreased with increased KNF loadings. This may be due to the incompatibility between the hydrophobic PP/WPT matrices and the hydrophilic KNF that resulted in weak interfacial adhesion. At similar KNF loadings, higher tensile strength was observed for composites with APTES coupling agent than without. APTES promotes better interfacial adhesion between hydrophobic PP/WPT and hydrophilic KNF. As a result, better stress transfer through KNF was achieved, and the tensile strength of PP/WPT/KNF composites was improved.

Table 2. Tensile Properties of PP/WPT/KNF Composites with and without 3-Aminopropyl-triethoxysilane (APTES)

KNF Loading (phr)	Tensile Strength (MPa)	Tensile Modulus (MPa)	Elongation at Break (%)
0	14.1	606.5	12.1
10	11.7	622.1	8.6
10 + APTES	12.5	665.7	7.4
20	9.9	718.8	5.6
20 + APTES	10.9	759.4	4.5

The interfacial interaction between PP/WPT matrices and KNF occurs in two steps. The first step involves the reaction of hydroxyl (–OH) groups on KNF with the ethoxy (CH_3CH_2O-) groups of APTES, which is shown in Fig. 1. A complete reaction can possibly form 3 linkages for each molecule of APTES at the KNF surface, thus forming an intermediate.

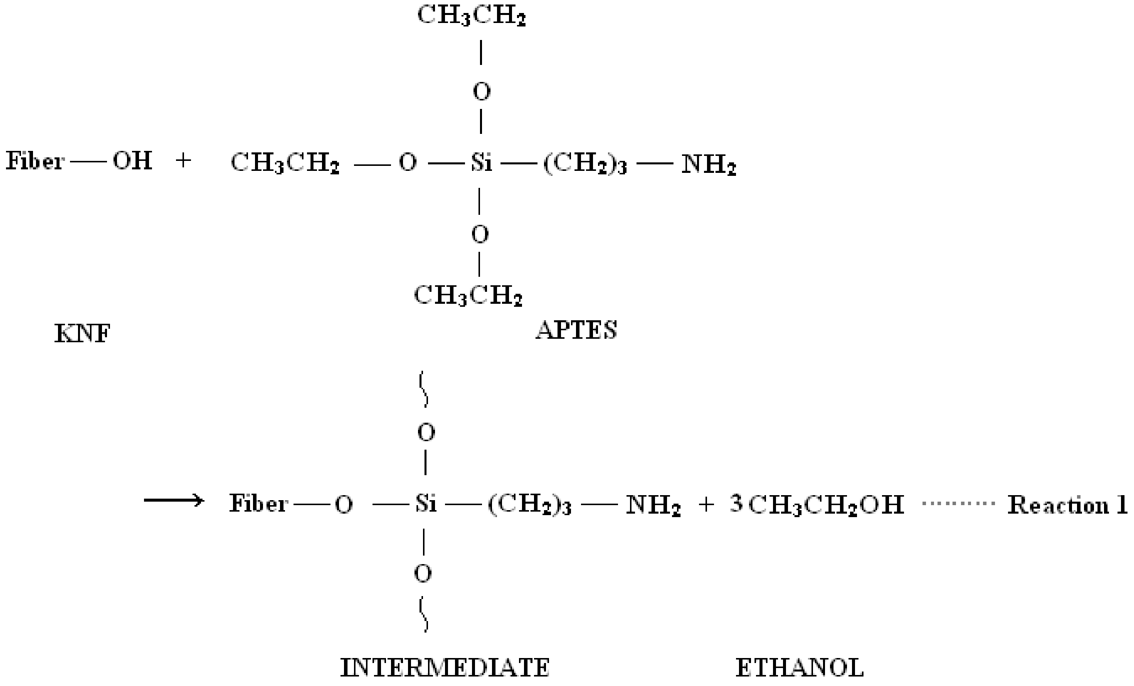

Fig. 1. Proposed possible interactions scheme between KNF and 3-aminopropyltriethoxysilane (APTES) coupling agent

The second step of reaction occurs between the intermediate molecules with PP/WPT matrices. The amino (-NH_2) hydrophobic group of the intermediate is attracted to the hydrophobic PP/WPT matrices due to their similar hydrophobicity, which contributes to the enhancement of KNF-PP/WPT interaction in the presence of the APTES coupling agent.

The tensile modulus was increased with an increase in KNF loading (Table 2) due to the nature of KNF, which added stiffness and rigidity to the composites. This became more obvious with higher KNF loadings. The chain mobility of PP/WPT matrices was lowered with the addition of KNF, resulting in a higher tensile modulus. At similar KNF loadings, composites with the APTES coupling agent exhibited a higher tensile modulus compared to those without. Again, the presence of the APTES coupling agent that resulted in a higher tensile modulus was due to better interfacial interaction between PP/WPT matrices and KNF.

The elongation at break of the composites was found to decrease with increased KNF loading. This is because the restriction of the chain mobility of PP/WPT matrices increased with KNF loading, thus leading to a higher breaking tendency (lower deformation) of the composites. At similar KNF loadings, higher elongation at break was observed for composites with the APTES coupling agent compared to those without. Again, as explained earlier, the presence of the APTES coupling agent resulted in the enhancement of interfacial adhesion of the PP/WPT matrices and KNF, leading to the elongation at break of the composites.

Water Uptake

Figure 2 shows the water uptake of PP/WPT/KNF composites with and without the APTES coupling agent for different KNF loadings. It can be seen that the water uptake by all the composites increased with increasing immersion time and KNF fibre loadings. Similar findings have been reported by other researchers (Santiagoo *et al.* 2011; Ismail *et al.* 2010; Demir *et al.* 2006). All composites showed a similar trend of water uptake; in each case initially high water uptake by the samples was observed, followed by a gradual increase until an equilibrium stage was achieved at the end of 30 days.

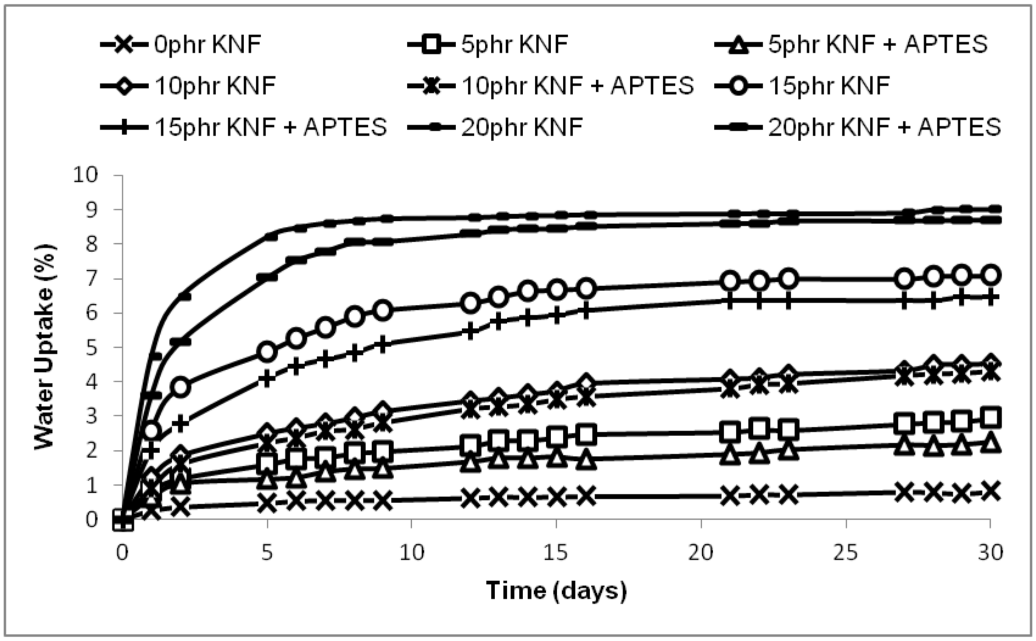

Fig. 2. Water uptake of PP/WPT/KNF (70/30) composites with and without 3-aminopropyltriethoxysilane (APTES)

Natural fibres are hydrophilic in nature due to the hydroxyl groups (OH) found in cellulose, which are able to form hydrogen bonds between water and the KNF fibres (Ismail *et al.* 2010). Besides the hydrogen bonding sites in natural fibres, the presence of voids, lumens, pores, and micro-cracks in the matrix, along with gaps and flaws at the interfaces, may result in water uptake by natural fibre polymer composites (Stokke and Gardner 2003; Zabihzadeh 2010; Santiagoo *et al.* 2011). According to Fig. 2, the results indicate that the percentage of water uptake by the composites increased with increased KNF loadings. This is because as the KNF loadings increased, the number of hydrogen bonds formed between KNF fibres and water molecules also increased along with the number of OH groups from the cellulose. In addition to that, greater water uptake by samples with higher KNF loading was due to the increasing amount of voids between KNF fibres and PP/WPT matrices.

At similar filler loadings, the composites with the APTES coupling agent demonstrated a lower water uptake percentage compared to the composites without. Santiagoo *et al.* (2011) and Demir *et al.* (2006) reported similar findings. This result clearly indicates that the presence of the APTES coupling agent enhances the filler-matrix interactions at the interface. APTES also consumed some –OH during composite manufacturing, thus decreasing hydrophilicity and the amount of equilibrium water uptake by the composites.

Thermal Properties

The thermal analysis results of the KNF and PP/WPT/KNF composites with and without the APTES coupling agent at 10 phr and 20 phr KNF loadings, are shown in Fig. 3 and Table 3. The TGA curve for KNF exhibits two weight loss steps.

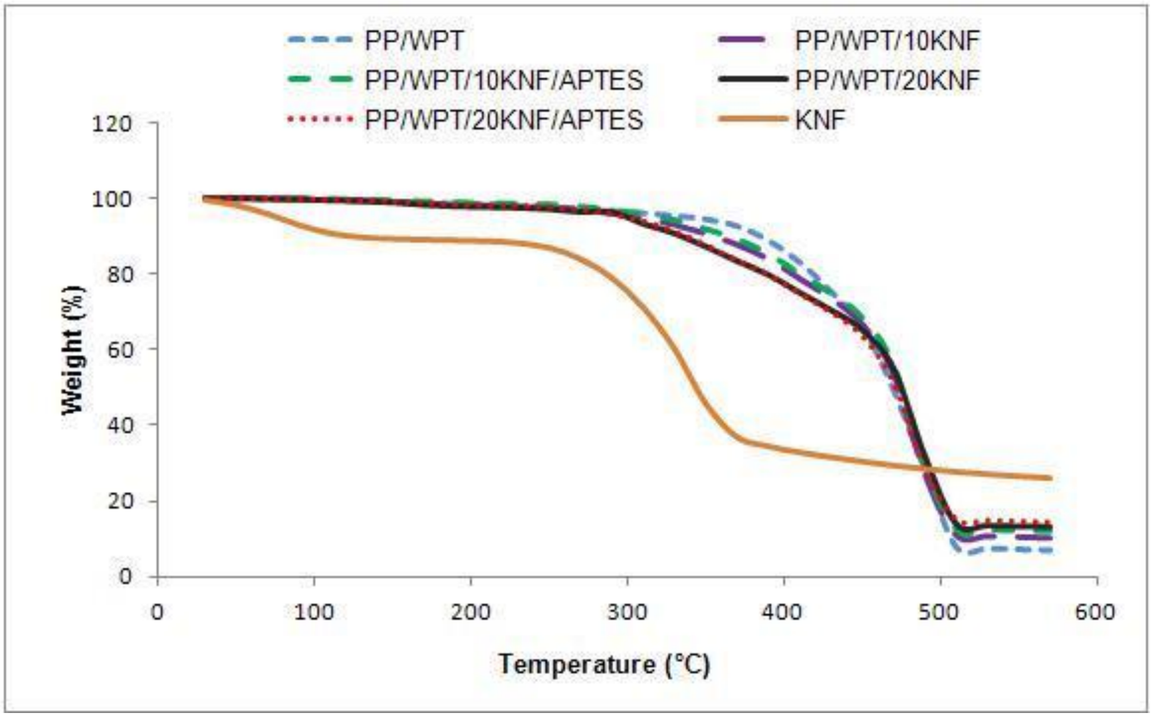

Fig. 3. TGA curves of KNF and PP/WPT/KNF composites with and without 3-aminopropyltriethoxysilane (APTES)

The weight loss was measured as a function of temperature, and the initial weight loss of KNF began below 100°C due to the gradual evaporation of absorbed moisture. The next stage of weight loss occurred approximately between 200°C and 400°C due to the thermal decomposition of the three major constituents (hemicelluloses, cellulose, and lignin) of KNF. Generally, the thermal decomposition of cellulosic materials takes place between the temperature range of 200°C and 400°C (Fisher *et al.* 2002). Based on Table 3, the char residue for KNF was found to be higher compared to the KNF-filled composites, and this was attributed by the presence of lignin as well as ash, which have very slow thermal decomposition properties (Yang *et al.* 2007; Ashori *et al.* 2006).

Table 3. Thermal Stability Parameters of KNF and PP/WPT/KNF Composites with and without 3-Aminopropyltriethoxysilane (APTES)

PP/WPT (phr)	KNF loading (phr)	Char residue (%)	Temperature at 50 % weight loss (°C)
-	KNF	26.11	344
70/30	0	6.91	468
70/30	10	10.14	473
70/30	10 + APTES	13.38	474
70/30	20	12.29	475
70/30	20 + APTES	14.50	476

The results in Fig. 3 and Table 3 also indicate that the temperature at 50% and char residue of composites increased along with increased KNF loading. Composites with the 20 phr KNF loading exhibited highest temperature at 50% weight loss and char residue, *i.e.* 475°C and 14.50%, respectively. The formation of char residue provides a barrier between the heat source and polymeric material, hence slowing down or inhibiting the out-diffusion of volatile decomposition products from the composites (Ismail *et al.* 2008; Beyler and Hirschler 2001). Increased KNF loading resulted in increased char residue; therefore a thicker barrier was formed between the heat source and polymeric material. This had the effect of increasing the thermal stability of PP/WPT/KNF composites, such that a higher temperature was then required for decomposition.

At similar KNF loadings, composites with APTES exhibited slightly higher temperature at 50% weight loss and char residue compared to the composites without APTES. These results indicate that the thermal stability of composites with APTES was increased. However the increment was insignificant, implying that APTES does not affect the thermal properties as much as it affects the tensile properties and water absorption of PP/WTD/KNF composites.

FTIR

Figure 4 shows the spectra of PP, PP/WPT blend, and KNF. From Fig. 4, PP shows its characteristic peaks at 1450 cm^{-1} which was assigned to the asymmetric stretching vibration of -C-H- in CH_3 and 1375 cm^{-1} which was assigned to the symmetric bending of CH_3 (Zainal and Ismail 2011 and Ansari and Ismail 2010). It can be seen that the spectra for both PP and PP/WPT were almost identical, with no significant change in intensity. This indicates that the possible reaction between PP and WPT was not a chemical reaction, but instead, could possibly be due to the formation of covalent bonds between two hydrophobic structures. Zainal and Ismail (2011) reported that the C-H bond type exists abundantly in WPT and PP. The characteristic peaks at 3349 cm^{-1} was due to the –OH group, and the 1750-1720 cm^{-1} region might be due to a nitrile type substituent in the aliphatic chain (Jose *et al.* 2007), or a carbonyl compound (possibly ester or ketone) of WPT (Jose *et al.* 2007; Salmah *et al.* 2006) which exists in the PP/WPT spectrum.

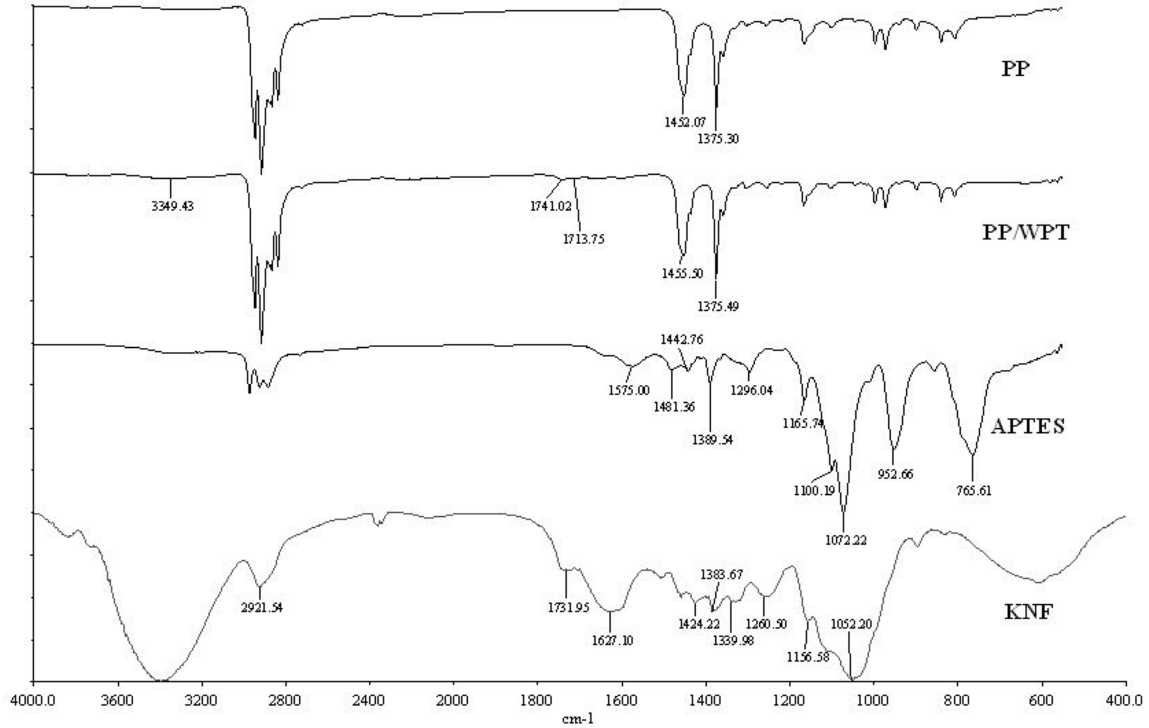

Fig. 4. FTIR spectra of PP, PP/WPT (70/30), APTES, and KNF

APTES shows its characteristic peaks at 1574 to 1576 cm^{-1}, which can be assigned to the deformation vibration of the N-H bond, 1296 and 765 cm^{-1} which were assigned to the Si-C vibration, and 1165, 1072, and 952 cm^{-1} which were assigned to the Si-O-C bond. The peak at 1100 cm^{-1} is assigned to the stretching vibration of C-NH_2 bond (Demjen *et al.* 1999; Mathialagan and Ismail 2012).

Characteristic peaks of KNF which appeared in the range of the 3400-3200 cm^{-1} region were attributed to the stretching of O-H groups, 2900-2800 cm^{-1} region were due to stretching of C-H (Khalil et al. 2001), 1731-1729 cm^{-1} region were due to C=O stretching in carbonyl (Abdrahman and Zainudin 2011) or C=O stretching of the acetyl group in hemicellulose or ester linkage of the carboxylic group (Alemdar and Sain 2008; Janoobi *et al.* 2009), 1650-1620 cm^{-1} region were due to absorbed water in cellulose (Troedec et al. 2008; Janoobi *et al.* 2009), 1425-1432 cm^{-1} region were due to CH_2 symmetric bending (Abdrahman and Zainudin 2011; Sgriccia et al. 2008), 1380-1320 cm^{-1} region were due to the bending vibration of C-H and C-O groups of aromatic rings in polysaccharides (Troedec *et al.* 2008; Janoobi *et al.* 2009), 1242 cm^{-1} was due to C-O phenolic or C-O stretching of the aryl group in lignin (Troedec et al. 2008; Janoobi *et al.* 2009; Abdrahman and Zainudin 2011), 1160 cm^{-1} was due to the anti-symmetrical deformation of the C-O-C bond (Silva et al. 2008; Troedec et al. 2008), and 1010-1070 cm^{-1} region were due to C-O alcohol (primary and secondary), O-H or aliphatic ethers (Nacos et al. 2006; Abdrahman and Zainudin 2011).

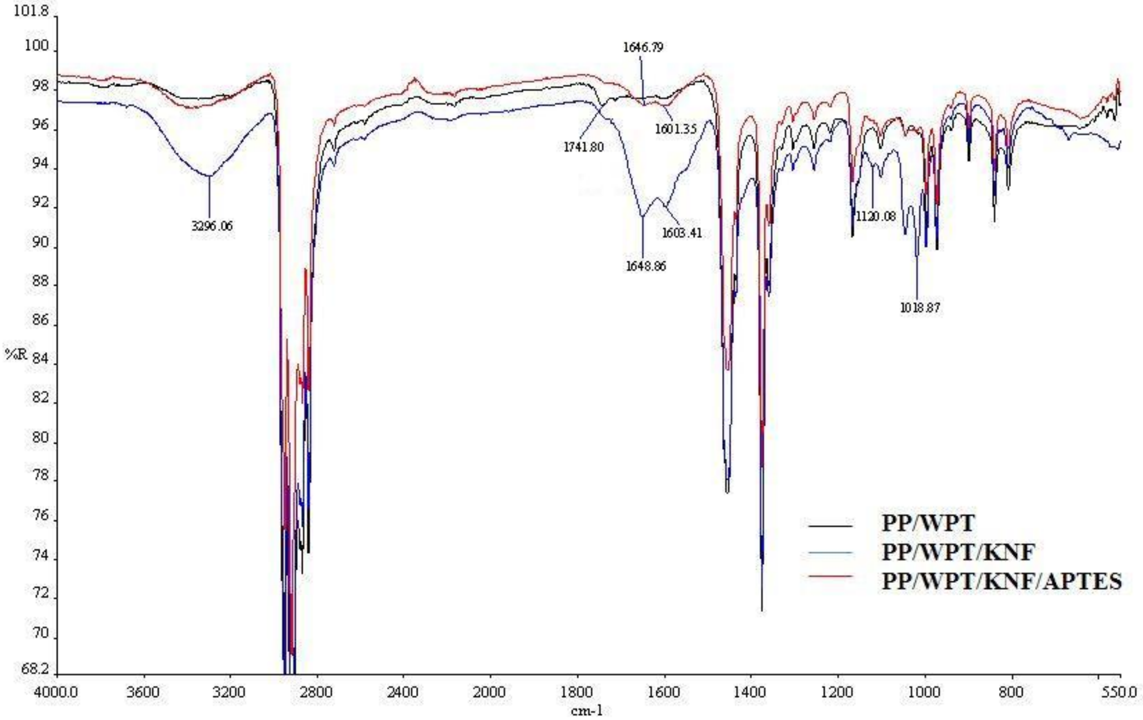

Fig. 5. FTIR spectra of PP/WPT (70/30), PP/WPT/KNF (70/30/10), and PP/WPT/KNF/APTES (70/30/10/1.5) composites

Figure 5 shows a comparison of FTIR spectra of PP/WPT, PP/WPT/KNF, and PP/WPT/KNF composites. The characteristic peaks at the region 1741 cm^{-1} in the PP/WPT spectra might be due to the presence of a nitrile type substituent in the aliphatic chain (Jose *et al.* 2007) or a carbonyl compound (possibly ester or ketone) in the WPT (Jose *et al.* 2007; Salmah *et al.* 2006). In the PP/WPT/KNF spectrum, the characteristic peaks of KNF were observed at 3294 cm^{-1} and 1648-1603 cm^{-1}, which were attributed to structural water and absorbed water. Besides, the peaks at 1120 cm^{-1} and 1018 cm^{-1} in PP/WPT/KNF composites are also characteristic peaks of KNF.

However, by comparing PP/WPT/KNF and PP/WPT/KNF/APTES composites, a decrease in the intensity of the absorption peaks at 3296 cm^{-1} and 1648-1603 cm^{-1} was observed in composites with APTES. The reduction of O-H stretching and absorbed water in the presence of APTES confirmed the interaction between O-H groups of KNF and $-SiOCH_2CH_3$ groups of APTES as shown in Fig. 1. Moreover, the characteristic peak of APTES at 1575 cm^{-1} from the deformation vibration of N-H bond disappeared in PP/WPT/KNF/APTES composites, indicating the reaction between the APTES and PP/WPT network. This observation corroborates the fact that the presence of an APTES coupling agent increases the tensile properties and lowers the water uptake in composites.

CONCLUSIONS

1. Tensile strength and elongation at break were decreased, and tensile modulus increased with kenaf (KNF) loading. Polypropylene/waste pulverized tire/kenaf (PP/WPT/KNF) composites with the addition of coupling agent APTES showed higher tensile strength and tensile modulus, but lower elongation at break.

2. Water uptake properties of the PP/WPT/KNF composites increased with an increase in KNF loading. Composites with the APTES coupling agent reduced the water uptake of the composites by improving interfacial compatibility so that with pores blocked and fewer voids, less –OH groups are exposed to water molecules, thus reducing the water uptake properties of PP/WPT/KNF composites.
3. Thermal stability of PP/WPT/KNF composites was found to increase with increased KNF loading. Thermal stability of PP/WPT/KNF composites with APTES was increased, but the increment was insignificant.
4. From this research, APTES was found to have an effect on the tensile properties and water uptake of PP/WPT/KNF composites. However, APTES does not have a significant effect on the thermal stability of PP/WPT/KNF composites.

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