Surface Modification of *Elateriospermum tapos* Seed Shell Recycled Polypropylene Composites

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The influence of the filler content and surface modification of *Elateriospermum tapos* seed shell (ETSS)-filled recycled polypropylene (rPP) on the tensile, thermal, and morphological properties was investigated. Maleic acid (MA) was used for the chemical modification of ETSS. It was found that increasing the ETSS content decreased the tensile strength and elongation at break of composites. However, the modulus of elasticity increased with the addition of ETSS. The thermal properties of composites were examined using thermal analysis (TGA) and differential scanning calorimetry (DSC). The addition of ETSS indicated better thermal stability of rPP/ETSS composites. The degree of crystallinity (X_c) of the composites decreased with increasing ETSS content. The tensile strength and modulus of elasticity of modified composites was higher than unmodified composites. Surface modification with maleic acid increased the thermal stability and crystallinity of the modified rPP/ETSS composites. Scanning electron microscopy showed that the filler-matrix interaction improved with the modification of ETSS with maleic acid.

Keywords: Recycled polypropylene; Elateriospermum tapos seed shell; Maleic acid; Surface modification; Composites

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INTRODUCTION

Currently, the rapid development of polymer usage has led to the dramatic rise of what is commonly referred to as the "green movement." As part of the green movement, people are conscious about environmental pollution due to overconsumption and the disposal of plastic waste in landfills. The green movement shows how environmental awareness has increased among individuals, which has forced the industry to be much more concerned about waste disposal management and the development of plastic-based materials. The conventional method was largely concentrated on landfill disposal. Thus, the industry has moved to the much more acceptable option of recycling (Aurrekoetxea *et al.* 2001). This movement has also become a very large influence for scientists to research how to recycle plastic and make biodegradable products, such as biodegradable composites (Liu *et al.* 2005; Saxena *et al.* 2008; Kalia *et al.* 2009).

Traditionally, polymer composites were prepared using synthetic fillers such as glass, carbon, and other synthetic fibers. However, these are considered harmful because they are not biodegradable and can cause harm to the environment. Lately, studies using

organic fillers or cellulose-based fillers in the production of polymer composites have been very popular (Ismail *et al.* 2001; Al-Maadeed *et al*. 2014; Etaati *et al.* 2014; Salmah and Ismail 2008). The usage of organic or cellulosic fillers also has some advantages when compared with synthetic fillers, such as low cost, low density, acceptable specific strength, easy extraction, renewable sources, carbon dioxide sequestration, and better bio-degradability when compared with synthetic reinforcement or inorganic fillers (Mohanty *et al.* 2000; Mohanty *et al.* 2001; Mishra *et al.* 2003; Chun *et al.* 2013a; Chan *et al*. 2013; Koay *et al.* 2013; Mohamad Rasidi *et al.* 2014).

Elasteriospermum tapos is one type of lignocellulosic filler that is abundant in Malaysia. This plant is one of the Malaysian canopy tree species in the tropical rainforest (Osada *et al.* 2003; Mohd Hasmadi *et al*. 2008). It has been reported to be found in Peninsular Malaysia (Mohd Hasmadi *et al.* 2008), Brunei (Chumkiew *et al.* 2007), Peninsular Thailand (Ngamriabsakul and Kommen 2011), and Java (Sosef *et al.* 1998). The tree is approximately 24 to 43 m high, with red young leaves. The perah fruit are approximately 5.08 to 6.35 cm long, oblong, with a buff colour. They hang on a single stalk, and the seeds are about 4.45 cm long in a shiny brown shell (Yong and Salimon 2006), like rubber seed with an oval shape.

Using recycled products can reduce the solid waste disposal problem and the use of virgin plastics in many applications. Furthermore, studies found that recycled polypropylene (PP) provides performance similar to that of virgin PP, while being much less expensive (Azadeh *et al.* 2011). Recently, studies on the use of recycled polypropylene as a matrix have become popular (Chun *et al.* 2013a; Mohamad Rasidi *et al.* 2014; Mohamad *et al.* 2014).

Research on composites with different types of lignocellulosic fillers has been reported (Salmah *et al.* 2013). However, organic and cellulosic fillers both exhibit high moisture absorption, which can cause major issues in most applications. This is due to the high hydrophilicity of the materials and compatibility issues between hydrophilic fibres and a hydrophobic matrix such as polypropylene. In the past, many attempts have been made to modify the surface to improve the properties of cellulose fibres to enhance adhesion with the matrix. Various methods, such as corona treatment, plasma treatment, mercerization, heat treatment, graft copolymerisation, silane treatment, and treatments with other chemicals have been reported to positively affect the compatibility of natural fibre composites (Hernández-Sánchez *et al.* 2001; Zafeiropoulosa *et al.* 2002; Yong and Salimon 2006; Ares *et al.* 2010; Salmah *et al.* 2011). Extensive research has been carried out on interface modification in organic filler or natural fibre reinforced composites using coupling agents.

In this research, the effectiveness of *Elateriospermum tapos* seed shell as a filler in recycled polypropylene was studied. The effect of maleic acid (MA) as a surface modifier to improve the tensile, thermal, and morphological properties of *Elateriospermum tapos* seed shell (ETSS)-filled recycled polypropylene (rPP) composites was investigated.

EXPERIMENTAL

Materials

Recycled polypropylene (rPP) was supplied by SLT Plastic Sdn. Bhd (Penang, Malaysia). The density of rPP was 0.9 g/cm^3 and the melt flow index (MFI) value was 4.03 g/10 min at 230 °C. The *Elateriospermum tapos* seed shells (ETSS) were collected from BatuMalim Village (Pahang, Malaysia). Initially, the ETSS were ground into a smaller size using a grinder and then dried at 70 °C for 24 h. The average particle size of ETSS was 16 µm, as determined by a particle size analyzer (Malvern Mastersizer model 2000, UK). Maleic acid (MA) and ethanol were supplied by HmBG Chemicals (Penang).

Methods

Filler modification

Maleic acid was dissolved in ethanol at a concentration of 3% (v/v). The ETSS was added to the solution and stirred for 2 h. The filler and solvent ratio was 1:10 (w/v). Then, the ETSS was filtered using filter paper and dried in an oven at 70 °C for 24 h.

Preparation of Composites

The composites were made by mixing rPP and ETSS with a Brabender Plasticoder EC-PLUS (Germany). For the mixing conditions, the temperature was set at 180 °C and the rotor speed at 50 rpm. The rPP melted homogeneously while the torque remained constant for 4 min. Then, the ETSS filler was added and mixed for 5 min. The rPP/ETSS composites were shaped into 1 mm thick using the compression moulding machine GT 7014A (Gotech, Taiwan). The compression process involved: 4 min pre-heating, 2 min compression under 100 kgf/cm^2, and a 2 min cooling period. Then, the composites were cut into dumbbell shapes according to the ASTM D638 (2007).A similar preparation process was followed for the modified rPP/ETSS composites. The formulations of the composites are shown in Table 1.

Table 1. Formulation of Unmodified and Modified rPP/ETSS Composites

Material	Unmodified	Modified with MA
rPP (php)	100	100
ETSS (php)	0, 10, 20, 30, 40	10, 20, 30, 40
Maleic Acid, MA (%)*	0	3

Php= parts per hundred polymer
*3% by weight of filler

Tensile Properties

The tensile properties of the composites were tested by an Instron 5569 universal testing machine (USA). The tensile test was performed according to ASTM D638 (2007). The crosshead speed was 20 mm/min at room temperature. The values of tensile strength, elongation at break, and modulus of elasticity were taken as the average values of 5 samples.

Thermogravimetry Analysis (TGA)

The thermal properties of rPP/ETSS composites were investigated using a TA Instrument Q500 (USA). The temperature was scanned from 30 to 600 °C, with a heating rate of 20 °C/min. The samples were tested with a nitrogen air flow of 50 mL/min.

Differential Scanning Calorimetry (DSC)

The DSC analysis was carried out using the DSC Q 10, Research Instrument. The samples were scanned from 25 to 250 °C at a heating rate 20 °C/min, using a nitrogen air flow of 50 mL/min. The melting temperature (T_m) and the enthalpy of composites were automatically calculated by the instrument software. The crystallinity of the composites (X_c) was determined using Eq. (1):

$$X_c = (\Delta H_f / \Delta H_f^o) \times 100\% \quad (1)$$

Where ΔH_f is the heat of fusion for composites, and ΔH_f^o is the heat of fusion for 100% PP, which is 209 J/g (Chun *et al*. 2013).

Morphology Analysis

The tensile fracture surface of the composites was analyzed using a JSM-6460 scanning electron microscope (JEOL; Tokyo, Japan) operating at an accelerating voltage of 10 kV. The fracture surface was coated with a thin layer of palladium using a JFC-1600 AutoFine Coater (JEOL).

Fourier Transform Infrared Analysis (FTIR)

The FTIR analysis of unmodified and modified ETSS was carried out by using Perkin-Elmer Spectrometre 2000 FTIR spectrometer. The ETSS was scanned in range between 400 to 4000 cm^{-1}. Sample preparation was followed the ATR method. The spectra of samples were recorded with 16 scans.

RESULTS AND DISCUSSION

Tensile Properties

The effects of the ETSS content on the tensile strength of the unmodified and maleic acid modified rPP/ETSS composites are shown in Fig. 1. The tensile strength of the composites decreased as the filler content increased. The decrement of tensile strength was due to the weak interfacial adhesion between the filler and matrix (Ghani *et al*. 2014). However, after the ETSS was chemically modified using maleic acid, the tensile strength of composites increased. The modified composites had higher tensile strength than the unmodified composites. The improvement of the filler-matrix interaction was due to the reaction between maleic acid and the hydroxyl groups of ETSS (Chun *et al*. 2013b). Among all of the composites, the modified rPP/ETSS with 10 php ETSS showed the highest tensile strength. The increment of the tensile strength at 10 php of ETSS was 12% compared to that of the unmodified rPP/ETSS composites. Furthermore, the tensile strength of modified composites at a 10 php filler loading was

1.72% higher than that of the neat rPP, and the average value of tensile strength increased with MA surface modification by approximately 10.3%.

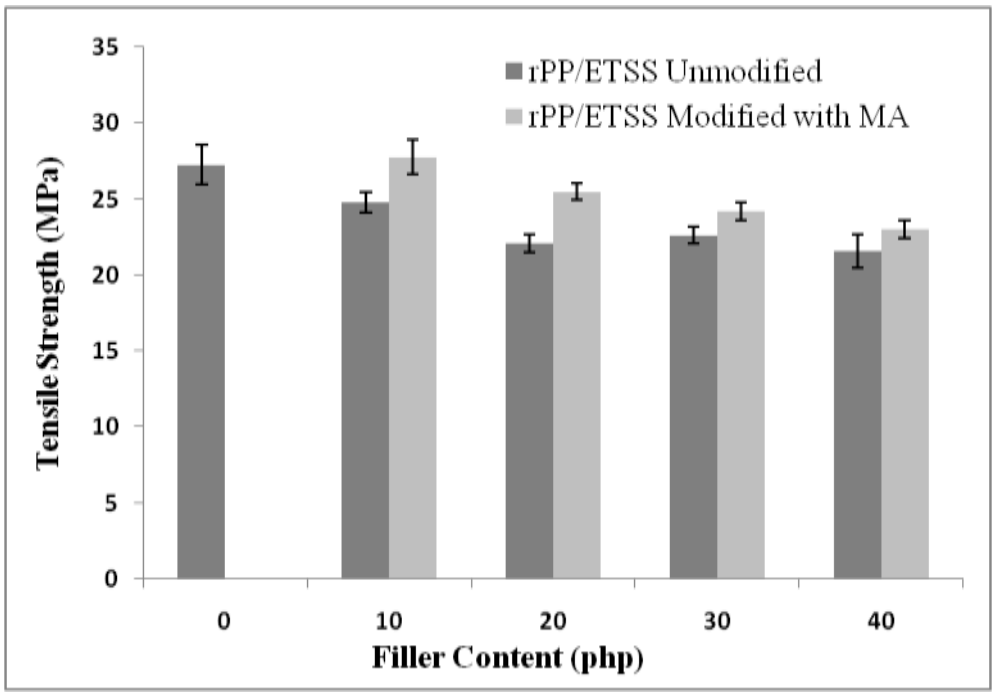

Fig. 1. Effect of ETSS content on tensile strength of unmodified and modified rPP/ETSS composites. Data provided as the average ± standard deviation

Figure 2 shows the effect of ETSS content on elongation at break of the unmodified and modified rPP/ETSS composites. The elongation at break of the unmodified composites decreased with increasing ETSS content. The plasticity of the polymer reduced as the chain mobility was restricted by the addition of the ETSS filler (Mohanty *et al.* 2000). The elongation at break of both composites decreased dramatically. Meanwhile, at a similar ETSS content, the modified composites had lower elongation at break compared to unmodified composites. This is due to the improvement of the interfacial interaction between the matrix and filler after the modification, which leads to reduced elongation of composites (Salmah and Azieyanti 2010).

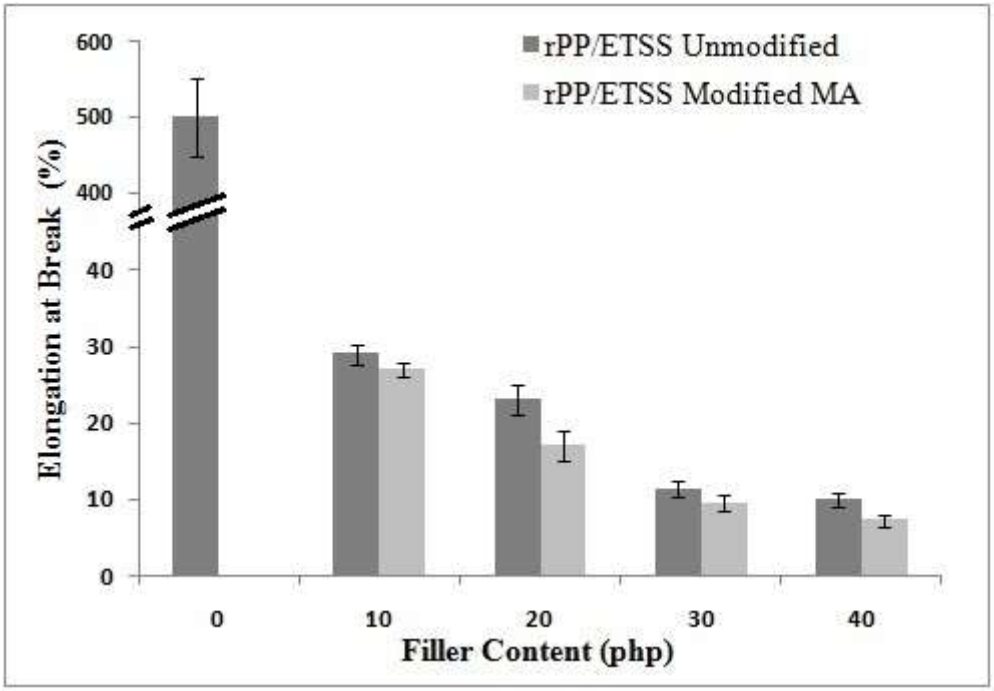

Fig. 2. Effect of filler content on the elongation at break of unmodified and modified rPP/ETSS composites. Data provided as the average ± standard deviation

The effect of ETSS on the modulus of elasticity of the unmodified and modified rPP/ETSS composites is illustrated in Fig. 3. The modulus of elasticity of the unmodified composites increased with increasing ETSS content. The presence of the organic filler restricted the mobility of the rPP chain when load was applied to the composites (Salmah and Faisal 2010). This chain mobility restriction caused the increment of the modulus of elasticity with ETSS addition. The figure also shows that the modified composites have a higher value of modulus of elasticity compared to the unmodified composites. The increment of the modulus of elasticity of modified composites averaged 25.85% than unmodified composites. These increasing values indicate that a better filler-matrix interaction and wettability of composites were achieved after MA surface modification (Chun *et al*. 2013a).

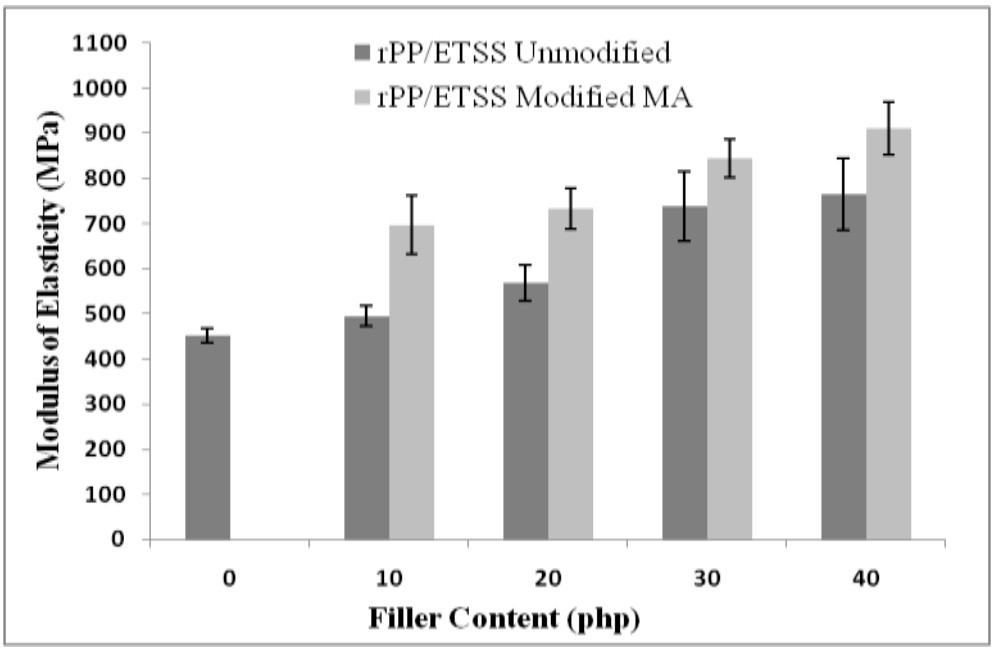

Fig. 3. Effect of filler content on the modulus of elasticity of unmodified and modified rPP/ETSS composites. Data provided as the average ± standard deviation

Fourier Transform Infrared Spectroscopy

The FTIR spectra of the unmodified and modified rPP/ETSS composites are shown in Fig. 4, and the primary peaks that are characteristic of ETSS are presented in Table 2.

Table 2. Functional Groups of Lignocellulosic *Elateriospermum tapos* Seed Shell

Wavenumber (cm^{-1})	Functional Group
3320	hydrogen bond from OH groups of ETSS
2924 and 2855	CH_2 and CH_3 structures of long methyl group in ETSS lignocelluloses filler
1514	benzene ring in lignin
1424	C-O stretching of hemicellulose
1636	C-O-C group of cellulose and hemicelluloses
1373	-OH in-plane bend
1711	C=O of carboxyl group of Lignin
899	Aromatic C-H group vibration in lignin and hemicelluloses.
1240	C-O group from acetyl group in lignin
1030	C-O component in lignin and hemicelluloses
899	C-H deformation

The FTIR spectrum of the modified composites showed a change of peaks from 1636 cm^{-1} to 1625 cm^{-1}. The change of peaks from 1714 cm^{-1} to 1711 cm^{-1} appeared after modification, due to the absorption band of the C=O group from ETSS overlapping with the C=O from ester bonding between ETSS and MA. This can also be observed in the reduction of OH groups from 3320 cm^{-1} to 3317 cm^{-1} due to the reaction between the OH

groups from ETSS and MA to form esterification. A schematic of the reaction between ETSS and maleic acid is shown in Fig. 5.

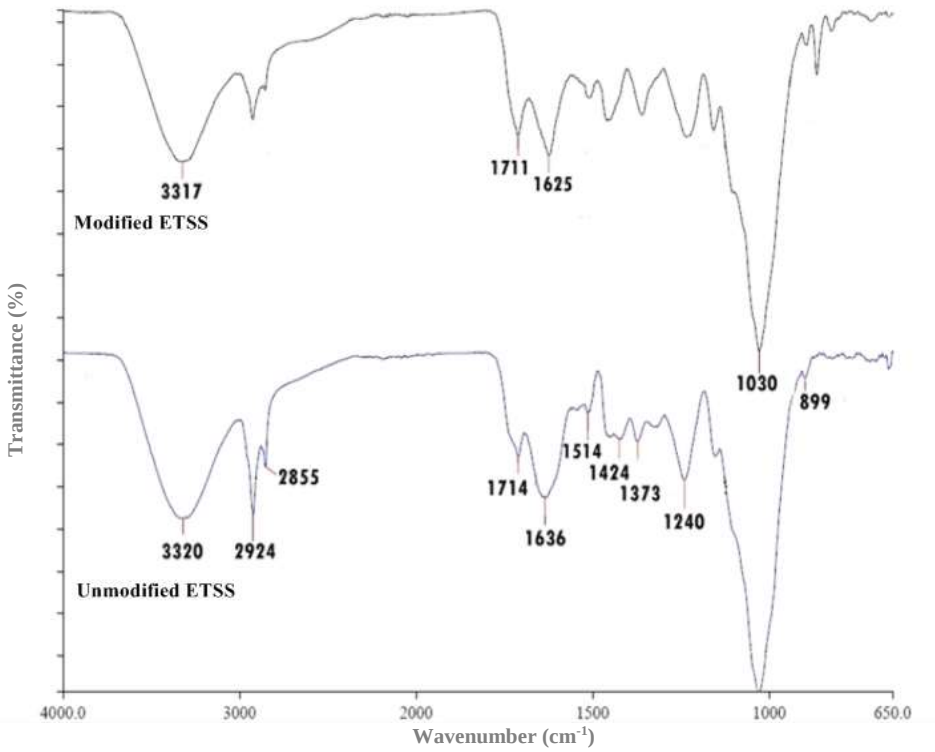

Fig. 4. FTIR spectra of unmodified and modified rPP/ETSS composites

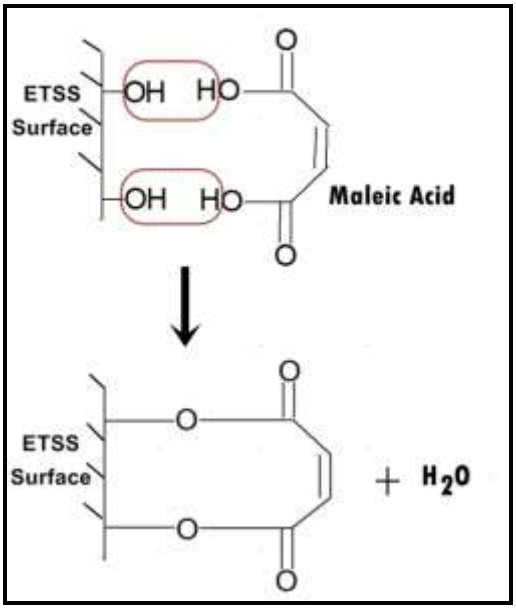

Fig. 5. Schematic of reaction between ETSS and maleic acid

Thermal Properties

The TGA curves of the unmodified and modified composites are shown in Fig. 6. The data obtained from Fig. 6 are summarized in Table 3. The temperature at the maximum rate of weight loss was labelled T_{dmax}. The weight loss of the unmodified composites at 300 °C increased as the ETSS increased compared to the neat rPP. At 600 °C, the weight loss of the unmodified composites decreased as the ETSS increased. The T_{dmax} of the composites increased with an increasing load of ETSS. This indicates that the improvement of the thermal stability occurred due to char formation during the pyrolysis of the ETSS. The char became like a protective barrier that prevented the thermal decomposition of the rPP matrix. At a similar ETSS loading, the modified composites showed lower weight loss at 300 and 600 °C. Furthermore, the T_{dmax} of the modified composites was higher than that of the unmodified composites. This proves that the MA improved the filler-matrix interaction, which leads to a better wettability and the dispersion of the ETSS through the rPP matrix. This due to the better char formation, thus leading to a higher thermal resistance (Mohamad Rasidi *et al.* 2014).

Table 3. Weight Loss of Unmodified and Modified rPP/ETSS composites at Different Temperatures

composites		Weight loss (%)	
	T_{dmax}	300 °C	600 °C
rPP	441	3.86	100
rPP/ETSS : 100/20 (Unmodified)	429	14.24	99.76
rPP/ETSS : 100/40 (Unmodified)	441	15.04	98.69
rPP/ETSS : 100/20 (Modified with MA)	461	6.81	97.69
rPP/ETSS : 100/40 (Modified with MA)	470	11.93	93.65

T_{dmax} = temperature at the maximum rate of weight loss

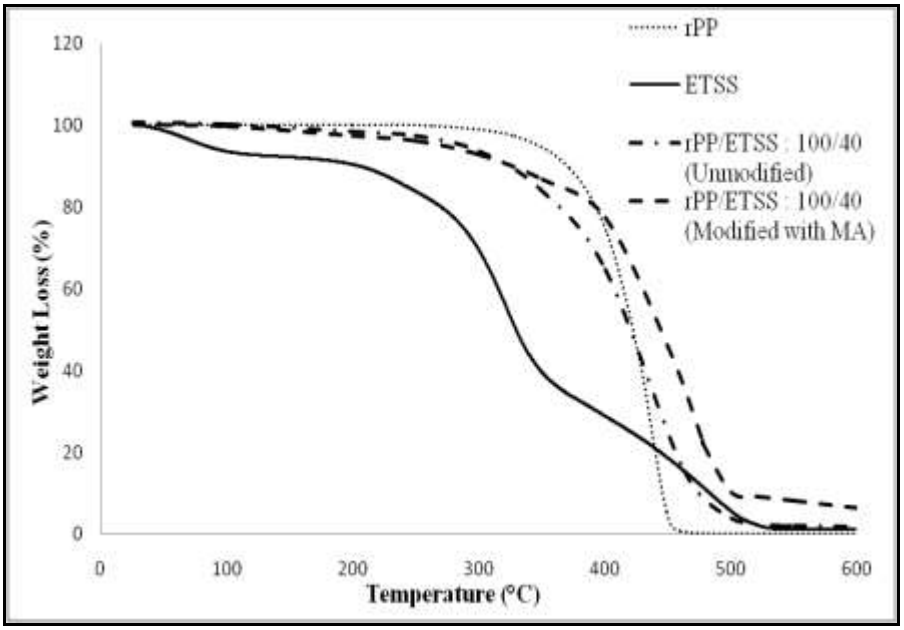

Fig. 6. TGA curve of unmodified and modified rPP/ETSS composites

Figure 7 illustrates the derivative thermogravimetric (DTG) curves of the unmodified and modified rPP/ETSS composites. The recycled polypropylene shows only one step of degradation, between 300 and 500 °C. The ETSS curves have three peaks of degradation. The first degradation occurred between 30 and 100 °C, due to the evaporation of water and moisture inside the ETSS. The second peak was identified between 180 and 350 °C and is indicative of the degradation of hemicelluloses and cellulose (Lv *et al.* 2010; Poletto *et al.* 2012. The third peak was identified at a temperature range of 400 to 500 °C and was due to the degradation of the lignin. From the DTG graph, it was also found that the unmodified and modified composites have two-step degradation processes. The peak in the temperature range 180 to 350 °C was due to the degradation of cellulose. The peak in the temperature range 300 to 500 °C was due to the degradation of rPP. It can be observed from the DTG curve that the $T_{d\ max}$ of modified composites shifted to the right. This shows that surface modification with MA enhanced the thermal stability of modified composites.

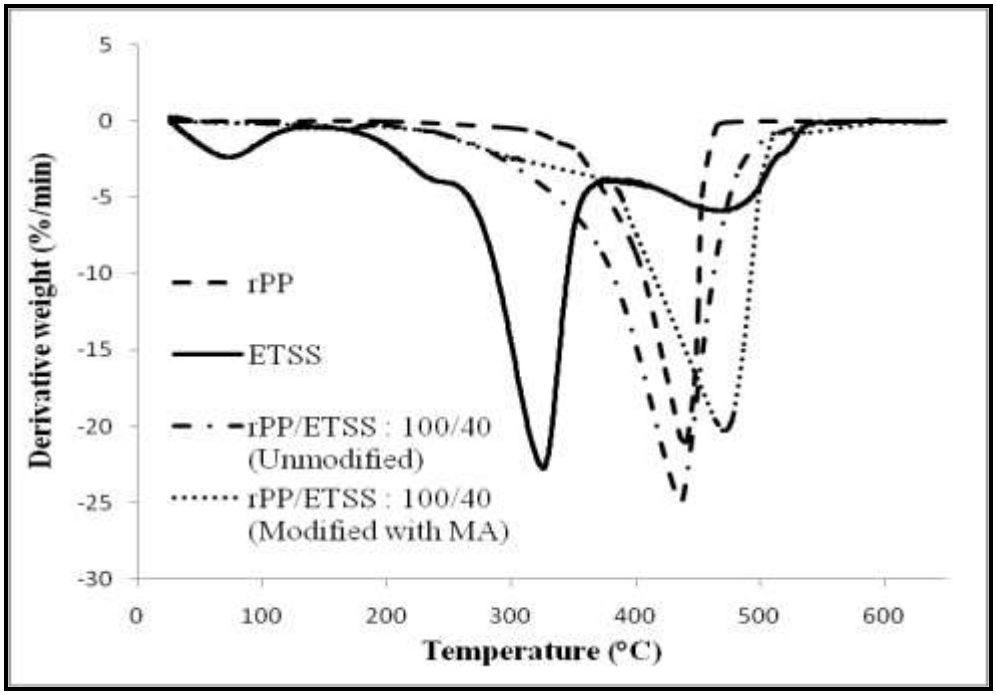

Fig. 7. DTG curves of unmodified and modified rPP/ETSS composites

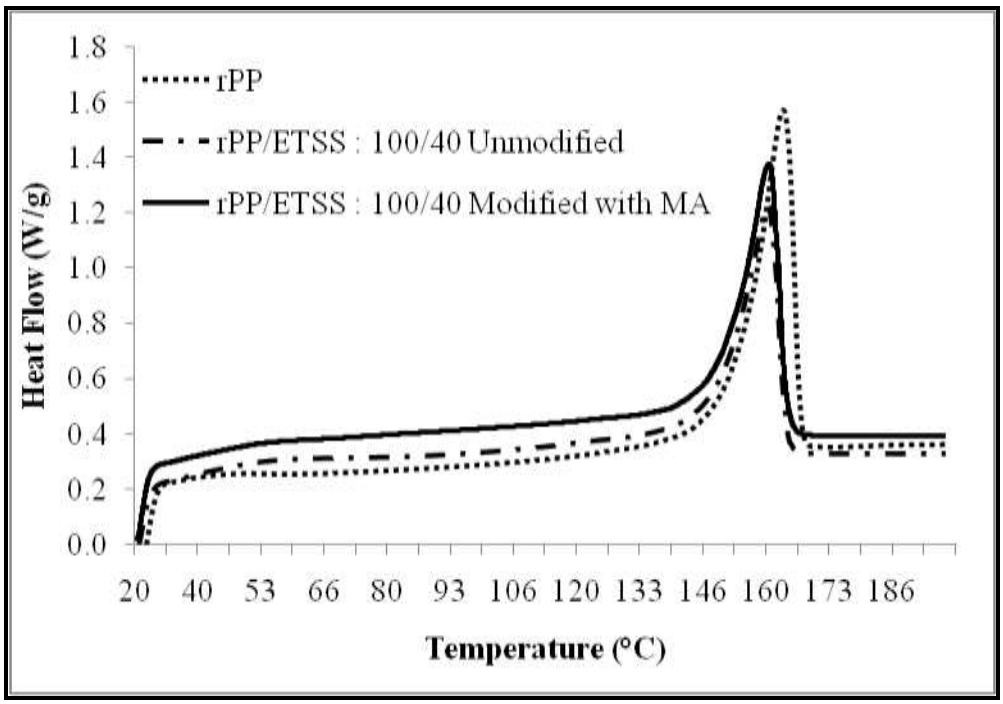

Fig. 8. DSC curve of unmodified and modified rPP/ETSS composites

The DSC curves of the unmodified and modified rPP/ETSS composites are illustrated in Fig. 8. Table 4 is summary of the melting temperature (T_m), enthalpy of fusion of composites (ΔH_f), and degree of crystallinity (X_c) of the composites. The enthalpy and crystallinity of the unmodified composites decreased with increasing ETSS content. This indicates that the ETSS does not act as a nucleating agent and has restricted the ability of the molecular chain of the composites to fold into a crystalline structure. Table 4 also shows that the modified composites have a higher enthalpy and crystallinity compared to the unmodified composites. This indicates that the addition of maleic acid led to an increase in the crystallinity of the composites. However, the addition of ETSS and MA did not slightly change upon the melting temperature of the composites.

Table 4. The T_m, ΔH_f, and X_c of Unmodified and Modified rPP/ETSS composites

composites	T_m (°C)	ΔH_f(J/g)	X_c (%)
rPP/ETSS: 100/0	163	79.55	38.4
rPP/ETSS: 100/20 (Unmodified)	163	69.50	33.6
rPP/ETSS: 100/20 (Modified with MA)	163	71.09	34.3
rPP/ETSS: 100/40 (Unmodified)	162	65.37	31.6
rPP/ETSS: 100/40 (Modified with MA)	163	70.09	33.9

T_m = melting temperature; ΔH_f = enthalpy of fusion; X_c = degree of crystallinity

Morphology

The morphology of the tensile fracture surfaces of unmodified and modified rPP/ETSS composites is shown in Figs. 9 to 12. Figures 9 and 10 show the morphology of the fracture surface of unmodified composites with 20 and 40 php ETSS. The ETSS withdrawal from the rPP matrix is observed. This occurred due to the weak interfacial interactions between the rPP and ETSS, which leads to poor filler wettability and dispersion. A similar result was also reported by Wang *et al.* (2014). Furthermore, some of the ETSS detachment from the rPP matrix was also found from the micrograph. This result was supported with the decreased tensile strength of the unmodified composite, which was discussed previously. As the amount of filler content increased, the filler-filler interactions increased, which tended to create filler agglomeration. The SEM micrographs of the modified composites, shown in Figs. 11 and 12, exhibit ETSS filler well dispersed in the matrix, which the present less pull out of ETSS. The better filler-matrix interaction between ETSS and rPP enhance after the surface modification with MA.

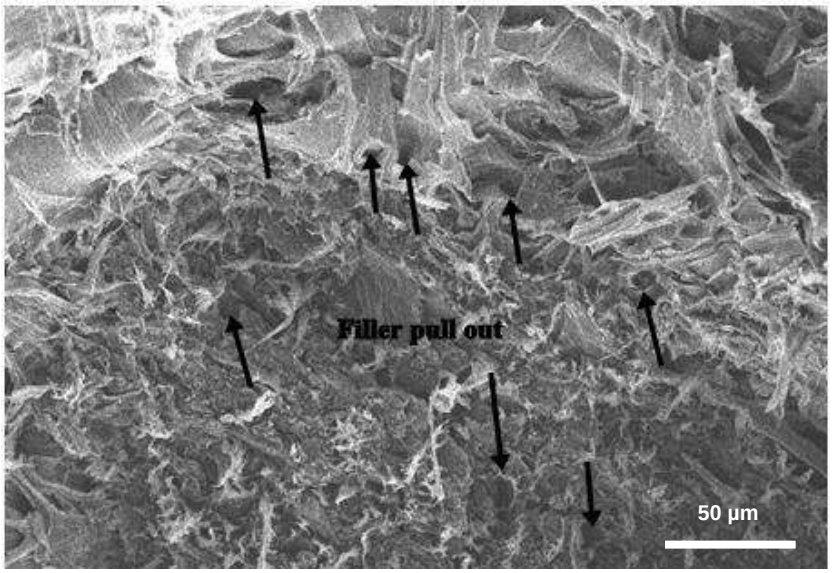

Fig. 9. SEM image of tensile fracture surface of unmodified rPP/ETSS composite with 20 php ETSS

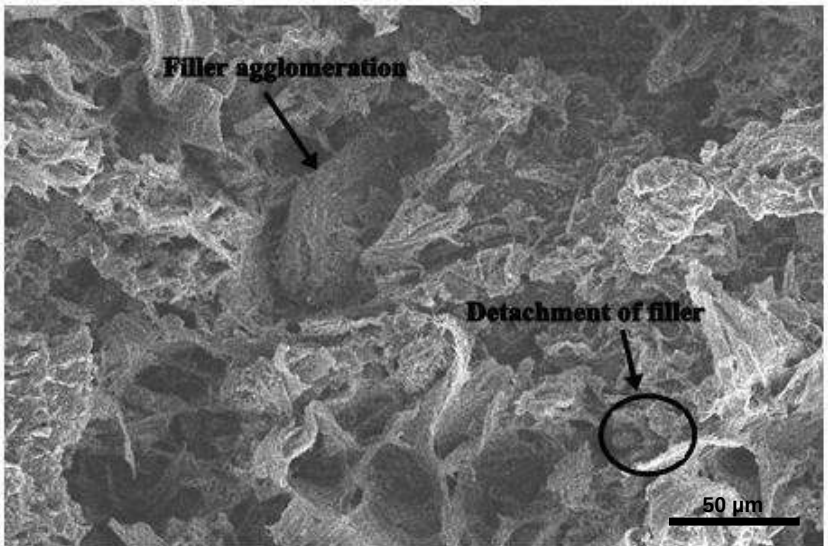

Fig. 10. SEM image of tensile fracture surface of unmodified rPP/ETSS composite with 40 php ETSS

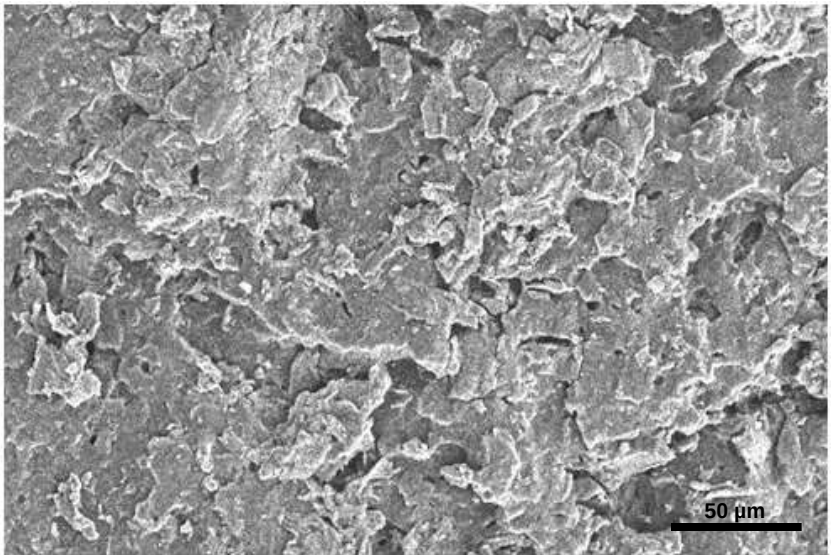

Fig. 11. SEM image of tensile fracture surface of modified rPP/ETSS composite with 20 php ETSS

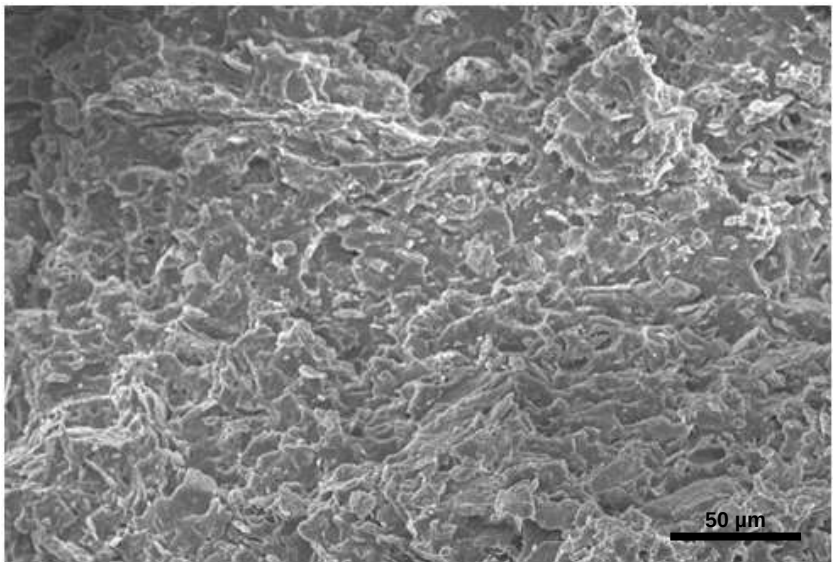

Fig. 12. SEM image of tensile fracture surface of modified rPP/ETSS composite with 40 php ETSS

CONCLUSIONS

1. The tensile strength and the elongation at break of the rPP/ETSS composites decreased with increasing ETSS content. However, the modulus of elasticity of the composites increased when the ETSS content increased.
2. The surface modification using MA improved the tensile properties of the modified composites. The modified composites had higher tensile strength and modulus of elasticity than the unmodified composites.
3. The modified composites with 10 php ETSS had the highest tensile strength compared to others composites.
4. The crystallinity and weight loss of composites decreased with increasing ETSS content. The modified ETSS composites had higher thermal stability and crystallinity than the unmodified composites.
5. The interfacial interaction of composites improved when chemically modified with maleic acid. This was shown in SEM micrographs.

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