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Mechanical and Thermal Properties of Polypropylene/Wax/MAPP Composites Reinforced with High Loading of Wood Flour

†Sun-Young Lee · In-Aeh Kang · Geum-Hyun Doh · D.Jagan Mohan

Division of Environmental Material Engineering, Department of Forest Products, Korea Forest Research Institute, 57 Hoegi-Ro, Dongdaemun-Gu, Seoul 130-712, Republic of Korea

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ABSTRACT : Polypropylene (PP) composites with wood flour/wax/coupling agent were manufactured by melt compounding and injection molding. The influence of wood flour(WF), wax, and coupling agent on the mechanical and thermal properties of the composites was investigated. The addition of wood flour to neat PP has the higher tensile modulus and strength compared with neat PP. The presence of wax also improved the tensile modulus. At the same loading of PP and WF, the addition of coupling agent highly decreased the tensile modulus, and increased the tensile strength. From thermogravimetric analysis (TGA), the addition of wax improved the thermal stability of the composites in the later stages of degradation. The presence of MAPP and wood flour in turn decreased thermal stabilities of composites. From differential scanning calorimetry analysis (DSC), neither the loading of wax, nor the presence of MAPP has shown significant effect on the thermal transition of composites.

Key words: wood flour, tensile properties, thermogravimetric analysis; differential scanning calorimetry, wax, coupling agent

1. Introduction

Since the 1970's, the use of lignocellulosics as fillers and reinforcements in thermoplastics has been gaining acceptance in the building products market [1-3]. The use of natural fibers like wood flour, rice husk, hemp and ramie to reinforce plastics offers a number of advantages over conventional materials because of their low cost, biodegradability, flexibility and low density [4-5]. In particular, wood polymer composites (WPCs) with 50% or less plastic by weight, have been accepted by the construction industry and homeowners, primarily for decking, railing, fencing, roofing, window profile, landscaping and automotives [6]. The market for WPCs in U.S.A. is estimated at about 6.48 x 10^5 ton in 2004 and is expected to reach about 1.9 x 10^6 ton by 2009 [7]. Generally, WPCs have the many specific properties such as high stiffness and impact resistance, dimensional stability,

†Corresponding author (e-mail address: nararawood@foa.go.kr)

resistance to rot, excellent thermal characteristics and low moisture absorption.

Comparing mechanical properties of wood with materials of variable formulation such as WPCs is not a simple task. Properties of wood change with moisture content and those of WPCs depend mainly on manufacturing procedures and parameters such as temperature, wood species, loading level of wood flour, coupling agent and polymer type [2,8,9]. Strength and modulus of WPCs may be inferior to those of wood without addition of coupling agent. However, hardness, abrasion resistance, shear and compression strengths of WPCs can exceed those of wood [10]. Another advantage of WPCs is an ease of molding for hollowed shapes, which is not easy with wood.

Polypropylene (PP) used as the polymer matrix is one of the most important commercial plastics with low price, high stiffness, excellent thermal properties and high melting temperature [11]. PP can easily be injection-molded into solid plastics and extruded into fiber/sheet/film. General applications of PP include WPC, packaging film, fabrics, housewares and automotives.

Coupling agents are used to improve bonding between wood and polymers by creating chemical bonds across the interfaces of the two materials. As the bonding between these materials increases, the strength of WPCs increases. Interactions between wood flour and polymer matrix have been the subject of continuing research because of its importance in composite performance [12-16]. These phenomena are complicated because wood flour and polymer matrix intrinsically bond poorly, but wood flour can nucleate crystal growth in polymer matrix [6]. One of the best-known coupling agent is MAPP (maleated polypropylene). Technically, coupling agents increase the strength and stiffness often referred to as modulus of rupture (MOR) and modulus of elasticity (MOE). They also improve dimensional stability, impact resistance, and fiber dispersion, while lowering creep [17].

Wax as a lubricant is a well-known processing agent added to improve the processing of polymers, especially polyolefins. Incorporation and dispersion of wood fillers are upgraded by the addition of external lubricants which promotes incorporation of fillers. The addition of wax has been found to have a considerable influence on the thermal and mechanical properties of LDPE [18,19] and cross linking as a means to improve the mechanical properties of LDPE-sisal fiber composites has been investigated [20,21].

This study was directed towards the manufacture of WPCs with high loading of wood flour up to 72%. The main objective of this study was to investigate the effect of wood flour, coupling agent and wax on the mechanical and thermal properties of PP composites with wood flour/wax/coupling agent. The mechanical properties and surface degradation after accelerated weathering test were determined. Thermogravimetric analysis (TGA), differential scanning calorimetry (DSC), stress-strain behavior and scanning electron microscopy (SEM) were used to evaluate the thermal, mechanical and morphological properties of WPCs.

2. Materials and methods

2.1. Materials

Polypropylene (PP 5014) was in the powder form courtesy of the Korea Petrochemical Ind. Co., (Republic of Korea). Neat PP has a melt index (MI) of 3.2 g/10min and a density of 0.90 g/cm^3. The wood flour (Lignocel C120) was supplied from J.Retenmaier & Sohne Co. (Germany) and manufactured from an European softwood. Maleated polypropylene (MAPP, Grade PH-200; Honam Petrochemical Co., Republic of Korea) was used as a coupling agent with MI of 1.0

g/10min. The molecular weight and maleic anhydride grafting level of MAPP were 40,000 g/mol and 5%, respectively. Polypropylene-based wax (Grade 502N) was supplied from Lion Chemicals Co., (Republic of Korea). The density and softening temperature of the wax were 0.89 g/cm^3 and 161°C, respectively.

2.2. Melt Compounding

The WPCs with PP/wax/wood flour/coupling agent were manufactured with wood flour (27 to 72 wt.%), wax (10 wt.% based on PP) and MAPP (1 to 5 percent) using a co-rotating twin-screw extruder (Bautek Co., Korea) at a compounding temperature of 180°C. The extruder had a screw diameter of 19 mm with an L/D ratio of 40. The screw speed for compounding of PP and other additives was in the range of 100-150 rpm. The strand extrudate was air-cooled, and then pelletized with a pelletizer (Bautek Co., Republic of Korea). The hybrid pellets were dried at 90°C for 24 h in a vacuum oven and then injection- molded at 190°C.

2.3. Thermal Properties

Thermogravimetric analysis (TGA) was employed to investigate thermal decomposition behavior of WPCs (TA Instrument SDT Q600, U.S.A.). Tests were done under nitrogen at a scan rate of 10°C/min in the temperature range of 30 to 600°C. A sample of 5 to 10 mg was used for each run. The weight change (%) was recorded as a function of temperature. Peak temperature (T_p) was a maximum temperature acquired from the differentiation of the weight change by time. Differential scanning calorimetry (DSC) experiments were performed in a DSC (Q10, TA instrument, U.S.A). Each sample was heated and cooled at a scanning rate of 10°C/min under nitrogen in order to prevent oxidation. Each test sample of 5 to 10 mg was placed in an aluminum capsule and heated from 30 to 200°C. The melting temperature (T_m), enthalpy (H_m), crystallization temperature (T_c), crystalline enthalpy (H_c) and crystallinity (X_c) were determined after the melt-crystallization process.

2.4. Mechanical Properties

The tensile strength testing for WPCs was determined according to ASTM D638 using a universal testing machine. Test specimens were molded to 13.0 mm (width) x 57.0 mm (length) x 3.00 mm (thickness) with a gauge length of 50.0 mm. For each treatment level, five replicates were tested and the results were presented as the average of five replicates. Tensile strength and modulus were accurately determined using an extensometer at a crosshead speed of 10 mm/min.

2.5. Morphological Properties

Study on the morphology of the fractured composites after tensile testing was observed by scanning electron microscope (SEM) (JSM-6300F, JEOL Japan) with a field emission gun and an accelerating voltage of 10 kV. A gold layer of a few nanometers in thickness was coated onto tensile fractured surfaces and the samples were scanned perpendicular to the fractured surface. SEM micrograph magnification was 200 times.

3. Results and discussion

3.1. Mechanical properties

The tensile properties of WPCs were measured by means of a Universal Testing Machine (Figs. 1 to 4). Fig. 1(a) and (b) show the relationship between tensile properties and the PP composites with and without wood flour/wax. As shown in Fig. 1(a), the minimum tensile modulus was shown in neat PP (1,272 kgf/cm^2). The PP50/WF50 composite has the tensile modulus of 3,209 kgf/cm^2, indicating the higher stiffness and brittleness than neat PP.

On the other hand, the addition of wax (2.5%) to PP50/WF47.5 mixture increased the tensile modulus of 6.2%. As the loading of wood flour decreased from 50 to 40% and the loading of wax increased from 2.5 to 10%, the tensile modulus of the composites slightly decreased (Fig. 1(a)). Fig. 1(b) represents the relationship between tensile modulus and the polymers with different loadings of wood flour (27 to 63 wt.%) and wax (3 to 7 wt.%). As the loading of wood flour increased from 27 to 63 wt.% and the loading of wax increased from 3 to 7 wt.%, the tensile modulus of the PP/WF/wax composites increased from 101.5 to 188.8%, compared with neat PP. The increase of tensile modulus of the PP/WF/wax composites was mainly due to the addition of wood flour, and partially a better dispersion of wood flour [14]. The PP70/WF27/WAX3 composite showed 13.1% higher tensile modulus than that of PP60/WF36/WAX4 composites. As shown in Fig. 1(a), the lowest tensile modulus was shown in neat PP (297 kgf/cm^2). However, the presence of wood flour (50 wt.%) to neat PP showed 28.6% increase in tensile strength, compared to neat PP. This result was obviously different from the previous literatures which showed the decrease of tensile strength with the increased loading of wood flour [2,22]. It is assumed that the surface of wood flour used in this study was chemically-modified to have the characteristic of hydrophobic. Therefore, wood flour and PP matrix may have a good compatibility to improve the tensile strength at the equal loading level (50 wt.%: 50 wt.%). As the loading of wood flour decreased from 47.5 to 40 wt.% and the loading of wax increased from 2.5 to 10 wt.%, the tensile strengths of the composites increased from 374 to 407 kgf/cm^2. Fig. 1(b) shows the relationship between tensile strength and the polymers with different loading of wood flour (27 to 63 wt.%) and wax (3 to 7 wt.%). At higher loading of wood flour (63 wt.%), hydrophilicity of the composites has more negative effect on the resultant tensile strength. It can be seen that the values decreased from 377 to 317 kgf/cm^2 at the loading of wood flour (27 to 63 wt.%) and wax (3 to 7 wt.%).

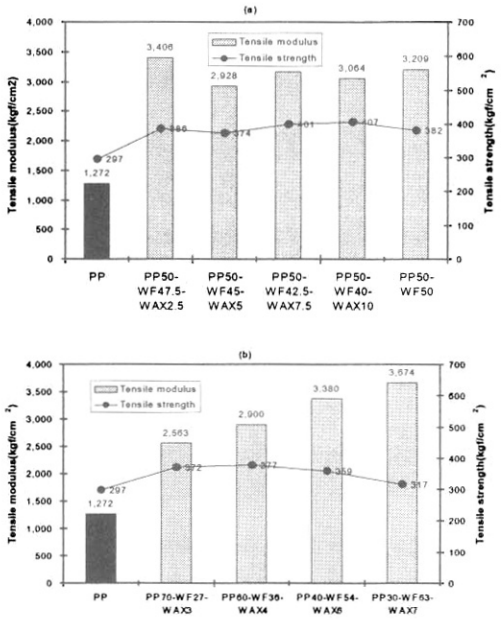


Fig. 1. Tensile modulus and strength of (a and b) PP/WF/WAX composites.

The effect of MAPP on the tensile modulus of PP/WF hybrids was shown in Fig. 2(a). The composite with PP (30 wt.%) and wood flour (70 wt.%) has 128.5% higher tensile modulus than neat PP. At the same loading of PP and WF, the addition of MAPP (1 to 5 %) decreased the tensile modulus of the composites. The decrease of tensile modulus of the composites with MAPP of 1 to 5 percent was in the range of 68.7 to 50.3%. However, no particular trend for the decrease of tensile modulus was observed. As shown in Fig. 2(b), the addition of wax (7 wt.%) to PP/WF decreased the tensile modulus up to 13.8%. In addition, at the highest loading of MAPP (5 %), the linear increase of tensile modulus of the hybrids

was witnessed by the addition of wax. The significant increase in tensile strength of the PP/WF composites was found by the addition of MAPP (Fig. 2(a)). The maximum tensile strength of the composites with coupling agent at wood flour level of 70 wt.% was 602 kgf/cm^2. The increase of tensile strength obtained by adding MAPP of 1, 3 and 5 percent was in the range of 74.1 to 89.9 % in comparison with PP/WF composites. This may be due to an increase of interfacial bonding strength between PP matrix and wood flour, and the increase of compatibility between two polymers through the reduction of the interfacial tension [2,14,23]. The effect of wax on the tensile strength of PP/WF/MAPP composites was shown in Fig. 2(b). The addition of wax (7 wt.%) slightly decreased the tensile strength (0 and 0.2 %)

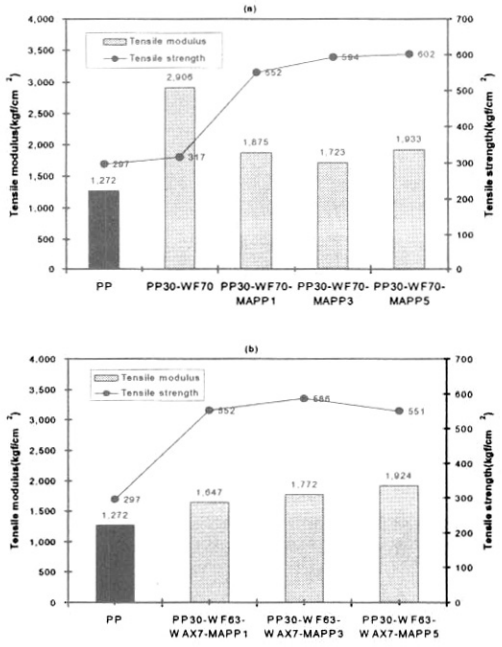


Fig. 2. Tensile modulus and strength of (a) PP/WF/MAPP and (b) PP/WF/WAX/MAPP composites.

at the low loading of MAPP (1 and 3 %). By contrast, at the loading of MAPP (5 percent), the tensile strength of PP30/WF63/WAX7 composites significantly decreased (9.1%).

3.2. Thermal properties

3.2.1. Thermogravimetric analysis

Fig. 3(a) and (b) show the TGA curves of neat PP and PP/wood flour/wax composites containing different loadings of wood flour and wax. The TGA curves of these hybrids show a single stage weight loss (Fig. 3(a)). It can be seen that for all the composites, the main degradation starts at 280C and leaves a residue at about 500°C or higher. The initial decomposition temperature (IDT) has been decreased with the addition of wax. It indicates that the thermal stability for the composites decreases with an increase in wax content. Despite the fact that it has lower degradation temperature, the final degradation of all the composites is higher than the neat PP. All the composites have decomposed at >500°C. It is noteworthy that after 50% decomposition all the curves have shifted towards higher temperatures except for PP50/WF40/WAX10, which has shifted after >85% decomposition. If IDT is the way to judge the thermal stability of the material, then these composites are less stable compared to the neat PP. On the other hand, if the thermograms based on initial, central and final stages of degradation were evaluated, then it seems that with the increase in temperature the thermal stability has improved with the addition of wax. This fact indicates a higher level of compatibility of PP, wood flour and wax, at lower composition of wax. The composite of PP50/WF40/WAX10 is less stabile as compared to PP50/WF47.5/WAX2.5, PP50/WF45/WAX5 and PP50/WF42.5/WAX7.5 composites. As shown in Fig. 3(b), the weight loss has occurred in two different stages for PP30/WF63/WAX7. The first stage

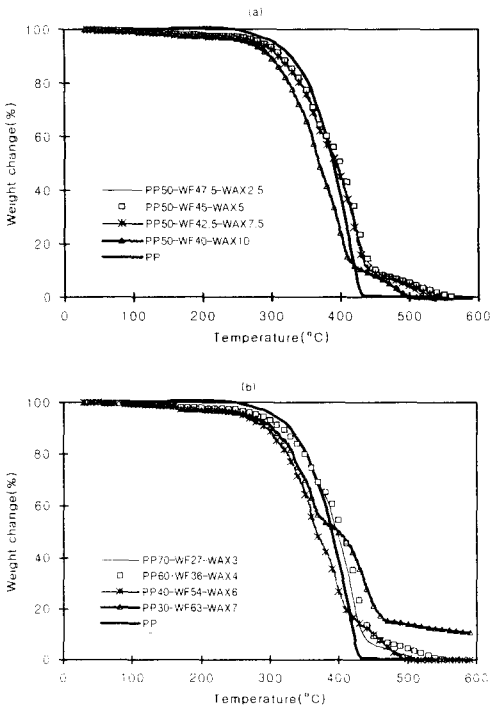


Fig. 3. TGA thermograms of (a and b) PP/WF/WAX composites.

was observed in the temperature range 280-380°C, and the second stage weight loss was observed above 380°C, with 40% decomposition. Remaining composites, i.e., PP70/WF27/WAX3, PP60/WF36/WAX4 and PP40/WF54/WAX6, followed the same pattern as witnessed in the previous hybrids in Fig. 3(a). These composites show better thermal stability as compared to neat PP. After 30-40% weight loss, all the composites shifted towards the higher temperature, except for PP40/WF54/WAX6 composite, which exhibited the least thermal stability. The final degradation of all composites was in the temperature of >500C. It was reported that the thermal stability of blends decreased with increase in wax content [24,25]. Although the thermal stability of hybrids showed lower IDT, the stability was improved with the increase in temperature. From the data, the final degradation of PP occurred faster as compared to all the composites. There seems to be a much stronger interaction between wax and wood flour. The resultant improved interfacial interaction, and therefore, explains the improved thermal stability of the composites.

The effect of coupling agent (MAPP) on the thermal stability of composites was also studied by TGA in Fig. 4(a) and (b). During the thermal degradation in inert atmosphere all the composites exhibited single step degradation except for PP30/WF70 and PP30/WF70/MAPP1 composites, which showed a three-stage degradation pattern. The TGA scans of neat PP and PP/WF/MAPP composites are shown in Fig. 4(a). The presence of MAPP causes the shift of initial weigh loss towards lower temperature (280°C). The stability increased in the later stages of heating. It was shown that PP30/WF70 and PP30/WF70/MAPP1 composites have shifted to higher temperature after 50% weight loss, whereas, PP30/WF70/MAPP3 and PP30/WF70/MAPP5 composites have shifted to higher temperature in the later stages of thermal heating. Hence, the result indicate that the thermal stability has much improved in the later stages of heating. This may be due to the enhanced interfacial adhesion and additional intermolecular bonding between hydroxyl groups of wood flour and the anhydride functional group of MAPP. In general, PP/wood flour/WAX/MAPP composites showed a significant decrease in the thermal stability (Fig. 3(b)). Nevertheless, there is a big difference in the final decomposition temperature of hybrids. The decomposition of neat PP occurs at 320°C, whereas, other hybrids degrade at 280°C and above. From the thermograms, it was shown that the last 20% decomposition occurred in a slow manner for all the composites. In case of PP/WF/WAX/MAPP composites, the degradation patterns are similar to one another. The addition of MAPP to

PP/WF/WAX composites decreased the thermal stability of composites. There are two main features worthy noting for all composites. Firstly, they showed slow and gradual degradation after 80% weight loss. Secondly, their final decomposition temperature was much higher than neat PP.

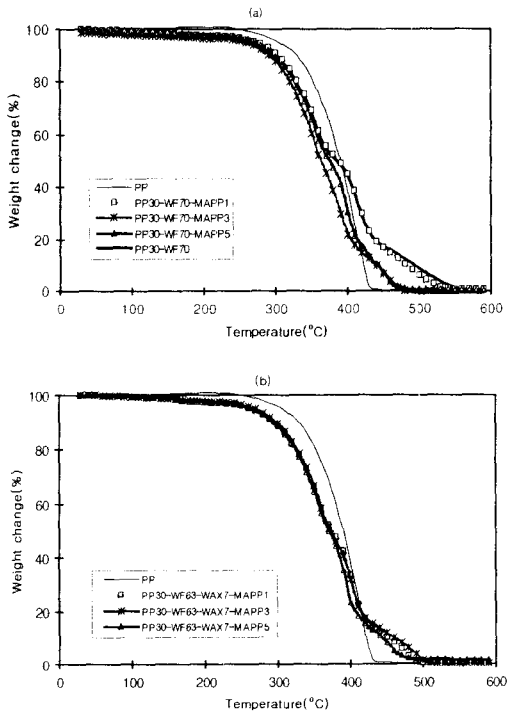


Fig. 4. TGA thermograms of (a) PP/WF/MAPP and (b) PP/WF/WAX/MAPP composites.

3.2.2. Differential scanning calorimetry

The DSC thermograms for PP/WF/WAX composites are shown in Figs. 5 (a) and (b). The melting temperature (T_m), enthalpy (H_m), crystalline temperature (T_c), crystalline enthalpy (H_c) and crystallinity (X_c) values of composites obtained from DSC scans are summarized in Tables 1 and 2. Generally, the melting behavior of composites depends on heating rate and thermal history of polymer matrix. There are very small differences in the onset and peak temperatures of melting and crystallization, for all the composites. There was no general trend observed in the variation of these parameters. Therefore, it is clear that neither the loading of wax, nor the presence of MAPP significantly influence the thermal transition of hybrids. It was observed that the increase in wax content in PP/WF composites shifted endothermic peak towards the lower temperature. It is known fact that the PP and wax are not miscible due to different crystal morphologies. PP crystallizes in helical form [26] and wax in various structures [27]. Possibly wax penetrates through the wood flour leaving less space for the polymer to contact with wood flour.

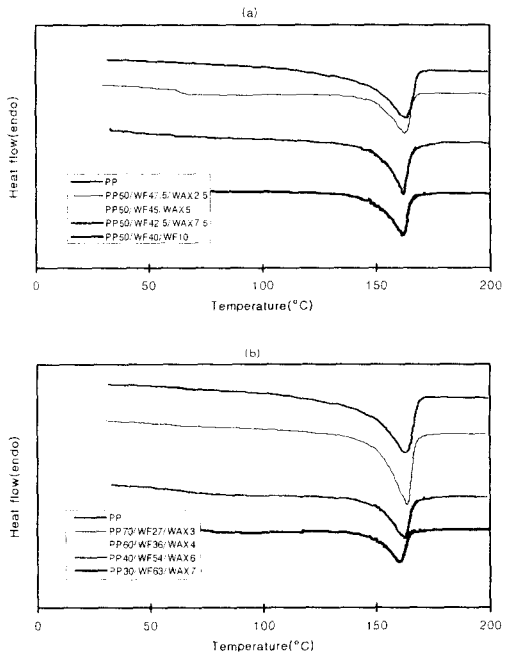


Fig. 5. DSC melting results of (a and b) PP/WF/WAX composites.

Similar trends are observed during the crystallization of the composites. The T_c decreases slightly with an increase in wax content. The T_m of the composites decreases with the increase in wax content, which indicates plasticization of PP by wax. The T_m of neat PP (162.8°C) was 2.6°C lower

Table 1. Tensile strength of PP/WF/WAX/MAPP composites

Systems	Tensile modulus (kgf/cm^2)	Tensile strength (kgf/cm^2)
PP	1,272 (245.8)	297 (45.1)
PP50/WF50	3,209 (350.6)	382 (24.6)
PP70/WF27/WAX3	2,563 (354.2)	372 (39.0)
PP60/WF36/WAX4	2,900 (313.7)	377 (25.2)
PP40/WF54/WAX6	3,380 (245.7)	359 (23.1)
PP30/WF63/WAX7	3,674 (349.4)	317 (52.1)
PP30/WF63/WAX7/MAPP1	1,647 (240.5)	552 (25.8)
PP30/WF63/WAX7/MAPP3	1,772 (342.1)	586 (29.3)
PP30/WF63/WAX7/MAPP5	1,924 (246.8)	551 (27.3)
PP30/WF70	2,906 (310.1)	317 (31.4)
PP30/WF70/MAPP1	1,875 (325.3)	552 (34.2)
PP30/WF70/MAPP3	1,723 (432.1)	594 (35.9)
PP30/WF70/MAPP5	1,933 (298.9)	602 (42.1)

Values in parenthesis are the standard deviation.

Table 2. Results of DSC analysis of PP/WF/WAX composites.

Systems	T_m (°C)	H_m (J/g)	T_c (°C)	H_c (J/g)	X_c (%)
PP	162.8	81.82	108.7	91.03	39.15
PP50/WF47.5/WAX2.5	162.7	39.98	117.6	47.65	19.13
PP50/WF45/WAX5	161.6	51.62	116.5	49.28	24.70
PP50/WF42.5/WAX7.5	161.6	53.27	116.3	49.23	25.49
PP50/WF40/WAX10	163.4	44.92	116.2	47.57	21.49
PP70/WF27/WAX3	163.1	68.67	117.2	67.10	32.86
PP60/WF36/WAX4	162.4	62.73	117.4	60.95	30.01
PP40/WF54/WAX6	160.2	37.98	117.7	38.72	18.17
PP30/WF63/WAX7	162.2	26.79	116.8	33.08	12.82
PP50/WF50	162.3	41.83	116.2	42.19	20.01

WF = wood flour, T_m = melting point, H_m = enthalpy, T_c = crystalline temperature, H_c = crystalline enthalpy; X_c = crystallinity.

than that of PP40/WF54/WAX6 composite (160.2°C). The T_c values are in the range of 116.2 to 117.6 °C (Figs. 6(a) and (b)). Neat PP possesses the lowest T_c value of 108.7 and PP50/WF47.5/WAX2.5 composite has the highest T_c value of 117.6. The specific enthalpy of melting increases with increase in wax content, since this value is bigger for wax than for PP.

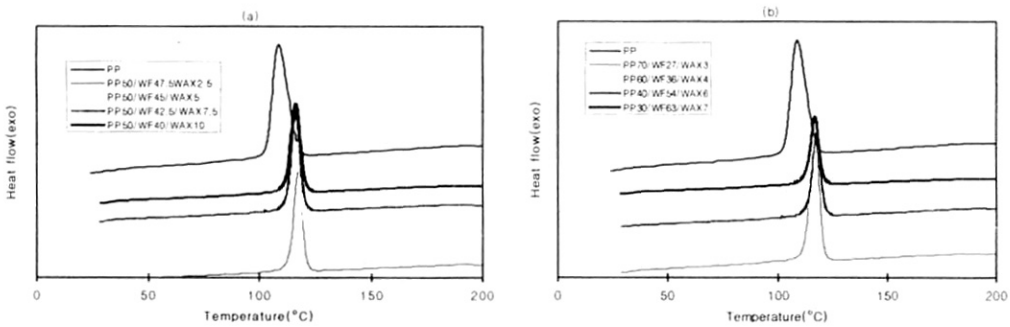
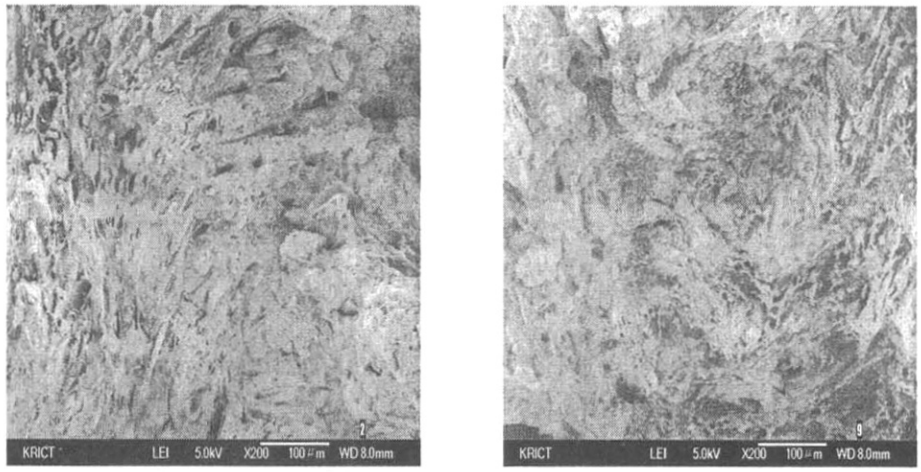


Fig. 6. DSC crystallization results of (a and b) PP/WF/WAX composites.



(a) (b)

Fig. 7. SEM images of (a) PP50/WF45/ WAX5 and (b) PP50/WF50 composites.

3.3.3. Morphological properties

The SEM photomicrographs of the composites with PP50/WF45/WAX5 and PP50/WF50 composites prepared by the compounding are shown in Figs. 7(a) and (b), respectively. The compounding method with a co-rotating twin extruder showed an uniform distribution of wood flour in the PP matrix and wood flour particles were encapsulated in the PP matrix. The addition of 5% wax showed no significant effect on the morphology of the resultant composites. However, both PP matrix and wood flour were better compounded by the addition of wax.

4. Conclusions

WPCs with PP/wood flour/wax/coupling agent were manufactured by melt compounding and injection molding. The work was focused to investigate the effects of wood flour, wax, and coupling agent on the mechanical and thermal properties of WPCs. The addition of wood flour to neat PP increased the tensile modulus and strength compared to neat PP. The presence of wax also improved the tensile modulus of the composites. The addition of MAPP with PP/wood flour highly decreased the tensile modulus, and increased the tensile strength.

From TGA analysis, the loading of wax improved the thermal stability of the composites in the later stages of degradation. The coupling agent and wood flour, however, decreased thermal stabilities of the composites. From DSC analysis, wax and MAPP provided no significant effect on the thermal transition of composites.

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