

## Highly Filled Polypropylene Rubber Wood Flour Composites

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**Abstract.** In this research, polypropylene composites filled with *Hevea Brasilliensis* wood flour at filler content up to 60wt% were prepared and investigated in order to determine the effects of polymer melt flow rate (MFR), number of reprocessing times, filler size, and filler content on thermal and mechanical properties. The results reveal that the composites of polypropylene with higher melt flow rate (lower viscosity) provided greater values of flexural and tensile properties. The study additionally exhibits the recyclability potential without losing mechanical properties. Furthermore, both flexural and tensile modulus increased, while both flexural and tensile strength decreased with increasing wood flour contents. In addition, the average particle size of wood flour that was suitable for improving the mechanical properties was approximately 200-300  $\mu\text{m}$ . In the last section of the research, the effects of maleic anhydride grafted polypropylene (MAPP) coupling agent were investigated. It is worth noting that, the flexural strength and tensile strength of highly filled composites with MAPP at 5wt% (based on wood flour) were approximately 110 % and 87% higher than those of the composites without MAPP, respectively. In the presence of coupling agent, the enhancement of interfacial adhesion was analyzed using scanning electron microscope (SEM).

**Keywords:** Polypropylene, highly filled system, wood composite, reprocessing.

ENGINEERING JOURNAL Volume 15 Issue 2

Received 14 December 2010

Accepted 10 January 2011

Published 1 April 2011

Online at <http://www.ej.eng.chula.ac.th/eng/>

DOI:10.4186/ej.2011.15.2.17

## 1. Introduction

The use of wood-based materials (wood flour and wood fibers) as reinforcing fillers for thermoplastics has received great attention from a number of researchers and manufactures in recent years. [1] The addition of the renewable natural filler in polymeric composites aims to produce unique characteristics of great versatility, light weight, biodegradability and recyclability with good specific properties. In comparison with other fillers such as traditional glass fiber and inorganic mineral fillers, wood is inexpensive, low density and non-abrasive to processing machinery. [2]

Rubber tree (*Hevea brasiliensis*) is an economic plant widely cultivated in more than 30 countries around the world confined to the tropical and subtropical regions. The total area of plantations world wide is ca. 90,000 million m<sup>2</sup>. Generally, the plant is mainly grown for the production of latex and cut down when it becomes unproductive [4]. The rubber wood products are mainly used as particleboards, packing materials, furniture, and construction materials. In average, the rubber tree plantation of 1,600 m<sup>2</sup> provides 40 m<sup>3</sup> of rubber timber to feed wood factories. In this amount, the sawdust waste of 7.5 m<sup>3</sup> (ca. 19% of 40 m<sup>3</sup>) is generated. This high amount of waste has to be disposed, which causes environmental pollution. Therefore, the use of rubber sawdust waste as filler in polymer composites will tremendously help solving the severe environmental problem. [3]

Polypropylene is one of the most versatile commodity thermoplastics with low cost, high thermal stability and low density. The polymer is used in a wide variety of applications, including packaging, textiles, stationery, plastic parts and reusable containers of various types, laboratory equipment, automotive components, and polymer banknotes. For over a decade, there have been a number of researches related to polyolefin composites reinforced with wood-based filler [4-14]; i.e. Bledzki et al. [4] studied about polypropylene composite with modified cellulosic filler. They found that the tensile strength of modified composites increased for 45% in comparison to unmodified fiber composites. Premalal et al. [5] reveals that the rice husk powder filled polypropylene composites provided lower Young's modulus and flexural modulus than talc composites. Adhikary et al. [6] exhibits the water absorption of the wood flour-recycled high-density polyethylene composites increased with weathering. Ichazo et al. [7] reported that the polypropylene/wood flour composite with maleated polypropylene (MAPP) treatment provided slightly increase of tensile modulus and tensile strength. Mantia et al. [8] indicated that the organic filler-polypropylene composites caused enhancement of the rigidity and thermomechanical resistance, which was similar to the one observed for inorganic filler. Nachtigall et al. [9] showed that tensile strengths of polypropylene/wood flour composites with coupling agent were higher than that of the one without coupling agent. Kim et al. [10] presented that MAPP-treated polypropylene composites showed improved mechanical and thermal stability. This enhancement is also observed in the system of polyethylene and wood flour with maleated polyethylene as compatibilizing agent. [11] Sombatsompop et al. [12] reported that the increased of wood fiber into the polypropylene matrix could reduce the overall strength and toughness of the composites. Balasuriya et al. [13] exhibited that the wood flake wetting and filler distribution had a profound effect on the mechanical properties. Guo et al. [14] revealed that the storage modulus of wood flour filled polypropylene composites was improved when MAPP was applied.

Most of the researches were focused on the wood flour composites without reprocessing information at low filler content, i.e. 10-40wt%; however, none of the studies was based on considerably higher wood flour content at various numbers of reprocessing times. Practically, the important point is that the increase of wood filler content is able to reduce the production cost of the composites. Moreover, the information of mechanical properties at various numbers of reprocessing times is important in order to understand the recycling potential of the composite system.

In this research, the information about the mechanical properties of reprocessed composites prepared from polypropylene of different melt flow rates is provided. This is useful for the composite manufacturer to realize the change of mechanical properties after recycling. Furthermore, the mechanical properties of the composite at various wood flour contents (from 0-60 wt%) and different particle sizes are compared in the work. This is crucial to comprehend the behaviors of the wood flour composites and to further select the filler size and content to suit the composite applications.

The objective of this study is to investigate the effects of the melt flow rate of polypropylene matrix, wood flour particle size, filler content, and number of reprocessing time on the mechanical properties of wood flour filled polypropylene composites. Furthermore, the effects of MAPP coupling agent on the

thermal and mechanical properties of the highly filled composites were additionally determined in the last section of the research.

## 2. Experimental

### 2.1. Materials

Three commercial types (Moplen HP740H, HP648N and HP644T) of polypropylene were supplied by HMC Polymers Co., Ltd. (Thailand). In order to study the effects of melt flow rate, all three polypropylene types with different melt flow rates (injection moulding grades) were applied. The typical characteristics are shown in Table 1. *Hevea brasiliensis* wood flour from rubber tree with actual density of 1.49 g/cm<sup>3</sup> [6] was size-classified and dried at 105°C for 24 hours in a vacuum oven to constant weight and was kept in a desiccator before using.

Table 1. Typical characteristics of three types of commercial polypropylene.

Characteristic	HP740H	HP648N	HP644T
Melt flow rate, g/10min	2.1	12	60
Flexural modulus, GPa	1.75	1.69	1.63
Tensile strength at yield, MPa	36	38	38
Flexural strength, MPa	60.5	59.6	59.2

Maleic anhydride grafted polypropylene (MAPP) coupling agent with trade name of Fusabond MZ109D was supplied by Creative Polymer Co., Ltd., Thailand. The melt flow rate (MFR) of MAPP was 120 g/10min, according to ASTM D-1238 and maleic anhydride grafted to polypropylene is 0.55% by weight.

### 2.2. Preparation of Wood Flour Composites

In order to determine the effects of melt flow rate of polypropylene matrix and number of reprocessing times, three types of polypropylene and wood flour filler (average particle size of 215 µm) were mixed thoroughly at 40wt% using a twin screw extruder Rheomix PTW 16/25, THERMO HAAKE, MA: L/D = 25 and D = 16 mm.) at a screw speed of 20 rpm. The blending temperature profiles from hopper to die zones on the extruder were set at 170, 175, 180 and 185°C, respectively. The extrudate strands were cooled in air prior to producing compound granules. Then, the granules were dried at 105°C for 24 hours and then injected to the mold using an injection moulder (MANUMOLD, model 45E) at 190°C and 0.65 MPa.

To investigate the effects of particle sizes and filler contents, wood flour filler at various particle sizes (49, 90, 165, 215, 275, 362, and 512 µm) was thoroughly mixed with polypropylene at various filler loadings (10, 20, 30, 40, 50, and 60wt%) before compounding. The extrusion and injection conditions are the same as previously mentioned.

In order to evaluate the effects of coupling agent contents on the properties of wood composites, polypropylene pellet, wood flour filler and MAPP were mixed thoroughly before compounding. The composites were prepared at 60wt% of 275-µm wood flour with maleic anhydride grafted polypropylene (0 to 20wt% based on wood flour).

### 2.3. Mechanical property analysis

Flexural properties of the wood composite specimens were determined following ASTM D790-M93, using a universal testing machine (Instron 5567). The test was carried out in three-point bending mode with a support span of 48 mm at the crosshead speed of 1.2 mm/min. The dimension of the each specimen was 20 × 60 × 3 mm<sup>3</sup>. Tensile properties of wood composites were investigated using Instron 5567 according to ASTM D638 with a speed of 5.0 mm/min. The test specimens are a dumbbell shape with a uniform thickness. The average flexural and tensile properties were determined from five tests on each material.

## 2.4. Differential scanning calorimetry (DSC)

Differential scanning calorimeter model 2910 from TA instruments was used to evaluate thermal behaviors of specimens (approximately 10 mg) at 10°C/min from 30-200°C under nitrogen atmosphere. The purge nitrogen gas flow rate was 50 ml/min. The melting temperature ( $T_m$ ) and melting enthalpy ( $\Delta H_f$ ) of each sample were obtained from the maxima and area of the melting peaks of thermograms, respectively.

## 2.5. Thermogravimetric analysis (TGA)

The decomposition temperature at 5 % weight loss of the sample of approximately 10 mg were determined using PerkinElmer TG/DTA thermogravimetric analyzer (model SII Diamond) at 20°C/min from 40-800°C under nitrogen atmosphere. Char yield of polypropylene and wood composites were reported at 800°C.

## 2.6. Morphological study

The fracture surface of composites was observed by using ISM-5400 scanning electron microscope (SEM) at an acceleration voltage of 10 kV. The specimen was coated with gold prior to scanning. The morphology such as filler dispersion and interfacial adhesion of the components were investigated.

## 3. Results and Discussion

### 3.1. Effects of melt flow rate of polymer matrix and number of reprocessing times

Figure 1(a) and 1(b) show the effects of polypropylene types with different melt flow rates and number of reprocessing times on flexural modulus and tensile modulus of the composites. In comparison with the composites of HP740H (highest viscosity) and HP648N (moderate viscosity), the composites of HP644T (lowest viscosity) seemed to render the highest flexural modulus and tensile modulus, respectively. This is due to the wetting ability of polymer matrix on wood flour particles. As the viscosity of polymer matrix is low, the polymer matrix is thoroughly around wood flour particles; therefore, relatively high mechanical properties can be expected. The results are similar to the polyethylene and wood flake composites [13].

Figure 2a and 2b show the effects of polypropylene types and number of reprocessing times on flexural strength and tensile strength of the composites at 40wt% of wood flour, respectively. It was found that the differences of the flexural and tensile strength in Figures 2a and 2b for the composites without reprocessing are in the range of error bars. However, the differences are more clearly observed in the composites with reprocessing for 1-3 times. Furthermore, it was found that the trends of flexural strength and tensile strength affected by polypropylene types are similar to those observed in flexural modulus and tensile modulus. The flexural strength and tensile strength of wood flour composite which was processed from low-viscosity polypropylene (HP644T) was higher than those of composites produced from moderate-viscosity polypropylene (HP648N) and high-viscosity polypropylene (HP740H). This is possibly due to better stress transfer of the low-viscosity polypropylene composites caused by higher wetting behavior of polypropylene on the filler [15].

The particle aggregation is an important factor having an influence on the final properties of wood flour composites; therefore, the re-compounding process was applied to the molding compound in order to eliminate the aggregation problems. From Figure 1 and 2, it can be seen that the number of reprocessing times showed no significant effect on the ultimate flexural properties and tensile properties (regardless of melt flow behavior of polymer matrix). In addition, the flexural and tensile properties showed no significant change in values even though the molding compound was reprocessed up to 3 times. This indicates relatively well distribution of wood flour in the polymer matrix without filler agglomeration in the compounding process. The results were similar to those observed in the 20wt% sisal fiber/LDPE matrix composites [16]. Therefore, in this work, one time of passing through an extruder was enough for achieving the ultimate mechanical properties. It also implies one major

advantage of the wood flour polypropylene composites, i.e. recyclability to reduce the environmental wastes without losing any mechanical properties. Furthermore, wood flour composites from HP644T (lowest viscosity polypropylene) possessed the highest mechanical properties. Therefore, the experiments to study the effects of filler particle sizes, and filler contents were based on HP644T polypropylene type.

### 3.2. Effects of particle sizes and filler contents

Figure 3 and 4 show the effects of particle sizes and wood flour contents on the flexural and tensile moduli of composites, respectively. It could be seen that at the same average particle size, both flexural and tensile moduli of the composites were increased with the contents of wood flour. The modulus values of the composites at all filler contents are higher than those of neat polypropylene. This similar phenomenon could be observed in the composite systems of polypropylene filled with rice husk powder [5], and other organic fillers (such as kenaf fiber and sago starch) [8]. This could be possibly caused by the fact that the wood flour possesses higher modulus in nature than the polymer [12]. Moreover, at low filler contents (i.e. 10-20wt%), there was no significant effect of particle size on the moduli of the composites. However, at high filler contents, the highest values of moduli presented at the average particle size of 275  $\mu\text{m}$ . The flexural and tensile moduli of wood flour composites at 275  $\mu\text{m}$  particle size with 60wt% are approximately 300% and 250% higher than those of neat polypropylene, respectively. Furthermore, it could be noticed that the flexural modulus and tensile modulus were decreased when average particle size was greater than 275  $\mu\text{m}$ ; it is possibly because surface area to transfer load between matrix and filler in case of large-sized particle is less than that of the optimum particle size (275  $\mu\text{m}$ ). This phenomenon was also found in the similar system between bamboo sawdust and polyvinyl chloride. [17] However, at high filler contents, the highest values of moduli presented at the average particle size of 275  $\mu\text{m}$ . The flexural and tensile moduli of wood flour composites at 275  $\mu\text{m}$  particle size with 60wt% are approximately 300% and 250% higher than those of neat polypropylene, respectively. Furthermore, it could be noticed that the flexural modulus and tensile modulus were decreased when average particle size was greater than 275  $\mu\text{m}$ ; it is possibly because surface area for polymer wetting in case of large-sized particle is less than that of the optimum particle size (275  $\mu\text{m}$ ). Therefore, the load transfer between the matrix and large-sized filler was lower than that of the composites filled with the particle size of 275  $\mu\text{m}$ .

In case of small-sized particle (much smaller than 275  $\mu\text{m}$ ), flexural and tensile moduli were relatively low due to the fact that there was difficulty in dispersing the small-sized particles due to particle-particle interactions. The particles could form agglomeration which constitute flaws and as the agglomerates becomes larger in size, wetting of small-sized filler by polypropylene macromolecules also become poor, resulting in voids between filler and matrix. These factors could have an effect on the low values of flexural and tensile moduli. The similar effects of particle size on the surface area for polymer wetting, which impacted on flexural and tensile properties, were also observed in the literatures [5, 12]. Furthermore, the filler and matrix are rather incompatible due to hydrophilicity of wood flour and hydrophobicity of the polypropylene matrix [18]. As a result, interfacial adhesion between wood flour and polypropylene is relatively weak, which results in some unsatisfactory properties of the composites. In addition, the small average particle size possesses relatively high surface area. Therefore, the polypropylene matrix may not be sufficient to totally cover the surface of wood flour. This leads to the presence of void in the specimens. Hence, the obtained flexural modulus and tensile modulus are slightly low [19]. This assumption can be proven by checking the actual densities of the composites at various sizes of filler, which are not shown in the paper. Furthermore, small wood flour filler tends to cling or agglomerate together, due to hydrogen bonding, or other secondary forces, and resists dispersion of the individual filler particle as the filler content is increased [12].

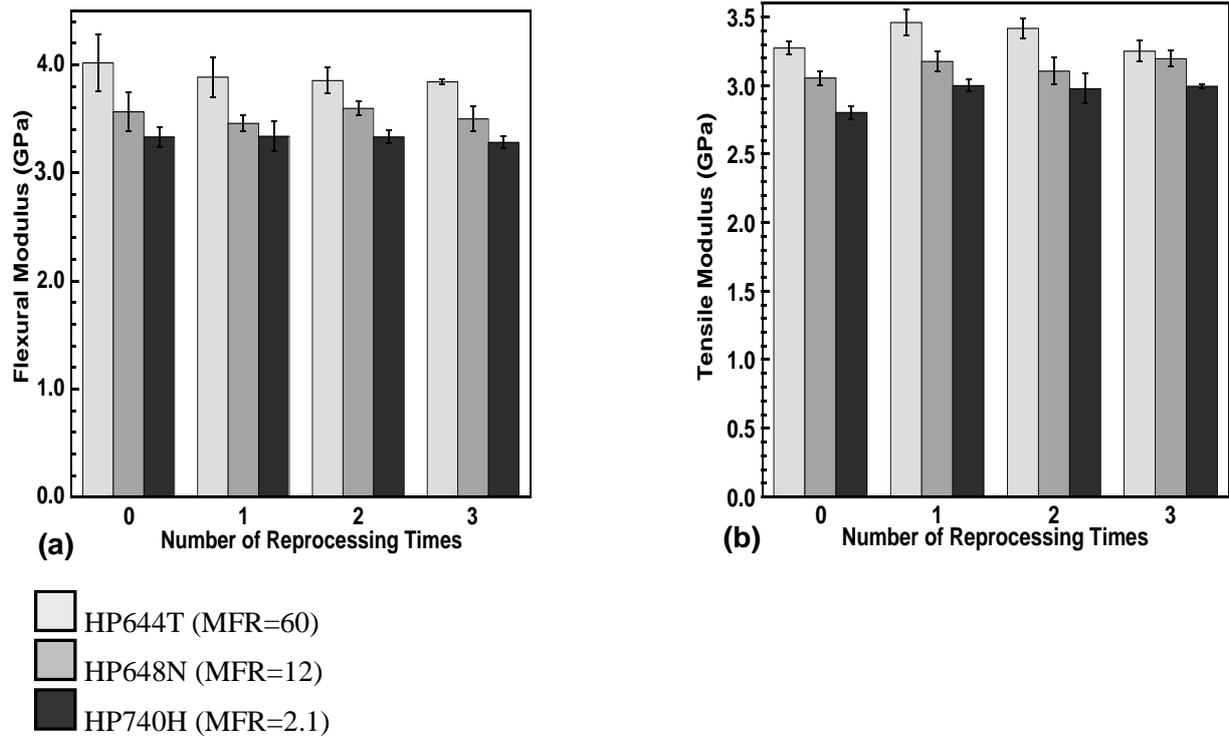


Fig. 1. Modulus of the composites (40wt%, 215 $\mu$ m) at various types of polypropylene and numbers of reprocessing times: (a) flexural modulus, (b) tensile modulus.

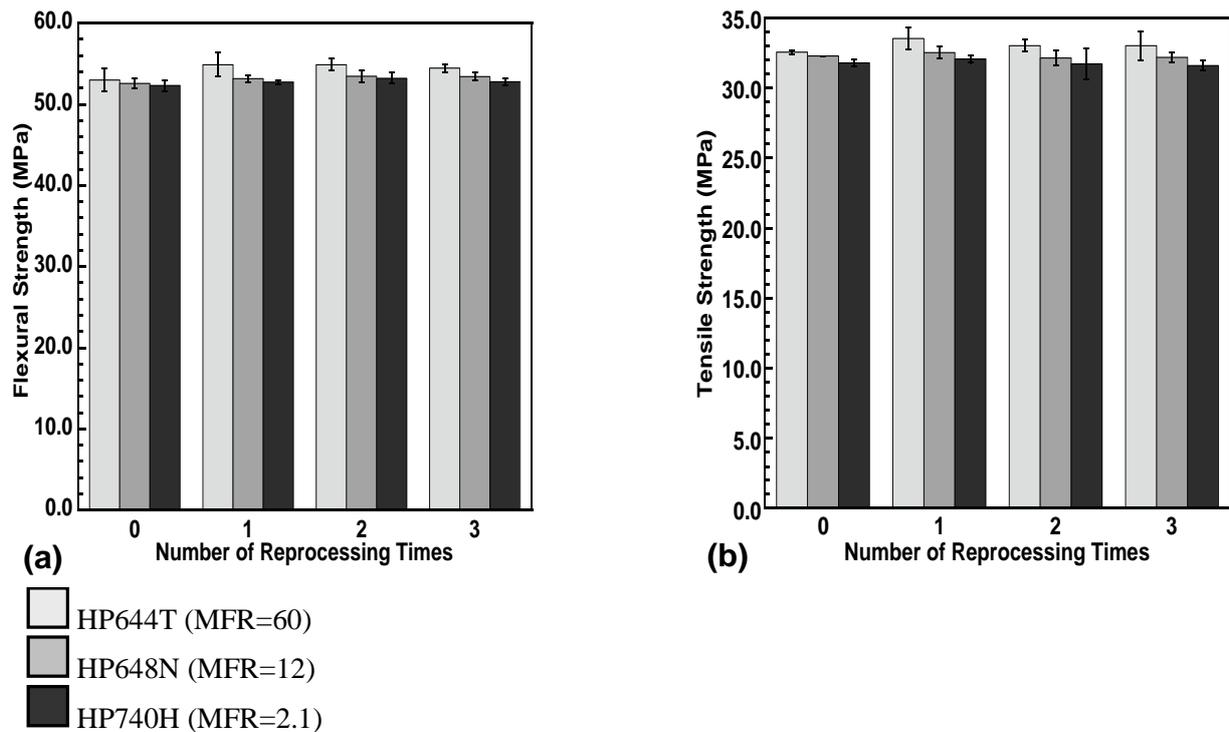


Fig. 2. Strength of the composites (40wt%, 215 $\mu$ m) at various types of polypropylene and numbers of reprocessing times (a) flexural strength, (b) tensile strength.

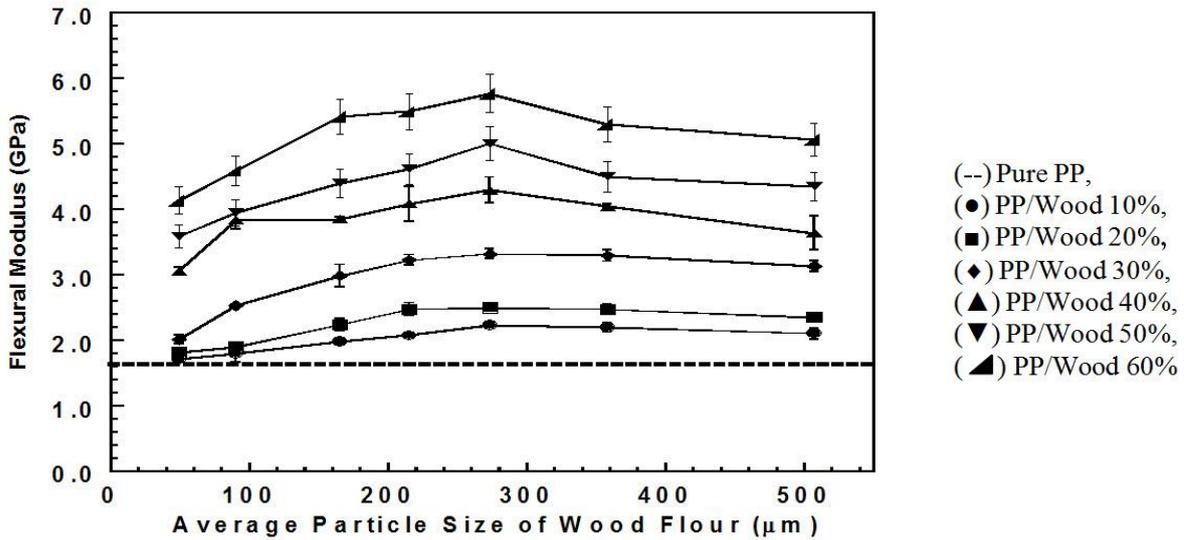


Fig. 3. Effect of wood flour particle size on flexural modulus of polypropylene/wood flour composites.

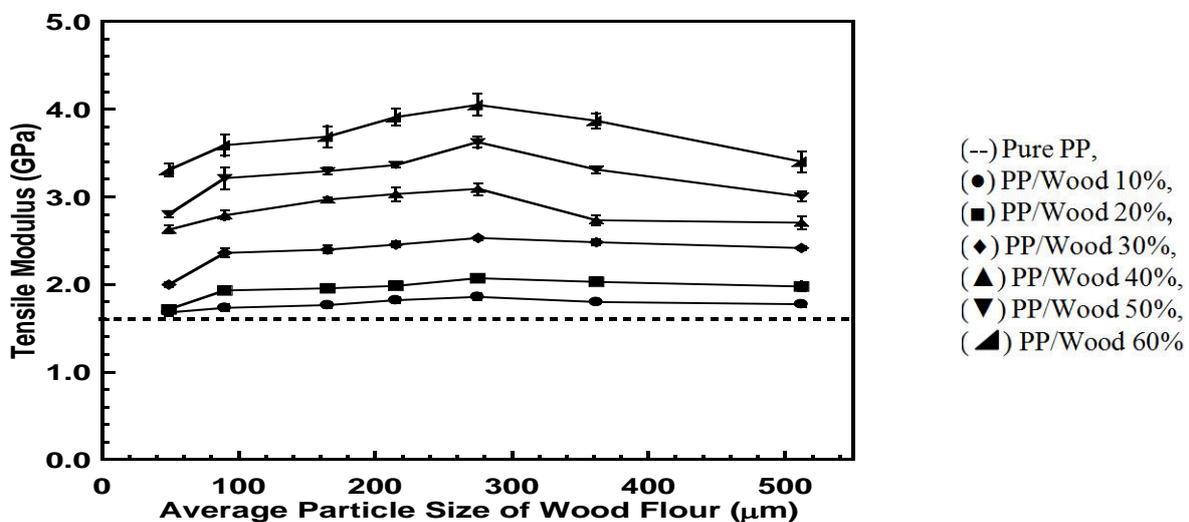


Fig. 4. Effect of wood flour particle size on tensile modulus of polypropylene/wood flour composites.

The flexural strength and tensile strength of wood flour filled polypropylene composites as a function of average wood flour particle sizes (50-500  $\mu\text{m}$ ) and wood flour contents (10-60wt%) are illustrated in Figure 5 and 6, respectively. These figures reveal that the flexural strength and tensile strength increased with increasing the average particle size in the range of 49-275  $\mu\text{m}$ . However, for the average particle sizes larger than 275  $\mu\text{m}$ , the flexural strength and tensile strength tend to decrease with increasing the particle size. This phenomenon exhibited the similar trend observed in the flexural modulus and tensile modulus. The reasons for these behaviors can be explained using the same reasons as previously mentioned. In addition, the flexural strength and tensile strength of wood flour filled polypropylene composites (at the same particle size) decreased with increasing wood flour contents. This result suggests that the interfacial adhesion between wood flour filler and polymer matrix was rather poor because of the discrepancy in polarity, which obstructs the stress transfer in wood composites [11, 20].

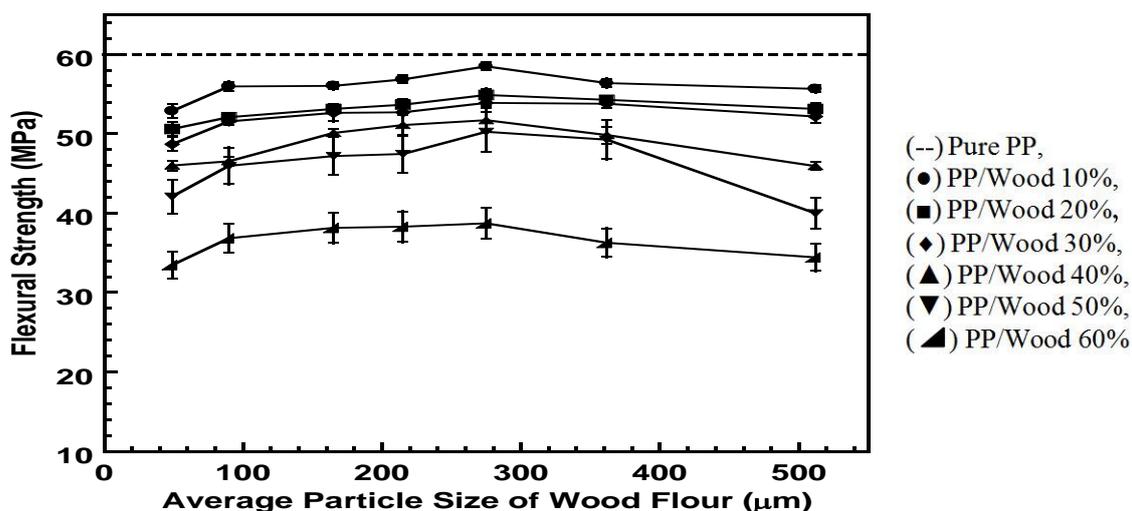


Fig. 5. Effect of wood flour particle size on flexural strength of polypropylene/wood flour composites.

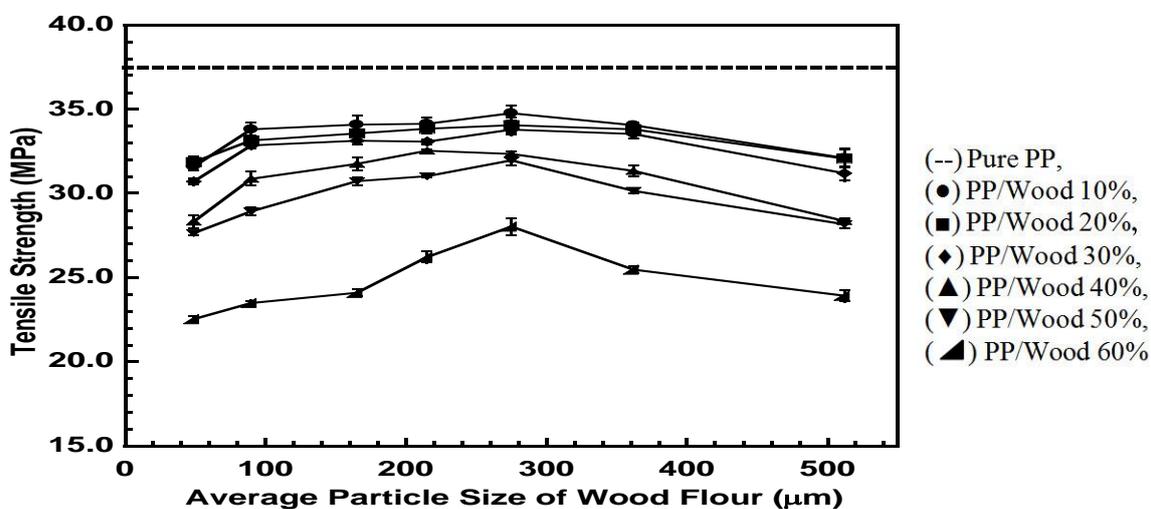


Fig. 6. Effect of wood flour particle size on tensile strength of polypropylene/wood flour composites.

### 3.3. Effects of wood flour contents on thermal stability

TGA thermograms of wood flour filled polypropylene composite at different filler contents are shown in Figure 7. The thermal degradation of composites reveals a three-stage degradation process, i.e. the range of 260-320°C, 360-430°C, and 400-470°C, which corresponds to the thermal degradation range of hemi-cellulose, lignin and polypropylene matrix, respectively. In nature, the thermal degradation temperature of wood flour is lower than that of polypropylene. Therefore, the composites rendered the decomposition properties with combining the characteristics of wood flour and polypropylene, which were typically observed in heterogeneous materials. Therefore, thermal stability and degradation temperature of composites (10-60wt%) decrease with increasing wood flour mass fraction in the range of 288-353°C while the degradation temperature of neat polypropylene was 409°C. Moreover, the percentage of residue at 800°C or the char yield, related to the material flame resistance, was enhanced with the presence of wood flour in the range of 2-12%. This could be because the ring structures of

cellulose and phenolic structures of lignin clearly render greater char formation than the linear structure of polypropylene. [21-22]

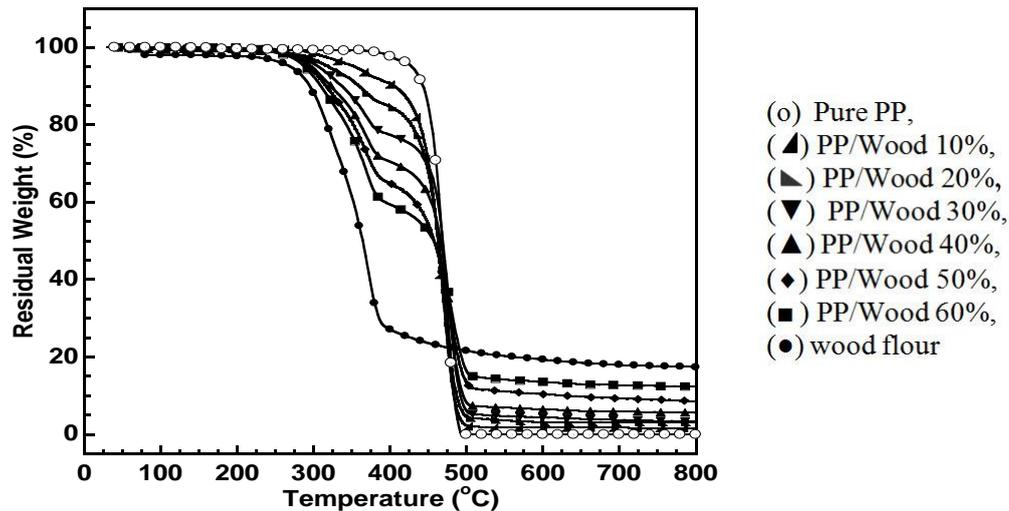


Fig. 7. TGA thermograms of polypropylene, woodflour and the composites at various wood flour contents.

### 3.4. Effects of MAPP coupling agent on thermal properties

Figure 8 presents DSC thermograms of pure polypropylene and wood flour-highly filled polypropylene composites at 60wt% with different MAPP coupling agent contents. It can be observed that all thermograms at various MAPP contents showed the same endothermic melting peak at approximately 165°C. The presence of MAPP in the composites seems to have no a major influence on the melting temperature of the composites as evidently shown in the thermograms due to small amount of in the wood composites. In addition, no significant effect on melting enthalpy, i.e. 43-46 J/g, was observed when MAPP was applied.

### 3.5. Effects of MAPP coupling agent on mechanical properties

Figure 9 exhibits the flexural modulus and tensile modulus of highly filled polypropylene composites at various coupling agent contents (0 to 20wt% based on wood flour). It could be observed that flexural modulus of the highly filled composites was slightly increased with MAPP at 5wt% based on wood flour, i.e. approximately 8 % increase, while the change in tensile modulus was not clearly observed. When MAPP content was beyond 5wt% based on wood flour, the flexural modulus of the composites was decreased. It is possibly attributed to the fact that the excess content of MAPP (i.e. at 10 to 20wt% based on wood flour) does not provide any further enhancement on the interfacial adhesion. Furthermore, the flexural modulus of MAPP is lower than that of polypropylene and wood flour. Therefore, the excess amount of MAPP is likely to cause the reduction of flexural modulus [23].

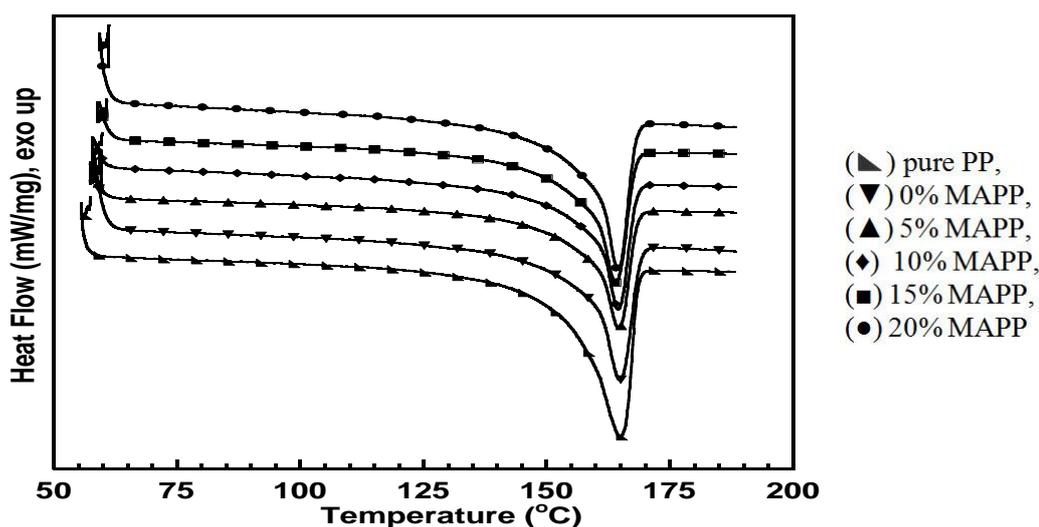


Fig. 8. DSC thermograms of the composites at various coupling agent contents.

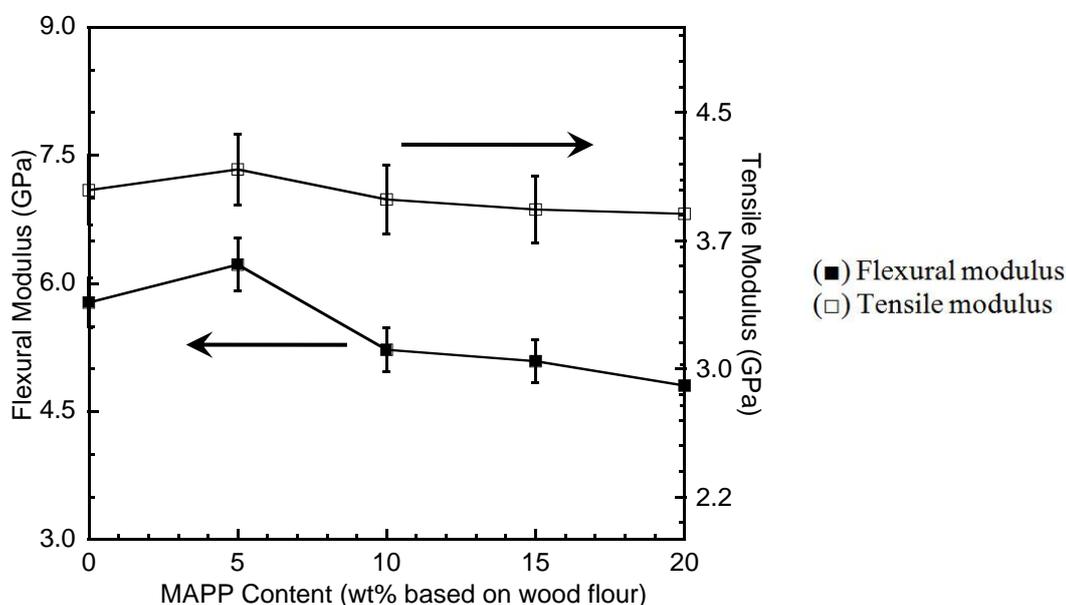


Fig. 9. Modulus of wood flour highly filled polypropylene composites at various coupling agent contents.

Figure 10 presents the effects of MAPP coupling agent on the flexural strength and tensile strength of highly filled polypropylene composites. At a low MAPP content, i.e. less than 5wt%, it could be observed that both flexural strength and tensile strength were increased. Beyond 5wt% of MAPP, the plateau region was reached. It is worth noting that, the flexural strength and tensile strength of the composites with 5wt% MAPP were approximately 110 % and 87% higher than those of the composites without MAPP, respectively. The strength improvement was attributed to the enhancement of interfacial interaction between wood flour and polypropylene matrix, which is associated with the ester linkages formed by chemical reaction of coupling agent and wood flour filler [12].

Figure 11 shows SEM micrographs of the fracture surfaces of MAPP-treated and non-treated composites. In Figure 11a, the fracture surface of the non-treated composites indicates the presence of interfacial gaps around wood flour filler, which exhibited relatively poor interfacial adhesion resulting in obtained poor flexural strength and tensile strength. In Figure 11b and 11c, the appearance of the fracture surface of the MAPP-treated composites exhibits strong interfacial adhesion between the filler

and the matrix, i.e. the coverage of polymer on wood flour particles and the tight interface. These results are in good accordance with the enhancement of mechanical properties as previously mentioned.

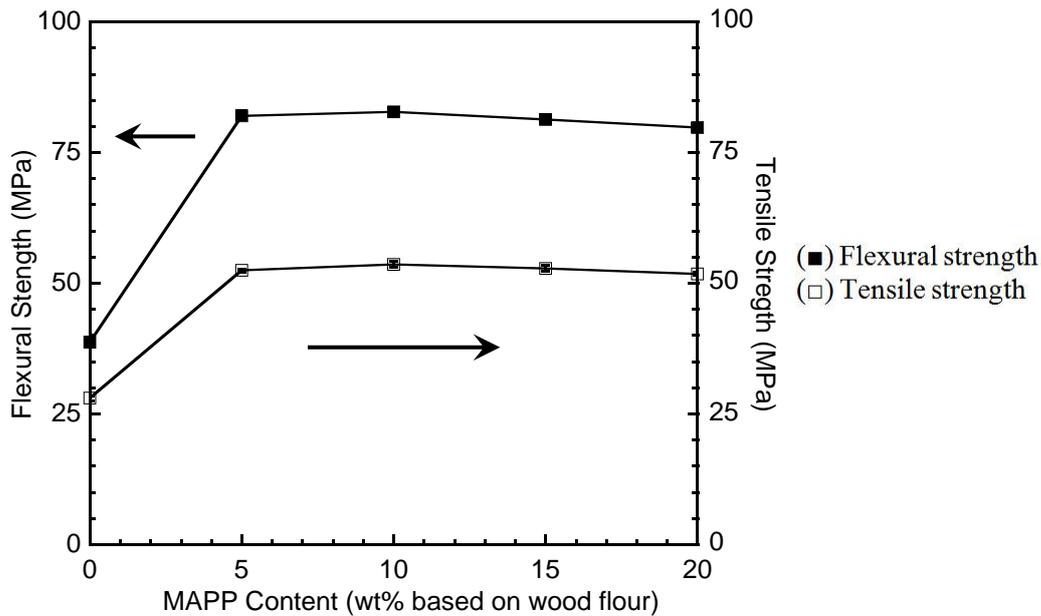


Fig. 10. Strength of wood flour highly filled polypropylene composites at various coupling agent contents.

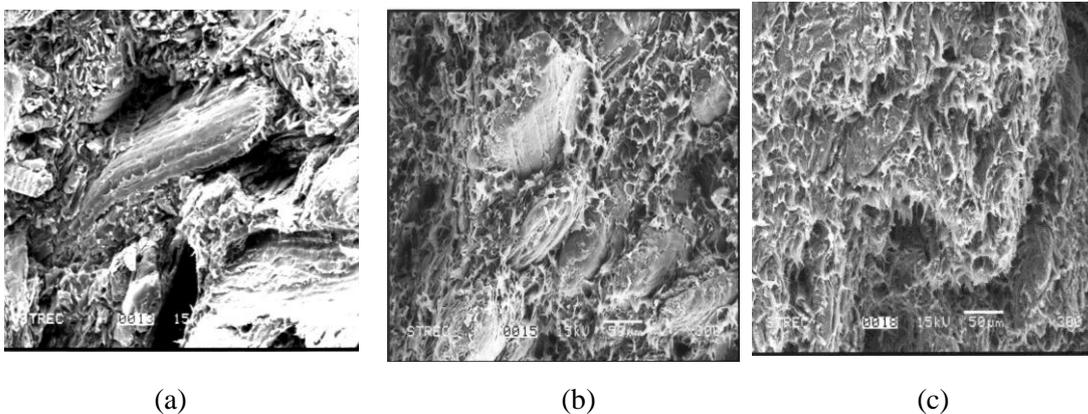


Fig. 11. SEM micrographs of the fracture surface of the composites at various coupling agent contents (a) MAPP 0%, (b) MAPP 5%, (c) MAPP 20%.

### 3.6. Effects of MAPP coupling agent on thermal stability

The corresponding degradation temperatures at 5 % weight loss of the highly filled composites are summarized in Table 2. It could be noticed that the thermal degradation temperature slightly increases with increasing the coupling agent content in the ranges from 291 to 297°C. The results suggest that the use of MAPP coupling agent can slightly improve the thermal stability of the composites. This implies that the interfacial adhesion enhancement could relate to the improvement of thermal stability [24-25].

Table 2. Degradation temperature of the composites at various coupling agent contents.

Coupling agent content (wt% based on wood flour )	Degradation temperature (°C)
0	288.0
5	291.0
10	293.0
15	295.0
20	297.0

### 3.7. The advantages of using high dosage of wood flour

Comparatively, the use of highly filled wood flour composites renders many advantages over the composites with low dosage. The main benefits are presented as follows:

(1) Low cost of raw material for composite manufacturing [26]

As previously mentioned, wood flour is waste from the furniture manufacturing; therefore, the more consumption of waste as filler in the polymeric composite means the greater reduction of cost for manufacturing.

(2) Environmental friendly aspect [3]

The use of higher wood flour contents leads to the higher consumption of rubber sawdust waste as filler in polymer composites will tremendously help solving the severe environmental problem.

## 4. Conclusion

In comparison of the composites processed from three types of polymer matrix, the wood flour composites based on HP644T (high melt flow rate) rendered higher mechanical properties than those of wood flour composites of HP648N and HP740H. It was found that the particle sizes and wood flour contents played a significant role in the mechanical properties of wood flour composites. The average particle size of wood flour that was suitable for improving the mechanical properties of the composite was in the range of 200-300  $\mu\text{m}$ . Moreover, the modulus increased, while the strength decreased with increasing wood flour contents. In the systems of highly filled composites treated with MAPP, the application of low content of MAPP (less than 5wt% based on wood flour) rendered the enhancement of interfacial adhesion, leading to the significant improvement of thermal and mechanical properties. However, when MAPP content was over 5wt%, the flexural modulus and tensile modulus of the composites were decreased.

## 5. Acknowledgements

The authors would like to thank for the financial supports from the 100<sup>th</sup> Anniversary of Chulalongkorn University Fund, the Ratchadapiseksomphot Endowment Fund of CU, and the Thailand Research Fund (TRF). Moreover, the Commission on Higher Education, Ministry of Education, is acknowledged for the financial support through the National Research University Funding Program. Furthermore, the author would like to thank HMC Polymer Co., Ltd. (Thailand) and Du Pont Co., Ltd. (Thailand) for their raw material supports.

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