Effects of Premixing Wood Flour with Compatibilizer with Different Characteristics on the Physical and Mechanical Properties of Wood/Plastic Composites

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Abstract

The addition of maleic anhydride (MA)-modified polypropylene (MAPP) as a compatibilizer increases mechanical and physical properties of wood/plastic composites (WPCs). It is thought that MA graft ratio (Gr), molecular weight (weight-average molecular weight [Mw]), and mixing methods of MAPP affect physical and mechanical properties of WPCs. However, evaluation, especially for mixing order, is not enough. The objective of this study was to investigate the effect of Gr and Mw of MAPP and its mixing methods on the mechanical and physical properties of WPCs. Two types of mixing methods were used: mixing the MAPP with the filler using a Henschel mixer before kneading with PP (premixing method) and mixing all of the materials at one time (one-step mixing method). The WPCs made with the premixing method had lower shear viscosity and higher flexural modulus than those made with the one-step mixing method. The particle size, moisture content, and surface morphology of fillers are thought to be more important in determining the shear viscosity of WPCs than the MAPP itself. The highest tensile, impact, and flexural strengths of WPCs were obtained in the WPC with premixing with low-Gr and high-Mw MAPP. It may be due to the effective reaction of the wood flour with MAPP and higher entanglement between the MAPP and PP. These results suggest that the premixing method could improve productivity and mechanical properties of WPCs.

Wood/plastic composites (WPCs) are produced from lignocellulose fillers, thermoplastic resin, and a small amount of additives. Lignocellulose fillers are biodegradable and carbon neutral (Kim and Pal 2010, Ndiaye and Tidjani 2012), and have excellent properties, such as higher specific strength and stiffness, and lower cost, compared with mineral fillers. However, WPCs have problems due to their poor mechanical properties, which are caused by the low compatibility between the polar (hydrophilic) lignocellulose fillers and the nonpolar (hydrophobic) thermoplastic resins (Stark and Berger 1997). Thus, it is important to overcome the low compatibility between the filler and the matrix by applying chemical or mechanical treatments (Ndiaye and Tidjani 2012).

Coupling treatment is a representative chemical treatment for WPCs. The first coupling treatment for WPCs was reported by Mayer (1968). At present, over 40 coupling agents have been used with WPCs (Lu et al. 2000). In the case of mechanical treatments, plasma and corona discharge treatments for lignocellulose fillers increase the tensile and bending properties of WPCs (Kim et al. 2013, Siham et al.

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2013). Many approaches toward improving the mechanical properties of WPCs have been attempted. Compatibilizers are generally used in WPC products. In the case of polypropylene (PP)-based WPCs, maleic anhydride (MA)-modified PP (MAPP) is the compatibilizer used most widely with WPCs (Kim et al. 2007).

The first MAPP treatment for WPCs was reported by Woodhams et al. (1984). Bera et al. (2010) reported that a WPC containing MAPP had superior mechanical properties to the corresponding WPC containing a silane coupling agent. The main mechanisms by which MAPP improves the compatibility of WPCs are as follows: (1) MA groups in the MAPP form chemical bonds, such as ester and hydrogen bonds, with hydrogen groups in the filler, and (2) PP chains in MAPP entangle with matrix chains (Rowell et al. 1997, Arbelaiz et al. 2005, Kim et al. 2007). The presence of an ester bond between MAPP and lignocellulose filler was reported by many researchers (Kim et al. 2007, Kim and Pal 2010). The details of chemical bonds between MAPP and cellulose, i.e., the presence of monoester and hydrogen bonds, were first reported by Niwa et al. (2017). Direct measurement of entanglements between MAPP and PP is difficult. However, it was reported that mixtures of MAPP and PP had different crystal structures depending on cocrystallization between the MAPP and PP (Harper et al. 2008). Researchers have suggested that the acid value or MA graft ratio (Gr, %) in the MAPP determines the number of bonds formed (Arbelaiz et al. 2005, Kim et al. 2007). It is also suggested that the molecular weight, such as the weight-average molecular weight (Mw) or number average molecular weight, of MAPP determined the degree of entanglement. The mechanical properties of WPCs containing different types of MAPP with different Gr and Mw have been reported (Kim et al. 2007). The results of that study showed that there are suitable combinations of Gr and Mw in MAPP to improve the mechanical properties of WPCs. In our previous research, we reported that a WPC had higher flexural modulus when it contained MAPP with high Mw and low acid value (Murayama et al. 2018). However, these studies suggested that some MAPPs do not attach to fillers because all materials are mixed at same time. At the pretreatment between fillers and MAPP before kneading with matrix, it is thought that the reactions between them are increased. Arbelaiz et al. (2005) tested the effect of mixing procedure on the strength properties of WPCs. They used two types of mixing: soaking a filler in MAPP before kneading it into the PP, and kneading all of the materials together at one time. They reported that the strength properties of WPCs were not significantly different by the mixing method. However, they used only one type of MAPP. In other words, the effect of mixing methods on mechanical and physical properties of WPCs containing different Gr and Mw of MAPP was not evaluated.

The objective of this study was to investigate the effect of two types of mixing methods and the different Gr and Mw of MAPP on mechanical and physical properties of WPCs. Two types of mixing methods were used: mixing the MAPP with the filler using a Henschel mixer before kneading with PP (premixing method) and mixing all of the materials at the same time (one-step mixing method). In a premixing step, a Henschel mixer, which is used to fabricate compounds in factories, was used (Tokihiro 2012). This mixer can heat materials by friction derived from collisions and thus may be suitable for premixing fillers and MAPP. However, the effect of premixing on characteristics, such as particle size and moisture content, of filler with MAPP was not reported. Therefore, the effect of premixing by Henschel mixer on characteristics of fillers was also evaluated.

Materials and Methods

Materials

Commercial wood flour (WF) with particle sizes under 150 μm was used as the lignocellulose filler. PP (Prime Polypro J107G; Prime Polymer) with a melt flow rate of 30 g/10 min (230°C/2.16 kg) was used as the matrix. Two types of MAPP, one with a Mw of 16,000 and Gr of 3.4 percent (MAPP1), and the other with a Mw of 40,000 and Gr of 0.9 percent (MAPP2), were used as compatibilizers.

Preparation of fillers

WF and MAPP were kneaded together using a high-speed Henschel mixer (SWV-20A; Kawata) until the temperature inside the mixer reached 190°C at a relative centrifugal force of 760.92 × g. The mixing ratio of WF/MAPP was fixed at 25/1 by weight. Also, kneaded WF without MAPP was prepared to evaluate the influence of the mixer. Kneaded WF itself, premixed WF containing MAPP1, and premixed WF containing MAPP2 were called PWF0, PWF1, and PWF2, respectively. They were stored at 20°C and 60 percent relative humidity (RH) for 2 weeks before evaluating the characteristics of the fillers.

Characteristics of fillers

A laser-diffraction particle size distribution analyzer (Partica LA-9502; Horiba) was used to measure the particle size distribution and median particle diameter of each filler. An infrared moisture determination balance (FD-720; Kett Electric Laboratory) was used to obtain the equilibrium moisture content. We poured each filler into a 100-mL graduated cylinder and then measured their volumes to calculate their bulk densities. The moisture content and bulk density of the fillers were measured with three replicates. A specific surface area and pore size distribution analyzer (Poremaster GT; Quantachrome) was used to measure the specific surface area of each filler.

WPC preparation

Figure 1 shows the process of fabricating the WPCs by the premixing method and one-step mixing method. The materials were kneaded with a twin screw extruder (AS30 m/m; Nakatani Machinery) and extruded into pellets. The temperature, screw speed, and WPC pellet output rate were 190°C, 80 rpm, and 4 kg/h, respectively. In this study, the WF/MAPP/PP ratio was fixed at 25/1/74 (wt%) for both WPC fabrication methods. Therefore, in the case of WPCs with PWF1 or PWF2, the filler/PP ratio was 26/74 (wt%). Also, in the condition of WPCs without MAPP, the WF (or PWF0)/PP ratio was fixed at 25/75 (wt%). The extruded pellets were oven dried at 80°C for 24 hours by an oven dryer with air circulation before injection molding. Dumbbell-shaped tensile specimens, measuring 60 mm in overall length and 2 mm in thickness, and with a narrow section measuring 15 mm in length and 5 mm in width, were molded at 200°C and 5.0 MPa by an injection molding machine (Babyplast 6/10P; Cronoplast). The rectangular

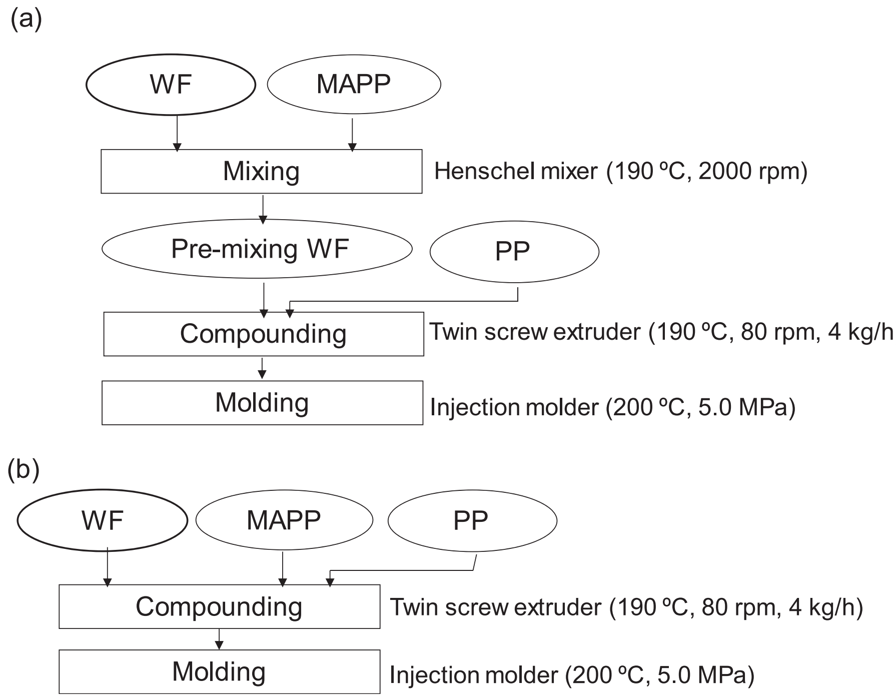

Figure 1.—Wood/plastic composites (WPCs) compounding and molding processes of (a) mixing the maleic anhydride (MA)-modified polypropylene (PP) (MAPP) with the commercial wood flour (WF) using a Henschel mixer before kneading with PP (premixing method) and (b) mixing all the materials at the same time (one-step mixing method).

specimens measuring 60 by 10 by 3 mm were prepared for bending and impact tests. They were molded at 200°C and 5.0 MPa. All specimens were stored at 20°C and 60 percent RH for 2 weeks before mechanical testing.

Rheological test

Shear viscosity of the WPCs was measured at five shear rates (100, 150, 224, 334, and 500 s^{-1}) by a capillary rheometer (LCR7001; Dynisco). The pellets were oven-dried at 80°C for 24 hours by an oven drier before the rheological test. The die diameter, melting temperature, sample amount, and premelting duration were 2 mm, 180°C, 9 g, and 300 seconds, respectively. Shear viscosity was measured in triplicate.

Mechanical tests

The tensile test was performed on a universal testing machine (AGS-5kNX; Shimadzu) with a loading speed of 10 mm/min. The length of the specimen between grips is 30 mm. Also, an impact tester (U-F Impact Tester; Ueshima Seisakusho) was used for an unnotched Izod impact test. The velocity of the striker at the moment of impact was 3.5 m/s and the impact energy was 2 J. In addition, we performed a three-point bending test with a universal testing machine (BT805; Yasuikikai) at a loading speed of 5 mm/min.

Tensile strength, nominal tensile strain at break, unnotched Izod impact strength, flexural strength, and flexural modulus were calculated according to the Japanese Industrial Standards JIS A 5741 (Japanese Standards Association [JSA] 2006) and JIS K 7110 (JSA 1999) protocol. Six specimens were used for each test. The Tukey-Kramer method and *t* test were used to evaluate the significant differences of mechanical tests.

Results and Discussion

Characteristics of fillers

Figure 2 shows the particle size distributions and median diameters of each filler. The particle sizes of PWF0, PWF1, and PWF2 were smaller than that of WF. It may be due to the influence of shear force on mixing in a Henschel mixer. PWF1 and PWF2 had larger particle sizes than PWF0. In this case, the frequency, around 20 μm, of PWF1 and PWF2 was lower than that of the other fillers. It is thought that >20-μm-size WF increased by coating or attaching MAPP on ~20-μm-size WF surface and ~20-μm-size WF aggregation via MAPP. PWF2 had larger particles than PWF1, with the median particle size of PWF1 being 4.2 μm larger than that of PWF0, and PWF2 being 17.9 μm larger than PWF1. Since MAPP2 has a higher Mw than MAPP1, MAPP2 is thought to have covered or attached to a larger area of the WF surface and made larger WF/MAPP aggregates. Table 1 shows the equilibrium moisture content at room temperature, bulk density, and specific surface area of each filler. The WF had a higher moisture content than the other fillers. The equilibrium moisture content of the wood decreased after heat treatment (Jämsä and Viitaniemi 2001) because of the chemical change caused by the decrease in hydroxyl groups. It is thought that the same effect occurs during premixing. Also, PWF1 had higher moisture content than PWF0 and PWF2. Because the Gr of MAPP1 to be used for PWF1 is high, it is thought that the water absorption of WF increased by increasing MA groups unreacted with WF. The bulk density of PWF0-2 was higher than that of WF. It may be due to the decreasing moisture content and particle size reduction (Fig. 2). However, PWF1 had a lower specific surface area than WF, despite the particle size decrease. These results suggest that low-Mw and high-Gr MAPP1 fills the internal voids of the WF. However,

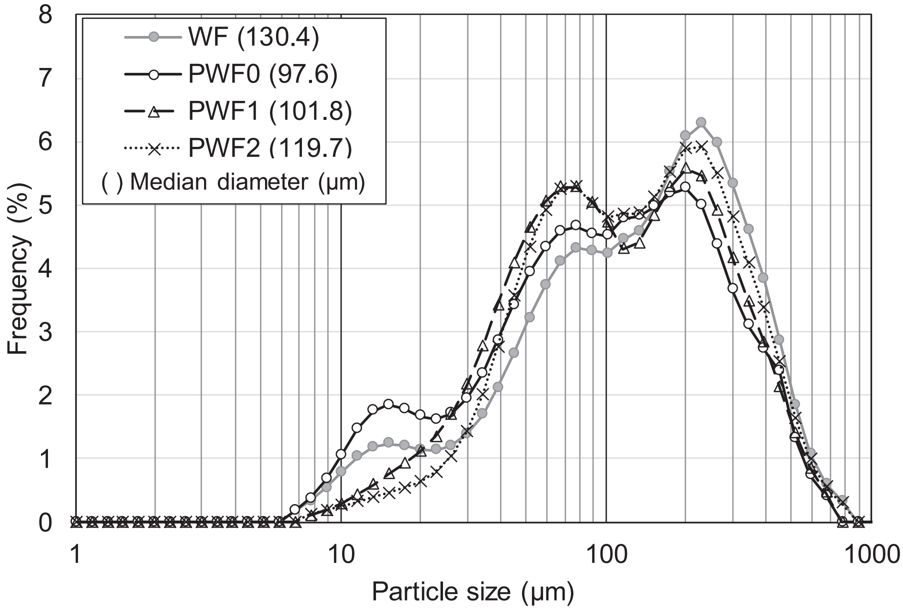

Figure 2.—Particle size distributions and median density of wood flour (WF), kneaded WF (PWF0), premixed WF containing a weight-average molecular weight (Mw) of 16,000 and maleic anhydride (MA) graft (Gr) of 3.4 percent MAPP (MAPP1) (PWF1), and premixed WF containing a Mw of 40,000 and Gr of 0.9 percent MAPP (MAPP2) (PWF2).

additional measurement of particle size and specific surface area of WF with other MAPP, such as low-Mw/low-Gr MAPP and high-Mw/high-Gr MAPP, will be required to prove this consideration.

As described, premixing with WF and MAPP affected particle size, moisture content, bulk density, and specific surface area of fillers. Thus, it is possible that the physical and mechanical properties of WPCs are affected by filler characteristics, and therefore it is necessary to discuss the effects of these filler characteristics on mechanical and physical properties of WPCs.

Rheological properties of WPCs

Figure 3 shows the shear viscosity as a function of shear rate for each type of WPC pellet. At all shear rates, the shear viscosity of the WPCs on the premixing method was lower than that on the one-step mixing method. Because the low viscosity makes it possible to mold complicated injection molding products and improve the productivity of composites, the premixing method is more efficient than the one-step mixing method. In the one-step mixing method, the shear viscosity of WPCs containing MAPP was lower than that without MAPP. This is consistent with previously reported results (Murayama et al. 2018). On the other hand, the shear viscosity of the WPCs with and without MAPP with the premixing method were almost the same. The particle sizes, moisture content, bulk density, and specific surface area of the fillers were affected by application of the premixing method (Table 1 and Fig. 2). Therefore, the characteristics of the fillers are thought to be more important in determining the shear viscosity of WPCs than the MAPP itself.

Table 1.—Moisture content, bulk density, and specific surface area of each filler.

	Mean (SD)		Specific
Code	Moisture content[b] (%)	Bulk density[b] (g/mL)	surface area[a] (m^2/g)
WF	11.2 (0.3) A	0.224 (0.003) D	11.00
PWF0	6.7 (0.0) C	0.239 (0.002) C	22.10
PWF1	7.8 (0.1) B	0.259 (0.001) A	9.01
PWF2	6.9 (0.3) C	0.250 (0.003) B	13.60

[a] Specific surface area was measured only once. Significant differences among groups (Tukey-Kramer multiple comparison test) are represented by different letters. Codes are defined in Figures 1 and 2.

[b] Moisture content and bulk density were measured with three replicates.

Mechanical properties of WPCs

Table 2 shows the mechanical properties of WPCs fabricated by different mixing methods and MAPP types.

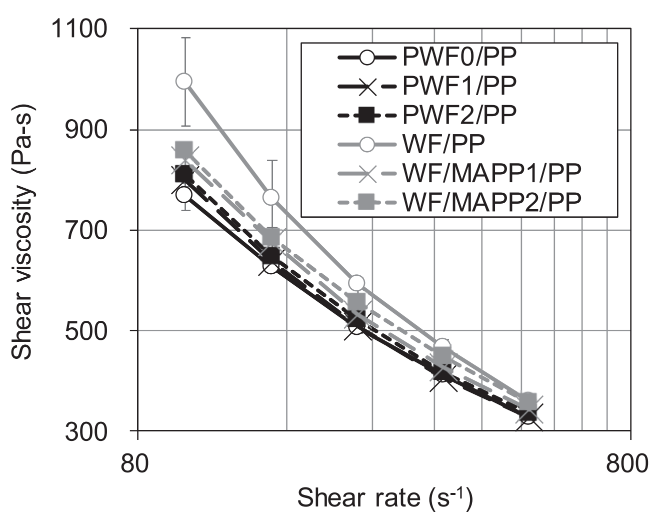

Figure 3.—Relationships between the shear rates and shear viscosity of the wood/plastic composites (WPC)s. Codes are defined in Figures 1 and 2.

Table 2.—Tensile, impact, and flexural properties of the wood/plastic composites (WPCs).[a,b]

Code	Mean (SD)				
	Tensile strength (MPa)	Nominal tensile strain at break (%)	Unnotched Izod impact strength (kJ/m^2)	Flexural strength (MPa)	Flexural modulus (GPa)
Premixing method WPC					
PWF0/PP	34.2 (0.5) C	4.3 (0.2) A	12.0 (1.5) B	57.0 (0.3) D	2.82 (0.08) A
PWF1/PP	40.2 (0.2) B	3.6 (0.1) B	13.6 (0.9) A	66.5 (1.0) C	2.71 (0.10) A
PWF2/PP	41.8 (0.2) A	3.9 (0.2) B	15.5 (1.3) A	71.0 (0.6) A	2.71 (0.09) A
One-step mixing method WPC					
WF/PP	34.2 (0.6) C	4.7 (0.3) A	11.8 (1.1) B	56.6 (0.5) D	2.57 (0.13) B
WF/MAPP1/PP	40.2 (0.4) B	3.9 (0.3) B	14.7 (0.5) A	68.5 (0.3) B	2.59 (0.12) B
WF/MAPP2/PP	39.9 (0.6) B	3.8 (0.5) B	13.5 (1.1) A	69.4 (0.9) B	2.66 (0.10) A

[a] Codes are defined in Figures 1 and 2.
[b] Significant differences among groups (Tukey-Kramer multiple comparison test) are represented by different letters.

In both mixing methods, the tensile strength of the WPCs containing MAPP was higher than that without MAPP. Though WF and PWF0 had different particle sizes, moisture content, bulk density, and specific surface area (Table 1 and Fig. 2), the tensile strengths of WPCs without MAPP were almost the same for both mixing methods. Thus, these differences did not affect tensile strength of WPCs in this study. Also, the tensile strengths of WPCs containing MAPP1 were almost same with both mixing methods. This result suggests that mixing methods did not affect the tensile strength of WPCs in the case of the addition of high-Gr MAPP. However, in the case of the tensile strength of the WPC containing MAPP2, the premixing method was higher than the one-step mixing method. The WPC containing MAPP2 in the premixing method had the highest tensile strength in this study. When the MAPP content was low, higher acid value or Gr resulted in WPCs with higher tensile strengths (Arbelaiz et al. 2005, Kim et al. 2007, Murayama et al. 2018). Low-Gr MAPP is considered to have the lower reactivity for hydroxyl groups of fillers than high-Gr MAPP. The tensile strength of WPCs containing MAPP2 shows that the premixing method yields high compatibility with its MAPP. On the other hand, it is suggested that high-Mw MAPP is able to diffuse deeper into the matrix and become entangled (Arbelaiz et al. 2005). MAPP2 had higher Mw than MAPP1. Therefore, the WPC containing MAPP2 in the premixing method had the highest tensile strength possibly due to the effective reaction between the WF and MAPP and higher entanglement between the MAPP and PP. In the premixing method, the tensile strength of WPCs containing MAPP2 was higher than those containing MAPP1. It is thought that the effect of Gr is low under premixing. On the other hand, high-Mw MAPP2 may have strong entanglement with PP. In one-step mixing, the tensile strength of WPC containing MAPP1 did not differ from that of WPC containing MAPP2. In the case of MAPP1, high reactivity to WF might contribute to the tensile strength of WPCs. On the other hand, it is thought that high entanglement between PP and MAPP2 contributes to the tensile strength of WPCs. The addition of MAPP decreased the nominal tensile strain at break of the WPCs under all conditions. The interface between fillers and the PP matrix became more brittle with the addition of MAPP, thereby decreasing the strain at break. The WPC without MAPP yielded by the premixing and one-step mixing methods had almost the same nominal tensile strain at break. The nominal tensile strain at break of the WPCs containing MAPP was not affected by the characteristics or the mixing methods of MAPP. We obtained different interfacial interaction between the WF and MAPP2 for the two mixing methods because the tensile strengths of the WPCs were different. However, this interaction did not affect nominal tensile strain at break. The impact strength of WPCs without MAPP was lower than those containing MAPP. The impact strength of WPCs without MAPP was almost the same for both mixing methods. Though the impact strength of WPCs containing MAPP was not significantly different by MAPP types or mixing methods, the highest impact strength was observed on WPCs containing MAPP2 fabricated by the premixing method. The trend of impact and tensile strength was almost the same. The flexural strength of the WPCs increased with the addition of MAPP. Similar to the cases of the tensile and impact strengths, the flexural strengths of the WPCs without MAPP were almost all the same, regardless of the mixing method. The WPC containing MAPP1 in the premixing method had lower flexural strengths than that in the one-step mixing method. As with tensile and impact strengths, the highest flexural strength in this study was achieved by applying the premixing method to fabricate WPCs with MAPP2. Though the flexural modulus of the WPCs containing MAPP2 was not significantly different by mixing methods, the premixing method yielded WPCs with a higher flexural modulus than the one-step mixing method under all conditions. Therefore, the flexural modulus of WPCs may be affected by the particle size, moisture content, bulk density, and specific surface area of the fillers (Table 1 and Fig. 2).

The moisture content of the WF was 4.5 percent higher than that of PWF0. Therefore, we confirmed the effects of the mixing methods on the mechanical properties of the WPCs, except for the effect of the moisture content of the fillers, by conducting an additional experiment to evaluate the mechanical properties of WPCs with WF and PWF0 at 0 percent moisture content. The WF and PWF0 were dried by an oven drier at 105°C/24 h. Then, the WPCs containing WF and PWF0 at 0 percent moisture content were prepared by the ‘‘2.4 WPC preparation’’ process. The results are summarized in Table 3. The WPCs with WF and PWF0 at 0 percent moisture content had almost identical strength properties, and these properties were also almost the same as

Table 3.—Tensile, impact, and flexural properties of wood/plastic composites (WPCs) with WF and PWF0 at 0 percent moisture content.[a,b]

Code	Mean (SD)				
	Tensile strength (MPa)	Nominal tensile strain at break (%)	Unnotched Izod impact strength (kJ/m^2)	Flexural strength (MPa)	Flexural modulus (GPa)
Premixing method WPC (0% moisture content)					
PWF0/PP	33.8 (0.2)	4.4 (0.4)	11.8 (0.8)	56.7 (0.3)	2.98 (0.04)
One-step mixing method WPC (0% moisture content)					
WF/PP	33.5 (0.7)	4.0 (0.2)	12.0 (0.9)	57.3 (0.2)	2.90 (0.03)*

[a] Codes are defined in Figures 1 and 2.
[b] * = Significantly different at $P < 0.05$ from PWF0/PP at moisture content 0%.

those of the specimens with room temperature moisture content (Table 2). On the other hand, the flexural modulus of the WPC with PWF0 at 0 percent moisture content was higher than that at room temperature moisture content. At 0 percent moisture content, the WPC with PWF0 had a marginally higher flexural modulus than the WPC with WF. However, the increase of the flexural modulus of WPCs with 0 percent moisture content fillers was smaller than that with room temperature moisture content fillers. Specifically, at 0 percent moisture content, the WPC with PWF0 was 0.08 GPa higher than that with WF. Also, at room temperature moisture content, the WPC with PWF0 was 0.25 GPa higher than that with WF. Therefore, the change in moisture content caused by premixing the WF appears to have a significant effect on the flexural modulus of the WPCs. However, some other properties of fillers, such as chemical component before and after premixing, should be measured to understand the improvement mechanism on the flexural modulus of WPCs.

WPCs containing MAPP2 in the premixing method had the highest strength properties in this study. It may be due to the effective reaction between the WF and MAPP by premixing and higher entanglement between the MAPP and PP as high Mw of MAPP. However, the effect of Mw and Gr on physical and mechanical properties of WPCs could not be evaluated independently, because only two types of MAPP (low Mw/high Gr and high Mw/low Gr) were used in this study. To evaluate the effect of Mw and Gr independently, at least two more different MAPPs (high Mw/high Gr and low Mw/low Gr) should be prepared.

Conclusions

In this study, we investigated the effect of different mixing methods (premixing and one-step) and different Gr and Mw of MAPP on mechanical and physical properties of WPCs. The main results of this study are as follows:

1. The shear viscosity of the WPCs with the premixing method was lower than that with the one-step mixing method, regardless of MAPP types. Also, the shear viscosity of the WPCs with and without MAPP with the premixing method were almost the same. The particle size, moisture content, bulk density, and specific surface area of the fillers were changed by the premixing. Therefore, the changes of the fillers are thought to be more important in determining the shear viscosity of WPCs than the MAPP itself.
2. The highest strength properties of WPCs were obtained in the WPC with the premixing method with low Gr and high Mw. It may be due to the effective reaction of the WF with MAPP and higher entanglement between the MAPP and PP.
3. The decreases of moisture content caused by premixing appears to have an increase on the flexural modulus of the WPCs.

These results suggest that the premixing method could improve productivity and mechanical properties of WPCs.

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