The Effects of Different Types of Maleic Anhydride-Modified Polypropylene on the Physical and Mechanical Properties of Polypropylene-based Wood/Plastic Composites

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Abstract: Maleic anhydride-modified polypropylene (MAPP) is a compatibilizer used to improve the physical and mechanical properties of many wood/plastic composites (WPCs). The properties of WPCs containing MAPP differ according to the characteristics of the specific MAPP that it is used. In this study, the physical and mechanical properties, including shear viscosity, of polypropylene-based WPCs containing different types of MAPP were investigated before and after water absorption. The shear viscosity of MAPP increased with increasing molecular weight, but remained nearly constant for WPCs containing different types of MAPP. In dry conditions, the strongest WPC contained the MAPP with the highest acid value. The highest flexural modulus was observed with the WPC containing the MAPP with the highest molecular weight. In wet conditions, the WPC exhibiting the best mechanical properties contained a MAPP with a molecular weight of 58,000.

Key words: wood/plastic composite, maleic anhydride-modified polypropylene, mechanical property, rheological property, water absorption

Introduction

A wood/plastic composite (WPC) is a material composed of a matrix of thermoplastic resin, a lignocellulosic filler, and a small amount of compatibilizer or other additives. WPC is an environmentally friendly material because it can be fabricated from wood waste and recycled thermoplastic resin, thereby reducing the proportion of petroleum-derived thermoplastics used in finished products.[1-6] Compared with composites incorporating an inorganic filler, WPC boasts several advantages, including high specific strength and modulus, low density, and low friction during compounding. WPCs have higher water and decay resistance than solid wood.[1,2] Generally, WPCs are used as exterior decking and as door and automobile composite parts. Recently, the development of injection moulding processes for WPCs has extended their use to include electrical casings, packaging for daily necessities, and engineering applications.[1-3,7-9]

Many researchers have focused on the effects of compatibilizers in WPCs. These materials improve the compatibility between hydrophilic lignocellulosic fillers and the hydrophobic thermoplastic resin.[1,2,10-12] Generally, maleic anhydride-modified polypropylene (MAPP) is used in polypropylene (PP)-based WPCs. PP is a commodity thermoplastic resin with high water tolerance and chemical resistance compared with other thermoplastic resins. The hydroxyl groups of the lignocellulosic filler react with the maleic anhydride (MA) groups of MAPP to form ester and hydrogen bonds.[1,2,4,13,14] The evidence of ester bonds and hydrogen bonds between MAPP and lignocellulosic filler was difficult to determine, however, the evidence of ester bonds and hydrogen bonds was confirmed from the compounds of microcrystalline cellulose and MAPP by spectroscopy and gel (swollen)-state NMR method.[15] In addition, the PP matrix can entangle with MAPP chains. At the mixture of PP and MAPP, it is reported that cocrystal was formed between PP and MAPP.[16] The acid value/MA graft (%) and the molecular weight of MAPP determine the number of bonds formed and the degree of entanglement.[4,14] In the case of WPCs, MAPP can physically cross-link the porous lignocellulosic filler.[1] Also, the inclusion of MAPP in the PP matrix, leads to increased nucleation on the surface of the wood and in the bulk.[17] MAPP can increase the tensile strength and modulus of the final WPC compared with other compatibilizers. Several reports have stated that the mechanical properties of the final WPC are strongly influenced by the acid value/MA graft (%) and molecular weight of MAPP.[1,4,14,18] However, the physical and mechanical properties of WPCs containing different types of MAPP, for example, different acid values and molecular weights, have not been sufficiently evaluated. While rheological characterisation yields information critical for WPC production and further developments of WPC applications,[3,19,20] examinations of the relationship between WPCs and the rheological properties of MAPP have not been reported. The mechanical properties of a WPC after water absorption are important for applications that commonly experience wet conditions, such as outdoor decking material. Generally, the poor water resistance of lignocellulosic filler has undesirable effects on the mechanical properties of wet WPCs.[9] However, the mechanical properties of WPCs containing various types of MAPP in wet conditions have not been sufficiently evaluated. The objective of this study is to evaluate the effects of different types of MAPP on the physical and mechanical properties of WPCs.

Experimental

Materials

Commercial wood flour (WF) with a particle size of under 150 μm was used as lignocellulosic filler. PP (Prime Polypro J107G, Prime Polymer) with a melt flow rate of 30 g/10 min (230 °C/2.16 kg) and a density of 0.9 g/cm^3 was used as the matrix material. Three types of MAPP, named MAPP-A, MAPP-B and MAPP-C, were used as compatibilizers. Table 1 shows the relevant characteristics of the compatibilizers.

Sample preparation

Table 2 shows the WPC formulations. The ingredients were melt-blended using a twin screw extruder (AS30 m/m, Nakatani Machinery) and extruded to pellets. In this study, the WPCs were low WF contents compared to the general WPC products which contain 50-60 wt% WF. The melting temperature was 190 °C and the screw speed was 80 rpm. The pellet output was approximately 4 kg/h. WPC-0 corresponds to WPC without MAPP. Pellets of WPC and PP were oven-dried at 80 °C for 24 h by an oven dryer with air circulation. The pellets were moulded into two types of specimen using an injection moulding machine (Babyplast 6/10P, Cronoplast). Tensile test specimens, measuring 60 mm in overall length and 2 mm in thickness with a narrow section measuring 15 mm in length and 3 mm in width, were moulded at 200 °C and 25 bar. Rectangular specimens, used for bending and impact tests, measured 60 × 10 × 3 mm and were moulded at 200 °C and 50 bar. All specimens were stored at 20 °C and 60% RH for two weeks prior to mechanical testing. These conditions are herein defined as dry conditions.

Water absorption tests

Specimens were submerged in hot water at 70 °C, conditioned to a constant weight (for 524 h), and submerged in room-temperature water for 1 h. These conditions are herein defined as wet conditions. Specimens were continuously weighed during the water absorption tests. Water absorption at time t (WA_t) was calculated according to the following equation,

$$WA_t = \frac{(W_t - W_0)}{W_0} \times 100\ (\%), \qquad (1)$$

where W_t is the specimen weight at time t and W_0 is the initial specimen weight.

Rheological tests

Shear viscosity measurements were carried out with a capillary rheometer (LCR7001, Dynisco) for PP, pellets of WPCs, and different types of MAPP. The values of shear viscosity of PP and the pellets were measured using a 2-mm-diameter die, while a 1-mm-diameter die was used with MAPP. The melting temperature, sample amount, and the pre-melting duration were 180 ˚C, 9 g, and 300 s, respectively. Shear viscosity was measured in triplicate at five shear rates (100, 150, 224, 334, and 500 s^{-1}).

Mechanical tests

A universal testing machine (AGS-5kNX, Shimazu) was used for tensile tests with a loading speed of 10 mm/min. The tensile strength (σ_t) and nominal tensile strain at break (ε_t) were calculated using the following equations,

$$\sigma_t = \frac{F_t}{\mathrm{A}} \ (\mathrm{MPa}) \qquad (2)$$

$$\varepsilon_t = \frac{\Delta L_t}{\mathrm{L}_t} \ (\%) \qquad (3)$$

where F_t is the maximum load of the tensile test, A is the cross section, ΔL_t is the displacement from initiation to break, and L_t is the length of the specimen between grips (30 mm).

Three-point bending tests were performed with a universal testing machine (BT805, Yasuikikai) at a loading speed of 5 mm/min with a span of 48 mm. Flexural strength (σ_f) and modulus (E_f) were calculated as follows,

$$\sigma_f = \frac{3F_f L_f}{2bh^2} \text{ (MPa)} \qquad (4)$$

$$E_f = \frac{L_f^3 m_f}{4bh^3} \times 10^{-3} \text{ (GPa)} \qquad (5)$$

where F_f is the maximum load of the bending test, L_f is the support span, b is the specimen width, h is the thickness of the specimen, and m_f is the slope of the initial straight-line portion of the load deflection.

Unnotched Izod impact tests were conducted using an impact tester (U-F Impact Tester, Ueshima Seisakusho) at 3.5 m/s. The impact energy was 2 J. The unnotched Izod impact strength (a_{iN}) was calculated using the following equation,

$$a_{iN} = \frac{W}{bh} \times 10^3 \text{ (kJ/m}^2\text{)} \qquad (6)$$

where W is the impact energy absorbed by the specimen.

Tensile, bending, and impact tests were performed in dry conditions. Tensile and bending tests were also performed in wet conditions. Six specimens of each formulation were used in each test.

Morphological test

The broken surface of tensile specimens was observed by scanning electron microscopy (SEM, JSM-6510LV2, Jeol) operating at 15 kV. Specimens were coated with platinum prior to measurements.

Results and Discussion

Rheological properties

Figure 1 shows the shear viscosity as a function of shear rate for each sample and formulation. The data in Figure 1a show that the shear viscosity of the WPCs was higher than that of PP at all shear rates. At low shear rates, the values of shear viscosity of WPCs containing MAPP were lower than those of WPC-0 without MAPP. The same trend was observed for the shear viscosity of WPC containing maleic anhydride-modified polyethylene with 30% WF.[20] It is thought that the addition of maleic anhydride-modified thermoplastic resin is effective for WF dispersion and WF orientation at low shear rates. However, the differences between the values of shear viscosity of WPCs containing MAPP and WPC-0 gradually decreased with increasing shear rate, disappearing altogether at a shear rate of 500 s^{-1}. This suggests that the influence of MAPP addition on shear viscosity decreases at higher shear rates. The shear viscosity of MAPP-C was higher than those of other formulations (Figure 1b). For example, at a shear rate of 224 s^{-1}, the values of shear viscosity of MAPP-C, MAPP-B, and MAPP-A were 209.6, 17.7, and 16.8 Pa s, respectively. Note, however, that at all shear rates, the shear viscosity of WPCs remained nearly the same regardless of the type of MAPP incorporated. For example, at a shear rate of 224 s^{-1}, the values of shear viscosity of all MAPP-containing WPCs ranged from 543.2 to 556 Pa s. Thus, the shear viscosity of a given WPC samples was independent of the type of MAPP used. This may have been due to the relatively small amount of MAPP in each formulation.

Mechanical properties under dry conditions

Table 3 shows the mechanical properties of WPCs and PP under dry conditions. The tensile strengths of WPCs showed increases of 4% to 27% over that of PP. Likewise, flexural strength increased by 2% to 33% and flexural modulus increased by 63% to 75%. Several researchers have reported increases in tensile strength, flexural strength and flexural modulus in PP-based composites incorporating a lignocellulosic filler.[21,22,23] However, some researchers have fabricated PP-based composites with lower tensile strengths than PP.[21,22] It is thought that the higher tensile strength of WPC-0 compared with that of neat PP is due to the high dispersibility and physical cross-linking of WF in the matrix. In contrast, the nominal tensile strain at break of the WPCs was 67% to 78% lower than that of PP. This phenomenon is in agreement with previously published results.[5,8,21,22,24]

Similarly, the unnotched impact strength of the WPCs decreased by 65% to 77% compared with that of PP.[21,22,25] The decreases in strain at break and impact strength are likely due to crack development at the stress concentration point, initiated at the filler/matrix interface or by filler ends and/or defects. The tensile strengths of WPC-A, WPC-B and WPC-C were significantly higher than that of WPC-0. Generally, the tensile strengths of PP-based composites containing MAPP are higher than those of PP-based composites without MAPP.[4,14,22,26] However, excessive amounts of MAPP decrease the tensile strength of a WPC because the proportion of filler surface that is available to react with the MAPP is limited.[18] The relative amounts of MAPP employed in this study were not excessive. The flexural strengths of WPC-A, WPC-B and WPC-C were significantly higher than that of WPC-0. The flexural strength of PP-based composites with MAPP is also generally higher than that of PP-based composites without MAPP.[4,14,22,26] Of the tested composites, WPC-A yielded the greatest tensile strength, flexural strength and impact strength, followed by WPC-B and WPC-C. MAPP-A, which contained WPC-A, had the highest acid value (Table 1). This suggests that the addition of MAPPs with high acid values is effective for increasing the final composite strength. A similar trend was reported for short flax fibre bundle/PP composites containing either high- or low-acid-value MAPP.[4] Figure 2 shows SEM images of the fracture surface of WPCs tensile specimens under dry conditions. Large gaps were observed in the break surfaces of WPC-0, WPC-B and WPC-C, but not WPC-A. These findings suggest that WPC-A has the highest degree of interfacial bonding between MAPP and WF. MAPP-B and MAPP-C have the same acid value (Table 1). Therefore, the higher tensile strength, flexural strength and impact strength of WPC-B compared with WPC-C may be due to the effects of molecular weight. The nominal tensile strains at break of WPC-B and WPC-C were lower than that of WPC-0. The flexural modulus of WPC-C was significantly higher than that of WPC-0. In this study, these findings suggest that the influence of entanglement between MAPP and PP is higher compared with the interfacial bonding, such as ester bonds and hydrogen bonds between MAPP and WF on decreasing the nominal tensile strain at break and increasing the flexural modulus. This conclusion is supported by the gaps observed in the SEM images of WPC-B and WPC-C (Figure 2c, d). Also, it is thought that differences in crystallinity index of WPCs containing MAPP and WPC without MAPP[17] are one of the reasons of differences on mechanical properties of WPCs. However, the difference of crystallinity index between WPCs containing different types of MAPP was not evaluated. This is a future topic of discussion.

Mechanical properties after water absorption

Table 4 shows that the mechanical properties of WPCs were equal to or lower than those of PP after water absorption. Figure 3 shows that the water absorption of PP remained the same from 0 to 524 h in water at 70°C. In contrast, the WPCs absorbed water gradually under these same conditions, with 5%–6% absorption after 524 h. Water absorption in plastic composites typically occurs through one or more of the following mechanisms: (1) the diffusion of water molecules into microgaps between polymer chains, (2) transport of water through microcracks in the matrix, formed during the compounding process, and (3) capillary transport into gaps and flaws at the filler/polymer interface.[9] In addition, water can be absorbed into the lignocellulosic filler. Both the strength and the stiffness of natural fibre composites are typically lower after water absorption due to a reduction in fibre stiffness and the development of shear stress at the fibre/matrix interface due to fibre swelling.[8] In contrast, we consider that the mechanical properties of MAPP-containing WPCs are degraded by the hydrolysis of ester bonds and a decrease in the degree of hydrogen bonding between the MAPP compatibilizer and the WF. This hypothesis is supported by the gaps observed in the SEM images of WPC-B, which were only observed in wet conditions (Figure 4). However, for the evaluation of the decrease of chemical bondings between MAPP and WF, it is necessary to additional study. In addition, the flexural strength of all MAPP-containing WPCs was significantly higher than that of WPC-0. However, the increases on tensile strength and flexural strength of WPCs containing different types of MAPP under wet conditions were smaller than these under dry conditions (Table 3).Both tensile strength and flexural strength were very nearly independent of the type of MAPP used. The nominal tensile strains at break of WPC-B and WPC-C were significantly lower than that of WPC-0. The flexural moduli of WPC-B and WPC-C were significantly higher than that of WPC-0. These results are likely due to differences in molecular weight, because probably acid value does not affect on these mechanical properties in wet conditions due to internal debonding between MAPP and WF during water absorption tests. The initial rate of water absorption varied among the WPCs. However, WPC-B was the only composite to show a lower degree of water absorption at constant weight (after 524 h) than WPC-0 (Figure 3). These data suggest that, in the current study, the addition of MAPP-B, which has weight-averaged molecular weight of 58,000, yields a composite with the best physical and mechanical properties under wet conditions.

The mechanical properties of WPCs under dry and wet conditions were compared. The retention of the mechanical properties (R_m) is defined as:

$$R_m = \frac{X_w}{X_d} \times 100\ (\%) \qquad (7)$$

where X_w is the tensile strength, the nominal tensile strain at break, the flexural strength, or the flexural modulus under wet conditions and X_d represents those same variables under dry conditions.

Table 5 shows the retention of the mechanical properties of WPCs. WPCs which had high retentions of tensile strength and flexural strength had low tensile strength and flexural strength under dry conditions (Table 3). The degree of interfacial bonding between WF and MAPP appears to be independent of the type of MAPP used. The retention of the nominal tensile strain at break increased following water absorption. It is thought that hot water absorption softens the WF and leads to interfacial debonding between MAPP and WF. The retention of flexural modulus remained nearly the same across all WPC samples, suggesting that interfacial bonding between MAPP and WF does not affect the flexural modulus of WPCs. In this study, the effect of MAPP on mechanical properties and retention of mechanical properties of WPCs after water absorption were evaluated. However, it is thought that the difference of water absorption on WF is affected to mechanical properties of WPCs. There is a matter for future investigation.

Conclusions

The rheological, physical and mechanical properties of WPCs with and without different types of MAPP were evaluated in wet and dry conditions. The main results of this study were as follows:

1. The shear viscosity of WPCs was effectively independent of the type of MAPP used. In different types of MAPP, the highest shear viscosity was observed with the highest-molecular-weight MAPP. The shear viscosity of MAPP was unrelated to the shear viscosity of the WPC formulation.

2. Under dry conditions, the WPC incorporating the MAPP with the highest acid value showed the

highest tensile, flexural and impact strengths. The WPC incorporating the MAPP with the highest molecular weight showed the highest flexural modulus.

3. Under wet conditions, the WPC containing MAPP with a molecular weight of 58,000 exhibited significantly higher tensile strength, flexural strength and flexural modulus, as well as lower water absorption, than the WPC without MAPP.

4. When the tensile and flexural strengths of WPCs were higher under dry conditions, the retention of those properties after water absorption was lower. The retention of the nominal tensile strain at break increased for all WPCs, while the retention of flexural modulus remained constant.

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Table and Figure

Table 1. Samples and the characteristics of compatibilizers.

Sample	Color	Weight average molecular weight	Acid value (mgKOH/g)	Melting point (˚C)
MAPP-A	Light-yellow-white	36,000	42	167
MAPP-B	Yellow	58,000	20	166
MAPP-C	Light-yellow-white	153,000	21	168

These are nominal values by the manufacturer.

Table 2. Reference codes and composition of WPC formulations.

	Amount of substance (wt%)				
Code	WF	PP	MAPP-A	MAPP-B	MAPP-C
WPC-0	25	75	0	0	0
WPC-A	25	74	1	0	0
WPC-B	25	74	0	1	0
WPC-C	25	74	0	0	1
PP	0	100	0	0	0

WF: Wood flour, PP: Polypropylene. Characteristics of MAPP-A to C are referred to in Table 1.

Table 3. The mechanical properties of WPCs and PP under dry conditions.

Code	Tensile strength (MPa)		Nominal tensile strain at break (%)		Flexural strength (MPa)		Flexural modulus (GPa)		Unnotched Izod impact strength (kJ/m^2)	
WPC-0	35.6	(1.1)	4.1	(0.3)	56.6	(0.5)	2.57	(0.13)	11.8	(1.1)
WPC-A	43.6*	(1.0)	4.2	(0.3)	73.7*	(0.6)	2.59	(0.10)	16.6*	(0.4)
WPC-B	41.0*	(0.5)	3.5*	(0.2)	69.4*	(0.9)	2.66	(0.10)	13.5*	(1.1)
WPC-C	37.6*	(1.0)	2.8*	(0.2)	62.8*	(0.4)	2.77*	(0.12)	11.2	(0.6)
PP	34.2*	(0.7)	12.7*	(1.2)	55.6*	(1.1)	1.58*	(0.06)	47.7*	(1.4)

The standard deviation is shown in parentheses. Single asterisks indicate a significant difference of $p < 0.05$ from WPC-0. Codes are referred to in Table 2.

Table 4. The mechanical properties of WPCs and PP after water absorption.

Code	Tensile strength (MPa)	Nominal tensile strain at break (%)	Flexural strength (MPa)	Flexural modulus (GPa)
WPC-0	31.7 (1.1)	4.6 (0.3)	47.6 (0.4)	1.38 (0.05)
WPC-A	33.1* (0.2)	4.5 (0.2)	50.0* (0.6)	1.43 (0.08)
WPC-B	33.4* (0.9)	4.2* (0.4)	50.2* (0.3)	1.46* (0.04)
WPC-C	32.3 (0.2)	4.4* (0.2)	49.1* (0.6)	1.50* (0.06)
PP	36.1* (0.3)	9.0* (1.0)	57.8* (1.5)	1.48* (0.05)

The standard deviation is shown in parentheses. Single asterisks indicate a significant difference of $p < 0.05$ from WPC-0. Codes are referred to in Table 2.

Table 5. The retention of mechanical properties by WPCs following water absorption.

Code	Tensile strength	Nominal tensile strain at break	Flexural strength	Flexural modulus
WPC-0	89%	112%	84%	54%
WPC-A	76%	107%	68%	55%
WPC-B	81%	118%	72%	55%
WPC-C	86%	154%	78%	54%

Codes are referred to in Table 2.

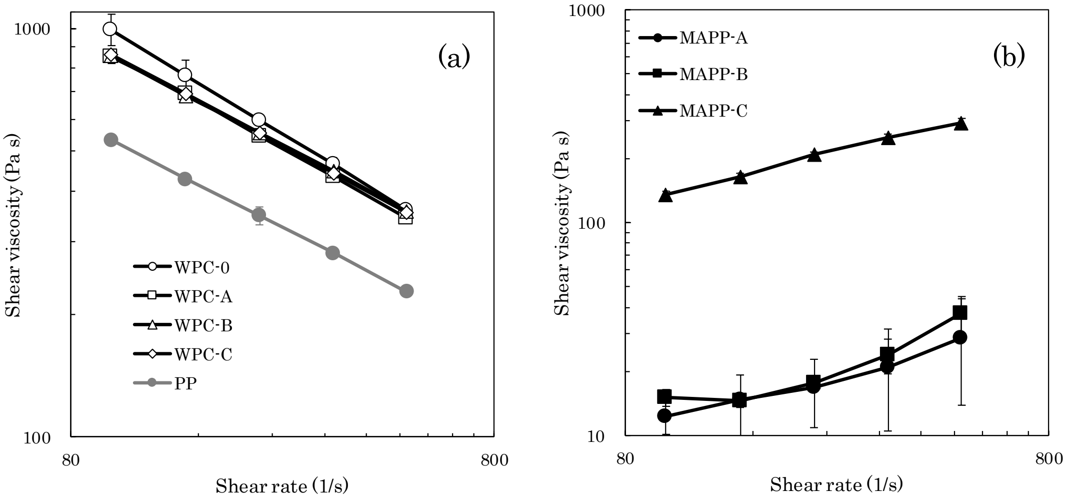

***Figure* 1.** Shear viscosity at the different shear rates for PP, pellets of WPCs, and different types of MAPP: (a) WPCs and PP, (b) different types of MAPP. Vertical bars indicate standard deviations. Samples and codes are defined in Tables 1 and 2, respectively.

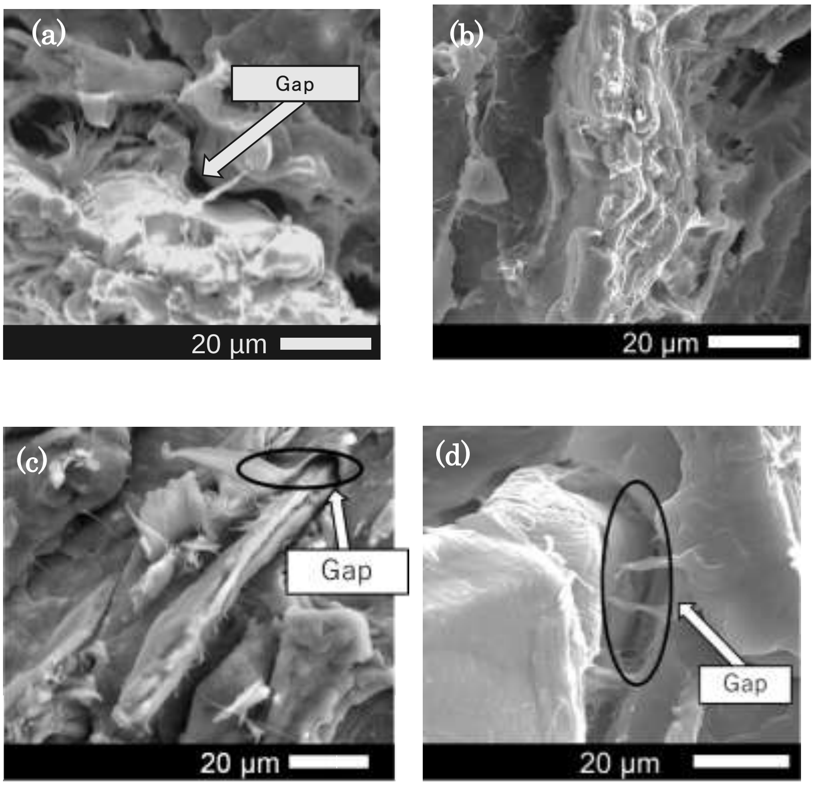

***Figure* 2.** SEM images of the fracture surface of tensile specimens under dry conditions: (a) WPC-0, (b) WPC-A, (c) WPC-B, (d) WPC-C.

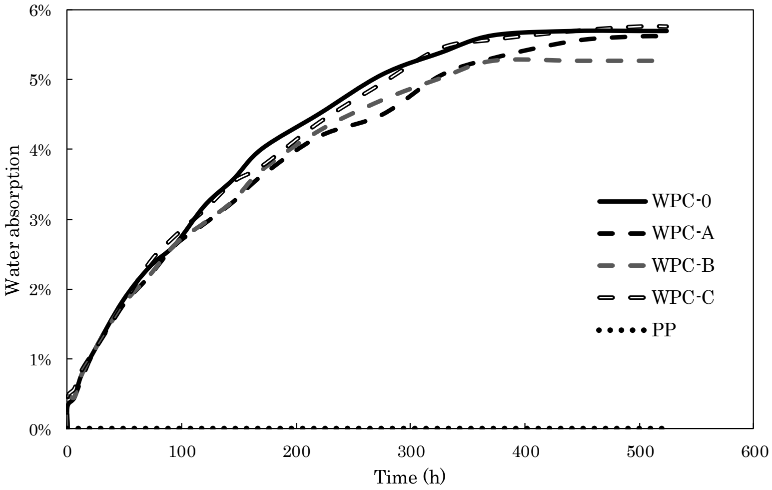

***Figure* 3.** Water absorption curves of rectangular WPC and PP specimens.

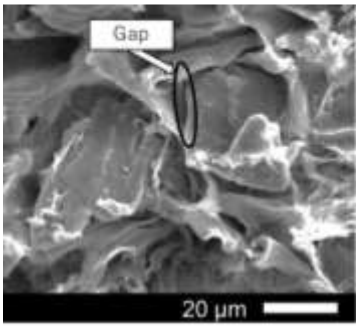

***Figure* 4.** SEM image of the fracture surface of a WPC-A tensile specimen after water absorption.