

# Mechanical and Physical Properties of Wood–Plastic Composites Containing Cellulose Nanofibers Added to Wood Flour

Kazushige Murayama      Momoka Yamamoto      Hikaru Kobori  
Yoichi Kojima      Shigehiko Suzuki      Kenji Aoki      Hirokazu Ito  
Shinji Ogoe      Masaki Okamoto

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## Abstract

Recently, the usage of wood–plastic composite (WPC) products has increased, mainly for exterior decking. The shape of fillers is one of the important factors deciding the mechanical and physical properties of WPCs. Surface-fibrillated wood flour (WF) improves the mechanical properties of WPCs, although it requires a lot of energy and time to produce the fibrous structure during the pulverization process. Therefore, the adsorptive interaction between cellulose nanofiber (CNF) and WF by hydrogen bonding was investigated. We considered that CNF could form the fibrous structures on the WF surface by mixing CNF and WF. Also, it is thought that the addition of CNF could increase mechanical and physical properties of WPC because CNF has better physical and mechanical properties than most other fibers. The objective of this study was to produce WF-CNF fillers and to evaluate the mechanical and physical properties of WPCs containing WF-CNF fillers. WF-CNF fillers could be produced by freeze-drying after mixing WF, CNF, and water. The fibrous structures on the WF surface were observed through scanning electron microscope images of WF-CNF filler containing 3 weight percent CNF. A WPC with WF-CNF filler containing 22 percent (by weight) WF and 3 percent (by weight) CNF showed improved mechanical properties compared with WPCs without CNF. The water absorption of WPCs containing CNF was found to decrease with increasing CNF content.

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Wood–plastic composite (WPC) is a material made by combining wood flour (WF), thermoplastic resin, and a small amount of additives. WPC can be produced from wood waste and recycled thermoplastic resin, thereby reducing the consumption of thermoplastics derived from petroleum (Kim and Pal 2010, Gardner et al. 2015). WPC has high specific strength, specific modulus, and low density and friction compared with inorganic filler composites. WPC also has higher water and decay resistance than solid wood. An early example of combining WF with phenolic resin used as an automobile gearshift knob appeared around 100 years ago (Gordon 1988). Since the late 1990s, the number of WPC studies has been increasing because of the increased demand for WPC decking as a replacement for preservative-treated wood decking (Watanabe 2014). Generally, WPC is used in building products such as decking and louvers, but is expected to be used in other industries with improvements in its mechanical and physical properties (Kim and Pal 2010, Soucy et al. 2014).

The main studies of WPC that focus on the improvement of its mechanical properties investigate the influence of the

types and amounts of thermoplastic resins, additives, and fillers. Normally, thermoplastic resins with melt temperatures under 220°C are used to avoid degradation of WF (Kim and Pal 2010, Watanabe 2014, Gardner et al. 2015). The use of polyethylene (Zabihzadeh et al. 2010), polypropylene (PP; Kim and Pal 2010), polyvinyl chloride

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The authors are, respectively, PhD Student, Former Undergraduate Student, Assistant Professor, Associate Professor, Professor, and Appointed Professor, Faculty of Agric., Shizuoka Univ., Shizuoka, Japan (murayama.kazushige@shizuoka.ac.jp, t0316042@ipc.shizuoka.ac.jp, kobori.hikaru@shizuoka.ac.jp, kojima.yoichi@shizuoka.ac.jp [corresponding author], suzuki.shigehiko@shizuoka.ac.jp, aoki.kenji@shizuoka.ac.jp); Senior Researcher, National Inst. of Advanced Industrial Sci. and Technol., Hiroshima, Japan (itou.hirokazu@aist.go.jp); and General Manager and Member, TOCLAS Co., Ltd., Shizuoka, Japan (Shinji\_Ogoe@toclas.co.jp, masaki\_okamoto@toclas.co.jp). This paper was received for publication in February 2018. Article no. 18-00006.

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(Jiang and Kamdem 2004), polystyrene (Kim and Pal 2010), acrylonitrile butadiene styrene (Kaseem et al. 2015), and recycled resins (Adhikary et al. 2008) has been reported. PP has excellent processability, mechanical properties, water resistance, chemical resistance, and recycling efficiency (Kim and Pal 2010, Watanabe 2014). For WPC products, the improvement of compatibility between hydrophobic thermoplastic resins and hydrophilic WF was investigated. Generally, maleic anhydride–modified polypropylene (MAPP), MA-modified polyethylene, and styrene ethylene butylene styrene (Kim and Pal 2010, Chen et al. 2013) were used as the compatibilizers. In the case of PP as a matrix, MAPP is commonly used as a compatibilizer. Some researchers reported that the characteristics of WPCs were influenced by the amounts, species, and shapes of fillers (Stark and Berger 1997, Stark and Rowlands 2003). Stark and Berger (1997) reported that the modulus of elasticity of WPC increased with increasing filler amounts and the strength properties of WPCs showed the highest values between 20 and 40 percent (by weight; wt%). Furthermore, the strength properties of WPCs showed higher values with hardwood fillers or fillers with particle sizes of about 250  $\mu\text{m}$ . In addition, it was reported that the effect of filler aspect ratio was more important than that of filler size for the mechanical properties of WPCs (Stark and Rowlands 2003). Therefore, filler shape is one of the most important factors affecting the mechanical properties of WPCs. In recent studies, it was reported that WPCs containing WF with fibrous structures on the surface, derived from mechanical pulverization, had better mechanical properties than those containing WF without fibrous structures (Isa et al. 2014, 2016). However, it requires a lot of energy and time to prepare the fibrous structures during the pulverization process. Therefore, we focused on the nanoscaled structures of cellulose nanofiber (CNF). CNF has received attention in numerous fields worldwide. Over a trillion tons of CNF exist worldwide. CNF is known to have better physical and mechanical properties than most other fibers (Kojima et al. 2016). In the case of plastic composites, CNF composites have better mechanical properties than thermoplastic resin itself (Hees et al. 2017, Soman et al. 2017). However, these CNFs were prepared by chemical treatment to avoid aggregation. The plastic composites containing CNF without chemical treatment had worse strength properties than thermoplastic resins (Robles et al. 2015). It is thought that CNFs are attached to a hydrophilic material by hydrogen bonding. We considered that CNF could form the fibrous structures on the WF surface by mixing CNF and WF. Also, it is thought that the addition of CNF could increase mechanical and physical properties of WPC because CNF has better physical and mechanical properties than most other fibers. The objective of this study was to produce WF-CNF fillers by mixing and drying and to evaluate the mechanical and physical properties of WPC containing WF-CNF fillers. A drying process was required to prepare the WF-CNF fillers because CNF was mixed with WF with high moisture contents. Thus, the effects of two drying methods, heat and freeze-drying, were evaluated.

## Materials and Methods

### Materials

The following raw materials were used: commercial WF with particle sizes under 150  $\mu\text{m}$  (ARBOCEL C-100;

Rettenmaier Co., Ltd., Japan), commercial CNF (BiNF-i-s WMa-10010; Sugino Machine Co., Ltd., Japan), PP (J-107G; Prime Polymer Co., Ltd., Japan), and MAPP (Kayabrid 006PP-N; Kayaku Akzo Co., Ltd., Japan) as a compatibilizer. The CNF was contained in 10 wt% CNF and 90 wt% water. The PP was a homopolymer powder with a melt flow rate of 30 g/10 min (230°C/2.16 kg) and a density of 0.9 g/cm<sup>3</sup>. The MAPP powder contained 2 wt% MA and had an average molecular weight of 75,000.

### Preparing the WF-CNF filler

In this study, the WF-CNF filler used in the WPCs was fixed at 25 wt%. Fillers were produced with CNF contents of 0, 3, 5, and 10 wt%. The WF content of these fillers was therefore 25, 22, 20, and 15 wt%, respectively. In the case of 3 and 5 wt% CNF, water was added into commercial CNF to maintain the same moisture content as in the 10 wt% WF-CNF filler. The WF and CNF (and water) were blended at 80°C for 10 minutes at a rotary speed of 30 rpm using a Laboplast mill (Laboplast Mill 30R150; Toyo Seiki Seisaku-sho, Ltd., Japan). The moisture content of the WF-CNF (and water) mixture was approximately 75 percent. Therefore, the mixture was dried using two different drying processes, heat drying and freeze-drying. Heat drying is an easy and fast drying method. Freeze-drying, however, is a more difficult drying method, but it is believed to reduce aggregations. Heat drying was carried out at 80°C for 24 hours in an oven dryer (SOFW-600; AS ONE Co., Ltd., Japan). Freeze-drying was carried out at -30°C for 120 hours in a freeze-dryer (FDD1200; Tokyo Rikakikai Co., Ltd., Japan). After the drying process, the mixture of WF-CNF was crushed with a laboratory mixer (IFM-800DG; Iwatani Co., Ltd., Japan). After crushing, the WF-CNF mixture is referred to as WF-CNF filler. Figure 1 shows the general procedure of WPC production.

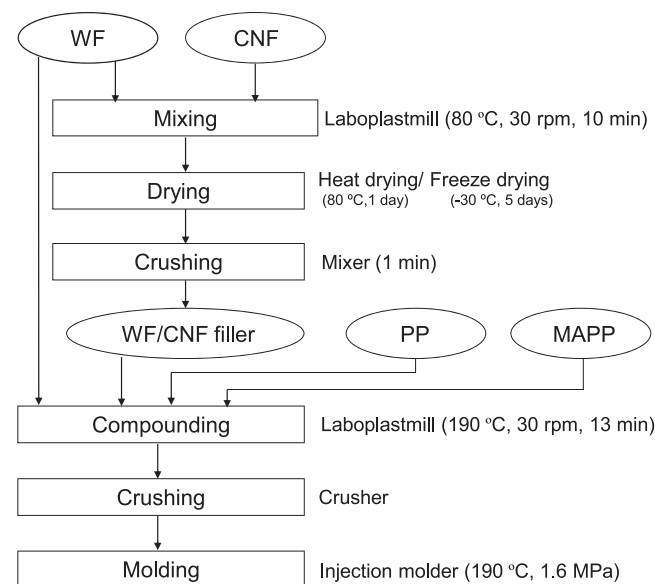


Figure 1.—General procedure of wood flour (WF)–cellulose nanofiber (CNF) filler and wood–plastic composite production. PP = polypropylene; MAPP = maleic anhydride–modified polypropylene.

## Filler characterization

A laser-diffraction particle-size distribution analyzer (Partica LA-9502; Horiba, Ltd., Japan) was used to obtain the median particle diameter and particle-size distribution of the WF-CNF filler. The surface morphology of the WF-CNF filler was observed using a scanning electron microscope (SEM; JSM-6510LV2; JEOL, Ltd., Japan).

The bulk density of WF and WF-CNF filler after conditioning at 20°C and 65 percent relative humidity (RH) for 2 weeks was calculated by measuring a volume of 4.35 mL in a standard cell (F10-UV-10; GL Sciences, Japan).

## Sample preparation

The lower part of Figure 1 shows the compounding and moulding processes. The materials were blended at a filler-PP-MAPP ratio of 25/74/1 (wt%) using a Laboplast mill. Mixing of filler-PP-MAPP was performed at 190°C for 13 minutes at a rotary speed of 30 rpm. The compounds were crushed into particle sizes of less than 1 mm using a low-speed axial crusher (SA-23; Stolz Co., Ltd, Japan). The crushed compounds were melted and mixed using a microcompounder (Micro5cc Twin Screw Compounder; DSM Xplore, The Netherlands) at 190°C for 5 minutes at a rotary speed of 30 rpm. The WPC specimens were formed in an injection moulder (DSM Xplore) at 190°C at an injection pressure of 1.6 MPa. WPCs without CNF were prepared using the same compounding and moulding processes as control specimens. Rectangular specimens with typical dimensions of 50 by 6 by 2 mm were prepared for flexural, impact, and water absorption tests. Dumbbell-shaped tensile specimens with typical dimensions of 50 by 4 by 2 mm were prepared for tensile tests.

All specimens were conditioned at 20°C and 65 percent RH for 1 week before each test.

## Physical and mechanical testing

Composite performance was evaluated using flexural, tensile, and impact tests. The flexural test was carried out using a universal testing machine (Yasui Kiki Co., Ltd., Japan). The flexural strength and flexural modulus were measured using a three-point bending strength test. A cross-head speed of 5 mm/min and a span length of 32 mm were used. The tensile test for calculating the tensile strength was carried out using a universal testing machine (AGS-5kNX; Shimadzu Co., Ltd., Japan). Dumbbell-shaped tensile specimens were used. A cross-head speed of 20 mm/min and a gauge length of 30 mm were used for the test. An unnotched Izod impact test was conducted using an impact tester (U-F impact tester; Ueshima Seisaku-sho, Japan). The test was performed at a speed of 3.5 m/s. The impact energy was 2 J. Water absorption was determined by measuring the changes in weight of the rectangular specimens before and after soaking in water at 70°C for 5 days (120 h). Five rectangular and dumbbell-shaped tensile specimens were used for each test and average data along with the corresponding standard deviation are reported.

## Results and Discussion

### Evaluation of WF/CNF

Figure 2 shows the particle size distributions of commercial CNF, WF, and WF-CNF fillers with different

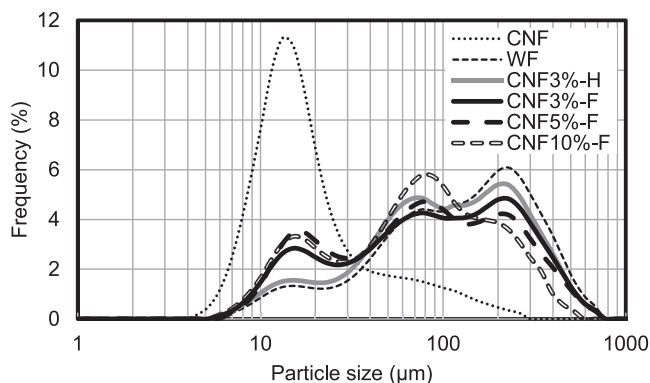


Figure 2.—Particle size distributions shown for cellulose nanofiber (CNF), wood flour (WF), and WF-CNF fillers. CNF is commercial CNF; WF is commercial WF. CNF 3, 5, and 10 percent are WF-CNF fillers containing 3, 5, and 10 percent (by weight) CNF, respectively. H = heat drying; F = freeze-drying.

drying processes. A notable peak around 10 µm was observed in the WF-CNF filler. It is thought that this peak comes from CNF unattached to WF, because CNF has the same peak. In addition, the peak around 10 µm for freeze-dried WF-CNF filler was larger than that for the heat-dried filler. The frequency peaks around 70 to 90 µm increased with increasing CNF content. However, the peak around 10 µm, which was derived from CNF, did not increase in size. It is suggested that CNF is excessively attached to WF under high CNF content. Comparing the drying methods, the frequency of high particle sizes was higher in the heat-dried filler than in the freeze-dried filler. It is thought that many aggregates of WF and CNF were formed after heat drying compared with freeze-drying because of rapid vaporization during the heat-drying process. Figure 3 shows SEM images of CNF, WF, and WF-CNF fillers with different drying processes. In the case of CNF, nanofibers around 100 nm in diameter and aggregates of CNF can be observed (Fig. 3a). WF had a relatively smooth surface and fibrous structures were not observed on the surface (Fig. 3b). In the case of WF-CNF filler containing 3 wt% CNF, fibrous structures were observed from both drying processes (Figs. 3c and 3d). Many more fibrous structures were observed after freeze-drying compared with heat drying. It is clear that the surface fibrous structures were derived from the CNF because these structures are not observed in the WF image (Fig. 3b). Figure 4 shows a schematic diagram of the fibrous structure formation process. Under high moisture content, a part of a CNF becomes attached to the WF to form the surface fibrous structure. However, these fibrous structures were not maintained after heat drying, since CNF aggregated on the WF surface during rapid vaporization. On the other hand, it is thought that freeze-drying causes less aggregation. Therefore, many surface nanofibers were observed after freeze-drying (Fig. 3d). In the case of freeze-drying, aggregates of CNF on the surface of WF were observed in WF-CNF filler containing 5 wt% CNF (Fig. 3f). In WF-CNF filler containing 10 wt% CNF (Fig. 3h), aggregates of CNF with larger diameters than the WF were observed. It is thought that some of CNF aggregates had equal or larger diameters than WF. These aggregates contained many voids between CNF. Many CNF aggregates were also observed in WF-CNF filler from heat drying with 5 and 10 wt% CNF



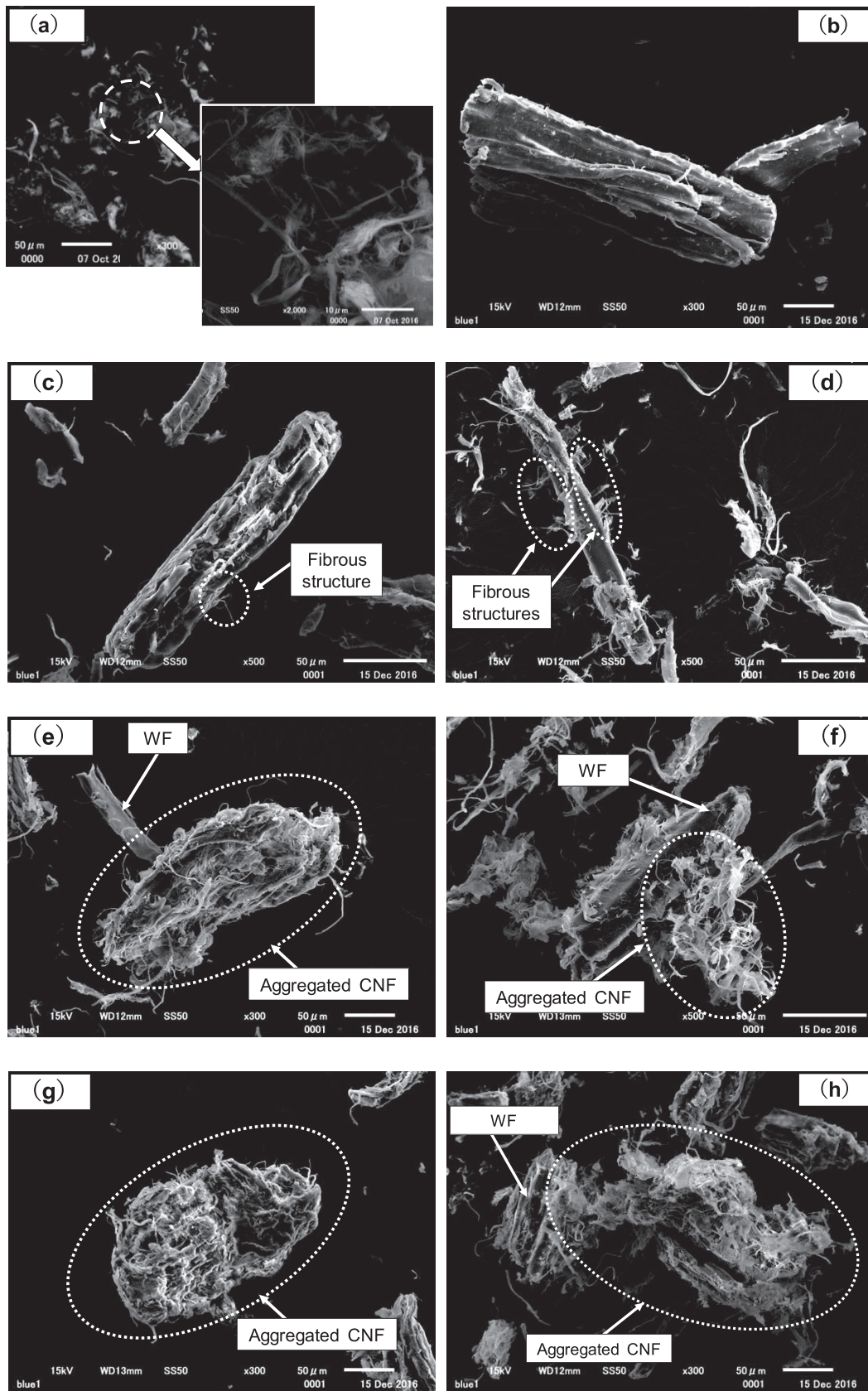


Figure 3.—Scanning electron microscope images of cellulose nanofiber (CNF), wood flour (WF), and WF-CNF fillers. (a) CNF, (b) WF, (c) WF-CNF filler containing 3 percent (by weight) CNF after heat drying, (d) WF-CNF filler containing 3 wt% CNF after freeze-drying, (e) WF-CNF filler containing 5 wt% CNF after heat drying, (f) WF-CNF filler containing 5 wt% CNF after freeze-drying, (g) WF-CNF filler containing 10 wt% CNF after heat drying, (h) WF-CNF filler containing 10 wt% CNF after freeze-drying.

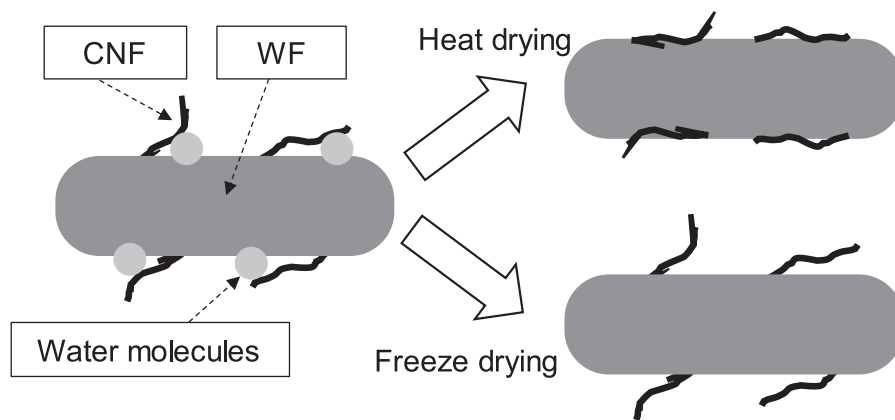


Figure 4.—Schematic diagram of the fibrous structure formation process. CNF = cellulose nanofiber; WF = wood flour.

content (Figs. 3e and 3g). The aggregated CNFs after heat drying were densely aggregated. Figure 5 shows the bulk density of WF and WF-CNF fillers with different drying processes. As the CNF content increased, the bulk density from heat drying increased, whereas the bulk density from freeze-drying decreased. These results suggest that WF-CNF fillers after heat drying have densely aggregated structures and WF-CNF fillers after freeze-drying have large specific surface, including fibrous structures derived from CNF. These results agree with the SEM images of the WF-CNF fillers (Figs. 3c through 3h).

These results suggest that it is difficult to prepare fibrous structures on WF-CNF fillers using the heat-drying process. Therefore, only the mechanical properties of WPCs containing WF-CNF fillers after freeze-drying were evaluated.

### Mechanical and physical properties

Figure 6 shows the relationship between CNF content and WPC flexural properties. WPCs with 3 wt% CNF had the highest flexural strength and modulus in this study. The flexural strength and modulus of WPC containing 3 wt% CNF were 5 and 9 percent higher than those of WPC without CNF, respectively. WF-CNF fillers containing 3 wt% CNF maintained their fibrous structures (Fig. 3d). It is

thought that the fibrous structures exert a strong reinforcement effect on the matrix, which results in the better flexural properties of the WPC with WF-CNF filler containing 3 wt% CNF. The decreases on flexural strengths of WPCs containing 5 and 10 wt% CNF may be due to increases of excessive amounts of CNF attached to WF surfaces and CNF unattached to WF. However, WPCs containing 5 and 10 wt% CNF had nearly the same flexural strengths as WPC without CNF. It is thought that excessive CNF on the WF surface and aggregated CNF has the same reinforcing effect as commercial WF. However, the flexural moduli of WPCs containing 5 and 10 wt% CNF were higher than that of WPC without CNF. According to the bulk density shown in

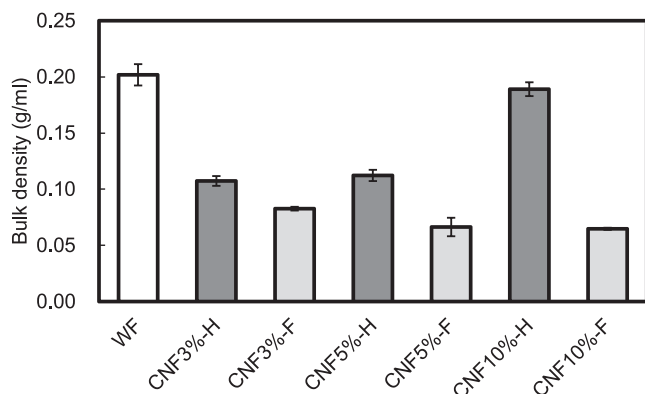


Figure 5.—Effects of cellulose nanofiber (CNF) content and drying process on the bulk density of wood flour (WF) and WF-CNF. WF is pure WF. CNF 3, 5, and 10 percent are WF-CNF fillers containing 3, 5, and 10 percent (by weight), respectively. H = heat drying; F = freeze-drying.

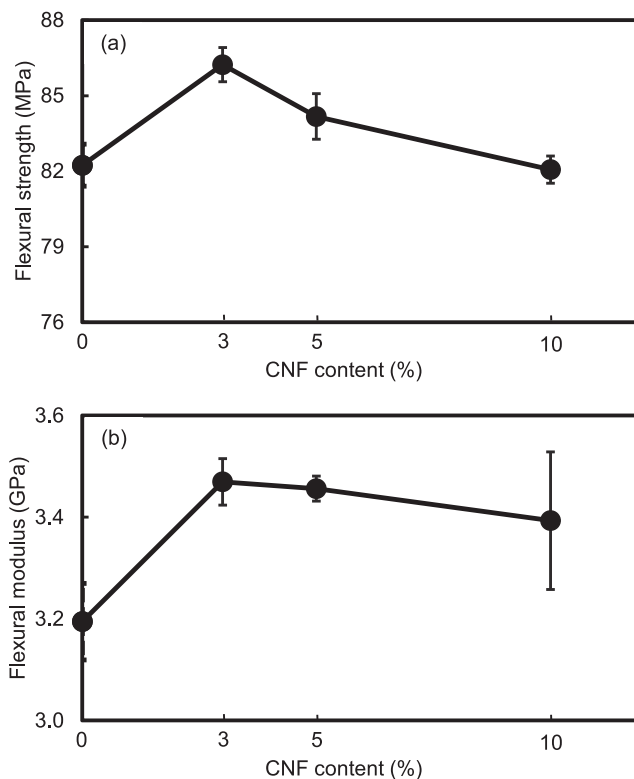


Figure 6.—Relationship between cellulose nanofiber (CNF) content and flexural properties of wood–plastic composite specimens. (a) Flexural strengths and (b) flexural moduli.

Figure 5, this may be owing to the higher volume fraction of WF-CNF fillers in the resins compared with commercial WF.

Figure 7 shows the relationship between CNF content and the tensile strengths of WPC specimens. WPC containing 3 wt% CNF had the highest tensile strength, as with flexural strength. The tensile strength of WPC containing 3 wt% CNF showed increases of 6 percent over that of WPC without CNF. The tensile strengths of WPCs containing 5 and 10 wt% CNF were still higher than that of WPC without CNF. Sparsely aggregated CNF, which contained many voids, was observed in WF-CNF fillers containing 5 and 10 wt% CNF (Figs. 3f and 3h). It is thought that these sparsely aggregated CNFs become the break point in the tensile test, although WPCs containing 5 and 10 wt% CNF have higher tensile strength than WPC without CNF. This may be owing to the large extent of rigid interfacial bonding in sparsely aggregated CNF. Figure 8 shows the relationship between CNF content and the unnotched impact strengths. The impact strength of WPC containing 3 wt% CNF was highest, as with flexural and tensile strength. The impact strength of WPC containing 3 wt% CNF showed increases of 12 percent over that of WPC without CNF. For the unnotched Izod impact test, some researchers have reported that the impact strengths of WPCs decreased with increasing filler size or decreasing filler aspect ratio (Stark and Berger 1997, Stark and Rowlands 2003). WF-CNF filler containing

3 wt% CNF has a higher aspect ratio than WF, which results in the higher impact strength of the WPC. The decreases on impact strengths of WPCs containing 5 and 10 wt% CNF may be owing to the large size and small aspect ratio fillers that were derived from the aggregated WF with CNF. The aggregated CNFs in WF-CNF fillers containing 5 and 10 wt% CNF were observed through SEM images (Figs. 3f and 3h). In addition, according to the particle size distributions shown in Figure 2, the frequency of large-size particles increased with increasing CNF content.

These results suggest that WPCs containing WF-CNF filler with suitable CNF content (3 wt% CNF content in this study) can form fibrous structures, which results in higher flexural strength, flexural modulus, tensile strength, and impact strength than WPCs without CNF.

Figure 9 shows the water absorption curve from 0 through 120 hours. WPC containing 3 wt% CNF and WPC without CNF had almost the same water absorption rate. At a CNF content of 5 wt% or more, the water absorption rate of WPCs was reduced with increasing CNF content. This indicates that WPCs containing CNF have higher water resistance than that of WPC without CNF. Figure 10 shows the relationship between CNF content and water absorption after 120 hours. The water absorption of WPCs and CNF content showed a highly negative correlation through linear approximation. WF absorbs water because of many internal voids. However, it is difficult to absorb water into CNF

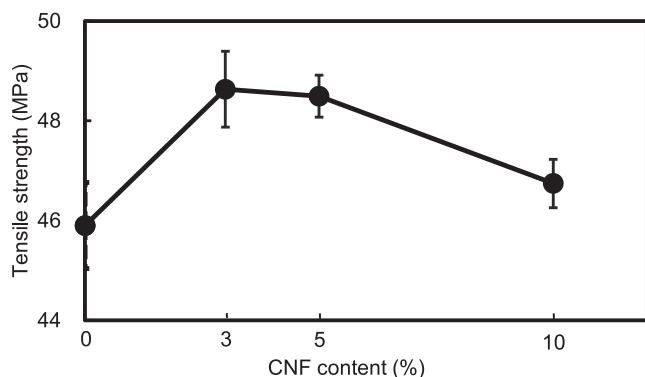


Figure 7.—Relationship between cellulose nanofiber (CNF) content and tensile strength of wood–plastic composites.

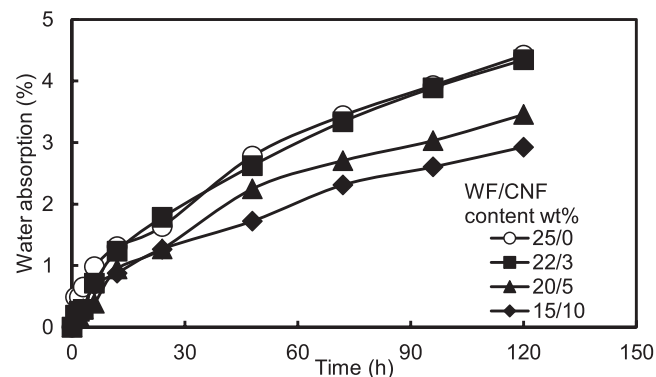


Figure 9.—Water absorption curve from 0 through 120 hours. WF = wood flour; CNF = cellulose nanofiber.

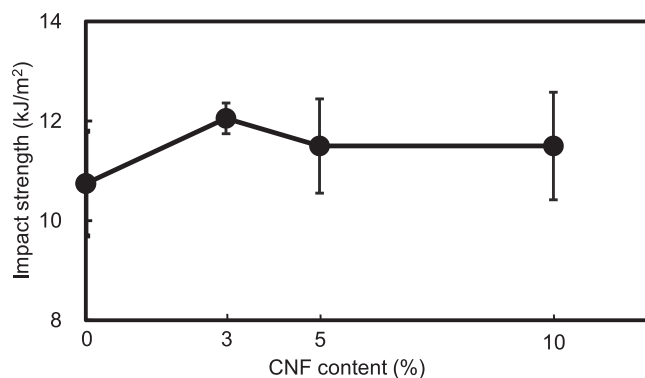


Figure 8.—Relationship between cellulose nanofiber (CNF) content and unnotched Izod impact strength of wood–plastic composites.

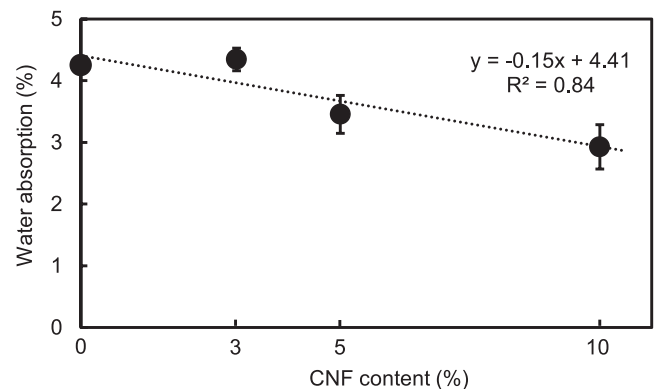


Figure 10.—Relationship between cellulose nanofiber (CNF) content and water absorption of wood–plastic composites after 120 hours.

because it has fewer internal voids. Therefore, it is reasonable to improve the water resistance of WPCs by replacing WF with CNF. In WF-CNF fillers containing 5 and 10 wt% CNF, aggregated CNFs of nearly the same size as WFs were observed (Figs. 3b, 3f, and 3h). It is thought that aggregated CNFs have firm structures that make it difficult for WPCs to absorb water.

### Conclusions

CNF was attached to WF by freeze-drying after mixing WF, CNF, and water. The fibrous structures on the WF surface were observed through SEM images of WF-CNF filler containing 3 wt% CNF. In strength tests, WPCs containing WF-CNF filler had better mechanical properties than WPC without CNF. In this study, WPC with WF-CNF filler containing 3 wt% CNF displayed the best mechanical properties. The water absorption of WPCs containing CNF decreased with increasing CNF content. It is thought that aggregated CNFs have firm structures that make it difficult for WPCs to absorb water. At a CNF content of 5 wt% or more, the mechanical properties of WPC were not increased. In contrast, the water resistance of WPC was increased with increasing CNF content.

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