

Properties of Compression-Molded Plates Made from Wood Powders Impregnated with Liquefied Wood-based Novolak-Type Phenol-Formaldehyde Resins

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ABSTRACT: Novolak-type phenol-formaldehyde (PF) resins with solution form were prepared by reacting phenol-liquefied *Cryptomeria japonica* (Japanese cedar) wood with formalin in the presence of methanol. Wood powders of *Albizia falcate* (Malacca albizzia) impregnated with these resins were air dried followed by an oven-dried at 60°C. DSC analysis showed the PF resin existing in wood powders could be melted, and could be cured if hexamine was mixed and heated at high temperature. Compression-molded plates made with PF resin impregnated woods had a high degree of curing reaction. However, compression-molded plates hot-pressed at 180°C for 8 min or 200°C

for 5 min had better internal bonding strength and dimensional stability than others. Premixing hexamine with PF resin and impregnating into wood powders simultaneously could enhance the reactivity of PF resin, but it was not useful for improving the properties of compression-molded plates. © 2010 Wiley Periodicals, Inc. *J Appl Polym Sci* 118: 3471–3476, 2010

Key words: biomaterials; compression-molded plates; impregnated wood powders; liquefied wood; novolak-type PF resin

INTRODUCTION

PF resins are the oldest synthetic resins, and there are many various types with different properties depending on synthesis conditions. Novolak-type PF resin is one of the PF resins, which is widely used for manufacturing moldings. It is synthesized from phenol with an insufficient amount of formaldehyde under acidic condition to form a thermoplastic prepolymer without hydroxymethyl group in its molecular structure. A crosslinking agent such as hexamine is needed for it to form a cured network structure under heating. On the other hand, resol-type PF resin is widely used as an adhesive, which is prepared under basic condition with the molar ratio of formaldehyde to phenol over 1.0 and can be cured under heating.

Solvent liquefaction is an effective method to convert lignocellulose from a solid to liquid. Phenol is one of the solvent commonly used for wood liquefaction, and the phenol-liquefied products can be used as raw materials for preparing phenolic resins.

Alma et al.^{1–3} investigated the flowability of phenolated wood and indicated mixing phenolated wood with hexamine could be used as raw materials for moldings. However, if the phenol-liquefied wood was prereacted with formalin, the moldings made had better physical and mechanical properties. Lin et al.^{4–6} investigated the mechanism of liquefaction of cellulose in the presence of phenol and the heat flowability of phenolated wood. They indicated that moldings made with phenolated wood that containing a more complex phenol would have a better performance. But if the liquefied wood reacted with formaldehyde to form phenolated wood/phenol/formaldehyde cocondensed novolak-type PF resins, the flowing temperature and melting viscosity were similar to conventional novolak-type PF resins. Lee et al.^{7–10} used phenol as a solvent to liquefy wood, corn bran, and waste paper. They indicated the free phenol that existed in liquefied products could react with formaldehyde and these liquefied products could be used as raw materials for preparing both novolak-type and resol-type PF resins. Pan et al.¹¹ investigated the curing kinetics of liquefied wood/phenol/formaldehyde resins and indicated that the properties of prepared resins were influenced by synthesis conditions. Lee et al.^{12,13} indicated that mixing the liquefied wood with formalin would cause an exothermal reaction, and formed a novolak-type PF resin without extra heating. The dried resin had

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the characteristic of thermomelting. Zhang et al.^{14,15} investigated the characteristics of phenol-liquefied biomass and employed them to prepare liquefied wood-based resins and indicated the resin prepared could be used for moldings manufacturing. The phenol-liquefied biomass was also employed in preparing resol-type PF resins and used in manufacturing plywood and particleboard.^{16–18}

Novolak-type PF resins are widely used in moldings. In general, they are used by mixing solid resin powders with wood powders and hexamine, and cured by hot-pressing. However, a fully uniform mixing of all these components is difficult although it is necessary for moldings manufacturing. In this study, novolak-type PF resins with solution form were prepared by reacting phenol-liquefied wood with formaldehyde in the presence of methanol and then impregnated into wood powders free from or with hexamine. After being dried, the thermal properties of these impregnated wood powders and the feasibility for them to make compression-molded plates were investigated.

EXPERIMENTAL

Materials

Woods of Japanese cedar and Malacca albizzia were used for liquefaction and compression-molded plates manufacture, respectively. Chemicals such as sulfuric acid (H_2SO_4), hydrochloric acid (HCl), phenol, formalin (37% aq), methanol, ethanol, hexamethylenetetramine (hexamine; $\text{C}_6\text{H}_{12}\text{N}_4$), and acetone were the reagent grade and were used without further purification.

Liquefaction and properties of phenol-liquefied japanese cedar wood

Wood meals of Japanese cedar with a dimension passing 20 meshes were liquefied in phenol with H_2SO_4 or HCl as a catalyst. Before liquefaction, phenol and catalyst were charged in a 1000-mL glass reactor equipped with a stirrer, a thermometer, and a reflux condenser. For the liquefaction using H_2SO_4 as a catalyst, the weight ratio of phenol/wood/ H_2SO_4 was 25/10/1.25, and the reaction took place at 130°C for 60 min. The liquefied product contains 2.4% of unliquefied wood residue and has the viscosity of 149,700 cps. For the liquefaction using HCl as a catalyst, the weight ratio of phenol/wood/HCl was 30/10/3, and the reaction took place at 110°C for 60 min. The liquefied product contains 3.8% of unliquefied wood residue and has the viscosity of 25,100 cps.

Synthesis of phenol-liquefied wood-based novolak PF resins

When preparing phenol-liquefied wood-based novolak PF resins, 94 g of liquefied wood and 50 g of methanol were charged into a 500 mL round-bottom reactor equipped with a stirrer, a thermometer and a reflux condenser. The mixture was heated to 50°C followed by gradually dripping down 48.6 g of formalin which containing 0.6 mol of formaldehyde from a separating funnel. After all formalin was added, the reaction proceeded at reflux temperature for 60 min. The solution of prepared resin was then cooled to room temperature and diluted with ethanol to a concentration of 20% followed by adjusting the pH to 7–7.5. Codes of S-PF and C-PF were used to represent PF resins that prepared from wood that liquefied with H_2SO_4 and HCl as a catalyst, respectively. Because novolak PF resin can not be cured unless a crosslinking agent, the hexamine, is added. For the purpose in getting a more uniform contact between hexamine and PF resin, 20% of hexamine-based on the weight of nonvolatile content of PF resin was dissolved in water and added into the diluted PF resins. Codes of S-PF-H and C-PF-H were used to represent PF resin those had premixed with hexamine.

Impregnated wood powders with PF resin

Wood powders of Malacca albizzia with a dimension passing 100 meshes were immersed in various kinds of resin solution for overnight. After filtering, the impregnated wood powders were air-dried, followed by being oven-dried at 60°C to remove the solvent completely. The weight percent gain of resin for wood powders was controlled as 100% for those impregnated with S-PF and C-PF, and 120% for those impregnated with S-PF-H and C-PF-H in order to have the weight ratio of wood to PF resin as 1/1.

DSC thermal analysis of PF resin impregnated wood powders

The thermal properties of wood powders impregnated with PF resin that were free from or containing hexamine were measured with differential scanning calorimetry (DSC; PerkinElmer DSC-7). The test was conducted by heating from 40 to 200°C at a heating rate of 10°C/min in a nitrogen environment. In addition, the impregnated wood powders that free of hexamine were mixed with hexamine powder with a weight ratio of 9/1, and then measured under similar conditions.

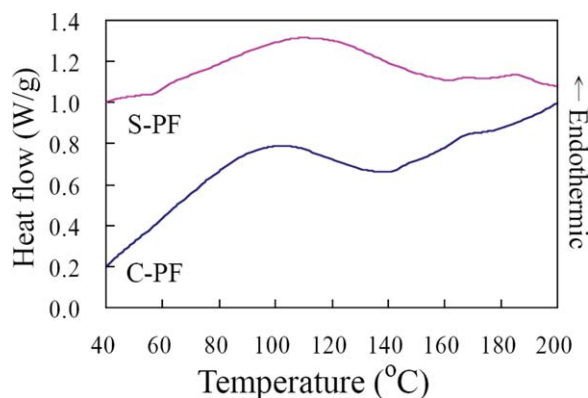


Figure 1 DSC curves of wood powders impregnated with PF resin. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

Compression-molded plates manufacture

The dry wood powders those impregnated with PF resins, which had the weight ratio of wood to resin as 1/1 were used to manufacture compression-molded plates. For wood powders that impregnated C-PF and S-PF, which were free of hexamine were mixed with hexamine with a weight ratio of 9/1, but for those impregnated with C-PF-H and S-PF-H were used directly without additional hexamine. Compression-molded plates with a dimension of $10 \times 10 \times 0.4 \text{ cm}^3$ and density of 1.0 g/cm^3 were made by hot-pressing at 180 and 200°C for 5 and 8 min, respectively, under a pressure of 1.96 MPa.

Properties measuring of compression-molded plates

Compression-molded plates were cut into specimens with a dimension of $2 \times 2 \times 0.4 \text{ cm}^3$. The internal bonding strength was measured by a universal strength testing machine (Shimadzu UEH-10) using a load vertical to the flat surface of plate at a speed of 2 mm/min. The water immersion test was carried out by immersing specimens into water and the percentage of water absorption and swelling thickness at times of 2 and 24 h were measured. The measurement of internal bonding strength, water absorption, and swelling thickness were conducted for five replications. In addition, some of compression-molded plates were ground to particles and dried with a vacuum-oven at 60°C for 24 h. These particles were immersed in acetone for 24 h, followed by filtrating and drying. The percentage of weight retention was used as an index of the curing degree of resins. For thermogravimetric analysis (TGA; PerkinElmer Pyris 1), about 5 mg of the sample was put into a platinum sample pan and heated from 50 to 800°C at a heating rate of 10°C/min under a nitrogen atmosphere with a flow rate of 20 mL/min. Curves of

weight loss (TG) and derivative TG (DTG) were plotted. The onset temperatures and weight loss percentage at each thermal degradation stage were calculated.

RESULTS AND DISCUSSION

DSC thermal properties of wood powders impregnated with PF resin

Figure 1 shows DSC curves of wood powders impregnated with S-PF and C-PF that were free of hexamine. Both of them showed a broad endothermic peak, indicating the resin absorbed in wood powders can be melted under heat. However, wood powders impregnated with C-PF had the heat flow increased more obviously than that of S-PF and had a lower peak temperature, they were 91.5 and 109.7°C for C-PF and S-PF, respectively. This result indicated that wood powders impregnated with C-PF had better heat flowability than that of S-PF.

Figure 2 shows the variation in heat flow for wood powders that impregnated with S-PF-H and C-PF-H. An exothermic peak appeared instead of the endothermic phenomenon. This is because the hexamine was premixed with PF resins and therefore a crosslinking reaction took place during the period of heating. Comparing wood powders impregnated with C-PF-H and S-PF-H, the former had the exothermic phenomenon more drastic than the latter, indicating C-PF-H had higher reactivity than S-PF-H.

Zhang et al.¹⁴ indicated that degraded components of wood would combine with phenol to form derivatives, i.e. combined phenol, those had hydroxyl benzyl groups and could react with formaldehyde. In our previous study,¹² both the combined phenol and the free phenol were existed liquefied woods. However, liquefied with HCl as a catalyst would had more free phenol than combined phenol, but it had

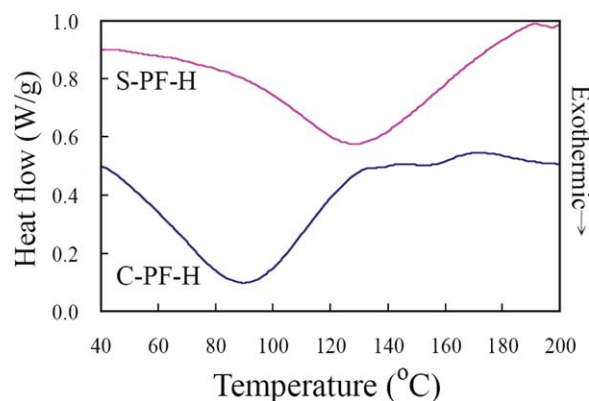


Figure 2 DSC curves of wood powders impregnated with PF resin that containing hexamine. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

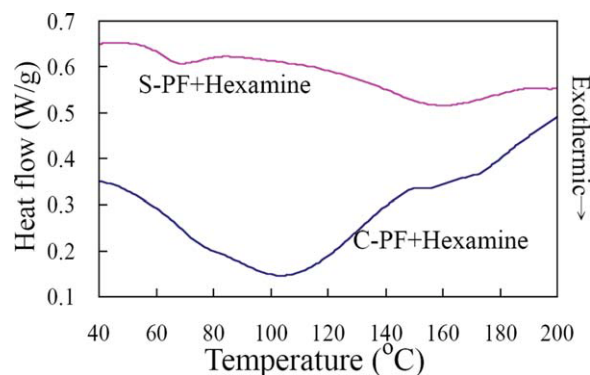


Figure 3 DSC curves of the mixture blending with PF resin impregnated wood powders and hexamine. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

more combined phenol than free phenol for that liquefied with H_2SO_4 as a catalyst. Because the combined phenol had the structure more complex than free phenol, it may be the reason that caused C-PF had better heat flowability than S-PF and C-PF-H had the higher reactivity than S-PF-H.

Figure 3 shows the DSC curves measured from the mixture of wood powders those impregnated with S-PF and C-PF blending with hexamine powder. They had the exothermic phenomenon more moderate than those impregnated with S-PF-H and C-PF-H. Especially for S-PF impregnated wood, it appeared a two-stage but ambiguous curing reaction. This result indicated that premixed hexamine with PF could provide a higher reactivity and could undergo a more crosslinking reaction during the period of heating.

Table I shows the thermosetting parameters of impregnated wood powders during the period of DSC scanning. Wood powders impregnated with S-PF-H and C-PF-H had more heat released and had lower onset and peak temperatures than that

TABLE I
DSC Thermosetting Parameters of Wood Powders Impregnated with PF Resin

Resin ^a	Onset ^b (°C)	Peak ^b (°C)	Heat ^b (J/g)
S-PF ^c	55.9	66.4	24.3
C-PF	120.1	153.7	
S-PF-H	51.3	103.2	68.7
S-PF-H	83.1	130.7	135.4
C-PF-H	47.3	89.4	113.9

^a S-PF and C-PF impregnated wood powders were measured by mixing with hexamine powder; S-PF-H and C-PF-H impregnated wood powders were measured directly.

^b The onset and peak were the initial and the maximum heat flow temperature of the exothermic peak, respectively; The heat was the released heat during the period of curing reaction.

^c It appeared two exothermic peaks, the value at upper and lower represent the first and second peak, respectively.

impregnated with S-PF and C-PF, indicating the former had higher reactivity than the latter. In the previous description, wood impregnated with S-PF and then mixing with hexamine had a two-stage curing reaction, but it had the total heat released only 24.3 J/g, indicating it underwent a less degree of crosslinking reaction during the period of curing reaction.

Properties of compression-molded plates made with impregnated wood powders

Table II shows the properties of compression-molded plates made with wood powders impregnated with novolak-type PF resin. The percentage of weight retention after acetone immersion can be used as an index for the cured degree of resin after being thermosetting. All compression-molded plates showed the high percentage of weight retention,

TABLE II
Properties of Compression Molded Plates Made with Wood Powders Impregnated with Novolak-Type PF Resin

Resins	Hot-pressing		Density (g/cm ³)	Weight retention ^b (%)	Internal bonding strength (MPa)	Water absorption (%)		Thickness swelling (%)	
	Temp. (°C)	Time (min)				2 h	24 h	2 h	24 h
S-PF	180	5	1.07	88.6	4.7 ± 1.4	4.1 ± 1.9	13.1 ± 4.3	1.1 ± 1.0	3.3 ± 0.6
S-PF	180	8	1.03	92.7	6.3 ± 1.3	2.4 ± 0.6	8.7 ± 2.0	1.1 ± 1.1	2.5 ± 0.6
S-PF	200	5	1.09	98.4	6.7 ± 0.9	2.3 ± 0.6	9.5 ± 2.4	1.6 ± 0.3	3.0 ± 0.5
S-PF	200	8	1.08	95.1	3.4 ± 0.3	4.5 ± 0.7	14.3 ± 1.3	1.5 ± 0.4	2.7 ± 0.5
S-PF-H ^a	200	5	1.14	96.5	1.1 ± 1.0	2.1 ± 0.7	7.8 ± 2.2	0.7 ± 1.1	3.3 ± 0.6
C-PF	180	5	1.06	95.6	3.7 ± 0.2	2.6 ± 0.2	9.8 ± 1.0	0.5 ± 0.2	2.3 ± 0.8
C-PF	180	8	1.03	98.6	5.4 ± 0.6	2.8 ± 0.9	10.5 ± 3.2	2.0 ± 0.5	4.5 ± 1.6
C-PF	200	5	1.11	99.5	5.7 ± 1.2	1.2 ± 0.2	5.7 ± 0.7	0.4 ± 0.3	2.9 ± 0.6
C-PF	200	8	1.07	97.9	3.7 ± 1.2	3.8 ± 1.2	12.3 ± 3.6	2.4 ± 1.1	3.7 ± 0.7
C-PF-H ^a	200	5	1.05	98.7	4.7 ± 0.2	3.2 ± 0.3	11.7 ± 1.2	2.0 ± 0.3	4.3 ± 0.3

^a PF resin containing hexamine.

^b After solvent immersion.

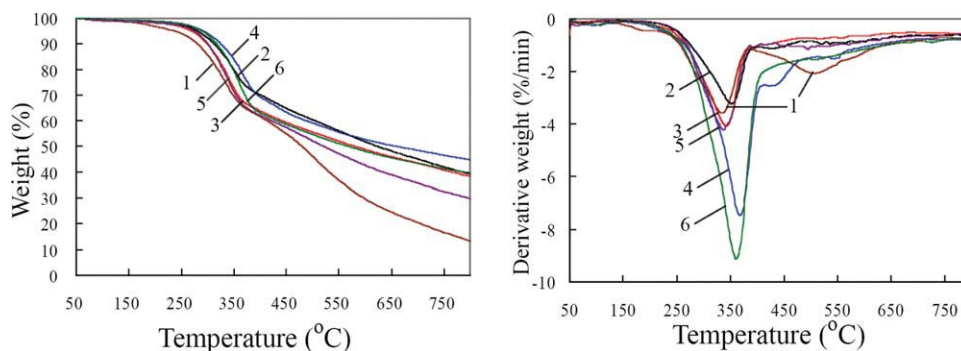


Figure 4 TG and DTG curves of compression-molded plates made with wood powders impregnated with PF resin; 1:S-PH, 180°C, 5 min; 2:S-PF,200°C, 5 min; 3:S-PF, 200°C, 8 min; 4:S-PF-H, 200°C, 5 min; 5:C-PF, 200°C, 5 min; 6:C-PF-H, 200°C, 5 min. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

indicating the resin impregnated into wood powders had a high degree of curing reaction after hot-pressed. However, compression-molded plates made with wood powders impregnated with C-PF had higher weight retention than those with S-PF. It may be because C-PF resin had better heat fluidity and reactivity than S-PF resin, resulting in a more complete crosslinking reaction. Comparing the influence of hot-pressing conditions, hot-pressed with a temperature of 200°C had higher weight retention than those at 180°C, indicating increased the temperature could promote the curing reaction. When hot-pressed at 180°C, 8 min of hot-pressing time had higher weight retention than that with 5 min. But when hot-pressed at 200°C, 8 min of hot-pressing time had less weight retention than that with 5 min, indicating the cured resin might be degraded at high temperature for a long time.

The internal bonding strength can be used as an index for the bonding effect among wood powders. However, it depends not only on the curing degree of the resin, but also the hot-melting phenomenon of resin, the distribution of melted resin over the surface of wood powders, and the interaction between resin and wood. Although wood powders impregnated with C-PF had higher weight retention after acetone immersion, they had less internal bonding strength than those impregnated with S-PF. This may be due to the higher reactivity and faster curing reaction for C-PF than S-PF, which caused the resin with an insufficient melting to spread over the surface of wood powders before it cured, and resulted in a worse bonding strength. Furthermore, when hot-pressed at 180°C, using the pressing time of 8 min had higher internal bonding strength than that with 5 min. But when hot-pressed at 200°C, using 5 min of hot-press time had better internal bonding strength than that with 8 min.

The water absorption and thickness swelling of compression-molded plates were depending on their porosity, both inter- and intra-wood powders, and

the bonding strength among the wood powders. As shown in the Table, when using S-PF as the impregnating resin, hot-pressed at 200°C for 5 min or 180°C for 8 min would be better conditions. However, when C-PF resin was used, hot-pressed at 200°C for 5 min would be the better condition, it had lower water absorption and thickness swelling.

S-PF-H and C-PF-H were premixed the hexamine into resins and then impregnated into wood powders. In theory, they have more uniform distribution of hexamine within resins, and both of them would be expected to have better contact during the curing reaction. In the previous discussion of DSC thermo-setting, wood powders impregnated with S-PF-H and C-PF-H had higher thermal reactivity than those of S-PF and C-PF. However, this effect had not reflected in the properties of compression-molded plates, especially for the internal bonding strength. It may be due to the hexamine was predissolved in water and blended with the resin in a molecular form. It causes the hexamine more sensitive and more easily to decompose to formaldehyde and amine compounds under heating, which resulted in the resin with a high reactivity and a too fast curing reaction during hot-pressing. However, some of formaldehyde would evaporate because it did not have enough time to participate in the curing reaction. Furthermore, the moving of vapor of amine compounds may destroy the bonded structure. Both of these phenomena may lead a worse internal bonding strength.

Thermogravimetric analysis of compression-molded plates

Figure 4 shows the TG and DTG curves of various molds. All compression-molded plates had a drastic weight loss at a temperature between 250 and 350°C, which may be due to the decomposition of wood components. But the weight loss was sustained even when the temperature went up to 800°C which contributed to the decomposition of PF resin accompanied

TABLE III
TGA Thermodegradation Parameters of Compression Molded Plates

Resins	Hot-pressing		First-stage	Second-stage		Third-stage		Residue content (%) (800°C)
	Temp. (°C)	Time (min)	Weight loss (%)	Onset (°C)	Weight loss (%)	Onset (°C)	Weight loss (%)	
S-PF	180	5	5.9	264.0	27.7	360.5	53.2	13.2
S-PF	200	5	1.8	274.1	25.0	372.3	34.0	39.2
S-PF	200	8	2.6	278.0	29.8	364.4	29.3	38.3
S-PF-H	200	5	2.9	300.6	26.3	388.2	26.0	44.8
C-PF	200	5	2.6	283.9	31.9	367.4	35.6	29.9
C-PF-H	200	5	1.6	285.8	34.1	384.6	24.6	39.7

with the continued wood degradation. However, they had dissimilar thermodegradation behavior among various compression-molded plates. The DTG curves showed compression-molded plates made with wood powders impregnated with S-PF-H and C-PF-H (Code 4 and 6) had a rapid weight loss rate. The compression-molded plates that made with wood powders impregnated with S-PF and hot-pressed at 180°C for 5 min (Code 1) had another obvious weight loss rate peak appeared at the temperature of 500°C.

Table III shows the onset temperature and percentage of weight loss at each thermodegradation stage and the residue content after heating to 800°C. Among which, compression-molded plates made with S-PF impregnated wood powders and hot-pressed at 180°C for 5 min had lower onset temperature of degradation, higher weight loss in first and third stage, and lower residue content when heating to 800°C than the others. It indicated that hot-pressed at 180°C for 5 min was not enough to cure the resin completely, resulting in lower heat resistance. When hot-pressing temperature was raised to 200°C, the weight loss at first stage decreased, the onset temperature for second and third stage of thermodegradation increased, and had higher residue content after heating to 800°C. It indicated that compression-molded plates made with a hot-pressing temperature of 200°C had a higher heat resistance. Advance compression for compression-molded plates that hot-pressed at 200°C, using S-PF-H and C-PF-H impregnated wood powders as raw materials had higher onset temperature and residue content but lower weight loss in the third-stage, which contributed to the degradation of cured resin, indicting they had better heat resistance than those made with S-PF and C-PF impregnated wood powders.

CONCLUSIONS

The phenol-liquefied wood could be used to prepare novolak PF resins. After impregnating the resin into wood powders, the dried resin could be melted under heating and could be cured as hexamine was mixed. However, if the hexamine was premixed

with PF resins and impregnated into wood powders, they (S-PF-H and C-PF-H) had higher reactivity than that of S-PF and C-PF. All the compression-molded plates had high degree of setting reaction after being hot-pressed. However, those made with wood powders that impregnated with C-PF and S-PF had higher internal bonding strength than those with S-PF-H and C-PF-H. Compression-molded plates made with wood powders impregnated with C-PF had higher weight retention after acetone immersion, but those made with S-PF impregnated wood powders had higher internal bonding strength. The better conditions for manufacture compression-molded plates those used wood powders impregnated with C-PF and S-PF as raw materials in this study were hot-pressed at 180°C for 8 min or 200°C for 5 min.

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