

Physicomechanical and Thermal Properties of Moldings Made from Liquefied Wood-Based Novolak PF Resins Under Various Hot-Pressing Conditions

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ABSTRACT: The wood powder of *Cryptomeria japonica* (Japanese cedar) was liquefied in phenol, with H_2SO_4 and HCl as a catalyst. The liquefied wood was used to prepare the liquefied wood-based novolak phenol formaldehyde (PF) resins by reacting with formalin. Furthermore, novolak PF resins were mixed with wood flour, hexamethylenetetramine, zinc stearate as filler, curing agent, and lubricating agent, respectively, and hot-pressed under 180 or 200°C for 5 or 10 min to manufacture moldings. The results showed that physicomechanical properties of moldings were influenced by the hot-

pressing condition. The molding made with hot-pressing temperature of 200°C for 10 min had a higher curing degree, dimensional stability, and internal bonding strength. The thermal analysis indicated that using a hot-pressing temperature of 180°C was not sufficient for the liquefied wood-based novolak PF resins to completely cure. © 2009 Wiley Periodicals, Inc. *J Appl Polym Sci* 113: 1257–1263, 2009

Key words: liquefied wood; molding; novolak PF resin; thermal properties

INTRODUCTION

Solvent liquefaction is one of the effective methods for converting wood from solid to liquid. Phenol is one of the solvents commonly used for wood liquefaction, with acid, such as H_2SO_4 and HCl, used as catalysts. The acid can degrade the components of wood into small ingredients and can promote these ingredients to react with phenol to form derivatives. After then, phenol derivatives can dissolve in the free phenol. The product of phenol liquefied wood can be used as a raw material to prepare phenolic resins.

Lin et al.^{1–3} used β -O-4 lignin and cellobiose as model compounds to study the liquefaction mechanisms of lignin and cellulose in the presence of phenol with acid as a catalyst. Acemioglu and Alma⁴ investigated the kinetic of liquefaction of Monarch birch wood in phenol with HCl as a catalyst. They pointed out that wood phenolysis followed a second-order reaction of bimolecular type. Lee and Wang⁵ investigated the effect of water on wood liquefaction; they thought that water present in the liquefaction system would not only decrease the reaction temperature and reduce the liquefaction rate, but could also suppress the recondensation

reaction. Zhang et al.⁶ researched the liquefaction mechanism of cellulose in phenol, and they indicated that pyranoses decomposed from the cellulose could combine with phenol to form a hydroxyl benzyl form derivative, but retained the characteristics of phenolic functional groups, and could react with formaldehyde, just like phenol.

Novolak phenol formaldehyde (PF) resins are synthesized from the reaction of phenol with an insufficient amount of formaldehyde under acidic condition. They have no reactive methylol groups in their linear structure, and are therefore incapable of condensing with other novolak molecules under heating but can only melt. However, if formaldehyde is further added and heated at high temperature, the melted novolak PF resin will undergo the condensation reaction and complete resinification. Novolak resins are extensively used as molding materials and applied in electrical engineering, household/electrical appliances and automobile. Alma et al.^{7–9} liquefied wood and bark in phenol with HCl, H_2SO_4 , and NaOH used as catalyst and investigated the feasibility of using these liquefied products as raw materials to replace pure phenol in the manufacture of moldings. The results showed that phenolated wood materials had thermoflowability. When mixed with hexamethylenetetramine and cured under hot-pressing, they could be employed to make moldings. Nevertheless, the melting temperature of phenolated wood materials was dependent

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on the degree of combined phenol. Lin et al.^{10,11} prepared phenolated wood/phenol/formaldehyde cocondensed novolak PF resins in a two-stage procedure. The flowing temperature and melted viscosity of these cocondensed resins were similar to those of conventional novolak PF resins. Lee et al.¹² liquefied waste paper in phenol, and they pointed out that moldings could be made by mixing the liquefied waste paper with wood flour, hexamethylenetetramine, calcium hydroxide, and zinc stearate, and hot-pressed under 180°C for 5 min. However, when the liquefied waste paper was reacted with formaldehyde to form a cocondensed novolak PF resin, and used as raw materials for moldings, the flexural properties and thermal stability of moldings would be improved. Pan et al.¹³ conducted wood liquefaction in an atmospheric reactor and a sealed Parr reactor, and then both of these liquefied products were condensed with formaldehyde under an acidic condition to prepare novolak PF resins. They investigated curing kinetic mechanism using dynamic and isothermal differential scanning calorimetry (DSC) analysis and indicated that the curing reactions of these resins followed an autocatalytic mechanism.

In our previous studies,¹⁴ the phenolated bark was employed to prepare the resol type PF resins. The results showed that PF resins could be prepared for manufacturing particleboards. In addition,^{15,16} phenol liquefied wood had been used to prepare novolak PF resins, the results showed an exothermic reaction for the mixture of liquefied wood and formalin, and a solid-like novolak resin would form without extra heating. The liquefied wood-based novolak PF powder displayed a thermomelting characteristic, and could be used as a binder for making moldings. However, as well known, the fluidity and curing degree of the binder are the most important factors influencing the properties of moldings. The effect of the hot-pressing temperature and the hot-pressing time for liquefied wood-based novolak PF resins may be different from those for conventional novolak PF resins. In this study, effects of hot-pressing conditions on properties of moldings made using liquefied wood-based novolak PF resins as a binder were investigated.

EXPERIMENTAL

Materials

Wood of Japanese cedar was air-dried and ground to powder with a hammer mill. The wood powder with a dimension passing 20 mesh was used for liquefaction, and the dimension passing 200 mesh was used as the filler for moldings. All wood powder was oven dried at 105°C for 12 h before being used. Chemicals such as sulfuric acid (H₂SO₄),

hydrochloric acid (HCl), phenol, formalin (37% aq), hexamethylenetetramine, zinc stearate, and acetone were of reagent grade and were used without further purification.

Liquefaction of Japanese cedar

The powder of Japanese cedar was liquefied in phenol with H₂SO₄ or HCl as a catalyst. The procedure used for liquefaction was the same as described in our previous article.¹⁵ For the liquefaction using H₂SO₄ as a catalyst, the weight ratio of phenol/wood/H₂SO₄ was 2.5/1/0.125, and the reaction took place under 130°C for 60 min. Contents of the unliquefied wood residue and the unreacted free phenol in the liquefied product were 1.4% and 21.8%, respectively. For liquefaction using HCl as a catalyst, the weight ratio of phenol/wood/HCl was 3/1/0.3, and the reaction took place under 110°C for 60 min. The content of unliquefied wood residue and the unreacted free phenol in the liquefied product were 4.2% and 49.8%, respectively.

Preparation of liquefied wood-based novolak PF resin

When preparing liquefied wood-based novolak PF resins, the molar ratio of free phenol in the liquefied wood to formaldehyde was 1/0.8. The liquefied wood and formalin were mixed in a beaker and kept in a sustained stirring without any external heating. An exothermic reaction took place during stirring. The temperature of the reactant rose spontaneously and finally formed a solid-like resin when the temperature reached its maximum. The solid-like resins were dehydrated in an oven at 105°C, and then ground to powder with the dimension passing a 100-mesh screen. Two liquefied wood-based novolak PF resins were prepared from the liquefied wood that used H₂SO₄ and HCl as catalyst, which were represented as SF and CF, respectively.

Manufacturing of moldings

To manufacture moldings, the liquefied wood-based novolak PF resin powder, wood flour, hexamethylenetetramine, and zinc stearate were mixed and used as the binder, filler, curing agent, and lubricating agent, respectively. Weight ratios of novolak PF resin/wood flour/hexamethylenetetramine/zinc stearate used for the mixture were 40/50/10/1 and 60/30/10/1. After all components were mixed completely, flat moldings with a dimension of 10 × 10 × 0.4 cm³ and a density of 1.0 g/cm³ were made by hot-pressing at the temperature of 180 and 200°C for 5 and 10 min at a pressure of 20 kgf/cm².

Physicomechanical properties of moldings

The moldings were cut into specimens with a dimension of $2 \times 2 \times 0.4 \text{ cm}^3$ and utilized to determine the density, internal bonding strength, and water absorption properties. The internal bonding strength was measured by a universal strength testing machine (Shimadzu UEH-10) using a load vertical to the surface of the flat molding at a crosshead speed of 2 mm/min. The water immersion test was carried out by dipping specimens into water, and measuring the percentage of water absorption and swelling thickness at the time of 2, 24, and 36 h. In addition, some moldings were ground to powder and dried in a vacuum oven at 60°C for 24 h. About 0.2 g of the powder was immersed in acetone (about 20 mL) for 24 h, followed by filtrating and calculating the weight retention.

Thermal properties of moldings

Thermal properties of moldings were determined with the differential scanning calorimeter (DSC) (Perkin-Elmer DSC-7) and thermal gravimetric analyzer (TGA; Perkin-Elmer Pyris 1). For DSC analysis, 3 mg of sample was sealed in an aluminum capsule and heated from 30 to 500°C at a heating rate of 10°C/min. The variation in heat flow during the period of heat scanning was detected and the relative thermal parameters were calculated. For TGA, about 10 mg of sample was put into a platinum sample pan and heated from 50 to 700°C at a heating rate of 10°C/min under a nitrogen atmosphere. Curves of weight loss (TG) and derivative TG (DTG) were plotted, and the weight loss at various temperatures was calculated.

RESULTS AND DISCUSSION

Properties of liquefied wood-based novolak PF resins

Two kinds of novolak PF resin, SF and CF, were prepared using liquefied wood as raw materials. As described in our previous study,¹⁵ the liquefied wood could react with formalin to prepare novolak PF resins without any extra heating. Among which, SF and CF have the melting point of 149.4 and 127.5°C, respectively, and have the weight-average molecular weight of 3638 and 3941, respectively. The DSC analysis shows that SF and CF, after mixing with wood flour, hexamethylenetetramine, and zinc stearate, have the onset temperature of curing reaction of 143.1 and 127.9°C, respectively, and have the peak temperature of curing reaction of 237.2 and 223.7°C, respectively.

Properties of moldings

Moldings were made by first mixing liquefied wood-based novolak PF resins with wood flour, hexamethylenetetramine, and zinc stearate, followed by hot-pressing. Table I shows manufacturing conditions and properties of moldings. When soaked with acetone, wood powder of Japanese cedar used as the contrast had weight retention of 94.3%, indicating that some extractives were dissolved from the wood. As shown in Table I, all the moldings had weight retention higher than 90.2%. This result indicates that the liquefied wood based-novolak PF resin in the molding was resinificated by hot-pressing. Comparison shows that moldings made with the formula of 40 : 50 : 10 (S1 and C1) had lower weight retention than those made with the formula of 60 : 30 : 10,

TABLE I
Manufacturing Conditions and Properties of Moldings Made with Liquefied Wood-Based Novolak PF Resin

Code of resin*	Code of molding	Manufacturing conditions			Properties of moldings		
		Binder : wood flour : curing agent (by wt)	Hot-pressing temperature (°C)	Hot-pressing time (min)	Density (g/cm ³)	Weight retention (%)	Internal bonding strength (kgf/cm ²)
SF	S1	40 : 50 : 10	180	5	1.00	90.2	–
	S2	60 : 30 : 10	180	5	0.94	91.3	–
	S3	60 : 30 : 10	180	10	0.92	92.3	7.0
	S4	60 : 30 : 10	200	5	0.92	93.9	13.2
	S5	60 : 30 : 10	200	10	0.97	95.3	15.9
CF	C1	40 : 50 : 10	180	5	0.99	90.3	–
	C2	60 : 30 : 10	180	5	0.89	93.8	–
	C3	60 : 30 : 10	180	10	0.91	96.8	5.7
	C4	60 : 30 : 10	200	5	0.91	97.2	11.3
	C5	60 : 30 : 10	200	10	0.94	97.6	11.5
Wood powder	–	–	–	–	94.3	–	

* SF and CF were the novolak PF resin that prepared from the Japanese cedar wood liquefied with H₂SO₄ and HCl as catalyst, respectively.

TABLE II
Water Absorption and Thickness Swelling of Moldings
Made with Liquefied Wood-Based Novolak PF Resin

Code of molding	Water absorption (%)			Thickness swelling (%)		
	2 h	24 h	36 h	2 h	24 h	36 h
S1	3.2	11.0	13.8	1.2	3.4	4.4
S2	2.6	9.5	11.7	1.3	2.3	2.5
S3	2.4	7.9	9.9	1.0	2.0	2.0
S4	2.2	6.9	8.6	1.3	1.8	1.8
S5	1.8	6.2	7.4	0.8	1.3	1.3
C1	2.2	6.8	8.0	0.7	1.7	2.5
C2	1.7	5.3	6.7	0.3	0.3	1.3
C3	2.0	6.3	7.4	0.3	1.0	1.7
C4	2.0	6.3	8.1	0.3	1.0	1.7
C5	2.2	6.4	7.6	0.3	1.3	1.3
Solid wood	35.3	70.6	—	4.2	7.7	—

suggesting that properties of moldings improved when using a higher rate of resin. Comparison also shows that moldings made with CF as the binder had higher weight retention than those made with SF, indicating that the resin of CF had a more complete crosslinking structure than that of SF after being hot-pressed. Moreover, increasing the hot-pressing temperature or prolonging the hot-pressing time could lead to more complete curing reaction of the resin, and could increase weight retention. However, the molding of C5 made with CF as a binder and hot-pressed at 200°C for 10 min had the highest weight retention.

The internal bonding strength of moldings was dependent on complex factors such as the curing degree of the resin, the hot-melting phenomenon of glue powder, the distribution of melted glue over the surface of wood filler, and the interaction between PF resin and the surface of wood filler. Table I shows that moldings made with hot-pressing temperature of 200°C had higher internal bonding strength than those hot-pressed at 180°C. This is because hot-pressing at a higher temperature could enhance the fluidity of the melted resin, and form a more uniform distribution of the melted glue on the surface of wood filler, thus providing a better inter-

nal bonding strength for moldings. Comparison shows that moldings made with SF as the binder had higher internal bonding strength than those made with CF.

Water adsorption and dimensional stability of moldings

Table II shows the water adsorption and swelling thickness of moldings after being immersed in water. As can be seen, water adsorption percentages of the solid wood of Japanese cedar that was used as a contrast were 35.3 and 70.6% after being immersed in water for 2 and 24 h, respectively. However, the water adsorption percentage was much lower for moldings than for solid wood. The water adsorption percentage for various moldings at the immersion time of 2 h was between 1.8 and 3.2%. When the immersion time was prolonged to 36 h, S1 had the highest water adsorption percentage though it was only 13.8%. Comparing between SF and CF, except for S5 and C5 which were hot-pressed under 200°C for 10 min, shows that moldings made with CF as a binder had lower water adsorption percentage than those made with SF. Nevertheless, for moldings made with SF as the binder, increasing the hot-pressing temperature or prolonging the hot-pressing duration could reduce water adsorption percentage, though this tendency was not found for moldings using CF as the binder. This was because the resin of SF had lower reactivity than that of CF; therefore, providing more heat could lead to more complete curing reaction, and could reduce water adsorption.

The swelling thickness of solid wood of Japanese cedar immersed in water for 2 and 24 h was 4.2 and 7.7%, respectively. However, all the moldings had reduced swelling thickness even after being immersed in water for 36 h. Moreover, the swelling thickness of S5 and C5, which were hot-pressed under 200°C for 10 min, was only 1.3%, indicating that moldings made with liquefied wood-based

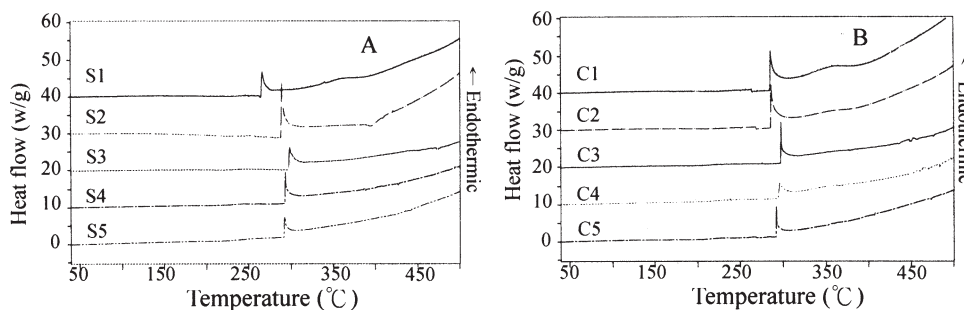


Figure 1 DSC thermograms of moldings made with liquefied wood-based novolak PF resins [A and B were moldings made with SF and CF, respectively].

TABLE III
DSC Thermoanalysis Parameters of the Endothermic Peak of Moldings Made with Liquefied Wood-Based Novolak PF Resin

Code of molding	Onset temp. (°C)	Peak temp. (°C)	Height of peak (w/g)	Heat of reaction (j/g)
S1	265.2	266.6	5.882	127.4
S2	288.5	289.5	14.886	300.1
S3	297.6	299.3	5.566	158.2
S4	293.2	293.7	8.500	163.7
S5	291.4	292.7	4.980	108.5
C1	285.2	285.7	10.520	212.5
C2	285.8	286.7	11.157	288.6
C3	297.4	297.9	10.600	119.9
C4	294.3	296.0	3.991	118.0
C5	291.8	292.3	7.903	107.4

novolak PF resin as a binder had good dimensional stability.

DSC thermoanalysis of moldings

In our previous research,¹⁵ the DSC analysis showed that dried phenolated wood had an endothermic peak at the temperature between 270 and 280°C, which was caused by thermodegradation of wood components. Figure 1 shows DSC thermograms of moldings made with SF and CF as the binder. Among which, all moldings showed an endothermic peak at the temperature of 300°C. It was attributed to the thermodegradation of wood components in the moldings, but the temperature was higher than that of the liquefied wood mentioned earlier. This indicates that the liquefied wood-based novolak PF resin existing around the wood fillers underwent a crosslinking reaction with hexamethylenetetramine and formed a cured resin by hot-pressing. Therefore, the heat resistance of the wood filler increased. Table III shows the thermoanalysis parameters of the endothermic peak of moldings. Moldings made with the hot-pressing temperature of 180°C for 10 min, i.e., S3 and C3, had the highest onset temperature and peak temperature.

Figure 2 shows heat flow curves during the DSC analysis at the temperature lower than that at which thermodegradation occurred. As shown in Figure 2(A,B), moldings made with a hot-pressing temperature of 180°C for 10 min (S1, S2, C1, and C2) had a significant exothermic peak due to the postcuring reaction, indicating that the binder in these moldings did not cure completely after being hot-pressed. Comparison shows that moldings using SF as the binder had a more significant postcuring phenomenon than that using CF as the binder. For moldings hot-pressed at the temperature of 200°C, a slight postcuring phenomenon was observed in S4 and S5, but not in C4 and C5.

Table IV shows the onset temperature and peak temperature of the exothermic peak. The molding of S2 had the onset temperature for postcuring reaction at 167.9°C, but it was 188.6°C for S3 when the hot-pressing duration was prolonged to 10 min. In addition, increasing the hot-pressing temperature to 200°C, such as the case of S4 and S5, led to a higher onset temperature than that of S3. The higher onset temperature for moldings made with a longer hot-pressing duration and a higher temperature might be due to the more complete crosslinking reaction under hot-pressing. This causes them to be less movable, and a higher temperature will be needed to undergo the postcuring reaction.

TGA of moldings

Figure 3 shows the TGA and DTG thermal gravity analysis curves of moldings made under various hot-pressing conditions. Lee et al.¹² pointed out that moldings made from liquefied wood or liquefied waste paper with hexamethylenetetramine as the curing agent would thermodegrade at the temperature of 350°C. In Figure 3(A-1, B-1), all the moldings showed a rapid weight loss starting at the temperature of 300°C. This temperature is the same as that obtained by DSC analysis, which shows a significant endothermic peak. Alma et al.⁷ had pointed out that moldings made from novolak PF resin with a more

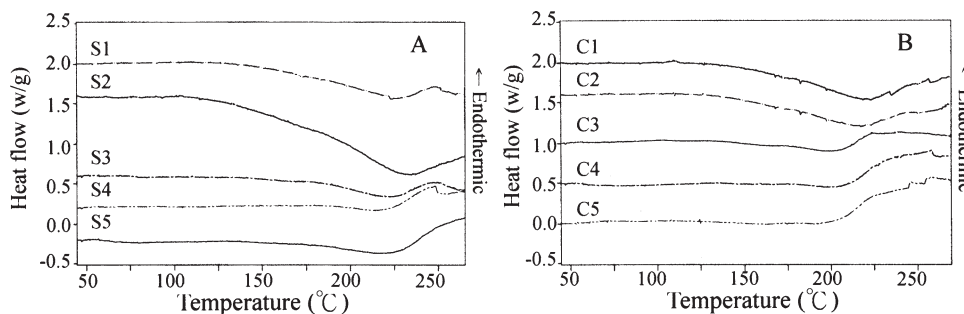


Figure 2 Heat flow curves of postcuring of moldings made with novolak PF resins at the DSC thermoanalysis [A and B were moldings made with SF and CF, respectively].

TABLE IV
The Postcuring Temperature of Moldings Made with Liquefied Wood-Based Novolak PF Resin Measured with DSC Thermoanalysis

Code of molding	Onset Temp. (°C)	Peak temp. (°C)	Code of molding	Onset temp. (°C)	Peak temp. (°C)
S1	155.9	225.3	C1	171.9	218.5
S2	167.9	226.7	C2	174.6	217.1
S3	188.6	222.7	C3	177.0	199.8
S4	196.7	213.7	C4	–	–
S5	187.7	222.8	C5	–	–

complete crosslink curing reaction would had higher heat resistance. Comparison shows that thermodegradation was more moderate for moldings made with CF as raw material [Fig. 3(B-1)] than that made with SF [Fig. 3(A-1)]. Moreover, moldings made with CF had more char residue than those made with SF at the temperature of 800°C. These results suggest that moldings made with CF had a more complete crosslinked structure, and had better heat resistance than those made with SF. This result can also be used to explain the higher weight retention after moldings made with CF were soaked in acetone, as shown in Table I.

Comparing the thermal property of moldings made with formulas 40 : 50 : 10 and 60 : 30 : 10

shows that the former (S1 and C1) had a more rapid thermodegradation and less char residue than the latter. This may be due to the former having a lower resin dosage than the latter, which resulted in an insufficient covering of melted resin over the surface of wood fillers, and caused the molding to have a loose construction and a lower heat resistance. Comparing among moldings made with SF hot-pressed under various conditions shows that moldings of S2, S3, and S5 had a similar thermal weight loss curve, but S4, which was hot-pressed under 200°C for 5 min, had a more moderate thermal weight loss. However, with CF as raw material, C3, which was hot-pressed at 180°C for 10 min, had a more moderate thermal weight loss.

The derivative thermogravimetry analysis (DTA) curves are shown in Figure 3(A-2, B-2). As can be seen, S1 showed only a single significant thermodegradation peak over a wide temperature range, indicating that the thermal degradation reaction of S1 was a continuous process with increase in temperature. However, for S2, S3, S4, and S5 made with a higher dosage of resin, their DTG curves showed two thermal degradation peaks. The first peak appearing at the temperature of 200–400°C was due to the thermodegradation of wood components, and the second peak appearing at the temperature about 450°C was due to the thermodegradation of cross-

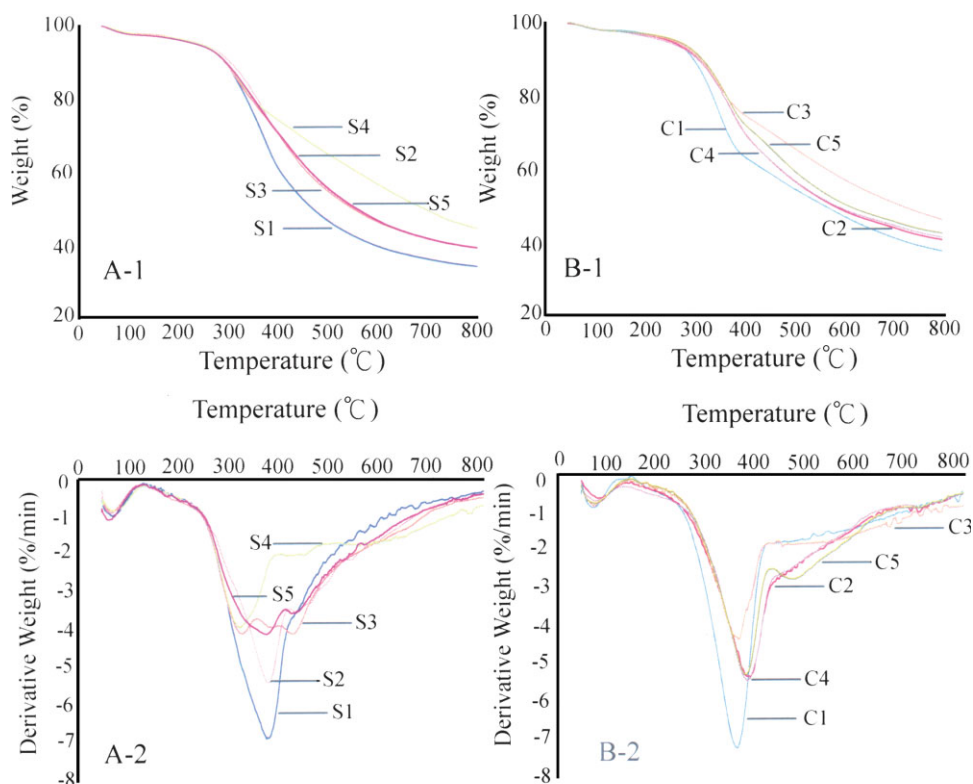


Figure 3 TGA and DTG curves of molding made with various hot-pressing conditions [A and B were moldings made with SF and CF, respectively] [Color figure can be viewed in the online issue which is available at www.interscience.wiley.com.]

TABLE V
Thermal Weight Loss Percents of Moldings Made with Liquefied Wood-Based Novolak PF Resins at Various Degradation Temperatures

Code of molding	Thermal weight loss (%)					
	300°C	400°C	500°C	600°C	700°C	800°C
S1	11.54	39.37	53.84	60.85	64.39	66.49
S2	9.81	30.21	45.33	54.24	59.02	61.52
S3	11.50	30.20	45.92	54.39	58.89	61.38
S4	11.00	26.18	35.60	43.93	51.14	56.12
S5	11.02	29.74	45.01	53.90	58.79	61.40
C1	10.57	36.89	45.78	53.30	59.13	62.86
C2	9.35	30.26	43.61	52.16	56.49	59.89
C3	8.72	25.58	34.75	43.28	49.81	54.50
C4	9.51	29.63	43.45	51.52	55.83	59.17
C5	8.19	28.01	40.84	49.98	54.79	58.05

linked PF resin in moldings. However, S2, which was hot-pressed at 180°C for 5 min, had the first decomposition peak more obvious than the others, indicating that its degree of crosslinking was lower than those hot-pressed for a longer duration or at a higher temperature. In Figure 3(B-2), with the resin of CF used as the binder, C1 showed a single but significant thermodegradation peak as seen in S1. For other moldings, C5 showed two thermodegradation peaks, but the variation in thermodegradation rate after the first peak appeared was slowed down for the other three moldings. These results showed that the moldings made with CF resin as the raw material had higher heat resistance than those made with SF resin.

Table V shows the relationship between the heating temperature and the weight loss of moldings during the TGA. They had a drastic weight loss at the temperature between 300 and 400°C, after then, the weight loss became moderate gradually. After being heated up to 800°C, moldings made with CF resin had lower weight loss than those made with SF resin under the same manufacturing condition. Moreover, comparison in this table shows that, for moldings made with SF as raw material, hot-pressing at a temperature of 200°C for 5 min resulted in the lowest weight loss. On the other hand, those made with CF as raw material, hot-pressing at 180°C for 10 min led to the lowest weight loss.

CONCLUSIONS

Novolak PF resins of SF and CF were prepared by reacting phenol liquefied Japanese cedar with formalin. These novolak PF resins could be employed to manufacture moldings by mixing with wood flour, hexamethylenetetramine, and zinc stearate and set under hot-pressing. Moldings made with CF as raw material had a higher weight retention than those

made with SF after being immersed in acetone. However, moldings made with SF had higher internal bonding strength than those made with CF. Moldings hot-pressed at temperature of 200°C had better physicomechanical properties than those at 180°C. The DSC analysis indicated moldings hot-pressed at temperature of 180°C were not completely cured. The TGA showed that the thermodegrading of moldings occurred at the temperature up to 300°C. Their thermodegradation involved two stages, the first stage appeared at 200–400°C whereas the second stage took place around 450°C. Moldings made with CF resin as the binder had higher char content than those made with SF at the heating temperature of 800°C.

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