Thermal Curing Behavior of Modified Urea-Formaldehyde Resin Adhesives with Two Formaldehyde Scavengers and Their Influence on Adhesion Performance

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Received 5 October 2007; accepted 15 November 2007 DOI 10.1002/app28748 Published online 25 July 2008 in Wiley InterScience (www.interscience.wiley.com).

ABSTRACT: This study investigated, by differential scanning calorimetry, the thermal curing behavior of urea–formaldehyde (UF) resins modified with two formaldehyde scavengers and its impact on the adhesion performance of particleboard bonded with modified UF resin adhesives. As the concentration of the two scavengers [i.e., urea–formaldehyde prepolymer (UFP) and urea solution (US)] increased, the gel time, peak temperature, and onset temperature of the modified UF resins, but these parameters increased for the US-modified UF resins. These results indicated that the reactivity of the UF resin modified with UFP was maintained, but it deteriorated for the US-modified UF resins. The formaldehyde emission of particleboard bonded with the modi-

INTRODUCTION

Urea-formaldehyde (UF) resin is a polymeric condensation product of the chemical reaction of formaldehyde with urea, and it is regarded as one of the most important types of adhesives in the woodbased panel industry. Among the formaldehydebased resin adhesives, UF resin adhesives have been extensively used in the manufacture of particleboard and medium-density fiberboard. Therefore, UF resin adhesives are mainly consumed by the wood panel industry. In Korea, the production of formaldehydebased resins was about 207,000 tons in 2005, which was 39% of the total production of adhesives. The production of UF resin adhesives was about 75% (i.e., ca. 155,000 tons) of the total production of formaldehyde-based resin adhesives. Compared to other wood adhesives, such as phenol-formaldehyde resfied UF resin decreased with an increase in the scavenger concentration. UFP was more effective than US in scavenging the formaldehyde emission and in achieving adhesion to the UF resin. The results showed an optimum UFP level of 20% in the UF resin for maintaining a balance between the formaldehyde emission and adhesion of the particleboard, and they indicated that both the thermal curing behavior of scavenger-modified UF resins and the properties of particleboard bonded with them must be taken into account in the evaluation of a formaldehyde scavenger system. © 2008 Wiley Periodicals, Inc. J Appl Polym Sci 110: 1573–1580, 2008

Key words: adhesion; calorimetry; curing of polymers; thermal properties; thermosets

ins and diphenylmethane diisocyanate, UF resin adhesives possess some advantages such as fast curing, good performance in wood panels, water solubility, and a lower price. The disadvantages of using UF resins include formaldehyde emissions from wood panels and lower resistance to water. Lower resistance to water limits the use of wood-based panels bonded with UF resins to interior applications.

Free formaldehyde that is present in UF resins and hydrolytic degradation of UF resins under moist and acidic conditions are known to be responsible for formaldehyde emissions from wood-based panels.¹ In other words, the unreacted formaldehyde in a UF resin after its synthesis can be emitted from wood panels even after hot pressing at a high temperature. In addition, the reversibility of the aminomethylene link and its susceptibility to hydrolysis also explain the lower resistance against the influence of water and moisture and, consequently, formaldehyde emissions from wood-based panels.² Therefore, the formaldehyde emission issue has been one of the most important aspects of UF resins in the last few decades.^{3–8}

Much attention has been paid to reducing or controlling formaldehyde emissions from UF-resin-

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Contract grant sponsor: Agricultural R&D Promotion Center (Seoul, Korea); contract grant number: 303003.

Journal of Applied Polymer Science, Vol. 110, 1573–1580 (2008) © 2008 Wiley Periodicals, Inc.

bonded panels through resin technologies. In general, UF resins are synthesized by a two-step reaction procedure (i.e., methylolation under alkaline conditions and condensation under acidic conditions with a large amount of formaldehyde).⁹ This synthesis method has been widely employed for the preparation of UF resins for many years. In the early 1970s, however, this method was faced with the serious problem of formaldehyde emissions. Therefore, lowering the formaldehyde-to-urea (F/U) molar ratio for the synthesis of UF resins was adopted as one of the approaches to reducing formaldehyde emissions of UF-resin-bonded panels.⁶ Thus, lower F/U molar ratios from 1.1 to 1.2 were employed for the resin synthesis. An excellent literature review on the influence of the F/U molar ratio on formaldehyde emissions, as well as panel properties, was published.¹⁰ According to the review, the gel time increases with a decrease in the F/U molar ratio. In general, lower F/U molar ratios cause less formaldehyde emission, resulting in a loss of panel properties, particularly the internal bond (IB) strength, as well as thickness swelling (TS) after water immersion for 24 h. Lower F/U molar ratios also reduce the modulus of rupture (MOR).⁶ In recent years, it has been reported that a lower F/U molar ratio (i.e., 1.0) can reduce the formaldehyde emissions of particleboard.¹¹

Another approach to abating the formaldehyde emissions of UF resin adhesives is the use of formaldehyde scavengers that are added to UF resins before they are blended with wood materials.¹² Formaldehyde scavenger systems that are being used for wood adhesives include scavenger resins, scavenger wax, and urea in either a solution or dry form. The use of scavenger systems varies, depending on the location and mill conditions.¹² In general, low F/U molar ratio UF resins, scavenger resins, and urea solution (US) are the three main systems used in North America. The use of scavenger systems also requires maintaining the reactivity or cure rate of modified UF resins; otherwise, the productivity will be degraded. Thus, the thermal curing behavior of scavenger-modified UF resins is extremely important in reducing formaldehyde emissions and maintaining productivity.

Many studies on the thermal curing of UF resins using differential scanning calorimetry (DSC) have been published.^{13–19} For example, exothermic peak temperatures of UF resins, depending on the types of hardeners, range from 75 to 100°C, and the heat of reaction (ΔH) ranges from 26 to 154 J/g.¹⁶ Similar results for peak temperatures, ranging from 80 to 85°C, have also been reported.¹⁴ DSC has also been used to monitor the thermal curing behavior of UF resins modified with the addition of amines to lower residual stress once they have been cured. It has been reported that there is a decrease in the peak temperature with an increase in the amount of amines added.¹⁷ In recent years, the thermal behavior of commercial UF resins has been characterized with thermogravimetry/differential thermal analysis techniques, and exothermic peak temperatures between 83 and 86°C and decomposition of resins above 200°C have been reported.¹⁸ In addition, a ΔH value of 15.27 J/g and an activation energy (E_a) of 66.7 kJ/mol have been reported for a control UF resin not precured for a certain period of time.¹⁹

Even though these scavenger systems have been known to be effective in reducing the formaldehyde emissions of wood panel products, their specific influence on the thermal curing behavior of UF resin adhesives has not been well elucidated. In fact, there are few results regarding the thermal curing behavior of modified UF resins with formaldehyde scavengers. In addition, the influence of the thermal curing behavior of scavenger-modified UF resins on the properties of particleboard bonded with them has not been fully evaluated. Therefore, in this study, using DSC, we have attempted to investigate the thermal curing behavior of a UF resin modified with two different scavengers and to evaluate the properties of particleboard bonded with modified UF resin adhesives.

EXPERIMENTAL

UF resin synthesis

The UF resin used in this study was prepared with commercial urea pellets and formalin (37%) to obtain a target F/U molar ratio of 1.0. Its synthesis followed a traditional two-step reaction under alkaline-acid conditions. The UF resin was synthesized in a four-necked, 1-L reactor, which was charged with formaldehyde under stirring. The pH was adjusted to 7.8 with 20% sodium hydroxide. After the components were heated to 40°C in the reactor, the first urea was slowly added to the reactor over a 10-min period to obtain an initial F/U molar ratio of 2.0. When the temperature rose to 90° C, the reaction was maintained for 60 min. The temperature was lowered to 80°C, and the pH was also adjusted to 4.6 with 20% formic acid. The temperature was held at 80°C until the turbidity point. The resin was then cooled to room temperature, and the second urea was added to get the final F/U molar ratio of 1.0. After cooling, the pH of the resin was adjusted to 8.0. The nonvolatile solid content of the prepared resins was determined by a pan solid technique. The nonvolatile solid content, pH, and viscosity of the prepared UF resin were 60.4%, 8.0, and 470 cps, respectively.

Concentrations of the Two Scavengers									
	Scavenger mixing ratio (UF resin : scavenger)								
Scavenger type	100:0	90:10	80:20	70:30	50 : 50				
UFP US	60.4 60.4	58.4 58.2	56.5 54.8	54.8 51.9	50.7 48.5				

 TABLE I

 Nonvolatile Solid Contents of Modified UF Resins Depending on the Various Concentrations of the Two Scavengers

Preparation of the scavengers

Urea–formaldehyde prepolymer (UFP) was synthesized under alkaline conditions with a procedure similar to the initial addition reaction of the UF resin synthesis. The final F/U molar ratio was adjusted to 0.5. Details of the preparation of UFP have already been patented.²⁰ The pH and viscosity of UFP were 7.1 and 16.7 cps, respectively. The amount of free formaldehyde was 0.045% with respect to UFP, and this was determined by the method described previously. US was simply prepared through the dissolution of dry urea in distilled water to obtain a concentration of 40 wt %.

Mixing the UF resin with the scavengers

The two prepared scavengers were mixed with the UF resin in different proportions (100 : 0, 90 : 10, 80 : 20, 70 : 30, and 50 : 50) on the basis of the non-volatile solid content, and this resulted in different nonvolatile solid contents of the modified UF resins, as shown in Table I.

Measurement of the gel time

To compare the reactivity of the synthesized UF resins, the gel times of both neat and modified resins were measured by the addition of 3% NH₄Cl as a hardener with a gel time meter (Davis Inotek Instrument, Charlotte, NC) at 100°C. An average of three replications was reported.

DSC measurement

A DSC instrument (Q10, TA Instrument, United States) with high-pressure cells was used to evaluate the curing behaviors of the UF resins modified with the two different scavengers at a heating rate of 10° C/min under nitrogen gas as a purge gas at a flow rate of 50 mL/min. After the addition of 3% NH₄Cl to the modified UF resin, 3–5 mg of the modified UF resins was weighed in the high-pressure cell before scanning with a minimum of two replications per sample. For each sample, the onset temperature, peak temperature, ΔH , reaction order (*n*), and degree of conversion (α) were obtained by an analysis of the two DSC curves, and the average

value was given. A single dynamic scan method was used to calculate E_a of the prepared UF resins.²¹ This method is based on using a single heating run to analyze a curing reaction that is assumed to have *n*th-order kinetics:

$$d\alpha/dt = \operatorname{Zexp}(-E_a/RT)(1-\alpha)^n \tag{1}$$

where the rate of the curing reaction $(d\alpha/dt)$ is dependent on the pre-exponential factor or Arrhenius frequency factor [Z (s⁻¹)], E_a (J/mol), the gas constant [R (8.314 J/mol K)], and the absolute temperature [T (K)].

Manufacture of particleboard and determination of its properties

Commercial wood particles including at least 50% recycled chips, donated from a local particleboard mill, were dried to about 2% moisture content before being blended with UF resins. Liquid modified UF resins with formaldehyde scavengers were sprayed onto the dried particles in a rotary drum blender. The resins were applied at a concentration of 10 wt % resin solids with respect to the oven dry weight of the particle. Before the resin application, an emulsified wax (40 wt % solids) was applied at a concentration of 1 wt % (with respect to the oven dry weight of the particle) onto the furnish in the same blender. A pumping system in conjunction with a pressurized atomizing air stream was used to deliver and atomize the wax and resin.

The dried particles were felted into a mat (30 cm \times 35 cm) with a target density of 700 kg/m³. A singlelayer mat was prepared, and then it was hot-pressed at 180°C for 5 min. The initial pressure was allowed to increase to 4.5 MPa, and then the pressure was completely released over the last 30 s. The specific conditions for hot pressing are presented in Table II.

All mechanical properties of the particleboard, including MOR, the modulus of elasticity (MOE), and the IB strength, were determined according to standard procedures.²² TS and the water absorption (WA) were also measured through the soaking of six specimens (50 mm \times 50 mm) in cold water (20°C) for 24 h. The formaldehyde emissions of the particleboard were determined with a 24-h desiccator method.²²

Particleboard Preparation					
Parameter	Value				
Board size	18.0 mm \times 300 mm \times 350 mm				
Target board density	700 kg/m^3				
Wax content	1 wt % (based on the oven dry weight of the particle)				
Particle moisture content	2% before blending				
Resin content	8 wt % (based on the oven dry weight of the particle)				
	dry weight of the parti				

TABLE II Specific Values of Hot-Pressing Parameters for Particleboard Preparation

RESULTS AND DISCUSSION

Figure 1 shows the gel time of modified UF resins with UFP or US. As the scavenger portion in the UF resin increased, the gel time did not change much for the UFP modification, whereas it gradually increased up to a 30 wt % US addition and then dramatically increased up to a 50 wt % US addition, but the gel time of the UF resin modified with 50 wt % UFP slightly increased. Within the range of the scavenger concentrations, the gel time of the UF resin was greater for UFP than that of US. These results suggest that the reactivity of the modified UF resins with UFP was greater than that of those resins with US. This result can be attributed to the presence of methylolated ureas that were formed in UFP, which were a product of the initial addition reaction between formaldehyde and urea under alkaline conditions. In other words, methylolated ureas were able to subsequently form methylene linkages during the curing of the modified UF resin. In contrast, the urea in US did not possess methylolated ureas; more time was required to cure the modified UF resin as the concentration of US increased.

The DSC curves of the modified UF resins with either UFP or US at different scavenger concentrations



Figure 1 Gel times of modified UF resins with two different scavengers.



Figure 2 Onset temperatures of modified UF resins with two different scavengers.

provided thermal curing parameters such as the onset temperature, peak temperature, heat of reaction, and rate constants. First, the onset temperatures of the modified UF resins are shown in Figure 2. The onset temperature is defined as the temperature that is linearly extrapolated by any transition or phase change as determined from a DSC curve. Thus, the onset temperature may be expressed as an extrapolated and starting temperature of the curing of the UF resin under acidic conditions. As the concentration of the two scavengers increased, the onset temperature continuously increased from about 83.2 to 89°C for the US-modified UF resins, whereas it slightly decreased from about 83.2 to 82.3°C, except with the addition of 30 wt % UFP. The previous study showed that the onset temperature of the UF resin depended on the F/U molar ratio, ranging from about 84.1 to 72.6°C for F/U molar ratios ranging from 1.0 to 1.6.¹¹ In this study, the onset temperature of the neat UF resin in this study was within the range, whereas the onset temperatures of the modified UF resins were much greater than those of the previous study.

This result indicates that the UFP-modified UF resin had a faster start of polymerization than the US-modified UF resin. In other words, the addition of US to the UF resin retarded the initiation of curing, whereas the addition of UFP slightly accelerated the initiation of curing. Again, this result was possibly due to the presence of different chemical species in the scavenger. UFP possessed reactive methylolated ureas, whereas US did not.

The peak temperature, at which the conversion rate of the UF resin reached a maximum, is presented in Figure 3, which shows changes in the peak temperature of the modified UF resins. This parameter also indicates the reactivity of the UF resin, as does the gel time. As the scavenger concentration



Figure 3 Peak temperatures of modified UF resins with two different scavengers.

increased, the peak temperature of the UFP-modified UF resin did not change much, except with the addition of 50 wt % UFP, whereas that of the US-modified UF resin continuously increased. The USmodified UF resin showed a greater increase in its peak temperature than the UFP-modified one. These results show that the addition of US to the UF resin reduced the reactivity of the UF resin much more than the addition of UFP. In other words, the addition of UFP maintained the reactivity of the UF resin, except with the addition of 50 wt % UFP. This result indicates that the addition of UFP was more effective than that of US in simultaneously scavenging formaldehyde and maintaining UF resin reactivity.

To compare the reactivities of the modified UF resins with the two different scavengers, the rate constants of the modified UF resins, depending on the scavenger concentration, are presented in Figures 4 and 5. Figure 4 illustrates changes in the rate constant of the UF resin modified with the addition of



Figure 5 Rate constants of modified UF resins with US.

UFP. In general, the UF resin rate constant increased as the UFP concentration and temperature increased. As expected, this result is compatible with the results for the gel time, onset temperature, and peak temperature. Figure 5 shows the rate constant of the US-modified UF resin as a function of the temperature and US concentration. The rate constant increased with an increase in temperature, but it decreased with an increase in the concentration of US in the UF resin. This result explains the occurrence of higher peak temperatures of UF resins when they were modified with the addition of US. In other words, a lower rate constant resulted in a higher peak temperature for US-modified UF resins.

The E_a levels of the modified UF resins, depending on the concentration of either UFP or US, are shown in Figure 6. When 10 wt % US was added to the UF resin, E_a of the resultant UF resin jumped from about 300 to about 542 kJ/mol, and then it gradually decreased to 358.7 kJ/mol. When 10 wt % UFP was added to the UF resin, however, E_a only



Figure 4 Rate constants of modified UF resins with UFP.



Figure 6 E_a of modified UF resins with two different scavengers.

Journal of Applied Polymer Science DOI 10.1002/app



Figure 7 ΔH of modified UF resins with two different scavengers.

slightly increased to 349 kJ/mol, and then it decreased to 258.2 kJ/mol. E_a of the modified UF resins increased at a lower concentration and then decreased as both the US and UFP concentrations increased. In general, the E_a values of the UFP-modified UF resins were much lower than those of the US-modified ones. This result suggests that the UFP-modified UF resins required less energy to start the curing process than the US-modified UF resins.

The E_a values of the UF resins that were prepared under alkaline, weakly acidic, and strongly acidic conditions were about 78, 94.8, and 152.2 kJ/mol, respectively,²³ which were greater than the reported values.²⁴ However, the E_a values of this study were greater than these results. This could be attributed to many factors, such as different F/U molar ratios, reaction conditions, and resin formulations. Despite these inconsistencies, there is an inherent inaccuracy in the methods that are used to determine E_a values of a reaction system.²⁵ The author reported that the maximum rate method was more accurate than the dynamic method, which was used in this study.

Figure 7 shows the changes in ΔH of the UF resins modified with two scavengers. ΔH is defined as the area under an exothermic peak of a DSC curve. ΔH of the UFP-modified UF resin increased to 30% and then decreased. The US-modified UF resin also showed a similar trend, increasing to 20% and then decreasing. In general, the ΔH values of the UFPmodified UF resins were greater than those of the US-modified UF resins.

Because the onset and peak temperatures increased with an increase in the US concentration in the UF resin, it was expected that the US-modified UF resins would have greater ΔH values than the UFP-modified UF resin. Previous research has also reported an increase in ΔH of a UF resin when the peak temperature of the UF resin decreased with

a decrease in the F/U molar ratio,¹¹ but this was not the case for this study. The result in the current study could be attributed to the presence of methylolated ureas in UFP. Methylolated ureas of UFP facilitated the start of the curing reaction of the UF resin, which reduced E_a of the UFP-modified UF resins, as shown in Figure 6, but an increase in the number of methylolated ureas provided more energy to form methylene or methylene ether linkages to complete the curing of the UFP-modified UF resins. In contrast, it is thought that the urea grain that dissolved in US stayed in part as a separated substance that did not react with free formaldehyde, which was insufficient in the US-modified UF resins. Further research on the chemical structure and species of modified UF resins is needed.

The formaldehyde emissions of particleboard bonded with either UFP- or US-modified UF resins are shown in Figure 8. When the UF resin was modified with the addition of 50% US, particleboard could not be prepared because of delamination at the core layer. This could have been due to the high moisture content in the core layer with the addition of US, which was 40% urea and 60% water. As expected, the formaldehyde emissions of the particleboard continuously decreased with the increasing concentrations of the two scavengers. In general, the formaldehyde emission values were smaller for particleboard bonded with the UFP-modified UF resin than for particleboard bonded with the US-modified UF resins at all scavenger concentrations. This result suggests that UFP was more effective in scavenging formaldehyde than US. However, the formaldehyde scavenging effectiveness of the two scavengers should be judged with particleboard properties taken into account.

Figure 9 shows the IB strength values of particleboard bonded with either UFP- or US-modified UF



Figure 8 Formaldehyde emissions of particleboards bonded with modified UF resins with two scavengers.



Figure 9 IB strengths of particleboards bonded with modified UF resins with two scavengers.

resins; this is one of the critical properties of particleboard. The IB strength continuously decreased with an increase in the scavenger concentration, regardless of the type of scavenger. However, the IB strength values of particleboard bonded with UFPmodified UF resins were greater than those of USmodified UF resins. The IB strength of particleboard decreased from about 7.7 kgf/cm² to about 3.8 kgf/ cm²; it was half when 20% UFP was added to the UF resin. This result could be attributed to the better reactivity of the UFP-modified UF versus the USmodified UF resin, and this was confirmed by the onset temperature, peak temperature, or rate constants from the results of DSC. In other words, a 20% addition of UFP to the UF resin reduced the formaldehyde emissions of particleboard and simultaneously did not cause significant deterioration of the IB strength. This result indicates that UFP was better in scavenging the formaldehyde emissions of the UF resin than US. When the formaldehyde emission and IB strength values of the particleboard were taken into account, the optimum addition level of UFP was determined to be 20%, which resulted in

a balance of lowering the formaldehyde emissions and maintaining the IB strength at the same time.

Other properties of particleboard, such as MOR, MOE, TS, and WA, are presented in Table III. MOR values did not change much at the 10% concentration of either UFP or US in the UF resin and then continuously decreased with an increase in the concentration of the two scavengers. However, the MOR values of the particleboard bonded with the UFP-modified UF resin were much greater than those of the particleboard bonded with the US-modified UF resin. These results could also be attributed to the better reactivity of the UFP-modified UF resin.

MOE values of particleboard bonded with either UFP- or US-modified UF resin slightly increased at the 10% concentration. As the scavenger concentration increased above 10%, MOE values gradually decreased for the UFP-modified UF resin, whereas they drastically decreased for the US-modified UF resin. This result could be due to a greater densification of particles during hot pressing, which resulted from more water being sprayed when a 10% concentration of the scavenger was added to the UF resin. As shown in Table I, the nonvolatile resin solids of the modified UF resins decreased with an increase in the scavenger concentration.

The TS values of particleboard bonded with modified UF resins continuously increased above a 10% addition of the two scavengers. Also, the TS values were lower for the particleboard bonded with the UFP-modified UF resin than for the particleboard bonded with the US-modified UF resin, but they did not much change up to a 20% concentration of the two scavengers. The WA values of the particleboard followed a similar trend. These results could be attributed to a negative relation between the IB strength and TS or WA. In other words, greater IB strength generally resulted in lower TS or WA for the particleboard. A greater adhesive bond between particles could hold them together tightly, and this resulted in lower TS and WA values for the particleboard. In addition, the TS and WA values of this

TABLE III

Properties of Particleboard Bonded with Modified UF Resins by the Addition of Various Concentrations of the Two Formaldehyde Scavengers

Properties	Scavenger type	Scavenger mixing ratio (UF resin : scavenger)				
		100:0	90:10	80 : 20	70:30	50 : 50
MOR (kgf/cm ²)	UFP	176.4	178.3	155.1	119.4	71.9
	US		179.7	134.0	75.5	
MOE (× 10^3 kgf/cm ²)	UFP	29.9	34.4	33.8	32.0	25.1
	US		35.1	30.0	16.5	
TS (%)	UFP	8.18	7.4	9.2	14.5	42.8
	US		7.2	10.9	27.1	
WA (%)	UFP	31.3	28.9	32.5	40.1	77.2
	US		23.8	32.1	50.7	_

study were much lower than previously reported results.²⁶ These results indicated that UFP was more effective than US in scavenging the formaldehyde emissions of the UF resin adhesives, and the addition of 20% UFP to the UF resin was optimum when the properties of the particleboard were taken into account.

CONCLUSIONS

To investigate the influence of formaldehyde scavengers with respect to the thermal curing behavior of UF resins, a neat UF resin was modified by the addition of two formaldehyde scavengers (i.e., UFP and US) in different weight proportions (100 : 0, 90 : 10, 80 : 20, 70 : 30, and 50 : 50), and then these modified UF resin adhesives were used for the manufacture of particleboard to maintain the balance between the thermal curing behavior and particleboard properties. The thermal curing behavior of these modified UF resins with two scavengers was characterized with DSC, and particleboard was manufactured to determine its properties. As the concentration of the two scavengers increased, the gel time, peak temperature, and onset temperature of the UFP-modified UF resins did not change much, whereas these parameters increased for the USmodified UF resins. These results indicated that the reactivity of the UF resin modified with UFP was maintained, but it was weakened for the US-modified UF resin. Furthermore, the rate constant of the modified UF resin confirmed this result. E_a of the UFP-modified UF resins was lower than that of the US-modified UF resins, whereas the ΔH values were reversed.

The formaldehyde emissions of the particleboard bonded with the modified UF resin decreased with an increase in the scavenger concentration. UFP was more effective than US in scavenging the formaldehyde emissions of UF resin adhesives, and the addition of 20% UFP to the UF resin was optimum when the properties of the particleboard were taken into account. These results indicate that the thermal curing behavior and particularly the resin reactivity of scavenger-modified UF resins and the properties of particleboard bonded with them must be taken into account in the evaluation of a formaldehyde scavenger system.

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