

Melamine-modified urea formaldehyde resin for bonding particleboards

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Abstract

For the development of a cost-effective melamine-modified urea formaldehyde resin (MUF), the study evaluated the effects of reaction pH and melamine content on resin properties and bond performance of the MUF resin adhesive systems. Eight resins, each with three replicates, were prepared in a factorial experiment that included two formulation variables: two reaction pHs (i.e., 4.5 and 8.0) and four molar ratios of formaldehyde to urea to melamine (i.e., 3/2.2/0.3, 3/2.2/0.2, 3/2.2/0.1, and 3/2.2/0.05). Variables in particleboard preparation were two hot-press cured times (2 and 4 min). Thus, with two panel replications, a total of 96 panels were fabricated. Melamine content significantly affected resin properties and glue bond quality: gel time decreased, solid content increased, internal bond strength increased, thickness swell and water absorption decreased, and formaldehyde emission decreased as melamine content increased. In general, the resins catalyzed under acidic conditions (pH 4.5) resulted in faster gel times, higher internal bond strengths, lower formaldehyde emissions, and lower thickness swell and water adsorption than those catalyzed under alkaline conditions (pH 8.0). Significant correlation between gel time with both internal bond strength and formaldehyde emission suggest that resins with fast curing speeds provided favorable conditions for attaining a higher degree of resin cure, and in turn led to better bond strength and lower formaldehyde emission.

Low cost and proven performance have made urea formaldehyde resins (UF) the most important of wood adhesives for interior applications. However, concern about formaldehyde emission from particleboard and weakening glue bond caused by hydrolytic degradation of UF polymers have stimulated efforts to develop improved and/or new adhesives based on UF resins. For instance, in the late 1940s and early 1950s, various fortified UF resins were developed by simply incorporating various portions of melamine resin or resorcinol to improve the durability of hot-press urea resin glue bonds in plywood (Lecher 1946, Blomquist and Olson 1955). It was shown that optimum water resistance was obtained only when the urea and melamine were coreacted in the resin system (Lecher 1946). Paper chromatography, infrared spectroscopy, and elemental analysis were later used to confirm the cocondensation of urea and melamine with formaldehyde (Yanagawa and Matsumura 1962). It should be noted, however, the formaldehyde emission of MUF resins was not evaluated in these studies, because these resins were developed mainly for plywood gluing and the formaldehyde emission was not an issue at the time. The market domination of UF resins has so far made the application of fortified UF or MUF resins to particleboard gluing economically not feasible.

This has led to very little technical data on gluing particleboard with MUF resins in the literature.

With the development of structural wood composites in recent years, phenol-modified melamine urea formaldehyde resins have been used for the manufacture of exterior grade particleboard in Europe (Clad and Schmidt-Hellerau 1977). In Japan, phenol melamine formaldehyde in combination with urea formaldehyde resins has been used for the manufacture of concrete-form plywood (Tamura et al. 1981). These applications have expanded the interest in melamine-modified UF resins for structural applications other than conventional hot-setting MUF resins in edge gluing and laminating. Recent development of a cost-effective melamine-modified UF resin

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*Forest Products Society Member.

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Forest Prod. J. 58(4):56-61.

system for exterior grade flakeboard has shown that the MUF resins worked particularly well with various hardboards of wide-ranging wood density (Hse and He 1991).

These exterior-grade MUF resin adhesives are considered too costly to replace UF resins for interior application. The melamine-modified UF resins with reduced melamine content levels have been developed to improve durability and moisture resistance properties. These low-melamine content UF resins have been relatively popular in Europe (Dunky 1995) and in the Asia-Pacific region (Maylor 1955, Parker and Crews 1999) for many years. More recently, the melamine-modified UF resins were also shown to yield particleboards with significantly lower formaldehyde emissions than the control UF resins (Graves 1993, Rammon 1997).

Although the low-mole-ratio MUF resins have not been widely accepted in North America, interest in the development of cost-effective resin systems for upgrading particleboard and MDF remains high. This paper describes recent experiments on such a low melamine content MUF resin system to improve glue bond durability and formaldehyde emissions of particleboard. The main objectives of the study were to evaluate the effects of reaction pH and melamine content on resin properties and bond performance of the MUF resin adhesive systems.

Experimental procedure

Design of experiment

The formulation factors for MUF resins considered in the study were melamine content level and reaction pH. The experimental variables were:

Reaction pH

Weak acid—pH 4.5

Alkaline—pH 8.0

Molar ratio of formaldehyde to urea to melamine (F/U/M)

3 / 2.2 / 0.3 (F/U/M)

3 / 2.2 / 0.2

3 / 2.2 / 0.1

3 / 2.2 / 0.05

Resin replication: 3 resins per condition (total of 48 resins)

Resin preparation

All MUF resins were synthesized in the laboratory. To prepare each resin, all formaldehyde and water were placed in a reaction kettle and pH was adjusted with sulfuric acid or sodium hydroxide. Urea was added in 15 equal parts at 1-minute intervals. To initiate the reaction, the mixture was heated and maintained at 80 °C to promote the formation of urea-formaldehyde prepolymer. At 30 minutes reaction time, melamine was added and reacted for an additional 120 minutes at the same temperature (80 °C). No additional pH adjustment was made in the resins synthesized with pH 8.0. For resins formulated with pH 4.5, however, the pH was adjusted to 6.0 at 60 minutes reaction time. Additional urea was introduced with three consecutive additions of 0.4 mol urea each at 20 minutes intervals beginning at the reaction time of 90 minutes. All reactions were terminated by rapidly cooling the mixture to 25 °C at 150 minutes. Gel time, pH, solid content, and viscosity were determined. **Figure 1** summarizes the reaction schedule.

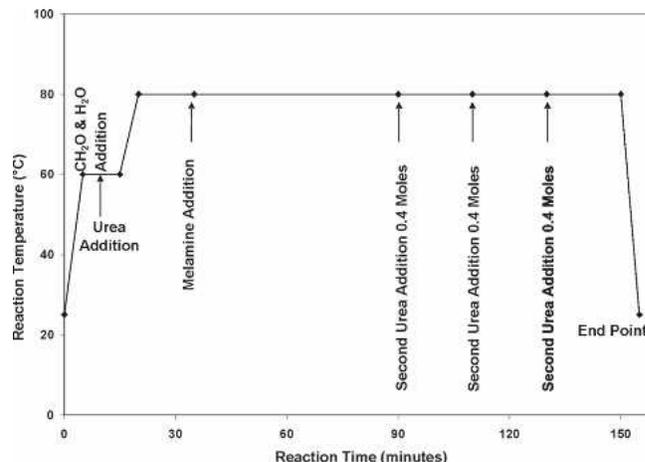


Figure 1. — Reaction temperature and chemical addition as related to reaction time.

Particleboard manufacture

All panels were prepared in the laboratory with wood particles obtained from a particleboard plant of Willamette Industries at Ruston, Louisiana. The wood particles, with averaged MC of 3 percent, were stored in heavy duty polyethylene bags directly from the dry-end of the mill dryer and were used in the laboratory without further treatment.

To prepare each panel, the wood furnishes were weighed out (target board density was 48 PCF (0.769 g/cm³)) and placed in a rotating drum-type blender. The resin, 4.5 percent based on oven dry weight of wood, was then weighed and applied by air-atomizing nozzle. Wax and catalyst were not used in the study. After blending, the wood furnishes were carefully felted into a 19- by 20-inch (48.3 by 50.8-cm) box to form the mat. The mat was transferred immediately to a 40- by 40-inch (101.6 by 101.6-cm) single-opening hot-press with the platen temperature regulated at 375 F (190.6 °C). Sufficient pressure (about 550 psi (3,792 kPa)) was applied so that the platen closed to 1/2-inch (1.27-cm)-thickness stops in approximately 45 seconds. Press times were 2 and 4 minutes. Board manufacture replication was 2 boards per condition (total of 192 boards).

Particleboard testing

All boards were conditioned in a chamber at 50 percent relative humidity (RH) and 80 °F (26.7 °C) before testing, ending with a MC that averaged 5.5 percent. After conditioning, each board was cut to yield 10 2- by 2-inch (5.08 by 5.08-cm) specimens for tensile strength perpendicular to the face (IB), eight 2.75- by 5-inch (6.99 by 12.7-cm) desiccator samples, and four 6- by 6-inch (15.24 by 15.24-cm) dimensional stability specimens (thickness swell). The internal bond strength test was performed in accordance with ASTM standard for evaluating the properties of wood-base fiber and particle panel materials (D 1037-93). For thickness swell (TS) evaluation, a 24-hour water soak was employed. The TS values measured changes in thickness after the specimens were submerged in water at room temperature for 24 hours.

Formaldehyde emission measurement

The determination of formaldehyde absorbed in distilled water is based on the specific reaction of formaldehyde with a chromotropic acid-sulfuric acid solution, forming a purple monocationic chromogen. The absorption of the purple solution

Table 1. — Resin properties as related to resin formulation variables.

Initial reaction pH	Molar ratio F/U/M ^a	Melamine content ^b (%)	Resin pH	Resin solid (%)	Viscosity (Cp)	Gel time (min.)
8.0	3/2.2/0.3	14.5	6.82	53.4	77	31.4
	3/2.2/0.2	10.2	6.75	51.7	73	48.0
	3/2.2/0.1	5.40	6.49	51.2	63	69.7
	3/2.2/0.05	2.75	6.42	51.4	73	92.7
	3/2.2/0	0	6.25	47.8	67	135.5
4.5	3/2.2/0.3	14.5	6.39	53.2	86	20.5
	3/2.2/0.2	10.2	6.22	52.1	69	35.3
	3/2.2/0.1	5.40	6.22	50.4	70	71.4
	3/2.2/0.05	2.75	6.05	50.6	89	75.7
	3/2.2/0	0	6.37	48.1	64	134.3

^aLetter F represents formaldehyde, letter U represents urea, and letter M represents melamine.

^bBased on molecular weight.

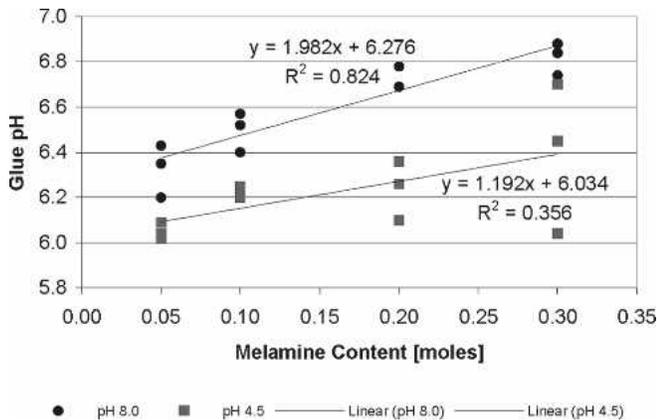


Figure 2. — Relationship of melamine content to resin pH.

was read using a spectrophotometer at 580 nm. The test was performed in accordance with the National Particleboard Association (NPA) 2-hour-desicator test.

Result and discussion

Effects of reaction pH and melamine content on resin properties and panel properties were evaluated by analysis of variance at the 0.05 level of probability. Significant effects were further characterized by the least significant difference test.

Resin properties

Average physical properties of the MUF resins are summarized in **Table 1**. As expected, the pHs of resins catalyzed with alkali were slightly higher than those catalyzed with acid. When compared under the same reaction pH conditions, the pH of the resins catalyzed with alkaline increased as melamine increased (**Fig. 2**). Such relationship observed for the acid catalyzed resins was not significant. Regression analysis also showed that the effects of resin pH on gel time were evident in resins prepared with alkaline catalyst. The gel time decreased as reaction pH increased (**Fig. 3**). Again such relationship observed for the acid catalyzed resins was not significant.

In general, the resin solid content increased as the melamine content increased (**Table 1**). The solid content increase was due mainly to the addition of powdered melamine without

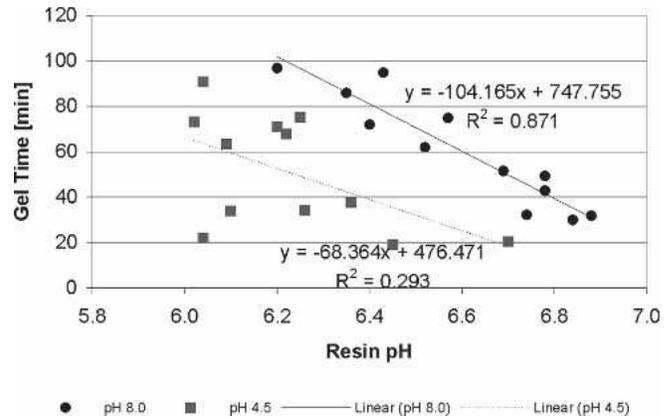


Figure 3. — Relationship of resin pH to gel time.

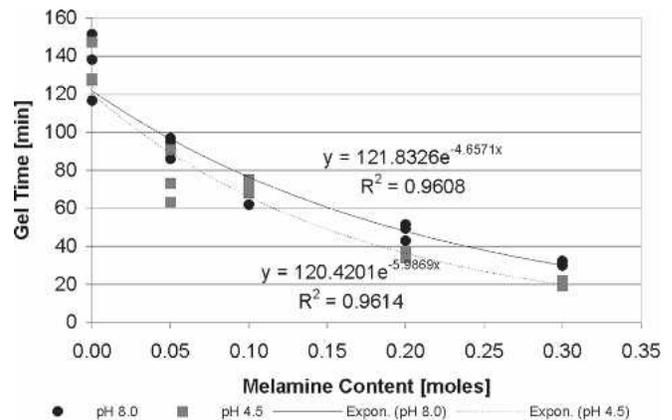


Figure 4. — Relationship of melamine content to gel time.

making adjustment for the water content in the reaction. Differences in resin viscosity were not significant.

The significant effects of melamine content on gel time were evident in the study. The resins catalyzed under acidic conditions (i.e., pH 4.5) resulted in gel times consistently faster than those catalyzed with alkaline conditions (pH 8.0). When compared at the same reaction pH, the gel times decreased as the melamine content increased. Regression analysis confirmed the relationship between gel time and melamine content was exponential (**Fig. 4**). It is interesting to note that the decrease in gel time for each mole increase in melamine content was greater under acidic conditions (i.e., at relative rate of 5.9869) as compared to that of alkaline condition (i.e., at relative rate of 4.6571).

By regression analysis, gel time was found to correlate significantly with resin solid content. In both reaction pH conditions, the gel time decreased as resin solid content increased.

Mechanical properties and formaldehyde emission

Average internal bond strength, thickness swell, water absorption, and formaldehyde emission are summarized in **Table 2**. The effects of melamine content, reaction pH, and hot-press time on panel properties evaluated by the least significant difference tests are summarized in **Table 3**.

Internal bond strength

On average, the acid catalyzed resins yielded significantly higher IB (i.e., 94.3 psi (650 kPa)) than that of the alkaline catalyzed resins (79.6 psi (549 kPa)). When compared at the

Table 2. — Physical mechanical properties and formaldehyde emission of particleboard related to formulation variables.

Reaction pH	Press time	Ratios F/U/M ^a	Panel property									
			Density		IB ^b		EMI ^b		TS ^b		WA ^b	
			Ave.	Stde.	Ave.	Stde.	Ave.	Stde.	Ave.	Stde.	Ave.	Stde.
			(PcF)	(psi)	(ppm)		----- (%) -----					
4.5	2	3/2.2/0.3	47.5	1.17	88.2	8.8	2.12	0.509	23.2	0.9	81.1	6.0
		3/2.2/0.2	47.3	1.49	90.4	6.2	3.04	0.637	22.3	1.5	81.6	3.5
		3/2.2/0.1	45.8	1.41	68.0	12.8	4.18	0.477	26.1	1.2	93.8	5.6
		3/2.2/0.05	46.3	1.15	53.7	10.7	4.73	0.968	31.4	0.7	101.0	8.2
	4	3/2.2/0.3	49.1	1.30	115.8	10.6	1.43	0.345	22.7	0.7	78.4	6.0
		3/2.2/0.2	49.6	1.36	138.9	14.0	2.50	0.300	22.8	1.2	74.3	5.1
		3/2.2/0.1	47.0	1.09	107.2	17.0	3.41	0.469	23.2	0.8	85.8	9.2
		3/2.2/0.05	47.6	1.23	92.1	14.2	3.42	0.629	28.4	1.7	83.1	5.6
	3/2.2/0 (control)	47.9	0.91	52.9	4.7	3.92	0.559	34.2	2.8	101.5	3.8	
8.0	2	3/2.2/0.3	47.7	1.97	83.4	11.2	2.99	0.543	19.4	1.6	77.9	5.3
		3/2.2/0.2	45.6	1.50	75.8	10.9	3.58	0.493	21.7	2.5	85.5	4.8
		3/2.2/0.1	46.0	1.86	46.7	13.2	3.79	0.498	30.0	4.5	100.4	16.0
		3/2.2/0.05	46.5	1.76	30.3	7.9	4.22	0.236	41.0	4.2	121.8	13.4
	4	3/2.2/0.3	49.7	0.69	133.5	12.4	2.69	0.830	20.1	1.4	73.9	6.8
		3/2.2/0.2	46.3	2.42	116.1	14.4	2.94	0.768	21.9	1.4	79.0	5.6
		3/2.2/0.1	49.5	2.15	93.2	12.0	2.96	0.537	28.9	5.0	85.9	7.0
		3/2.2/0.05	47.3	2.18	57.8	9.9	3.71	0.622	36.3	3.2	106.7	7.9
	3/2.2/0 (control)	46.2	2.10	28.7	4.8	4.78	0.159	44.8	1.6	127.5	7.7	

^aLetter F represents formaldehyde, letter U represents urea, and letter M represents melamine.

^bIB represents internal bond strength; FEMI represents formaldehyde emission; TS represents thickness swelling; and WA represents water absorption.

Table 3. — Summary results of the least significant difference test.

Factors	Levels	Property ^a				
		Density (PcF)	IB ^c (psi)	FEMI ^c (ppm)	TS ^c (%)	WA ^c
PH	4.5	47.5 A ^b	94.3 A	3.104 A	25.0 A	84.9 A
	8.0	47.3 A	79.6 B	3.361 B	27.4 B	91.4 B
Time	2	46.6 A	67.0 A	3.582 A	26.9 A	92.9 A
	4	48.3 B	106.8 B	2.883 B	25.5 B	83.4 B
Ratio	3/2.2/0.3	48.5 A	105.2 A	2.310 A	21.4 A	77.8 A
	3/2.2/0.2	47.2 B	105.3 A	3.016 B	22.2 A	80.1 A
	3/2.2/0.1	47.1 B	78.8 B	3.584 C	27.0 B	91.5 B
	3/2.2/0.05	46.9 B	58.5 C	4.021 C	34.3 C	103.1 C

^aMeans with the same capital letter are not significantly different by Tukey's tests at 0.05 level.

^bThe same letter in the column indicates no significant difference at the 5 percent level.

^cIB represents internal bond strength; FEMI represents formaldehyde emission; TS represents thickness swelling; and WA represents water absorption.

same reaction pH, the reaction pH interacted with the melamine content to affect IB. As shown in **Figure 5**, the IB increased as the melamine content increased for alkaline catalyzed resins; while, for acid catalyzed resins, the IB increased to the maximum at 0.2 mol of melamine content and thereafter IB decreased as melamine content increased. It is further shown that the decrease in IB for the acid-catalyzed resin at F/U/M molar ratio of 3/2.2/0.3 resulted in lower IB than that of alkaline-catalyzed resin. However, by the least significant difference test (**Table 3**), the difference in the average IB between 0.2 and 0.3 mol of melamine was not significant.

The effects of melamine content on IB were further considered through relevant resin properties. By regression analysis,

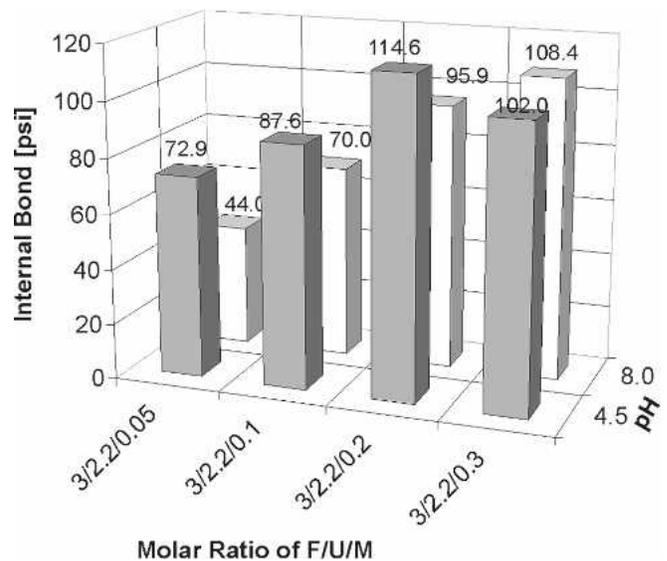


Figure 5. — Interaction of internal bond with pH and molar ratio of F/U/M.

the IB increased as gel time decreased (**Fig. 6**). It is generally agreed that gel time can be correlated to curing speed of resin with actual pressing condition. Thus, the results suggest that high melamine content yielded resins with faster curing speed as expected. The fast curing speed, in general, provided favorable condition for attaining higher degree of the completion of resin cure, and in turn led to better bond strength.

Figure 7 shows the correlation between resin solid content and the IB. The water that enters the furnish as part of the liquid resin, also makes up a portion of the final mat MC entering the hot-press. Hence, on a 4.5 percent addition of the

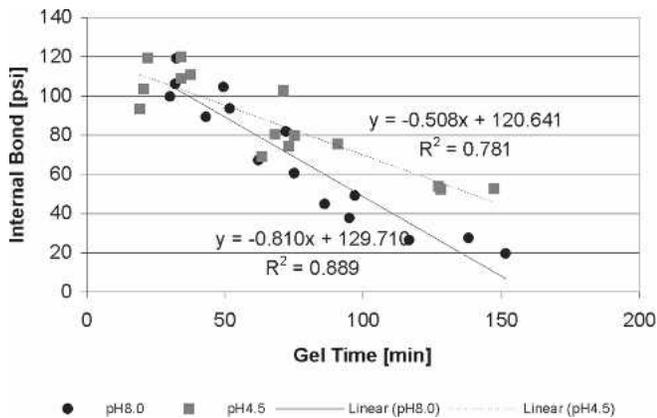


Figure 6. — Relationship of gel time to internal bond.

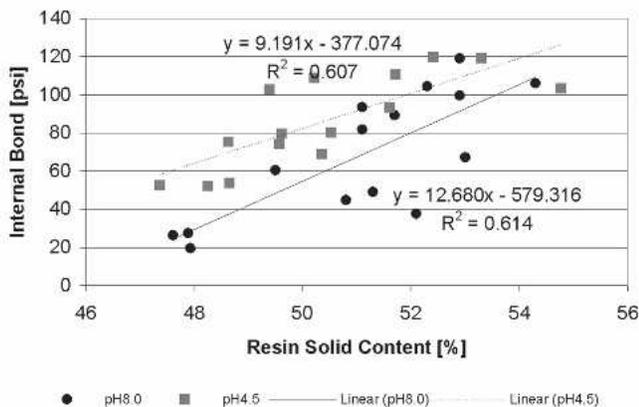


Figure 7. — Relationship of resin solid content to internal bond.

MUF resin solids, a maximum of 2.15 percent of water (based on resin solids content of 47.8%) is added to the furnish. Since the average MC of the wood particles used was 7.5 to 8.5 percents, the moisture from the resin could raise the final MC to 10 or 11 percent. At this level, the mat MC is approaching the allowable limit that the process can tolerate, particularly when using a short, hot-press cycle. The significant correlation between resin solids content and IB seems to highlight the importance of mat MC in the IB development of the panels.

It should be mentioned that two control UF resins were also made with same reaction conditions without melamine addition in the study. However, the control UF resins failed to produce acceptable panels when cured at 2 minutes press time. The panel properties of the UF resins with 4 minutes cure time are included in **Table 2**. Comparison of IB of UF bonded panels with that of MUF resins confirmed that melamine addition increased resin cure speed and improved bond strength as expected.

Formaldehyde emission

As expected, 4-minute cure time resulted in significantly lower formaldehyde emissions compared to the 2-minute cured panels. By analysis of variance, formaldehyde emissions were also significantly affected by reaction pH. Average formaldehyde emissions were 3.104 ppm and 3.361 ppm for acid catalyzed resins and alkaline catalyzed resins, respectively. When comparing formaldehyde emissions under same

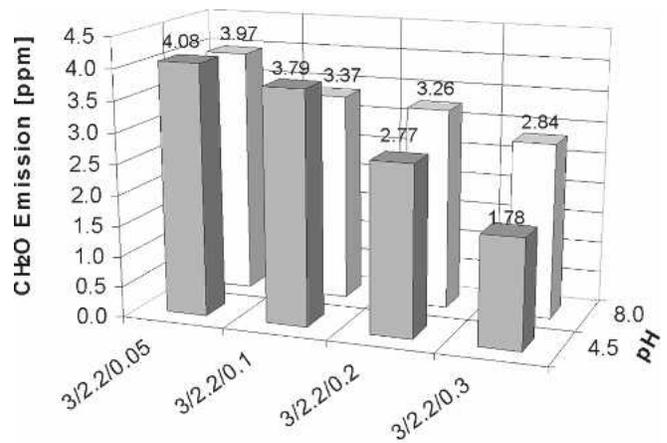


Figure 8. — Interactions of formaldehyde emission with pH and molar ratio of F/U/M/

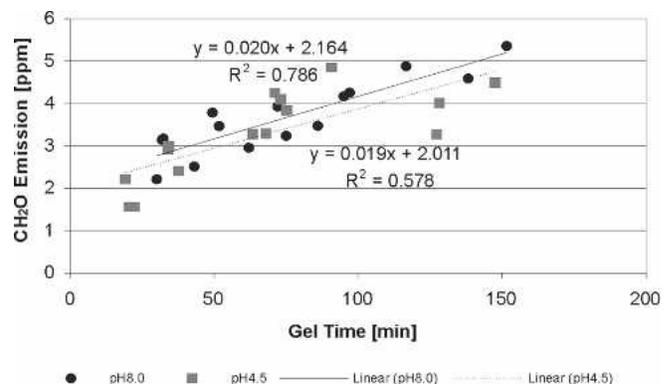


Figure 9. — Relationship of gel time to formaldehyde emission.

reaction pH, the formaldehyde emission decreased consistently as melamine content increased. Furthermore, the melamine content interacted with reaction pH to affect the formaldehyde emission. As shown in **Figure 8**, at lower melamine content levels (i.e., 0.1 and 0.05 mol) the alkaline pH yielded slightly lower formaldehyde emission than that of acidic pH. At higher melamine content levels (i.e., 0.2 and 0.3 mol), however, the acidic pH yielded substantially lower formaldehyde emission than that of alkaline pH.

Regression analysis showed strong correlation between gel time and formaldehyde emission (**Fig. 9**) As with IB, fast gel time led to the completion of higher degree of resin cure, and in turn lower formaldehyde emissions.

Thickness swell and water absorption

Analysis of variance showed that thickness swell and water absorption after 24-hour water soak test were significantly affected by hot-press time and resin pH (**Table 3**). The panels fabricated with 4-minute press time had significantly lower thickness swell (TS) and water absorption (WA) compared to the panels made with 2-minute press time. Furthermore, panels bonded with acid catalyzed resins resulted in significantly lower TS and WA compared to alkaline catalyzed one. On average, TS and WA decreased as the melamine content increased (**Table 3**). Analysis of variance showed that the differences in TS and WA of panels bonded with MUF resins

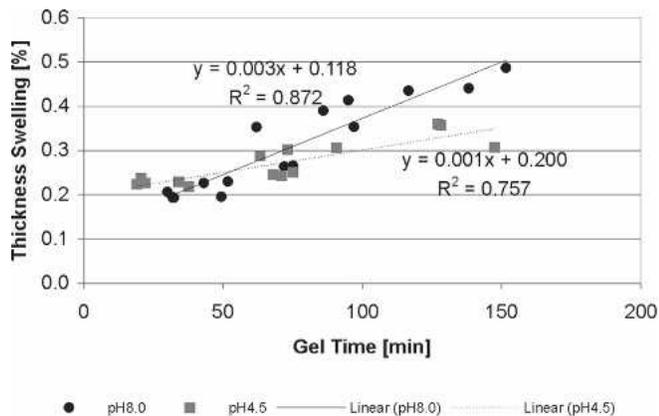


Figure 10. — Relationship of gel time to thickness swelling.

between 0.3 and 0.2 mol of melamine were not significant. This suggests that addition of melamine content greater than 0.2 mol resulted in little gain in dimensional stability.

By regression analysis, the TS increased proportionately as gel time increased (Fig. 10). The correlation between gel time and WA was not significant.

Discussion

Of the two pH conditions evaluated in the study, weak acid catalyst systems are not commonly used in present MUF resin formulations. Based on the bond strength and formaldehyde emission data, however, weak acid catalysis seems to result in overall better MUF resin systems. One of the major differences in the two catalyst systems were the reaction products yielded during the initial urea and formaldehyde reaction period (i.e., prior to the addition of melamine). Reacting the UF under acid catalyst, without first using an alkaline catalyst, promoted higher degrees of condensation of urea with formaldehyde as shown in a previous study (Hse et al. 1994). The alkaline catalyst enhanced the formation of methylolurea. In recent studies on curing behavior and polymeric structures of conventional MUF resin adhesives with alkaline catalyst, Higuchi et al. (1992) indicated that the majority of melamine in the MUF resins tend to form melamine to melamine linkages and incorporate a small amount of urea to form a three-dimensional, cross-linked network. The large portions of urea in the system are then linked as pendants or grafts to form the polymeric structure. Thus, the higher condensation under acid catalyst may result in incorporating larger amount of urea to form a three-dimensional cross-linked network with melamine. This would account for the faster cure speeds with better glue bonds observed here. However, there is no experimental data from this study or elsewhere to confirm these explanations. An attempt was made to use carbon-13 nuclear magnetic resonance spectroscopy to evaluate the reaction mechanism of cocondensation of urea and melamine through carbon-13 enriched formaldehyde in a previous study (Tomita and Hse 1995). However, precise analyses among three resins (i.e., MF, UF, and MUF) by C-13 NMR are still not fully attainable at the present time.

Optimum curing of resin adhesive is an important factor effecting on overall resin performance, particularly to the bond strength and formaldehyde emission. As expected, the 4 minutes cure time consistently resulted in higher IB and lower formaldehyde emission than that of 2 minutes cure time. The longer cure time, in general, provided favorable condition for

attaining higher degree of the completion of resin cure, and in turn led to better resin performance. However, it should be noted that the curing effects of the resin on performance of the cured resin will require further evaluation of the comparison of full cure panels fabricated with each resin in order to obtain the conclusive comparisons of each resin performance and formaldehyde emission.

Summary and conclusions

Gel-times were significantly correlated with IB, formaldehyde emission, and panel durability. The fast curing speed provided favorable conditions for attaining a high degree of completion of resin cure and led to better bond strengths and lower formaldehyde emissions.

Reacting the UF under acid catalyst without first using an alkaline catalyst promoted higher degrees of condensation. The higher condensation resulted in incorporating larger amount of urea to form a three-dimensional cross-linked network with melamine and enhanced glue bond performance.

Melamine addition yielded overall improvements in board properties. The melamine-modified UF resins provide an option for upgrading the performance of particleboards

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