

Effects of Melamine on the Properties of Medium-Density Fiberboard Fabricated with Urea-Based Resins

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Abstract

Urea-melamine-formaldehyde (UMF) resins were synthesized and used for manufacturing medium-density fiberboard (MDF). As melamine content was increased at the same formaldehyde/urea (F/U) molar ratio, the solids content of the resin increased. Physical properties and storage stability of the resins tended to improve slightly when the final pH of resin was 8.5 compared with 7.5 and 9.5. At the same F/U molar ratio, free formaldehyde content and gel time of the resins decreased as melamine content increased. Chemical structures of the resins were proposed from the results of ^{13}C nuclear magnetic resonance and Fourier transform infrared analyses. Formaldehyde emissions of MDF prepared with UMF resins with the same F/U molar ratio decreased as melamine content of resin was increased. MDF fabricated with UMF resin showed slightly better resistance against decay fungi and termites compared with MDF fabricated with urea-formaldehyde (UF) resin. Other physical properties of MDF fabricated with UMF resin were comparable to those of MDF fabricated with a commercial UF resin. An optimum F/U molar ratio of 1.2 with melamine content of 15 percent, equivalent to an F/(U+M) molar ratio of 0.98, was found from the results.

With the 2004 implementation of an indoor air quality control law as a countermeasure for sick house syndrome in Korea, the general public's preference has moved toward less harmful building materials. At the same time, wood-based composite factories, especially those utilizing formaldehyde, have tried to use lower amounts of formaldehyde, thereby producing products that emit less formaldehyde. Until now, formaldehyde-containing resins have been the most widely used adhesives for manufacturing wood-based composites due to the low cost of raw materials.

In 2009, a total 568,000 tons of adhesives were produced in Korea, with 222,000 tons of formaldehyde-based adhesives. Among those, urea-formaldehyde (UF) resins were used in up to 77 percent due to low costs and colorless bond lines after curing. However, their weakness to moisture restricts their use to interior applications only. Cured UF resins are not water resistant, which causes many problems such as changes in dimensions and then disintegration. In addition, when UF resin-bonded boards are in contact with moisture for long periods, they emit formaldehyde and cause sick house syndrome. Therefore, formaldehyde emission has been one of the most important issues with UF resins in the last few decades (Myers and

Koutsky 1987, Pizzi 1994). The amount of formaldehyde emitted from wood-based composites is almost proportionally related to the amount of formaldehyde in the resin formulation. Both the reversible curing mechanism of UF resins and the low resistance against hydrolysis of cured UF resins are major factors affecting formaldehyde emission of UF resin-bonded wood-based composites. Formaldehyde emission during curing in board manufacturing or during the in-service use of the boards has been controlled and greatly reduced by technical advancements. The hydrolysis mechanisms of cured UF resins were investigated (Myers 1983, Myers and Koutsky 1987), and the reversible reaction

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schemes of UF resins were revealed. Numerous studies focusing on formaldehyde emission as well as water resistance were performed on UF resins, but only a few of them were reported (Pizzi 1994; No and Kim 2004; Park et al. 2006, 2009). Most of the formaldehyde emission control from wood-based composites was achieved by lowering the molar ratio of formaldehyde to urea (F/U) in the resin; however, the lower F/U molar ratio negatively influenced some other properties. One of these is the resistance to fungi and termites of the wood-based composites. Decay and termite resistance of MDF made from different wood species and oriented kenaf fiberboards fabricated with phenol-formaldehyde resin have been evaluated (Kartal and Green 2003, Walther et al. 2007), but more research is needed on the decay and termite resistance of wood-based composites produced with urea-based resins.

Materials and Methods

Urea-melamine-formaldehyde (UMF) resins used for this study were prepared in the laboratory with reagent grade urea (99%), melamine (99%), formaldehyde (37%) solution, sodium hydroxide aqueous solution (20%, wt/wt), and formic acid aqueous solution (20%, wt/wt). An industrial UF resin (F/U = 1.2) for MDF from Sunwood Co. (Inchen, Korea) was used as a control.

UMF resins were synthesized at F/U molar ratios of 1.0, 1.2, and 1.4 with melamine contents of 10 and 15 percent. UMF resins with different F/U molar ratios and melamine contents were converted to F/(U+M) molar ratios (Table 1). The formaldehyde solution (37%) was charged in a 2-liter reactor equipped with a stirrer, thermometer, and condenser, and the pH was adjusted to 7.8 with the sodium hydroxide solution. Then the mixture was heated and kept at 75°C for 10 minutes. The first urea was added to obtain an initial F/U molar ratio of 2.0, and the mixture was heated to 90°C. Melamine was added at 90°C to obtain different melamine content based on the total weight of UMF resin. Following this, the reaction pH was adjusted to 6.0 with the formic acid solution, and the viscosity of resin was measured every 15 minutes until the viscosity of the reaction mixture reached CD by the Gardner-Holdt scale. Then, the second urea was added; as soon as the viscosity of the reaction mixture reached C, the resin was cooled to room temperature. The final pH of the resins was adjusted to 7.5, 8.5, and 9.5.

Resin viscosity was measured by using Gardner-Holdt and Brookfield viscometers at 25°C. Nonvolatile resin solids were measured by heating about 1.0 g of resin in an oven at 125°C for 2 hours. Gel times were measured at 100°C using a Sunshine gel timer. Resins were catalyzed with 3 percent ammonium chloride as a 20 percent water solution based on the nonvolatile solids content of the resin. The content of free formaldehyde of the resins was determined by the sodium sulfite method. Storage stability was measured by

gel times and viscosity changes of the prepared resins at regular intervals.

¹³C nuclear magnetic resonance (NMR) spectra of the resins were obtained using a 300-MHz model (Bruker AMX-R300) spectrometer at ambient temperature using a 9.5-microsecond pulse width and 5-second delay time for maximum quantification results with about 2,000 scans accumulated. Samples were prepared by mixing a liquid resin in dimethylsulfoxide-d₆ in a 1:1 weight ratio. The spectral intensities were integrated and peaks were assigned.

Fourier transform infrared (FT-IR) spectra of cured resins were measured in the form of KBr pellets on a Thermo Nicolet NEXUS FT-IR spectrometer. Functional groups of resins were assigned and IR absorption bands of those functional groups were compared.

Differential scanning calorimetry (DSC) curves of resins were obtained using a TA DSC Q10. Approximately 5 mg of resin was sealed in an aluminum pan, and runs were carried out by increasing the temperature from 30°C at a heating rate of 10°C/min to 200°C.

MDFs were made for evaluating the synthesized resins by using the manufacturing parameters summarized in Table 2. The MDF manufacturing process was as follows: radiata pine fibers, dried to a moisture content below 7 percent based on oven-dried fiber weight, were tumbled in a rotary blender, and the resin was sprayed in using a compressed-air sprayer. Mats were made by felting weighed amounts of fibers in a 30 by 35-cm forming box. The mat was then hot-pressed in a laboratory hot press (76 by 76 cm) using platen temperatures of 180°C and press times of 10 s/mm.

The pressed MDFs were tested according to KS F 3200 (Korean Standards Service Network [KSSN] 2006) for internal bond (IB), thickness swelling (TS), and modulus of rupture (MOR), and to KS M 1998-4 (KSSN 2005) for the formaldehyde emission level.

MDFs manufactured with a UMF resin (F/U molar ratio of 1.2 and 10% melamine content) and manufactured with an industrial UF resin as a control were chosen for decay and termite resistance tests. The specimens used in the decay resistance test were cut to 20 by 16 by 6 mm; for the termite resistance test the specimen size was 10 by 10 by 6 mm.

A decay test was conducted with the brown-rot fungus, *Fomitopsis palustris* (Ber. et Curt) Murr., and the white-rot fungus, *Trametes versicolor* (L. ex Fr.) Quelet., according to Standard JIS K 1571 (Japanese Industrial Standards [JIS] 2004). A 100-mL aliquot of medium containing 2.5 percent glucose, 0.5 percent peptone, 0.3 percent dipotassium hydrogen phosphate, 0.2 percent magnesium sulfate, and 1.0 percent malt extract was inoculated with stock culture. The inoculated medium was incubated on a shaker at 26°C and 70 percent relative humidity for 10 days. The medium of 300 g of sea sand in a glass jar was permeated with 85 mL

Table 1.—Properties of resins prepared at different formaldehyde/urea (F/U) molar ratios and melamine (M) contents.

Resin	F/U molar ratio	M content (%)	F/(U+M) molar ratio	Solids content (%)	pH	Viscosity (cP)	Free HCHO (%)	Gel time (s)
U10M15F	1.0	15	0.84	61.20	8.50	130.1	0.75	1,800
U12M10F	1.2	10	1.05	57.80	8.50	131.5	1.06	331
U12M15F	1.2	15	0.98	59.70	8.51	132.0	1.00	300
U14M10F	1.4	10	1.21	55.45	8.52	130.4	0.96	303
U14M15F	1.4	15	1.13	57.50	8.50	131.2	0.94	298
UF	1.2	0	1.20	63.00	7.72	143.3	1.80	149

Table 2.—Medium-density fiberboard (MDF) manufacturing parameters.

Parameter	Detail
Dimension of MDF	6 mm (T) × 35 cm (L) × 30 cm (W) ^a
Resin loading level	13% based on oven-dried fiber weight
Catalyst amount	3% ammonium chloride as a 20% water solution based on resin solids content
Target density	0.700 g/cm ³
Press condition	Temperature, 180°C; pressure, 0.98 MPa; time, 10 s/mm

^a T = thickness; L = length; W = width.

of the nutrient solution. The jar was then inoculated with 3 mL of the previously prepared fungal stock culture. The specimens were sterilized with ethylene oxide gas. When the mycelium fully covered the medium in the jar, three specimens were placed on top of the growing mycelium. The jar was then incubated at 26°C for 12 weeks. Nine replicates were tested for each fungus for each MDF. The extent of the fungal attack was expressed as the average of mass loss calculated from oven-dry weights of nine specimens before and after the test.

The specimens were exposed to termites (*Reticulitermes speratus kyushuensis* Morimoto) according to Standard JIS K 1571 (JIS 2004). An acrylic cylinder (80 mm in diameter, 60 mm in height) that had the lower end sealed with a 5-mm-thick dental plaster was used as a container. A test specimen was placed at the center of the plaster bottom of the test container; a total of 150 worker termites were introduced into the container. Five specimens per each MDF were assayed with termites. The containers were set on damp cotton pads to supply water to the specimens and kept at 28°C ± 1°C in a dark room for 3 weeks. The mass loss of the specimens due to termite attack was calculated based on the differences in the initial and final oven-dry weights of the specimens after cleaning off the debris from termite attack. The mortality of the termites was calculated based on the differences in the number of termites introduced initially and those that survived after 3 weeks.

Results and Discussion

An F/(U+M) molar ratio of 0.84 was found to be inadequate for resin formulation; the reaction hardly proceeded and the prepared resin showed phase separation phenomenon after cooling. Adhesion was evident when the F/(U+M) molar ratio was higher than 0.98 in our resin system. Various comparisons of synthesized resins were justified for different F/U molar ratios and melamine contents to keep the resin material parameters and the extent of polycondensation relatively constant. The physical properties of prepared UMF resins and a commercial E1 grade UF resin, used as a control resin, are summarized in Table 1. Prepared resins were divided into three parts in order to investigate the effect of pH on the storage stability of resins. The nonvolatile solid content of UMF resins prepared at different F/(U+M) molar ratios ranged from 55.5 to 61.2 percent.

One critical factor for storage stability of a water-based resin is its pH. The physical properties and storage stability of the resins were better at pH 8.5 than at pH 7.5 and 9.5, regardless of the F/U molar ratio and melamine content. In the early stages of curing, both melamine and urea components condense through hydroxymethylation reactions under weak basic pH conditions (7.0 to 8.0). Nucleophilic addition of amino groups from melamine or

urea to formaldehyde form hydroxymethyl groups. The melamine-formaldehyde condensation reaction occurs under neutral conditions (pH 6.0 to 8.0) in the second stage of resin synthesis, but the UF condensation does not occur under neutral pH conditions because urea condenses with formaldehyde only under acidic conditions. After synthesis, a resin is composed of some co-condensed urea-melamines, some hydroxymethylated ureas, and some hydroxymethylated melamines. At pH 7.5 and 9.5, some of these components most likely self-condense or agglomerate. However, at pH 8.5 co-condensed urea-melamines and hydroxymethylated ureas remain stable while hydroxymethylated melamine components probably condense further to form somewhat stable melamine-formaldehyde resins as shown by Pizzi (1994).

The viscosity of the resins ranged from 130.1 to 132.0 cP, regardless of the F/(U+M) molar ratio. At high F/(U+M) molar ratios, excess formaldehyde may have formed more branched hydroxymethylated urea or hydroxymethylated melamine, resulting in an increased viscosity of the resins. If most of the hydroxymethylated urea and melamine condensed further, nonvolatile solid content would also have increased, but most of those species seemed to have not reacted. In other words, the structure of the resin tended to be more branched and to have less condensed structures with a high F/(U+M) molar ratio; in contrast, less branched but more condensed structures were favored at a low F/(U+M) molar ratio. Most of the added melamine seemed to participate in condensation reactions because solid content tended to increase as melamine content increased at the same F/U molar ratio. Gel times of the prepared resins ranged from 298 to 1,800 seconds and were shortened as the F/U molar ratio increased. The shortened gel time due to the increase in F/U molar ratio can be attributed to the higher degree of branching and higher functionality. The influence of melamine content on gel time was slightly larger for an F/U molar ratio of 1.2 compared with 1.4. Gel times of resins with an F/U molar ratio of 1.2 ranged from 300 to 331 seconds and those with a ratio of 1.4 ranged from 298 to 303 seconds. Free formaldehyde content in the prepared resins ranged from 0.75 to 1.06 percent. Except for the resin with 10 percent melamine content, the resins with an F/U molar ratio of 1.4 had lower amounts of free formaldehyde than the resins with an F/U molar ratio of 1.2. Most of the formaldehyde in resins with an F/U molar ratio of 1.4 may have produced more branched hydroxymethylated ureas or the free formaldehyde remained less reactive toward urea, and consequently reacted mostly with melamine. On the other hand, the resins prepared with an F/U molar ratio of 1.2 may have produced less branched hydroxymethylated ureas and thus the free formaldehyde content remained relatively high.

The storage stability of prepared UMF resins was measured by viscosity and gel time during the storage period (Fig. 1). Viscosity of the UMF resins increased gradually and gel time of the UMF resins decreased gradually, but when pH stayed around 8.5, the UMF resins, regardless of F/(U+M) molar ratio, were stable and could be sprayed even after storage at room temperature for 1 month.

DSC curves obtained at 10°C/min showed exothermic peaks at slightly different temperatures. The exothermic peak could be attributed to the heat released from the curing reaction of amino groups of unreacted melamine or urea with hydroxymethylated groups. The peak temperature is defined as a temperature at which the polycondensation of any resin reaches the maximum conversion rate. The peak temperatures of UMF resins at different F/(U+M) molar ratios obtained from DSC curves are shown in Figure 2. The exothermic peak temperature of the prepared resin was lowered by increasing the F/(U+M) molar ratio, which indicated an increase in the resin reactivity at a higher F/(U+M) molar ratio.

Chemical structures of prepared resins were tentatively assigned from ¹³C-NMR spectra. All the spectra of prepared resins showed similar spectral patterns, regardless of F/(U+M) molar ratio. Chemical shift values of different carbon atoms of selected resins were assigned (Table 3) and compared with published articles (Pizzi 1994, No and Kim 2004). The content of hydroxymethyl groups [-N-(CH₂-OH)₂, -N-CH₂-OH] and methylene groups [-(CH₂)-N-CH₂-N-, H-N-CH₂-N-H] calculated from the integral values of

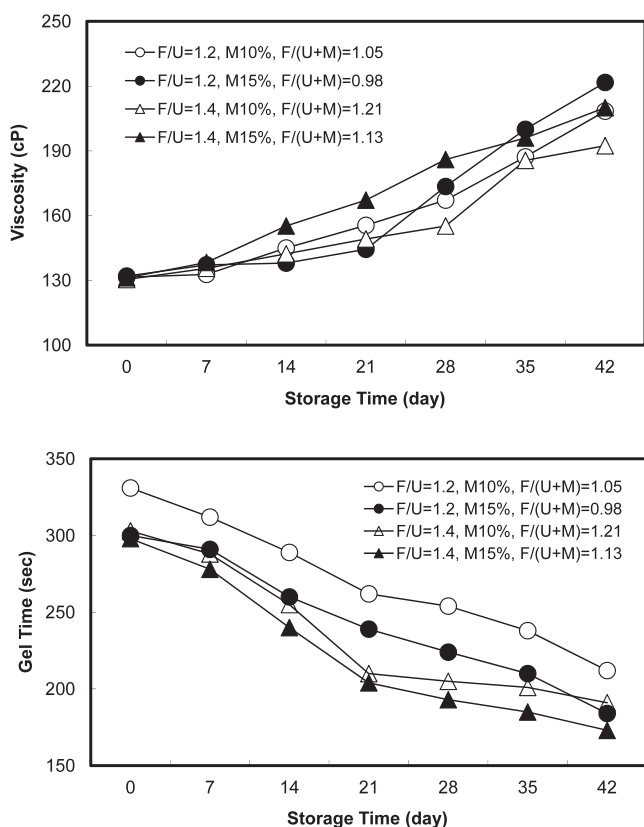


Figure 1.—Storage stability of urea-melamine-formaldehyde (UMF) resins: change of viscosity (top) and change of gel time (bottom).

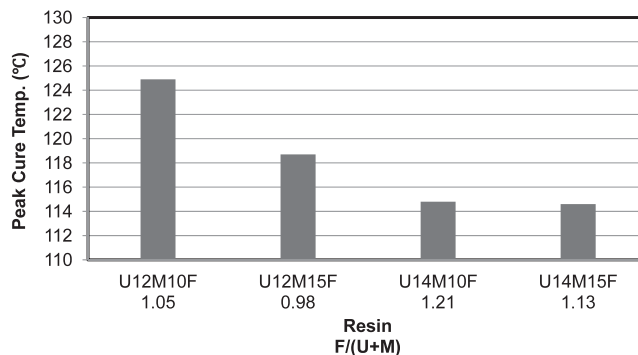


Figure 2.—Peak curing temperatures of prepared urea-melamine-formaldehyde (UMF) resins.

assigned carbon signals increased as the F/U molar ratio increased.

Functional groups of the resins were monitored with FT-IR spectra. At 10 percent melamine content, when the F/U molar ratio was increased, the N-H stretching band in UMF resin tended to move toward a higher wave number region. The N-H stretching band for the resin with F/U molar ratios of 1.4 and 1.2 appeared at 3,336.7 and 3,336.0 cm⁻¹, respectively (Fig. 3). When 15 percent melamine was added, the N-H stretching band (not shown in Fig. 3) for the resin with F/U molar ratios of 1.4 and 1.2 appeared at 3,339.7 and 3,335.2 cm⁻¹, respectively. This result can be explained by excess amounts of formaldehyde at the higher F/U molar ratios reacting to form more branched hydroxymethylated ureas. Theoretically four hydrogen atoms on a urea might be substituted by formaldehyde and form hydroxymethyl end groups; with higher molar ratios, more branching will occur and hydrogen bonding of free primary N-H groups will decrease. As the result, the N-H band would shift to a slightly higher wave number region. From the results of ¹³C-NMR spectra and FT-IR spectra, certain chemical structures of the prepared UMF resins can be assumed. The structures of UMF resins can be proposed as more linear and two dimensional with the lower F/U molar ratio of 1.2 (Fig. 4a) compared with the more branched structure with the higher F/U molar ratio of 1.4 (Fig. 4b).

All MDFs showed density values in the range of 0.73 to 0.78 g/cm³. Dry IB strength values of MDFs were directly proportional to density of MDF and ranged from 0.46 to 0.66 MPa (Fig. 5). All dry IB values exceeded the minimum value of 0.50 MPa for TYPE 25 MDF according to Korean Standard KS F 3200 (KSSN 2006). Resins with F/(U+M) molar ratios over 1.13 may have produced more branched hydroxymethylureas and more dimethylether groups in the resin structure compared with an F/(U+M) molar ratio below 1.05. Therefore, high molar ratios used in a resin should give higher reactivity with melamine and will consequently form stronger bonds than resins at low molar ratios. TS values of MDFs tended to decrease with increased F/(U+M) molar ratio. However, TS values were higher when high melamine content was used at the same F/U molar ratio. It is believed that the more melamine-containing resin should form stronger bonds than the resin containing less melamine, but the cured resin network forms a more random configuration with the resin containing more melamine. For that reason, moisture may penetrate into the pores of a cured resin network when in contact with water, promoting hydrolytic degradation of cured resin bonds and

Table 3.—Chemical shift values of prepared urea-melamine-formaldehyde resins.

Resin ^a	Chemical shift (ppm)				
	-(CH ₂)-N-CH ₂ -O-CH ₂ -N-	-N-(CH ₂ -OH) ₂	-N-CH ₂ -OH	-(CH ₂)-N-CH ₂ -N-	H-N-CH ₂ -N-H
F/U = 1.0, M 15%	—	—	63.5	—	48.7
F/U = 1.2, M 10%	72.1	67.9	63.7	54.3	48.9
F/U = 1.2, M 15%	72.2	67.9	64.0, 63.8	54.6, 54.5	49.0, 45.8
F/U = 1.4, M 10%	72.2	67.9	63.9, 63.8	54.6	48.9
F/U = 1.4, M 15%	72.2	67.9	63.9, 63.7	54.3	48.9

^a F/U = formaldehyde-to-urea molar ratio; M = melamine.

give high TS values. TS values of MDF manufactured with UMF resins met the minimum Korean standard value of 17 percent for MDF with thicknesses below 7 mm (Fig. 6), regardless of resin formulations. MOR values of MDFs ranged from 27.0 to 34.8 MPa and varied with different F/(U+M) molar ratios; the values were above the minimum value of 25.0 MPa for TYPE 25 MDF according to Korean standards (Fig. 7). The amounts of formaldehyde emitted

from MDF manufactured with laboratory prepared UMF resin adhesives are shown in Figure 8. Formaldehyde emissions for all cases were lower than E0 grade (0.5 mg/liter) of Korean Standard KS M 1998-4 (KSSN 2005).

MDF manufactured with a UF resin and a UMF resin did not meet the 3 percent required by Standard JIS K 1571 (JIS 2004) for the decay and the termite resistance tests. The mass losses that resulted in the specimens during the 12-

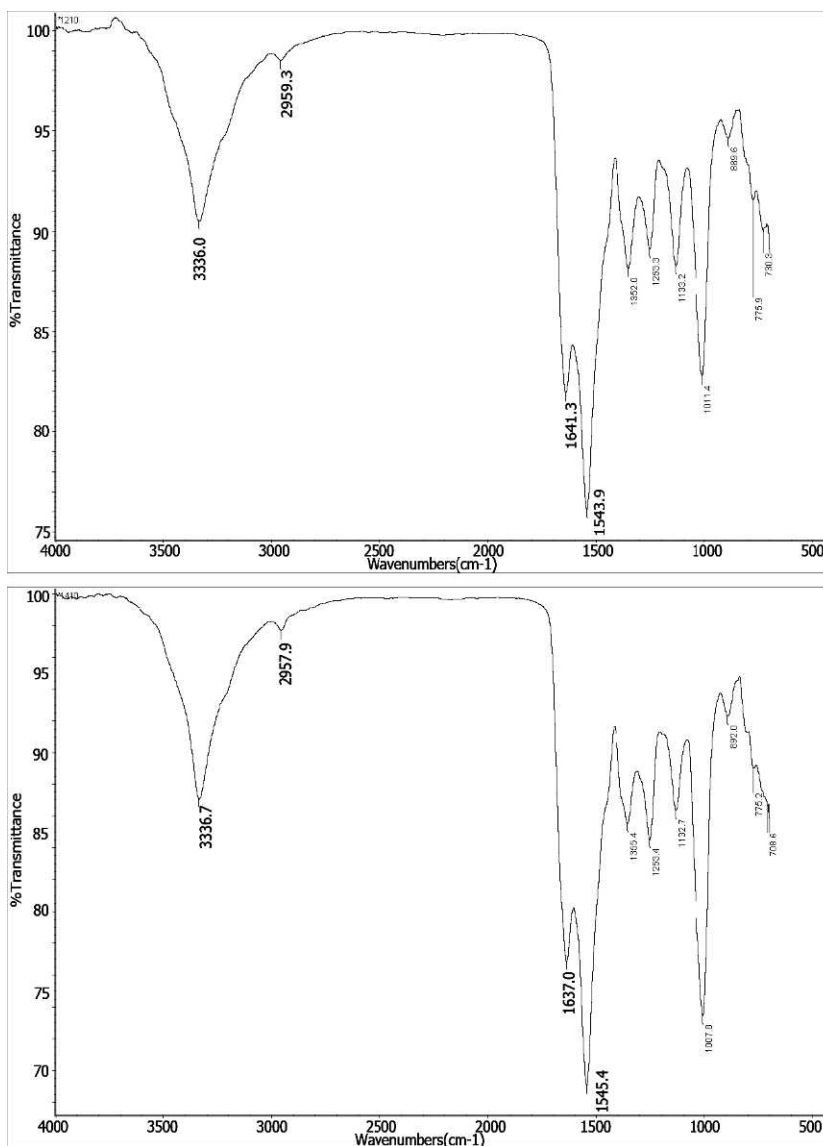


Figure 3.—N-H bands on Fourier transform infrared spectra of prepared urea-melamine-formaldehyde (UMF) resins: F/U molar ratio = 1.2 (top) and 1.4 (bottom).

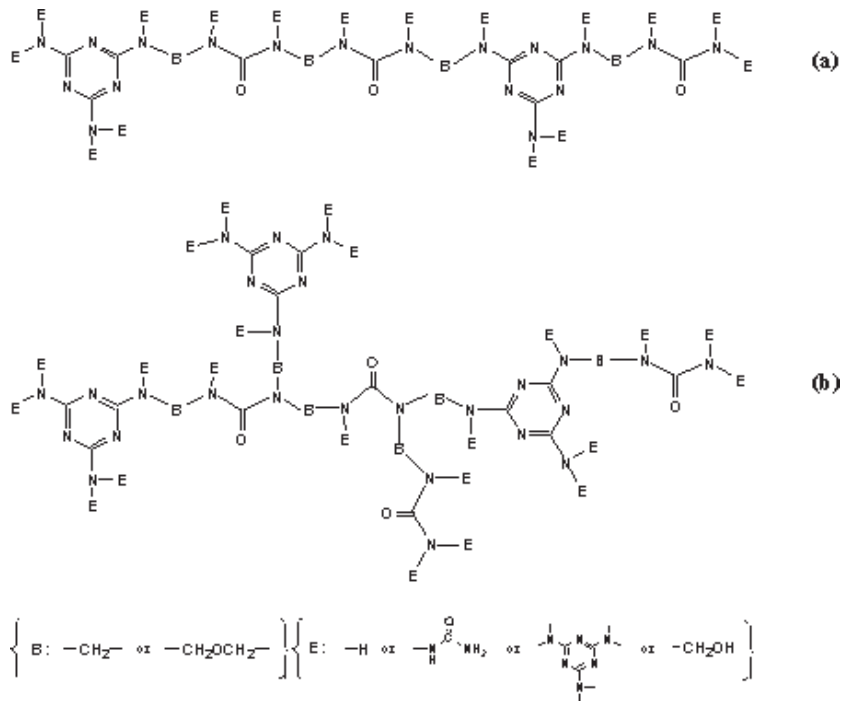


Figure 4.—Proposed chemical structures of prepared urea-melamine-formaldehyde resins: formaldehyde/urea molar ratio = 1.2 (a) and 1.4 (b).

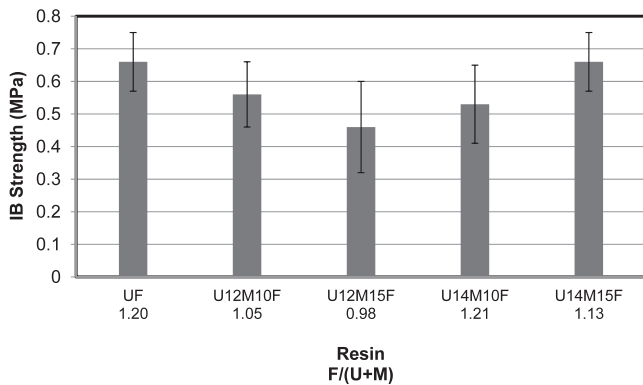


Figure 5.—Dry internal bond (IB) strength values of medium-density fiberboards. U = urea; F = formaldehyde; M = melamine.

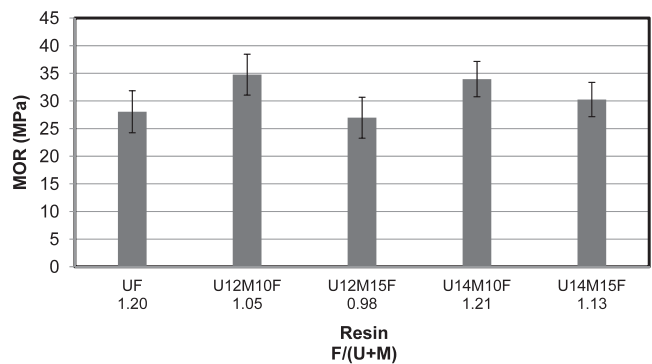


Figure 7.—Modulus of rupture (MOR) strength values of medium-density fiberboards. U = urea; F = formaldehyde; M = melamine.

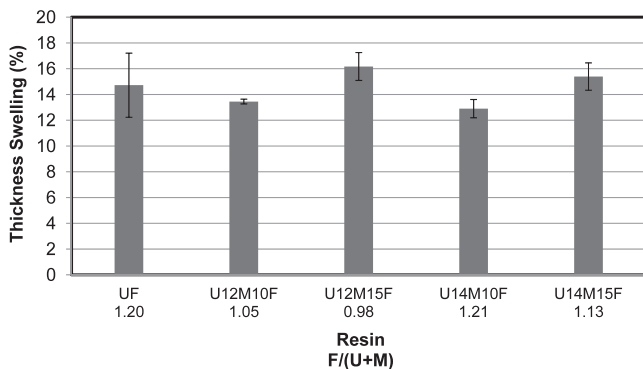


Figure 6.—Thickness swelling values of medium-density fiberboards. U = urea; F = formaldehyde; M = melamine.

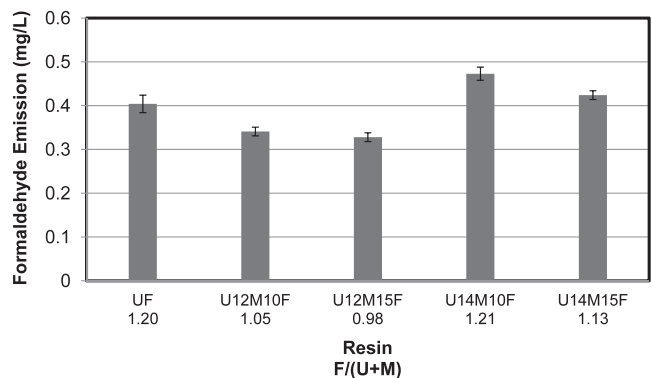


Figure 8.—Formaldehyde emission amount of medium-density fiberboards tested by KS M 1998-4 (desiccator method; Korean Standards Service Network 2005).

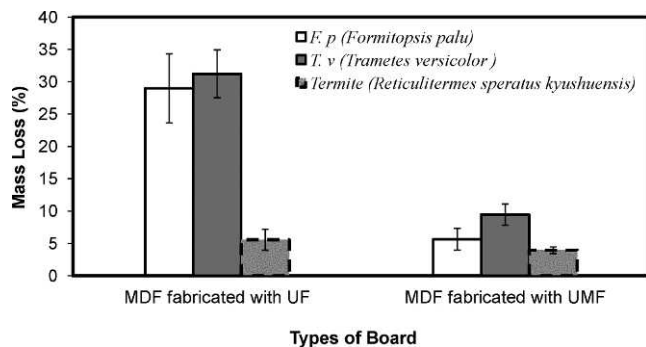


Figure 9.—Mass losses of the specimens after 12-week exposure to wood decay fungi and after 3-week exposure to termites. MDF = medium-density fiberboard; UF = urea-formaldehyde; UMF = urea-formaldehyde-melamine.

week fungal decay tests and during the 3-week termite resistance tests are shown in Figure 9. A slightly better termite resistance and higher termite mortalities were found for the MDF manufactured with a UMF resin compared with the MDF manufactured with a UF resin. The termite mortality of the MDF manufactured with a UMF resin was 99 percent and the termite mortality of the MDF manufactured with a UF resin was 96 percent. Fungal decay and termite resistance tests revealed that melamine, which is co-condensed with urea for manufacturing the UMF resin, can increase bond durability.

Conclusions

This study was conducted to investigate the effects of melamine on the properties of MDF fabricated with UMF resins. Physical properties of UMF resins tended to be influenced by the F/M molar ratio, but not by the F/(U+M) molar ratio. At the same F/U molar ratio, free formaldehyde content and gel time of resins decreased as melamine content was increased. Curing temperature of UMF resins was also influenced by the F/U molar ratio, but not by the F/(U+M) molar ratio. Chemical structures of resins could be

proposed from the results of ¹³C-NMR and FT-IR analysis. Linear two-dimensional structure of UMF resin was mainly formed at the F/U molar ratio of 1.2, and a branched structure of UMF resin was mainly formed at the F/U molar ratio of 1.4.

It was difficult to find a correlation between mechanical properties of MDFs and F/(U+M) molar ratio, except for formaldehyde emission; formaldehyde emissions of MDFs decreased as the F/(U+M) molar ratio decreased.

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