

Formaldehyde Emissions from Urea-Formaldehyde- and No-Added-Formaldehyde-Bonded Particleboard as Influenced by Temperature and Relative Humidity

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Abstract

It is well documented that temperature and humidity can influence formaldehyde emissions from composite panels that are produced using urea-formaldehyde (UF)-type adhesives. This work investigates the effect of temperature and humidity on newer commercial California Air Resources Board (CARB) phase II-compliant particleboard produced with UF-type adhesives. These results were compared with laboratory particleboards prepared with the no-added-formaldehyde (NAF) Soyad adhesive technology. A modified version of EN 717-3 (“Formaldehyde Release by the Flask Method,” ÖNORM 1996) was used to collect formaldehyde emissions that were quantified using the acetylacetone method. The formaldehyde emissions from the commercial particleboard panel bonded with a UF-type resin increased greatly when panels were exposed to higher heat and humidity than in normal testing protocols. Furthermore, the rate of emission for these UF-bonded panels increased with longer exposure at 100 percent relative humidity. In contrast, formaldehyde emissions from particleboard bonded with the NAF adhesive were relatively stable and significantly lower compared with those bonded with UF at all temperature and relative humidity conditions. This work highlights the potential for increased long-term formaldehyde emissions even from the new UF CARB phase II-compliant adhesive systems.

Increasing concerns over the quality of indoor air has led to some recent changes in legislation and a general preference for more stringent limits on the quantity of formaldehyde that can be emitted from consumer products intended for indoor use. Wood composites bonded with urea-formaldehyde (UF)-type adhesives have been identified as a source of indoor formaldehyde emissions (Kelly 1996). Formaldehyde release from interior wood composites has been a long-standing issue leading to the adoption of voluntary standards in the United States and Europe in the 1980s that placed limits on formaldehyde emissions (Österreichisches Normungsinstitut [ÖNORM] 2002; American National Standards Institute [ANSI]/Hardwood Plywood and Veneer Association [HPVA] 2004; ANSI 2009a, 2009b). These voluntary standards led to lower formaldehyde-emitting wood composites, but in subsequent years product emissions in the United States stayed constant while

products in Europe and Japan have moved to lower emission levels.

The recent standards adopted by the California Air Resources Board (CARB) are intended to significantly reduce and regulate formaldehyde emissions in composite wood products (CARB 2009). The CARB standard was also the basis for the national 2010 “Formaldehyde Standards for Composite Wood Products Act” (US Congress 2010).

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The new standards have led to new UF adhesives with lower formaldehyde emissions (Dunky 2005), as well as opened the door for no-added-formaldehyde (NAF) adhesives such as soy-based adhesives (Allen et al. 2010, Wescott et al. 2010), polymeric diphenylmethane diisocyanate, and certain types of poly(vinyl acetate) adhesives. Although both classes of adhesives, UF and NAF, can meet the CARB phase II formaldehyde emissions limits, there is a concern about the long-term emission potential of UF adhesives, in particular when exposed to higher temperature or humidity levels than specified in the current testing methodology for measuring formaldehyde emissions.

Although significant progress has been made on UF adhesives, most notably the fortification and copolymerization with melamine, the fundamental chemistry of UF is probably unchanged and could remain susceptible to hydrolysis, but this has not been experimentally determined. The reaction of urea with formaldehyde first produces hydroxymethylated urea that condenses to yield methylene and dimethylene ether-bridged urea polymers (Meyer 1979, Pizzi 2003). Although these reactions are not unlike the steps to produce other formaldehyde-containing wood adhesives, the UF polymers are distinct in that they are susceptible to hydrolysis under some typical use conditions (Myers 1986a). The reaction shown in Figure 1 for urea and formaldehyde illustrates the problem with depolymerization in that it can yield additional free formaldehyde, especially if free water is present.

The presence of free water (moisture) in the composite panel, as would be the case in higher humidity conditions, will tend to drive the reverse reaction, yielding more formaldehyde that is emitted over time. Myers showed that formaldehyde adsorbed onto wood reaches an emission plateau in about 7 days at 80 percent relative humidity (RH) and 27°C as does phenol-formaldehyde-bonded particleboard. In contrast, UF-bonded particleboard continues to emit formaldehyde for more than 30 days without reaching a plateau (Myers 1986b). In addition, Myers' own data and his analysis of the literature data showed that formaldehyde emissions increase from UF-bonded wood composites at higher humidity and temperature conditions (Myers and Nagaoka 1981, Myers 1985). This work was done with composites bonded with more traditional UF-based adhesives. It was unclear if the new lower emitting UF systems suffer from the same level of hydrolytic instability and subsequent high formaldehyde emissions. A similar study conducted on the formaldehyde emissions of ultra-low-emitting-formaldehyde (ULEF)- and NAF-bonded hardwood plywood (Frihart et al. 2010) showed that higher temperatures and humidity led to significantly higher formaldehyde emissions from plywood bonded with UF adhesive. The current article reports on particleboard with

more extensive higher temperature and humidity conditions.

Wood itself has been shown to generate significant levels of formaldehyde when exposed to certain conditions common to the composite panel manufacturing process (Schäfer and Roffael 2000, Roffael 2006). This so-called "native" formaldehyde has been shown to be transient, and it rapidly decreases to levels below those set by the standards (Birkeland et al. 2010). The production of formaldehyde from wood has been shown to occur at conditions of very high heat and would not be expected to be a significant source of formaldehyde in composite wood products during service. However, based on the work of Birkeland et al., our expectation is that the native formaldehyde in particleboard will be significantly greater than that seen in plywood, likely a result of the process used to prepare the particles as well as the higher press temperatures typically used in the production of particleboard.

Currently, the primary standard test method in the United States for measuring and regulating formaldehyde emissions in composite wood panels is the ASTM E1333 large chamber test (ASTM International 2002). Secondary methods can be used as well; however, all methods must prove equivalence to the primary method. In E1333, samples are conditioned at 25°C and 50 percent RH for 7 days and then tested at the same temperature and RH conditions. Based upon the available literature, some questions arise regarding formaldehyde emissions from composite wood panels:

- Given that the standard test method, ASTM E1333, uses 25°C (77°F) and 50 percent RH, is this representative of the exposure that all interior composite wood products will experience in service?
- How do the formaldehyde emissions in CARB phase II-certified composite wood products bonded with UF adhesives compare with those bonded with NAF adhesives over the range of temperatures and RH that they may experience in service?

The focus of this study was aimed at answering these questions, using a modified version of EN 717-3 (ÖNORM 1996) to test the effects of temperature and RH on commercial CARB phase II-compliant particleboard bonded with UF and compared with similarly prepared (same furnish and pressing conditions) laboratory-produced particleboard bonded with an NAF adhesive.

Methods

Samples

Commercially produced particleboard samples used for testing were 3/4 inch (19.0 mm) thick supplied from a commercial collaborator. These samples were collected and

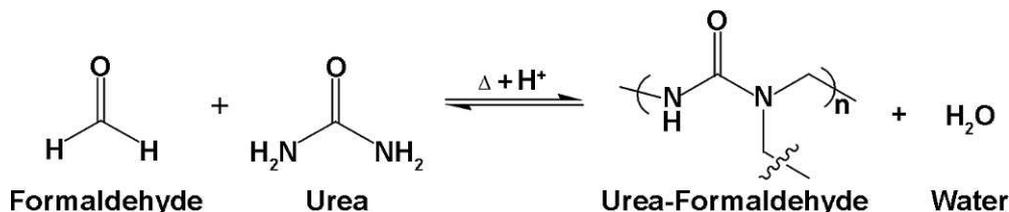


Figure 1.—The reaction of urea with formaldehyde to form the urea-formaldehyde polymer.

shipped according to ASTM E1333. Samples of the same face and core furnish were supplied to our laboratory where comparable NAF particleboard panels were made for comparison. Although the authors agree that a laboratory process is not an exact replication of commercial conditions, the laboratory NAF panels were processed as close to the commercial panels as possible using similar press temperatures, press times, board thicknesses, moisture contents, face-to-core furnish ratios, and resin loads. A pinch collar was used to match the higher internal steam pressures of commercial production. This steel collar was pressed into the panels around their edges to inhibit the release of steam, mimicking the conditions for a commercial size panel (Cai et al. 2009). Table 1 shows the commercial UF-bonded and laboratory NAF-bonded panels had similar physical properties of internal bond strength, modulus of rupture, and apparent density.

The NAF adhesives used were Soyad SD419 adhesive (Ashland, Wilmington, DE) in the face section and DPA-424 adhesive in the core. It should be noted that Soyad SD419 adhesive contains some urea to scavenge the native formaldehyde. Both the commercial and laboratory-produced panels were sanded, wrapped, and sealed in plastic within 48 hours after hot pressing and remained in plastic until testing. The commercial particleboard samples were reported by the mill to be CARB phase II compliant (<0.09 ppm).

Soyad NAF particleboard adhesives are a proprietary blend of soy flour, a polyamidoamine co-reactant, and specific diluents. Other NAF options are available, such as isocyanate-based resins and poly(vinyl acetate)s. The authors chose Soyad NAF adhesives because the face adhesive utilizes a formaldehyde scavenger, just as the commercially produced UF-based particleboard does. Using a scavenged Soyad NAF adhesive establishes the lowest possible formaldehyde emissions baseline for comparison to UF-bonded particleboard.

Modified EN 717-3 (WKI bottle method)

A modified version of EN 717-3 was used in this study, and Table 2 defines the specific modifications. RH was controlled as follows: 30 percent RH (with saturated MgCl₂), 75 percent RH (with saturated NaCl), and 100 percent RH (reverse osmosis H₂O; Wexler 1961).

The test procedure (Fig. 2) used a Nalgene 500-mL wide-mouth polypropylene bottle (Sigma Aldrich, Milwaukee, WI), with the cap modified with a stainless steel eyelet and a stainless steel nylon lock nut. Fifty milliliters (via burette) of either H₂O (100% RH), concentrated MgCl₂ (30% RH), or concentrated NaCl (75% RH) solution was placed in the bottom of the bottle. Three 1 by 1-inch (2.54 by 2.54-cm) samples were weighed to 0.1 g, stacked on top of each other (faces together), and bound with a rubber band. The bound samples were then suspended above the solution in the

Table 1.—Physical properties of commercial and laboratory panels.^a

	Internal bond (psi)	Modulus of rupture (psi)	Apparent density (lb/ft ³)
Commercial UF-bonded	86.5 ± 9.0	1,999 ± 179	44.6 ± 1.3
Laboratory NAF-bonded	91.2 ± 10.1	1,973 ± 111	46.8 ± 1.1

^a UF = urea-formaldehyde; NAF = no added formaldehyde.

Table 2.—Summary of modifications to EN 717-3.

	EN 717-3	Our method
Temperature (°C)	40	25 and 35
Test duration	3 h	1–4 d
Relative humidity (%)	100	30–100

bottle by attaching the rubber band to the eyelet. The bottle was kept in a temperature-controlled room (25°C) or in a water bath (35°C) for the allotted time (24, 48, or 96 h). Upon completion of the test, the samples were carefully removed, and the solution was collected for analysis. The samples were analyzed for formaldehyde on the same day using the acetylacetone method. One replicate was performed for each test.

Acetylacetone method

For the acetylacetone method (Nash 1953, Belman 1963, ÖNORM 1992), a calibration curve was generated using a standard formaldehyde solution prepared from 37 percent formaldehyde (Sigma Aldrich) titrated to determine formaldehyde concentration. A seven-point calibration curve was generated using standards with concentrations from 0 to 34.65 µg/mL formaldehyde and yielding an R² value of 0.99998. Equal parts (2 mL) of 0.4 percent acetylacetone (Fisher Scientific, Fair Lawn, NJ), 20 percent ammonium acetate (Daigger Chemical, Vernon Hills, IL), and a standard or sample solution were combined in a test tube. The mixture was heated to 40°C for 15 minutes in a water bath and then cooled to 25°C. The cooled solution was placed in the dark for 1 hour. The mixture was then analyzed for absorbance with a spectrophotometer at a wavelength of 412 nm. The formaldehyde concentration of



Figure 2.—Sample set-up for modified EN 717-3 method (ÖNORM, 1996).

the sample was determined based on the calibration curve. The results were converted to micrograms of formaldehyde emitted per gram of wood. Previous work using the same saturated salt solutions showed that there was no interference from the salts on the formaldehyde concentrations (Frihart et al. 2010).

Results and Discussion

A static formaldehyde emissions technique was used to assess the changes in emissions for composite wood products as a function of temperature and humidity. The method was a modified version of the EN 717-3 method. The modifications to this method are outlined in Table 2. The purpose of the modifications was to allow the test to be run under varying temperatures, RH, and durations to better understand the formaldehyde emission potential of composite panels under a variety of possible exposures. The 100 percent RH was higher than typical, but accelerated tests are generally run under more severe conditions than normal exposures because of shorter times under those conditions.

Studies were run both separately (one test water/bottle for the duration of the experiment) and sequentially (periodically new test water/bottle) over time. At the end of a separate test, the specimens were removed and discarded, and the solution was collected for analysis. At the end of a sequential test, the solution was collected for analysis, but the test specimens were transferred into a fresh bottle with fresh solution for additional exposure time. The purpose of this study was to assess the ability of the test method to continue to retain the formaldehyde and to better assess the longer term formaldehyde emission potential.

Two particleboard specimens were evaluated in this study. Both specimens were of the same furnish source with one being commercially produced using a UF-type adhesive and the other being laboratory produced (34 by 34 in., 86.4 by 86.4 cm) with a soy flour-based Soyad adhesive technology. Although careful attention was paid to reproduce the commercial process in a laboratory setting, the authors recognize that this is a difference, but do not consider the results or conclusions to be in question as a result of this difference. The commercial UF specimens were confirmed by the manufacturer to make a CARB phase II-compliant particleboard.

The ASTM E1333-96 large chamber method and any correlated small chamber method per ASTM D6007-02 (ASTM International 2008) must be run at 25°C and 50 percent RH. It is likely that these conditions may be typical for homes in the United States, in particular, those that contain and operate an air conditioning system. However, there are many instances in which these panels used within the interior of a home or trailer/recreational vehicle may be subjected to extended periods of time at temperatures higher than 25°C and/or RH levels higher than 50 percent. The data in Figures 3A (www.weather.com) and 3B (www.cityrating.com/relativehumidity.asp) show that ambient conditions in the majority of the United States are in fact often actually much higher than 50 percent RH. Most notably, the southeastern region is substantially higher during the summer months in both RH and temperature (Table 3) compared with the test conditions. It is this finding that led us to evaluate composite panel emissions as a function of temperature and humidity.

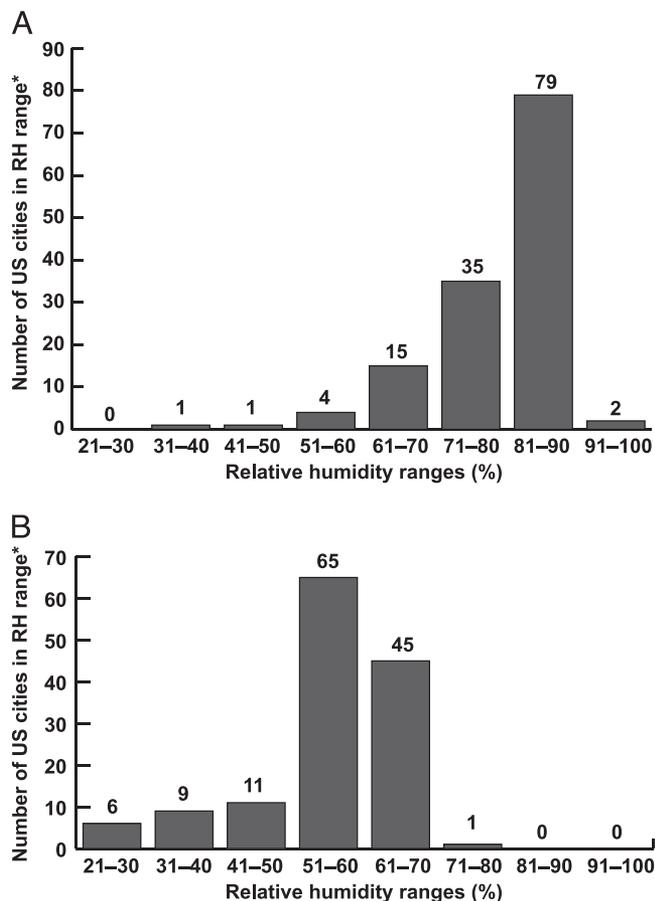


Figure 3.—Relative humidity distribution of 137 US cities: (A) morning (average = 79%) and (B) evening (average = 55%).

In this section of the study, we used water (100% RH) and saturated solutions of $MgCl_2$ (30% RH) and $NaCl$ (75% RH) to control the RH inside the sample bottles. See the experimental section for details. These experiments were run at both 25°C (77°F) and 35°C (95°F). Table 4 shows the tabulated results with the days indicating the duration of the test.

To better analyze the data, several charts were constructed from the data shown in Table 4. Figures 4A and 4B show the emissions as a function of RH for both the 25°C and 35°C data sets. These results clearly show that the commercial UF panel emitted significantly higher formaldehyde levels when subjected to higher RH levels and that this was further exacerbated by a concomitant increase in temperature.

A comparison between the results of the previously published plywood emissions and our particleboard emissions is shown in Figures 5A and 5B. These new particleboard results are consistent with the findings in the previous article on hardwood plywood, although the emission level increase is actually slightly less in the particleboard case (at least at the 100% RH level), suggesting that the adhesive system used in these commercially produced particleboards may be slightly more resistant to hydrolysis or may contain an even greater level of scavenger than was used for the hardwood plywood panels. The fact that the emissions for the particleboard are actually higher at 75% RH is of notable concern. It was also

Table 3.—Summer temperature and relative humidity (RH) averages for select US cities.

	Avg. low temp. Jun–Aug, °F (°C)	Morning RH (%)	Avg. high temp. Jun–Aug, °F (°C)	Evening RH (%)
Chicago, IL	64 (18)	80	82 (28)	62
Fargo, ND	57 (14)	81	80 (27)	64
Houston, TX	75 (24)	90	93 (34)	63
Kansas City, MO	70 (18)	81	88 (31)	63
Las Vegas, NV	76 (24)	39	102 (39)	21
Los Angeles, CA	64 (18)	79	83 (28)	65
Miami, FL	76 (24)	83	91 (33)	61
New York, NY	66 (19)	72	80 (27)	56
Raleigh-Durham, NC	67 (19)	85	86 (30)	54
Seattle, WA	55 (13)	83	73 (23)	62

interesting to note that even the NAF-bonded panel showed a slight increase in emissions as the RH and temperature increased, most likely a result of softening of the wood and increasing the porosity or ability of the native formaldehyde to be released. Furthermore, the NAF adhesive system in this case does contain some scavenger and this too may be contributing to the slow increase in the emissions rate at these higher temperature and humidity conditions.

We were also interested in the rate of emissions in this study. To assess the rate of emissions, individual samples were tested at 1-, 2-, and 4-day increments (separate experiments). The results of this study are shown in Figures 6 and 7 (note the differences in scale for each set of figures). These results show that the rate of emissions was significantly greater for the UF-bonded panels in all cases and that the UF-bonded panels were much more prone to high levels of emissions when exposed to the higher humidity environments than the NAF-bonded panels. Of interest was that in the 25°C study, all the panels continued to increase in emissions over time. This is most likely a result of the wood softening with extended exposure to higher humidity levels, and possibly because of the hydrolysis of the scavenged native formaldehyde and also swelling of the wood exerting more force on the chemical bonds. However, for the 35°C study, the rate of emissions, although much higher, appeared to decrease over time. To further test this, the emission levels were also measured using a sequential process. Rather than leaving the specimen in the test bottle for continuous days, the water was removed and replaced with fresh water and a clean bottle after day 4 and day 6. Thus, we had a sequential run of a 4-day test, a 2-day test, and a 1-day test. Of interest was the emission level of the 1-day test after 6 days of exposure compared with the results of the 1-day test with no previous days of exposure. The theory

was that if the 1-day sequential test (6 days of exposure) was significantly less than the separate 1-day test (0 days of exposure), this would indicate a decreasing rate of formaldehyde emission from the panel. The results in Figure 8 and Table 5 show that this was not the case. In fact, the emission rate for the sequential 1-day test (after 6 days of exposure) was actually 11 percent higher than it was initially for the UF-bonded panel and 9 percent lower for the NAF-bonded panel. These results suggest that the slight decrease in the emission rates observed in Figure 7A are an artifact of the test method and not the result of decreased emission rate over time.

The effect of temperature and humidity on formaldehyde emissions reported in Table 4 is similar to the effects seen by Myers and Nagaoka (1981). Myers and Nagaoka using a dynamic chamber method and UF-bonded particleboard, showed that moving from 25°C and 30 percent RH to 25°C and 75 percent RH resulted in an approximate twofold increase in formaldehyde emissions. An examination of the data in Table 4 shows that at 25°C, the increase from 30 to 75 percent RH yields a 3.6-, 3.5-, and 2.9-fold increase in emissions for the 1-, 2-, and 4-day data, respectively.

Furthermore, when Myers and Nagaoka (1981) changed the temperature from 25°C to 40°C and the RH from 30 to 75 percent, the resultant increase in formaldehyde emission was approximately sixfold. An analysis of data from Table 4 shows that moving from 25°C and 30 percent RH to 35°C and 75 percent RH yields 24.1-, 25.0-, and 13.3-fold increases in emissions for the 1-, 2-, and 4-day data, respectively. These latter results suggest that the new UF-type adhesives still struggle with formaldehyde emissions when the temperature and the humidity are increased.

Although Myers and Nagaoka (1981) did not conduct analyses at RH levels above 75 percent, in a comprehensive literature survey, Myers was able to derive quantitative

Table 4.—Formaldehyde emissions via the modified EN 717-3 method.^a

No. of days	Temp. (°C)	UF at RH (%):			NAF at RH (%):		
		30	75	100	30	75	100
1	25	0.5	1.8	4.0	0.0	0.2	1.0
2	25	1.0	3.5	8.3	0.1	0.9	1.9
4	25	3.3	9.5	19.6	0.7	2.6	4.3
1	35	5.7	12.1	22.4	1.0	2.7	4.3
2	35	6.3	25.0	45.3	1.6	4.5	7.1
4	35	13.5	43.9	72.6	3.0	7.8	14.9

^a Values are in micrograms of CH₂O per gram of wood. UF = urea-formaldehyde; NAF = no added formaldehyde; RH = relative humidity.

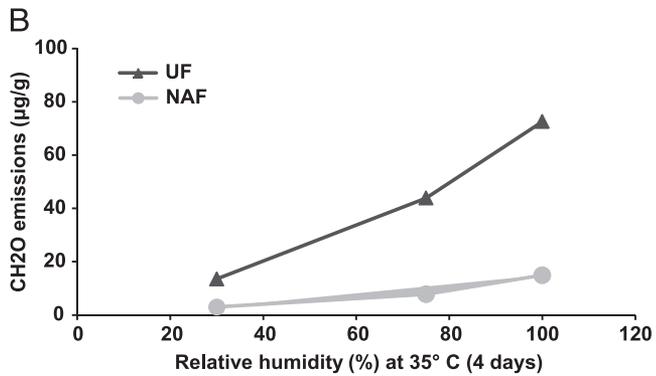
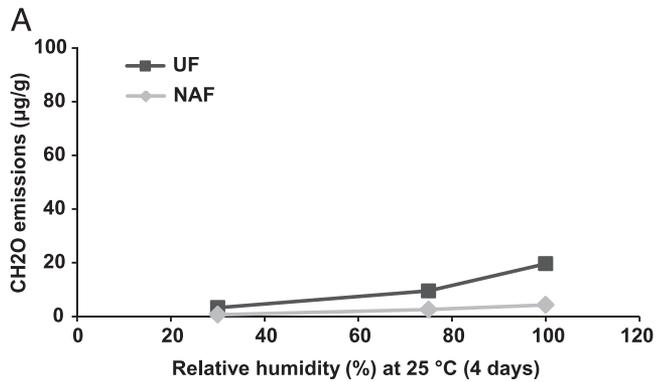


Figure 4.—Formaldehyde emissions of 4-day samples at various relative humidity levels: (A) at 25°C and (B) at 35°C. UF = urea-formaldehyde; NAF = no added formaldehyde.

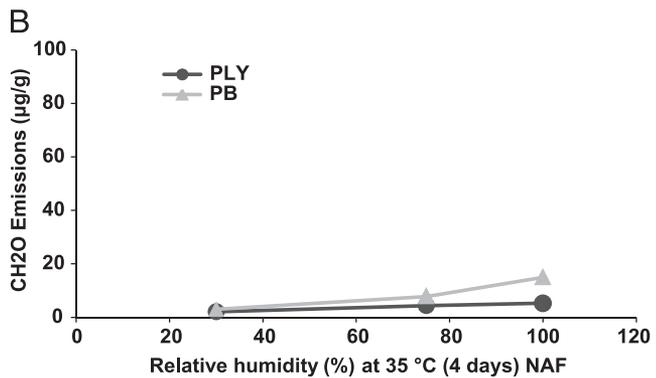
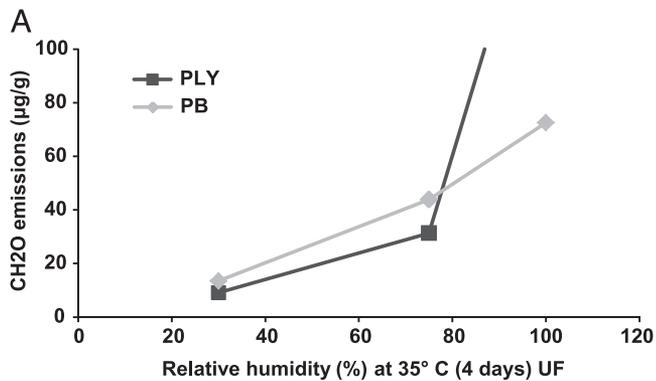


Figure 5.—Formaldehyde emissions for panels (plywood [PLY] vs. particleboard [PB]) at 35°C: (A) urea-formaldehyde (UF) and (B) no added formaldehyde (NAF).

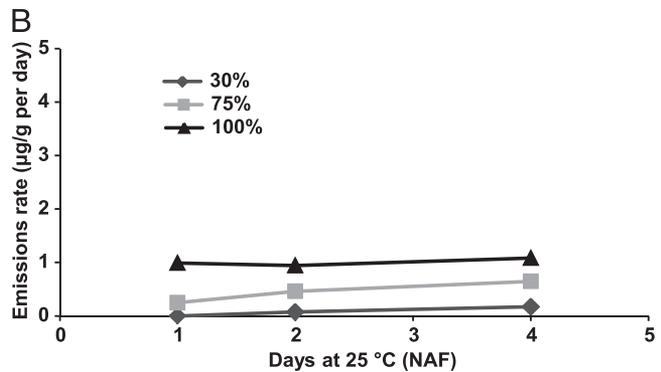
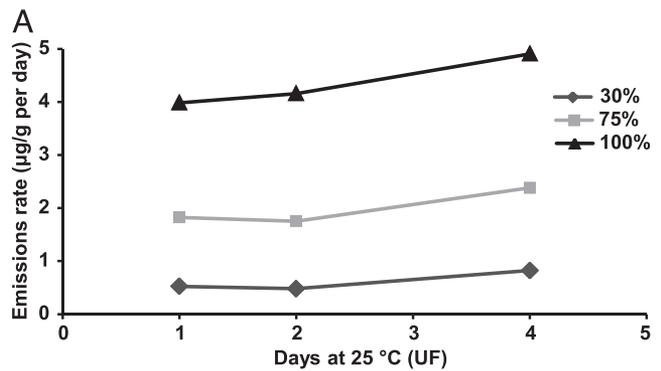


Figure 6.—Formaldehyde emissions rate as a function of percent relative humidity for panels at 25°C: (A) urea-formaldehyde (UF) and (B) no added formaldehyde (NAF).

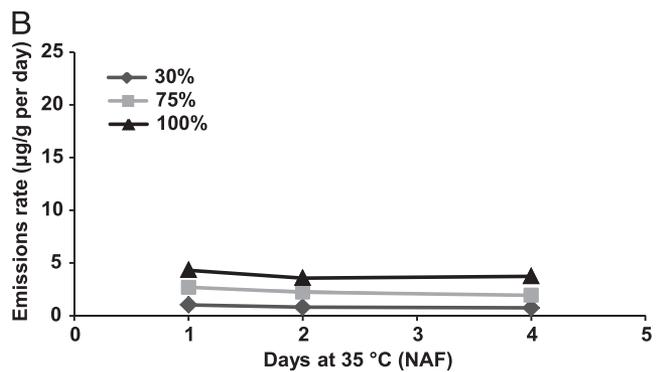
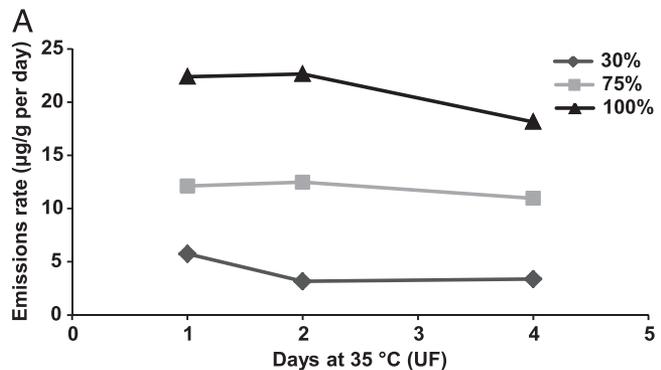


Figure 7.—Formaldehyde emissions rate as a function of percent relative humidity for panels at 35°C: (A) urea-formaldehyde and (B) no added formaldehyde (NAF).

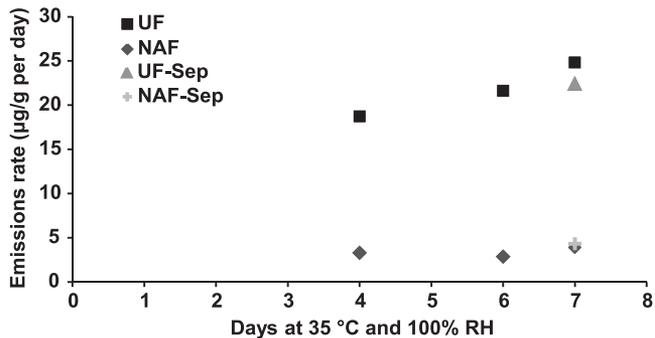


Figure 8.—Sequential versus separate (Sep) formaldehyde emissions rates of urea-formaldehyde (UF) and no-added-formaldehyde (NAF) panels at 35°C and 100 percent relative humidity.

Table 5.—One-day formaldehyde emissions as a function of exposure days.^a

	Exposure days ^b	UF	NAF
Separate	0	22.4	4.3
Sequential	6	24.8	3.9
Change (%)		10.7	-9.3

^a Values are in micrograms of CH₂O per gram of wood. UF = urea-formaldehyde; NAF = no added formaldehyde.

^b Number of days at 35°C and 100 percent relative humidity prior to test.

temperature and RH factors at a wide range of temperature and RH conditions (Myers 1985). Based on these equations, the move from 30 to 90 percent RH at 25°C is predicted to yield a threefold increase in formaldehyde emissions. The data in Table 4 show that moving from 30 to 100 percent RH at 25°C yields increases in formaldehyde emissions of 8.0-, 8.3-, and 5.9-fold for the 1-, 2-, and 4-day data, respectively. The results agree reasonably well with Myers' prediction and support the notion that the newer UF adhesives are able to meet the emissions requirements of the test method but do not maintain low levels of formaldehyde emissions at higher ambient temperature and humidity.

Conclusions

Testing of formaldehyde emissions from CARB phase II-compliant particleboard panels using a low-emitting UF-type resin and a NAF resin were carried out at six different conditions: 25°C at 30, 75, and 100 percent RH, and 35°C at 30, 75, and 100 percent RH using a modification of the EN 717-3 method. Results show that the UF-bonded particleboard product emitted more formaldehyde as the temperature and RH increased. At 100 percent RH and 35°C, a sequential test showed that the emission rate per day was 11 percent greater after 6 days of exposure; thus, no evidence of reduced emissions could be demonstrated in this study. Additionally, the NAF-bonded particleboard product showed significantly lower levels of formaldehyde emissions and had a much lower response to both temperature and humidity.

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