Significance of Native Formaldehyde in Particleboards Generated during Wood Composite Panel Production

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Abstract

Hot-pressing wood, especially as in the production of wood composites, generates significant "native" (wood-based) formaldehyde that remains in the composite panel, even in the absence of adhesive. The level of native formaldehyde relates directly to the time and temperature of the hot-pressing. This native formaldehyde dissipates in a relatively short time and is not part of the long-term formaldehyde emission issue commonly associated with hydrolysis of urea-formaldehyde bonds. We show that the common desiccator/chromotropic acid method used in our study distinctly measures formaldehyde and not other similar compounds. Furthermore, the use of no adhesive or one type of no-added formaldehyde (NAF) adhesive in making particleboard produced significant native formaldehyde levels. Properly formulated adhesives can suppress the native formaldehyde emissions leading to soy-based adhesives that provide low formaldehyde emission levels in both the short and long timescales. This work highlights an important, but often overlooked, aspect that should be considered for production quality control programs: the importance of standardizing the time and conditions employed immediately after pressing but prior to the onset of emissions testing. Implementing this change should improve reliability between process monitoring and large-chamber correlations.

Introduction

Emission of formaldehyde from composite wood products for interior applications has been a topic of concern for many years. The source of the concern is due mostly to the widespread use of urea-formaldehyde (UF) adhesives in these products and increased presence of UF-bonded wood products within the home (Meyers 1984a). In response to this concern, the acceptable emission level of formaldehyde from interior composite wood materials has decreased as defined by the American National Standard (ANSI) voluntary standards, ANSI A208.1-2009, ANSI A208.2-2009, and ANSI/HPVA HP-1-2004 for particleboard, fiberboard, and hardwood plywood, respectively (ANSI/HPVA 2004, ANSI 2009a, 2009b). Levels of acceptable formaldehyde emissions have been set in Europe and Japan that are even lower. In 2008, the California Air Resources Board (CARB) introduced and passed regulation requiring low formaldehyde emission levels for any interior composite wood product sold in California and requiring significant regulatory compliance in the form of "third party certification" with much tighter quality controls than have typically been employed in the industry (ACTM 2009).

A variety of test methods for determining formaldehyde emissions from wood products have evolved over time. Each method has its own unique set of board conditioning and test conditions that possess both positive and negative attributes. The ANSI standards, specifically A208.1-2009 (ANSI 2009a), call for use of a large-chamber test ASTM E 1333 (ASTM 2002) and most of the industry uses this method for determining compliance with the voluntary standard along with ongoing tests at the plant using a small chamber with a predetermined correlation factor. CARB also requires use of the large-chamber test

for certification, but may also allow other approved secondary methods for ongoing plant quality assurance if proper correlation to the large-chamber method can be demonstrated. Some of the negative attributes for the large-chamber test include the significant cost of the equipment, the amount of sample required and the time required to run the test. These restrictions make the large-chamber test impractical for quality assurance in commercial production.

American industry has used several other tests as a way of measuring relative formaldehyde emission levels in lieu of the costly and time-consuming large-chamber test. The desiccator test as described in ASTM D 5582-00 (ASTM 2006) is a widely used test for rapid relative formaldehyde determination. With its inexpensive materials and simple procedure, this method is an ideal quality assurance test for commercial production. The desiccator test has been shown to provide reasonable correlation to the large chamber test (Meyers 1983, Que and Furano 2007). No matter which method is used to collect formaldehyde from the composite wood sample, formaldehyde content is ultimately determined using colorimetric analysis based on the chemistry of either chromotropic acid (Eegriwe 1937) or acetyl acetone (Nash 1953, Czech 1973), with the former being more common.

Many studies have been conducted with the aim of measuring, understanding, and/or reducing formaldehyde release from UF-bonded products (Meyers 1986). As expected, the studies clearly show that the more formaldehyde in the UF resin, the more formaldehyde is emitted from the panel. As a result of this research, typical F:U ratios (formaldehyde to urea) in urea formaldehyde resins have been lowered significantly, and the use of a number of scavenger and catalyst technologies have been developed to meet increasingly stringent emissions standards (Meyers 1984b, Elbert 1995). Undoubtedly for all wood-bonding adhesives, UF has the greatest problem with long-term emission of formaldehyde, as UF is highly susceptible to degradation and formaldehyde release, especially under conditions of high temperature and high humidity (Meyers and Nagaoka 1981, Meyers 1985), conditions that are not currently probed in the widely used test standards.

As formaldehyde emission regulations drive toward ever-lower emission levels, a natural question arises about the background level of formaldehyde from wood. Several studies have been done measuring formaldehyde emissions from solid wood and dried wood chips (Meyer and Boehme 1997, Funch 2002, Roffael 2006, Weigl et al. 2009). From this work, the formaldehyde levels derived from wood were inferred to be insignificant contributors to the total concentration of formaldehyde obtained from traditional UF-bonded composite wood panels. However, these levels become more significant with the new CARB limits (ATCM 2009).

One study has shown that wood emits increasing levels of formaldehyde as temperature and time of heat-treatment increase (Schäfer and Roffael 2000). Schäfer and Roffael propose a number of chemistries using model compounds that could explain the emissions of formaldehyde from wood.

Formaldehyde and other volatile organic compound (VOC) emissions during pressing have been measured in a number of studies (Carlson et al. 1995, Baumann et al. 2000, Jiang et al. 2002). These studies were able to identify formaldehyde as a significant constituent of in-press emissions, even in the absence of any resin applied to the wood. No testing of these boards was done, however, likely because in the absence of any adhesive the boards are not suitable for measurement by traditional methods. Although there is reasonable evidence that formaldehyde is produced from wood during hot-pressing of composite panels, the generally accepted idea is that formaldehyde from wood is an insignificant contributor to the total measurable level of formaldehyde in a composite wood product, and any level of detectable formaldehyde must be coming from the adhesive.

In the work presented herein, results obtained using the desiccator test on particleboard and hardboard samples prepared with no-added-formaldehyde (NAF) adhesives in addition to a water-only control clearly show that formaldehyde from wood is produced at significant levels and retained within the board when exposed to typical hot-pressing conditions. This phenomenon is much less apparent in plywood, likely because of lower hot-press temperatures. To rule out any interference from other compounds that may yield a false positive in the chromotropic acid analysis, we ran a series of controls with a variety of compounds known to be present in wood or similar in structure to formaldehyde. We further determined that the wood-based formaldehyde, referred to from here on as "native formaldehyde," is present only for a short time, after which the wood returns to background levels, as have been measured in the studies mentioned previously.

The time it takes for native formaldehyde to be completely dissipated from the sample can be as long as 30 days or more after production. Given the loose guidelines for the time in between production of a commercial panel and the time that board conditioning for the large-chamber test begins, it is possible to get a high value for formaldehyde from a sample due to native formaldehyde, even in a board produced using an NAF adhesive. An NAF adhesive can be formulated with added scavenger to prevent emission of native formaldehyde and to provide a background level of formaldehyde emission right out of the press. Given the stringent regulations on formaldehyde emissions, timing of sample testing for quality control programs will become increasingly important for maintaining appropriate certification. More stringent timing controls will allow for more reliable correlations to be established between the plant's quality control methods and the large-chamber standard required for certification.

Experimental

The PAE- (polyamidoamine-epichlorohydrin-) containing NAF adhesive used in this study was a commercially available resin, ChemVisions™ CA1000, obtained from Ashland, Inc. (formerly Hercules Inc., Wilmington, DE). Wood furnish used to produce particleboards and

hardboards was obtained from a variety of industrial collaborators around North America as follows: Furnish A–northwest U.S.; Furnish B–southeast U.S.; Furnish C–western U.S.; Furnish D–northeast Canada; Furnish E–southeast U.S.; Furnish F–northern Canada. The UF adhesive used for control work was provided by an industrial collaborator at 65% solids and had a face F:U of 1.05:1.00 and a core F:U 1.15:1.00. Additionally in the core, 1.0% v/v NH_4Cl (Sigma-Aldrich, Milwaukee, WI) was used in the core material as an accelerator. The scavenger used with UF was a 40% urea solution at 20% in the face and 15% in the core. The Soyad® NAF adhesive used is a proprietary blend of soy flour and other non-reactive ingredients with a solids content of 45 to 60% and pH of 5.5, combined with ChemVisions CA1000 at various levels. Materials for chromotropic acid analysis and control testing of the method were obtained from Sigma Aldrich, Inc.

Hardboard

For preparation of hardboard-type samples, particleboard face furnish (Furnish B–southeast U.S.) was dried to 3.0 ± 0.5% moisture content (MC). For the board with no adhesive, the pH of the water was adjusted to match that of the adhesive (pH 2.5) and applied such that the final MC of the furnish was 10.7% dry basis. Adhesive was applied to the dried furnish at a resin/wood load of 2.0% (s/s) using an atomizing spray nozzle and a rotating drum blender such that the MC of the final furnish was 10.7% dry basis. The material was then formed into two boards 0.56 × 0.56 m and pressed for either 180 or 300 s at 170°C using a 0.91 × 0.91 m oil-heated press. The panels were removed from the press, allowed to cool for 30 to 60 min and wrapped in plastic. Boards were stored at 30% RH and 27°C and kept in plastic until desiccator samples could be cut. The samples remained in plastic until the first desiccator test and were then conditioned at 50% RH and 23°C for further desiccator testing.

Particleboard-UF and Soyad/ChemVisions CA1000-NAF

Face and core material (Furnish F–northern Canada) were dried to 3.0 ± 0.5% MC. Urea-formaldehyde (face F:U 1.05:1.00 and core F:U 1.15:1.00) and Soyad/ChemVisions CA1000-NAF adhesive was applied using an atomizing spray nozzle at a face load of 10.8% (s/s) and a core load of 7.25% (s/s). The material was formed into a 0.86 × 0.86 m face-core-face board with a face:core ratio of 40:60. Boards were pressed for 300 s at 170°C using a 1.27 × 1.27 m steam-heated press. The boards were allowed to cool for 30 to 60 min and wrapped in plastic. Boards were stored at 30% RH and 27°C and kept in plastic until desiccator samples could be cut. The samples remained in plastic until the first desiccator test and were then conditioned at 50% RH and 23°C for further desiccator testing.

Particleboard-PAE-NAF

Face and core material (Furnishes A–F) were dried to 3.0 ± 0.5% MC. PAE-NAF adhesive was applied using an atomizing spray nozzle at a face load of 1.75% (s/s) and a core load of 2.5% (s/s). The material was formed into a 0.86 × 0.86 m face-core-face board with a face:core ratio of 40:60. Boards were pressed for 300 s at 170°C using a 1.27 × 1.27 m steam-heated press. The boards were allowed to cool for 30 to 60 min and wrapped in plastic. Boards were stored at 30% RH and 27°C and kept in plastic until desiccator samples could be cut. The samples remained in plastic until the first desiccator test and were then conditioned at 50% RH and 23°C for further desiccator testing.

Formaldehyde Analysis

Desiccator testing was conducted as described in ASTM D 5582-00 (ASTM 2006) using eight samples, measuring 6.99 × 12.7 cm, with the exception that the edges of the samples were not sealed with paraffin wax. Chromotropic acid analysis was conducted as detailed in ASTM D 5582-00 (ASTM 2006). For control samples, stock solutions of formaldehyde and each possible interference compound were prepared and diluted as necessary to obtain absorbance readings within instrumental capabilities.

Results and Discussion

Chromotropic Method Evaluation Results

Early in the commercialization process, experiments using Soyad/ChemVisions CA1000-NAF adhesives had produced curiously high formaldehyde emissions as measured both by the desiccator and dynamic microchamber (DMC) methods when samples were evaluated within days of coming out of the press (results not shown). Initial explanations for this unexpected result could not exclude the possibility of a false positive in the chromotropic acid analysis. A variety of compounds are known to be emitted from hot-pressed wood (Wang and Gardner 1999), so to confirm the measurement of formaldehyde, a series of control compounds with similar structures and/or functionality to formaldehyde were tested using the chromotropic acid analysis. **Figure 1** shows the structures of the nine compounds chosen as well as formaldehyde for reference. A solution of known

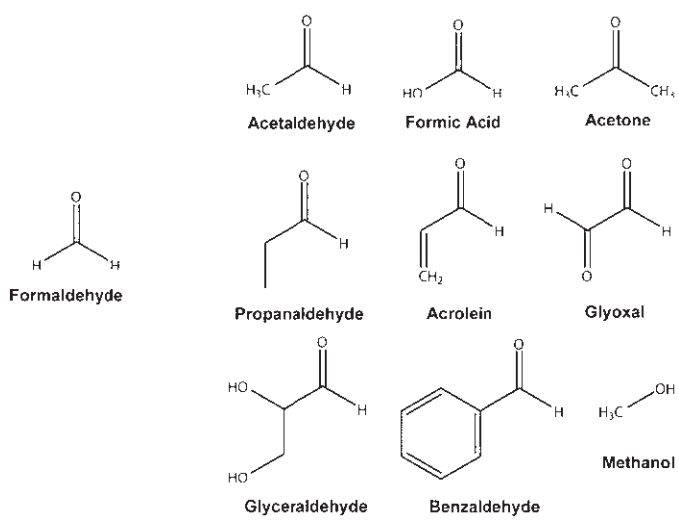

Figure 1. ~ *Structures of formaldehyde and potential interference compounds used to test the specificity of the chromotropic acid test for formaldehyde.*

concentration was prepared for each of the nine compounds and formaldehyde and then subjected to the chromotropic acid analysis.

Figures 2 and **3** show the results of the testing. In **Fig. 2**, the absorbance of all samples was normalized on the basis of concentration of the formaldehyde sample at 1.96 µg/ml. **Figure 3** shows the lower absorbance end of **Fig. 2** expanded to elucidate the shapes of the curves for the other nine compounds. Taken together, it is clear that even if our original results were caused by interference from another compound, the offending compound would have had to be produced at levels that were at least

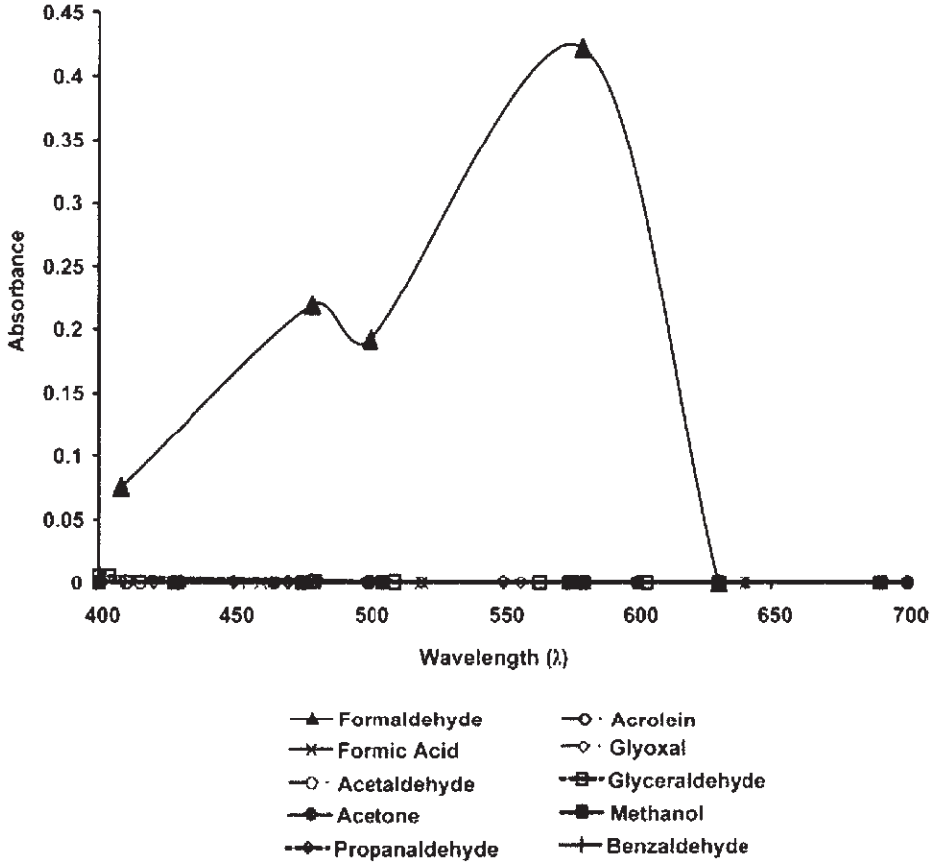

Figure 2. ~ *Spectra of solutions of formaldehyde and potential interference compounds at equivalent concentrations of 1.96 µg/ml after treatment by the chromotropic acid method.*

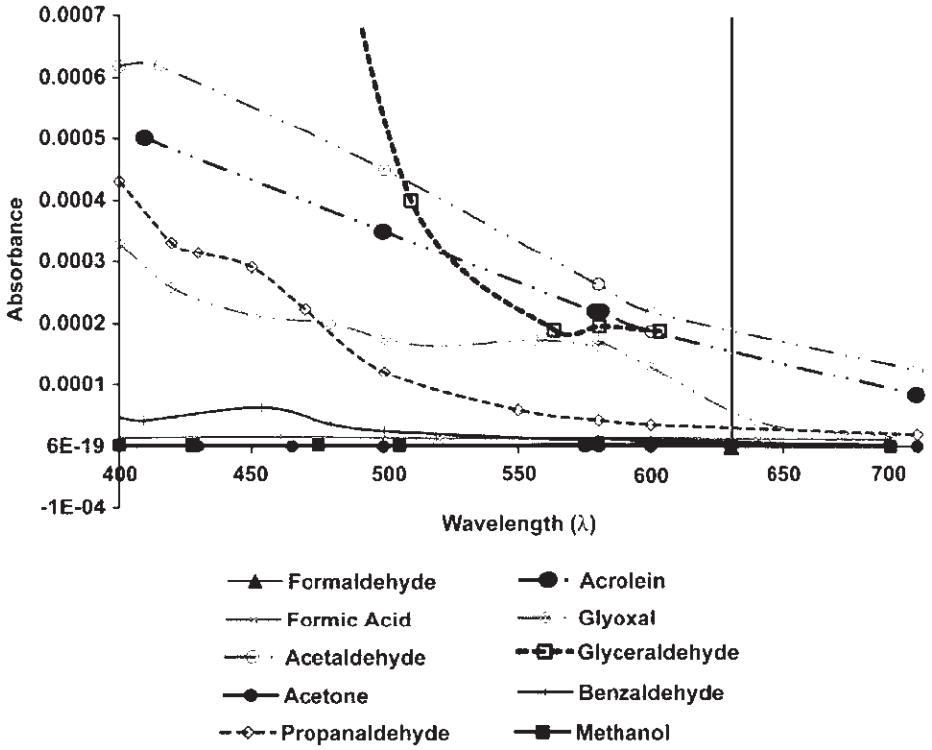

Figure 3. ~ *Figure 2 expanded to show details of the spectra for potential interference compounds.*

1000 times greater than typical formaldehyde-detection levels. Furthermore, it is obvious from the spectra that formaldehyde yields a specific spectral fingerprint that makes the chromotropic acid analysis highly specific for formaldehyde. These results confirm and expand earlier reports of the chromotropic analysis as being a very specific method for formaldehyde (MacFayden 1945).

Board Test Results

Once the chromotropic acid analysis was confirmed to be formaldehyde-specific, a set of control hardboard panels was prepared to determine the source of the formaldehyde. Although PAE is not manufactured using any formaldehyde, we wanted to ensure that it was not degrading or catalyzing any wood-based reactions to produce formaldehyde within the press. The first control board contained 2.0% PAE (s/s) dry basis to wood and had a final MC of 10.7%. Two boards were prepared from this batch, one pressed for 180 s and the other pressed for 300 s at 170°C. A second set of panels was prepared, this time spraying only water that had been pH-adjusted to 2.5 to match that of the PAE, such that the final MC of the furnish was 10.7%. Again, two panels were pressed for 180 and 300 s at 170°C.

The panel pressed with no adhesive, although somewhat weak, had enough integrity that little difficulty was encountered in preparing desiccator test samples from it. **Figure 4** shows the initial formaldehyde concentrations detected from the two panels tested with no conditioning period, having been wrapped in plastic from the time they were pressed until they were first measured, less a small time for cutting to size. In both panels, little to no detectable formaldehyde is coming from the samples pressed at 180 s; however, the formaldehyde level of the panels pressed for 300 s increased substantially. Surprisingly, the water-only panel actually gave higher levels of formaldehyde than did the PAE-containing board. This may have been due to a slight formaldehyde scavenging ability of the free amines on PAE, or it could have been related to the slightly different densities of the two boards, which has been shown to affect the formaldehyde diffusion rate (Christensen et al. 1987). In either case, **Fig. 4** is clear evidence that formaldehyde is produced or released from wood while wood is being pressed under hot and wet conditions and that formaldehyde is retained by the board and emitted at levels that are significant for a period of time after the panel is removed from the press.

After the initial formaldehyde test, the PAE and water-only samples were tested again 10 days later after conditioning (not in plastic) at 23 ± 1.7°C and 50% RH and then again 17 days later (a total of 30 days after pressing). The data in **Fig. 5** show that after 10 days of conditioning, the native formaldehyde produced during the hot-press had completely dissipated. What was not clear, however, was how the amount of native formaldehyde compares quantitatively in particleboards prepared using UF and NAF adhesives.

To answer this question, a series of four particleboards were prepared: commercial UF without scavenger, commercial UF with scavenger, Soyad/ChemVisions CA1000-NAF non-scavenging, and Soyad/ChemVisions CA1000-NAF scavenging. **Figure 6** shows the formaldehyde emissions data. The data highlight a few key observations. First, it is clear that native formaldehyde is produced at significant levels, actually showing higher initial formaldehyde levels for the non-scavenged NAF than in the scavenged UF panel. Second, although the NAF non-scavenged board initially yields higher formaldehyde values than that of the scavenged UF, the values drop quite rapidly from the board and the detectable level of formaldehyde goes to near zero because this is all native formaldehyde. In the case of the scavenged UF, there is a small initial drop in formaldehyde level followed by a considerable and sustained level of detectable formaldehyde in the board.

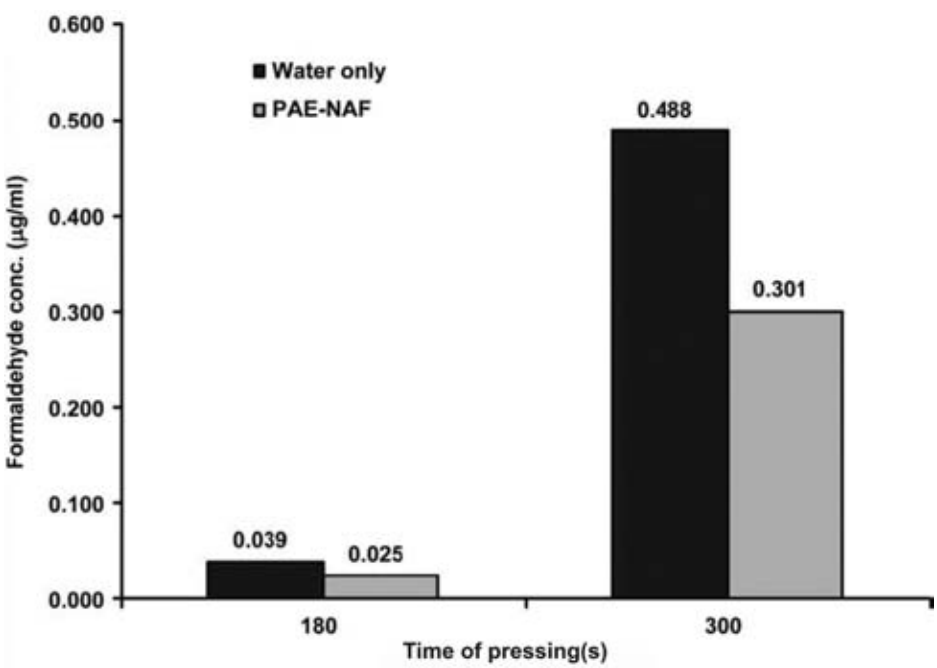

Figure 4. ~ *Formaldehyde levels of hardboard samples pressed at 180 and 300 s as measured by the desiccator method described in ASTM D 5582-00 (ASTM 2006). NAF is no-added-formaldehyde and PAE is polyamidoamine-epichlorohydrin.*

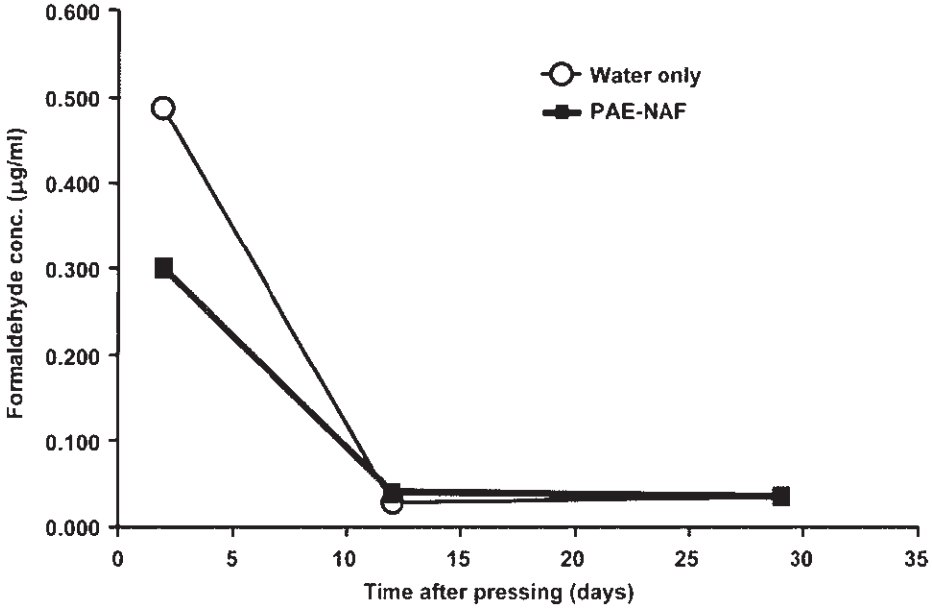

Figure 5. ~ *Formaldehyde levels for the hardboard samples measured over time using the desiccator method, ASTM D5583-00 (ASTM 2006). Samples were wrapped in plastic after pressing until the first test (day 2) but then were removed from the plastic and conditioned at 23 ± 1.7°C and 50% RH in between subsequent tests. NAF is no-added-formaldehyde and PAE is polyamidoamine-epichlorohydrin.*

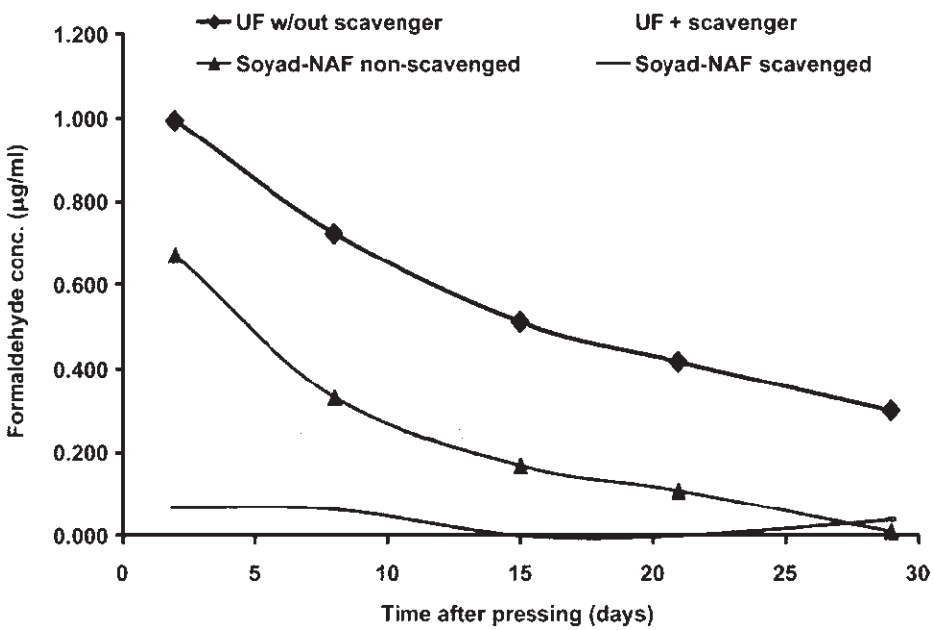

Figure 6. ~ *Formaldehyde levels of particleboards produced with either UF (urea formaldehyde) or NAF (no-added formaldehyde) adhesives and measured using the desiccator method, ASTM D 5582-00 (ASTM 2006). Samples were wrapped in plastic after pressing until the first test (day 2) but then were removed from the plastic and conditioned at 73 ± 3°F and 50% RH in between subsequent tests.*

These data suggest that the scavenger in the UF is likely scavenging most of the native formaldehyde and the longer-term formaldehyde that is detected is that produced by the UF adhesive as it releases formaldehyde over time. This idea is supported by the data for non-scavenged UF trending toward a similar equilibrium value as that of scavenged UF. Obviously for any NAF adhesive, the data indicate that once all the native formaldehyde has dissipated and background levels are achieved, the board will remain at background levels since the adhesive does not provide a long-term formaldehyde source as does the UF. Finally, the graph shows that an NAF adhesive can be formulated to contain a scavenger for the native formaldehyde, thus effectively producing a true zero-emission composite wood product.

To eliminate the possibility that high native formaldehyde was due to an unusual wood furnish source used in the above studies, particleboards were prepared using PAE-NAF and furnish from six mills located around North America (see materials and methods for details). Samples were tested for formaldehyde levels after hot-pressing boards at 170°C for 300 s. The results in **Fig. 7** show that although the formaldehyde level was somewhat furnish-dependent, all the samples produced significant formaldehyde emissions under these conditions.

Conclusions

Results from this study have shown that boards produced with PAE-NAF, Soyad-NAF, and even water provide strong evidence that native (wood-derived) formaldehyde is released, and possibly produced, from wood under conditions of hot-pressing. Furthermore, this native formaldehyde is actually retained by the board and detectable at levels similar to scavenged UF for a period of time within the standard time window of testing under commercial certification. The chromotropic

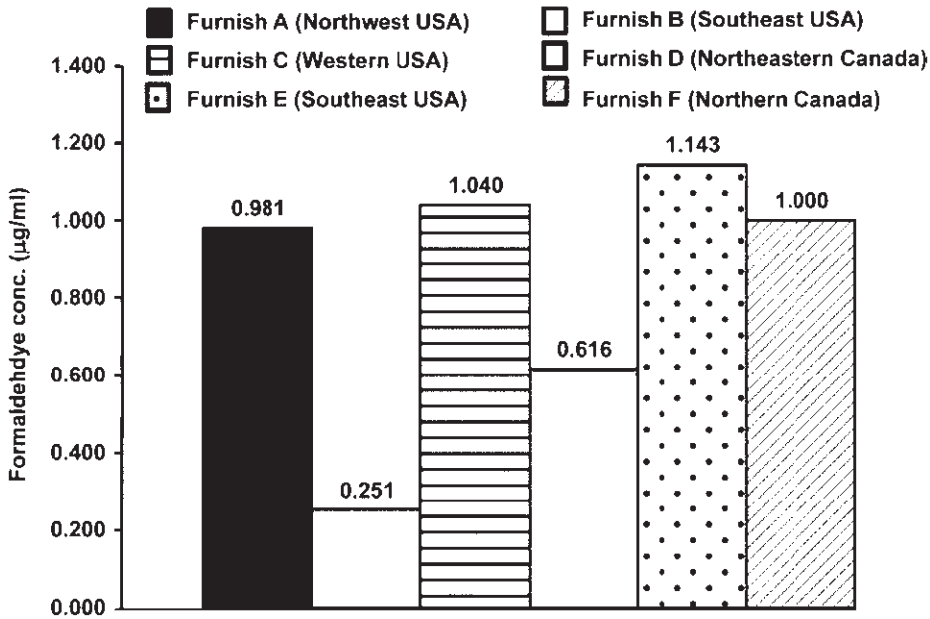

Figure 7. ~ *Formaldehyde levels of particleboards prepared using PAE–NAF and a variety of furnishes obtained from mills located in various regions of North America. Formaldehyde was measured using the desiccator method, ASTM D 5583-00 (ASTM 2006), on samples that had been wrapped in plastic after being removed from the press. Samples were all tested two days after pressing.*

method used for the quantification of this formaldehyde was confirmed to provide great confidence that the substance being measured is in fact formaldehyde and not some other interfering compound. The native formaldehyde produced during hot-pressing has been shown to be transient, dissipating on a relatively short time-scale, and likely dependant on the individual board and/or conditioning factors. Production of native formaldehyde is directly related to the length of time a board is pressed, suggesting that press temperature may also play an important role.

There are still many unknowns about native formaldehyde. For example, it is unknown whether it is actually produced from degradation products of wood constituents or simply released from an unknown loosely bound wood-formaldehyde state requiring heat and time. Additional unknowns include the effects of moisture content, pH, wood species, and other processing variables that may increase or decrease the level of native formaldehyde production in a hot-pressed composite panel.

Implications

Although presence of native formaldehyde may raise some questions about current emission standards set by CARB, attention belongs on the time period and sample treatment before testing as well as on the limits themselves. There is little doubt that UF adhesives can continue to release formaldehyde over time. To achieve reliable quality-control testing results, however, the time between press and testing will need to be more standardized. The alternative, of course, is an NAF adhesive that is formulated to achieve background levels of formaldehyde emissions right out of the press. This alternative, however, may limit the field of potential NAF adhesives.

The discovery that native formaldehyde in hot-pressed composite wood panels has the potential to yield significant formaldehyde readings, even for panels produced using NAF adhesives, certainly requires review and discussion about the best way to preserve the spirit of the CARB ruling. Improving indoor-air quality and lowering formaldehyde emissions is important given our current knowledge of the risks of formaldehyde exposure. Careful testing using well-defined and appropriate procedures is crucial to the ability of producers to consistently achieve the low-formaldehyde targets with minimal disruption in day-to-day manufacturing or inventory control.

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