

# Phenol-formaldehyde resin curing and bonding under dynamic conditions

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423

**Summary** To better understand the curing and bonding behavior of phenol-formaldehyde (PF) resin under dynamic conditions, flakeboards were manufactured either by conventional pressing at 7% or 12% mat moisture content or by steam injection pressing with 10 or 20 seconds steaming duration. Resin-impregnated glass-cloth samples and lap-shear tension specimens were embedded in the core of each flakeboard. After the flakeboards were pressed for various periods of times, the samples and specimens were quickly retrieved. The degree of resin cure was determined on the resin-impregnated glass-cloth samples by dynamic mechanical analysis. The bond strengths were measured from the lap-shear tension specimens on a mechanical testing machine. The results of resin curing and bonding were then correlated to the performance of the resin-bonded boards, which were evaluated by internal bond, modulus of rupture, modulus of elasticity, and thickness swelling. Resin curing and lap-shear bonding did not proceed simultaneously. In conventional pressing, the mat moisture content (MC) at 12% favored resin curing, but slightly retarded lap-shear bonding, as compared to 7%MC. In steam injection pressing, the rates of resin curing and lap-shear bond strength development were much faster for 20 seconds than for 10 seconds of steaming duration. Longer press times were needed to obtain boards with maximum strength in the 12%MC conventional pressing and the 20-s steam duration steam injection pressing than in those conditions where moisture content was lower or

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steam time was less. The need for longer press times must be attributed to higher internal vapor pressures and/or lower wood and resin component strengths, rather than to incomplete cure or bonding.

## Introduction

The response of resin curing and bonding mechanisms to process variables such as time, temperature, moisture content (MC) and/or relative humidity (RH) is important in optimizing the wood composite manufacturing process. Little work has been done to directly measure the extent of curing and bonding achieved during pressing and to relate this to board performance. The purpose of this study was to develop a method, based on the previous studies by Follensbee (1990) and Geimer and Christiansen (1994), to progressively monitor and characterize the resin curing and bonding process under dynamic conditions.

## Background

A number of analytical techniques have been used to successfully monitor and characterize PF resins' curing behavior by responding to either mechanical, physical, or chemical changes occurring during the curing process. These techniques include dynamic mechanical analysis (DMA) or dynamic mechanical thermal analysis, torsional braid analysis, ultraviolet spectroscopy, differential thermal analysis, differential scanning calorimetry (DSC), nuclear magnetic resonance, and Fourier transform infrared spectroscopy. Previous work (Follensbee 1990; Geimer and Christiansen 1994) showed that the degree of cure of a dried resin, as measured by DMA or DSC, increased with an increase of environmental relative humidity. A similar result was also observed by Wang et al. (1994) using DSC. On the other hand, ultraviolet spectroscopy measurements indicate that excess moisture present in liquid resins will reduce their curing rate (Chow and Hancock 1969; Chow and Mukai 1972). This finding was also confirmed, under certain conditions, for partially precured resin by Wang et al. (1994) using DSC measurements. The effect which moisture has on resin curing diminishes with increasing temperature (Chow and Hancock 1969; Chow and Mukai 1972; Wang et al. 1994).

Follensbee (1990) has distinguished between two different modes of resin cure: mechanical cure measured with a DMA, and chemical cure measured with a DSC. The mechanical cure, dependent on resin stiffness, generally develops at a faster rate than the chemical cure. Likewise resin cure and bond strength may develop at different rates. Two studies have shown that the reactivity of phenolic resin appears to change in the presence of wood (Chow 1969; Mizumachi and Morita 1975). Techniques developed by Humphrey and Ren (1989) enable one to measure the bond strength development between two disks of wood veneers after a selected time while the resin is curing. This "active" measurement of bond strength while the resin is still hot was extended to determine the rate of bond formation, under isothermal conditions, between two flakes in a lap-shear configuration (Humphrey and Zavala 1989). Geimer et al. (1990) have also measured the extent of bond strength developed in flake lap shear specimens exposed to various periods of controlled curing conditions. Their studies differ from Humphrey and coworkers in that the lap shear specimens were removed from the curing conditions and equilibrated prior to the bond strength being tested. Studies indicate that an optimum equilibrium moisture content (EMC) (Humphrey and Ren 1989) or RH (Geimer and Christiansen 1994) exists for promoting resin bonding. Wang et al. (1995) also showed that, similarly to resin cure, bond strength development was affected less by RH as bonding time and temperature increased.

Resin curing and bonding behavior characterized by currently used techniques or methods under isothermal and isohydro conditions may not quantitatively represent the actual resin performance in the boards under dynamic or varying conditions. During flakeboard manufacture, internal mat temperature, MC, and vapor pressure are related to press processes (Kamke and Casey 1988; Wolcott et al. 1990; Kamke and Wolcott 1991) and vary with press time.

In this study, resin samples and lap-shear specimens were simultaneously embedded in the core of flakeboards manufactured by using either a conventional hot pressing method or a steam injection pressing method. The boards were pressed for successively increasing periods of times. After board pressing, resin samples were retrieved from within the board and tested to determine the degree of cure using dynamic mechanical analysis. Similarly retrieved lap-shear specimens were used to determine bond strengths. The resin curing and bonding results were then related to previously conducted curing and bonding tests using isothermal and isohydro conditions and correlated to the board mechanical properties.

## Materials and methods

### Preparation of DMA resin samples

The synthesis procedure and initial properties of the phenol-formaldehyde (PF) resin used as an adhesive in this study have been previously described (Wang et al. 1994). To prevent chemical changes during storage, the resin was frozen at  $-16^{\circ}\text{C}$  in plastic bottles immediately after preparation. To prepare the samples to be inserted in the core of a flakeboard, the PF resin was thawed at room temperature and impregnated into a pure borosilicate glass microfiber filter (Whatman GF/C) (Follensbee et al. 1993). A 15-cm disk of the glass filter was immersed for a half hour in a container of the resin. Following removal, the excess resin was uniformly squeezed out of the sample by using a soft rubber roller under hand pressure. The resin-loaded disk was dried for a half hour at ambient conditions and put into a desiccator over phosphorous pentoxide ( $\text{P}_2\text{O}_5$ ) overnight. The  $\text{P}_2\text{O}_5$ -dried disks, with an assumed 0% initial moisture content, were then weighed to determine the resin loading. All disks were cut into strips (12 mm wide), sealed in small plastic bags, and frozen at  $-16^{\circ}\text{C}$  until used. Before being submitted to cure, the strips were thawed, cut to 70-mm lengths and conditioned at 50% RH ( $22^{\circ}\text{C}$ ) overnight. Prior to being inserted into a mat, the strips were weighed. Selective trials indicated that the MC after conditioning was approximately 3%MC ( $\text{P}_2\text{O}_5$ -dried basis). The strips were flexible and could withstand handling without being broken or damaged.

### Preparation of lap-shear specimens

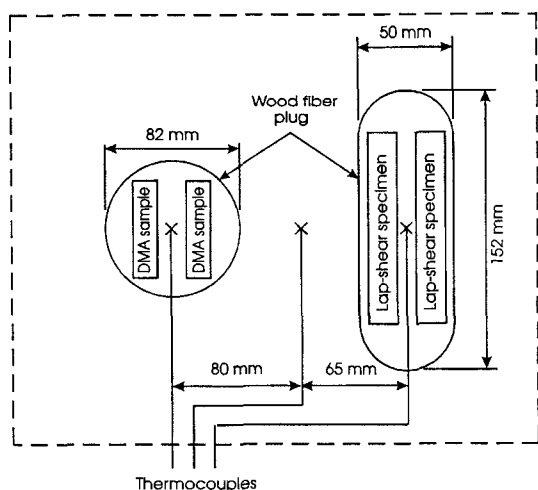
Specially cut aspen (*Populus spp.*) flakes, 76 mm long by 15 mm wide by 0.89 mm thick, were used to fabricate lap-shear joined specimens. The procedure of flake preparation has been reported in a separate paper (Wang et al. 1995). All the flakes were conditioned at 50%RH ( $22^{\circ}\text{C}$ ) for at least 24 hours before being used. Each pair of flakes to be bonded in lap-shear were coated with the liquid PF resin on their smooth sides. The resin (17–21 mg) was uniformly brushed on a  $15 \times 15$  mm area at one end of each flake. After an open assembly time of 20 min, the two flakes were lapped and the assembly was ready to be placed in the flakeboard mat for pressing.

### Procedure for embedding and retrieving DMA samples and lap-shear specimens

In the flakeboard pressing process several phenomena may occur which can affect the quality of DMA samples and lap-shear specimens and hinder their extraction from the

core of the board. First, the samples and specimens may stick to the surrounding flakes, making it impossible to quickly and properly retrieve them from the core of the boards immediately after pressing is stopped. Second, resin may be squeezed out of the DMA glass matrix under high pressures. Finally, the lap-shear specimens will be deformed and damaged by the uneven local pressures, resulting from random distribution of surrounding flakes. Techniques developed at the U.S. Forest Products Laboratory have solved these problems by isolating the DMA samples and lap-shear specimens in a screen of Teflon<sup>®</sup> and by embedding the covered samples and specimens in wood fiber plugs whose density has been adjusted. The Teflon<sup>®</sup> material neither bonds to the samples or specimens nor interferes with the transfer of heat and mass during pressing (Follensbee 1990; Geimer and Christiansen 1994). The procedure used in this study for embedding the DMA samples and lap-shear specimens in the core of boards before pressing and retrieving them from boards after pressing is described as follows.

Two cardboard molds (20 cm high) were placed near the center of the bottom caul on a single layer of flakes as shown in Figs. 1 and 2. Aluminum cauls, 6.5-mm thick, were used for conventional pressing; screen cauls were required for steam injection pressing. A circular mold was used to form the plug for the DMA samples and an ellipse-shaped mold was used for the lap-shear specimens. A cover was placed over each mold while half of the required flakes to make a board were formed around them. The covers were then removed and half of the required wood fibers were put into the molds and compressed with plungers. With the plungers holding the fibers in place, the molds were removed. Next, the two DMA samples and two lap-shear specimens held between Teflon<sup>®</sup> screens were placed on the fiber plugs. Copper-constantan thermocouples were placed adjacent to both the DMA samples and the lap-shear specimens, and in the mat core flakes as shown in Fig. 1. The two molds were then placed above their original positions (on the top of the half-formed plugs) and the remaining half of the required flakes and wood fibers were added in the same manner mentioned before. Finally, a single layer of colored flakes was placed on top of each plug to indicate the location of the DMA samples or lap-shear specimens. The colored flakes not only facilitated easy recovery but in conjunction with the bottom flake layer served to equalize the rate of heat and mass transfer into the plugs and surrounding flakes during pressing. This was especially pertinent in steam injection pressing.



**Fig. 1.** Configuration of wood plugs, DMA samples (70 by 12 mm) and lap-shear specimens (137.4 by 15 mm) in flakeboards

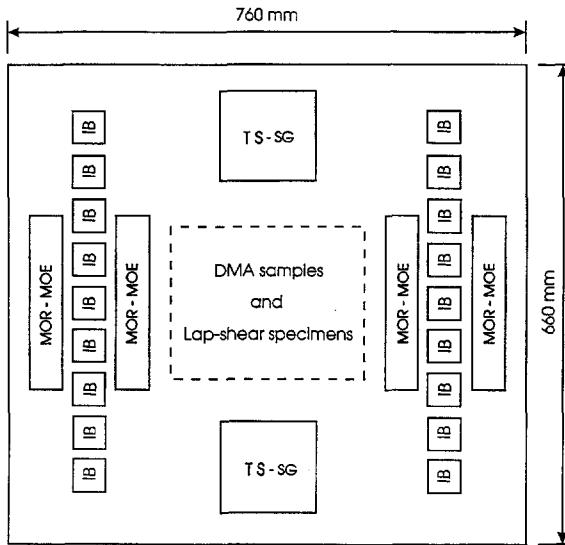


Fig. 2. Sampling diagram for both conventional and steam injection pressed flakeboards. MOR = modulus of rupture; MOE = modulus of elasticity, 330 by 76 mm; IB = internal bond, 50 by 50 mm; TS = thickness swell; SG = specific gravity, 152 by 152 mm

Following pressing, wood blocks with configurations similar to those of the wood fiber plugs, were used to push the plugs out the board. The DMA samples, retrieved from within the plugs, were restrained between glass plates for several hours to keep them flat. The samples were then frozen until needed. These precured samples were dried again over the  $P_2O_5$  desiccant prior to conditioning for DMA. The weights ( $P_2O_5$ -dried basis) after retrieval from the boards were used to calculate the resin loading. The difference in weights before and after pressing were used to determine resin loss.

### Flakeboard manufacture and evaluation

All flakeboards were constructed using 57-mm-long by random width by 0.51-mm-thick aspen (*Populus spp.*) disk-cut flakes. After being cut, the flakes were dried to about 2%MC and then sealed in plastic bags. Before being used, the flakes were adjusted to final moisture content by adding the appropriate amount of water and equilibrating them overnight in the sealed plastic bags.

Two series of 760 by 660 by 13 mm flakeboards were made to a target specific gravity (SG) of 0.64, oven-dry (OD) basis (see Table 1). One series was pressed conventionally and one series was pressed using steam injection. Each series had two subsets of boards: one made with resin and one made without resin (the latter to conserve limited resin). The 45.8 percent solids content resin was the same resin as that used to prepare the flake bonding specimens. Resin was applied at a 5% level (solid PF resin based on OD wood) in a rotary drum blender using an air atomizer sprayer. Western Hemlock (*Tsuga heterophylla*) fibers, without resin, were used to construct the plugs. The SG of the fiber plug holding the lap-shear specimens was the same as that of the flakeboard. The SG of the plug holding the DMA samples was reduced to 0.48 (OD basis) to prevent the resin from being squeezed out of the glass cloth.

Each subset of the conventionally pressed boards had a further subset differing in moisture content. Moisture content of the mats and associated wood fiber plugs was either 7% or 12%. (For those boards made with resin, mat MC included the water in the resin). All the mats and plugs subjected to steam injection pressing had a moisture

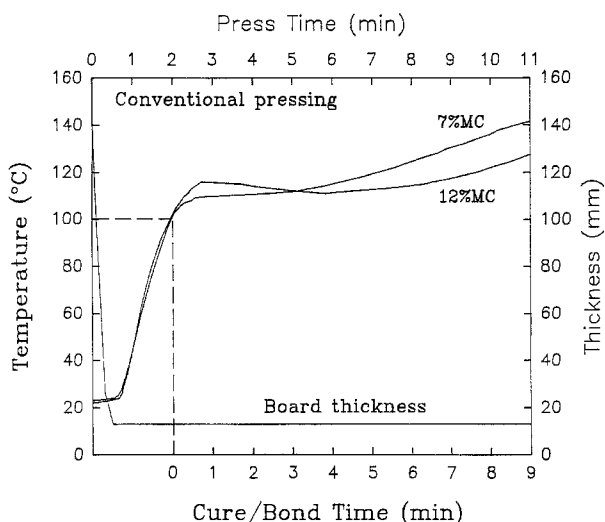
content of 7%. Each subset of the steam injection boards also had further subsets, differing in steam times of 10 or 20 seconds.

Cure/bond time differs from press time and is defined in this study as beginning when the temperature adjacent to the DMA samples reached 100 °C, as shown in Figs. 3 and 4. It took about 30 seconds to retrieve the DMA samples and lap-shear specimens after pressing, and this time was not included in the cure/bond time. For the conventional pressing (Fig. 3), the press closing rate was controlled at 6 mm/s as the mat was compressed from 140 mm to 26 mm, then at 1 mm/s from 26 mm to the board target thickness of 13 mm, which corresponds to 0.64 SG. For the steam injection pressing (Fig. 4), the press closure rate was 7 mm/s during mat compression from 140 mm to 40 mm, and then reduced to 1.5 mm/s until a mat thickness of 25 mm was reached. The steam, at a flow rate of 112 kg/h at 410 kPa maximum pressure, was then

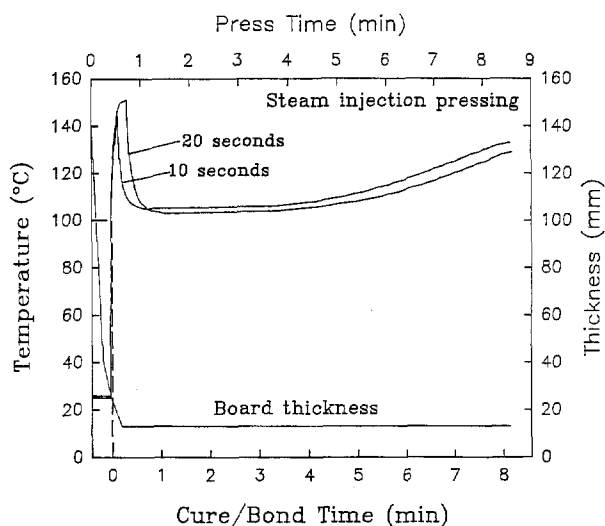
**Table 1.** Pressing conditions of boards

Programs	Resin content (%)	MC of mat (%)	Steaming time (s)	Cure/bond time (min)
Conventional pressing	0.0	7.0	–	2, 4, 6, 9
Conventional pressing	0.0	12.0	–	2, 4, 6, 9
Conventional pressing	5.0	7.0	–	2, 4, 6, 9
Conventional pressing	5.0	12.0	–	2, 4 <sup>a</sup> , 6, 9
Steam injection pressing	0.0	7.0	10	2, 3, 5, 5, 7, 8
Steam injection pressing	0.0	7.0	20	1 <sup>a</sup> , 2 <sup>a</sup> , 3, 5
Steam injection pressing	5.0	7.0	10	2, 4, 6, 8
Steam injection pressing	5.0	7.0	20	1, 3, 5, 8

<sup>a</sup> A replicate board was also made at this cure/bond time



**Fig. 3.** Typical pressing curves for conventionally pressed boards with two levels of initial mat moisture content (7%, 12% MC). The boards were manufactured with 5% PF resin solids (OD wood basis). The temperatures were detected by thermocouples at the location of DMA samples



**Fig. 4.** Typical pressing curves for steam injection pressed boards with two levels of steam injecting time period (10, 20 seconds). Initial mat moisture content was 7% for all boards. The boards were manufactured with 5% PF resin solids (OD wood basis). The temperatures were detected by thermocouples at the location of DMA samples

introduced through the top and bottom of the perforated platens, and the closure rate was reduced to 1.0 mm/s. The press continued to close at 1.0 mm/s until the board target thickness of 13 mm was reached. The 10-s steam time terminated before the target thickness was reached, while the 20-s steam time terminated about 8 s after closing. In both conventional and steam injection pressing, platen temperature was controlled at 190 °C.

With one exception boards were exposed for one of four curing times at each of the eight pressing conditions (five curing times were used for 10 s steaming of resinless boards). Replications were only made for three of the exposures (marked with asterisks in Table 1). A total of 36 boards were made, including 17 pressed in a conventional manner and 19 pressed using steam injection. A sampling diagram for the flakeboards is illustrated in Fig. 2. Physical properties of flakeboards were tested according to ASTM D1037 specifications (1970) after conditioning at 23 °C, 65%RH. The results of MOR, MOE, and IB were subjected to an analysis of variance (ANOVA), and means significantly different at the five percent level were separated by Duncan's Multiple-Range Test (SAS 1989). Thickness swelling (TS) was measured after 24 hours of immersion in water at 20 °C.

#### Measurement of the degree of cure

Dynamic mechanical analysis follows the resin curing process by detecting changes in the storage modulus ( $E''$ ) and the loss modulus ( $E'$ ) of the resin samples. The ratio of two dynamic parameters,  $E''/E'$ , is then designated as loss tangent or tan delta. Areas under tan delta curves in an isothermal DMA experiment using resonant frequency mode have been used to determine the degree of cure for a partially cured PF resin sample (Follensbee 1990; Geimer et al. 1990). This method is based on the fact that the residual tan delta area for a partially cured resin sample measured by DMA is a function of the curing time of the sample prior to DMA analysis.

The previous studies have used resin weight to normalize the tan delta area. The tan delta area was corrected by dividing the measured tan delta area ( $\text{cm}^2$ ) by the resin loading ( $\text{mg}/\text{cm}$ ). Since the resin loading was defined as the  $\text{P}_2\text{O}_5$ -dried resin weight per length of substrate with a standard width of 1.25 cm, the corrected tan delta area had units of cubic centimeters per milligram. In this study, the tan delta area was normalized using a redefined resin loading and an adjustment for resin loss during pressing. The use of a resin loading determined after pressing accounts for squeeze-out and loss of water of condensation. Resin loading was calculated by dividing the  $\text{P}_2\text{O}_5$ -dried resin weight ( $\text{mg}$ ) by the area ( $\text{cm}^2$ ) of glass filter being tested to give a value in  $\text{mg}/\text{cm}^2$ . Tan delta area after normalizing for resin loading and resin loss is calculated as follows:

$$A = A_0 / (L \times W \times R) \quad (1)$$

where

- A normalized tan delta area ( $\text{cm}^2/\text{mg}$ )
- $A_0$  measured tan delta area ( $\text{cm}^2$ )
- L tested length of DMA sample between the two arms of clamps (cm)
- W tested width of DMA sample (cm)
- R resin loading after pressing ( $\text{P}_2\text{O}_5$ -dried basis,  $\text{mg}/\text{cm}^2$ ).

Thus, the corrected tan delta area was in square centimeters per milligram. For simplicity, the tan delta area, normalized for resin loading and resin loss, is still referred to as tan delta area.

All DMA measurements were conducted on a Du Pont 983 dynamic mechanical analyzer. The DMA samples were conditioned in a desiccator over a saturated solution of ammonium chloride (79%RH) overnight prior to DMA analysis. For each measurement, the scan program provided an equilibrating period of 2 min at  $30^\circ\text{C}$  followed by a jump to  $150^\circ\text{C}$  for 45 min. The degree of cure (percent),  $\alpha$ , of a partially cured resin sample is calculated by the following equation:

$$\alpha = 100 \times (A_T - A_R) / A_T \quad (2)$$

where

- $A_T$  total tan delta area ( $\text{cm}^2/\text{mg}$ ) for an uncured sample (not subjected to heat exposure prior to the DMA test)
- $A_R$  residual tan delta area ( $\text{cm}^2/\text{mg}$ ) for a partially cured sample (pressed in the core of flakeboard for a period of time).

Only one sample from each board was tested, except in two cases where two samples were tested from boards. For these pairs, the coefficients of variation were 0.7% and 8.3%.

### Measurement of flake bond strength development

All bonded flake specimens were conditioned at 50%RH ( $22^\circ\text{C}$ ) for at least 24 hours prior to testing. Tests were performed on an Instron mechanical testing machine with a head speed at 10 mm/min. The specimens were vertically clamped between two serrated grips. Length between the grips was 73 mm. Flake bond strength was determined as the average maximum tensile load for each of the two specimens retrieved from each pressed board. In case of board replication, bond strength was taken as the average maximum tensile load of the lap-shear specimens for both boards. Curves of bond strength development were obtained by plotting bond strength as function of cure/bond time.



## Results and discussion

### Press and temperature curves

#### Conventional pressing

Typical press curves for conventional pressed and steam injection pressed flakeboards with 5% of PF resin are shown in Figs. 3 and 4, respectively. Heat is transferred from the platens to the board surface by conduction, generating steam from mat moisture. During the first stages of pressing, heat transfer from the face of the board to the core is primarily by convection with the steam as the heat transfer medium. For conventional pressing, it took approx. 2 min press time for the core in either the 7%MC or 12%MC board to reach 100 °C. The temperature in the 7%MC board stabilized at 110 °C after 3 min and then began to climb slowly after 4 min. This plateau occurs as long as steam that escapes along the center layers to the edges of the board is replaced by steam entering the core from the face. When the core begins to dry out, as occurs at 4 min press time, temperature begins to rise again. The higher initial maximum temperature in the 12%MC board is attributed to an increase in vapor pressure (Casey 1987). Core temperature in the 12%MC board declines gradually from 115 °C to 110 °C by 6 min and then begins a gradual climb. Longer “plateau” durations are typical responses to increased mat MC. The final core temperatures at 11 min press time for the 7%MC and 12%MC boards were approx. 140 °C and 130 °C, respectively.

#### Steam injection pressing

Core temperature in the steam injection pressed boards responded rapidly to the relatively high steam pressure differential (Fig. 4). It took less than one second for the core temperature to rise from 26 °C to 100 °C. Core temperatures peaked at 145 °C in the 10-s steamed board and 150 °C in the 20-s steamed board. After the steam injection was stopped, core temperature rapidly dropped to approximately 105 °C in both 10-s and 20-s steamed boards. The temperature remained at this level for about 2.5 min until loss of moisture permitted the temperatures to rise again. After 9 min press time, the core temperature was 130 °C in the 20-s steamed board and 135 °C in the 10-s steamed board.

Temperatures measured for the lap-shear specimens were close to those for the DMA samples in all pressing conditions. However, the initial core temperature rise in the flakes at the center of the mat was sometimes delayed for conventional pressing. In addition, board-to-board temperature variation was also greater for the core flakes than for DMA samples or lap-shear specimens in both conventionally and steam injection pressed boards. We attribute this to the larger difference in the material geometry of individual flakes as compared to wood fibers. Press curves for the conventionally pressed or steam injection pressed resinless boards were similar to those for the resin-bonded boards.

In this section, times referred to press time. In subsequent sections, times will refer to cure/bond times, defined on Figs. 3 and 4.

#### Resin curing and bonding

The choice of resin loadings determined after curing within boards, used for Eq. (1), was made because of our concern for possible resin loss during pressing. Even though adjustments were made to reduce squeeze-out by reducing the specific gravity of the wood fiber matrix surrounding the DMA samples, resin solid losses did occur, as evidenced by the data in Fig. 5. Resin solids loss attributed to squeeze-out is increased primarily by increased pressure and by decreased viscosity. Viscosity can be decreased

by increasing temperature or MC. Resin loss attributed to squeeze-out stops after curing increases the viscosity sufficiently to resist the pressures imposed. Any loss of resin solids occurring beyond where squeeze-out stops must be attributed to loss of water of condensation that results from curing reactions. Resin loss in the early portion of the press cycle likely combines resin squeeze-out and loss of water of condensation. As shown later, increases of temperature, MC, and cure occur very early in steam injection pressing.

### Conventional pressed boards

Samples of resin on glass filters taken from conventionally pressed board were analyzed by DMA (Fig. 6). The areas under tan delta curves at either moisture condition, calculated by Eq. (1), decreased with cure/bond time. This indicates that the resin underwent curing (Follensbee 1990; Christiansen et al. 1993), confirmed by the development of mechanical stiffness or rigidity. The data indicate that at some

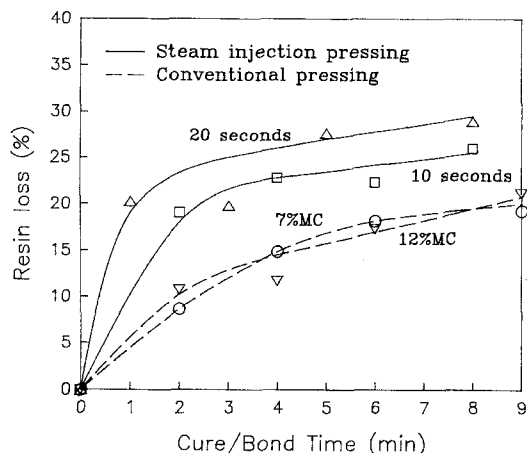


Fig. 5. Percent of resin loss during hot pressing, as determined from the difference of P<sub>2</sub>O<sub>5</sub>-dried DMA samples' weights before embedding in the wood plugs and after retrieving from the resin-bonded boards

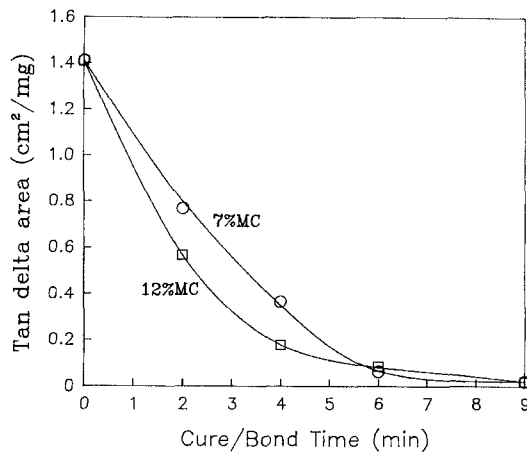


Fig. 6. Tan delta areas for the DMA samples taken from cores of conventionally pressed resin-bonded flakeboards. DMA samples were conditioned at 79%RH (NH<sub>4</sub>Cl) overnight prior to analysis

cure/bond (prior to 6 min) more potential (residual) cure remains in boards pressed at 7%MC than those pressed at 12%MC. Beyond 6 min, both tan delta curves appeared identical and slowly decreased towards a zero value. For the convenience of comparison of resin curing with bonding characteristics, the rest of the discussion in this study will use the degree of cure instead of the tan delta area.

The degree of resin cure and bond strength development as a function of cure/bond time are illustrated for conventionally pressed resin-bonded boards in Fig. 7. Degree of cure and bond strength both increased with increasing cure/bond time. Initial mat moisture content influenced the rate of resin cure and, to a far lesser extent, the rate of bonding. The data show that prior to 6 min cure/bond time resin cure progressed further in a 12%MC board than in a 7%MC board. Enhancement of cure of dried PF resins by higher moisture contents under isothermal and isohydro conditions has been found in other investigations: Wang et al. (1994), using DSC analysis, observed that resins cured faster at 90%RH than at 41%RH (at 105 °C). Geimer and Christiansen (1994), using DSC and DMA techniques, observed that resins cured faster as RH levels increased from 0% to 91% (at 115 °C). We believe that higher moisture environments favor the cure of dried PF resins, because water plasticizes the resin, thereby enhancing its reactivity. However higher moisture content does not favor resin cure in all situations. A DSC study (Wang et al. 1994) shows that when the uncured PF resin is dried to a lesser degree before being cured under humid conditions (90%RH at 105 °C), the rate of the cure decreased as the initial resin moisture content increased from 0% to 71%. This indicates that excess moisture in the resin likely dilutes the reactive components of resin, thus retarding the resin cure rate as has been shown in previous work (Chow and Mukai 1972).

On the other hand, lap-shear specimen bond strengths measured in this study were slightly higher for specimens retrieved from boards pressed at 7%MC than for those pressed at 12%MC at bonding times between 2 and 6 min (Fig. 7). That is, 7%MC favored resin bonding. There could be an optimum moisture condition for resin bonding at these conventional pressing conditions. A previous study on PF resin

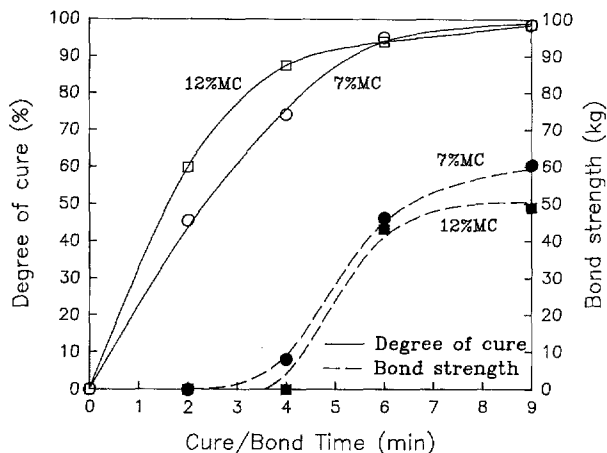


Fig. 7. Degree of resin cure and bond strength development measured on DMA samples and lap-shear specimens, respectively, from the cores of conventionally pressed flakeboards bonded with 5% resin. DMA samples were conditioned at 79%RH overnight prior to analysis. Lap-shear specimens were conditioned at 50%RH for at least 24 h prior to tensile testing

bonding under isothermal and isohydro environments by Humphrey and Ren (1989) indicated that 10% equilibrium moisture content (EMC) appeared to be better for the resin bonding at 110 °C and 115 °C, as compared with samples at either 4% EMC or 16% EMC. Geimer and Christiansen (1994) observed that PF resin bond strengths rose faster at 41%RH than at 0%RH or 91%RH at an isothermal temperature of 115 °C.

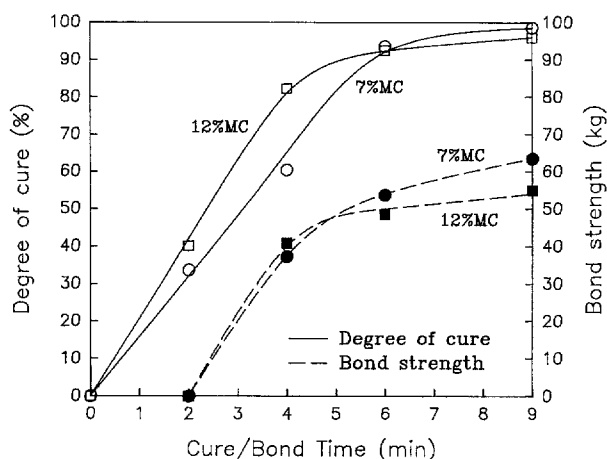
One aspect of the bond strength test is the mode of failure of the lap-shear specimens. In those tests, specimens failing at bond strengths below 33 kg failed by shear failure in the bond line, whereas specimens failing at bond strengths above 50 kg (with one exception) failed at least partly in tension by breakage of the wood substrate. Thus between 33 and 50 kg both resin cure and wood strength were important in determining bond strengths, and above 50 kg the resin and the bond were stronger than the wood. [In previous work (Wang et al. (1995)) the inherent tensile strength of a single aspen flake 76 mm long with the same cross-sectional area as these flakes was about 120 kg. The likely cause for the lower failure loads on the bonded joints was attributed to joint geometry causing peeling forces and stress concentrations at the ends of the lap-shear joints.] It is important to remember that these strengths are for specimens that were already conditioned to 50%MC and 22 °C, at which condition wood is much stronger than it would be just out of the hot press.

The fact that degree of cure and bond strengths respond differently to moisture content indicate that optimum environmental conditions for curing are not necessarily the optimum conditions for resin-wood bonding. For each phenolic formulation there is an optimum moisture content to achieve the ideal flow properties to adequately penetrate the wood substrate. Note also that at both moisture contents there were initial delays before resin bond strength developed, although mechanical cure of the resin was occurring. The lowest average non-zero bond strength (about 10 kg) occurred at 4 min cure/bond time, which corresponds to an average 75% degree of cure. This indicates that although the resin was developing integrity, that integrity had to rise to a high level before measurable lap-shear bond strength could be observed. Whether the cause of the lagging bond strength was due directly to insufficient integrity of the resin or to a need for the resin to interact in a specific way with the wood to promote sufficient bonding is not yet understood.

DMA cure samples and lap-shear bond strength specimens retrieved from mats of resinless wood showed behavior (Fig. 8) similar to those from resin-bonded boards. Once again resin cure was faster for 12%MC than for 7%MC before 6 min cure/bond time. An indication of slightly lower rates of cure in the resinless mats compared with the resin-bonded boards is not considered significant.

As previously noted for resin-bonded boards, bond strength development within the resinless mats was not significantly affected by the moisture content. However, bond strength developed more rapidly for samples situated within the resinless mats than in the resin-coated mats. For example, at 4 min bonding time, a 40 kg lap-shear bond strength was obtained for specimens retrieved from the resinless mat with 7%MC (Fig. 8), whereas only 10 kg bond strength was measured for specimens from the resin-bonded board with 7%MC (Fig. 7). Since the temperature gradients in both mats with resin and without resin were similar, the reason for this difference is not understood, but it signals that cautious interpretation should be used if experiments are tried without resin on the mat flakes.

Figures 7 and 8 indicate that 6-min cure/bond time produces near maximum bond strength. At that time about 95% cure was achieved at each moisture content in both resin-bonded and resinless boards. Beyond 6 min, both degree of resin cure and bond strength still increased, but at much reduced rate. However, increases of bond strength



**Fig. 8.** Degree of resin cure and bond strength development measured on DMA samples and lap-shear specimens, respectively, from the cores of conventionally pressed resinless flakeboards. DMA samples were conditioned at 79%RH overnight prior to analysis. Lap-shear specimens were conditioned at 50%RH for at least 24 h prior to tensile testing

beyond 6 min cure/bond time most likely reflect the fact that flake tension strength dominates bond strength for these specimens. In this same region, specimens from boards pressed for 9 min at 7%MC appear to be stronger than those from boards at 12%MC. It may be that more damage is done to the inherent wood structure under the higher moisture condition of the 12%MC bond.

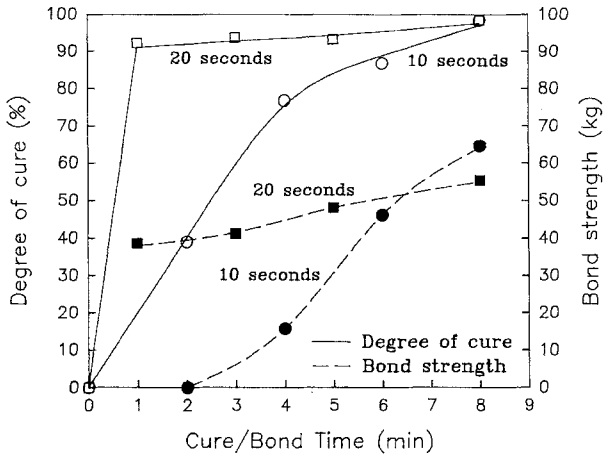
### Steam injection pressed boards

Resin cure and bonding were significantly enhanced in boards that underwent 20-s steaming duration as compared with boards made with 10-s steaming duration (Fig. 9). For 20-s steaming, the resin attained 90% cure and 40 kg of bond strength at 1 min of cure/bond time. However, to achieve similar values of resin cure and bonding for 10-s steaming takes about 6 min cure/bond time. Although moisture content can affect the rate of resin cure, a more important factor is the energy input (temperature and its duration). Interestingly, the results show that the steam injection pressed boards with 10-s of duration of steam (Fig. 9) achieved similar values for the degree of cure and for bond strength at the same cure/bond time as did the conventionally pressed resin-bonded boards which contained 7%MC (Fig. 7). The sharp spike of high temperature (Fig. 4), before reduction to a plateau level of 105 °C, by 10-s steam injection had the same effect on cure as the much more gradual rise to a plateau of 110 °C in the 7%MC conventionally pressed board.

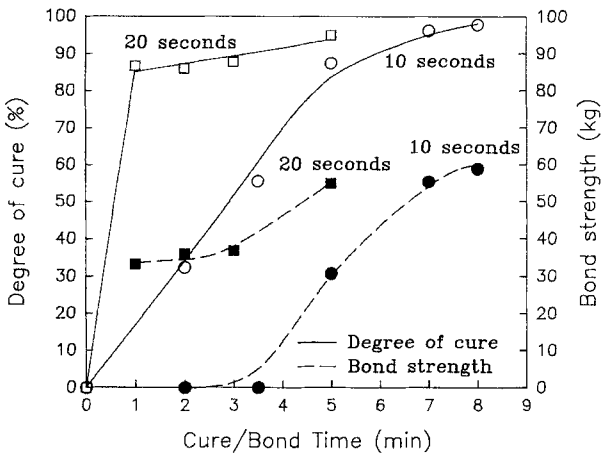
Surprisingly, 10 additional seconds of steaming increased the degree of cure from practically nothing (the 10-s steamed board) to 90% in the 20-s steamed board (Fig. 9). Likewise, bond strength increased from zero to 40 kg (near the maximum measured strength). During these 10s, the maximum temperature rose 5 °C higher (Fig. 4) than the spike attained for the shorter steam duration, and the area for the cure time/temperature being twice as large. Apparently, the energy supplied in this time, at the needed high temperature required by the high energy of activation of PF resins, was sufficient to cause the entire transformation from a liquid resin to a crosslinked solid.

The higher energy input from longer steam injection significantly enhances the resin bonding rate despite moisture added during steaming. A study on PF resin bonding by Wang et al. (1995) found that increasing the temperature from 110 °C to 140 °C diminished the retarding effect of high environmental RH on the rate of resin bonding.

There appears to be little significant difference between the cure or bond strength development of specimens retrieved from the resin-bonded boards and the resinless mats (compare Fig. 10 with Fig. 9). The data for specimens from the resinless mat



**Fig. 9.** Degree of resin cure and bond strength development measured on DMA samples and lap-shear specimens, respectively, from the cores of steam injection pressed flakeboards bonded with 5% resin. DMA samples were conditioned at 79%RH overnight prior to analysis. Lap-shear specimens were conditioned at 50%RH for at least 24 h prior to tensile testing



**Fig. 10.** Degree of resin cure and bond strength development measured on DMA samples and lap-shear specimens, respectively, from the cores of steam injection pressed resinless flakeboards. DMA samples were conditioned at 79%RH overnight prior to analysis. Lap-shear specimens were conditioned at 50%RH for at least 24 h prior to tensile testing

reinforce the previous conclusion that both resin cure and bond strength development for 10-s steaming (Fig. 10) were comparable to those for 7%MC in the conventionally pressed resin-bonded boards (Fig. 7).

### **Correlation of static controlled conditions with dynamic pressing conditions**

#### **Conventionally pressed boards**

Tests of flake specimens bonded under controlled static conditions of temperature and RH can give us insights into results from specimens bonded within, and retrieved from, flakeboards. Because phenolic resin polymerization and crosslinking activity is relatively minimal until temperatures exceed 100 °C, the bonding temperature-time curves from conventionally pressed flake specimens are closely related to those seen previously in specimens exposed to controlled conditions. Wang et al. (1995) showed that tensile-stressed bonded specimens, precured at controlled 110 °C and 90%RH conditions, are characterized by adhesive failure and never reach maximum strength during the first 10 min of exposure. However, at 41%RH, partial or total wood failure occurs and bond strengths are near maximum. In the present work, bond strengths of specimens exposed to conventional pressing for 9 min reached near maximum levels with failure occurring predominantly in the wood. The results from controlled conditions would indicate that the RH in the flakeboards were at less than 90%RH. Observations by Geimer et al. (1992) indicate that in the early or temperature plateau portion of the press cycle, saturated steam conditions are prevalent in the core of the board. Only after the steam has had a chance to escape does the temperature rise. This occurs at 4 min bond time in the 7%MC conventionally pressed resin-bonded board. The data presented in Fig. 7 shows that strength began to develop after 4 min.

#### **Steam injection pressed boards**

The 20-s steam injection pressing schedule is characterized by a burst of high temperature with the possibility of some superheating occurring (Geimer et al. 1992). As mentioned previously, Wang et al. (1995) showed that the retarding effect of high RH on bonding is lessened or eliminated at a temperature of 140 °C. The data presented in Fig. 9 shows that bond strengths of 40 kg were attained during the first minute in a 20-s steamed board when temperatures rose to 150 °C. Further strength increases occurred only slowly when temperature fell to 105 °C. Near maximum bond strengths of 65 kg were finally reached in 8 min after moisture was reduced and the temperature began to rise.

### **Correlation of resin curing and bonding to board properties**

The results of the physical property tests of both conventionally pressed and steam injection pressed resin-bonded boards are shown in Table 2. Note that no data are available at very short cure/bond times because the boards delaminated either in the center or through the whole board. Those low MC (7%) conventional boards and short time-steamed (10 s) boards developed maximum physical properties when pressed at the shortest successful cure/bond time. The high MC (12%) conventional boards and boards steamed for 20 s developed only marginal properties at their shortest successful cure/bond times. Board strengths in these latter two groups were improved by longer pressing times.

#### **Conventionally pressed boards**

Correlation of the conventional board properties (Table 2) and the degree of resin cure as measured by the DMA (Fig. 7) suggests that a conventional board pressed at 7%MC

**Table 2.** Physical properties of flakeboards<sup>a</sup>

Pressing conditions	Cure/bond time (min)	IB <sup>b</sup> (Kpa)	MOR <sup>c</sup> (Mpa)	MOE <sup>d</sup> (Mpa)	Thick <sup>e</sup> (mm)	SG <sup>f</sup>	Ts <sup>g</sup> (%)
Conventional pressing	2	Del. <sup>h</sup>	Del.	Del.	Del.	Del.	Del.
7%MC of mats	4	350 (100)A	30.1 (5.58)A	4350 (724)A	15.4	0.550	21.8
	6	320 (76)A	28.9 (2.58)A	4530 (455)A	15.4	0.561	21.6
	9	320 (83)A	27.1 (6.51)A	4400 (627)A	15.4	0.580	20.2
Conventional pressing	2	Del.	Del.	Del.	Del.	Del.	Del.
12%MC of mats	4	Del.	Del.	Del.	Del.	Del.	Del.
	6	230 (69)B	25.0 (6.48)A	4660 (862)A	14.8	0.610	25.7
	9	320 (90)A	28.5 (6.40)A	5010 (738)A	14.7	0.591	23.8
Steam injection pressing	2	Del.	Del.	Del.	Del.	Del.	Del.
10 seconds	4	340 (90)A	30.2 (4.74)A	4930 (455)A	12.9	0.701	35.5
20 seconds	6	310 (83)A	32.8 (11.0)A	5020 (1040)A	13.2	0.676	33.8
steaming	8	350 (69)A	28.8 (6.62)A	4820 (579)A	13.2	0.685	30.1
Steam injection pressing	1	Del.	Del.	Del.	Del.	Del.	Del.
20 seconds	3	180 (62)C	24.3 (6.49)A	4900 (179)A	13.6	0.671	37.3
steaming	5	320 (69)A	30.4 (6.96)A	4830 (558)A	13.3	0.679	33.7
	8	320 (76)A	29.7 (7.45)A	4760 (889)A	13.1	0.707	31.1

<sup>a</sup> All specimens were conditioned at 23°C, 65%RH before testing. Values in parentheses are standard deviations. Means within a column followed by the same capital letter are not significantly different at the 5 percent level (ANOVA, Duncan's Multiple-Range Test)

<sup>b</sup> IB, internal bond (average of 18 samples)

<sup>c</sup> MOR, modulus of rupture (average of 4 samples)

<sup>d</sup> MOE, modulus of elasticity (average of 4 samples)

<sup>e</sup> Thick, thickness of TS specimens (average of 2 samples)

<sup>f</sup> SG, oven-dry specific gravity (average of 2 samples). The average of moisture content was 9.3% after conditioning at 23°C, 65%RH before testing

<sup>g</sup> TS, 24-hour thickness swelling (average of 2 samples)

<sup>h</sup> Del., delamination (either in the center or through the whole board)

required a minimum of 75 percent resin cure (4 min bond time) to develop adequate board properties. Individual flake bond tests indicated that a very low flake bond strength, less than 10 kg, was adequate to achieve high board strengths. Conventional boards pressed at 12%MC attained a minimum of 95 percent resin cure (6 min bond time) before they developed adequate board properties. In contrast to the boards pressed at 7%MC, these 12%MC boards attained a rather high flake bond strength, greater than 45 kg, to produce boards with only marginal properties.

For today's short press cycles, resin curing and bonding mechanisms appear to be more important for resisting adverse internal environments (such as high internal vapor pressures) than they are for achieving final board properties. It would also appear that in some cases the in-press strength of the wood or cured resin components is lowered by the internal environment to levels at which they are not adequate to withstand internal pressures during press opening, even though the resin component is completely cured or bonded.

The strength properties of the flakes and/or the resin measured after pressing, Figs. 7 and 8, will generally be higher than those properties occurring in the high temperature and moistures present in the press immediately prior to opening. Four major factors,



vapor pressure, wood strength, resin strength, and bond strength are involved in the delamination of a fully cured and bonded board.

1. Vapor pressure. Casey (1987) has shown that internal mat vapor pressure, and consequently within-flake EMC, increases with an increase in mat MC. High internal pressure disrupts resin bonds when platen pressure is released.

2. Wood strength. Casey's work (1987) and that of Geimer (1985) have shown that the properties of individual flakes after removal from a hot pressed mat may be altered (negatively or positively) depending on the extent and duration of compression and the internal environment at the time of compression. Increased flake MC during pressing will decrease the glass transition of the wood and weaken its resistance to stresses (Back and Salmén 1982) in the mat when the press is opened.

3. Resin strength. High temperature (Nelson 1974) and moisture content (Irle and Bolton 1988) both lower the strength of crosslinked PF resins. One indication of the plastic state of resin during part of a full pressing operation may be seen in the paper by Humphrey and Ren (1989), which shows fingers of PF resin drawn out from a broken joint that was tensile tested while hot, part way into the pressing process.

4. Bond strength. The strength of a fully formed bond between the resin and wood may also be susceptible to the high levels of temperature and moisture content.

The data shown in Fig. 3 gives us an indication of the relative levels of vapor pressures and/or strength of the components during pressing between two boards pressed at different MCs. The temperature plateau of a 7%MC mat is short, and temperature begins to rise after 2 min cure/bond time (corresponding to 4 min press time). In the 12%MC board this increase in temperature, and the accompanying decrease in vapor pressure, is delayed until 4 min cure/bond time (corresponding to 6 min press time). In both cases the ability of the boards to survive being taken out of the press was delayed until 2 min after they reached the point where the temperature started to rise again. This rise in temperature is due to the drying of the core. Therefore, it was not until the vapor pressures decreased and wood, resin, and bond strengths increased, that good boards could be produced. We can only conclude that the two-minute-longer press time necessary to make the 12%MC board as compared to the 7%MC board is due to the more persistent internal pressures and the inability of the wood and cured resin components to resist stress, rather than to the extent of resin cure and bonding.

### Steam injection pressed boards

As mentioned above, 10 s of steam injection resulted in resin curing and flake bonding curves (Fig. 9) very similar to those obtained with the 7%MC conventional board (Fig. 7). Resin cure after 4 min cure/bond time was about 75 percent in both cases. Flake bond strength was 15 kg for the 10 s steamed board and 10 kg for the 7%MC conventional board. Because in both cases the tested board properties were near maximum (Table 2) and they had similar degrees of cure and flake bond strengths, one is led to believe that the internal pressures and the ability of the wood and resin to resist stresses were similar.

When steam was injected for 20 s, boards having marginal properties were made in about 3 min (Table 2). The increased duration of high temperatures resulted in a DMA resin cure of 95 percent and flake bond strengths of about 40 kg (Fig. 9). Values essentially this high were reached as early as the first minute of exposure, which indicates again that minimum press time to make a board was more dependent on environmental conditions affecting internal stress and the wood and resin strengths at the time of pressure release, rather than the extent of resin cure or bond formation. The

increase of IB strength between 3 min and 5 min cure/bond time is attributed to reduction of bond damage at the time of press opening.

It is important to note that in some cases high final board strengths could be obtained with relatively low flake bonding strengths. Perhaps the extent of resin cure controls the degree of final strength in those boards pressed under conditions of low vapor pressures. As shown in Fig. 7 resin cure develops at a faster rate than bond strength. This has also been shown by Follensbee (1990) and Geimer and Christiansen (1994). A question arises, unfortunately not addressed in this study, whether the development of high flake-to-flake bond strength is critical in development of long-term durability. Observations made with plywood have shown that the degree of wood failure on wet lap-shear specimens is a much better indication of durability than the level of shear strength. This has also been confirmed in a preheating fingerjoint process (Troughton and Chow 1980) using phenol resorcinol formaldehyde glue whereby % wood failure is a much better indicator of durability than bond strength.

Dimensional stability of the boards is often a reflection of the environmental conditions encountered during pressing. While high MC reduces the glass transition and, consequently, the strength of wood components, it also permits lignin flow and hemicellulose deformation, which in turn reduces internal flake stresses. The consequent reduction of out-of-press springback is evidenced by slightly higher SG and reduced thickness of those conventional boards made with 12%MC as compared to those made with 7%MC. The boards made with steam injection experienced very little, if any, out-of-press springback (Table 2). Usually steam injection pressing results in slight improvements in dimensional stability, that is, reduces thickness swell. This has been attributed to the reduction of internal stresses resulting from early plasticization of the flakes (Geimer et al. 1992). In our study the conventional boards had lower TS than the steamed boards. However, total thickness swell (out-of-press springback compounded with thickness swell caused by soaking) averaged only 35 percent in the steam injected boards as compared to 43 percent for the conventionally pressed boards.

## Conclusions

A technique has been developed to progressively follow the process of resin curing and bonding under real time conditions, by embedding the DMA samples and lap-shear specimens in the core of flakeboards.

In conventional pressing, a higher mat moisture content favored resin curing and slightly retarded resin bonding. It appears that the extent of resin cure rather than bond strength determines the final strength of a low moisture content (MC) board: a 75% resin cure level was attained in 4 min minimum cure/bond time with a 7%MC board. Bond strength at this time was very low. A minimum of 6 min cure/bond time was necessary to produce a board at 12%MC. Resin cure level was 95%, and bond strength was very high. The longer time required to press the 12%MC boards must be attributed to the increased vapor pressure and/or lower resistance of the wood and resin components to stress, rather than to incomplete cure and bonding.

In steam injection pressing, longer steam duration increased the rates of resin cure and bond strength development. It took 6 minutes of cure/bond time to achieve 85% resin cure and a high bond strength in the 10-s steam board. It took only one minute to achieve 90% resin cure and a very high bond strength with the 20-s steamed board. A good board was made in 4 min with 10-s of steaming. A marginal board was made in 3 min with 20-s of steaming; however it took 5 min to make a board having maximum properties with 20-s steaming. Again, the additional time required to achieve maximum properties in the 20-s steamed board must be attributed

to the increased vapor pressure and/or lower resistance of the wood and resin components to stress, rather than to incomplete cure and bonding.

Higher thickness swelling calculated for the steam injection boards compared to the conventional boards neglects the higher out-of-press springback of the conventional boards. Maximum board thickness measured after 24-hour soaking was less for the steam injection boards than for the conventional boards.

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