# MONITORING RESIN CURE DURING PARTICLEBOARD MANUFACTURE USING A DIELECTRIC SYSTEM

# Sigun Wang<sup>†</sup>

Assistant Professor Tennessee Forest Products Center The University of Tennessee Knoxville, TN 37996-4507

and

# Paul M. Winistorfer<sup>†</sup>

Professor/Head Department of Wood Science and Forest Products Virginia Tech Blacksburg, VA 24061-0323

(Received November 2001)

### ABSTRACT

The hot press is a key piece of equipment in composite panel production. The press influences processing efficiency of the whole production line and the performance of products. The objective of this study was to nondestructively monitor the bonding development of particleboard during hot-pressing using a dielectric system.

With all other variables held constant, laboratory particleboard pressed with 20-s closure time reached the initial conductivity peak and the conductance valley the earliest and had the highest value at the initial conductivity peak and the conductance valley versus other closing times studied. There were strong relationships between the impedance signal and panel strength developments. There were significant effects of panel thickness on the characteristics of impedance curve.

Keywords: Particleboard, resin cure, UF, impedance, dielectric, pressing, density.

#### INTRODUCTION

The hot press is undoubtedly a key piece of equipment in composite panel production. The press influences the performance of products and the processing efficiency of the whole production line. There is a philosophy that says in simple terms: when the press closes, 90% of the cost of production has been incurred and 96% of the oriented strandboard (OSB) panels must be good panels because quality can not be added in the warehouse (Lin 1996). Most wood-based composites processing done today is controlled by a fixed "time and temperature recipe" based on empirical observation of the process variables and resin chem-

istry (Magill and Sauter 1999). These recipes must be overly conservative to allow for the variability inherent in the process. The result is a costly compromise in both process time and product quality (Magill and Sauter 1999). Wood-based composites such as OSB, plywood, laminated veneer lumber (LVL), medium density fiberboard (MDF), and particleboard are typically cured under pressure in multi-platen presses using thermosetting adhesives. In addition to the common lot-to-lot variations associated with adhesive systems, the processing of wood-based composites is further complicated by variations in wood species, moisture content, pH, and seasonal plant environmental conditions.

In the panel industry, trial-and-error meth-

Wood and Fiber Science, 35(4), 2003, pp. 532–539 © 2003 by the Society of Wood Science and Technology

<sup>†</sup> Member of SWST.

ods are used to determine the appropriate press cycle for a particular condition. Because there is no real-time feedback of panel property information for the press operator, pressing control is generally not optimized (Wang and Winistorfer 2002). There is a need for an online sensor system that can determine the state of adhesive cure within the composite during pressing, without compromising the integrity of the finished products due to invasive methods. In order to provide real-time monitoring and intelligent control for the curing of wood composites during hot pressing, several techniques are currently being developed. Wang and Winistorfer (2002) recommended the inprocess measurement and control of hot pressing through monitoring the internal density change during press opening. Chen and Beall (2000) investigated bond strength development in particleboard during pressing using acousto-ultrasonics.

For control purposes, dielectric analysis (DEA) appears to be the most promising and popular method for monitoring the resin state in composites (Kim and Lee 1993; Magill and van Doren 2000; Rubitschun 1981; Wang and Winistorfer 2000a; Wolcott and Rials 1995). DEA has been used to provide large amounts of data about basic phenomena in resin-based systems (Maffezzoli et al. 1994).

Dielectric cure monitoring, also referred to as impedance cure monitoring, is currently being developed for the wood composite industry. Dielectric cure monitoring involves monitoring changes in the viscosity and cure state of thermosetting resin systems through changes in the dielectric properties of the material. By use of remote dielectric sensors, the measurements can be made in actual processing environments such as presses, autoclaves, and ovens. Dielectric measurements are made by applying an alternating voltage between two electrodes in contact with a material and measuring the resulting alternating current. Dipoles in the material will attempt to orient with the electric field, while ions, present as impurities, will move toward the electrode of opposite polarity. Changes in the degree of alignment of dipoles and the ion mobility provide information pertaining to physical transitions in the material and to material properties such as viscosity, rigidity, reaction rate, and cure state. The objective of this study was to nondestructively monitor the bonding development of particleboard during hot pressing using a dielectric system.

### EXPERIMENTAL METHOD

Laboratory particleboard panels were manufactured while monitoring the dielectric signal. The face particle furnish was from a particleboard mill. The furnish moisture content was 6%. A commercial liquid urea-formaldehyde resin was applied to the furnish at the rate of 8% per dry wood weight in a rotating blender. Ammonium chloride was applied at a rate of 0.5%. The mat size was 406 mm by 406 mm, the target thickness was 15.9 mm, and the target panel density was 0.736 g/cm<sup>3</sup>. Every mat was produced at a platen temperature of 177°C. A set of particleboards were produced with a pressing cycle of 300 s and press closing times of 20, 40, and 60 s. Closure rate was defined as the time required to reach final position from the initial contact of the mat with the upper platen for the up-acting press. Another set of particleboards were produced with 20-s press closure time and pressing cycle of 100, 120, 145, 170, 195, 220, 245, 270, 295, 300, 320, and 345 s. There were at least two replications for each condition. The in-situ density measuring radiation sources installed on our hot press were maintained at positions of 20, 50, and 80% of mat thickness, measured from the top of the mat (Winistorfer et al. 2000). The impedance signal and coreline temperature data were recorded in real time. The frequency for the impedance test was 100 Hz based on preliminary tests.

The block diagram of the dielectric system and its integration with the press is shown in Fig. 1. A bulk field measurement sensor is placed on top of the mat while the bottom platen acts as the other electrode. The sensors are operated by applying a voltage across the elec-

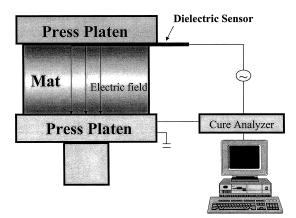


Fig. 1. Schematic diagram of the dielectric system.

trodes at a chosen frequency(s) and measuring the resultant change in amplitude and phase. If the geometry of the sample is controlled and known, then the capacities and conductive responses of a material can be expressed by the dimensionless quantities: the permittivity (E')and loss factor (E''). The dielectric loss factor is a measure of the energy lost into a system during cyclic electric excitation. The lost energy is typically due to the viscous drag of moving dipoles and ions within the sample. Ion viscosity, or electrical resistivity, is extracted from the loss factor and is directly related to the mobility of ions in the mechanical viscosity and cure state. The following equation was used for calculation of ion viscosity:

Ion viscosity

= 
$$1/(\text{loss factor} \cdot \text{frequency} \cdot 2 \cdot \pi \cdot 8.85\text{E})$$
  
- 14) (ohm)

The log ion viscosity is the log of electrical resistivity in ohm-cm. The log ion viscosity is a direct measurement of the resistivity due to ionic movement in the material under test. This quantity is inversely related to the ionic concentration and ionic mobility. Since the ionic mobility is inversely related to viscosity, the ionic concentration of a system is often constant; the log ion viscosity generally relates directly to viscosity and rigidity changes in the system under test. The dlog ion viscosity/dt

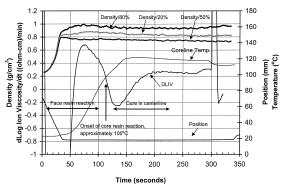


Fig. 2. Dielectric signal, *in-situ* density profiles, coreline temperature and press position of particleboard mat during pressing (Resource: Wang and Winistorfer 2000a).

(DLIV) is the first derivative (slope) of ion viscosity with respect to time. The slope or rate of change has been correlated to reaction rate and cure state during the course of thermosetting cure (Nolometrix Micromet). The dlog ion viscosity/dt is used to represent dielectric signal in this paper. The dielectric instrument used in this research was an ICAM-1500 Cure Analyzer made by Micromet Instruments. Data were collected and analyzed using Icam Instrument System Software.

Four 3-by-14-in. MOR/MOE and six 2-by-2 in. internal bond (IB) specimens were obtained from each panel. Density, MOR/MOE, and internal bond (IB) tests were performed in accordance with ASTM 1037-96 A (1996).

### RESULTS AND DISCUSSIONS

A typical dielectric signal obtained from pressing a particleboard mat is shown in Fig. 2. The *in-situ* density measurements, press position, and coreline temperature from the mat are also shown. Specific characteristics observed from the dLog ion viscosity/dt curve include a high first peak, a conductance valley, and a subsequent rise. As wood and water also are dielectric materials, the dielectric signal obtained from pressing a particleboard mat resulted from wood heating, water vapor, and resin cure. The first peak was caused by the closing of the press, the initial generation of steam, wood heating, and the face resin reac-

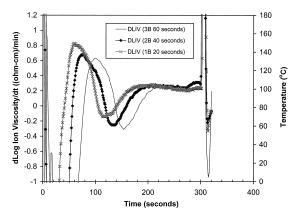


Fig. 3. The effect of pressing closure time on the conductance signal.

tion. The conductance valley corresponded with the onset of the core resin reaction, which occurred at approximately 100°C coreline temperature. When the pressing time was 110 s, coreline temperature reached 100°C. The subsequent rise in the data signal correlated with the ongoing cure of the core resin and development of IB. During the first 40 s of the closing period, the densities of three layers increased quickly with nearly the same consolidation rates before the press reached final position. Immediately upon reaching final closure position, there was a marked change in mat density at the three monitoring locations. Both surface densities continued to increase after the press had reached final position, while the core layer density decreased. When the onset of core resin reaction occurred and coreline temperature reached 100°C, both face densities gradually declined during the remainder of the cycle; indicating the end of Stage III (a period of surface layer consolidation) (Wang and Winistorfer 2000b) when coreline temperature reached 100°C. This implied that the face resin reaction occurred while both surface areas were at the continual compression state during the consolidation period (Stage III). When core resin started to cure, the mat was at Stage IV and the core area was in the compression state. The continual compression state would appear a better environment for bond formation than the con-

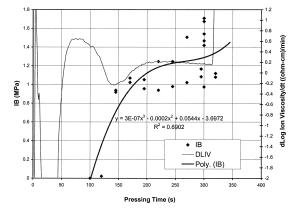


Fig. 4. Correlation of IB development with the dielectric signal for particleboard.

tinual springback state. The mat in Fig. 2 was in a good environment for the resin to cure and to form effective bonds. The data in Fig. 2 were based on a final panel thickness of 15.9 mm.

### Pressing closure time

DLIV curves obtained from particleboards that were pressed with different pressing closure times are shown Fig. 3. There was a significant effect of pressing closure time on the dielectric signal changes. With all other variables held constant, the particleboard pressed with 20-s closure time reached the initial conductivity peak and the conductance valley the earliest and had the highest value at the initial conductivity peak and the conductance valley versus other closing times. Press closure time controls the mat consolidation and consequently controls the rate of heat application to furnish. The shorter the press closure time, the faster the heat transfer during pressing. Although the values at the initial conductivity peak and the conductance valley were different for three pressing closure times, the three products reached the same subsequent final rise. Further research should examine whether or not the final rise in DLIV curves is more related to mat structure, for example, mat thickness, mat density, and moisture content.

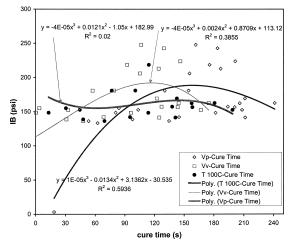


Fig. 5. Correlation between IB and cure times (Vp-Cure Time—time between the detection of the first peak in the ion viscosity slope signal and the initiation of the degas pressing cycle; Vv-Cure Time—time between the detection of the first valley in the ion viscosity slope signal and the initiation of the degas pressing cycle; T100-Cure Time—time between the detection of 100°C in the core and the initiation of the degas pressing cycle).

## Strength formation

Figure 4 shows results of measured IB values from 24 boards at different pressing times with the conductance curve. According to the IB data, the two shortest press cycles (100 and 120 s) exhibited an undercure condition. The IB value was only 0.022 MPa at the pressing time 120 s. IB began to develop when the conductance data nearly reached a valley. IB was 0.896 MPa when the conductance data reached the valley at the pressing time 140 s. The correlation coefficient between the IB and pressing time was 0.831.

In order to use the conductance curve for monitoring and assessing hot pressing, three cure times were calculated: 1) Vp-cure time is the time between the detection of the first peak in the ion viscosity slope signal and the initiation of the degas pressing cycle; 2) Vv-cure time is the time between the detection of the first valley in the ion viscosity slope signal and the initiation of the degas pressing cycle; and 3) T100-cure time is the time between the detection of 100°C in the core and the initiation

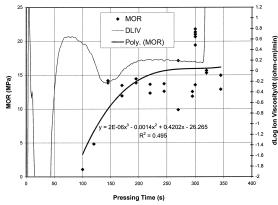


Fig. 6. Correlation of MOR development with the dielectric signal for particleboard.

of the degas pressing cycle. The T100-cure time is currently used to determine total pressing cycle by panel industry. Figure 5 shows the correlation between IB and cure times. The cure time between the detection of the first peak in the ion viscosity slope signal and the initiation of the degas pressing cycle had the greatest correlation with internal bond strength. The time cure between the detection of 100°C in the core and the initiation of the degas pressing cycle had the lowest correlation with internal bond strength. The thermocouple results have more disperse clusters and variations at each pressing than the sensor results, thus leading to a lower correlation coefficient. This demonstrates the possible advantage of the sensor in reducing the variability of the monitoring of the mat internal conditions, and thus improving the correlation with panel properties.

Figures 6 and 7 show results of measured MOR and MOE values from 24 boards at different pressing times with the conductance curve, respectively. Both MOR and MOE began to develop much earlier than IB. The MOR and MOE were 1.08 MPa and 174.1 MPa at the pressing time 100 s, respectively. The MOR and MOE were about 13.8 MPa and 2482 MPa, respectively, when the conductance data reached the valley at the pressing time 140 s. The correlation coefficients be-

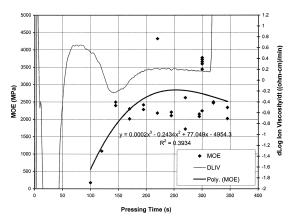


Fig. 7. Correlation of MOE development with the dielectric signal for particleboard.

tween the pressing time and MOR/MOE were 0.704 and 0.627, respectively.

# Panel thickness

Figures 8 and 9 show dielectric signal, *insitu* density profiles, coreline temperature, and press position of 15.9-mm and 31.8-mm thick particleboard, respectively. For the 15.9-mmthick particleboard, heat easily transferred from press platens to the mat. The coreline temperature reached 100°C at the pressing time of 130 s. For the 31.8-mm-thick particleboard, heat transfer from the press platens to the mat center was much slower than for the 15.9-mm-thick particleboard. The coreline temperature reached 100°C at the pressing time of 340 s, which is 2.6 times longer than the time required for the 15.9-mm-thick particleboard.

The DLIV curve for the 15.9-mm-thick particleboard (Fig. 8) was smooth and included a high first peak, a conductance valley, and a subsequent rise. On the contrary, the DLIV curve for the 31.8-mm-thick particleboard was not smooth (Fig. 9). The conductance valley and subsequent rise were not as significant as for the 15.9-mm-thick particleboard. Wang and Winistorfer (2000a and b) reported that the vertical density profile of wood composites is formed from a combination of actions that occurs both during consolidation and also after

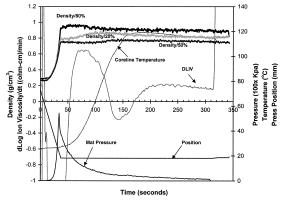


Fig. 8. Dielectric signal, *in-situ* density profiles, coreline temperature and press position of 15.9-mm-thick particleboard mat.

the press has reached final position. Recognition of the consolidation changes during pressing implies that there is not a steady state for resin bonding formation during pressing. During pressing, the wood furnish elements are not in a steady state of contact. The more severe the unsteady phase during hot pressing, the poorer quality of the bond formation. The thicker the panel pressed, the more severe the unsteady contacting phase. The *in-situ* density profile for the 15.9-mm-thick particleboard shows that after the press had reached final position, *in-situ* density changes mainly occurred during pressing times of 40 to 100 s (Fig. 8). During the same pressing period, the

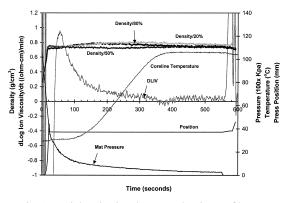


Fig. 9. Dielectric signal, *in-situ* density profiles, coreline temperature and press position of 31.8-mm-thick particleboard mat.

coreline temperature increased from room temperature to  $65^{\circ}$ C and the DLIV curve was at the high first peak, suggesting that the severe unsteady contacting phase ended before onset of the core resin reaction. For the 31.8mm-thick particleboard, the *in-situ* density profile shows that after the press had reached final position, *in-situ* density changes mainly occurred during pressing times of 20 to 300 s (Fig. 9). During the same pressing period, the coreline temperature increased from room temperature to 87°C and the DLIV curve was near the conductance valley.

#### CONCLUSIONS

Specific characteristics observed from the dLog ion viscosity/dt curve include a high first peak, a conductance valley, and a subsequent rise. The first peak was caused mainly by the closing of the press, the initial generation of steam, wood heating, and the face resin reaction. The conductance valley corresponded with the onset of the core resin reaction, which occurred at approximately 100°C coreline temperature. When the pressing time was 110 s, coreline temperature reached 100°C. The subsequent rise in the data signal can be correlated with the ongoing cure of the core resin and development of IB.

With all other variables held constant, the data clearly indicated that the particleboard pressed with 20 s closure time reached the initial conductivity peak and the conductance valley the earliest and had the highest value at the initial conductivity peak and the conductance valley versus other closing times studied. There were strong relationships between the impedance signal and panel strength developments. There were significant effects of panel thickness on the characteristics of the DLIV curve.

#### ACKNOWLEDGMENTS

This work was partly supported by the Tennessee Agricultural Experiment Station, Project MS No. 72 and No. 83. The authors would like to thank colleague Chris Helton for his helpful assistance. The authors would also like to thank Georgia-Pacific Resins Inc. for its contribution to this research.

#### REFERENCES

- AMERICAN SOCIETY FOR TESTING AND MATERIALS (ASTM). 1996. Standard test methods for evaluating properties of wood-based fiber and particle panel materials. ASTM1037-96 a. ASTM, W. Conshohocken, PA.
- CHEN, L., AND F. C. BEALL. 2000. Monitoring bond strength development in particleboard during pressing, using acousto-ultrasonics. Wood Fiber Sci 32(4):466– 477.
- KIM, J. S., AND D. G. LEE. 1993. On-line cure monitoring and viscosity measurement of carbon fiber composite materials. J. Mater. Proc. Technol. 37:405–416.
- KING, R. J., AND R. W. RICE. 1996. Monitoring resin cure of medium density fiberboard using dielectric sensors. Materials Res. Soc. Symposia Proc., 430:601–605.
- LIN, F. S. 1996. Meeting OSB customer requirements. Pages 163–170 in T. M. Maloney, ed. Proc. 30th Particleboard/Composite Materials Symposium. Washington State University, Pullman, WA.
- MAFFEZZOLI, A., A. TRIVISANO, M. OPALICKI, J. MIJOVIC, AND J. M. KENNY. 1994. Correlation between dielectric and chemorheological properties during cure of epoxybased composites. J. Mater. Sci. 29:800–808.
- MAGILL, R., AND S. L. SAUTER. 1999. In-situ impedance sensors provide real-time monitoring and intelligent control for the curing of engineered wood products. Page 181 *in* Proc. 33rd International Particleboard/ Composites Materials Symposium, April 13–15, 1999, Pullman, WA.
- , AND J. VAN DOREN. 2000. Intelligent monitoring and control wood panel press curing in-situ impedance sensors. Pages 89–95 *in* Proc. 34th International Particleboard/Composites Materials Symposium Proc. April 4–6, 2000, Pullman, WA.
- NOLOMETRIX MICROMET. ICAM-1500 Cure analyzer users guide. P. 11.
- RUBITSCHUN, R. A. 1981. Measuring adhesive cure by dielectric analysis. M.S. Thesis, Oregon State University, Corvallis, OR.
- WANG, S., AND P. M. WINISTORFER. 2000a. Unsteady contacting phase during hot pressing and its influence on resin cure and bond formation. Pages 155–161 *in* Proc. Wood Adhesives 2000, June 22–23, 2000, South Lake Tahoe, NV.
- , and \_\_\_\_\_. 2000b. Fundamentals of vertical density profile formation in wood composites. Part 2. Methodology of vertical density formation under dynamic conditions. Wood Fiber Sci. 32(2):220–238.
- , and \_\_\_\_\_. 2002. Monitoring *in-situ* density change for in-process measurement and control of hot pressing. Forest Prod. J. 52(7/8):77–82.
- WINISTORFER, P. M., W. W. MOSCHLER, S. WANG, E.

DEPAULA, AND B. L. BLEDSOE. 2000. Fundamentals of vertical density profile formation in wood composites. Part 1. *In-situ* density measurement of the consolidation process. Wood Fiber Sci. 32(2):209–219.

WOLCOTT, M. P., AND T. G. RIALS. 1995. In-situ monitoring of adhesives for wood-based composite. Pages 185–193 in Proc. 29th International Particleboard/Composites Materials Symposium. Pullman, WA.