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CURING OF PHENOL-FORMALDEHYDE ADHESIVE IN BOARDS OF DIFFERENT THICKNESSES

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ABSTRACT

The influence of board thickness on the rate of curing of a thermosetting adhesive was studied. A yellow poplar veneer and a phenol-formaldehyde adhesive were used to manufacture multi-layer boards with four different thicknesses: 1.8, 3.6, 5.4, and 7.2 mm. The curing of the adhesive was investigated by means of dielectric analysis during the pressing process in a hot-press. An LCR meter and an IDEX sensor were used to measure changes in the dielectric properties of the adhesive bond. A thermocouple was used to measure the temperature in the bondline. It was found that dielectric analysis was a suitable method for monitoring the curing of phenol-formaldehyde adhesives. The rate of cure was the highest for the thinnest boards and it decreased with the thickness of the board. A power equation was used to describe the influence of board thickness on the time needed to achieve the desired degree of cure.

KEY WORDS: board thickness, curing, dielectric analysis, phenol-formaldehyde adhesive, temperature

INTRODUCTION

Hot pressing is the most energy-consuming and therefore cost-intensive part of the production of wood-based composites. For this reason it is important to minimize the curing time of thermosetting adhesives by optimizing the pressing conditions. Achievement of sufficient bond strength during curing depends on several pressing parameters: time, pressure and temperature. The adhesive in the bondline starts to cure when a certain temperature level is reached, depending on the type of adhesive used for bonding. Phenol-formaldehyde adhesives need higher temperatures for hardening than melamine-formaldehyde or urea-formaldehyde adhesives (Vick 1999).

The transfer of heat from the conventional hot-press plates to the bonded assembly depends mainly on the thermal conductivity of the wood. This conductivity varies among wood species, and is significantly affected by density, moisture content, extractive content, grain direction, structural irregularities such as checks and knots, the fibril angle, and temperature (Simpson and TenWolde 1999). The rate of increase of the temperature in the adhesive bondline also depends on the distance from the hot plate, where the heat is generated, to the (innermost) bondline.

The rate of temperature increase affects the adhesive cure process and the development of bond strength.

The curing or hardening process of phenol-formaldehyde adhesive in a hot-press is a continuation of the reaction involved in the synthesizing of the adhesive. Liquid phenol-formaldehyde molecules with a low molecular weight convert to macromolecules through chain extension, chain branching and crosslinking, and form a three-dimensional network of infinite molecular weight (Wang et al. 1995). The cured adhesive is solid and capable of transferring substantial load.

A faster increase in the temperature in the bondline accelerates the curing reaction. At higher temperatures, adhesives cure more rapidly and develop higher bond strength (Heineman et al. 2002). Such polymers are longer and more crosslinked. The rate of phenol-formaldehyde adhesive strength development increases with the temperature at the bondline (Wang et al. 1995).

The objective of this research was to determine the influence of board thickness on the temperature increase in the bondline and on the rate of curing of the thermosetting phenol-formaldehyde adhesive. The suitability of dielectric analysis for monitoring the cure of this adhesive was also examined.

MATERIAL AND METHODS

Phenol-formaldehyde (PF) adhesive made for OSB production and yellow poplar (*Liriodendron tulipifera* L.) sapwood veneer were used in the study. The PF adhesive had a pH value of 10.0, a specific gravity of 1.22 g.cm^{-3} and a solids content of 55%. Veneer with a thickness of 0.9 mm was produced by peeling, and cut to rectangular sheets with dimensions of 110×125 mm. The veneer sheets were conditioned to a moisture content of 7.5 % before bonding with the PF adhesive.

Monitoring of PF adhesive cure depending on board thickness

The PF adhesive cure was evaluated by dielectric analysis (DEA), using a fringe-field sensor having an interdigitized design (IDEX, Netzsch 066S). The IDEX sensor was positioned at the central (innermost) bondline of the board and controlled by a precision LCR meter (Agilent, type 4285A) operating at the frequency of 100 kHz. The depth of the electric field was 0.115 mm. The temperature at the bondline was recorded by a type J thermocouple connected to a data acquisition switch unit (Agilent, type 34970A). Changes in the conductance (G) and the temperature (T) were monitored every 2 s for a period of 10 min during pressing with 0.8 MPa in a conventional hydraulic hot-press.

Four bonding assemblies (boards with parallel orientation of veneers) with different thicknesses (d) were prepared by using 2, 4, 6, and 8 sheets of veneer (Fig. 1). The PF adhesive was used only for the central bondline and it was applied at an application rate of 180 g.m^{-2} to the surface of one veneer. The distance from the board surface to the PF adhesive bondline (d/2) varied from 0.9 mm to 3.6 mm (Tab. 1). Three boards were made for each thickness.

The degree of cure (α) or the degree of conversion of the PF adhesive was determined as a function of curing time (t) from conductance data (Sernek and Kamke 2007):

$$\alpha(t) = \frac{G_{\max} - G_t}{G_{\max} - G_{\min}} \qquad (1)$$

where G_{max} is the maximum conductance, G_t is the conductance during cure, and G_{min} is the minimum conductance.

Tab. 1: Composition and thickness of the boards

Label of the board	Number of veneer sheets in the board	Board thickness (d) in mm	The distance to PF bondline (d/2) in mm
YP-0.9-PF	2	1.8	0.9
YP-1.8-PF	4	3.6	1.8
YP-2.7-PF	6	5.4	2.7
YP-3.6-PF	8	7.2	3.6

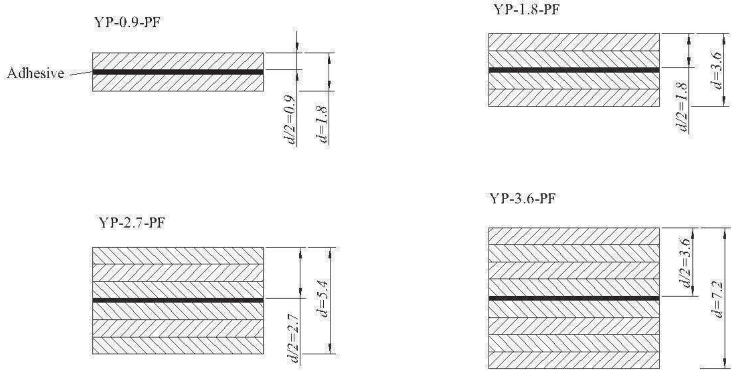

Fig. 1: Composition of boards with different thicknesses

RESULTS

The increase of the temperature at the PF adhesive bondline

The thickness of the board significantly affected the rate of increase of the temperature at the PF adhesive bondline (Fig. 2). There was very rapid increase of temperature for the thinnest board (YP-0.9-PF), since the heat had to be conducted through a very short distance of 0.9 mm in order to reach the adhesive bondline. In the case of this board, the temperature jumped from 20°C to about 100°C before the press totally closed and the measurements started. It has to be pointed out that it took 10 s to place the bonded assembly on the hot plate and close the press. About 30 s after press closure, the temperature at the bondline of the thinnest board approached the temperature of the press plates, which was set to 200°C. The actual temperature of the plates was about 5°C lower due to a deviation from the set temperature of the press.

For the three thicker boards, the rate of the increase of the temperature was slower and the pattern of the temperature increase changed, too. At a temperature slightly above 100°C, the rate of the increase of the temperature at the PF bondline of board YP-1.8-PF slowed down, whereas a brief temperature “plateau” was detected in the case of the thickest two boards (YP-2.7-PF and YP-3.6-PF). This happened because of a cooling effect due to the consumption of energy for the evaporation of water from the veneer and adhesive. It took about 20-30 s before the water evaporated and escaped from the PF bondline of the thicker boards, whereas this occurred instantaneously for the thinnest one. When most of water had evaporated from the bondline, the temperature slowly increased towards the set temperature.

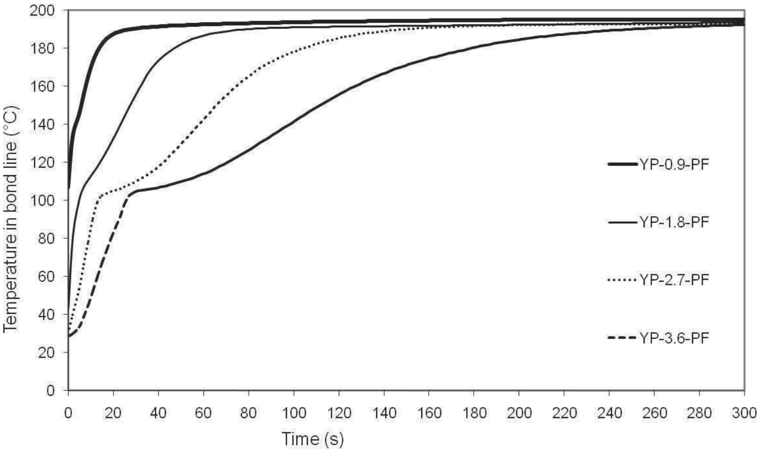

Fig. 2: The increase of temperature at the PF adhesive bondline for boards with different thicknesses

The change in the conductance of the PF adhesive bondline

The DEA measurements revealed that the conductance of the PF adhesive bondline changed significantly during hot-pressing (Fig. 3). The obtained dielectric response was due to the changes in the transitional mobility of ions and the rotational mobility of dipoles in the presence of an electric field. The loss of water from an aqueous adhesive system also affected ion mobility (Sernek and Kamke 2007). The conductance changed partially due to the increase of temperature (the increasing temperature decreased the viscosity and increased the conductance of the adhesive), but it was mainly caused by the curing reaction of the adhesive. As the chemical reaction of the PF adhesive progressed, ion and dipole mobility became increasingly restricted.

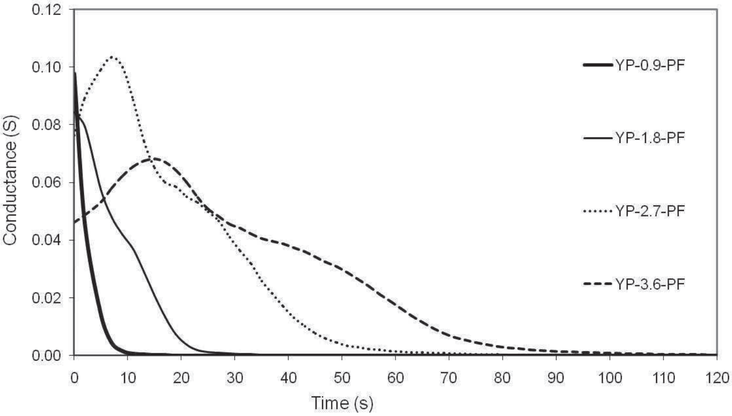

Fig. 3: The change in conductance of the PF adhesive bondline during curing

For the thinner boards (i.e. YP-0.9-PF and YP-1.8-PF), the effect of the rapid increase of temperature on conductance was not present since it occurred during the closing of the press, when measurements has not yet been started.

For the thicker boards (i.e. YP-2.7-PF and YP-3.6-PF) conductance increased with temperature, and then started to decrease. The decrease in conductance occurred due to the onset of curing, but also partly because of intensive water diffusion/evaporation from the bondline. The point when conductance reached a maximum and then began to decrease was set as the start of the conversion of the PF adhesive from a liquid to a solid state (Sernek and Kamke 2007). At that moment, the temperature at the PF adhesive bondline was over 70°C, which provided enough energy to start a polycondensation reaction.

DISCUSSION

The influence of board thickness on the rate of curing of the PF adhesive

The curing of PF adhesives, which involves chemical and physical phenomena, is related to the dielectric changes monitored by DEA and an IDEX sensor, as confirmed previously (Sernek and Kamke 2007). The degree of cure (a) was calculated from the conductance data by using Equation 1. It is clear that the degree of cure of PF adhesive depends on the pressing time and on the thickness of the board (Fig. 4). The curing process was very rapid for thin boards. The PF adhesive achieved 99 % conversion in about 10 s for board YP-0.9-PF, and in 30 s for board YP-1.8-PF. This was due to the fast conduction of heat and rapid temperature rise in the bondline. Note that temperature has been already over 100°C in the PF adhesive bondline of the thinnest board when the press closed.

The PF adhesive cured much more slowly in the case of the thicker boards. With increased board thickness, the heat had to be transferred over a greater distance, which took more time. Thus, the temperature in the PF bondline of the thicker boards increased slowly, so that it took 15 s of pressing time to begin the cure reaction in the PF bondline of the thickest board (YP-3.6-PF). A small "stagnation" of the increase of the temperature was observed around 100°C in the case of the thicker boards (Fig. 2). Most of the added heat energy around this temperature was consumed by water evaporation, and only a small part of it was used for heating the PF adhesive bondline. With thicker boards, a larger quantity of water had to evaporate, so this stagnation became more explicit and also had a significant effect on the rate of curing. The PF adhesive reached 99 % of conversion in 65 s in the case of board YP-2.7-PF, and 105 s in the case of board YP-3.6-PF.

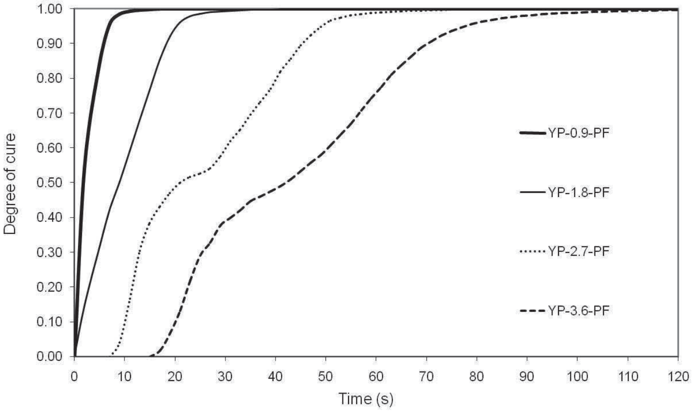

Fig. 4: Differences in the degree of cure of PF adhesive due to different increases of temperature caused by different board thicknesses

The rate of cure of the PF adhesive was expressed as /t. It was the highest at the onset of the curing process, and decreased with cure propagation (Fig. 5). As expected, the rate of cure was the highest in the case of the thinnest board, and the lowest in the case of the thickest board. The reaction rate is strongly affected by temperature (Sernek and Kamke 2007), since higher temperatures make more energy available for the reaction.

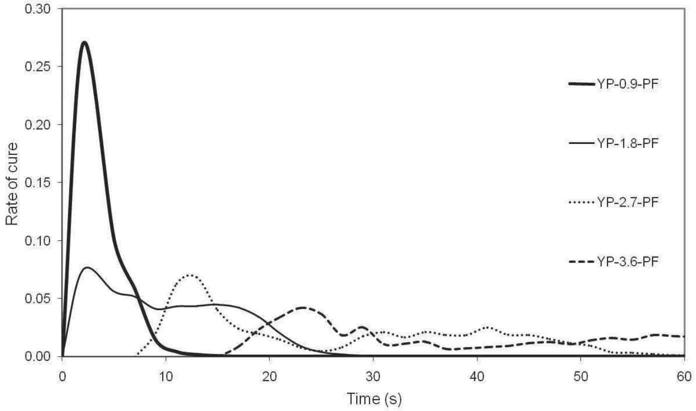

Fig. 5: Rate of cure of the PF adhesive as a function of the curing time

It is evident that increased board thickness increases the time needed to achieve a particular conversion of the PF adhesive (Fig. 6). A power equation was used to describe the relationship between curing time and the distance from the surface of the board to the bondline.

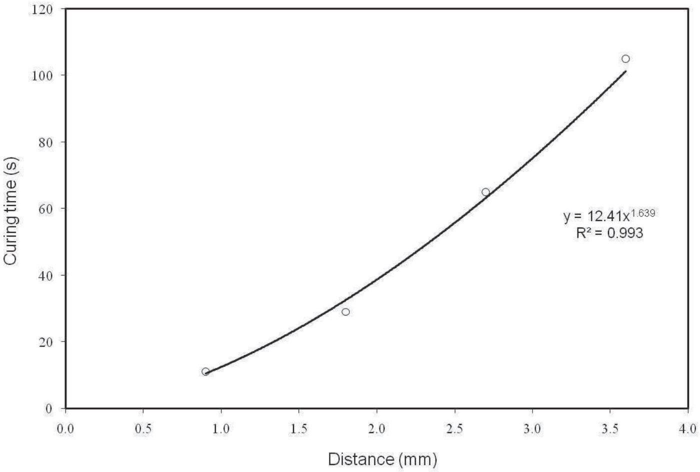

Fig. 6: Estimated curing time for the achievement of 99 % of conversion of the PF adhesive as a function of the distance from the surface of the board to the bondline (d/2)

CONCLUSIONS

The temperature in the PF adhesive bondline is one of the main factors which affects curing. The increase of temperature of the bondline strongly depends on the thickness of the board. The temperature increased very rapidly in the case of the thinnest board (d/2=0.9 mm), and reached the set temperature in about 30 s of pressing time. This value increased substantially with increasing board thickness. A power equation was used to describe the influence of board thickness on the time needed to achieve the desired degree of cure.

Dielectric analysis was found to be a suitable method for monitoring the curing of the PF adhesive. The measured conductance gave a reliable estimate of the degree of cure. Board thickness influenced the degree of cure, which increased with extension of the pressing time. The rate of cure was the highest in the case of the thinnest boards, and it decreased with increasing board thickness.

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