

CHARACTERIZING LIQUID RESIN PENETRATION IN WOOD USING A MERCURY INTRUSION POROSIMETER

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ABSTRACT

A method has been developed to characterize resin penetration by measuring the change in the pore size distribution and porosity of samples with and without resin using a mercury intrusion porosimeter. The method was used to study the liquid phenol-formaldehyde resin penetration in birch and aspen veneer samples as a function of different curing conditions. For both the birch and aspen samples, the liquid PF resin was found to penetrate mostly into the bigger pores (diameter $\geq 40 \mu\text{m}$ for birch/LPF and diameter $\geq 10 \mu\text{m}$ for aspen/LPF) for specimens cured in the oven. Resin penetrated into the smaller pores under the influence of pressure when cured in a hot-press, especially when the specimens were cured at face location in the hot-press. Under all curing conditions, some resin only partially filled some pores and resulted in an increase in pores of smaller sizes.

Keywords: Mercury intrusion porosimeter (MIP), pore structure, pore volume change, resin penetration, curing condition.

INTRODUCTION

Since wood is a porous material, adhesive penetration plays an important role in wood adhesion. To facilitate adequate mechanical interlocking, the adhesive must have penetrated into the wood before it is cured. At least, the low molecular weight molecules of the adhesive should have penetrated into the wood pores (Ellis 1993). The degree of penetration depends on the type and quality of wood, time of contact, properties of the adhesive, and curing speed (Marra 1992).

Wood is a heterogeneous material and wood pore structure varies greatly among species, logs, and different parts within the same log, resulting in large differences in location and quantity of the penetrated resin. Smith and Côté

(1971) observed that the resin penetration was greater in the earlywood than in the latewood. Penetration of UF adhesive in the tangential direction was greater than in the radial direction for beech veneer (Sernek et al. 1999). The natural variability of wood has the largest influence on the uniformity of resin penetration and increases the difficulties of measuring resin penetration. Several techniques have been employed to examine resin penetration. White et al. (1977) used neutron activation analysis (NAA) to study resin penetration in southern pine.

SEM (scanning electron microscopy) and TEM (transmission electron microscopy) in combination with other techniques were also used to measure resin penetration. One of the techniques was SEM with EDAX (energy dispersive analysis of X-ray). SEM combined with EDAX was able to obtain qualitative and semi-quantitative information on adhesive penetration

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(Smith and Côte 1971; Koran and Vasishth 1972; Bolton et al. 1988; Wright and Mathias 1993).

Rapp et al. (1999) used electron energy loss spectroscopy (EELS) in combination with transmission electron microscopy (TEM-EELS) to examine the penetration of partly methylated hydroxymethyl melamine resin in spruce. An advantage of this technique compared to SEM-EDAX is that resin penetration can be examined without adding any trace elements. TEM-EELS has been shown to have a high resolution of less than 1 nm. Gindl (2001) used SEM to investigate the gluelines in southern yellow pine. To observe the penetration of PF resin into the cell lumen, the cell-wall material was dissolved to expose the cured PF resin. The resin penetrated through cut-open tracheids and rays.

Fluorescence microscopy was also used (Gollob et al. 1985; Murmanis et al. 1986). Using this technique, lumen penetration can be examined. Brady and Kamke (1988) stained the PF resin/aspen or PF resin/Douglas-fir with 0.2% acridine red fluorochrome dye solution, while Sernek et al. (1999) soaked the UF resin/beech sections in 0.5% Brilliant Sulphaflavine solution for 21 h followed by 3 h of soaking in 0.5% Safranin O Solution. Typically, this type of technique involved adding a fluorescent marker into the resin to make it visible.

Though a large amount of information on resin penetration has been collected over the years using various techniques, the location of the penetrated resin under influence of process conditions is still largely unclear. The relationship between wood pore structure and resin penetration is still not well understood. The objective of this study is to investigate the feasibility of characterizing resin penetration in wood by measuring the pore structure change before and after resin application using a mercury intrusion porosimeter.

MATERIALS AND EXPERIMENTAL

Materials

Commercial birch veneer with a thickness of 0.62 mm was used for the resin penetration

study. Aspen veneer with an original thickness of 3–5 mm was sanded with sandpaper to 0.6–0.8 mm and cleaned with compressed air before the resin application. Sanding was done on the surface opposite to the side with resin application.

A commercial liquid phenol-formaldehyde (LPF) resin designed for both OSB core and face applications was used in this study. Its solids content is around 50%.

Specimen preparation

In order to have statistically representative results, birch and aspen veneers were randomly selected. Care was taken to ensure that no visible flaws were on the surface. Each piece of the veneer was cut into six equal parts (three pairs) of dimensions 35 × 30 mm (Fig. 1); each part was used for a designated test as specified in Table 1. The motivation for using the six parts from the same piece was to reduce variability and minimize the effect of tree growth characteristics on the pore structure. The six parts were assumed to have a similar pore size distribution. As a result, the effect of resin application and curing conditions can be compared. Five samples were measured for each type of test in order to obtain statistically meaningful results.

All veneers were dried in an oven at $103 \pm 2^\circ\text{C}$ for 2 h prior to resin application in order to remove the effect of initial moisture content variation among the specimens. The LPF adhesive was applied by hand with a hard-plastic roller at room temperature. Two curing conditions were used in this study: cured in oven at 150°C for 20 min and cured in a Dieffenbacher hot-press. For the oven-curing study, specimens without LPF were also put in the oven under the same curing condition prior to the determination

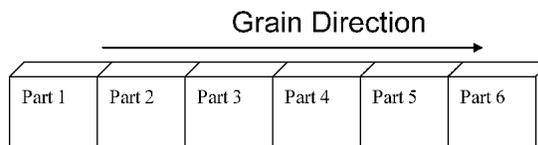


FIG. 1. Specimen preparation: a piece of veneer was cut into six equal parts of dimension. 35 × 30 × 0.6 ~ 0.8 mm.

TABLE 1. Specimen treatment conditions for different parts.

No.	Purpose	Resin application	Oven cure	Hot-press cure		
				Core	Face	Replication
1	To determine the original pore structure of the oven-dry wood		y			5
2	To measure resin penetration without external pressure	y	y			5
3	To determine the pore structure of wood hot-pressed in the core location			y		5
4	To measure resin penetration with external pressure in the core location	y		y		5
5	To determine the pore structure of wood hot-pressed in the face location				y	5
6	To measure resin penetration with external pressure in the face location	y			y	5

of the original pore distribution in order for these specimens to have the same thermal history as the specimens with resin. Similarly, under the hot-press curing conditions, specimens without LPF were also placed in the face and core locations of the hot-press to determine the change in the pore size distribution due to the pressure alone.

Specimens (parts 3 ~ 6) with and without LPF resin were compressed and cured in the hot-press (at the core and the face position, respectively). The test specimens were separated from the neighboring strands using silicon paper. The hot-press condition was similar to the typical OSB panel manufacturing settings with a target density of 640 kg/m³ at 200°C for 5 min. The dimension of the three-layer (face/core/face, 1:2:1 by weight) panel was 610 × 610 × 11 mm. Face strands were at 6% moisture content. Core strands were dried at 50°C for 48 h to obtain a moisture content range of 2–3%.

Resin and moisture contents of the test specimens were determined as shown in Table 2. The change in pore volume and pore size distribution of the test specimens with and without resin was used to characterize resin penetration. Since resin content may influence the degree of resin penetration, pore volume change of the test specimens with and without resin was normal-

TABLE 2. Applied resin content and moisture content of the specimens by weight.

	Oven		Core		Face	
	Resin (%)	MC (%)	Resin (%)	MC (%)	Resin (%)	MC (%)
Birch/LPF	4.64	4.64	6.05	6.05	5.46	5.46
Aspen/LPF	4.51	4.51	5.29	5.29	4.45	4.45

MC: Moisture content

ized by the resin content of 5% using the following formula:

$$\Delta V = (V_1 - V_2) \frac{5\%}{RC} \quad (1)$$

where ΔV is the normalized pore volume change of the test specimens with and without resin, V_1 is the cumulative or incremental intrusion volume of specimen without resin, V_2 is the cumulative or incremental intrusion volume of specimen with resin, RC is the applied resin content,

$RC = \frac{W_{resin}}{W_{wood}}$, W_{resin} is the solid resin weight and W_{wood} is the oven-dry weight of wood.

Cumulative or incremental intrusion volume change was calculated by the following equation:

$$\% = \frac{V_1 - V_2}{V_1} \times 100\% \quad (2)$$

The total volume of the specimens was characterized using the cumulative intrusion volume, and the volume of pores in a specified diameter

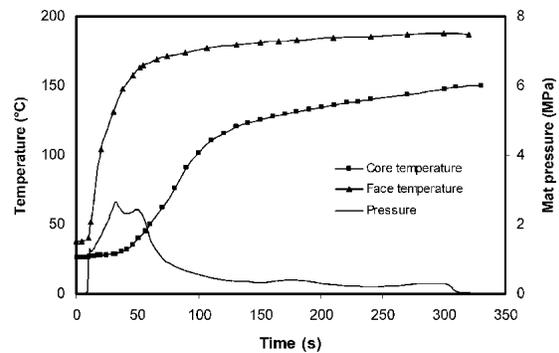


FIG. 2. Hot-press profiles.

range was quantified using the incremental intrusion volume. The cumulative intrusion volume was expressed as mL/g by normalizing the total intrusion volume against the sample weight. The incremental intrusion volume was calculated by subtracting the cumulative volume V_i measured at pressure P_i from the cumulative volume V_{i+1} measured at a higher pressure P_{i+1} and was also normalized by the sample weight. The plot of the incremental intrusion volume vs. the pore diameter illustrates pore size distribution. Figure 3 gives an example of the plot.

The in situ monitoring probe in the hot press was located either at the position of 50% of mat thickness (the core position) or at the face position under three layers of strands from the top. The pressman system allowed the pressing procedure to be programmed and the pressure and temperature to be recorded in real time (Fig. 2).

All the specimens after curing were sealed in plastic bags and stored in a desiccator prior to mercury intrusion porosimeter experiments.

Mercury intrusion porosimeter

Pore volume and pore size distribution measurements were performed using a mercury intrusion porosimeter (AutoPore III, Micromeritics). The mercury intrusion porosimeter used in this study can detect pores between 6 nm and 160 μm in size. The mercury intrusion porosimetry method is based on the Washburn equation

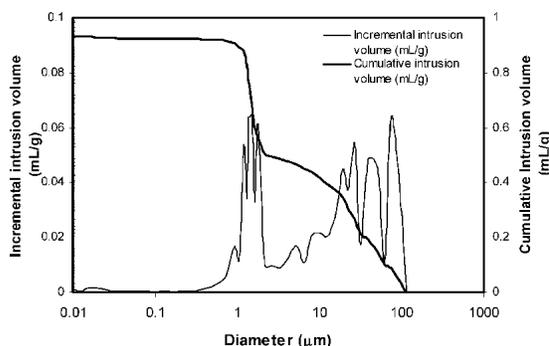


FIG. 3. The typical curves for the incremental intrusion volume and the cumulative intrusion volume for birch samples.

(Washburn 1921) that relates pore size to intrusion pressure with the assumption that pores are cylindrical. Mercury is used because it is a non-wetting liquid that will not penetrate pores by capillary action. For each test, a specimen of 10×5 mm in dimensions was placed in a suitable penetrometer for measurement. The penetrometer and the sample were then evacuated under vacuum to remove air and residual moisture from the pores. Prior to the test, all specimens were placed inside the oven at $103 \pm 2^\circ\text{C}$ for 5 min to remove moisture and were cooled down to room temperature in a desiccator. The pressure range for intrusion was set between 0 and 138 MPa. This pressure range was selected based on a large number of trials. Typically, the cumulative intrusion volume vs. pressure reached a plateau before the intrusion pressure increased to 138 MPa.

RESULTS AND DISCUSSION

The degree of resin penetration in wood material was characterized based on the difference in pore volume and pore size distribution for specimens with and without resin application. This difference was thought to be caused mainly by the resin penetrated into the wood pores. An assumption that the different parts taken from the same veneer piece have the same porosity and pore structure was made so that the original pore structure of the wood could be determined from the neighboring parts (Part 1, 3, and 5). In this study, the pore volume is always reported as the volume per unit weight of the test sample. After curing, the penetrated resin will either fill or partially fill the pore space. Unfortunately, that is indistinguishable with the technique. As a result, a pore that is partially filled by resin will make the interpretation of the results more difficult.

Before the resin penetration measurement, the pore volume of the cured resin was also characterized. A thin liquid PF resin film was formed in an aluminum dish and was cured in the oven at two different conditions: $103 \pm 2^\circ\text{C}$ for 4 h and $103 \pm 2^\circ\text{C}$ for 24 h. Resin porosity was

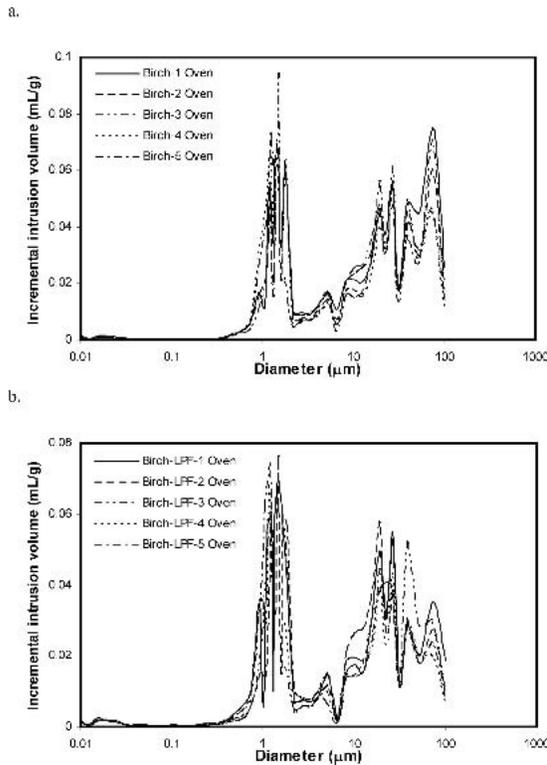


FIG. 4. Five replications of birch samples with and without LPF resin cured in the oven. a. Five replications of birch samples cured in oven. b. Five replications of birch/LPF samples cured in oven.

measured using the mercury intrusion porosimeter. The pore volume of the cured resin based on three replications was 0.0048 ± 0.003 (mL/g) for the 4-h curing condition and 0.0058 ± 0.0031 (mL/g) for the 24-h curing condition, respectively. But the actual porosity of the resin penetrated in wood is still unknown, and we have no good method of measuring it. In addition, the

density of an oven-cured thin resin film may not be the same as the density of the cured resin in wood. Nevertheless, the porosity of the cured PF resin was considered to be negligible in this study. One thing worth noting is that not knowing the actual density of the cured resin in wood makes it impossible to calculate the amount of penetrated resin using the measured change in pore volume.

Figure 4 shows a typical semi-log plot of the pore diameter (μm) versus the incremental intrusion volume (mL/g) for the birch veneer samples cured in the oven. The birch specimens under different curing conditions had similar curves with two distinctive regions: a large peak region in pore size between $0.5 \mu\text{m}$ and $3 \mu\text{m}$, and another region composed of many peaks in pore size between $5 \mu\text{m}$ and $110 \mu\text{m}$. The latter region may be corresponding to the lumen and vessel. Smook (1992) reported that the lumen diameter of birch is in the range of $14\text{--}28 \mu\text{m}$. Table 3 shows the distribution of the incremental intrusion volume (mL/g) of the birch specimens in three size ranges.

The aspen specimens with and without resin cured under different conditions also exhibited two distinctive regions in the plot of pore diameter versus the incremental intrusion volume. Table 4 shows the distribution of the incremental intrusion volume (mL/g) of the aspen samples in the same three size ranges.

It is clear from Tables 3 and 4 that the total pore volume is consistently smaller for specimens with resin for both species under all conditions. In order to take natural variability within the wood pore structure into account, five replications of each test condition were conducted.

TABLE 3. The distribution of the incremental intrusion volume (mL/g) for the birch samples.

Diameter (μm)	Incremental intrusion volume of birch samples (mL/g)											
	Oven-curing				Core-hot press				Face-hot press			
	Birch		Birch/LPF		Birch		Birch/LPF		Birch		Birch/LPF	
	Vol.	STDEV	Vol.	STDEV	Vol.	STDEV	Vol.	STDEV	Vol.	STDEV	Vol.	STDEV
$d \geq 3$	0.4399	0.0076	0.3422	0.0183	0.3058	0.0114	0.2032	0.019	0.1281	0.0136	0.0891	0.0085
$0.5 \leq d < 3$	0.4060	0.0406	0.3916	0.0407	0.3346	0.0114	0.3774	0.004	0.2725	0.0165	0.2080	0.0214
$d < 0.5$	0.0105	0.0012	0.0164	0.0030	0.0257	0.0059	0.0380	0.008	0.0339	0.0071	0.0550	0.0111
Total	0.8564	0.0643	0.7502	0.0654	0.6661	0.0357	0.6187	0.022	0.4345	0.0340	0.3521	0.0146

TABLE 4. The distribution of the incremental intrusion volume (mL/g) for the aspen samples.

Diameter (μm)	Incremental intrusion volume of aspen samples (mL/g)											
	Oven-curing				Core-hot press				Face-hot press			
	Aspen		Aspen/LPF		Aspen		Aspen/LPF		Aspen		Aspen/LPF	
	Vol.	STDEV	Vol.	STDEV	Vol.	STDEV	Vol.	STDEV	Vol.	STDEV	Vol.	STDEV
$d \geq 3$	0.6612	0.0481	0.5142	0.0357	0.2070	0.0224	0.1419	0.0173	0.0477	0.0102	0.0396	0.0077
$0.5 \leq d < 3$	0.6234	0.0440	0.6633	0.0045	0.6388	0.0048	0.6124	0.0232	0.2548	0.0189	0.2271	0.0097
$d < 0.5$	0.0244	0.0316	0.0126	0.0282	0.0214	0.0061	0.0241	0.0123	0.0969	0.0201	0.0960	0.0291
Total	1.3090	0.0962	1.1900	0.0587	0.8672	0.0476	0.7783	0.0896	0.3993	0.0566	0.3627	0.0503

Results of the five replications of the birch and aspen samples with and without LPF resin cured in the oven are given here as an example (Figs. 4 and 5). The pore size distribution curves indicated that the test results obtained by the mercury intrusion porosimeter were fairly consistent within each condition.

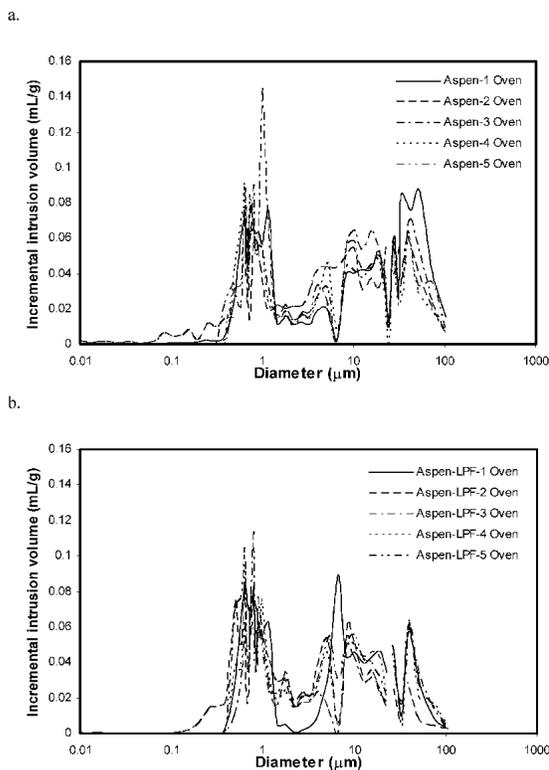


FIG. 5. Five replications of the aspen samples with and without LPF resin cured in the oven. a. Five replications of the aspen samples cured in oven. b. Five replications of the aspen/LPF samples cured in oven.

Resin penetration without external pressure (oven)

Results for the birch and aspen specimens with and without resin cured in different conditions are given in Figs. 6 and 7, respectively. Compared with the specimens without resin, the pore volume of the specimens with the resin application decreased for all cases. In the case of oven-cured birch/LPF specimens, a main part of pore volume change came from the large pore fraction (diameter $\geq 40 \mu\text{m}$). When the pore diameter was greater than $40 \mu\text{m}$, the difference in the incremental intrusion volume between the

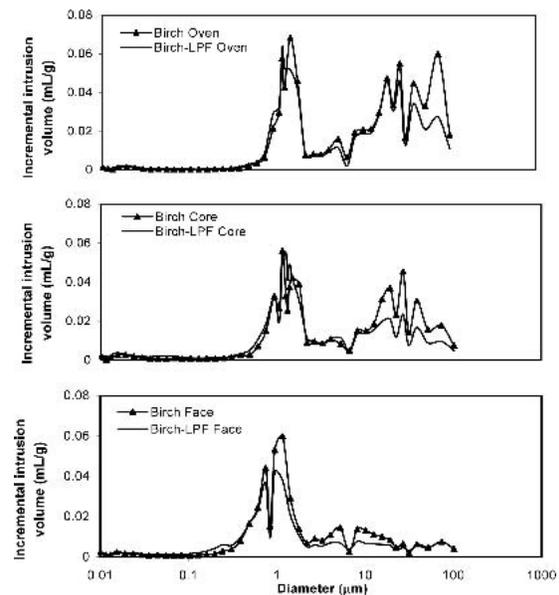


FIG. 6. Comparison of the incremental intrusion volume of the birch/LPF samples cured in different conditions. The plot is based on an average of five replications.

birch and birch/LPF specimens was 58% of the total difference in the intrusion volume (Figs. 6 and 8). For the oven-cured aspen/LPF specimens, the large part of the difference in the incremental intrusion volume was for pore size greater than 10 μm (Figs. 7 and 9). These calculations indicated that in the oven-cured condition, resin penetrated mainly into the larger size pores for both the birch and aspen samples. In addition, resin also penetrated into the pores of sizes between 1 μm and 3 μm for the birch/LPF specimens (Fig. 8). There is no evidence to show that resin penetrated into the pores of sizes smaller than 1 μm for both the birch and aspen samples. Resin may have penetrated into these submicron pores, but the amount may have been too small to be distinguished by the mercury intrusion technique.

Resin penetration under external pressure (hot-press)

Wood composites are typically made by hot-pressing. Wood materials experience different

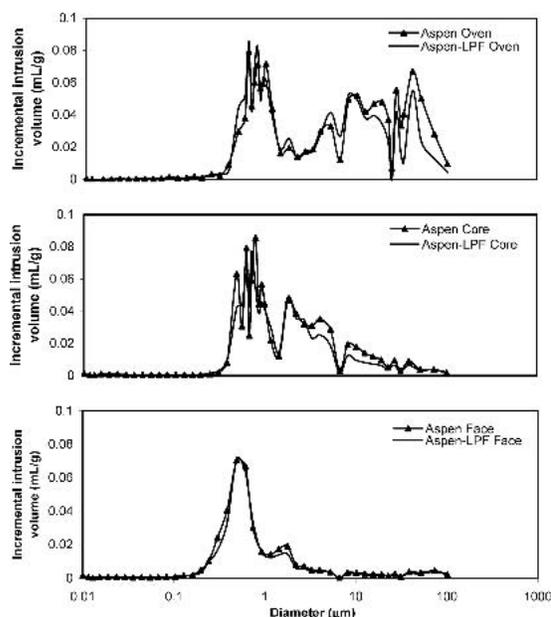


FIG. 7. Comparison of the incremental intrusion volume of the aspen/LPF samples cured in different conditions. The plot is based on an average of five replications.

temperature, moisture, and pressure profiles depending on the location in the mat: face vs. core (Fig. 2).

Cured in the hot-press core position.—In the case of core-cured birch/LPF samples, a main part of pore volume change for the birch specimen with and without resin came from the pores in the size range between 10 and 40 μm (Figs. 6 and 8). For the core-cured aspen/LPF samples, the difference in the incremental intrusion volume was mostly in the size range of 3 ~ 40 μm , which gave 70% of the total intrusion volume difference (Figs. 7 and 9). When the pore size was in the range of 3 ~ 10 μm , the difference in the incremental intrusion volume between the aspen and aspen/LPF samples was 40% of the total difference in the intrusion volume. Compared with the oven-cured specimens, these calculations indicated that resin penetrated into the smaller pores when the external pressure was applied.

Cured in the hot-press face position.—Compared with the specimens cured in the oven and cured in the hot-press at the core position, the majority part of the pore volume change of the face-cured samples came from the smaller pores (diameter <3 μm) (Figs. 6, 7, 8, and 9). When the pore size was in the range of 0.5 ~ 3 μm , the difference in the incremental intrusion volume between the birch and birch/LPF samples was

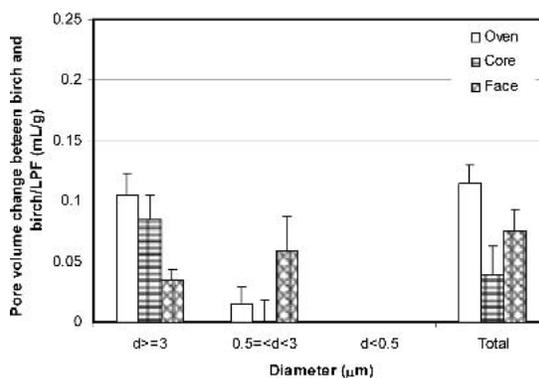


FIG. 8. Comparison of the pore volume change (reduction) between the birch and birch/LPF samples cured in different curing conditions. The pore volume change between the birch and birch/LPF samples was normalized to a resin content of 5%.

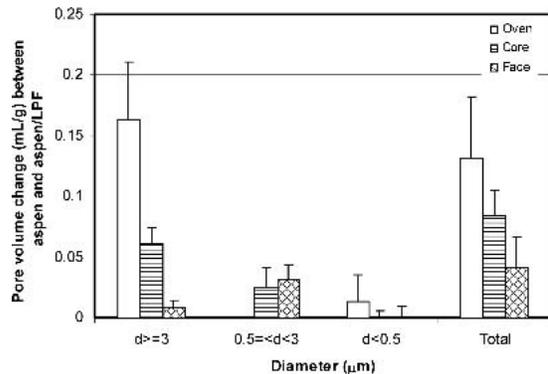


FIG. 9. Comparison of the pore volume change (reduction) between the aspen and aspen/LPF samples cured in different curing conditions. The pore volume change between the aspen and aspen/LPF samples was normalized to a resin content of 5%.

78% of the total intrusion volume difference (Fig. 8). Similar observation is true for the face-cured aspen/LPF samples. When the pore size was greater than 3 μm , the difference in the incremental intrusion volume between the aspen and aspen/LPF samples was 22% of the total intrusion volume difference. The difference in the incremental intrusion volume in the pore size range of 0.5 ~ 3 μm was 75% of the total intrusion volume difference (Fig. 9). These calculations suggested that under the face-cure condition resin penetrates mainly into the smaller pores.

The cumulative intrusion volume of the face-cured birch/LPF specimens was 17.37% less than the cumulative intrusion volume of the face-cured birch alone specimens. The cumulative intrusion volume of the core-cured birch/LPF specimens reduced 5.88% from that of the core-cured birch specimen without resin. Interestingly, the cumulative intrusion volume of the oven-cured birch/LPF specimens was 13.33% lower than that of the oven-cured birch samples (Table 5). The curing conditions didn't influence significantly the change in the cumulative intrusion volume for the aspen/LPF specimens. For both the birch/LPF and aspen/LPF specimens, resin had penetrated into small pores ($d < 1 \mu\text{m}$) under the hot-press curing conditions, implying that external pressure favored resin penetration into the smaller pores.

TABLE 5. Comparison of the reduction in the cumulative intrusion volume for birch/LPF and aspen/LPF specimens. The data were normalized to a 5% resin content.

	Cumulative intrusion volume change (%)		
	Oven	Core	Face
Birch/LPF	13.33	5.88	17.37
Aspen/LPF	10.08	9.69	10.30

CONCLUSIONS

Based on the difference in wood pore volume for specimens with and without resin under the same curing conditions, the mercury intrusion porosimeter can be used to measure the degree of resin penetration. For both the birch and aspen species, liquid resin penetrated mainly into the big pores ($\geq 40 \mu\text{m}$ for the birch/LPF and $\geq 10 \mu\text{m}$ for the aspen/LPF) when specimens were cured in the oven. Resin also penetrated into the small pores ($1 \mu\text{m} < \text{pore size} < 3 \mu\text{m}$) in the oven-cured condition. There is no evidence to show that resin can penetrate into the pores below 1 μm for the oven-cured birch/LPF specimens. However, in the case of the oven-cured aspen/LPF specimens, resin can penetrate into pores below 1 μm in size.

Under the hot-press curing condition, resin penetrated into the small pores. In the case of the birch/LPF samples cured in the hot-press at the core position, resin penetrated mainly into the pores of sizes between 10 and 40 μm . For the core-cured aspen/LPF samples, resin greatly filled in the pores of size range of 3 ~ 40 μm . When both the birch/LPF and the aspen/LPF specimens were cured in the hot-press at the face position, resin penetrated mostly in the range of 0.5 to 3 μm . Resin can penetrate into the small pores (diameter $< 0.5 \mu\text{m}$) for the face-cured aspen/LPF specimens, which is not true for the face-cured birch/LPF specimens. In all cases, some resin may have filled in the bigger pores partially, resulting in an increase in the smaller pore-fraction, which can confound some of the findings.

In addition to pressure, samples that were cured in the hot-press had different temperature and moisture profiles from the ones that were cured in the oven. Therefore, temperature and

moisture may have also played a role in affecting the resin penetration.

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