DEFORMATION OF WOOD-BASED MATERIAL DURING SUPERCRITICAL CARBON DIOXIDE TREATMENT

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ABSTRACT

The deformation of various wood-based materials during supercritical carbon dioxide treatment was assessed in situ at a range of pressurization and venting rates. Deformation was minimal with oriented strandboard (OSB), medium density fiberboard (MDF), and solid Douglas-fir heartwood, and even this slight deformation was rapidly recovered once the pressure was released. Higher degrees of deformation were observed in laminated veneer lumber (LVL) composed of Douglas-fir veneers and this deformation was not completely recovered at the end of the process. The resulting deformation resulted in permanent veneer separations. The results indicate that there is little risk of damage during supercritical carbon dioxide treatment of OSB, MDF, and Douglas-fir heartwood, but that further process studies will be required to identify treatment cycles suitable for treatment of LVL.

Keywords: Supercritical fluids, OSB (oriented strandboard), medium density fiberboard, laminated veneer lumber, deformation.

INTRODUCTION

The use of supercritical fluids as possible carriers has tremendous potential for delivering biocides, stabilizers, and other compounds into wood-based materials (Acda et al. 2001; Bernburg and Krukonis 1991; Henriksen 2000a,b; Ito et al. 1984; Kayihan 1992; Kiran 1995; Li and Kiran 1988; Morrell et al. 1993; Ritter and Campbell 1986, 1991; Sahle Demessie et al. 1995, 1998; Smith et al. 1993a,b; Ward 1989). Supercritical fluids (SCFs) have diffusivities similar to gases and some have solvating properties that approach those of liquids (Clifford 1998; Debenedetti and Reid 1986; DeFilippi 1982; Hoyer 1985; McHugh and Krukonis 1994). This combination offers the potential for SCFs to be used to completely impregnate a variety of materials that currently defy effective treatment. Paramount among these are the diverse array of wood-based composites. Although these materials are generally easily treated, the resulting treatments can produce unacceptable swelling or deformation or leave residual solvents that limit potential uses.

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Although SCFs offer tremendous potential for composite treatment, many potential users of this technology have expressed concerns about the potential effects of elevated pressure on panel properties. Acda et al. (1997a,b) found that SC CO_2 could be used to impregnate a number of composites with the biocide tebuconazole (Bayer Inc., Pittsburgh, PA) with little or no effect on panel properties; however, he did note panel densification with some specimens, which implied the presence of differential pressure gradients at some point in the treatment process. Tsunoda and Muin (2003) noted slight losses in mechanical properties of oriented strandboard following SCF treatment, but gave no explanation for the losses.

The development of pressure gradients during impregnation is clearly not confined to SCF processes; there are numerous reports describing pressure development during conventional liquid impregnation (Cobham and Vinden 1995; Peek and Goetsch 1990; Schneider and Morrell 1997). The key feature to developing such gradients is ensuring that the total pressure differential does not exceed any material properties of the wood. Pressure differences can have significant effects on wood if the gradients exceed the material properties (Walters 1967; Walters and Whittington 1970; Willeitner and Murphy 1987; Yashiro and Takahashi 1996; Smith et al. 1993a,b). Although it is clear that process conditions can be varied to limit the development of pressure gradients, there are few data on when the gradients develop or when deformation occurs.

Kim and Morrell (2000) studied deformation in white spruce during SCF impregnation and found that rapid increases in pressure produced the most dramatic deformation, while rapid venting at the conclusion of the process produced the lowest level of permanent displacement. These results suggest that process conditions can be varied to limit treatment effects on solid wood, but there are few data on deformation of other wood products during treatment. In this report, we compare the deformation of several wood composites with Douglas-fir heartwood lumber during treatment using SC CO_2.

MATERIALS AND METHODS

Oriented strandboard composed of aspen (OSB) and medium density fiber board (MDF) were obtained from a local building supply center. Laminated veneer lumber (LVL) was composed of 9 plies of Douglas-fir veneer. The sample specifications are shown in Table 1. In addition, Douglas-fir heartwood lumber was obtained from a lumberyard in western Oregon. The OSB and MDF panels were sealed on three edges with a two-part epoxy to limit penetration to the wide faces and one narrow face, thereby simulating the penetration process in standard size panels. Penetration near the edge of a composite panel should be dominated by parallel flow due to the higher permeability parallel to the panel surface. Transverse flow becomes increasingly important at greater distances from the edge until parallel flow ceases to contribute to panel penetration. Strain gauge transducers were placed 10, 158.5, or 307 mm from the unsealed edge of a panel to detect differences in deformation.

LVL and Douglas-fir were epoxy sealed on selected faces to produce flow in all directions (no sealing), sealed on the faces and radial edges to encourage longitudinal flow, or sealed on the faces and cross sections to encourage radial flow. All materials were conditioned at 65% relative humidity and 23°C until tested. The measuring points on LVL and Douglas-fir were similar to those on the panel products. For each pressing and venting rate, samples were completely unsealed, sealed on the face to prevent longitudinal flow, sealed on the face and the two

TABLE 1. *Specifications for materials used for deformation measurements.*[a]

Material	Average density (kg/m^3)	Number of specimens	Thickness (mm)	Width (mm)	Length (mm)
OSB	643. 2 (42. 5)	110	10. 9	76	317
MDF	798. 8 (22. 9)	24	11	76	317
LVL	583. 0 (19. 3)	24	50	50	320
Douglas-fir	596. 3 (71. 9)	7	50	50	320

[a] Numbers in parentheses represent 1 standard deviation.

sides perpendicular to the orientation of the lamellae, or sealed on the face and the two sides parallel to the orientation of the lamellae. All flow directions or the radial or the tangential directions were open in some Douglas-fir samples. Small brass squares (approximately 5 by 5 mm) were glued to the wood or panel surface to ensure proper mounting and positioning of the measuring devices. Dimples at centers of these brass squares allowed the sensor tips to seat in a predetermined position.

The strain-measuring device was based upon previous trials under supercritical conditions (Kim and Morrell 2000). Strain gauges are used in a wide array of applications (Walters and Huang 1971; Loferski 1989; Link et al. 1998). The basic design of the so-called dendrometer, which consists of two aluminum bars and a flexible steel band carrying four bonded strain gauges, was readily adapted to our studies.

Two different types of transducers were built for the measurements on OSB and MDF samples or the LVL and Douglas-fir samples. The gauges were tailored to the relative degree of change of the four materials. A strain-measuring device consisting of two aluminum rods and a steel spring was bolted onto the rods. All aluminum parts were CNC-machined (Griffo Brothers Ironmongerwerks, Corvallis, OR). Stainless steel springs measured 27 mm in length, 13 mm in width and 0.71 mm in thickness for the sensors used on OSB and MDF samples. High elongation constantan strain gauges EA-06-125AC-350 or EP-08-250B6-120 (Measurements Group Inc., Raleigh, NC) were placed on either side of the spring (Table 2).

The gauges were bonded onto stainless steel springs measuring 65.4 mm in length, 13 mm in width, and 0.81 mm in thickness. The strain gauges were connected in a half bridge circuit. Upon completion of the soldering, the transducers were tested for proper installation using a 1300 Gage Installation Tester (Measurements Group Inc. 1979, 1986) to ensure that the gauges were correctly installed.

A micrometer was used to calibrate each transducer. One transducer exhibited a linear relationship between imposed deformation and output in either direction, i.e., compression and tension. However, final calibration for all transducers was performed by applying tensile force at the sensor tips, since the transducers would only be operated in this mode under experimental conditions (Fig. 1). The difference between the actual and the measured deformation was plotted as "Error [mm]" and was well within the targeted accuracy of 1% of the maximum deflection. An inaccuracy in the calibration instrument caused a wave-like deviation that can be seen in both "Error"-curves and was repeated with every full rotation of the micrometer screw (0.635 mm).

Strain gauge transducers employed for each treatment consisted of three active transducers on wood samples and one passive reference transducer. The reference transducer was placed inside the treatment vessel to compensate for changes in resistance caused by temperature changes. A high-pressure feedthrough (Connex Buffalo Technologies, Buffalo, NJ) was used to feed wires from

TABLE 2. *Properties for strain gages used for deformation measurements on OSB and MDF samples.*

	OSB/MDF	LVL/Douglas-fir
Property	EA-06-125AC-350	EP-08-250B6-120
Resistance [Ω] at 24°C	350 ± 0.15%	120 ± 0.15%
Gage factor at 24°C	2. 090 ± 0.50%	2. 055 ± 0.50%
Transverse sensitivity at 24°	(0.7 ± 0.2)%	0. 9 ± 0.2%
Temperature range	–75°C to 175° C	–45°C to 95°C
Strain limits	3%	20%

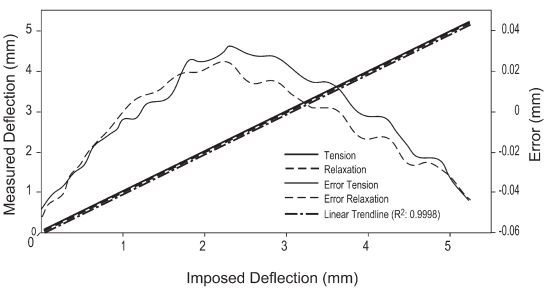

FIG. 1. Typical calibration curve for a strain gauge transducer also giving the deviation between the imposed and the measured deflections.

inside the treatment vessel to the data-acquisition system. An external 5V power source was used (HC 5–6/0 VP-A, Power-One, Camarillo, CA), since the data-logger could not provide the required current level for the transducers.

To ensure proper and safe mounting in the treatment vessel, the wood material with strain gauge transducers was mounted in one of two steel sample-holders that accommodated the thinner OSB or MDF panel samples or the LVL- and Douglas-fir samples (Fig. 2). The sample-holder for the panel products provided space for two additional samples, placed on either side of the deformation sample.

Initial measurements on OSB samples and subsequent calibration runs on stainless steel samples under typical treatment conditions revealed that the output on the transducers changed, although no deformation had occurred.

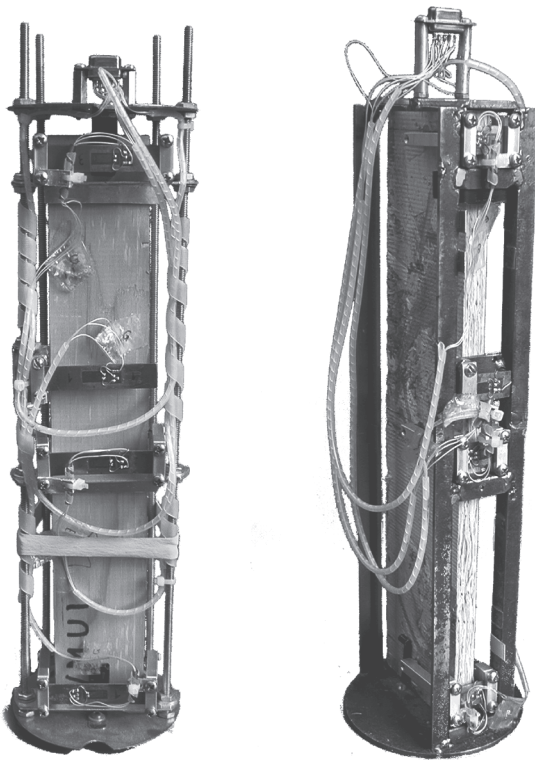

FIG. 2. Sample holders for LVL and Douglas-fir samples (to the left) and for OSB and MDF samples (to the right).

The transducers employed by Kim and Morrell (2000) for measurements on spruce exhibited similar behavior (Kim, unpublished notes). Carbon dioxide treatment at elevated pressures appears to cause changes in transducer output. The arch-like geometry of Kim's transducers meant that equal deformations translated into bigger changes in strain gauge resistance than we found with our transducer.

Several phenomena may account for the observed changes. Carbon dioxide can diffuse into and subsequently swell polymers. Several sources indicate that polyimides, which served as the strain gauge backing material may also be susceptible to CO_2 swelling (Cooper 2000; Shieh et al. 1996a,b; Berens et al. 1989; Perman et al. 1996). Although Boggess et al. (1997) found lower rates of CO_2 absorption than initially expected in their study on silver infusion into polyimide films, their trials confirmed that polyimides undergo the same changes as many other glassy polymers when exposed to pressurized CO_2. Although rising CO_2 pressure at low levels causes a linear increase in sorption in glassy polymers, which levels off at higher pressures (Zhang 1996), no quantitative data are available on the resulting increase in volume. Carbon dioxide may have also interacted with the epoxy adhesive used to bond the strain gauges to the steel springs, resulting in a swelling of the adhesive, elongating the grid material. Micro-bubbles in the adhesive layer could also be compressed and infused by the CO_2 during the pressure treatment, which again would induce a stress on the grid material. The latter two possibilities could also contribute to the variation in magnitude of the pressure-induced output change.

To confirm the observations made on OSB panels, two additional strain gauge transducers were used to measure dimensional changes in width and length (Kim and Morrell 2000).

Process conditions

The strain gauges were used to monitor deformation under varying pressurization and venting regimes using a supercritical fluid impregnation device in which carbon dioxide flowed through the treatment vessel. Each cycle consisted of the

introduction of liquid CO_2 to the treatment vessel at rates designed to produce pressurization rates of 1.03, 1.79, and 5.86 MPa/min until the desired maximum pressure of 9 MPa was reached. Pressure was held at the maximum for 30 min, then CO_2 was vented at either 1.03, 1.79, or 5.86 MPa/min to atmospheric pressure. Each pressurization/venting combination was evaluated on at least two specimens in separate treatment trials.

Data analysis

Data (as deformation and vessel pressure) were continuously monitored over a given treatment condition. These data were plotted and compared for each material tested.

RESULTS AND DISCUSSION

Deformation of both OSB and Douglas-fir samples was small enough to fall within the error range of the strain gauges themselves (data not shown). OSB is generally regarded as having numerous openings that allow for fluid flow, and internal pressure measurements inside OSB panels during SCF treatment indicate that internal pressure equilibrated rapidly with surface pressure. The absence of substantial deformation corresponds well with the internal pressure response and indicates that SCF impregnation of OSB should not affect panel properties.

Douglas-fir is generally regarded as a relatively impermeable material that resists traditional liquid penetration. Previous internal pressure measurements indicate that pressure in the interior lags behind the surface in this species, suggesting that there is some possibility for surface deformation (Schneider 1999). The absence of substantial deformation on the test specimens suggests that the pressure differential did not exceed the shear strength or resistance to crushing of the wood. As a result, SCF impregnation of this material, while likely to generate some surface to interior pressure gradients, should not adversely affect wood properties. These results confirm bending tests on SC carbon dioxide treated Douglas-fir heartwood beams (Anderson et al. 2000).

Pressurization

Deformation measurements of MDF, OSB, and LVL under supercritical conditions were affected by temperature changes as well as pressure. These effects could not be easily distinguished from actual deformation, making interpretation of the data at the lower output range difficult. Although pressure and especially temperature effects could be identified very clearly in several trials, the various effects could not be partitioned.

Very slight deformation expansion in MDF, ranging from 0.11 to 0.15 mm, occurred during pressurization, at levels near the limits of the sensitivity of the measurement technique. This deformation disappeared when pressure was released (Fig. 3). Calibration tests using stainless steel samples produced similarly shaped curves, which reached maximum values of about 0.05 mm. Consequently, our results suggest corrected increases in panel thickness of 0.06 to 0.11 mm (0.55% to 1%) of original panel thickness during treatment. Swelling of wood or wood-based materials due to CO_2 adsorption onto cellulose might help to explain the dimensional changes, although this conflicts with the concept that SCFs are non-swelling treatments.

Once pressure was released, the CO_2 would desorb and the materials would return to their original dimensions. Supercritical fluid treatment of four refractory wood species led to increases in cross-sectional area on many samples, which was explained by a recovery mechanism of collapsed cells (Anderson et al. 2000). Acda et al. (1997a) reported that SCF treatment of four types of wood composites produced no significant changes in thickness. Our results indicated that detecting dimensional changes would be rather difficult, since potential swelling disappeared after depressurization. Further in situ measurements with more sensitive transducers may be required to elucidate the possible interactions of gaseous and supercritical CO_2 with lignocellulosic materials.

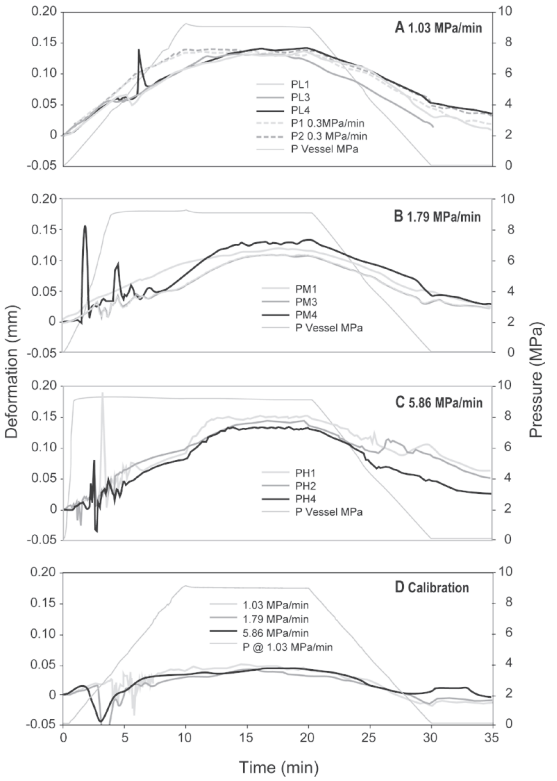

FIG. 3. Average deformation in MDF and on stainless steel calibration samples during pressurization and venting at (A) 1.03 (and 0.3), (B) 1.79, (C) 5.86, and (D) a calibration run at 1.79 MPa/min using non-wood samples as the controls.

No compression occurred on LVL samples during pressurization when a flow path other than the vertical pathway was left unsealed. The samples expanded and their deformation plots resembled those for MDF; however, variability was higher with LVL and thickness increases ranged from 0.2% to 0.4% of the original dimension. The thickness of 10 samples that had not suffered any apparent damage increased by an average of 0.075 mm (± 0.025 mm), suggesting that this material behaved more like solid wood than did MDF or OSB.

Very high pressure gradients and crushing in LVL samples, with flow restricted to the vertical direction, indicated that considerable forces had acted on these samples during SCF treatment; substantial displacements in LVL samples confirmed this effect (Fig. 4). Pressurization of these samples resembled a compression test (Fig. 5). Pressure versus deformation plots showed that initial elastic deformation was followed by plastic deformation. Results from two pressurization rates resembled the results of static compression tests (Fig. 6) over a wide deformation range. Sealant failure occurred as vessel pressure reached approximately 7 MPa; then pressure gradients equilibrated across the samples, and the associated compressive forces decreased. Values above this pressure were probably not representative of a stress-strain relationship.

A distinct rebounding effect was observed as pressure equilibrated (thin lines in Fig. 5). This effect was also noted during displacement measurements on white spruce lumber (Kim and Morrell 2000). Full recovery of deformation on the specimen pressurized at 1.03 MPa/min was achieved by venting the treatment vessel at the

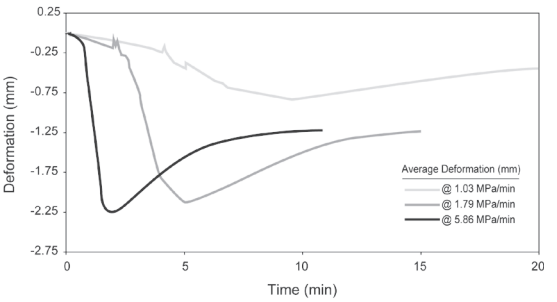

FIG. 4. Average deformation of LVL samples during pressurization at 1.03, 1.79, and 5.86 MPa/min with flow restricted to the vertical direction.

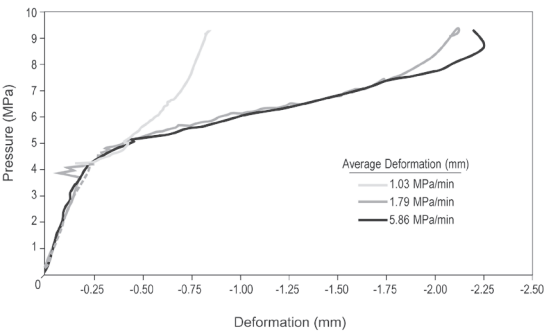

FIG. 5. Pressure and average deformation in LVL samples during pressurization at different rates.

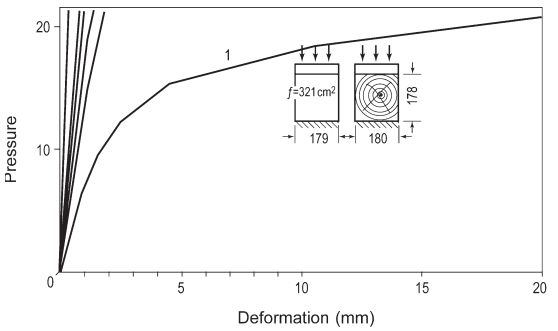

FIG. 6. Pressure and deformation in wood tested in compression perpendicular to the grain direction (Source: Kollmann 1951).

same rate. Pressurization and venting at 1.79 MPa/min did not produce full recovery of deformation, and the least recovery resulted when samples were pressurized at 5.86 MPa/min and vented at 0.35 MPa/min. This rate was chosen to protect the transducers from potential damage. The observation that higher venting rates produced better recovery of deformation supports previous work (Kim and Morrell 2000).

The deformed LVL specimens exhibited an hour-glass-like cross-section, indicating that the specimens experienced stronger compression in the flow direction than in the portions closer to the panel surfaces. Limited observations suggested that compression failures were mainly found in plies with higher percentages of earlywood. In contrast to LVL, no severe displacements were measured on Douglas-fir heartwood samples treated at the different pressurization rates. The differences between Douglas- fir lumber and LVL samples may reflect interference by gluelines.

Venting

Deformation was absent on OSB and MDF samples, and any expansion that developed during pressurization disappeared during venting, suggesting that rapid venting did not negatively affect either material (Fig. 3). Thus, venting rate could be adjusted to optimize the precipitation process with less concern about potential effects on material properties.

Although pressure gradients exceeded 1.2 MPa in only a few internal pressure measurements, several LVL samples were damaged during venting, suggesting that gradients exceeded the tensile strength of the LVL (2.1 to 2.4 MPa) (U.S. Department of Agriculture 1999). Deformation in samples sealed to restrict flow to the longitudinal direction was expected and occurred during venting at all three rates (Fig. 7). Internal forces during venting at 1.79 and 5.86 MPa/min caused steep initial deformation rates that peaked as maximum pressure gradients reached 1.2 and 1.9 MPa, respectively. Internal pressure gradients peaked towards the end of the venting, and deformation measurements supported the presence of elevated internal strain (Fig. 8). All specimens exhibited severe damage in the form of splits along two or more plies.

Splitting of unsealed samples was unexpected since internal sensors failed to detect elevated

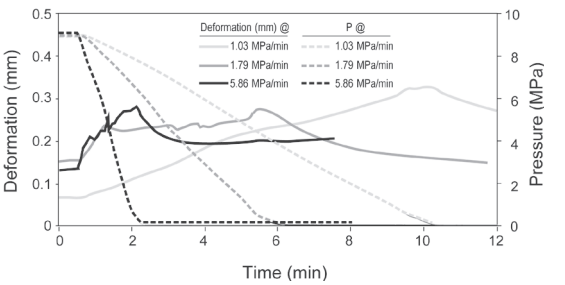

FIG. 7. Average deformation in LVL specimens with flow restricted to the vertical direction during ventilation at three different rates.

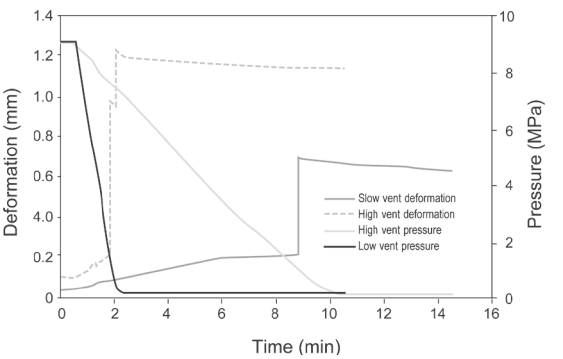

FIG. 8. Deformation of unsealed LVL samples during venting at 1.03 (VL) and 5.86 MPa/min (VH).

pressure gradients. However, the strength properties of Douglas-fir veneer may have been lower than reported for lumber, or gaps or knots may have resulted in localized stress concentrations where failures initiated. Our measurements indicated that flow of gaseous and supercritical CO_2 through LVL led to substantial pressure gradients with associated deformation and mechanical damage. LVL proved to be more vulnerable during venting than pressurization, probably as a consequence of the lower strength in tension than in compression. Decreasing the venting rate towards the end of depressurization may reduce the development of pressure gradients and minimize potential damage.

CONCLUSIONS

Deformation and the development of pressure gradients in OSB, MDF, and Douglas-fir heartwood during SCF treatment were minimal. LVL experienced permanent deformation and pressure differentials during treatment. The results suggest that most composites are suitable for SCF treatment, while further trials will be necessary with LVL to develop suitable schedules for this material.

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