INTERNAL PRESSURE DEVELOPMENT DURING SUPERCRITICAL FLUID IMPREGNATION OF WOOD

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ABSTRACT

Supercritical fluid impregnation has tremendous potential for effectively impregnating a variety of species, but little is known about the pressure response in wood during this process. Pressure response was studied in a number of wood species using specially designed high pressure probes, which allowed in-situ monitoring of the treatment process. Pressure response was relatively rapid in permeable species such as pine, but tended to lag in less permeable species. In some cases, the differences between surface and internal pressure exceeded the material properties of the wood, and crushing or fractures resulted. The results indicate that the rates of pressure application and release can be tailored to control pressure differentials to avoid wood damage.

Keywords: Douglas-fir, ponderosa pine, white fir, sweetgum, Pacific silver fir, supercritical fluids, pressure.

INTRODUCTION

Supercritical fluid impregnation (SCF) offers tremendous potential for effectively treating a variety of wood-based materials with biocides or other modifiers. Numerous studies have shown that complete biocide impregnation can be achieved on an array of materials that are typically ranked as extremely difficult to treat using conventional liquid treatment processes (Acda 1995; Acda et al. 2001; Ito et al. 1984; Kayihan 1992; Kim et al. 1997; Kim and Morrell 2000; Sahle-Demessie 1994; Sahle-Demessie et al. 1995a,b; Smith et al. 1993a,b; Tsunoda et al. 1999; Tsunoda and Muin 2003; Kang and Morrell 2003). Many of these tests have used smaller specimens that allowed relatively rapid ingress of treatment fluid. One aspect of treatment of larger specimens that must be considered is the development of internal pressure gradients (Kim and Morrell 2000; Anderson et al. 2000; Anderson 1998; Walters 1967; Walters and Whittington 1970). Pressure gradients are of little concern unless they exceed the material properties of the wood. In these instances, the wood can either collapse if external pressure exceeds the compressive strength or fracture if internal pres-

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sure exceeds the tensile strength perpendicular to the grain.

An important aspect of SCF process development is to ensure that treatment conditions do not result in excessive internal pressure gradients. One approach to this process development is to assess the relationships between treatment and internal pressure, then use this knowledge to prevent excessive pressure levels. In this report, we assess internal pressure development and make comparisons between different wood species, grain orientations, sample size, and pressing and venting rates during supercritical carbon dioxide treatment.

MATERIALS AND METHODS

Equipment

The high pressure equipment in this investigation consisted of an electronic instrument control cabinet, CO_2 source, gas compressor, back-pressure regulator, treatment vessels, vessel heaters, heating tapes, tubing, metering valves, and ball valves (Fig. 1). Standard grade carbon dioxide was purchased from Industrial Welding Supply Inc. in 23-kg gas cylinders. A single-stage diaphragm compressor (Fluitron Model A1-400) was used to move CO_2 from the gas cylinders to a high pressure storage vessel. Pressure in the storage vessel was controlled using a back-pressure regulator (Tescom Model 26-1722-24). Two metering valves, having flow coefficients of 0 to 0.04 and 0 to 0.37, were used to control CO_2 flow to the treating vessel from the storage vessel and from the treating vessel to outside the building during venting. The cylindrical storage and treating vessels, supplied by High Pressure Equipment Co., Inc., had inside diameters of 150 and 100 mm, respectively, and lengths of 600 mm. The temperature of each vessel was controlled through a cascading loop. A slave controller, (West Model 2072), measured the vessel thermal well temperature and sent a signal to the master controller (West Model 3100). The master controller was used to moni-

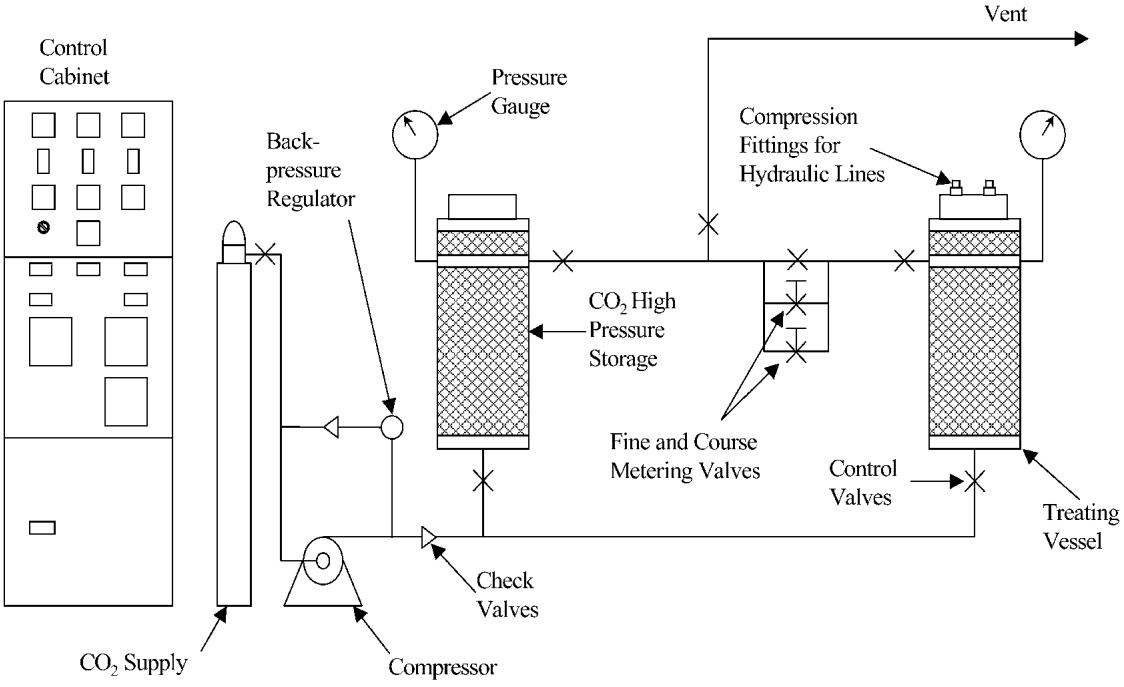

FIG. 1. Schematic of the supercritical fluid pilot plant used to study pressure response during supercritical CO_2 impregnation of wood.

tor vessel surface temperature. If both controllers called for heat, the master controller turned on a heating blanket surrounding the pressure vessel. The stainless steel tubing connecting the two vessels and in the vent lines was wrapped with heating tape to maintain the desired temperature during fluid transfer and avoid clogging during venting.

Temperature inside the heating vessel was measured with a Type K thermocouple. Thermocouples made from 24 gauge Type K thermocouple wires have a response time of about 3.3 s and an error limit of 2.2°C (OMEGA Engineering). Temperature readings were measured as thermocouple potential in millivolts using a Campbell 21x data logger and were stored in a personal computer.

Pressure measurements were made using OMEGA PX 420-5K GI pressure transmitters with error ranges of 0.5% of full-scale readings. The transducers were individually calibrated using a Heise test gauge with a pressure range of atmospheric to 41,368 MPa, divided into 35 kPa divisions. Because the pressure sensors produce an output signal of 4 to 20 mA, a temperature stable precision resistor was placed in series with each sensor, and a Campbell 21x data logger measured the voltage across the resistor. The transmitters were then connected to a common pressure vessel and their responses compared with each other to confirm proper installation and calibration. An analog-to-digital converter meter was also placed in series with each vessel's pressure transmitter. This meter was adjusted so that its zero and span corresponded to zero and the maximum gauge pressure readings from the pressure transmitters. The digital meters were used to visualize vessel pressure during pressing and venting. As a safety precaution, analog pressure gauges were attached directly to each pressure vessel.

Because of the high pressures generated in the treating vessel, the pressure transmitters were placed on the outside of the vessel and hydraulic lines were fed through the vessel top (Fig. 2). The hydraulic line was constructed using stainless steel tubing (3.2 mm OD) and compression fittings. A tee union was placed at the highest point of the hydraulic line outside of the vessel allowing attachment of a hydraulic fluid reservoir. A union at the end of the tubing extending below the vessel top was used to attach samples by their pressure probes.

Pressure probes

A variety of methods were initially evaluated for attaching pressure probes to wood samples (Schneider et al. 2003), based upon previous tests at conventional treatment pressures (Bergman 1991; Cobham and Vinden 1995; Peek and Goetsch 1990; Orfila and Hosli 1985). The solubility of many sealant materials in supercritical carbon dioxide makes it difficult to find materials that can effectively seal probes into wood, yet withstand the pressure, temperature, and solvent conditions during treatment. In addition, the sealant must be able to move with the sample if it changes dimension during the pressing and venting phases of treatment. Gluvit Marine Epoxy (ITW Philadelphia Resins, Montgomeryville, PA) was chosen for most tests.

Probes made from stainless steel tubing (3.2 mm OD, 2.1 mm ID) were cut to 50-mm lengths, roughed with sandpaper, and cleaned with alcohol. Holes for the probes were centered in the end-grain and drilled longitudinally to near the sample center with a 3.9-mm bit. The probes were coated with epoxy and set in the holes. After the epoxy cured, a 1.9-mm drill bit was used to bore through the epoxy at the bottom of the tubing creating a 10-mm-long pressure chamber below the tubing (Fig. 3).

Assessments of internal pressure development and influential factors

Internal pressure assessment.—A single (30 × 30 × 60 mm long (R × T × L)) sample of kiln-dried yellow-poplar heartwood (*Liriodendron tulipifera* L.) was conditioned to constant weight at 23°C and 65% relative humidity and sealed with epoxy to allow only tangential flow of the treating medium. A single probe was placed at the center of the sample. The sample was then pressurized with CO_2 at a rate of 276 kPa/min to

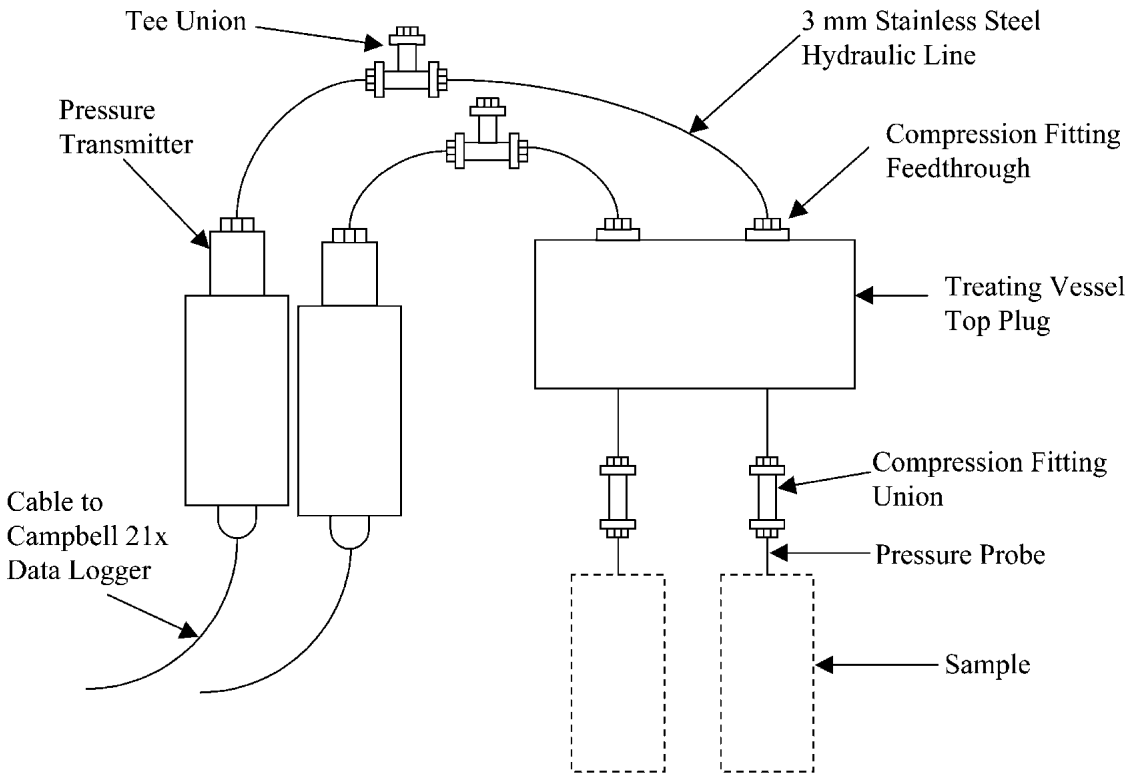

FIG. 2. Pressure transmitters and tubing to monitor pressure in wood during SFC treatments.

a maximum pressure of 10.3 MPa. Vessel pressure was maintained until pressure at the sample center equilibrated; then the vessel was vented to the atmosphere at 276 kPa/min.

Influence of wood species.—Five heartwood samples (30 × 60 × 60 mm long (R × T × L)) were cut from kiln-dried ponderosa pine (*Pinus ponderosa* Laws.), Douglas-fir (*Pseudotsuga menziesii* (Mirb.) Franco), white fir (*Abies concolor* Gord. & Glend.), and Pacific silver fir (*Abies amabilis* Dougl.) boards. The samples were sealed, allowing only radial flow, and fitted with a single pressure probe. The samples were individually pressurized with CO_2 at a rate of 276 kPa/min to a maximum pressure of 10.3 MPa. Vessel pressure was maintained until pressure at the sample center equilibrated; then the vessel was vented to atmospheric pressure at 276 kPa/min.

Influence of grain orientation.—Ten heartwood samples (30 × 60 × 60 mm long (R × T × L) were cut from kiln-dried yellow-poplar lumber. Five of the samples were sealed to allow only radial flow and five were sealed to allow only tangential flow. All were fitted with a single pressure probe. One sample from each orientation was simultaneously pressurized with CO_2 at a rate of 276 kPa/min to a maximum pressure of 10.3 MPa. Pressure was maintained until pressure at the sample centers equilibrated; then the vessel was vented to atmospheric pressure at 276 kPa/min.

Influence of sample size.—A single kiln-dried Douglas-fir heartwood board was used to cut samples measuring 30, 60, or 90 mm in the radial direction by 30 mm tangentially and 60 mm longitudinally. Each radial length was replicated 5 times. The samples were sealed to allow only radial flow of the treating medium, and single probes were placed at the sample centers. The samples were individually pressurized with CO_2 at a rate of 276 kPa/min to a maximum pressure

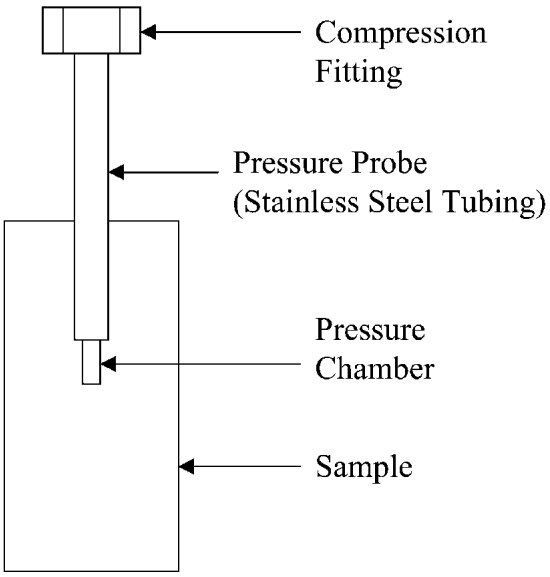

FIG. 3. Pressure probe – sample assembly to monitor pressure in wood during SC-CO_2 treatment.

of 10.3 MPa. Vessel pressure was maintained until pressure at the sample center equilibrated; then the vessel was vented to atmospheric pressure at 276 kPa/min.

Influence of pressing and venting rate.—Seventeen kiln-dried Douglas-fir heartwood samples (30 × 30 × 60 mm long (R × T × L)) were cut from a single board. The samples were sealed to allow only radial flow to the treating fluid; single probes were placed at the sample centers. The samples were individually placed in the treating vessel and pressurized at rates of 138, 276, or 827 kPa/min to the target pressure of 10.3 MPa. After internal pressure in the wood equilibrated with that of the vessel, the pressure was then vented at the same rate at which it was applied.

Confirmation of pressure differentials following venting.—In some cases, the residual pressures in Douglas-fir heartwood samples immediately after venting the treating vessel were as high as 4 MPa. Pressures this high should have caused the wood to split since this species has a tensile strength perpendicular to the grain of between 2.1 and 2.3 MPa (Markwardt and Wilson 1935; Bodig and Jayne 1982). However, most samples did not show fractures. To confirm the seemingly high residual pressures and to verify that measurement technique provided representative pressure data, pressure measurements were made on highly permeable ponderosa pine sapwood. This material was chosen because it is uniform and is very permeable; therefore, it should be immediately responsive to pressure changes. Twelve samples (30 × 30 × 60 mm long) were sealed with epoxy. Ten were allowed to have only radial flow; two were allowed to have only tangential flow. Half of the samples had pressure probes installed. One sample with a probe and another without were simultaneously pressurized with CO_2 at a rate of 690 kPa/min to 10.3 MPa, allowed to equilibrate for 10 min, then vented at the same rate. Following each treatment, the sample without the probe was immediately placed in a small pressure vessel (bomb). Pressure increases within the bomb were monitored and used to provide a measure of residual internal pressure in the wood. Residential pressure was calculated using the combined gas law for real gases where:

$$\frac{P_1V_1}{T_1Z_1} = \frac{P_2V_2}{T_2Z_2}$$

P = pressure (kPa absolute)
V = volume of vessel (cm^3) without wood volume
n = moles of gas
R = gas constant (8,314 cm^3 kPa/mole K)
T = temperature (K)
Z = gas compressibility factor
Subscripts represent conditions before ($_1$) and after ($_2$) equilibration

Carbon dioxide compressibility factors were obtained from interpolation of tabulated data (Perry and Green 1984). Because the compressibility factor is dependent on pressure, its value for gas initially in the wood was unknown. Therefore, it was estimated by calculating the approximate residual pressure in the wood assuming CO_2 to be an ideal gas. This pressure would be the maximum pressure and would not reflect any pressure gradients in the wood. The volume of gas in the wood was calculated by

multiplying the sample volume by the wood void volume factor (Siau 1984).

Data analysis

The data consisted of pressure measurements over time and are best presented graphically. However, to assist in making comparisons between treatments, characteristic measurements were made from the graphs. Times for an initial 35 kPa pressure increase and for pressure equalization and maximum surface-to-center pressure differences during pressing and immediately after venting were chosen to examine the effects of species, sample orientation, and depth on pressure response.

RESULTS AND DISCUSSION

Internal pressure assessment.—Almost 4 min passed until pressure at the center of the yellow-poplar sample reached 35 kPa as vessel pressure was increased at a rate of 276 kPa/min (Fig. 4). After this delay, internal pressure rose with that in the surrounding vessel. However, pressure was not equilibrated until 60 min after vessel pressure was held constant. Internal pressure during venting failed to keep up with decreasing vessel pressure and delays in pressure response. Delays in pressure response resulted in substantial surface-to-center pressure differences that exceeded 1 and 3 MPa during pressing and venting, respectively (Fig. 5).

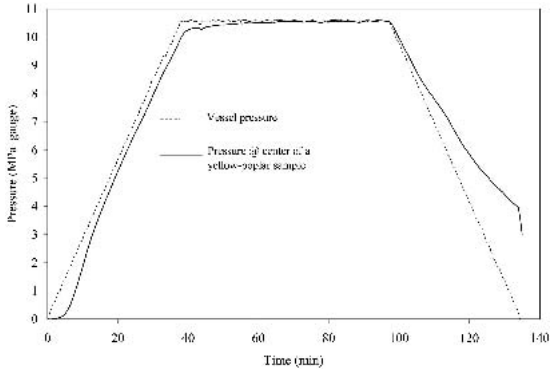

FIG. 4. Internal pressure measurements at the center of a yellow-poplar sample during SC-CO_2 treatment.

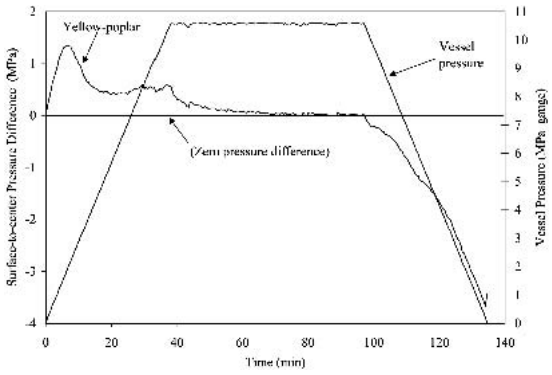

FIG. 5. Surface-to-center pressure differences during SC-CO_2 treatment of yellow-poplar.

Internal pressure response delays were due in part to permeability characteristics of the wood. In addition, the compressibility of the gaseous treating medium may cause delays in the observed pressure. The compressibility influence is likely to be greatest as pressure approaches the critical value (7.38 MPa for CO_2). The reason for this is that the density of CO_2 increases exponentially in the transition through the near critical region (Ely 1986). This phenomenon was manifested by the second increase in the surface-to-center pressure differences approximately 20 min after the initiation of pressure application (Fig. 5).

Influence of wood species.—Results from pressure measurements in the four species tested are summarized in Table 1. The amount of time to reach the initial 35 kPa and time for pressure equalization consistently increased as the permeability of the wood decreased. Markstrom and Hann (1972) list the nitrogen permeabilities of ponderosa pine, Douglas-fir, and white fir to be 0.016, 0.007, and 0.002 Darcy. No values were listed for Pacific silver fir. In addition, lower permeabilities were associated with increased maximum surface-to-center pressure differences. Wood species did not seem to influence maximum pressure differences immediately after venting.

Pressure differences exceeded the compressive strength of the Pacific silver fir causing the first two samples to collapse during the pressurization phase. Because of this, additional

TABLE 1. *Summary of internal pressure responses in wood samples from various species during SC-CO_2 treatments.*

Species	Time to reach 35 kPa (min)	Time to pressure equilibrium (min)	Max. ΔP during pressing (kPa gauge)	ΔP immediately after venting (kPa gauge)	Wood condition
Ponderosa pine	0	4.5[1]	165	−1,372	Good
	1.0	3.5[1]	400	−958	Good
	0.5	8.5	228	−2,448	Good
	1.0	5.5[1]	408	−2,703	Good
	0.5	1.5	138	−1,896	Good
Avg. (std.)	0.6 (0.4)	4.7 (2.3)	268 (115)	−1,875 (649)	
Douglas-fir	0.5	8.5	117	−2,599	Good
	0.5	2.5	172	−1,441	Good
	2.0	3.0	669	−2,523	Good
	2.0	6.0	558	−2,496	Good
	1.0	9.5	359	−1,875	Good
Avg. (std.)	1.2 (0.7)	5.9 (2.8)	375 (214)	−2,187 (454)	
White fir	6.0	80.0	1,868	−2,048[3]	Collapsed
	6.5	85.0	1,958	−524[3]	Good
	7.0	123	2,296	−324[3]	Good
	6.5	96.5	1,965	−972[3]	Good
	7.5	136	2,372	−1,407[3]	Good
Avg. (std.)	6.7 (0.5)	104 (21.7)	2,089 (204)		
Pacific silver fir	15.0	—[2]	5,971	—[2]	Collapsed
	17.5	—[2]	8,522	—[2]	Collapsed
Avg. (std.)	16.3 (1.3)		7,247 (1,276)		

[1] Samples were close to but not fully equilibrated.
[2] Data not applicable due to sample failure.
[3] Samples had pressure probe sealant failure during venting and were not included in the average.

samples were not treated. Pressure differences during venting of the more permeable white fir caused failures of the epoxy holding in the pressure probes. As a result of the sealant failures, pressure could be released and only one white fir sample collapsed.

Influence of grain orientation.—Pressure responses were more rapid in yellow-poplar samples with flow restricted to the tangential direction (Table 2). These results were different than would be expected from nitrogen permeability measurements by Choong et al. (1974),

TABLE 2. *Summary of internal pressure responses in yellow-poplar samples with the flow of CO_2 restricted to either the radial or tangential directions.*

Flow direction	Time to reach 35 kPa (min)	Time to pressure equilibrium (min)	Max. ΔP during pressing (kPa gauge)	ΔP immediately after venting (kPa gauge)	Wood condition
Radial	5.0	53.0	2,510	−3,992	Good
	4.0	50.0	2,420	−806[1]	Good
	5.0	56.5	2,806	−1,965[1]	Good
Avg. (std.)	4.7 (0.5)	53.2 (2.7)	2,579 (165)	−2,254 (1,317)	
Tangential	3.0	27.5	1,379	−3,523	Good
	1.5	18.5	951	−3,461	Good
	3.0	28.0	1,351	−3,434	Good
Avg. (std.)	2.5 (0.7)	24.5 (4.6)	1,227 (195)	−3,473 (37.3)	

[1] Samples may have had pressure probe sealant failure during venting.

who found similar permeabilities in both transverse directions, but were supported by the results of Cooper et al. (1997), who found that liquid permeability was greater in the radial direction. The faster pressure responses resulted in lower surface-to-center pressure differences. Pressure differences immediately after venting did not seem to be dependent on grain orientation.

Influence of sample size (flow path length).—Increasing the radial distance which CO_2 had to flow through Douglas-fir samples resulted in increased pressure response times and increased the surface-to-center pressure difference during pressing (Table 3). Pressure differentials immediately after venting, however, were similar. Time to reach 35 kPa and maximum pressure differences increased about three times for each doubling of the distance. Increases in equalization time were more variable, and it is unclear why pressure equalization took so long for the samples with pressure probes set at 45 mm.

Influence of pressing and venting rates.—As might be expected, time to reach 35 kPa tended to decrease with increasing pressurization rate; while time for pressure equalization and surface-to-center pressure differences tended to increase (Table 4). These results seem contradictory since faster press times would be expected to result in increased pressure responses. However, other Douglas-fir samples were easily crushed during pressing or fractured during venting when pressure was rapidly changed. Therefore, although the rates of pressure change in this experiment were not sufficient to show substantial differences, wood permeability should limit the ability of a fluid to flow freely and produce a corresponding lag in pressure response.

Confirmation of pressure differentials following venting.—The average pressure measured at the center of ponderosa pines sapwood samples immediately after venting varied from those calculated from matched samples placed in a pressure bomb after treating (Table 5). We suspect some of this variation reflects losses as the nonsensored sample was removed, but the calculated values were nearly double the gauge mea-

TABLE 3. *Summary of internal pressure responses at the centers of Douglas-fir samples having different radial dimensions during SC-CO_2 treatments.*

Depth of pressure probe (mm)	Time to reach 35 kPa (min)	Time to pressure equilibrium (min)	Max. ΔP during pressing (kPa gauge)	ΔP immediately after venting (kPa gauge)	Wood condition
15	0.5	8.5	117	−2,599	Good
	0.5	2.5	172	−1,441	Good
	2.0	3.0	669	−2,523	Good
	2.0	6.0	558	−2,469	Good
	1.0	9.5	359	−1,875	Good
Avg. (std.)	1.2 (0.7)	5.9 (2.8)	375 (214)	−2,181 (451)	
30	6.5	13.0	1,862	−4,661	Good
	4.0	10.0	1,234	−1,448[2]	Good
	4.0	2.0	1,048	−4,247	Good
	4.0	6.5	1,282	−2,599	Good
	4.0	6.5	1,269	−4,289[2]	Good
Avg. (std.)	4.5 (1.0)	7.6 (3.7)	1,339 (275)	−3,949 (796)	
45	11.5	59.5	4,226	−3,103	Collapsed
	11.0	89.0	3,978	−786	Good
	14.0	78.0	5,081	−4,082	Collapsed
	9.5	44.0	2,765	−3,413	Good
	7.0	—[1]	2,358	−4,082	Good
Avg. (std.)	10.6 (2.3)	67.6 (17.2)	3,682 (993)	−3,670 (426)	

[1] Samples did not reach equilibrium; therefore, values were not used with the average.
[2] Samples had pressure probe sealant failure during venting; therefore, values were not used with the average.

TABLE 4. *Summary of pressure measurements in Douglas-fir heartwood during SC-CO_2 treatments having different pressing and venting rates.*

Pressing & venting rate (kPa/min)	Time to reach 35 kPa (min)	Time to pressure equilibrium (min)	Max. ΔP during pressing (kPa gauge)	ΔP immediately after venting (kPa gauge)	Wood condition
138	3.0	4.5	317	−1,937	Good
	4.0	5.0	531	−731	Good
Avg. (std.)	3.5 (0.5)	4.8 (0.3)	424 (107)	−1,334 (603)	
276	0.5	8.5	117	−2,599	Good
	0.5	2.5	172	−1,441	Good
	2.0	3.0	669	−2,523	Good
	2.0	6.0	558	−2,496	Good
	1.0	9.5	359	−1,875	Good
Avg. (std.)	1.2 (0.7)	5.9 (2.8)	375 (214)	−2,187 (454)	
827	0.5	20.0	586	−2,151	Good
	1.5	20.0	1,193	−2,965	Good
	0.5	—[1]	476	−1,427	Good
	0.5	—[1]	372	−1,965	Good
	0.5	13.5	634	−1,558	Good
	0.5	14.0	855	−2,868	Good
	1.0	10.5	903	−1,503	Good
	1.0	11.0	979	−2,420	Good
	0	10.0	1,082	−2,027	Good
	1.0	11.0	827	−4,237	Good
Avg. (std.)	0.7 (0.4)	13.8 (3.8)	791 (253)	−2,321 (838)	

[1] Samples did not reach equilibrium; therefore, values were not used with the average.

surements in the radial direction. The opposite was true for the tangential direction where the sensored values were nearly double the pressure bomb values. It is unclear why radial or tangential orientation would make such a difference in these measurements; however, the comparisons in both directions show that there is considerable internal pressure in the blocks following treatment. Clearly, this effect is greater in the tangential direction and could become a factor in thicker samples with tangentially dominated flow paths.

TABLE 5. *Pressure measurements immediately after SC-CO_2 treating in matched ponderosa pine sapwood samples.*

Sample	Measurements using pressure probes: surface-to-center pressure difference immediately after venting (kPa gauge)	Calculated residual pressure from bomb equilibrium pressure (kPa gauge)
Pine–radial	258	373
Pine–radial	437	416
Pine–radial	266	528
Pine–radial	245	468
Pine–radial	135	455
Avg. (std.)	268 (108)	448 (58)
Pine–tangential	2,995	1,207

CONCLUSIONS/IMPLICATIONS

The results clearly demonstrate that pressure response in wood during supercritical fluid (SCF) impregnation is not instantaneous. As a result, substantial pressure differentials can develop, probably as a result of restricted flow through the intercellular pitting. SCFs have been hailed as a method for completely impregnating traditionally liquid impermeable materials such as the heartwood of Douglas-fir and the spruces. While results from previous investigations attest to the ability of biocide-laden SCFs to treat these species, it is clear that the process is still restricted by the permeability of the wood. Further development of this technology will require a much better understanding of the dynamic flow of SCFs into the wood matrix, in order to minimize detrimental effects to the wood and ensure

adequate pressure for maintaining biocide solubility throughout the wood. The results of this investigation help to better understand the influence of wood species, grain orientation, sample size, and pressing and venting rates. These results also help to explain sample failure and account for some of the variation in biocide deposition observed in previous trials.

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