Investigation of Pore Size Distribution by Mercury Intrusion Porosimetry (MIP) Technique Applied on Different OSB Panels

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Mercury intrusion porosimetry (MIP) is a technique used to characterize the pore size distribution and resin penetration in lignocellulosic materials, such as oriented strand board specimens (OSB), a multilayer panel utilized in structural applications. The method is based on the isostatic injection, under very high pressure, of a non-wetting fluid (mercury) into the porous material to determine parameters such as pore size distribution and percentage of porosity of the specimens. In this study, five different OSB were analyzed; they contained different wood species, resin type, and resin content. The panels manufactured with castor oil polyurethane resin showed porosity values in the range of 54.7 and 27.8%. This was a promising result compared with those obtained for panels made with phenolic resins, which are currently commercialized in Brazil.

Keywords: Mercury porosimetry; Oriented strand board; Castor oil polyurethane resin

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INTRODUCTION

The commercial exploitation of forest resources is growing globally as a result of the many applications of wood-based products (Santos *et al.* 2014). From an economic point of view, the activities of the lumber industry are of paramount importance. However, logging must be done in a controlled and sustainable manner, looking for ways that lead to more appropriate use of wood (González-Garcia *et al.* 2014). For example, wood can be utilized as a feedstock for the production of reconstituted wood panels (Mattos *et al.* 2008), including oriented strand board (OSB).

The European standard EN 300 (2006) defines OSB as a multilayer panel, generally composed of three to five layers and made from strand of wood with a length greater than 50 mm and less than 2 mm thick, joined together by an adhesive. The wood strands in the external layers tend to align parallel to the length of the panel and in the internal layers may be randomly distributed or aligned generally perpendicular to the external layers (Souza 2012). OSB products are used for structural applications such as walls, ceilings, floors, beams, structural components, *etc.*, due to their mechanical strength and good dimensional stability, competing directly with the plywood market (Mendes 2011).

The OSB market is growing worldwide, with an expected growth of 28% until 2022 (Grand View Research 2015). This increasing consumption in different sectors (mainly construction, furniture, and packing) is related to improved panel properties such as strength, workability, and versatility (Grand View Research 2015). Moreover, the growing substitution of OSB for plywood is expected to continue due to factors such as reduced availability of good quality logs for lamination, making the OSB an advantageous option, as it can be produced from lower quality logs and low commercial value species (Mendes 2013).

In Brazil the OSB is predominantly manufactured with *Pinus sp.* wood species (*Pinus elliotti* and *Pinus taeda*). However, with the growing demand for this product and the fact that *Pinus* species are widely used for various other purposes such as manufacture of plywood boards, cellulose industry, and sawmills (Vidal and Hora 2014), the amount of wood stored may not be enough to supply the market. Other species need to be studied for their use in these products. There have been some studies of the viability of OSB production with other species found in Brazil such as *Croton sonderianus* Muell. Arg, *Piptadenia stipulacea*, *Croton sonderianus* Muell. Arg (Nascimento *et al.* 2015), *Schizolobium amazonicum* (Ferro 2015), *Eucalyptus grandis,* and *Eucalyptus dunnii* (Iwakiri *et al.* 2004).

The common properties investigated in studies about OSB performance are bending strength, modulus of elasticity in bending in major and minor axis, internal bond, and thickness swelling after 24 h of immersion in water, as defined by the EN 300 (2006) standard. Bertolini (2014), Bertolini *et al.* (2014), and Varanda (2016) examined the structural porosity of particleboards. Jin *et al.* (2021) analyzed the porosity of wood species. The presence of pores may influence the mechanical properties, dimensional stability, thermal conductivity, permeability, and acoustical properties of particleboards.

The determination of pore size through mercury intrusion porosimetry (MIP) involves the forced intrusion of mercury into the pores and the measurement of the amount of liquid spent in the procedure (Zhao *et al.* 2021). According to Varanda (2014), this technique can be used to determine several important parameters in the characterization of porous materials, including total intrusion volume, total pore area, average pore diameter, real and apparent density, and porosity of the sample.

Because OSB is used for structural applications, the porosity must be included in the performance evaluation of these materials. The present study evaluated the porosity and pore distribution in different types of OSB manufactured in Brazil using mercury intrusion porosimetry.

EXPERIMENTAL

Materials

This study used five OSBs made with different wood species, resin type, and resin content. Four of them were produced in the Wood and Timber Structures Laboratory (LaMEM), Department of Structures Engineering (SET), São Carlos Engineering School (EESC), São Paulo University (USP), using wood species particles such as *Schizolobium amazonicum* sp., *Pinus* sp., and *Corymbia citriodora*, which were bonded with castor oil-based polyurethane. The other OSB analyzed in this study was found in the Brazilian market, manufactured by Louisiana Pacific Corporation (LP Brazil) with *Pinus* sp. wood and phenol-formaldehyde resin. For OSB manufactured, wooden beams were sectioned into pieces approximately 90 mm wide and 35 mm thick, which defined the length and width of the particles respectively. The particles, generated in a disk chipper, were obtained with a thickness of approximately 0.7 mm. These parameters were selected, once that are the parameters used by OSB industries. Table 1 shows the differences in the panels.

Table 1. OSB Types Subjected to MIP Test

OSB type	Wood species	Resin	OSB density (kg/m³)	Resin content (%)
OSB1	*Pinus* sp.	Phenol-formaldehyde	600	8
OSB 2	*Schizolobium amazonicum*	Castor oil-based polyurethane	650	10
OSB 3	*Schizolobium amazonicum*	Castor oil-based polyurethane	650	12
OSB 4	Pinus sp.	Castor oil-based polyurethane	700	12
OSB 5	*Corymbia citriodora*	Castor oil-based polyurethane	750	12

Methods

The mercury intrusion porosimetry method is based on the isostatic injection, under very high pressure (several hundred megapascals), of a non-wetting fluid (mercury) into the porous material (Zhao *et al.* 2021). For this study, it is important to highlight that the mercury intrusion porosimetry technique was developed based on the studies of Bertolini (2004) and Varanda (2016), which analyzed this property for wood-based panels.

The pore size distribution is determined from the volume intruded at each pressure increment, and total porosity is determined from the total volume intruded (Abell *et al.* 1999). MIP tests were performed with a Micromeritics Poresizer (model 9320, Sao Calos, Brazil) with a maximum 200 MPa injection pressure. Parameters such as total intrusion volume (mL/g), total pore area (m^2/g), average pore diameter (μm), bulk density (mL/g), skeletal density (mL/g), and porosity (%) were determined.

The specimens had dimensions of 14 mm width and 23 mm length. In total, it was used two samples for each OSB type. They were dried in an oven with air circulation at 50 °C for 24 h before the test. The MIP parameters for were as follows: mercury with a surface tension of 0.494 g/cm^2 and density of 13.533 g/mL, advancing and rewind contact angle of 130°, and equilibrium time between the low and high pressure of 10 seconds.

RESULTS AND DISCUSSION

Figure 1 shows the pore size distribution of the specimens obtained by plotting the log differential intrusion volume dV/dP versus the pore diameter. OSB 1 had a greater number of pores with diameters smaller than 0.05 μm, and there was a small pore amount in the range of 0.2 and 0.8 μm. There were few pores above 11 μm. For OSB 2, there were more pores with diameters below 0.04 μm and a considerable number with dimensions between 7 and 12 μm. In OSB 3, there were two pore size distribution ranges, the predominant band at less than 0.04 μm and another between 8 and 16 μm. For OSB 4, the most pores were below 0.02 μm; there was a small number of pores in the range of 0.2 to 1 μm, and others in the range 8 to 12 μm. Finally, for OSB 5, there were more pores in the size distribution ranges below 0.01 μm and a small number of pores between 0.2 and 0.5 μm.

Regarding differential intrusion volume, all specimens presented values between approximately 0.20 and 0.90 mL/g. Table 2 shows the results obtained in the porosity tests.

Table 2. Porosity Features of the Panels

Porosity Features	OSB 1	OSB 2	OSB 3	OSB 4	OSB 5
Total intrusion volume (mL/g)	0.492	0.756	0.752	0.312	0.330
Total pore area (m^2/g)	8.66	22.91	32.30	33.71	11.15
Average pore diameter (μm)	0.227	0.132	0.093	0.037	0.119
Bulk density (mL/g)	0.716	0.724	0.724	1.079	0.910
Skeletal density (mL/g)	1.107	1.599	1.593	1.630	1.306
Porosity (%)	35.29	54.73	54.52	33.77	27.85

According to Bertolini *et al.* (2019), porosity of the panel is associated with voids between particles and with wood microstructural elements. Table 2 shows that OSB 2 and OSB 3, both filled with castor oil polyurethane resin and produced with *Schizolobium amazonicum* wood, exhibited the highest intruded mercury volumes and porosity values. OSB 1 and 4, both manufactured with *Pinus* sp., showed similar porosity values, even though they were manufactured with different resin types and different resin content. However, OSB 4 presented a slightly lower value, which may be related to the higher density of the panel, as well as the higher amount of resin. OSB 5 had the lowest porosity value. In the other hand, the increase in the amount of resin from 10 (OSB 2) to 12% (OSB 3), both made with *Schizolobium amazonicum*, did not significantly influence the porosity performance of the panels.

According to Vidaurre (2010), *Schizolobium amazonicum* species has a lumen diameter of about of 25.6 μm. On the other hand, *Corymbia citriodora* and *Pinus* sp. had lumen diameters of about 5.4 μm and 27.84 μm, respectively (Amaral 2014; Segura 2015). However, it is important to highlight that the anatomy of wood of *Corymbia citriodora* and *Pinus* sp. is totally different, once the first one is hardwood and has fibriform fibers and vessels, whereas the second one is softwood and has tracheids cell. So, the anatomy characteristics of wood significantly contributed to the porosity results in OSB specimens.

The OSB 4 and 5 specimens showed a total intrusion volume of 0.31 and 0.33 mL/g, respectively, but OSB 4 presented a greater percentage of porosity. This could be explained by the pore geometry (Fig. 2); when the pore is open and interconnected (c, d, e and f in Fig. 2), the mercury fills the space even before applying pressure (Santos *et al.* 2014).

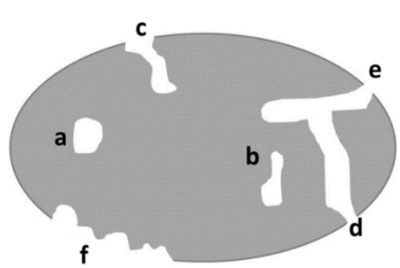

Fig. 2. Representative scheme of pores with different sizes and shapes: (a) and (b) closed pores; (c) open pore; (d) and (e) interconnected pores; (f) surface roughness. (Santos *et al.* 2014)

The empty spaces in the microstructure of a system are classified by the International Union of Pure and Applied Chemistry (IUPAC) as micropores, mesopores, and macropores (Santos *et al.* 2014), as shown in Table 3.

Table 3. Pore Classification

Classification	Pore size
Micropores	diameter < 0.002 μm
Mesopores	0.002 μm<diameter < 0.05 μm
Macropores	diameter > 0.05 μm

According to Table 3, all OSB types present macropores. The presence of macropores in each type of OSB is related to anatomical aspects of the wood. Because of the stochastic manner in which OSB is formed, as well as the differences in sizes of the strands, one can expect there to be a high frequency of empty spaces within the structure. The amount of resin applied, in combination with the applied pressure, is not likely to be large enough, during practical manufacturing conditions, to fill eliminate such pores between the strand elements. All panels in this study were pressed with 4.0 MPa. Nonetheless, Bertolini *et al.* (2019) analyzed the porosity for *Pinus* sp. particleboards pressed with 4.5 and 2.5 MPa. Porosity of panels were influenced by intensity of pressure during their manufacturing, being large for panel of 2.5 MPa. The use of uniform particles in the production of panels enables the occurrence of a larger voids, as these ordinarily would be filled with smaller particles.

Schizolobium amazonicum, used in the production of OSB 1 and 2, is a low-density wood (390 kg/m³). It shows no difference between heartwood and sapwood, and lacks knots. The low density contributes for more workability of the species, and these factors collaborate to generate particles with more uniform dimensions. *Pinus* sp. and the *Corymbia citriodora* (with a density of 490 kg/m³ and 700 kg/m³, respectively), show differences between the heartwood, sapwood, and knots, thus making particles with less uniformity. The utilization of smaller particles in the manufacture of the panels reduces the empty spaces; thus, the number of pores in the panel decreases. Allied to this are the pore diameters related to each species of wood, as already commented. It is also noteworthy that in addition to the reduced pore diameter compared to the other species under study, *Corymbia citriodora* has pores filled with tyloses. Expansion of the cell wall of a parenchyma cell adjacent to a vessel element, through the opening of a puncture, partially or completely blocking the lumen diameter. Tyloses in wood cause effects such as vessel closure causing low wood permeability.

The obtained results were compared to the literature. Varanda (2016), for example, analyzed the porosity of particleboard produced with *Pinus* sp. and peanut shell, glued with oil-based polyurethane resin beaver. Results showed a porosity of 33.7%, a value very similar to obtained in this study for treatments OSB 1 and OSB 4 (both produced with *Pinus* sp.). Bertolini *et al.* (2019) obtained approximate value of 54% for particleboard produced with *Pinus* sp. waste materials, which were treated with copper chrome boric oxide preservative and glued with castor oil-based polyurethane resin. This result is similar to that obtained for OSB 2 and OSB 3.

This result is very important for wood construction industry, especially in Brazil, where most of the houses are manufactured with OSB. Characteristics such as thermal conductibility and acoustic comfort are very important, and are used improved with the addition of other material as glass wool, pet wool. The porosity of the panel can improve theses performances of the construction system and enable the reduced usage of other materials.

According to Luamkanchanaphan *et al.* (2012), thermal and acoustic comfort are related to density of the panel: higher density leads to higher thermal conductivity. In insulation materials, heat transfers occur in the materials and voids filled with air.

CONCLUSIONS

1. The pore size distribution for oriented strand board specimens OSB 2 and OSB 3 indicated a large presence of macropores in the microstructure of the panels, contributing to a higher total porosity. This could be related to panel manufactured, as well as, microstructure of wood.
2. The OSB produced with *Schizolobium amazonicum* and castor oil polyurethane resin showed higher total porosity in the process conditions used in this study.
3. The commercial OSB found in the Brazilian market showed smaller structural porosity.

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