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Wood-Polymer Composite: Physical and Mechanical Properties of Some Wood Species Impregnated with Styrene and Methyl Methacrylate

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This study aimed to demonstrate the feasibility of obtaining wood-polymer composites (WPC) displaying a performance superior to that of untreated wood from reforested genera. To this end, wood samples of Pine (*Pinus caribaea*) and Eucalyptus (*Eucalyptus grandis*), whose density is compatible with this process, were impregnated with the polymeric monomers styrene and methyl methacrylate, using benzoyl peroxide to initiate the polymerization process forming free radicals. The vacuum-pressure method was used to impregnate the samples with monomer-initiator solution. The results indicated a significant improvement of all the properties investigated, including dimensional stability, for the Pine-WPC, while Eucalyptus-WPC, owing to the wood's low permeability, showed only increased values of hardness parallel and perpendicular to grain.

Keywords: *wood, physical and mechanical properties, styrene, methyl methacrylate*

1. Introduction

Brazil has considerable reserves of tropical species. However, selective and predatory exploitation has reduced the offer of traditional species, of which demand still persists. The reserves of some of these species have therefore become almost completely depleted, causing prices to soar prohibitively and making the use of these raw materials unfeasible.

In the search for solutions to this issue, the South and Southeastern regions have fallen back on reforested species, particularly the genera *Eucalyptus* and *Pinus*. Increasing interest has focused on the study of Wood-Polymer Composites (WPC) obtained from reforested species for the aforementioned reasons. However, for this alternative to be feasible on an industrial scale, it is essential that the performance of WPCs be well characterized, from their production process to the requisites for their various applications in the construction and furniture industries, among others.

The work reported on here, developed in the Wood and Timber Structures Laboratory (LaMEM) of the São Carlos School of Engineering (EESC), University of São Paulo (USP), aims at demonstrating the possibility of producing WPC with superior mechanical properties to those of untreated wood, using reforested species of the genera *Eucalyptus* and *Pinus* and in situ polimerization of styrene and methyl methacrylate monomers.

2. Literature Review

2.1. Structure and chemical composition of wood

Wood is composed of millions of individual units called cells, which differ in shape and size, depending on the tree's physiological function[1]. The cells that make up the xylem of conifers have structures simpler than those of the dicotyledons. The tracheids in conifers and the fibers in dicotyledons constitute the greatest part of the cell wall and can be credited with most of the wood's physical and chemical properties[2].

Conifers are composed mainly of cells that are tubular, elongated, pointed and closed at the ends, called tracheids[2]. The dicotyledons, on the other hand, are composed of cells of more varying shapes and sizes. Most dicotyledon cells

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are long and narrow, with pointed and closed ends – the fibers. Other important constituents are the parenchyma and, in relatively small quantities, the vessels that, in transversal cuts, are called pores[3]. These cells have open ends and are usually shorter than fibers, varying considerably in shape and size.

Wood cells are connected to each other through a cemented substance called intercellular layer or medium lamella. A mature cell is made up of two layers: the primary wall – a thin external layer, and the secondary wall – a thicker internal layer composed of three other layers. The interior of the cell contains the cellular lumen which, in most mature cells, is completely empty[1].

The main wall constituents of xylem cells are cellulose, hemicellulose and lignin[1]. In addition to these structural components, wood presents inclusions of organic and inorganic matter with high and low molecular weights.

2.2. Polymers

Polymers are a large class of materials consisting of many small molecules (called monomers) that can be linked together to form long chains, otherwise known as macromolecules[4]. Polymerization is the chemical process whereby monomers are joined to form a polymer. Polymers are widely used in composites as fibers or as the matrix[5]. More information about polymers can be found in the literature indicated[6-8].

2.3. Wood-polymer composites

Wood-polymer composites (WPC) result from the polymerization of liquid monomers or oligomers already impregnated in the wood. Wood porous structure, composed of lignin, cellulose and hemicellulose, is filled with a solid, plastic and fairly hard substance. In principle, WPCs should display superior mechanical properties, dimensional stability, greater resistance to chemical and biological degradation, and less moisture absorption than non-impregnated wood[9]. WPC production necessarily goes through two different phases: impregnation with a monomer/oligomer[10], followed by its polymerization inside the wood.

Wood is impregnated injecting certain chemical products (liquids), which can be done by immersion, vacuum-immersion and vacuum-pressure[1]. Insofar as impregnation with natural resins is concerned, it is worth mentioning the improvement of some physical properties of White Fig (*Ficus monckii*) impregnated with natural resin from Jatobá (*Hymenaea courbaril*)[11].

The impregnated monomer/oligomer can be polymerized through two different processes: by the incidence of radiation[10] or by the thermal decomposition of initiators[12]. In thermal polymerization processes, the thermal dissociation of initiators is the most commonly used method to generate radicals to start the reaction[13]. These initiators are compounds which decompose easily into free radicals as temperature rises.

The preliminary study carried out in Brazil presents few information related to modulus of rupture and hardness parallel to grain, in Pinus Elliottii (*Pinus elliottii*) specimens impregnated with methyl methacrylate[14]. The results showed the good potential of WPC but only now circumstances turned possible the research continuation.

3. Materials and Methods

3.1. Wood

Tests were carried out on samples of reforested *Eucalyptus grandis* and *Pinus caribaea* obtained from the Itirapina nursery of the São Paulo Forest Institute, some of them not impregnated and others impregnated with styrene and methyl methacrylate monomers.

3.1.1. *Eucalyptus grandis*

Eucalyptus grandis, within the class of the dicotyledons, has a parenchyma that is visible under a hand lens, vasicentric, scanty and occasionally dispersed. Its pores are visible to the naked eye, numerous and of medium size, predominantly solitary, frequently containing resins and sometimes obstructed by tyloses; rays visible under a hand lens in the cross section and the tangential face; and a strong pink colored heartwood. This species occurs in the reforested areas of southern and southeastern Brazil[15].

3.1.2. *Pinus caribaea*

Pinus caribaea, classified as a conifer, lacks parenchyma and pores. It has very small tracheids with a slightly radial orientation that are individually indistinguishable to the naked eye but visible under a hand lens, almost indistinguishable rays at the cross section and on the tangential face, growth layers marked by initial and late xylem with variable thicknesses, a medium texture, and a beige colored core slightly resinous and with a pleasant odor[15].

3.2. Monomers and initiator

Styrene, methyl methacrylate, and benzoyl peroxide were obtained from Companhia Brasileira de Estireno, Companhia Química Metacril and Degussa Initiators Ltda., respectively.

3.3. Removal and identification of the test specimens

The samples were prepared according to the specifications of Attachment B of the Brazilian NBR 7190:1997 standard[16]. The test specimens (TP) were taken from twelve pieces of each wood species, with nominal dimensions of 5 cm × 12 cm × 180 cm, each piece yielding three bars. Each bar supplied a set of three samples for each type of impregnation, totaling 576 test specimens, i.e., 12 sets of samples for each of the wood species studied, as illustrated in Fig. 1. Each test specimen was identified with a capital letter (corresponding to the beam), a small letter (corresponding to the type of test) and a number (corresponding to the type of impregnation). The samples identified with the number 1 were tested without impregnation, those displaying the number 2 were impregnated with styrene monomer, and the ones bearing the number 3 were impregnated with methyl methacrylate.

3.4. Phases of the test specimens' impregnation and polymerization process

Before the wood was impregnated with the monomer, part of the water had to be removed from its pores due to the wood's moisture content. The test specimens were therefore oven-dried at a temperature of 40 °C until their moisture content decreased to 12%. The oven's temperature was gradually raised to 50 °C to reduce the possibility of cracking or warping.

The method utilized to impregnate the monomer-initiator solution was vacuum-pressure. The autoclave employed for the impregnation work had a capacity of 159,000 mL. A total volume of 20,520 mL of each wood species was placed in the autoclave, which was then closed and the air removed from its interior. When vacuum was reached, the monomer-initiator solution (20,520 mL of monomer + 255 g of benzoyl peroxide)[17], was injected into the autoclave for each wood species. A pressure of 0.66 MPa was then applied for 30 min to complete the impregnation process.

The test specimens were then removed from the autoclave and wiped with paper towels to remove any excess impregnation resin. The next step consisted of weighing the test specimens, wrapping them in aluminum foil and placing them in the oven, where they were left for 48 h at a temperature of 60 °C. The samples were then removed from the oven, unwrapped, weighed and put back into the oven for another period of 72 h at 50 °C to consolidate the polymerization process inside the wood. Finally, the samples were removed from the oven, weighed, and their dimensions measured, in preparation for the tests to obtain the desired properties.

3.5. Tests to determine the properties of impregnated and non-impregnated wood

Tests were carried out to determine the following properties: Density (ρ); Total radial shrinkage ($\varepsilon_{r,2}$); Total tangential shrinkage ($\varepsilon_{r,3}$); Total radial swelling ($\varepsilon_{i,2}$); Total tangential swelling ($\varepsilon_{i,3}$); Strength in compression parallel to grain (f_{c0}); Modulus of elasticity in compression parallel to grain (E_{c0}); Strength in tension parallel to grain (f_{t0}); Modulus of elasticity in tension parallel to grain (E_{t0}); Strength in tension perpendicular to grain (f_{t90}); Shear strength (f_{v0}); Toughness (W); Impact bending (f_{bw}); Hardness parallel to grain (f_{H0}) and Hardness perpendicular to grain (f_{H90}). The tests were performed following the specifications of Attachment B of the Brazilian NBR 7190:1997 standard[16]. The toughness was calculated based on the ASTM D143-52:1981 standard[18].

3.6. Procedures for analyzing the results

The results were analyzed using the pairing test, procedure commonly employed in such cases[19]. The test consists of making a comparative analysis of the test specimens non-impregnated and impregnated with styrene and methyl methacrylate monomers to discover whether the difference between the averages of the physical and mechanical properties under study could be null, evidencing that they can be admitted to be statistically equivalent.

The first step in order to apply this methodology consists in calculating the difference between two population means (impregnated and non-impregnated specimens). Values obtained in this way are considered as a third popula-

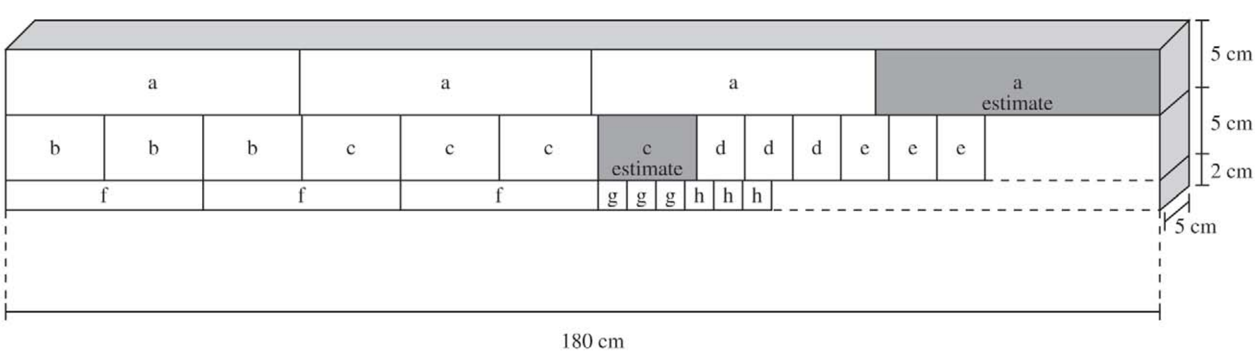


Figure 1. Diagram showing the removal of test specimens from the wooden beam, where: a) strength and modulus of elasticity in tension parallel to grain; b) hardness parallel and perpendicular to grain; c) strength and modulus of elasticity in compression parallel to grain; d) strength in tension perpendicular to grain; e) shear strength; f) toughness and impact bending; g) density; h) total radial and tangential shrinkage, total radial and tangential swelling.

tion. The second step consists in estimating the mean and confidence interval of this population, which is obtained through the following expression:

$$\bar{x}_m - t_{\frac{\alpha}{2},n-1}\frac{s_m}{\sqrt{n}} \leq \mu \leq \bar{x}_m + t_{\frac{\alpha}{2},n-1}\frac{s_m}{\sqrt{n}} \quad (1)$$

Where: $\bar{x}_m$ is the sample mean of the third population, s_m is the sample standard deviation of this population, n is the sample size, α is the confidence level usually adopted 95% and $t_{\frac{\alpha}{2},n-1}$ is the percentage point of the t distribution with n-1 degrees of freedom.

Analysis is carried out using this interval. If zero belongs to it, the means of the two populations (impregnated and non-impregnated) can be considered as equivalent. If zero does not belong to the interval, these means can be considered as different.

4. Presentation and Discussion of the Results

Tests were carried out on a total of 576 samples, 288 of *Eucalyptus grandis* (EG) and 288 of *Pinus caribaea* (PC), with 96 samples of each species without impregnation (wi), 96 impregnated with styrene monomer (i-E) and 96 impregnated with methyl methacrylate monomer (i-M). Small variations in the toughness values caused by differences in the nominal dimensions of the samples were corrected[20]. The results obtained from the tests performed to determine the physical and mechanical properties of the wood at 12% moisture content are listed as follows:

4.1. *Eucalyptus grandis*

Tables 1 and 2 list the results and pairing tests obtained when comparing samples of *Eucalyptus grandis* without impregnation and impregnated, respectively, with styrene and methyl methacrylate monomers.

The statistical analysis revealed that, except for the hardness parallel (f_{H0}) and perpendicular to grain (f_{H90}), all the other properties studied showed a zero within the confidence interval of the mean differences. Hence, equivalence is admitted between the properties of the wood without impregnation and impregnated with the two monomers involved in this study.

The analysis of the microscopic structure of the non-impregnated samples of *Eucalyptus grandis* compared with the samples impregnated with the styrene and methyl methacrylate monomers shown in Fig. 2 revealed that there was little penetration and subsequent polymerization of the monomers inside the wood's anatomical structure. However, some of the pores are clearly filled with polystyrene and with polymethyl methacrylate. The reason for this poor penetration is that, despite the large quantity of fibers, their small diameter almost completely precludes impregnation. In addition, the interior of the pores may present contents generically called gum-resins.

4.2. *Pinus caribaea*

Tables 3 and 4 show the results and pairing tests obtained when comparing samples of *Pinus caribaea* non-impregnated and impregnated with styrene and methyl methacrylate monomers, respectively.

The statistical analysis indicated that the confidence interval of the mean differences did not contain zero for any of the physical and mechanical properties studied. There-

Table 1. Results and pairing test – *Eucalyptus grandis* (wi) and (i-E).

Property	Mean percent variation (%)	Paired comparison		
ρ (g/cm³)	0.2	- 0.02	≤ μ ≤	0.01
$\varepsilon_{r,2}$ (%)	- 4.7	- 0.3	≤ μ ≤	1
$\varepsilon_{r,3}$ (%)	1.6	- 0.6	≤ μ ≤	0.3
$\varepsilon_{i,2}$ (%)	- 5.1	- 0.3	≤ μ ≤	1
$\varepsilon_{i,3}$ (%)	1.4	- 0.7	≤ μ ≤	0.4
f_{c0} (MPa)	13.2	- 15	≤ μ ≤	0.03
E_{c0} (MPa)	- 0.9	- 3046	≤ μ ≤	3398
f_{t0} (MPa)	- 7.2	- 13	≤ μ ≤	23
E_{t0} (MPa)	13.0	- 3988	≤ μ ≤	353
f_{t90} (MPa)	11.8	- 2	≤ μ ≤	0.4
f_{v0} (MPa)	5.5	- 3	≤ μ ≤	1
W (J)	- 21.2	- 0.4	≤ μ ≤	14
f_{bw} (kJ/m²)	- 16.9	- 3936	≤ μ ≤	32219
f_{H0} (MPa)	36.9	- 4239	≤ μ ≤	- 3079
f_{H90} (MPa)	49.1	- 6858	≤ μ ≤	- 5512

Table 2. Results and pairing test – *Eucalyptus grandis* (wi) and (i-M).

Property	Mean percent variation (%)	Paired comparison		
ρ (g/cm³)	- 2.0	- 0.001	≤ μ ≤	0.03
$\varepsilon_{r,2}$ (%)	- 5.9	- 0.2	≤ μ ≤	1
$\varepsilon_{r,3}$ (%)	0.8	- 1	≤ μ ≤	1
$\varepsilon_{i,2}$ (%)	- 6.5	- 0.2	≤ μ ≤	1
$\varepsilon_{i,3}$ (%)	0.4	- 1	≤ μ ≤	1
f_{c0} (MPa)	11.3	- 13	≤ μ ≤	0.1
E_{c0} (MPa)	- 2.7	- 4321	≤ μ ≤	5346
f_{t0} (MPa)	- 16.1	- 5	≤ μ ≤	27
E_{t0} (MPa)	9.5	- 2697	≤ μ ≤	27
f_{t90} (MPa)	2.6	- 1	≤ μ ≤	1
f_{v0} (MPa)	4.9	- 2	≤ μ ≤	0.3
W (J)	- 15.3	- 3	≤ μ ≤	13
f_{bw} (kJ/m²)	- 13.2	- 11762	≤ μ ≤	33916
f_{H0} (MPa)	33.5	- 4242	≤ μ ≤	- 2392
f_{H90} (MPa)	44.3	- 6133	≤ μ ≤	- 5035

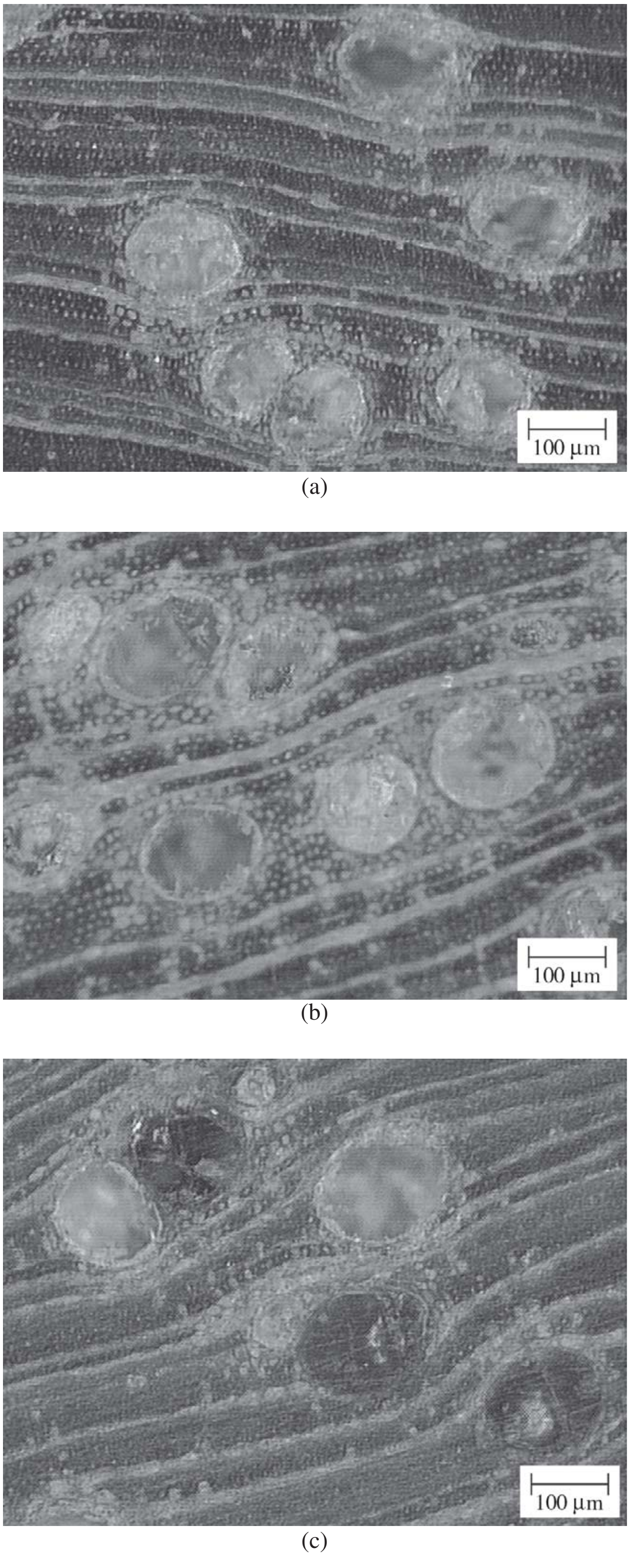


Figure 2. Microscopic structure of *Eucalyptus grandis* observed by optic microscope (100 ×): a) without impregnation; b) impregnated with styrene monomer; c) impregnated with methyl methacrylate monomer.

Table 3. Results and pairing test – *Pinus caribaea* (wi) and (i-E).

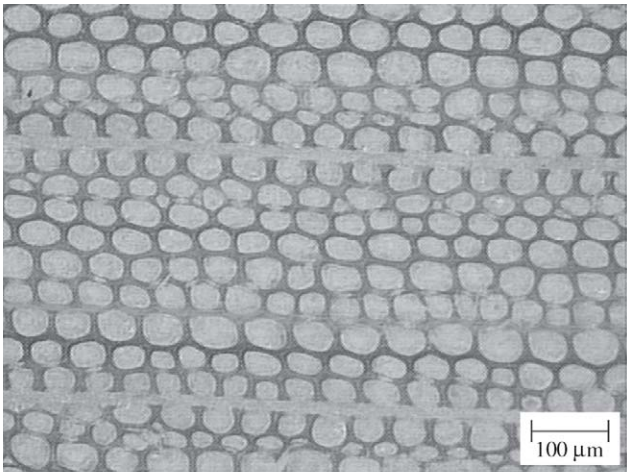
Property	Mean percent variation (%)	Paired comparison		
ρ (g/cm^3)	79.3	- 1	$\leq \mu \leq$	- 0.3
$\varepsilon_{r,2}$ (%)	- 10.5	0.1	$\leq \mu \leq$	1
$\varepsilon_{r,3}$ (%)	- 8.8	0.1	$\leq \mu \leq$	1
$\varepsilon_{i,2}$ (%)	- 11.0	0.1	$\leq \mu \leq$	1
$\varepsilon_{i,3}$ (%)	- 12.5	0.1	$\leq \mu \leq$	1
f_{c0} (MPa)	102.9	- 49	$\leq \mu \leq$	- 37
E_{c0} (MPa)	74.3	- 12392	$\leq \mu \leq$	- 3883
f_{t0} (MPa)	71.5	- 47	$\leq \mu \leq$	- 23
E_{t0} (MPa)	47.0	- 6599	$\leq \mu \leq$	- 3001
f_{t90} (MPa)	66.7	- 2	$\leq \mu \leq$	- 1
f_{v0} (MPa)	65.3	- 10	$\leq \mu \leq$	- 4
W (J)	81.9	- 14	$\leq \mu \leq$	- 7
f_{bw} (kJ/m^2)	85.2	- 37067	$\leq \mu \leq$	- 18938
f_{H0} (MPa)	425.8	- 12642	$\leq \mu \leq$	- 8924
f_{H90} (MPa)	299.9	- 15928	$\leq \mu \leq$	- 12306

Table 4. Results and pairing test – *Pinus caribaea* (wi) and (i-M).

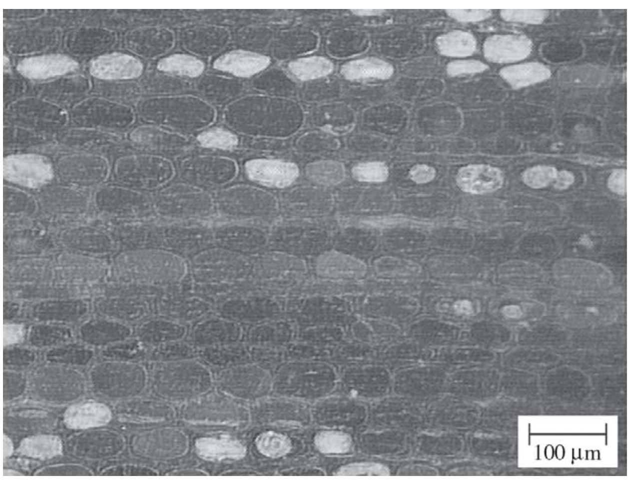
Property	Mean percent variation (%)	Paired comparison		
ρ (g/cm^3)	69.8	- 0.4	$\leq \mu \leq$	- 0.3
$\varepsilon_{r,2}$ (%)	- 15.9	0.2	$\leq \mu \leq$	1
$\varepsilon_{r,3}$ (%)	- 12.2	0.2	$\leq \mu \leq$	1
$\varepsilon_{i,2}$ (%)	- 11.5	0.01	$\leq \mu \leq$	1
$\varepsilon_{i,3}$ (%)	- 18.2	0.1	$\leq \mu \leq$	2
f_{c0} (MPa)	106.7	- 53	$\leq \mu \leq$	- 36
E_{c0} (MPa)	58.4	- 9638	$\leq \mu \leq$	- 3136
f_{t0} (MPa)	67.2	- 40	$\leq \mu \leq$	- 26
E_{t0} (MPa)	38.0	- 5269	$\leq \mu \leq$	- 2492
f_{t90} (MPa)	63.0	- 2	$\leq \mu \leq$	- 1
f_{v0} (MPa)	61.4	- 8	$\leq \mu \leq$	- 4
W (J)	100.0	-17	$\leq \mu \leq$	- 8
f_{bw} (kJ/m^2)	99.1	- 44829	$\leq \mu \leq$	- 20307
f_{H0} (MPa)	457.6	- 14123	$\leq \mu \leq$	- 9057
f_{H90} (MPa)	298.4	- 15251	$\leq \mu \leq$	- 12843

fore, one can state a statistical non-equivalence between the properties of the wood without impregnation and impregnated with the styrene and methyl methacrylate monomers.

The analysis of the microscopic structure of the non-impregnated samples of *Pinus caribaea* compared with the samples impregnated with the styrene and methyl methacrylate monomers shown in Fig. 3 indicated that penetration and subsequent polymerization of the monomers occurred inside the wood's anatomical structure. This Pine species has a permeable structure, facilitating its impregnation and the subsequent retention of the polystyrene and the polymethyl methacrylate.



(a)



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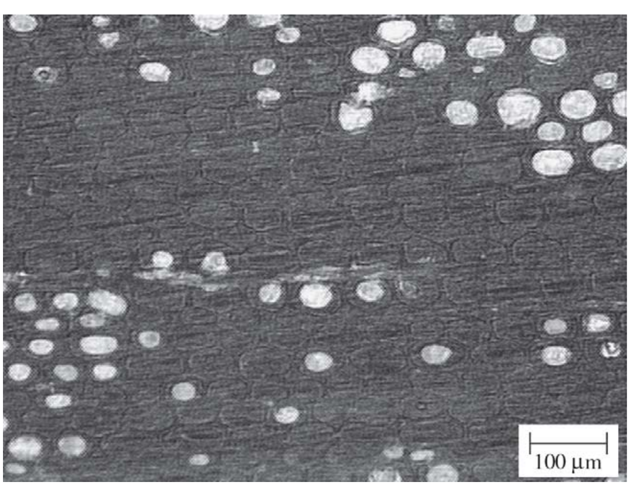

(c)

Figure 3. Microscopic structure of *Pinus caribaea* observed by optic microscope (100 ×): a) without impregnation; b) impregnated with styrene monomer; c) impregnated with methyl methacrylate monomer.

5. Conclusions

The results obtained for *Eucalyptus grandis* showed a statistically non-significant variation in all the properties studied here between impregnated and non-impregnated wood, except for the hardness parallel and perpendicular to grain. This poor outcome can, in principle, be attributed to the merely superficial penetration of the aforementioned monomers as a result of the species' anatomical peculiarities (small diameter fibers and ends, and vessels or pores frequently containing resins). The presence of styrene and methyl methacrylate in these regions caused minor variations of the measured properties.

The comparison between *Pinus caribaea* impregnated with the aforementioned monomers showed a significant increase in all the physical and mechanical properties related to the non-impregnated samples. The dimensional stability of the composite increased in comparison with the untreated wood, rendering it more impermeable to moisture absorption and retention. The two types of impregnation led to a strong improvement in the wood's hardness parallel and perpendicular to grain, with an average percent increase of over 400% and 300%, respectively, rendering this composite very interesting for flooring applications, for instance. It is also worth noting that the incorporation of polystyrene and polymethyl methacrylate was also highly satisfactory in *Pinus caribaea*, reaching an average of 80% and 50%, respectively, in wood mass.

Based on these results, it can be concluded that *Eucalyptus grandis* does not exhibit performance suitable for this type of impregnation, owing to its low permeability. *Pinus caribaea*, on the other hand, absorbs styrene and methyl methacrylate monomers easily, indicating the efficiency of the wood impregnation process and thus allowing for its use in applications that require materials with superior mechanical properties.

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