Analysis of the Treatability, Water Repellency Efficiency, and Dimensional Stability of Maritime Pine after Microwave Drying

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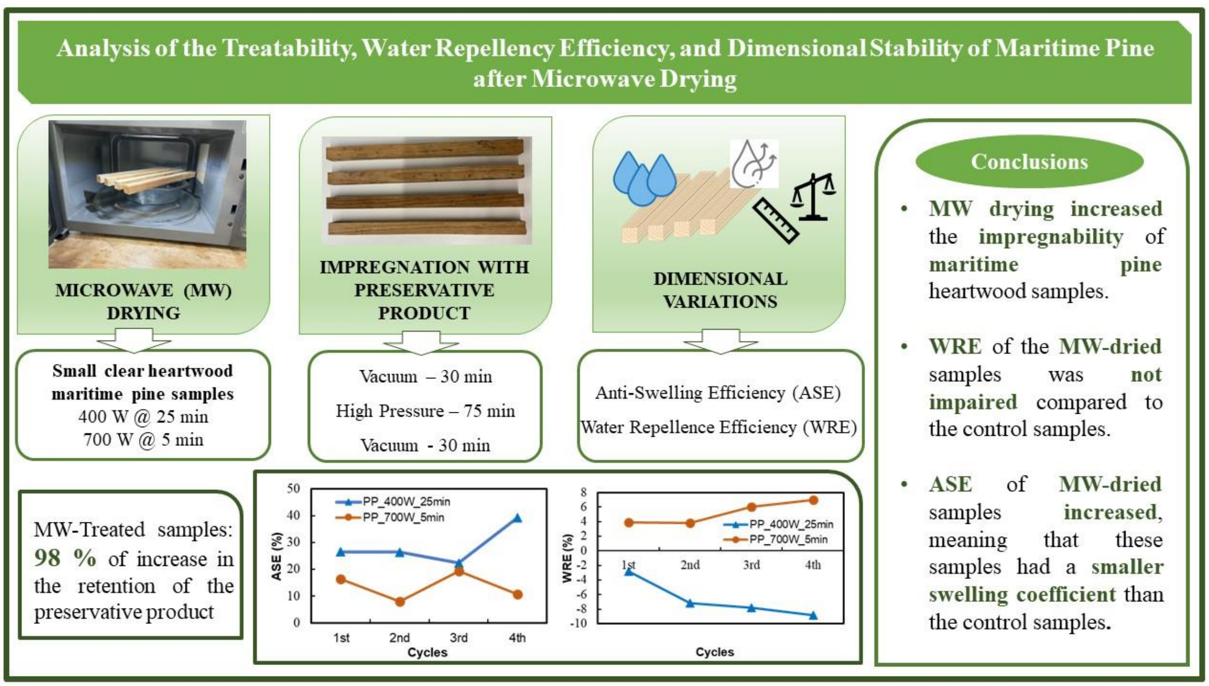
GRAPHICAL ABSTRACT



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The impregnability of Portuguese maritime pine (*Pinus pinaster* Ait.) subjected to microwave (MW) drying was tested, and the hydrophobicity, anti-swelling efficiency (ASE), and water repellence efficiency (WRE) were evaluated. Small wood heartwood samples of Portuguese maritime pine and two distinct MW treatment settings were employed. The levels of ASE and WRE of the wood elements were evaluated throughout four cycles of drying in an oven and soaking in water. Because of MW applied energy, the wood pine samples were satisfactorily impregnated with the preservative product. Regarding the absolutely dry densities of the samples, very subtle reductions were measured, and they were statistically equivalent to the average density of the non-MW-treated group. Slight improvements were identified in the WRE values of wood samples dried in the microwave. In terms of ASE, both MW-treated groups had a statistically significant increase. The MW treatment decreased the volumetric swelling of the maritime pine wood specimens. Hence, this study raises new insights and previously unexplored paths that can contribute to the expansion and greater application of MW technology in maritime pine and other species.

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Keywords: Microwave drying; Maritime pine; Impregnability; Water repellence efficiency; Anti-swelling efficiency

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INTRODUCTION

Wood is an ancient material in construction with different usages, from door frames to structural elements. Its usage in construction has been strengthening in recent years. It is currently used to replace other broadly used structural materials such as concrete and steel (Švajlenka and Kozlovská 2020). Wood has one outstanding point that makes it stand out among the other materials: its sustainable nature. It is crucially important in the sustainable transformation of the construction industry and, as a consequence, the entire world (Zubizarreta *et al.* 2019; Švajlenka and Kozlovská 2020; Svatoš-Ražnjević *et al.* 2022). Besides the environmental aspect of wood, it has advantageous properties as a material, such as its great strength-to-weight ratio, good thermal behavior, and aesthetic appeal (Arriaga *et al.* 2023).

Sawn wood production on a global scale is presently concentrated on softwood species. Projections indicate that by 2030, approximately half of all softwood production will be allocated for utilization in the construction industry, thereby substituting for steel, concrete, and masonry (Nepal *et al.* 2021; Martins *et al.* 2023). Besides the wide use of solid wood elements, engineered wood elements, for example, cross-laminated timber (CLT), are undergoing a fast increase in use in the contemporary market and are becoming very popular and used in different construction projects around the globe (Pramreiter *et al.* 2023). If properly engineered, mass timber products can be manufactured using various species of both softwoods and hardwoods. However, it is common to utilize construction-grade softwoods, such as spruce, pine, and fir (Comnick *et al.* 2022). Therefore, there is a growing demand for wood to subsidize the growth of this market.

Maritime pine (*Pinus pinaster* Aiton) corresponds to 22% of forests in Portugal (the main softwood) (ICNF 2019). It has been used in construction in Portugal since the 1800s (Fernandes *et al.* 2016). This particular pine species is indigenous to the western region of the Mediterranean basin, particularly in the southwestern parts of Europe, along with certain areas in northwestern Africa. Besides Portugal, it is found throughout Spain, southern France, and the nearby region that encompasses the island of Corsica and western Italy (Viñas *et al.* 2015; Alonso-Esteban *et al.* 2022).

In Portugal, maritime pine is used for furniture, as construction material, utility poles, and engineered wood products (Gaspar *et al.* 2010; Morgado *et al.* 2013, 2017; Viñas *et al.* 2015; Martins *et al.* 2019). Richardson and Rejmánek (2004) state that the increase in pine forests is acknowledged as a worldwide phenomenon. In the manufacturing process of glued laminated timber (glulam), for example, most of the engineered wood products used in Portugal are made of imported wood species (Martins *et al.* 2019). Therefore, a niche exists to be explored using Portuguese pine wood.

Despite the advantages of wood, the low permeability, dimensional variations in response to atmospheric changes, and susceptibility to biological degradation are hindrances. These limitations may be restrictive on the potential applications of wood (Hill 2006), playing a crucial role in the economic aspect of utilization (Shukla 2019).

The dimensional variations and hygroscopicity that several wood species exhibit are intimately associated with the final internal water content of wood elements and the hydroxyl groups (-OH) in the cell walls of wood. The hydroxyl groups present in wood have the ability to draw water molecules into its vicinity by means of hydrogen bonding, which results in the expansion of the wood and renders it vulnerable to changes in dimensions (Ding *et al.* 2012; Mattos *et al.* 2014; Forest Products Laboratory 2021). Anti-Swelling Efficiency (ASE) and Water Repellence Efficiency (WRE) tests have been widely used to assess the susceptibility of wood and wood-composed elements to variations in water content (Magalhaes and Da Silva 2004; Ding *et al.* 2012; Mattos *et al.* 2014; Dong *et al.* 2016; Sargent 2019).

The sapwood of maritime pine lacks natural durability. It may be attacked by termites and fungi, and the heartwood has low treatability, which is a measure of the impregnability with a preservative product (Tarmian *et al.* 2020). Although the heartwood of maritime pine is durable for *Hylotrupes* and *Anobium*, it is not durable against fungi and termites, requiring treatment against them. Nevertheless, maritime pine heartwood is extremely difficult to treat, as evidence of its reduced permeability. Hence, an elevated permeability is worthwhile when wood and wood-based materials must be deeply and homogeneously permeated with chemical or biological products (Bakir 2022).

Faced with the limitations of wood, modification processes are gaining importance. Wood modification processes are technologies employed to modify the properties of the wood to mitigate or improve its various drawbacks, aiming, for example, to improve its mechanical properties, enhance permeability, increase biological durability, and others (Hill 2006). The microwave (MW) drying or treatment is an emerging technology capable of improving wood treatability (Torgovnikov and Vinden 2009; Mascarenhas *et al.* 2021, 2023a; Weng *et al.* 2021; Kol and Çayır 2022). Several authors have pointed out that MW technology can offer substantial savings in material and energy efficiency, then improve the environmental and economic performance of the wood and timber industry (Leiker and Adamska 2004; Torgovnikov and Vinden 2010; Wang *et al.* 2022). The costs of MW drying are deemed acceptable by the sector, and it follows that there is real potential to have applications in the wood, timber, pulp, and biocomposite sectors (Torgovnikov and Vinden 2010).

In addition to being regarded as an environmentally friendly method, MW treatment can provide a more homogenous and fast drying compared to conventional drying methods (Torgovnikov and Vinden 2009; Wang *et al.* 2022). In the course of microwave drying, electromagnetic waves permeate the entirety of the sample, thereby enabling the absorption of heat throughout the entire volume of the wet wood specimen. This represents a significant departure from traditional wood drying techniques. In conventional drying, heat is transferred from the outer layers of the specimen to the inner layers through mechanisms such as convection, conduction, and radiation (Sahin and Ay 2004; Metaxas and Meredith 2008). This transfer of energy is driven by thermal gradients within the material. In contrast, microwave heating operates by converting electromagnetic energy into thermal energy in a localized manner. Consequently, the material experiences rapid heating across its entire thickness, resulting in a reduction in thermal gradients. This process of volumetric heating effectively minimizes drying times and conserves energy (Sahin and Ay 2004).

The MW device emits electromagnetic radiation that interacts with water; the molecules begin to agitate rapidly, generating increased temperature and, in turn, releasing water vapor at high temperatures (Oloyede and Groombridge 2000; Torgovnikov and Vinden 2009). This causes different wood cells and tissues to rupture, which leads to an expansion in pores and the emergence of new ones, resulting in the creation of new pathways through which water and vapor will come out of the wood (Mascarenhas *et al.* 2021, 2023a; Torgovnikov and Vinden 2010). Given this, there is a growth in wood porosity and permeability, which are directly linked to an increase in the capacity of wood elements to be penetrated with preservative products (Hansmann *et al.* 2002; Hess *et al.* 2021; Mascarenhas *et al.* 2021).

Notwithstanding the fact that microwave treatment of wood has benefits and has been becoming more popular in the past decades, there remains a scarcity of research exploring the effects of applying microwave energy on maritime pine, especially in three distinct areas. First, the alterations in the treatability of MW-treated pine samples, *i.e.*, their capacity to be impregnated with protective products, have not yet been assessed. Second, unlike other wood modification techniques (thermal and resin impregnation, for example), no studies in the literature examine the dimensional variations and hygroscopic behavior of MW-treated samples of maritime pine or any other wood species. Third, there is still little information about how maritime pine elements are affected by MW energy regarding their mechanical properties. Therefore, those aspects require further extensive studies and analyses to fully comprehend the impacts and benefits of MW methodology in maritime pine wood specimens to support the adoption of MW technology and the use of MW-treated wood pieces with structural functionalities.

Considering the hypothesis that MW treatment can make wood elements become more porous, and consequently, modifications in the hygroscopicity and improvements in the impregnability might happen, two different MW treatment configurations were applied to Portuguese maritime pine specimens. Therefore, acknowledging the benefits of MW treatment and the lack of technical and scientific information, the objective of this research work was to enhance the treatability (impregnability) of Portuguese maritime pine by drying the samples using MW energy and investigate the hydrophobicity (by the evaluation of the weight of water absorbed, WA), the water repellence efficiency and anti-swelling efficiency of the samples with and without MW drying. Finally, it was aimed to identify the feasibility of MW energy in treating maritime pine wood specimens. Although the investigation of a wood species' mechanical properties after MW treatment is significant, it is not within the scope of this paper.

EXPERIMENTAL

Materials

Maritime pine heartwood samples with no defects from Portugal were sourced from commercially available planks. The used specimens totalized thirty-six, with dimensions of 20 mm (R) × 20 mm (T) × 320 (L) mm (Fig. 1).

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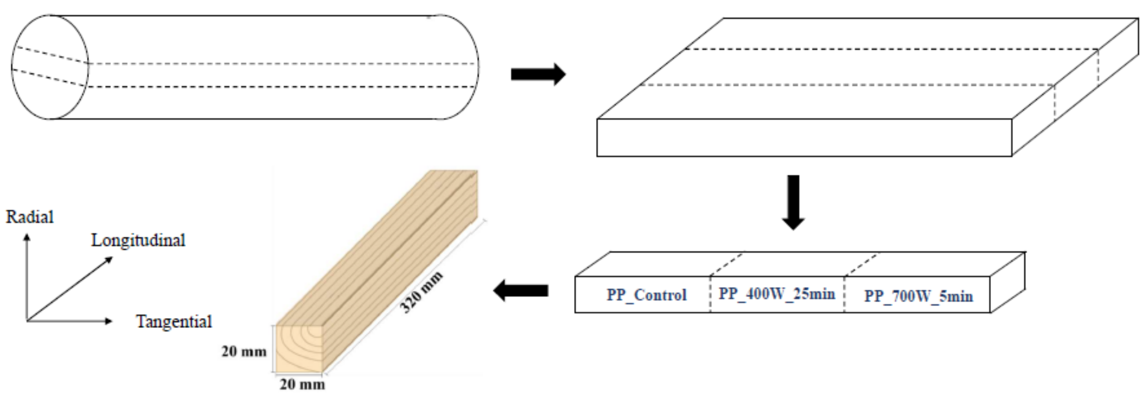

Fig. 1. Dimensions of the wood samples of maritime pine used in this study. R – Radial; T – Tangential; L –Longitudinal

MW Drying

The small wood samples were placed into groups with 12 wood specimens each: PP_Control (with no MW treatment, control group), PP_400W_25min, and PP_700W_5min (with MW drying). The term "PP" denotes *Pinus pinaster*. The methodology used was based on what Mascarenhas *et al.* (2023b) did, with some adaptations for their work. Two distinct MW treatment settings (parameters) were employed (Fig. 2).

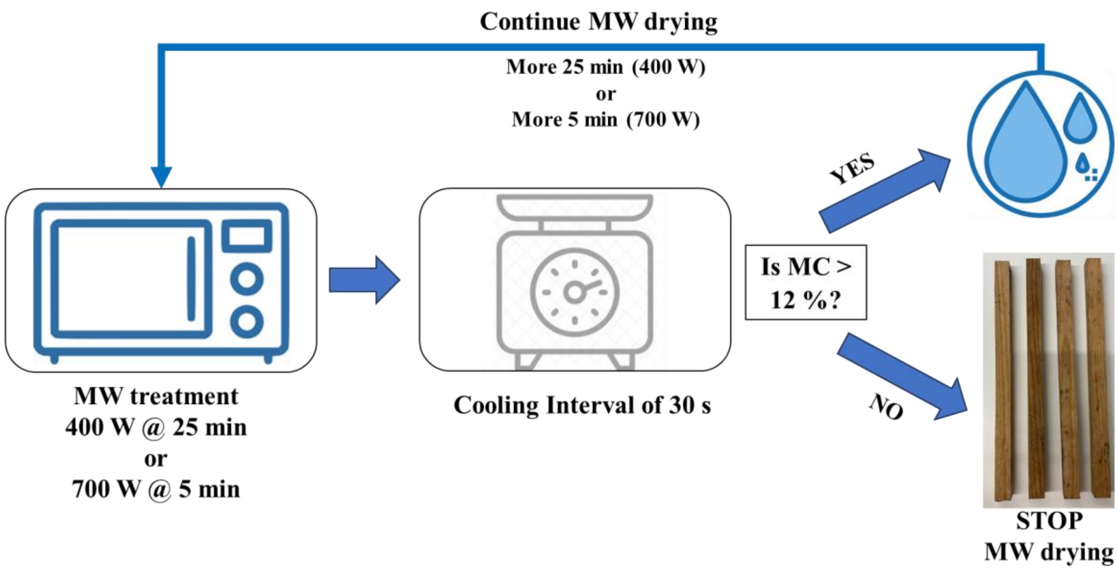

Fig. 2. MW treatment. The volume of wood samples MW-treated in each cycle was 0.000512 m^3.

The first setting employed a MW power of 400 W and a MW treatment cycle time of 25 min (continuous exposure), named PP_400W_25min, and an initial moisture content (MC: mass of water/mass of dried wood) of 95.4%. Conversely, the second setting employed a MW power of 700 W and a continuous exposure time of 5 min, named PP_700W_5min, and an initial MC of 95.5%. The samples of PP_Control were not subjected to MW treatment; instead, they were dried in a conventional oven with forced circulating air.

The maritime pine specimens underwent treatment in a traditional microwave oven equipped with a rotating platform, 2.45 GHz, and capable of delivering up to the top 1200 W (Fig. 3). Then, in order to prepare the pine samples for the preservation product impregnation procedure, the control and MW-treated groups' samples were dried in a traditional drying oven with an air circulation system until they reached absolutely dry condition.

Fig. 3. MW oven

The employed energy during the process (E) (Eq. 1) and the rate of MW drying (dr) were estimated as done by Mascarenhas *et al.* (2023b).

$$E = \frac{P \cdot t}{V \cdot 10^6} \quad (1)$$

In Eq. 1, E is the specific energy provided in the entire MW drying (MJ/m^3); P is the power provided by the MW (W); V is the volume of wood samples during each MW drying (m^3); and t is the total MW treatment time (s).

Impregnation Evaluation

The water-soluble chromium and arsenic-free wood preservative solution Tanalith E 8001 containing copper–and other compounds (Table 1) was used (YOU Solutions Germany GmbH 2023).

Table 1. Chemical Composition of the Preservative Product Solution

Compound name according to IUPAC[1]	CAS Number[2]	Content (% w/w)
Copper(II) carbonate- copper(II) hydroxide (1:1)	12069-69-1	14.57
1-(4-chlorophenyl)-4,4- dimethyl-3-(1,2,4-triazol- 1-ylmethyl)pentan-3-ol	107534-96-3	0.16
1-[[2-(2,4-diclorofenil)-4-propil-1,3-dioxolan-2-il]metil]-1H-1,2,4-triazole (propiconazole)	60207-90-1	0.16
Didecyldimethylammonium chloride (DDAC)	7173-51-5	0.50
Didecyl dimethyl ammonium carbonate (DDA Carbonate) Reaction mass of N,Ndidecyl-N,Ndimethylammonium carbonate and N,Ndidecyl-N,Ndimethylammonium bicarbonate	894406-76-9	0.50
2-aminoetanol	205-483-3	26.91

[1]International Union of Pure and Applied Chemistry; [2]CAS: Chemical Abstracts Service.

The pine exemplars were impregnated with a 5% water-soluble solution of the preservative product with an industrial vacuum-pressure process at "*Pinhal Nova Madeiras Tratadas*". First, a vacuum of -0.8 bar was applied for 30 min, and then a pressure of 15 bar was applied for 1 h 15 min, and finally, more 30 min of vacuum. This is the procedure that the industry uses to treat maritime pine. Before and after the impregnation process, the wood samples were weighted, and the values were used to calculate the retention and uptake done by Kol and Çayır (2022);

The absolutely dry densities of the impregnated samples were determined following the procedures established in ISO 13061-2 (ISO 2014) in wood specimens in the dimensions of 20 mm (R) × 20 mm (T) × 30 mm (L).

Dimensional Stability

The hydrophobic characteristics of the wood samples were determined using samples of 20 mm (R) x 20 mm (T) x 30 mm (L) through the calculation of water absorbed by weight (WA) and the rate at which water was absorbed (AR) (Mattos *et al.* 2014; Saiful Islam *et al.* 2012).

$$WA = \frac{(W_f - W_i)}{W_i} \times 100 \quad (2)$$

$$AR = \frac{(W_f - W_i)}{(t_f - t_i)} \times 100 \tag{3}$$

In the above equations, W_f is the weight of the wood specimens at the time t_f, in g; W_i is the weight of the wood specimens at the time t_i, in g; t_f is the time in which the samples were measured, in h; and t_i is the time in which the measurements started (zero).

The *WA* and *AR* profiles were established by determining the weight of the samples at specific periods (2, 4, 8, 24, 48, 72, and 96 hours) during immersion in fresh water to generate a comprehensive absorption kinetics analysis. After 96 h, the samples were dried until constant weight using a forced convection oven at 103±2 °C. This procedure was repeated four times (4 cycles) (Mattos *et al.* 2014). The values of water repellence efficiency (WRE) and anti-swelling efficiency (ASE) and swelling percentage (S) were evaluated during successive four oven-dry and water-soak cycles (Ding *et al.* 2012; Magalhaes and Da Silva 2004; Mattos *et al.* 2014; Saiful Islam *et al.* 2012; Sargent 2019).

$$WRE = \frac{(\Delta W_{ut} - \Delta W_t)}{\Delta W_{ut}} \times 100 \tag{4}$$

$$\text{Where: } \Delta W = \frac{(W_{sat} - W_{dry})}{W_{dry}} \times 100 \tag{5}$$

$$ASE = \frac{(S_{ut} - S_t)}{S_{ut}} \times 100 \tag{6}$$

$$\text{Where: } S = \frac{(V_{sat} - V_{dry})}{V_{dry}} \times 100 \tag{7}$$

In the above equations, S is the swelling percentage; V_{sat} is the volume of saturated wood specimens, in cm^3; V_{dry} is the volume of dry wood specimens, in cm^3; ut refers to the control specimens (untreated); t refers to the MW-treated specimens; ΔW is the weight variation percentage; W_{sat} is the weight of saturated wood specimens, in g; and W_{dry} is the weight of dried wood specimens, in g.

Statistical Analysis

This research investigated the alterations in the retention of the preservative product, the absolutely dry densities, and the water hygroscopic parameters, namely *WA*, *S*, *AR*, of wood specimens from groups PP_Control, PP_400W_25min, and PP_700W_5min, by employing the classical analysis of variance (ANOVA) at a 5% significance level. Based on ANOVA and the Tukey test, the samples from the control and MW-treated groups can be regarded as distinct when the p-values are less than the level of significance (p-value < 0.05).

The correlations between the amount of retained preservative product (ARPP) and density, ARPP and WA, ARPP and density, AR and density, AR and WA, and density and WA were done using the Pearson correlation coefficient (r). The significance of the correlations was done using ANOVA. The Minitab software was utilized for the purpose of conducting statistical analysis (Minitab 2018).

RESULTS AND DISCUSSION

Analysis of the MW Treatment Process

The results concerning the microwave process are given in Fig. 4. Although both wood groups had similar values of initial MC (95.4% for PP_400W_25min and 95.5% for PP_700W_5min), the intensification of the MW power from 400 to 700 W provoked a drastic increase in drying rate by 3.4 times, and, in consequence, a reduction in the total drying time from 110 min to 35 min. Regarding the samples dried with 400W and 25 min, it is important to explain that the last drying cycle lasted only 10 min (instead of 25 min), totaling 110 min. The intensification in the power of MW treatment may have increased the vapor pressure within the wood samples, enhancing the mass transfer rate.

The applied energy was decreased by around 50% with the rise in MW power. The average applied energy to group PP_400W_25min was 5153 MJ/m^3, and for group PP_700W_5min, it was 2871 MJ/m^3. Two factors can explain it. First, the overall time decreased as the MW power increased. Since the MW setup with 400 W required a longer time, more energy was applied to the wood samples, acknowledging that the larger the overall duration, the higher the applied energy will be (Eq. 1). The efficiency of the drying process is another consideration. A tentative explanation for the lower drying efficiency of the PP_400W_25min conditions, compared with the PP_700W_5min, might be that the water evaporation/condensation cycles occurring inside the wood sample at this power and/or the vapor pressure generated inside the sample was not enough to expel water in liquid phase from the inside of the wood to the outside, which can occur at higher vapor pressure.

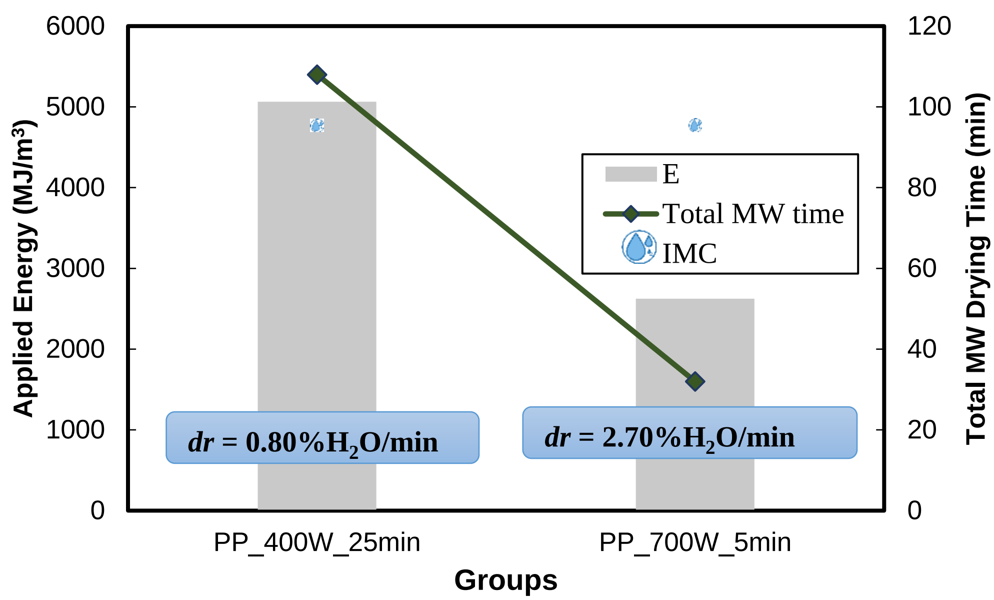

Fig. 4. MW treatment results for maritime pine heartwood samples. (E – Average Applied energy; Total MW time – Average Total MW drying time; IMC – Average Initial Moisture Content)

Absorption of the Preservative Product and Densities of Wood Samples

In Fig. 5, the maritime pine wood samples that underwent MW treatment (a) before and (b) after impregnation with the preservative product. The greenish appearance of the impregnated wood samples can be attributed to the presence of the preservative product.

Fig. 5. Pine wood pieces after MW treatment (a) before and (b) after impregnation with the preservative product.

The preservative product retention results for the studied maritime pine wood samples are shown in Fig. 6. The MW-treated groups PP_400W_25min and PP_700W_5min were statistically different from the PP_Control. The MW-treated retained 94% more preservative product than the control specimens. Then, it is evident that there has been a significant enhancement in the ability of the MW-treated specimens to be impregnated.

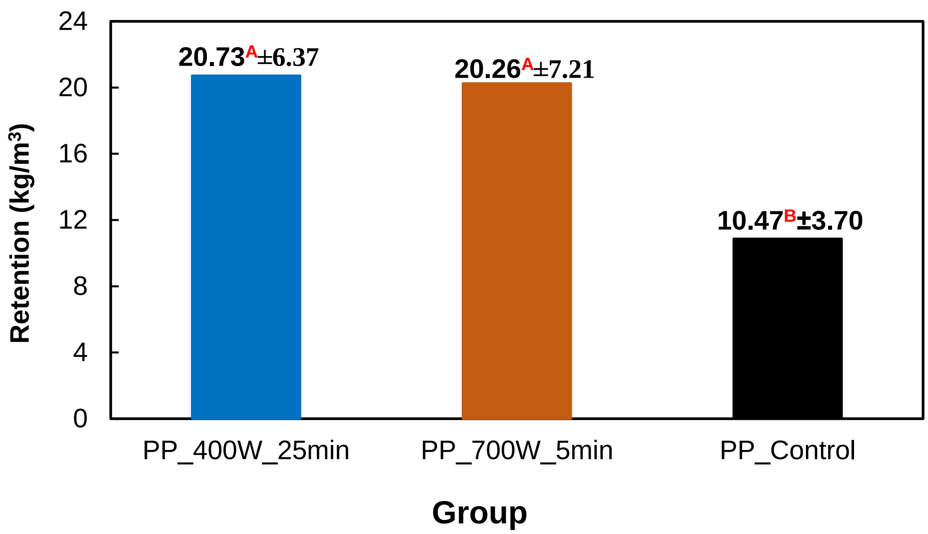

Fig. 6. Retention of Tanalith E 8001 of maritime pine samples. From Tukey tests, values preceded by different letters differ significantly at p-value=0.05

Table 2. Amount of Preservative Retained by the Wood Elements for Each UC

UC	MC of wood (%)	The appearance of xylophage agents[1]			Retention (kg/m³)[2]	
		Fungi	Termites	Bettes	Minimum	Maximum
1	≤ 20				8.50	18.75
2	Occasionally > 20				8.50	18.75
3	Frequently wet > 20				8.50	18.75
4	Predominantly or Frequently > 20				17.20	31.25

[1]Regarding the situation in Portugal, the risk level is categorized as follows: **Red** indicates a very high level of risk, **Orange** a high level of risk, **Yellow** a low level of risk, and **Green** no risk (CEN 2013b; Lima et al. 2022). [2]The minimum and maximum quantity of the used preservative product for each UC according to the manufacturer (YOU Solutions Germany GmbH 2023).

Table 3. Water Absorption after 4 Cycles of Testing

Groups	WA_{96h}^{4}(%)	Std. Dev. (%)	IC (%)	S_{96h}^{4}(%)	Std. Dev. (%)	IC (%)
PP_Control	58.46^{A}	9.63	(45.41; 71.51)	9.08^{A}	1.74	(7.59; 10.57)
PP_400W_25min	62.37^{A}	9.90	(55.96; 76.77)	6.49^{B}	2.17	(5.18; 7.55)
PP_700W_5min	55.42^{A}	9.60	(50.75; 69.89)	7.84^{AB}	1.78	(6.75; 8.93)

WA_{96h}^{4} is the average water absorbed by weight at 96 h, of the 4 cycles of soaking-drying; Std. Dev. is the Standard Deviation; IC is the Interval of Confidence; S_{96h}^{4} is the average swelling at 96 h, of the 4 cycles of soaking-drying.
From Tukey tests, values preceded by different letters differ significantly at p-value=0.05.

Although the power used to treat the PP_400W_25min group was lower than that used for the PP_700W_5min group, the energy applied to the first group was double. Therefore, it can be hypothesized that the higher cumulative value of energy applied to the PP_400W_25min group compensated for the lower power applied, and, as a result, the retention values of the preservative product were similar. However, further investigations using scanning electron microscopy (SEM) are necessary to deeper understand the effects of MW in the microstructure of wood.

The identification of the suitable method for preserving wood can be determined by referring to the EN 599-1 standard (CEN 2013a), which outlines the five different Use Classes (UC) as defined in EN 355-1 (CEN 2007). The technical documentation of the preservative product states that it can be applied to protect solid wood or wood-based elements utilized in UC 1 to 4. For each UC, the wood element must have a range of absorption of preservative products to protect against fungi and termites, as demonstrated in Table 2 (YOU Solutions Germany GmbH 2023).

The densities of heartwood samples of PP_Control retained 10.47 kg/m^3, allowing them to be applied in UC 1, 2, and 3 since the value they absorbed was higher than the minimum required. Because the heartwood samples of PP_400W_25min and PP_700W_5min retained, on average, 20.73 and 20.26 kg/m^3, they could be applied not only in UC 1, 2 and 3 (with a great margin of safety), but in circumstances of UC 4, for situations where there is proximity or direct contact between wood and the ground, wherein the wood remains consistently saturated with moisture (specifically fresh water).

It is necessary to remark that the impregnation process used in this work consisted of applying high pressures and vacuum-pressure cycles for long periods, and the wood industry continuously seeks methods to lower costs, aiming to achieve cost-effective and environmentally sustainable wood treatments (Teacă *et al.* 2019). Therefore, given the great results of the MW-treated samples in terms of retention of the preservative product, the possibility of making changes to the impregnation process is open, either by reducing the applied pressures or the impregnation time, for example. Such changes can lead to reductions in the costs associated with the impregnation process itself.

Regarding the uptake rise of the preservative solution of maritime pine samples, the MW treatment increased, on average, by 71%. MW energy was used to dry sapwood samples of Oriental spruce (*Picea orientalis* (L). Link.). Kol and Çayır (2022) observed a 61% heightening of the absorption of the preservative solution. Using four different preservative products, Samani *et al.* (2019) impregnated *Melia composita* wood samples. They found improvements in the uptake of the MW-treated samples that ranged from 7 to 215%, depending on the used preservative solution.

From the practical point of view, the improved ability of MW-treated maritime pine samples to be impregnated with preserving agents deserves special attention. In Portugal, for example, maritime pine constitutes the primary source for the production of wood utility poles, and they require long periods of durability in order to reduce the need to replace and/or reapply the product (Marques *et al.* 2016; Morgado *et al.* 2009). Although the heartwood of maritime pine is durable for *Hylotrupes* and *Anobium*, and moderately to slightly durable against fungi, it is not durable against termites, requiring protective measurements against them. Nevertheless, maritime pine heartwood is extremely difficult to be treated, which can also be an issue when choosing it for specific applications (CEN 2016). Therefore, the results found here (Fig. 6) show that it is easier to impregnate maritime pine specimens containing only heartwood after they have been MW-treated.

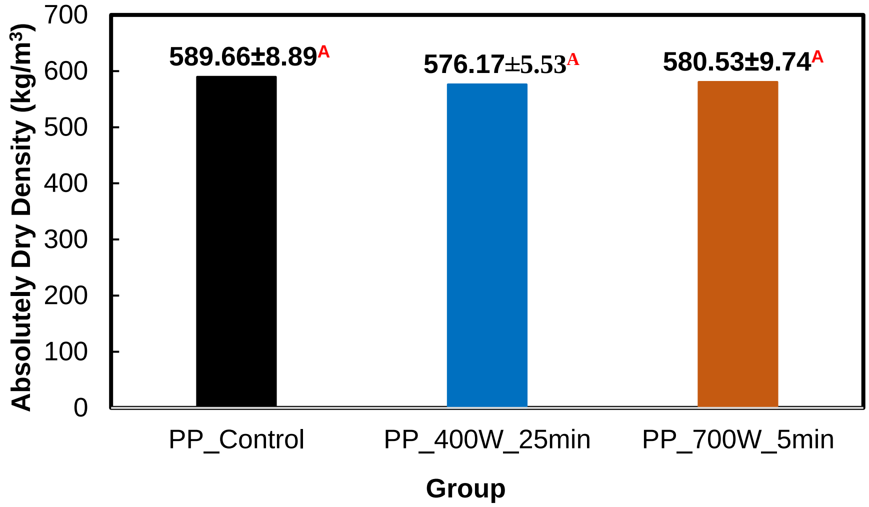

Fig. 7. Absolutely dry densities of control and MW-dried samples. From Tukey tests, values preceded by different letters differ significantly at p-value=0.05.

Regarding the absolutely dry densities subsequent to the impregnation procedure, slight reductions were observed (approximately 2% only), and the densities of three wood groups, PP_Control, PP_400W_25min, and PP_700W_5min, were statistically equivalent (Fig. 7). Similar findings were provided by He *et al*. (2023), which applied MW energy to Scots pine (*Pinus sylvestris* L.); the decreases in the densities of samples treated with MW were less than 1%, but the authors identified moderate increases in the porosity MW-treated samples. Other authors also reported similar results of small decreases in densities using maritime pine (Mascarenhas *et al.* 2023b), red Stringybark (*Eucalyptus macrorhyncha*) (Balboni *et al.* 2018), Chinese fir (*Cunninghamia lanceolata*) (He *et al*. 2014); and Douglas-fir (*Pseudotsuga taxifolia*), mountain ash (*Eucalyptus regnans*) and Paulownia (*Paulownia fortunei* and *Paulownia elongata*) (Torgovnikov and Vinden 2009).

When looking at the relationship between the density of control and MW-dried samples and the amount of preservative product retained, a negative, medium to strong value was identified, r = -0.540 (p-value = 0.004). The greater the density, the lower the retention of the preservative solution. Wood porosity, an attribute influenced by wood's anatomy and with direct influence in wood's density, generally governs the extent to which products like chemical preservatives can permeate the wood (Forest Products Laboratory 2021). The MW treatment causes only a very slight decrease in density. However, the huge increase in preservative retention and solution uptake strongly support a possible significant increase in wood permeability. Research conducted by Weng *et al.* (2021) utilizing SEM evidenced that MW drying resulted in an augmentation of impairments to the microstructure of wood. MW applied energy prompted the creation of multiple voids within the wood, thereby altering its mechanical and physical properties such as permeability and porosity (Torgovnikov and Vinden 2009).

Analysis of the Hygroscopicity and Dimensional Stability

The water absorption, WA of control and MW-treated samples were measured, and the results are presented in Table 3. The three wood groups were statistically equal regarding water absorption at the end of 4 testing cycles, WA^4_{96h} (Table 3), whereas the average WA of PP_400W_25min was 7% higher and the PP_700W_5min was 5% smaller than PP_Control. In contrast, the control group experienced the greatest swelling, S^{4}_{96h}, which was statistically distinct from the ones treated with MW. This finding is particularly noteworthy, as it revealed the ability of MW treatment to diminish the dimensional variations of the wood samples. There is no result in the literature involving MW drying to compare. This might be associated with modifications in the chemical composition of MW-treated samples, which were not evaluated in this work. However, it deserves to be further investigated in detail because Wang *et al.* (2022) and Xing *et al.* (2023) indicated that the chemical composition and structures of the wall of the cells had changed.

Regarding the water absorption, after 2 h of soaking, PP_Control showed a WA of 15% and the MW-treated samples of 20% (Fig. 8). In the first 24 h, the control and MW-treated samples had WA of 37 and 42%, respectively. In terms of the behavior of WA, it can be observed that the three groups had the same pattern.

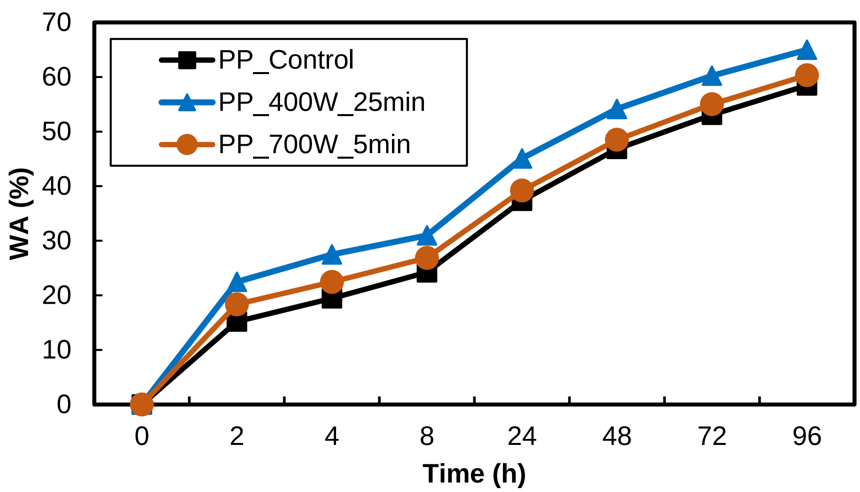

Fig. 8. Water absorption (WA) of control and MW-treated maritime pine heartwood samples

Contrary to what was observed in the retained Tanalith E values, the WA values of the MW-treated and untreated samples were similar. It is important to note that the methods used to impregnate with the preserving agent and the methodology used to carry out the WA tests were different. While WA assessment involves cycles of immersion in distilled water and drying the samples without applying any external pressure, treatment with Tanalith involves a high pressure (15 bar), which can make all the difference. Therefore, one can infer the hypothesis that WA tests cannot be used to predict impregnability, but only what it is intended for, which is water absorption.

The peak rate of water absorption (AR) happened in the initial 2 h of the soaking process (Fig. 9). Although the AR of PP_400W_25min and PP_700W_5min exhibited greater values compared to PP_Control during the initial 2-h period, the three wood groups demonstrated statistically equivalent AR (Table 4) (p-value < 0.05). After the initial 24-h period, the average AR values of the three different wood groups were similar throughout the duration of the immersion procedure. Furthermore, the average AR at the 96-h mark exhibited a very close value for all three wood groups.

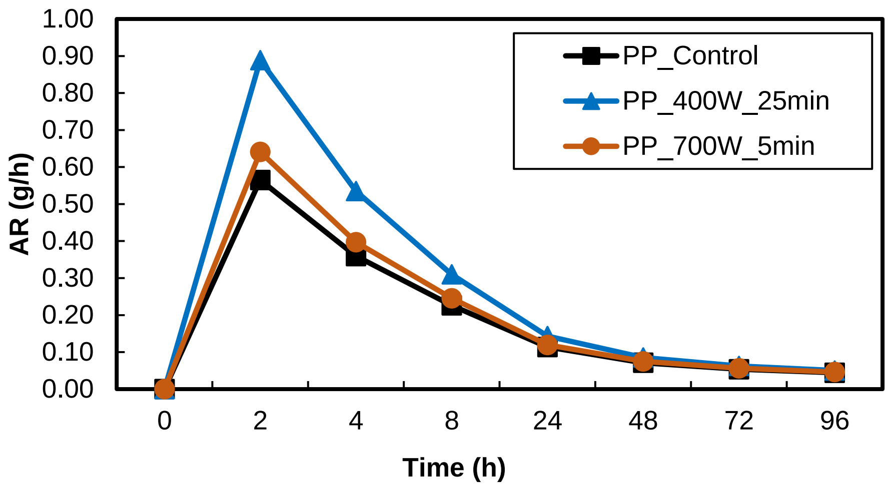

Fig. 9. Water absorption rates of control and MW-treated maritime pine heartwood samples

Table 4. Water Absorption Rate after 4 Cycles of Testing

Groups	AR_{2h}^{4}(g/h)	Std. Dev. (g/h)	IC (g/h)	AR_{96h}^{4}(g/h)	Std. Dev. (g/h)	IC (g/h)
PP_Control	0.57^{A}	0.25	(0.14; 0.99)	0.04^{A}	0.01	(0.04; 0.05)
PP_400W_25min	0.89^{A}	0.58	(0.55; 1.22)	0.05^{A}	0.01	(0.05; 0.06)
PP_700W_5min	0.64^{A}	0.61	(0.33; 0.95)	0.05^{A}	0.01	(0.04; 0.05)

AR_{2h}^{4} is the average water absorbed rate by weight at 2 h, of the 4 cycles of soaking-drying; AR_{96h}^{4} is the average water absorption rate by weight at 96 h, of the 4 cycles of soaking-drying. From Tukey tests, values preceded by different letters differ significantly at p-value=0.05.

When performing correlation analysis using the MW-treated samples with WA and the other studied variables, WA had positive-strong Pearson correlation coefficients with AR, $r = 0.967$ (p-value = 0.000). A positive-strong correlation coefficient was found between the uptake of the preservative solution and WA, $r = 0.782$ (p-value = 0.000), which indicates that the higher the water absorption capability, the higher the retention of the preservative product and the water retention rate.

The Pearson correlation coefficient demonstrated a positive-strong relationship between the AR at 2 h, of the 4 cycles of soaking-drying and the retention of the preservative product, $r = 0.804$ (p-value = 0.000), suggesting that the faster the water absorption rate at 2 h, the more preservative product was incorporated. On the other hand, there was no statistically significant correlation between the average water absorption rate by weight at 96 h, of the 4 cycles of soaking-drying and the amount of retained preservative product. In addition, a negative-strong correlation, $r = -0.732$ (p-value = 0.000), was identified between the AR at 2 h and the density. On the other hand, no statistically significant correlation was found between AR at 96 h and the swelling, S, neither between the MW power and WA and MW power and S (p-value > 0.05).

Finally, the WRE and ASE results are shown in Table 5 and Fig. 10. Both MW-treated wood groups had different behavior for WRE and ASE. Unlike other wood modification processes, such as thermal and chemical treatments, in which the behavior of various species with regard to WRE and ASE is widely known and disseminated, this is a field of knowledge that is still in its infancy and requires a great deal of study when it comes to MW-treated wood.

Table 5. WRE and ASE of MW-treated Wood Samples

Groups	ASE^4 (%)	Std. Dev. (%)	WRE^4 (%)	Std. Dev. (%)
PP_400W_25min	28.51	7.39	-6.70	2.67
PP_700W_5min	13.67	5.16	5.20	1.56

ASE^4 is the average water repellence efficiency of the 4 cycles of soaking-drying; WRE^4 is the average water repellence efficiency of the 4 cycles of soaking-drying.

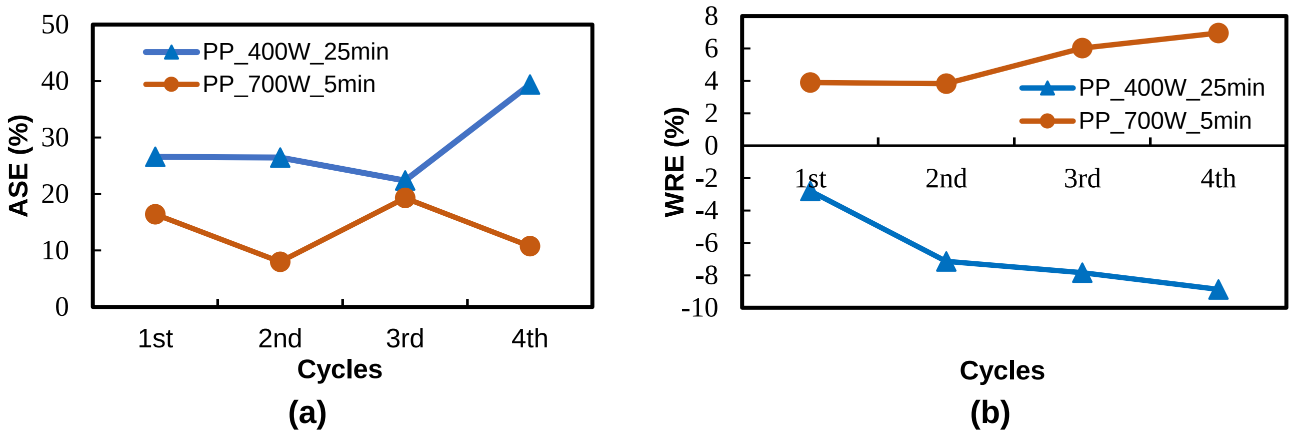

Fig. 10. Anti-swelling and Water repellence efficiency of MW-treated maritime pine heartwood samples, in comparison with the non-MW-treated wood

When assessing the ASE of the MW-treated samples, it is possible to see that both groups had good results, mainly the wood samples of group PP_400W_25min, which had an ASE of approximately 29%. The findings indicated that the application of MW treatment possibly yielded encouraging outcomes in enhancing the dimensional stability of the MW-treated pine wood specimens in comparison to the control samples. This behavior can tentatively be attributed to a process known as hornification, occurred in the fibers of the pine specimens in the MW treatment and after successive wetting and drying cycles. In this process, the capillary voids between the fibril in the fiber are progressively closed with drying and can no longer be completely reopened with rewetting (Fernandes Diniz *et al.* 2004). The role of the exposition time to the MW treatment on the ASE required further research and can justify the superior performance of the PP_400W_25 min samples.

Works from the literature that applied different methods to improve the ASE of wood specimens also identified improvements in the ASE. Pointing out three of them, Magalhaes and Silva (2004) studied the influences of chemically modified samples of Caribbean pine (*Pinus caribaea* var. *hondurensis*) in the ASE property, Mattos *et al.* (2014) also investigated the effects of chemical modification using polymer in the ASE property of *Pinus taeda* wood samples, and Tarmian and Mastouri (2018) thermally treated heartwood of oak (*Quercus castaneifolia*), hornbeam (*Carpinus betulus*), and poplar (*Populus nigra*) wood samples. All those works indicated that the ASE of the samples were higher in the first cycle of the soaking/drying process, and it decreased along the soaking/drying cycles; *i.e.*, ASE was better in the short period. The ASE of the MW-treated samples in this work did not display the same pattern. Overall, the ASE rose as the soaking/drying cycles increased.

In contrast to PP_700W_5min wood samples, which had a positive average WRE value equal to 5.20%, the PP_400W_25min wood samples showed a decrease (WRE = -6.70%), meaning that they absorbed more water than the reference group. A tentative explanation for these results can be higher loss of resin for the prolonged MW treatment in the PP_400W_25 min assay.

In this study, the effect of the MW treatment on the resin release was observed visually in the surface of the samples. Although it was not known what the initial resin content of the specimens was, when the specimens were visually examined before and after the MW treatment, it was noted that those in the PP_400W_25min group had lost on average more resin than those in the PP_700W_5min group. It is hypothesized that the longer time (110 min) removed more resin, while the higher power (35 min) did not remove as much resin because the time was limited. The slightly lower density of the PP_400W_25 min samples (Fig. 7) and the successive decrease of the WRE with the impregnation/drying cycles can suggest some cellulosic polymer degradation and water extraction.

In fact, recent studies made by some authors (Wang *et al.* 2022; Xing *et al.* 2023) have reported the effect of MW treatment on the polar wood components, such as cellulose, hemicelluloses, and lignin, pointing out that besides the role of permeability in the hygroscopicity and dimensional variations of wood, the wood chemical components also play an important role (Banks and Levy 1980). Hence, modifications in those three wood components might have happened, thus resulting in the WRE and ASE profiles of both groups of MW-treated maritime pine wood. Based on this, although tests to verify hornification, alterations in the wood's chemical constituents, and SEM images are not within the scope of this work, they are recommended in order to obtain more detailed and in-depth information.

These results deserve special attention because they bring novel insights regarding the dimensional stability and water absorption behavior of MW-treated samples and because, to date, there are no similar studies in the literature that present results that can be compared with those presented here. Hence, further studies are needed to identify better the consequences of MW drying on WRE and ASE of wood samples of different wood species.

Finally, the remarkable improvement in preservative retention, the stability in WRE (the possible increase in permeability did not lead to a worsening of this property), and the enhancement in ASE, all of them due to the MW treatment, can be very beneficial for improving the utilization of MW technology and the incorporation of maritime pine wood species in the construction industry. The possible cost savings from a simpler impregnation process, combined with the attributes of MW technology, both the benefits for the wood element and the ecological and cost-saving performance of the process, make it particularly attractive. Thus, MW treatment is a feasible and promising technology in the field of wood modification, mainly when it comes to improving the treatability of wood species.

Because the hygroscopicity and dimensional variations of wood samples that underwent microwave technology are topics that still need further analysis, additional studies that evaluate those properties in other wood species are recommended, applying different combinations of microwave power and time. Also, investigations about the chemical composition of MW-treated samples are required, since it is a field with large possibilities for research.

CONCLUSIONS

1. Regarding impregnability, the retention of the preservative solution of microwave (MW)-dried specimens for both settings increased significantly compared to the wood specimens that were not MW-treated. Based on the information from the preservative product manufacturer and the retention values, the wood samples could be used in the circumstances of use classes (UC) 1, 2, 3, and 4. However, durability tests are highly recommended to get a more complete understanding and a more accurate response in terms of durability.
2. The impact of microwave energy on the wood density was negligible. MW-treated elements had minor reductions compared to control ones, and the densities were statistically equivalent.
3. The water repellence efficiency (WRE) of the MW-treated samples was not impaired in comparison with the non-treated samples despite the significant enhancement in the water absorption (WA) of these samples.
4. The anti-swelling efficiency (ASE) of MW-treated specimens showed an increase, meaning that the MW-treated samples had a smaller swelling coefficient than the control samples. Hence, MW technology was effective in reducing the dimensional variations of the pine wood samples.
5. MW treatment showed itself to be a feasible and promising technology in the field of wood modification, mainly when it comes to improving the treatability of wood species, with advantages for the wood and consequently to the wood science and engineering fields.

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