Permeability of Wood Impregnated with Polyethylene Wax Emulsion in Vacuum

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Abstract

Polyethylene wax (PEW) has emerged as a commonly used material for maintaining the quality of wood. The emulsification of solid PEW before being injected into wood can effectively overcome the shortcomings of the high-temperature melting and injection process of solid PEW. The PEW emulsion permeability of Eucalyptus (*Eucalyptus saligna*) and Mongolian Scotch pine (*Pinus sylvestris* var. *Mongolica*) in vacuum environment was investigated using impregnation tests. Steaming pretreatment and microwave irradiation pretreatment were applied. The small particle size and low viscosity of the PEW emulsion facilitated its penetration into the wood capillary system. The weight percent gain (WPG) of Eucalyptus and Mongolian Scotch pine post impregnation in vacuum was positively correlated with both the vacuum-holding time and the impregnation temperature, but after 60 min of impregnation the WPG of both wood species decreased significantly. Both pretreatment methods could substantially improve the wood permeability and effectively promote the penetration of the PEW emulsion into the wood. For steaming, Eucalyptus steamed for 80 min and Mongolian Scotch pine steamed for 120 min gained the maximum WPG. For microwave irradiation, both species exhibited the highest WPG after water immersion for 60 min and microwave irradiation for 30 s. There was a significant variation in WPG between the two species with the same pretreatment method. The relatively lower density and better-developed capillary system of Mongolian Scotch pine allowed more efficient penetration of PEW emulsion. It is recommended that, considering the time cost and practical enhancement, microwave irradiation should be given priority to when pretreating wood.

1 Introduction

With the development of wood industry technology and analysis methods for safety, engineered wood products such as glulam, cross laminated timber (CLT) and laminated veneer lumber (LVL), have made high-rise wood structures and large-span wood structures into reality, and also endow the buildings with designability for shapes and functions (Crews et al. 2011; Tupenaite et al. 2021; Wang et al. 2022). In recent years, China has been vigorously developing rural revitalization. Wood landscapes such as timber kiosks, timber railings and timber walkways not only adds interest to the countryside, but also help improve the wood application with high additional value. However, wood is a kind of biomass material, which is prone to crack due to uneven dry shrinkage and be attacked by fungi. Shrinkage cracking and fungal decay seriously decrease the durability of wood in outdoor moist conditions (Patera et al. 2013; Thybring et al. 2018). In order to improve the durability of wood, various methods have been used to treat wood. In terms of biocidal treatment, preservative treatment is the most commonly used process for outdoor wood products that make wood resistant to fungi and termites. Traditional water-borne preservatives, which contain toxic substances such as chromium and arsenic, have been generally replaced by alternative preservatives such as alkaline copper quaternary (ACQ), copper azole (CuAz), micronized copper quaternary (MCQ) and boron-based preservatives (Janin et al. 2011; Caldeira et al. 2010). Organic solvent-borne preservatives are also an application trend (Yerman et al. 2021). According to Chinese standard GB/T 27651 – 2011 (2011), preservative-treated wood can be generally divided into five grades from C1 to C5 depending on use classes. C3 to C5 are for outdoor use, and retention and penetration are two key indicators. For example, the retention and sapwood penetration of MCQ treated wood components in key structures that contacts with soil or is immersed in fresh water (namely C4.2) should reach 9.6 kg/m³ and 95%, respectively. Another way to improve durability is to reduce moisture absorption. Thermally modified wood is one of the best-established modified wood products (Hill 2006). Stamm et al. (1946) developed the Staybwood process, and it improved the hygroscopicity of wood. Burmester (1973) published the FWD (Feuchte, Wärme und Druck) process, which endowed the wood with a lower mass loss and better mechanical properties than Staybwood. Nowadays there have been some commercialized mature processes. Mass loss is one of the most important features in thermal modification, and it depends on wood species, heating medium, temperature, and treatment time (Esteves and Pereira 2009). Thermal modification inevitably weakens wood bending strength to a certain extent (Gunduz et al. 2009; Arnold 2010; Bakar et al. 2013). According to Chinese standard GB/T 31747 – 2015 (2015), thermally modified wood for outdoor use should not contact with water and cannot be used for load-bearing.

Wax is excellent material for the quality protection of wood and wood products. There are various types of wax, including natural and synthetic wax, which are generally stable and hydrophobic. In the Ming Dynasty, Chinese craftsmen had already applied the process of melting wax to apply beeswax to the surface protection of wood furniture (Wang 2008). Polyethylene wax (PEW) is a synthetic mineral wax in the form of small white beads or flakes. Compared to natural wax, PEW has a higher melting point and has advantages in stability, wear resistance and hardness (Lang et al. 2022). Yang et al. (2017) used a 3-stage wax injection process of evacuation-high pressure impregnation-pressure release and cooling to inject molten PEW into Norway spruce (Picea abies), White spruce (Picea glauca), Mongolian Scotch pine (Pinus sylvestris var. Mongolica), New Zealand pine (Pinups radiata) and Eucalyptus (Eucalyptus saligna), and the wax impregnated wood samples were found to have higher MOE, hardness, hydrophobicity, dimensional stability and UV-ageing resistance. Zhang et al. (2022) used the 3-stage wax injection process to treat lodgepole pine (Pinus contorta) and eucalyptus (Eucalyptus saligna) with molten PEW, and results showed that treated wood had a much lower moisture absorption, better weathering resistance as well as fungal resistance than untreated ones. PEW tend to fall off due to the lack of reactive functional groups in the main chain of synthetic organic wax molecule that interact with the polar groups of the wood fibers (Niu and Song 2021). To address this problem, the researchers used PEW modified by graft maleic anhydride methyl acrylate (PEW-g-MAH-MMA) to hot waxed wood, the modified PEW filled the wood cell lumina and formed a continuous wax layer on the wood surface. The results showed that the best coating property of PEW was achieved with wood hot waxed at 180°C for 40 minutes, demonstrating better gloss, adhesion and thermal stability than that treated with beeswax.

The melting point of PEW is high and the process of high-temperature melting and injection is often complex, requiring high-performance equipment and consuming a lot of energy and time. The high temperature tends to cause water loss and make wood become brittle, resulting in cracking and deformation, which may result in partial loss of mechanical properties. On the other hand, the molten wax has difficulty penetrating deep into the wood and mostly remains on the surface of the treated wood

(Lesar and Humar, 2011), which still falls short of satisfactory protection. The emulsification of high melting point wax and the vacuum impregnation at a relatively low temperature is a less energy intensive and a simpler process that overcomes the shortcomings of the high-temperature melting. Current researches on wax emulsion impregnated wood mainly employ paraffin wax and some natural wax, with a few researches concerned with PEW emulsion impregnation. Lesar et al. (2011) vacuum impregnated Norway spruce (Picea abies) with different concentrations of montan wax emulsion (LGE), polyethylene wax emulsion (WE1) and oxidized polyethylene wax emulsion (WE6), and exposed them to artificially accelerated weathering for 500 cycles. LGE treated wood has the best hydrophobic effect, the lowest change in colour and FTIR, and the best resistance to weathering. Humar et al. (2016) used montan wax emulsion for the treatment of Norway spruce (Picea abies) by dipping impregnation or vacuum-pressure impregnation for 30 min, and then thermally modified the wood at 185°C to 230°C. Combing wax treatment and thermal modification gave wood better hydrophobicity along with higher resistance to fungi. Chen et al. (2020) used a paraffin/carnauba-mixed (5:5 and 7:3) wax emulsion system for the high-pressure impregnation of Cathay poplar (Populus cathayana) and found that wood impregnated with this mixed wax emulsion system demonstrated better surface hydrophobicity and dimensional stability. Promoting wood permeability to the wax emulsion is key to improving the modification quality. Permeability is usually determined by both the impregnation process and the structural characteristics of the wood itself. Impregnation treatments for wood can be divided into non-pressure treatments and pressure treatments. And the latter, which include full-cell process, modified full-cell process, empty-cell process and double vacuum process, generally produce a better level of protection (Teng et al. 2018). Wood is a porous material with an inherent degree of permeability, which is influenced by wood species, percent of late wood and grain direction (Leggate et al. 2020, Hansmann et al. 2002). Expanding or increasing the air-liquid channels between wood cells is a critical aspect of enhancing permeability. Researchers have verified that drilling, freezing, steaming and microwave irradiation can effectively improve wood permeability. Fukuta et al. (2011) manufactured Japanese cedar (Cryptomeria japonica) compressed wood by impregnating aqueous phenolic resin solutions into the heartwood through drilled holes and polymerizing it using a hot press. Results showed that resin permeation and the density of drilled holes were positively correlated, but the resin type was found to greatly affect the bending properties. Zhang et al. (2011) carried out the freezing treatment on Eucalyptus urophylla × E. grandis specimen at a temperature of -18℃ for 48 hours. Freezing was found to not only effectively destroy the pit membrane, but also freeze the water in the cell cavity to expand the diameter of the cell cavity. Steaming and microwave irradiation pretreatment are two commonly used methods of enhancing wood permeability. Mackay (1971) studied the effect of steaming treatment on water vapour diffusion in hardwoods and found that chemical changes such as recrystallisation of cellulose and expansion in the cell wall might increase permeability, but relocation of extractives and occlusions might instead worsen it. Microwave irradiation treatment is often used in wood drying and pretreatment of preserved wood, for saving time and reducing the occurrence of defects (Mascarenhas et al. 2021). Poonia et al. (2016) subjected chir pine (Pinus roxburghii) to various levels of intensity and radiation time at room temperature, the wood air permeability and the ACC preservative uptake were found to have a positive correlation to both the level of intensity and radiation time. Kol and Cayir (2021) conducted microwave treatment at 1156 MJ/m^3 and 1542 MJ/m^3 on Oriental spruce (Picea orientalis) sapwood with moisture contents of 55% and 83%. The samples with the two moisture contents increased the retention rates by 47.5% and 70% respectively. Additionally, they found that there were small changes in the mechanical properties after the microwave treatment.

The property of high melting point PEW is relatively more stable. Compared with high-temperature melting process, emulsification process is simpler and consumes less energy. If appropriate process parameters can be determined, PEW emulsion impregnated wood will be an excellent product for outdoor use. In the previous study on PEW emulsion impregnation of wood (Zhang 2022), our group applied an evacuation-high pressure impregnation-pressure release process and found that the vacuum stage, in which the negative pressure difference in the impregnation tank was used to pump enough PEW emulsion into the wood samples, had a great effect. Focusing on the vacuum stage, this study explored the process parameters to improve the PEW emulsion permeability of wood from the perspectives of vacuum process and the permeability of wood itself. For the vacuum process, the impregnation temperature and vacuum-holding time are the main parameters that affect the penetration. For wood itself, how to improve its permeability as much as possible is the key of the breakthrough. As the representative species of hardwood and softwood, Eucalyptus (Eucalyptus saligna) and Mongolian Scotch pine (Pinus sylvestris var. Mongolica) were selected. The structural differences between the two species cause varying degrees of emulsion penetration. Steaming and microwave methods were employed for the pretreatment.

2 Materials And Methods

2.1 Materials

The tested wood were 5-year-old Eucalyptus (Eucalyptus saligna) and 15-year-old Mongolian Scotch pine (Pinus sylvestris var. Mongolica) sapwood, obtained from Australia and northeast China respectively, with straight grain and a few live knots. Apparent defects were eliminated. The average ring widths of tested wood were 0.15 mm to 0.2 mm. The average air-dry densities of the two species were 0.78 g/cm^3 and 0.43 g/cm^3, and the average moisture contents were 8.4% and 11.84% (the wood were kept in a temperature and humidity regulator under a temperature of 20℃ and a relative humidity of 65% before being tested), respectively. The wax used for the tests was high-melting-point polyethylene wax (PEW, R-$(CH_2\text{-}CH_2)n\text{-}CH_3$, provided by Yousuo Chemical Technology Co. Ltd, Shandong, China), white granular solid at room temperature. The used emulsifier was maleic anhydride (MAH, $C4H_2O_3$) reactive emulsifier (provided by Yabang Chemical Co. Ltd, Jiangsu, China). This PEW emulsion is a cationic emulsion, which is a kind of homogeneous oil-in-water (O/W) fluid made from PEW uniformly dispersed in the aqueous phase under mechanical external force through the directional adsorption of MAH emulsifier. The carboxyl group of MAH emulsifier can esterify with the hydroxyl group of wood fiber to form a strong chemical bond (Fig. 1). It is ivory white emulsion at room temperature with good fluidity and homogeneity. It has a pH of 6.26, a density of 0.93 ± 2 g/cm3 and a solid content of 40 ± 2%.

2.2 Methods for determining the properties of PEW emulsion

Particle size test method: The penetration is correlated with the particle size of the PEW emulsion. PEW emulsion inside the wood was not easily observed, therefore the emulsion was designed to be directly dried. The changes in particle size and shape of the emulsion were observed and recorded. The particle size and shape of the PEW emulsion were tested in liquid state. About 5 ml emulsion was dropped into the sample cell of the wet and dry laser particle size analyzer (Bettersize 2600, Bettersize Instruments Co. Ltd., Liaoning, China), and ultrasonic waves uniformly dispersed the particles. The lens was then adjusted for testing. The PEW emulsion was then made into slide samples: take a small portion of emulsion and drop it in the center of the slide glass, and then slowly put on the cover glass from one side. The samples were air dried at room temperature and the dried wax particles were then tested by the wet and dry laser particle size analyzer. Ten samples were tested in liquid state and in solid state respectively. The average value was taken for emulsion. Image scanning and particle size analysis were carried out separately.

Viscosity test method: The viscosity of the wax emulsion has an effect on the penetration efficiency of the PEW emulsion. PEW emulsion viscosity was determined using the digital display rotary viscometer (NDJ-5S, Lichen Instrument Technology Co., Ltd., Shanghai, China), rotor 1. The speed was set at 6, 12, 30 and 60 r/s and the emulsion was tested three times at each speed. The average value was taken as the viscosity value for the emulsion.

2.3 Methods for vacuum impregnation and pretreatment

Defect free Eucalyptus and Mongolian Scotch pine were processed into samples with a size of 60 mm (longitudinal) × 20 mm (chordal) × 5 mm (radial), and the quality difference of samples from the same species shall be controlled within 2%. Before placed into electrothermic vacuum drying oven (DZF-6020, Saidelisi Experimental Analytical Instrument Manufacturing Factory, Tianjin, China), the samples were grouped, weighed and put into beakers, poured with PEW emulsion until being submerged for over 2 cm. The temperature inside the oven was set to 30℃, 40℃ or 50℃, and the vacuum level was − 0.09 MPa for all. The state was maintained for 30 min, 60 min or 120 min before releasing pressure, and then maintained at ordinary atmospheric pressure for 120 min. In order to visually test the penetration of PEW emulsion, an extra of 2 samples were taken from each species at each temperature to be impregnated by dyed emulsion and sawn from the middle. After the wax injection, the wood samples were removed with excess wax wiped off from the surface, dried at 80℃ by a DHG series heating and drying oven (DHG-9143BS- , CIMO Medical Equipment Ltd., Shanghai, China) and then weighed for subsequent test. The selected pretreatment methods included steaming and microwave irradiation. Steaming was carried out in an electro-thermostatic water cabinet (DK-600B, Senxin Experimental Instrument Co., Ltd., Shanghai, China) with a temperature of 100℃ for 40 min, 80 min or 120 min. Before microwave irradiation the samples needed to be immersed in water, and the immersion time was designed for 30 min, 60 min or 90 min depending on the pre-test. Microwave irradiation in microwave oven (P70F23P-G5, Galanz Electric

Appliance Co., Ltd., Guangdong, China) was held for 30s in all cases. This part consisted of 160 samples divided into 32 groups with 5 samples in each group as shown in Table 1.

Table 1
Design parameters for the impregnation test

Code	Species	Pretreatment			Impregnation Temperature /℃	Vacuum Time / min
		S /min	WI /min	MI /s		
E-30-30	E	-	-	-	30	30
E-30-60	E	-	-	-	30	60
E-30-120	E	-	-	-	30	120
E-40-30	E	-	-	-	40	30
E-40-60	E	-	-	-	40	60
E-40-120	E	-	-	-	40	120
E-50-30	E	-	-	-	50	30
E-50-60	E	-	-	-	50	60
E-50-120	E	-	-	-	50	120
E-S-40	E	40	-	-	30	120
E-S-80	E	80	-	-	30	120
E-S-120	E	120	-	-	30	120
E-W-M	E	-	-	30	30	120
E-W-30	E	-	30	30	30	120
E-W-60	E	-	60	30	30	120
E-W-90	E	-	90	30	30	120
P-30-30	P	-	-	-	30	30
P-30-60	P	-	-	-	30	60
P-30-120	P	-	-	-	30	120
P-40-30	P	-	-	-	40	30
P-40-60	P	-	-	-	40	60
P-40-120	P	-	-	-	40	120
P-50-30	P	-	-	-	50	30
P-50-60	P	-	-	-	50	60
P-50-120	P	-	-	-	50	120

Code	Species	Pretreatment			Impregnation Temperature /℃	Vacuum Time / min
		S /min	WI /min	MI /s		
P-S-40	P	40	-	-	30	120
P-S-80	P	80	-	-	30	120
P-S-120	P	120	-	-	30	120
P-W-M	P	-	-	30	30	120
P-W-30	P	-	30	30	30	120
P-W-60	P	-	60	30	30	120
P-W-90	P	-	90	30	30	120

"-" indicates no corresponding processing, "E" indicates Eucalyptus, "P" indicates Mongolian Scotch pine, "S" indicates steaming, "WI" indicates water immersion, and "MI" indicates microwave irradiation.

2.4 Test method for permeability of impregnated wood

In order to test such small samples, weight percent gain (WPG) was used to express. Weight percent gain post impregnation refers to the ratio of weight gain after impregnation to initial weight of the wood, and it provides a comprehensive indication of wood permeability to PEW emulsion. The average value was taken for each group of emulsion. The WPG was calculated according to Eq. (1).

$$\Delta M = \frac{M_1 - M_0}{M_0} \times 100\% \qquad (1)$$

where ΔM is the WPG of the wax impregnated wood (%), M0 is the dry mass before emulsion impregnation (g) and M1 is the dry mass after impregnation (g).

For WPG, multiple comparisons were tested by analysis of variance (ANOVA) at $p < 0.05$.

3 Results And Discussion

3.1 Particle size of PEW emulsion

The typical tracheids diameters of softwood are 10–50 μm, and the effective pore opening diameters are between 0.02 and 4 μm. Hardwoods contain vessels with larger diameters in the ranges of 20–400 μm, and their effective pore opening diameters are 5-170 μm (Plotze and Niemz, 2011). Particle size analysis results of PEW emulsion are shown in Table 2, and Fig. 2 is the particle size distribution of one sample. The average D50 value (i.e. the median particle size, which is a typical value indicating particle size) for

PEW emulsion was 0.256 μm, much smaller than the diameters of most wood fiber pores. The particle size of the typical sample mainly distributed in the range of 0.123 to 0.399 μm. The emulsion had a relatively uniform particle size and were well distributed. At the microscopic level, the shape and size of the PEW in the wet state provided favourable conditions for its penetration into the wood interior. From the macroscopic perspective, PEW emulsion demonstrated good mechanical and storage stability. Figure 3 shows that dried PEW had a wide variation in particle size and shape, and the particle size reached the micron level. Typical particle shape graph could roughly reflect the state of PEW emulsion on the wood surface and inside the wood after drying: The PEW particles that entered the wood agglomerated into large particles and then blocked the capillary system of the wood, achieving better protection by effectively blocking the entry of moisture. The natural condensation of the emulsion did not fully reflect the true state of its penetration into the wood after drying, so the analysis was somewhat restricted and further tests were required.

Table 2
Particle size analysis results of PEW emulsion

Diameter	D10	D50	D90
Mean value / μm	0.183	0.256	0.420
Repeatability / %	1.55	1.73	1.68

3.2 Viscosity of PEW emulsion

As can be seen from Fig. 4, rotor speed affected the viscosity of PEW emulsion. The viscosity of PEW emulsion tended to decrease significantly as the speed increased. PEW emulsion is a kind of non-Newtonian fluid. The viscosity of non-Newtonian fluid is positively correlated with the rotor speed at low speed due to the interactions between molecular chains; high speed cuts the tangled molecular chains that impede the flow of the fluid, and the viscosity decreases (Malkin 2013). The viscosity of PEW emulsion did not show the tendency to increase at low speed in this test, and the failure to reflect its inflection point may be due to the large span between the speed gears of the viscometer.

3.3 Effect of vacuum-holding time and temperature during impregnation on permeability

Figure 5 shows that PEW emulsion entered the cells in the inner part of wood under vacuum impregnation at 50°C for 120 min. After moisture drying, the wax particles (presented in dark gray) attached to the inner cell walls. PEW particles were attached to most of cells with a few exceptions. The WPG of Eucalyptus and Mongolian Scotch pine with different vacuum-holding time and impregnation temperature are shown in Table 3 and Fig. 6. For different vacuum-holding time, WPG increased as time increased, with a greater increase in the interval from 30 min to 60 min and a smaller increase (or a slight decrease, e.g. E-50-120) in the interval from 60 min to 120 min. Taking the Mongolian Scotch pine impregnated at 50°C as an example, the WPG was 7.09%, 9.97% and 10.24% respectively for samples with a vacuum-holding time of 30 min, 60 min and 120 min. The WPG increased by a factor of 0.41 when the vacuum-holding time increased from 30 min to 60 min and only by a factor of 0.027 when it increased from 60 min to 120 min. According to the statistical analysis results, the vacuum-holding time had significant influence on WPG of both species under the same impregnation temperature. The PEW emulsion maintained its low viscosity and good flowability throughout vacuum-holding time and gradually penetrated into the wood. As the PEW emulsion penetrated, the pores of the wood cells were gradually filled completely, so that the change in the uptake of emulsion gradually became smaller until almost constant after 60 min.

For different impregnation temperature, the WPG increased with temperature and the velocity of increase decreased. Taking the Mongolian Scotch pine impregnated with a vacuum-holding time of 120 min as an example, the WPG was 8.0%, 9.84% and 10.24% respectively for samples impregnated at 30℃, 40℃ and 50℃. The WPG increased 0.23 times when the temperature increased from 30℃ to 40℃ and 0.04 times when it increased from 40℃ to 50℃. According to the statistical analysis results, the impregnation temperature had significant influence on WPG of both species under the same vacuum-holding time. As the temperature increased, the irregular motion of the emulsion molecules intensified. At the same time the air inside the wood expanded, thereby increasing the wood capillary diameter. The two factors acted synergistically to promote the penetration of the emulsion into the wood, resulting in WPG increase. The vacuum-holding time had more significant influence. It was clear from the pattern of variation that continuing to extend the vacuum-holding time or increase the impregnation temperature had little effect on the permeability, so a breakthrough was needed in terms of the structural properties of the wood itself.

Table 3
WPG data analysis of Eucalyptus and Mongolian Scotch pine under different vacuum-holding time and impregnation temperature

Code	WPG / %			
	mean	min	max	SD
E-30-30	3.94	3.11	4.78	0.66
E-30-60	5.91	4.80	8.10	1.22
E-30-120	6.50	5.50	7.87	0.86
E-40-30	5.18	4.17	6.22	0.76
E-40-60	7.42	6.79	9.22	0.91
E-40-120	7.86	6.22	8.98	1.05
E-50-30	5.36	4.87	5.77	0.30
E-50-60	7.57	5.88	8.85	1.24
E-50-120	7.25	5.27	8.47	1.16
P-30-30	6.59	5.72	7.54	0.68
P-30-60	7.31	6.53	8.26	0.66
P-30-120	8.00	7.01	9.05	0.74
P-40-30	7.10	6.57	7.66	0.44
P-40-60	9.27	7.51	12.15	1.89
P-40-120	9.84	7.58	11.19	1.21
P-50-30	7.09	5.21	9.84	1.73
P-50-60	9.97	8.40	12.90	1.57
P-50-120	10.24	8.19	11.44	1.15

3.4 Effect of steaming pretreatment on permeability

The WPG of steaming pretreated Eucalyptus and Mongolian Scotch pine are shown in Table 4 and Fig. 7. The steaming pretreatment increased the WPG of both species, with the trends not entirely consistent. For Eucalyptus, the WPG tended to increase at first and then decrease with steaming time increasing. The WPG of Eucalyptus treated by steaming for 40 min, 80 min and 120 min was 7.42%, 8.07% and 7.57%, which were 0.14, 0.24 and 0.16 times higher than that of the unpretreated group, respectively. The WPG of Mongolian Scotch pine continued increasing, with growth becoming progressively slower. Mongolian

Scotch pine steamed for 40 min, 80 min and 120 min was 10.73%, 11.81% and 12.22% respectively in WPG, which increased by 0.34, 0.48 and 0.53 times compared to those unpretreated group. According to the statistical analysis results, the steaming had no significant influence on WPG of Eucalyptus but had significant influence on WPG of Mongolian Scotch pine. The explosion of the hot steam caused some of the grain pores in the wood to rupture and form fissures, thus opening up those blocked pathways for the flow of gas and liquid. Macroscopically, the steam-treated samples of both species exhibited a rough surface. In addition, the internal pressure generated by the steam drained the resin in Mongolian Scotch pine and the tylosis inside Eucalyptus, opening up the blocked channel. The slight decrease in the WPG of Eucalyptus steamed for 120 min may be due to the flow and relocation of extractives caused by the steam, resulting in the blockage of some of the pathways, which was consistent with the analysis from Mackay (1971).

Table 4
WPG data analysis of steaming pretreated Eucalyptus and Mongolian Scotch pine

Code	WPG / %				Improvement / %
	mean	min	max	SD	
E-30-120	6.50	5.50	7.87	0.86	-
E-S-40	7.42	6.67	8.30	0.53	14.15
E-S-80	8.07	6.11	11.01	1.85	24.15
E-S-120	7.57	6.94	8.73	0.67	16.46
P-30-120	8.00	7.01	9.05	0.74	-
P-S-40	10.73	9.45	12.65	1.11	34.13
P-S-80	11.81	10.51	12.85	1.06	47.63
P-S-120	12.22	9.66	15.13	1.77	52.75

3.5 Effect of microwave irradiation pretreatment on permeability

The WPG of microwave irradiation pretreated Eucalyptus and Mongolian Scotch pine are shown in Table 5 and Fig. 8. The WPG of the pretreated samples firstly rose and then declined with the increase of immersion time. Eucalyptus is a denser wood with an underdeveloped capillary system and the overall variation in WPG was small. The WPG of microwave irradiation pretreated Eucalyptus immersed for 0 min, 30 min, 60 min and 90 min was 7.05%, 7.56%, 8.30% and 7.93% respectively, which were 0.08, 0.16, 0.28 and 0.22 times more than the unpretreated group. The variation was more pronounced in Mongolian Scotch pine with a clear peak at 60 min. The WPG of microwave irradiation pretreated Mongolian Scotch pine immersed for 0 min, 30 min, 60 min and 90 min was 8.07%, 8.40%, 13.97% and 7.37% respectively, which were 0.09, 0.05, 0.75 and − 0.08 times higher compared to the unpretreated group. According to the statistical analysis results, the water immersion time had significant influence on both species. The internal water was heated up in the microwave field and evaporated rapidly. Under the combined effect of temperature and pressure gradients, the weakest wood cells and pit membrane were ruptured, meanwhile part of the resin and tylosis were discharged, and thus provided a connection between cell cavities (Torgovnikov and Vinden 2009). Water immersion time mainly affected the initial moisture content (IMC) of the wood, which was more appropriate around the fibre saturation point (FSP). If the IMC was too low, the amount and pressure of steam generated by vaporization may not be sufficient to break through the weakest parts of the wood; if it was too high, the mobile water in the wood had difficulty boiling completely in a short space of time, and the steam pressure generated was too low to be conducive to permeability improvement as well (Jiang et al. 2006).

Figure 9 shows the comparison of the permeability improvement between the two species pretreated by the two methods. Under the same impregnation condition, both pretreatment methods enhanced the permeability of Eucalyptus, with small differences between different groups. In this test, steaming increased the WPG of Eucalyptus by up to 0.24 times and microwave irradiation increased it by up to 0.28 times, with the latter achieving slightly better results. For Mongolian Scotch pine, the WPG of each group existed great differences. Steaming increased its WPG by up to 0.53 times and the microwave irradiation increased it by up to 0.75 times. The latter had a remarkable advantage for species with well-developed capillary system. When the wood IMC was near the FSP, microwave irradiation would be more effective. The difference in the optimum conditions achieved using the two methods may be due to the fact that the temperature and depth of interaction produced by steam had difficulty reaching the level of microwave irradiation. However, as can be seen from the minimum values, when the moisture in wood was not properly controlled, microwave irradiation may instead have a negative impact.

Table 5
WPG data analysis of microwave irradiation pretreated Eucalyptus and Mongolian Scotch pine

Code	WPG / %				Improvement / %	IMC / %
	mean	min	max	SD		
E-30-120	6.50	5.50	7.87	0.86	-	-
E-W-M	7.05	6.42	8.28	0.69	8.46	8.4
E-W-30	7.56	6.42	8.35	0.67	16.31	15.48
E-W-60	8.30	7.02	8.99	0.76	27.69	17.67
E-W-90	7.93	6.20	9.32	1.04	22.00	21.69
P-30-120	8.00	7.01	9.05	0.74	-	-
P-W-M	8.07	6.82	9.65	1.28	0.88	11.84
P-W-30	8.40	7.38	11.03	1.33	5.00	18.68
P-W-60	13.97	9.93	16.42	2.58	74.63	32.12
P-W-90	7.37	5.87	9.33	1.38	-7.9	40.67

4 Conclusion

1. The PEW emulsion had small particle size, low viscosity and good flowability, which facilitated its penetration into Eucalyptus and Mongolian Scotch pine. After moisture drying, the small particles agglomerated into large size particles, attached to cell walls and blocked the internal air-liquid channels of the wood.
2. Vacuum-holding time had a remarkable effect on the WPG. At the same impregnation temperature, the WPG increased with vacuum-holding time and then gradually stabilized. Impregnation was a slow process and a longer time favoured the penetration of the emulsion.
3. A higher impregnation temperature resulted in a higher WPG within the same vacuum-holding time. The high temperature intensified the irregular motion of the PEW molecules and opened up the air-liquid channels, making it easier for the emulsion to penetrate.
4. The penetration of PEW emulsion required that the wood itself had a permeable structure. Both steaming and microwave irradiation were effective in increasing or widening the air-liquid channels, significantly improving the permeability of the two species. The samples with IMC near the FSP can be better modified by microwave irradiation. Both pretreatment methods showed more remarkable improvement in Mongolian Scotch pine whose capillary system was well-developed.

Declarations

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Author Contribution Xiaojun Yang designed the study. Jiamin Liu wrote the original manuscript. Xiaohui Kong revised the manuscript. Chaojie Wang prepared figures and tables. All authors performed experiments. All the authors have accepted responsibility for the entire content of this submitted manuscript and approved submission.

Conflicts of interest The authors declare no confict of interest.

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Figures

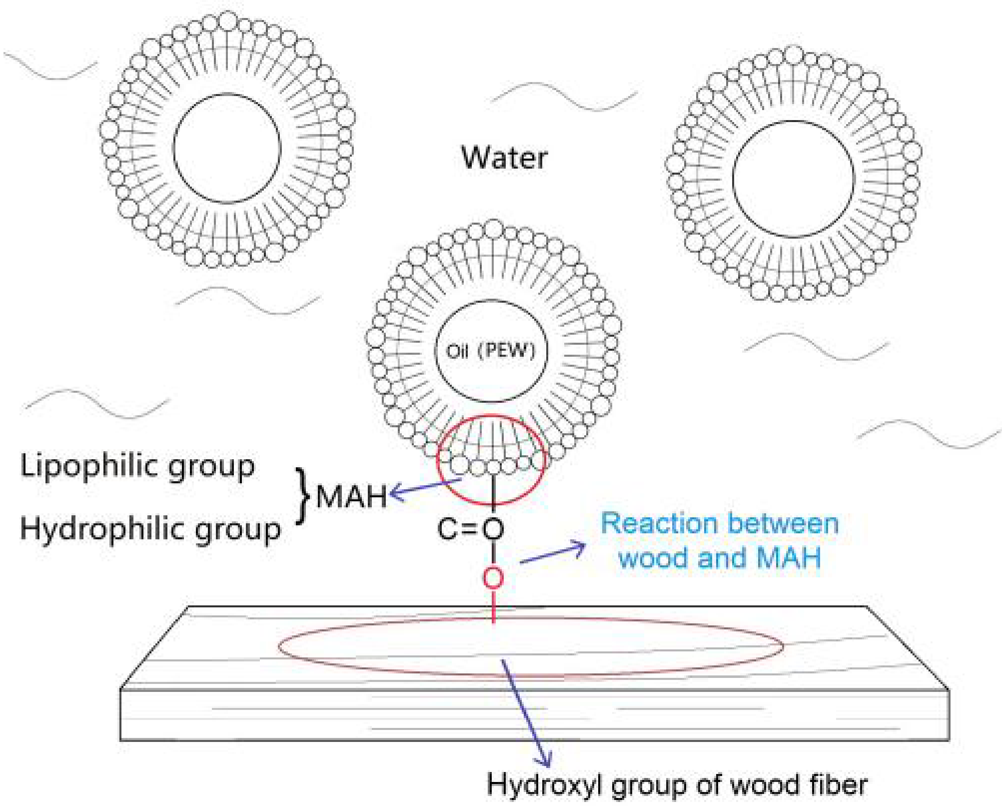

Figure 1

Schematic diagram of PEW emulsion

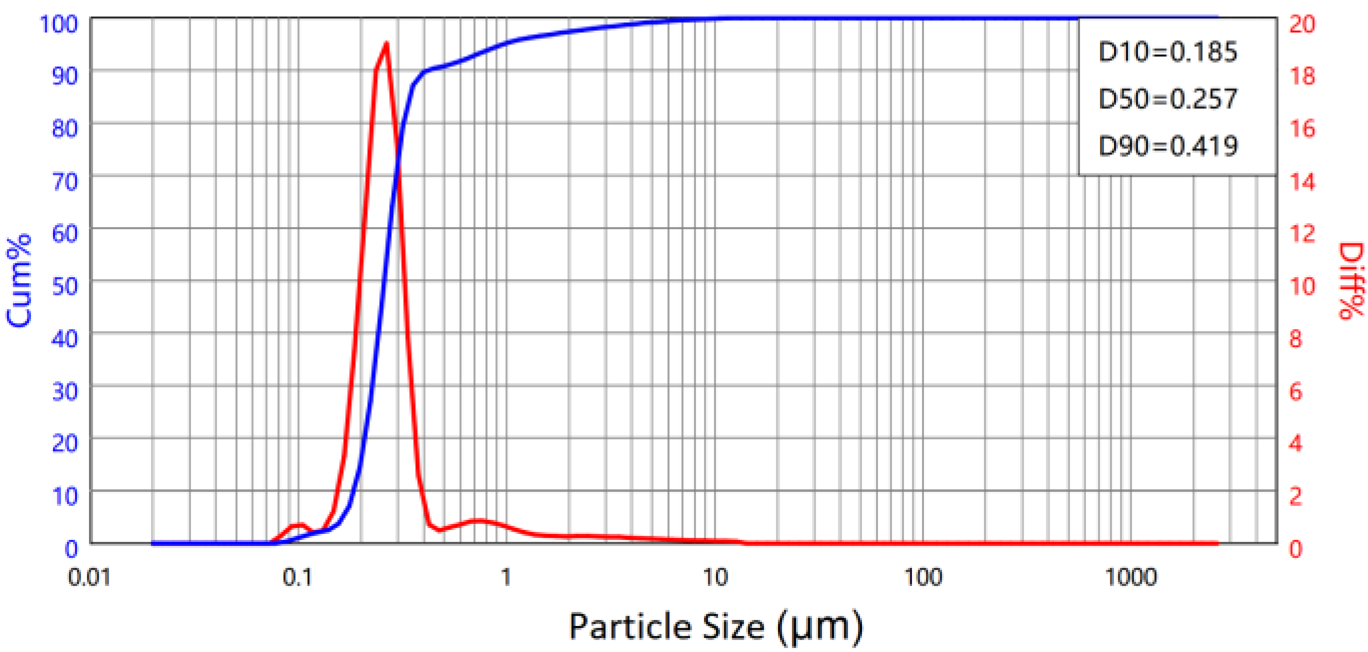

Figure 2

Typical PEW emulsion particle size analysis chart: Cum is the cumulative distribution curve and Diff is the differential distribution curve.

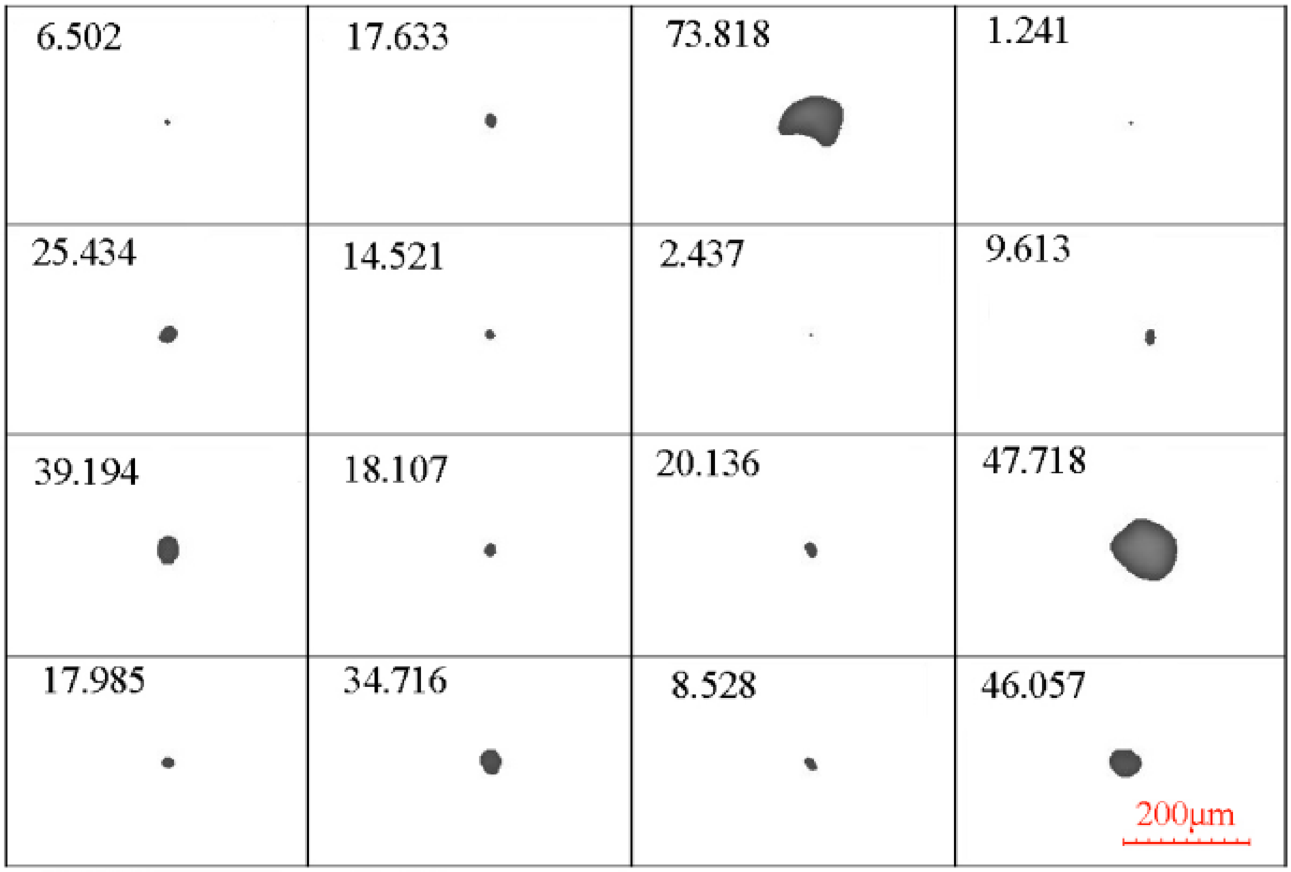

Figure 3

Typical particle shape of dried PEW: The particle size is shown in the upper left corner of each graph.

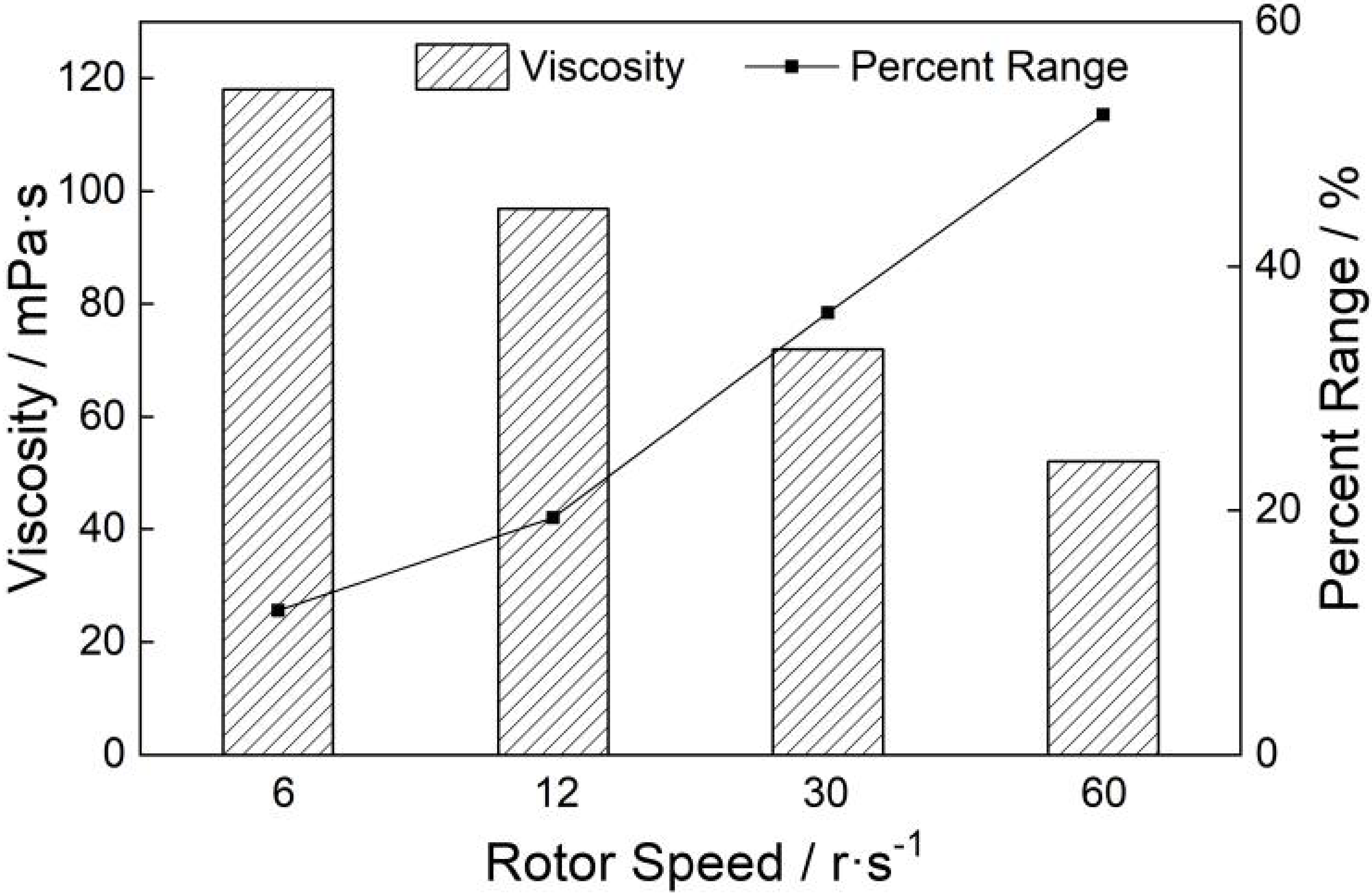

Figure 4

Viscosity of emulsion at different rotor speeds.

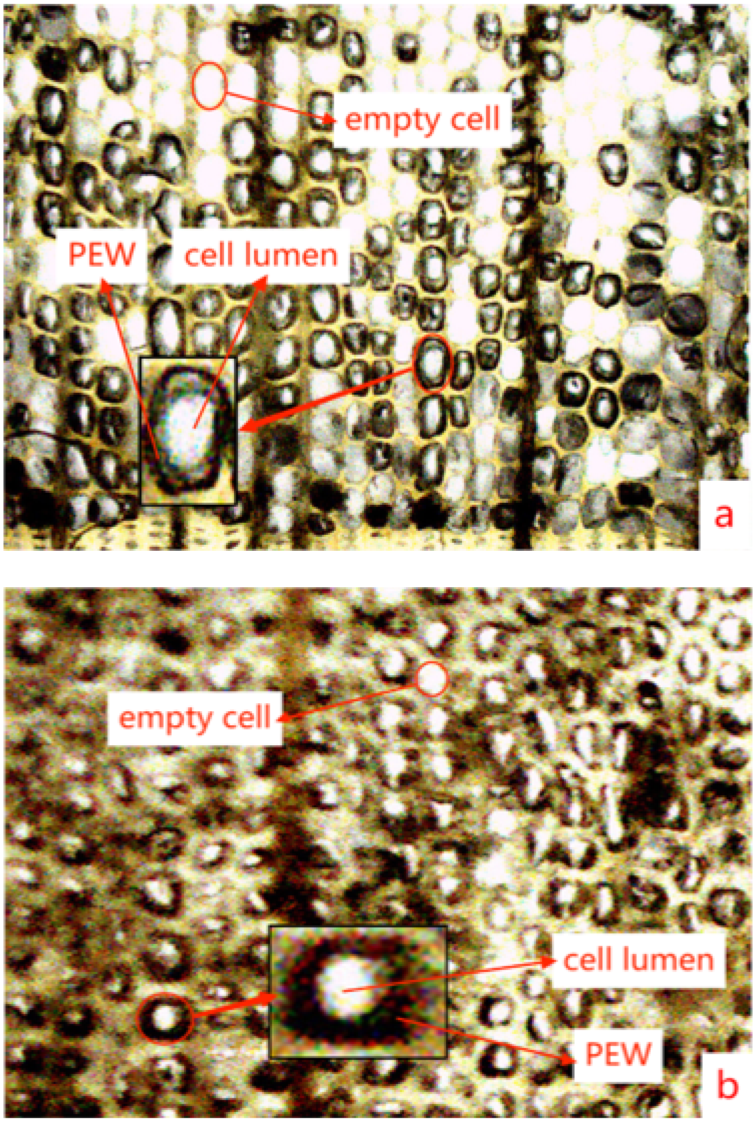

Figure 5

Typical microscopic images of cross-sections sawn from the middle of wood samples vacuumed at 50°C for 120 min: (a) Mongolian Scotch pine, (b) Eucalyptus.

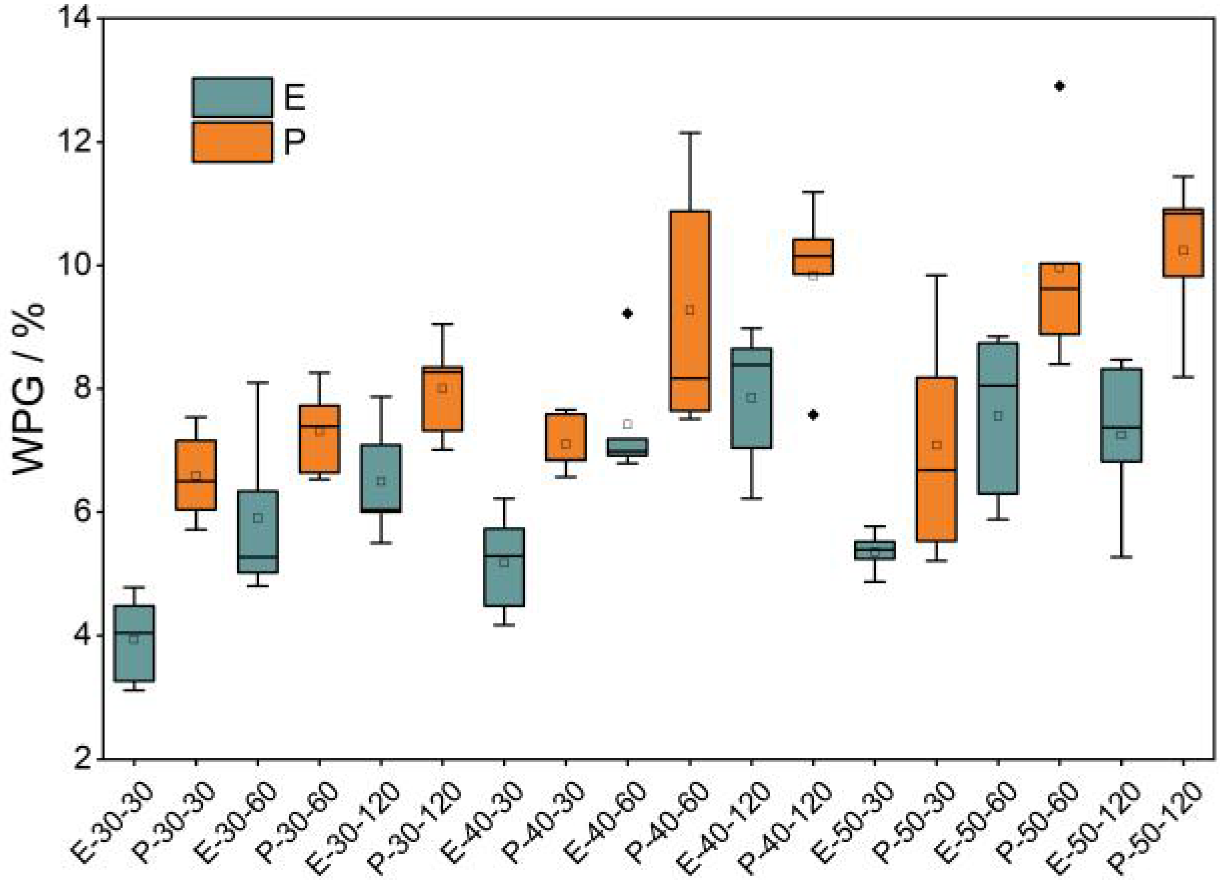

Figure 6

WPG chart analysis of Eucalyptus and Mongolian Scotch pineunder different vacuum-holding time and impregnation temperature

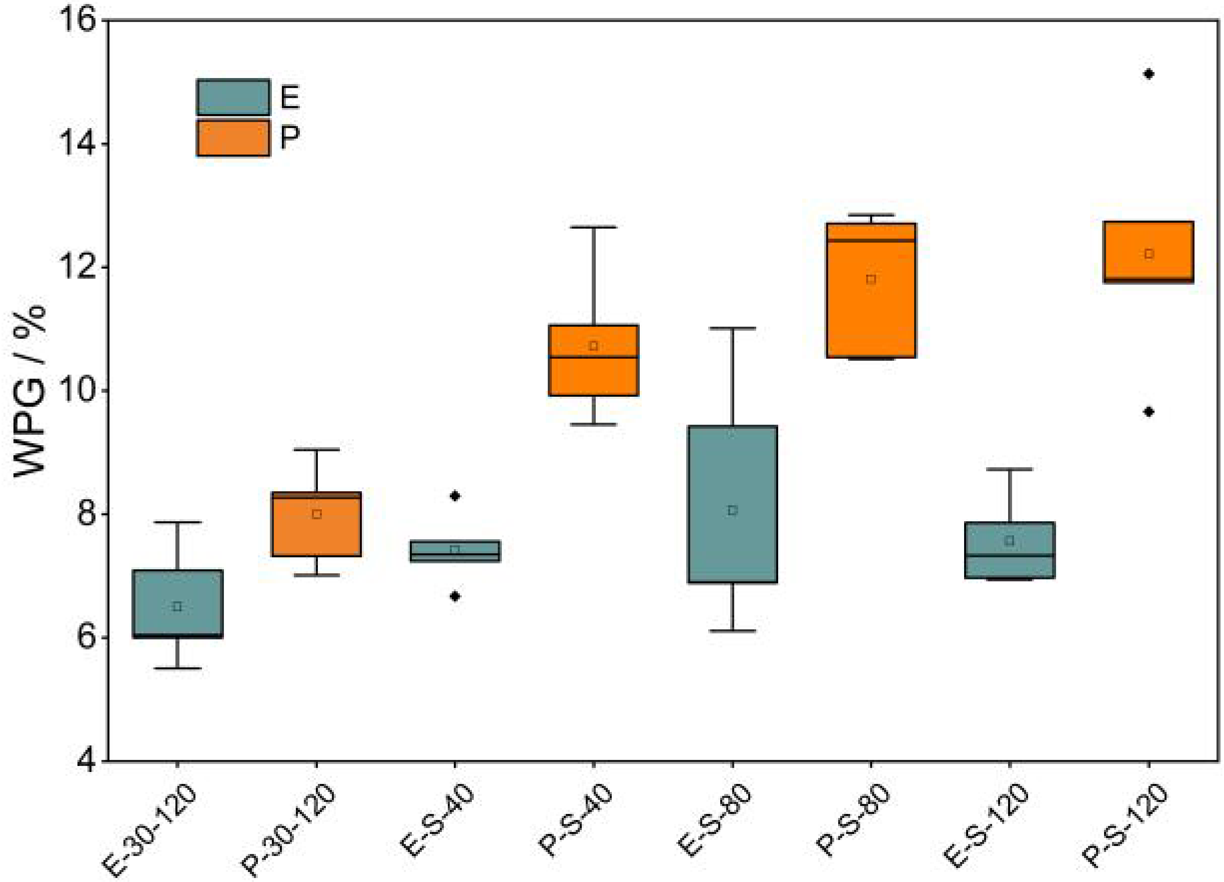

Figure 7

WPG chart analysis of steaming pretreated Eucalyptus and Mongolian Scotch pine

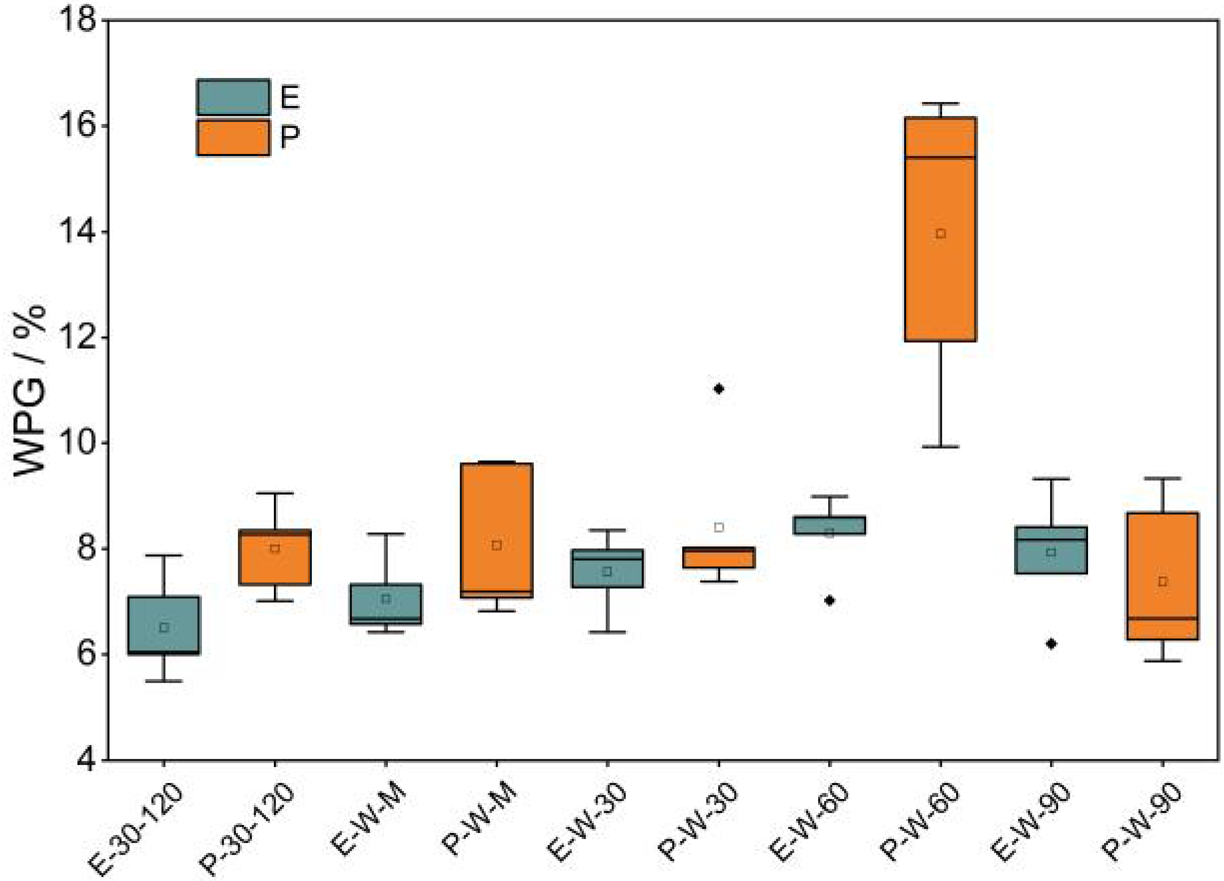

Figure 8

WPG chart analysis of microwave irradiation pretreated Eucalyptus and Mongolian Scotch pine

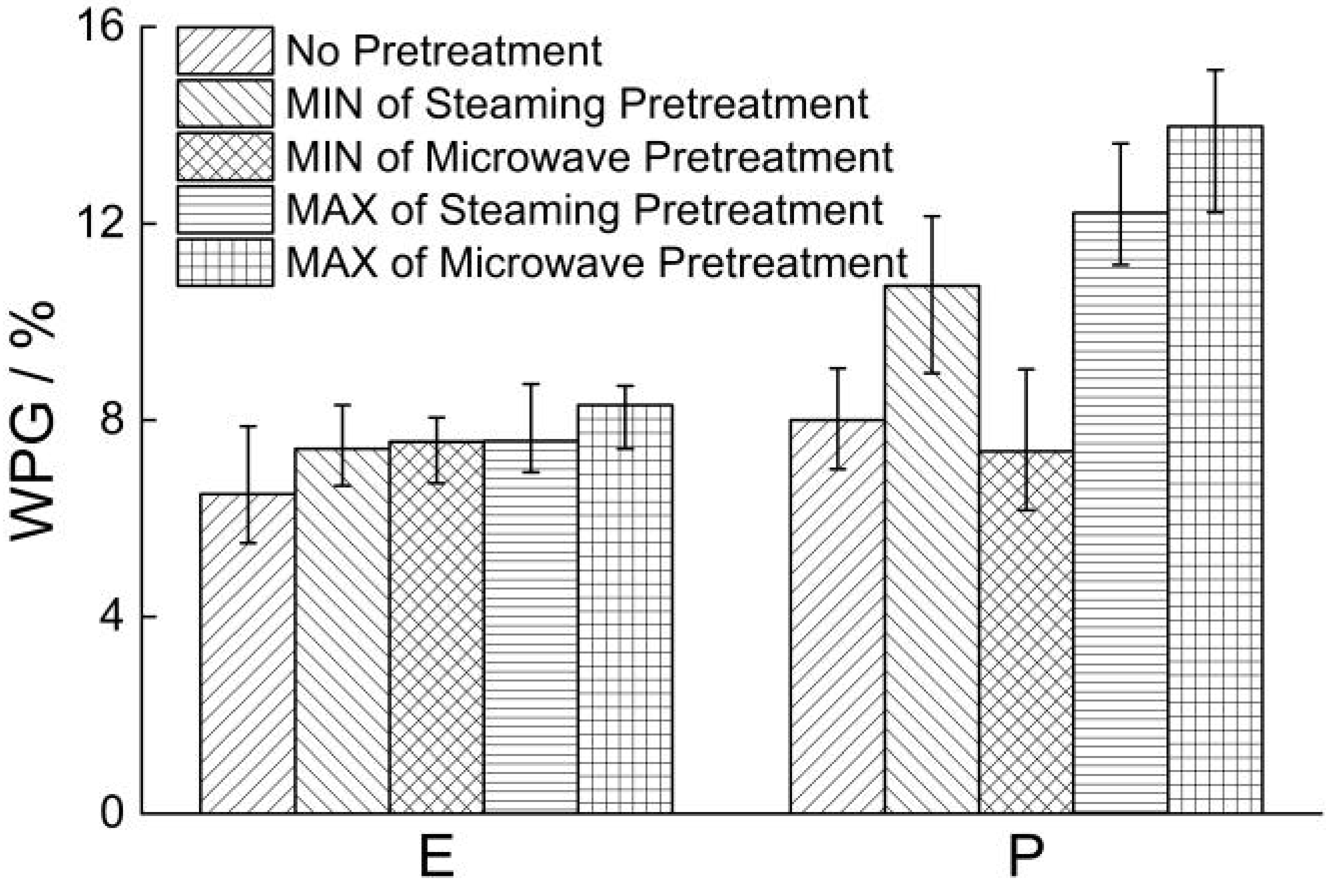

Figure 9

WPG of sampleswith different pretreatment methods