MOISTURE PROPERTIES OF HEAT-TREATED SCOTS PINE AND NORWAY SPRUCE SAPWOOD IMPREGNATED WITH WOOD PRESERVATIVES

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Abstract. An experiment was conducted on commercially heat-treated (HT) Scots pine (*Pinus sylvestris* L.) and Norway spruce (*Picea abies* [L.] Karst.) sapwood collected from Ht Wood AB, Arvidsjaur, Sweden. Secondary treatment on HT wood was performed in laboratory scale by impregnating with water-repellent preservatives (a commercial one and pine tar) to evaluate their retention and different moisture-related properties. Preservative solutions were impregnated using a simple and effective method. Wood samples were heated at 170°C in a dry oven and were immediately immersed in preservative solutions. Considerable retention was observed in HT wood, particularly in pine. Moisture adsorption properties were measured after conditioning in a high-humidity environmental chamber (4°C and 84% RH). Experimental results showed that secondary treatment enhanced moisture excluding efficiencies by decreasing equilibrium moisture content, suggesting better hydrophobicity. Soaking test in water showed that antiswelling and water repellence efficiencies improved, especially in tar-treated wood. In addition, this type of treatment significantly decreased water absorption. It was also possible to decrease volumetric swellings. Thus, secondary treatment of HT wood with preservative, in particular with tar, improved dimensional stability and water repellency.

Keywords: Heat-treated wood, pine tar, secondary treatment, water repellency, water uptake.

INTRODUCTION

The quality and in-service life of nondurable timber can be improved by means of wood modification or preservative treatment. Various types of chemicals, such as anhydrides, isocyanates, silicon, aldehydes, epoxides, alkyl chlorides, etc, have been used for wood modification (Donath et al 2004; Mai and Militz 2004). Additionally, wood treatment with heat is one of the most studied methods of wood modification (Zaman et al 2000; Epmeier and Kliger 2005; Hakkou et al 2006; Esteves and Pereira 2009; Karlsson et al 2011). This procedure claims to improve wood properties by decreasing hydrophilicity, improving dimensional stability, and enhancing resistance to biodegrading agents to some extent. For example, a noticeably lower mass loss after fungal attack was determined for oil heat-treated (HT) wood than for air HT wood (Rapp and Sailer 2001). However, wood modified with heat is reported to be susceptible to biodegrading agents such as marine borers (Westin et al 2006) and termites (Doi et al 1999) and has limited resistance against

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soil-inhabiting decay organisms. Therefore, use in ground contact is not recommended (Jämsä and Viitaniemi 2001).

Aside from wood modification, preservative treatment is effective and can extend the service life of wood and wood products. Certain chemicals used in conventional wood preservatives contain arsenic, chromium, or copper. Although found to be very successful against wood-destroying organisms, they cause environmental pollution and are hazardous to animals and human beings (Thompson and Dust 1971). Also, disposal of such wood is a major concern. Because metals cannot be broken down in the environment, disposal of any wood treated with a metal-based preservative will be more expensive and could potentially be a menace in the future. Therefore, use of these chemicals is banned or restricted in many regions of the world including Europe, North America, and Japan (Drysdale 2002). Special attention is thus focused on such biocides, which opens up an avenue for a wide range of environmentally benign new wood preservatives. For example, wood preservation methods with triazoles such as tebuconazole or propiconazole contain no metal, are found highly effective against a broad spectrum of Basidiomycetes fungi, and exhibit good stability and leaching resistance in wood (Schultz and Nicholas 2002). Tar is also used successfully as a wood preservative, and its application for wood protection has been known for centuries (Mazela 2007). Based on its chemical composition, it is considered a potential future wood preservative.

Success of wood preservation with chemicals mainly depends on permeability of wood tissue and ability of a preservative to penetrate deeply into wood structures (Rak 1976). For effective protection of wood against biodegrading agents, preservative must be evenly distributed in sufficient concentration. Preservative penetration is more critical in performance of treated wood than is preservative retention. Deep and uniform penetration should be preferred rather than the least amount of preservative retention. Shallow dispersion of preservative even at high concentration levels only in the outer zone did not provide adequate protection against biodegrading agents (Kollmann and Côté 1984; De Groot 1994).

A literature review indicated that thermal modification of wood was not adequate enough to ensure resistance against different biodegrading agents in outdoor use. It is expected that additional treatment with preservative would improve wood performance in outdoor application. Therefore, thermally modified wood was treated with preservatives to verify effectiveness against different wood-deteriorating organisms. As part of the experiment, this article only describes treatment with water-repellent preservatives and their effect on moisture properties of HT Scots pine and Norway spruce sapwood. Moisture properties include water adsorption at high humidity, water absorption, and swelling during water immersion. These properties are reported to be important for biological resistance of wood (Welzbacher and Rapp 2004). This study provides information on preservative selection and an easy method for secondary treatment of HT wood.

MATERIALS AND METHODS

Wood Sample

Commercially produced HT Scots pine and Norway spruce sapwood was collected from Ht Wood AB, Arvidsjaur, Sweden. Prior to heat treatment, boards ($\approx 125 \times 50$ mm^2) were kiln-dried to 18% MC. Heat treatment was carried out in a closed chamber in which temperature was increased to 170°C. The heat treatment phase started immediately after the high-temperature drying phase. Saturated steam was used during drying and heat treatment as a protective vapor that prevented wood from burning. It also affected chemical changes taking place in the wood. The heat treatment phase was applied for 2.5 h. More about the drying phase can be found in considerable detail in Johansson and Morén (2006). HT pine and spruce boards were then brought to the laboratory for secondary treatment with preservatives. Additional green pine and spruce boards (≈125 × 50 mm^2) were also collected. Sapwood portions were separated from heartwood and left in the laboratory. After attaining equilibrium moisture content at ambient conditions, they were used as the source of control samples.

Preservatives Used

Two types of water repellent preservatives were used. The first one was a water-miscible commercial wood preservative, Elit Träskydd (Beckers, Stockholm, Sweden). It contains additives such as propiconazole (0.6%) and 3-iodo-2-propynyl butylcarbamate (0.3%) in modified linseed oil as a binder and water as a solvent. Into that commercial formulation, 1.9% (w/v) tert-butylhydroquinone (Sigma-Aldrich, St. Louis, MO) was added as an antioxidant. The second one was commercial pine tar (Claesson Trätjära AB, Göteborg, Sweden) mixed in boiled linseed oil (Claessons Trätjära AB) as a carrier. Turpentine (Claessons Trätjära AB) was added as a solvent for thinning oil-based preservatives. The pine tar solution was as follows: 250 mL pine tar mixed with 1000 mL boiled linseed oil and 500 mL turpentine. The solution was stirred properly before use. Elit Träskydd and pine tar are hereafter referred to as Beckers and tar, respectively.

Preservatives Treatment

Stakes were prepared from 20 × 20 × 150-mm^3 boards (radial [R] × tangential [T] × longitudinal [L]). Stakes free of knots, cracks, or any visible defects and with similar masses were selected and numbered consecutively. After recording oven-dry mass and dimensions, stakes were heated at 170°C for 1 h in a dry oven. Then they were immediately submerged in preservative solutions for simultaneous cooling and impregnation for 2 h. Under this condition, a vacuum pressure gradient created in the sample automatically forces preservative solution to penetrate the wood a considerable amount. Thus, no external vacuum or pressure was applied. Six HT and seven control stakes from each species (pine and spruce) and each of the two treatments (Beckers and tar) were used for measuring mass increase and preservative retention. Thus, the total number of stakes used was 52.

After 2 h of soaking, preservative solution was gently wiped off surfaces with a paper towel and sample mass was recorded using a weighing balance to the nearest 0.01 g. Mass increase was determined from mass difference between preservative-treated and oven-dried stakes divided by oven-dry mass. Retention was calculated as follows: retention (kg/m^3) = 1000 G/V, where G is grams of preservative solution absorbed and V is volume of stake in cm^3.

Hygroscopic Behavior

To observe hygroscopic behavior, blocks with true R/T orientation with dimensions of 20 × 20 × 10 mm^3 (R × T × L) from HT and control wood were prepared and impregnated with preservatives (Beckers and tar) using the procedure outlined in the previous section. There were three different treatments (Beckers, tar, and no preservative) for control and HT wood from each species (pine and spruce) with 10 replications. Thus, a total of 120 samples were used. The bulking coefficient (BC) was calculated using the following formula: $BC(\%) = 100 \times (V_t - V_u)/V_u$, where V_t and V_u are oven-dried volume of sample after and before preservative treatment, respectively.

All treated and control samples were placed in an oven at 103°C for 24 h to obtain constant masses. They were then conditioned in a climate chamber at 4°C and 84% RH for 4 wk to acclimatize their equilibrium moisture content. MEE was defined as follows: $MEE\ (\%) = 100 \times (E_c - E_t)/E_c$, where E_c and E_t are equilibrium moisture content of control and preservative-treated samples, respectively.

Samples were then submerged in water at 21°C for 9 da to measure water absorption (WA; absorbed water divided by oven-dry mass) and volumetric swelling (S). Water was replaced daily during the soaking test. Water repellence efficiency (WRE) and antiswelling efficiency (ASE) were estimated based on water absorption (WA_t) and swelling (S_t) of treated samples relative to those of controls (WA_c and S_c).

$$WRE(\%) = 100 \times (WA_c - WA_t)/WA_c \quad (1)$$

$$ASE(\%) = 100 \times (S_c - S_t)/S_c \quad (2)$$

ASEs of preservative-impregnated HT and control wood were calculated from S values of unimpregnated HT and control samples, respectively. Volumetric swelling was calculated as S (%) = 100 × (V_2 – V_1)/V_1, where V_2 is wood volume after wetting with liquid water and V_1 is wood volume of the oven-dried sample before wetting. Volume was determined by immersing the wood sample in water and applying Archimedean principle, ie wood samples were weighed while immersed and suspended in water. Sample mass while immersed and suspended in water divided by the density of water was the volume of the wood sample (Wei et al 2000).

RESULTS AND DISCUSSION

Preservative Retention

Results for uptake (mass increase) and retention of two different preservatives are presented in Table 1. Without any external vacuum or pressure, all treated samples (except HT spruce) retained a considerable amount of preservatives, which fulfilled the retention of formulated oil-based preservative prescribed for usage under class A (ground or fresh water contact) according to the Nordic Wood Preservation Council (NWPC) (NWPC 2005). Treatment results showed that HT pine had the highest and spruce had the lowest mass increase and retention compared with their corresponding control samples. Mass increase was found slightly higher for HT spruce and much lower for HT pine compared with results in Karlsson et al (2011).

Table 1. Means (±SD) of preservative uptake by pine and spruce.[a]

Treatment	Mass increase (%)	Retention (kg/m^3)
C pine Beckers	32.05 ± 5.24	132.78 ± 14.03
C spruce Beckers	29.72 ± 3.67	114.86 ± 7.29
C pine tar	47.17 ± 4.50	214.16 ± 19.27
C spruce tar	25.01 ± 1.75	102.15 ± 8.04
HT pine Beckers	47.95 ± 3.91	228.15 ± 17.06
HT spruce Beckers	18.13 ± 2.31	73.06 ± 15.05
HT pine tar	47.22 ± 3.81	226.37 ± 15.55
HT spruce tar	15.35 ± 6.52	51.07 ± 17.82

[a] HT, heat-treated; C, control; SD, standard deviation.

Islam et al (2009) reported that preservative penetration was influenced by wood density. A lower specific gravity means fewer cell wall materials analogous to higher void volume, which facilitates absorption of a greater amount of preservative. However, observations in this study did not reflect correlation of preservative solution penetrability with wood density; rather, penetrability appeared to be related to intrinsic anatomical properties. Oven-dried density of control pine and spruce was 422.52 and 413.47 kg/m^3, respectively. Conversely, density of HT wood was 380.35 and 379.79 kg/m^3 for pine and spruce, respectively. Heat treatment decreased wood density, and this density change agrees with Esteves and Pereira (2009). They reported that the extent of mass loss caused by heat treatment depended on the method and temperature level used. Apart from the density-related property, lower permeability of spruce compared with pine was studied in considerable detail by Olsson et al (2001), in which the inconsistency of penetration between two species is explained based on their anatomical differences.

Thermal modification could cause pine to form secondary flow paths (interstitial spaces) by damaging thin-walled radial or axial parenchymas. Ahmed et al (2011) reported that kiln-drying was sufficient to initiate ray cell collapse, forming interstitial spaces in pine. These kinds of induced void spaces are considered important liquid flow paths in dried wood because the main flow channel by tracheids is restricted because of aspiration of bordered pits (Booker 1990). Until and unless bordered pits are closed, they are considered the most important structure regulating permeability of softwoods, but rays have also proven pivotal. However, this is not the case for HT spruce. The reason could be that spruce has rays with thicker cell walls and smaller cross-field pitting (piceoid) compared with pine (Olsson et al 2001). In contrast to pine, drastic damages such as detachment of the cell wall or cell wall layers and rupture of pit membranes caused by thermal treatments are not observed in spruce (Boonstra et al 2006). It is likely that the inability to form a secondary flow path and the appearance of some degree of plasticized bordered pit membranes (Boonstra et al 2006) meant that HT spruce had decreased permeability, even lower than control samples (Table 1).

Polarity of preservative liquid was expected to affect preservative uptake. Formulated pine tar is nonpolar, and some part of Beckers is constituted with polar liquid (see "Preservatives Used" section). Walters and Côté (1960) and Bailey and Preston (1970) showed that nonpolar liquids penetrate by bulk flow mainly through cell lumens and pits whereas polar compounds penetrate by bulk flow and diffusion through the wood cell wall. This causes an interesting uptake difference between tar and Beckers. The higher mass increase of tar-treated control pine compared with that of the Beckers-treated sample remains to be investigated.

Moisture Repellency

To illustrate the hydrophobicity of treated wood, MEE, one of the indices to evaluate hydrophobic or hydrophilic characteristics of wood, was measured at 84% RH (Table 2). Equilibrium moisture content of HT pine and spruce were 8.72 and 9.15%, respectively. After preservative treatment, equilibrium moisture contents of HT and control samples were decreased from their corresponding unimpregnated samples. This means that in the same ambient conditions, wood impregnated with preservatives absorbs less water which, of course, decreases dimensional changes. Thermal modification of wood had pronounced influence on the decrease of equilibrium moisture content, which agrees with Esteves and Pereira (2009). HT wood became more hydrophobic than preservative-treated wood, and this ultimately increased MEE. Higher MEE values mean higher hydrophobicity. HT woods performed better than their corresponding control wood samples. In this regard, Epmeier and Kliger (2005) reported that HT wood decreased hydrophilicity by breaking down hemicelluloses, modifying lignin, redistributing wood extractives, and decreasing the number of hydroxyl groups in wood cell walls. Nevertheless, additional uses of preservatives, especially tar, improved MEE of HT wood. Conversely, decreased MEE occurred in Beckers-treated

Table 2. Mean (±SD) MEE and ASE of preservative-treated wood.[a]

		Test at 84% RH		Water-soaked test	
Treatments	BC (%)	EMC (%)	MEE (%)	S (%)	ASE (%)
C pine	—	14.57 ± 0.48	—	16.09 ± 1.27	—
C spruce	—	14.82 ± 0.33	—	15.49 ± 2.44	—
HT pine	—	8.72 ± 0.13	—	9.28 ± 0.85	42.37
HT spruce	—	9.15 ± 0.40	—	8.87 ± 0.89	42.73
C pine Beckers	3.24	11.70 ± 0.14	19.70	13.31 ± 2.38	17.31
C spruce Beckers	2.22	12.02 ± 0.12	18.85	12.00 ± 0.93	22.53
C pine tar	1.45	9.96 ± 0.71	31.67	12.15 ± 0.60	24.54
C spruce tar	0.64	10.92 ± 0.12	26.31	10.33 ± 0.68	33.31
HT pine Beckers	1.49	6.83 ± 0.12	21.70	8.24 ± 0.62	11.12
HT spruce Beckers	1.97	7.15 ± 0.19	21.83	7.89 ± 0.48	11.02
HT pine tar	1.05	5.44 ± 0.18	37.63	8.20 ± 0.98	11.64
HT spruce tar	0.23	6.66 ± 0.15	27.15	7.18 ± 1.23	19.07

[a] EMC, EMC; MEE, moisture excluding efficiency; S, volumetric swelling; ASE, antiswelling efficiency; BC, bulking coefficient; HT, heat-treated; C, control; SD, standard deviation.

wood, suggesting lower hydrophobic ability. After soaking in water for 9 da, WRE of treated samples conspicuously increased for tar-treated wood, indicating better hydrophobicity than Beckers (Fig 1). Anomalous results of WRE were obtained in Beckers-treated wood; HT and control spruce had the highest and lowest WRE, respectively. It is postulated that lower hydrophobicity of Beckers decreased water repellency.

Dimensional Stability

BC values of wood treated with tar were lower than those of Beckers (Table 2). As discussed previously, polarity of preservative liquid was responsible for affecting BC values. Dimensional stability of preservative-treated wood was evaluated by the water-soaked method, and ASEs of treated wood are shown in Table 2. As expected, S of control wood was higher than that of HT wood. Before and after preservative treatments, S values of spruce were lower than pine. This result was attributed to lower permeability of spruce. Wood treated with tar showed better water repellency than that treated with Beckers. Both preservatives had a hydrophobicity effect, which decreased volume change by water absorption. Less shrinking resulted in better dimensional stability of treated wood, expressed as ASE. Because heat as well as preservative treatments contributed to ASE, they are supposed to decrease equilibrium moisture content of wood, specifically the amount of bound water in the cell wall (Rowell 1983). ASE of tar- and Beckers-impregnated HT wood was 11-19%, whereas it was 17-33% for their corresponding controls. Before impregnation with preservative solutions, all samples were heated to 170°C for 1 h. Higher ASE values for control wood suggested that some chemical reactions (decrease in hydroxyl groups) might take place during heating, accounting for dimensional stability rather than it being the sole effect of the preservative itself.

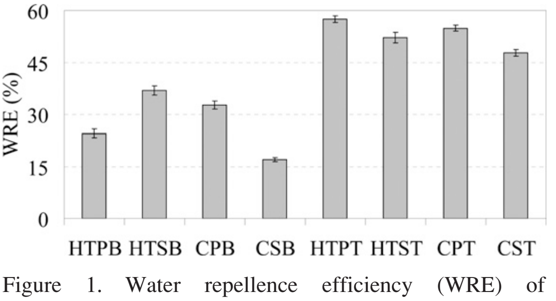

Figure 1. Water repellence efficiency (WRE) of preservative-treated wood after soaking in water at 21°C for 9 da. HTPB, heat-treated pine with Beckers; HTSB, heat-treated spruce with Beckers; HTPT, heat-treated pine with tar; HTST, heat-treated spruce with tar; CPB, control pine with Beckers; CSB, control spruce with Beckers; CPT, control pine with tar; CST, control spruce with tar. Error bars represent ±1 standard error.

Water Absorption

Figure 2 shows water absorption of HT and control wood samples during water soak at 21°C for 9 da. Compared with the control sample, HT pine and spruce had higher water absorption. Interestingly, an exception to increased water absorption by HT spruce compared with all samples was observed at the later stage of soaking, although HT spruce is reported to have decreased water absorption compared with HT pine (Metsä-Kortelainen et al 2006). The reason is not quite clear. However, it can be concluded from the later stage of soaking that HT spruce had much void space left to be filled with water and thus water absorption was still increasing. When HT and control samples were impregnated with preservative solutions, water absorption greatly decreased. Tar- and Beckers-treated samples performed differently with respect to water absorption. After 9 da of soaking, HT pine impregnated with tar had 58% lower water absorption than HT pine without preservative, whereas it was 52% lower for spruce. In the control sample, the highest water absorption was observed in pine, as expected. It is believed that water absorption of treated wood was related to oil properties. When control pine and spruce were treated with preservatives, water absorption also greatly decreased.

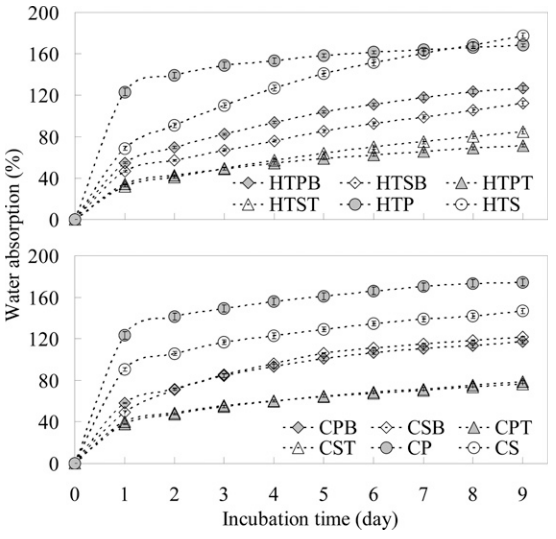

Figure 2. Water absorption of preservative-impregnated heat-treated and control wood samples during soaking in water at 21°C for 9 da. HTPB, heat-treated pine with Beckers; HTSB, heat-treated spruce with Beckers; HTPT, heat-treated pine with tar; HTST, heat-treated spruce with tar; HTP, heat-treated pine; HTS, heat-treated spruce; CPB, control pine with Beckers; CSB, control spruce with Beckers; CPT, control pine with tar; CST, control spruce with tar; CP, control pine; CS, control spruce. Vertical bars indicate ±1 standard error.

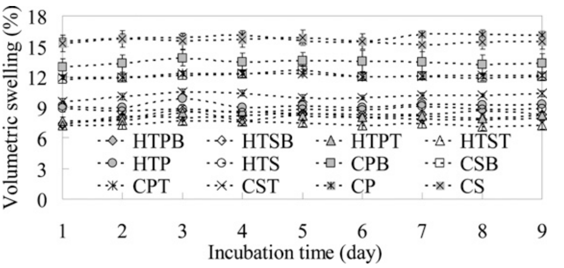

Figure 3. Volumetric swelling of preservative-treated wood samples during soaking in water at 21°C for 9 da. HTPB, heat-treated pine with Beckers; HTSB, heat-treated spruce with Beckers; HTPT, heat-treated pine with tar; HTST, heat-treated spruce with tar; HTP, heat-treated pine; HTS, heat-treated spruce; CPB, control pine with Beckers; CSB, control spruce with Beckers; CPT, control pine with tar; CST, control spruce with tar; CP, control pine; CS, control spruce. Vertical bars indicate ±1 standard error.

Volumetric Swelling

Figure 3 shows volumetric swelling of HT and control wood samples during water soak at 21°C for 9 da. Maximum swelling was achieved after 1 da of soaking for all treatments. Afterward, swelling rate was found to be almost stable. HT spruce and pine were more dimensionally stable than control samples. Tar-treated wood showed better dimensional stability than Beckers-treated wood. This could be attributed to superior hydrophobicity of tar formulation. This agrees with known circumstances discussed by Wang and Cooper (2005). They showed that oil treatment literally can improve dimensional stability of white spruce. Water repellent compounds block macropores by depositing hydrophobic compounds in the cell lumen. As a result, this kind of treatment decreased water uptake, consequently resulting in a lower swelling effect. Our experimental results showed that differential swelling decreased in HT samples compared with the control.

As previously discussed in the review of literature, the success of preservative-treated wood depends on how deep and uniform the distribution occurred. Therefore, preservative distribution in different cells of HT wood should be examined. Furthermore, outdoor performance of treated wood cannot be predicted from this study. Durability in laboratory and field tests is yet to be conducted. However, this study clearly shows that a considerable amount of preservative can be impregnated in wood without applying any external vacuum or pressure. This kind of secondary wood treating technique is new and simple and can be implemented after performing the durability test.

CONCLUSIONS

HT Scots pine and Norway spruce were impregnated with water-repellent wood preservative (Beckers and tar) successfully without applying any external vacuum or pressure. Particularly, HT pine retained a considerable amount of wood preservatives, fulfilling the oil-based preservative retention prescribed under class A by the NWPC. After preservative treatment, a conspicuous decrease in moisture adsorption and improved dimensional stability was observed.

Higher ASE values of control wood suggest that heat treatment before preservative impregnation accounted for the improved wood properties (decreased hydrophilicity and improved dimensional stability) rather than it being the sole effect of water-repellent preservative itself. In this regard, pine tar showed better performance than Beckers. Water absorption and volumetric swelling of HT wood dramatically decreased after preservative treatment, especially with tar. However, tar significantly increased WRE of HT wood compared with that of Beckers.

This study suggests that of the two wood preservatives studied, tar is superior to Beckers for improving moisture-repellent performance. Chemical reactions occurring in wood during thermal modification account for increased hydrophobicity and dimensional stability of HT wood. However, those properties can be enhanced by impregnating HT wood with tar in a simple and easy method. Secondary treatment of HT wood with water repellent tar enhances its performance by decreasing water absorption.

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