# CERNE

# Surface Wettability Of Boron And Oil-Treated Wood

# Eylem Dizman Tomak\*iD

Forest Industry Engineering Department, Bursa Technical University, Faculty of Forestry, Bursa, Turkey

# TECHNOLOGY OF FOREST PRODUCTS

# ABSTRACT

**Background:** Nowadays, demands for more environmentally friendly and cost-effective preservatives are increasing, and new non-traditional procedures are being explored in wood protection field. Plant oils improve the dimensional stability, water repellency and equilibrium moisture content of wood, and protect wood against decay fungi by means of its hydrophobic properties. The aim of this study is to investigate the influence of heat treatment and oil impregnation with or without prior treatment with boron compounds on wetting characteristic of Scots pine (*Pinus sylvestris* L.) and beech (*Fagus orientalis* L.) wood. Wood specimens were impregnated with boric acid, borax and agricultural boron at concentrations of 1%, 2% and 5% followed by oil heat treated with waste and sunflower oil at 160°C. Wettability was measured by contact angle with the sessile drop technique using water.

**Results:** Water contact angles on oil treated specimens increased while wetting tension decreased, and the wood more poorly wetted by water compared to the controls. A change in the drop volume on the surface of double treated specimens was around 5% based on the initial drop volume. Waste oil treatment resulted in having the greatest water repellent efficiency. High loadings of boron compounds decreased the contact angle and therefore the quantity of water absorbed by the wood increased.

**Conclusion:** Wettability was decreased in specimens pretreated with boron and this confirmed that the hydrophobic surface was created by oil. Wettability is a prerequisite for good adhesion, coating and painting and this feature may be reduced by the less hydrophilic surfaces created after oil heat treatment.

Keywords: Boron compounds, contact angle, hydrophobicity, oil heat treatment, wood protection

# HIGHLIGHTS

Waste oil treatment resulted in having the greatest water repellent efficiency. Change in the drop volume was around 5% for wood surface treated with boron and oil. Oil heat treatment improved surface hydrophobicity of boron treated specimens. High loadings of boron compounds, especially TB and BX, decreased the contact angle.

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e-mail: eylem.dizman@btu.edu.tr

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# **INTRODUCTION**

Wood is a truly renewable material that is commonly used in many diverse indoor and outdoor situations in construction and in many other applications. However, a combination of biological, chemical and physical processes can cause wood degradation and deterioration (Goldhahn et al., 2021). Water plays an important role in these processes (Zabel and Morell, 1992). Thus, to ensure a long, useful and safe life in end-use applications, wood needs to be protected against fungal decay and weathering (Ayanleye et al., 2022), and for this reason, treatments for the protection of wood and wood surfaces attract much attention, particularly nowadays, since depletion of natural resources is becoming a major problem all around the world (Nawaz et al., 2019).

Protection systems and preservatives for wood should to be safe in use and have low mammalian toxicity (Schultz et al., 2007). The use of boron compounds as an alternative wood preservative is promising, since the use of heavy metal containing wood preservatives is subject to restrictions (Tomak et al. 2011a; Broda, 2020). Boron compounds act as both an insecticide and fungicide and can also reduce thermal degradation, whilst they are widely accepted to have low mammalian toxicity (Ghani, 2021). However, boron compounds may accentuate the hygroscopicity of wood as well as leaching-out in outdoor conditions (Baysal et al., 2006; Ermeydan et al., 2019; Demirel et al., 2021; Ghani, 2021). Less water sensitive systems can be obtained by adding water repellents, such as oils, to common wood preservatives (Koski, 2008). Additionally, such a system may reduce the leaching of toxic biocides into the environment (Treu et al., 2004). The possible synergistic effect of dual treatments combining various biocides and oils on wood decay, biocide leaching and water repellency has been reported in number of studies (Palanti and Susco 2004; Lyon et al. 2007; Temiz et al. 2008a; Podgorski et al. 2008; Tomak et al. 2011a, b; Panov and Terziev, 2015; Can and Sivrikaya, 2017; Can et al., 2018; Demirel et al., 2018, 2021; Babar et al., 2021; Lucia et al., 2021).

Treatments with environmentally friendly and biodegradable oils are known to reduce capillary water uptake by creating a protective layer on the wood surface (Lee et al., 2018; Schwarzkopf et al., 2018). Since oil treatment improves the hydrophobicity of the wood surface, the rate of water absorption is limited and the risk or rate of fungal attack is reduced (Koski, 2008; Temiz et al., 2008a; Tomak et al., 2011b; Jebrane et al., 2015; Demirel et al., 2018). Using contact angle measurements, it was reported that impregnation with crude tall oil clearly enhances the hydrophobic properties of wood (Koski, 2008). A reduction in the hydrophilic behavior and swelling of wood as a result of thermal modification in soybean oil was found by Awoyemi et al. (2009). Demirel et al. (2018) impregnated wood specimens with

boric acid and epoxidized linseed and soybean oils and reported lower water absorption values than that of the controls along with an anti-swelling efficiency of 70%. Similar behavior in decreasing the water absorption rate in oil treated wood was also noted by Can and Sivrikaya (2016). The improvement in water repellency during an oil treatment depends on the oil type and its properties, process time and temperature as well as oil retention (Tomak et al., 2011b). For good long-term performance, deep penetration and a high retention of the oil in the wood is required. Rather strong negative linear relationships were found to exist between the oil retention level in wood and the water absorption rate in oil treated wood (Panov et al., 2010; Demirel et al., 2018).

Water repellents can considerably reduce hygroscopicity, water absorption and shrinkage, as well as increase the water repellency and anti-swelling efficiency of wood treated with boron compounds (Baysal et al., 2006; Tomak et al., 2011b; Demirel et al., 2018). Tomak et al. (2011b) studied wood treatment with boron compounds and hot vegetable oils finding that such a treatment reduced water absorption and improved water repellency efficiency. The reduction in the water absorption rate of spruce specimens that had been oil heat-treated at 200-220°C for 2-4 hours was believed to be due to both oil absorption and chemical changes occurring in wood during heat treatment (Wang and Cooper, 2005). It is now well known that heat treatment causes various changes in wood; the hydrophobicity and equilibrium moisture content of heat-treated wood is reduced and its dimensional stability and biological durability are improved (Ali et al., 2021). Wettability is decreased by the degradation of hemicelluloses resulting from the reduction in -OH groups and the formation of O-acetyl groups (Petrissans et al., 2003; Kocaefe et al., 2008a; Awoyemi et al., 2009). It is also reported that the change in wettability of heat-treated wood may be due to the plasticization of the lignin and a reorganization of the polymeric compounds (Hakkou et al., 2005).

The property of the wood surface of absorbing liquids (water, paint, glue) into it has an effect on its paintability and glueability (Darmawan et al., 2020). Two important surface properties, wettability and surface free energy, can be studied by contact angle measurements with sessile drop technique (Kocaefe et al. 2008b; Darmawan et al., 2020, Martha et al., 2020). In earlier investigations, boron-treated wood becomes more hydrophobic with an oil treatment, and treatments combining boron and oil seem to have some promising synergistic effects on boron leaching and resistance to biological attack (Tomak et al. 2011a, b; Demirel et al., 2021). Contact angle is expected to increase as a result of such treatment and this is likely to have an impact on industrial applications such as gluing, painting and coating (Petrissans et al., 2003; Martha et al., 2020). Consequently, it is important and necessary to study the influence of heat treatment and oil impregnation on wood's wetting characteristics. To the author's knowledge, there is a gap in research on the wetting behavior of boron and oil-treated wood, which the study reported herein sought to address.

In this study, the wettability of beech and Scots pine wood specimens treated with boron compounds and vegetable oils was investigated by using the sessile drop technique. Contact angle measurements were carried on the tangential surfaces of wood specimens treated with boric acid, borax and agricultural-boron compound solutions, having concentrations of 1%, 2% and 5%. Waste vegetable oil and sunflower oil were used both separately and in combination with the boron compounds.

# **MATERIAL AND METHODS**

# Material

Sapwood specimens of Scots pine (*Pinus sylvestris* L.) and beech (*Fagus orientalis* L.), having dimensions 20 x 20 x 20 mm (R x T x L) were used in this study. Boric acid (BA), borax decahydrate (BX) and agricultural-boron product (TB) were provided by the Turkish National Boron Research Institute (BOREN), Turkey. For impregnation, aqueous boron compound solutions, having concentrations of 1%, 2% and 5% (w/v) were prepared using distilled water. Sunflower oil was provided by Ordu Oil Company, Turkey, whilst used (waste) vegetable oil was obtained from various fast- food restaurants in Trabzon, Turkey.

Scots pine (Pinus sylvestris L.) is the most widely distributed member of the Pinaceae family in the world (Bilgen and Kaya, 2007). It is one of the main tree species in European forests and occupies 1.48 million ha of the total of 21.68 million ha of Turkish forests (Lee et al., 2016). Scots pine has superior technological properties and high usage potential in wood industry (Korkut et al., 2008). The eastern beech (Fagus orientalis L.) is a common hardwood tree specie that regenerates naturally in Turkish forests where species diversity is rich due to the variety of growing conditions (Ertekin et al., 2015). The beech forests in Turkey cover 615.000 ha and have a standing volume of 154 million m<sup>3</sup>, making up almost 20% of the country's total standing wood volume (Esen et al., 2004). Beech wood has economic importance since it is hard and heavy, and it has a wide variety of usages in wood industry (Topaloglu et al., 2016).

## Impregnation procedure for boron compounds and oils

Wood specimens having a moisture content of 12% were first vacuum impregnated with the boron solutions at 760 mmHg for 60 min at room temperature. Following this, the specimens remained immersed in the solutions for 60 min at atmospheric pressure. The boron retention level of the specimens (kg/m<sup>3</sup>) was calculated. The treated specimens were subsequently conditioned for 2 weeks at  $20\pm2^{\circ}$ C and 65% relative humidity. Oil heat treatment was performed in a two-stage process in oil baths at

atmospheric pressure as described by Lyon et al. (2007) and Podgorski et al. (2008). Boron treated and untreated wood specimens were first placed in an oil bath containing oil at ambient temperature and the temperature raised to 160°C at a rate of 5°C/min. Once the target temperature had been reached, the temperature was held constant for 30 minutes after which the specimens were quickly transferred to another bath containing oil at room temperature where they were submersed for a further 30 min. The specimens were not placed directly into the hot oil at 160°C in order to prevent crack formation. Oil retention values (kg.m<sup>-3</sup>) were also calculated.

### **Contact angle measurement**

Four replicates in each treatment group were conditioned at a temperature of 20±2°C with 65% relative humidity for 30 days before the test. A KVS -CAM 200 system was used to determine contact angle, drop volume and surface tension at 1 second (s) intervals for a period of 60 seconds. The measurement technique is based on rapid video capture of images and automatic image analysis. Four drops of 5 µl volume were formed automatically using a pump and deposited on the tangential surface of the specimens at room temperature. The change in volume of the drop (%) was calculated on the basis of the initial drop volume. Wetting tension, which is defined as the surface tension multiplied by the cosine of contact angle (Kocaefe et al., 2008b), was also calculated. Contact angle was measured three seconds after the drop was placed on the wood surface in order to see the actual effect of treatments on contact angle. The tangential surface was chosen for the study since these surfaces were smoother than the radial surfaces which exhibited wavy growth or grain.

# **RESULTS AND DISCUSSION**

The retention of oil and boron compounds (kg.m<sup>-3</sup>) in the treated wood specimens is summarized in Table 1. As expected, high retention values were obtained with Scots pine due to it being one of the most easily impregnated wood species. For both wood species, waste oil showed lower retention than sunflower oil in general. The probable reason for the different retention values between the oil treatments could be the different viscosity and penetration properties of the oils. Boron pretreatment had a greater negative effect on oil uptake in Scots pine than on beech. A possible explanation for this is that boron crystals, formed in wood following pre-impregnation, might inhibit oil uptake into the small void spaces of wood. Permeability and density differences between the wood species could also affect the retentions.

The wettability of wood can be characterized by contact angle or wetting tension (Petrissans et al., 2003; Kocaefe et al., 2008b). Figures 1-3 show the contact angle (°) and change in volume of the drop relative to the initial volume (%).

#### Table 1. Boron and oil retentions (kg.m<sup>-3</sup>) in the treated specimens.

Treatment Groups		Retentions (kg/m <sup>3</sup> )		
		Boron	Waste Oil	Sunflower Oil
1% BA	Pine	6.0 (0.3)*	494.2 (9.4)	530.3 (19.5)
	Beech	5.7 (0.2)	361.5 (16.5)	364.5 (16.5)
2% BA	Pine	11.4 (0.4)	378.8 (16.8)	469.6 (33.8)
	Beech	11.3 (0.4)	364.3 (16)	377.8 (10.4)
5% BA	Pine	38.5 (1.0)	510.8 (19.9)	515.3 (39.4)
	Beech	29.4 (0.8)	381 (24.4)	391.5 (12.2)
1% TB	Pine	6.6 (0.1)	399.2 ( 7.6)	527.8 (10.1)
	Beech	5.8 (0.1)	387.5 (15.8)	402.9 (14.2)
2% TB	Pine	12.7 (1.7)	380.9 (44)	496.3 (23.9)
	Beech	11.7 (0.3)	352.2 (25)	411.3 (11.1)
5% TB	Pine	31.9 (1.8)	567.8 (6)	531.9 (38.6)
	Beech	22.2 (2.5)	396.3 (37)	369.1 (27.5)
1% BX	Pine	5.2 (0.2)	414.2 (35)	536.6 (23.5)
	Beech	4.7 (0.1)	303.8 (13.2)	362.8 (15.0)
2% BX	Pine	12.0 (0.9)	508.8 (8.8)	505.8 (23.4)
	Beech	8.2 (0.2)	305.4 (24.2)	366.9 (11.3)
5% BX	Pine	35.8 (0.7)	412.2 (55.5)	494.1 (19.7)
	Beech	19.7 (0.4)	310.8 (16.6)	343.7 ( 9.0)
-	Pine	-	435.1 (34.6)	556.0 (31.5)
		-	338.4 (26.4)	349.0 (28.4)

\* Values in parentheses are standard deviations.



**Figure 1.** Comparison of contact angles (a) for BA + oil treated pine specimens (b) for TB + oil treated pine specimens. (c) for BX + oil treated pine specimens.



**Figure 2.** Comparison of contact angles (a) for BA + oil treated beech specimens (b) for TB + oil treated beech specimens. (c) for BX + oil treated beech specimens.

As may be seen from Figures 1 and 2, the contact angle of oil heat-treated specimens was greater and more stable over time than those of the controls and specimens treated with only boron compounds. The average contact angle of specimens treated with boron + waste oil was in a range of 57-84°. Regardless of the boron compound and its concentration, waste oil treated wood surfaces seemed to be more hydrophobic, showing greater contact angle and stability than the surfaces of boron + sunflower oil treated specimens did. This could be explained by the different penetration rates and viscosity of the oils during impregnation, and the drying properties of the oils when exposed to air (Lee et al., 2018). The drying characteristics of oils may have an effect on the creation a hydrophobic layer on the wood cells, preventing water uptake. BA + waste oil treated specimens displayed an average contact angle of around 81-84° for the pine and 78-84° for the beech specimens. For BA + sunflower oil, the contact angle on treated pine was found to be 62-64° and on treated beech around 64-67°. Contact angle values decreased slightly with increasing boric acid concentration in the BA + waste oil treated specimens (Figures 1a and 2a). However, this tendency was not clear for the BA + sunflower oil treated specimens. The average contact angle of TB + waste oil treated pine and beech specimens was in the range 58-83° and 72-80°, respectively. For TB + sunflower oil, the contact angle was found to be 61-69° on treated pine and 66-75° on beech. In the combined treatment using the TB biocide, contact angle values were seen to decrease with increasing agricultural boron compound concentration in both wood species (Figures 1b and 2b). BX + waste oil treated specimens showed contact angles of around 57-81° for pine and 63-78° for beech. For the BX + sunflower oil treated pine the contact angle was between 61 and 71° and for beech between 73 and 77°. Contact angle on both pine and beech wood was observed to decrease with increasing borax concentration in the combined treatments (Figures 1c and 2c). Average contact angle of controls was found to be 22° for pine and 24° for beech. In systems involving water as the liquid phase, surfaces forming contact angles of less than 90° are said to be wettable or hydrophilic, whereas those giving rise to contact angles greater than 90° are said to be water-repellent or hydrophobic (Koski, 2008). None of the treatment groups exhibited a water repellent or hydrophobic surface since the observed contact angles were less than 90°. Nevertheless, oil heat treatment increased the hydrophobicity of the wood surfaces and, therefore, the wettability of wood by water decreased compared to the controls. This has also been demonstrated by increased water repellency efficiencies and decreased water absorption in specimens treated with boron and oils (Tomak et al., 2011b). Findings similar to the ones described herein were reported by Demirel et al., (2018) on wood which had been treated with boric acid and epoxidized linseed and soybean oils. In addition, impregnation with waste engine oil clearly improves the hydrophobic properties of wood (Belchinskaya et al., 2021). Two possible reasons for the poor wettability are the high oil uptakes of wood that create water repellent surfaces, and thermal treatment. Thermal processes applied to wood affect wettability and enhance its hydrophobicity (Petrissans et al., 2003; Aydin and Colakoglu, 2007; Kocaefe et al., 2008b; Chu et al., 2016;

Karlinasari et al., 2018; Fu et al., 2019). Hydroxyl groups have an important role in the wettability of wood surfaces. Lignin is not as hygroscopic as cellulose and hemicellulose. FTIR-PAS spectra of the specimens showed some changes, suggesting that thermal degradation of the wood components had taken place during the oil heat treatment at 160°C (Tomak et al., 2011a). A reduction in the number of free hydroxyl groups accessible to water makes wood more hydrophobic (Kocaefe et al., 2008a). Similar or higher contact angles were obtained with low concentrations of boron compounds in specimens that had undergone the combined treatments compared to only oil treated ones. A negative linear relationship was found to exist between the concentration level of TB and BX in wood and the water contact angle in double treated pine wood. This might be a result of the hygroscopic character of boron compounds. In general, BA demonstrated higher contact angles than the other boron compounds in combined treatments. Similar findings were also observed in the water absorption tests by Tomak et al. (2011b). In the case of pretreatment with 5% BX and 5% TB in the combined treatments of pine and beech specimens, contact angles decreased to around 57-61° and 63-75°, respectively. Boron crystals could be seen by the naked eye on the wood surface at high boron loadings. The presence of boron crystals on the surface could change the contact angle and cause the spreading of the drop on the surface. The collection of boron crystals on veneer surfaces and some bonding problems between the adhesive and wood was also reported by Aydin and Colakoglu (2007).

Boron treatment alone showed a tendency for the contact angle to decrease over time, but the average contact angle of boron treated specimens was still higher than the contact angle of controls. The hygroscopic nature of boron salts might have an adverse effect on the hydrophobicity of wood (Demirel et al., 2018). Some boron might dissolve from the wood surface to water drop, thereby affecting the wettability measurements. It is well known that borates are easily leached, due to their high-water solubility, when treated wood is exposed to liquid water. Walinder (2007) reported that severe contamination of probe liquids may occur during measurements. The contamination is caused by dissolution or by the presence of extractives at the wood-liquid interface. and it generally results in a distinct decrease in the liquid surface tension, probably due to reorientation of functional groups due to oxidation, at the wood/extractives-air interface (Walinder, 2007). The contact angle of TB and BX treated specimens decreased with increasing chemical concentration, whilst BA treated specimens behaved somewhat differently. Surfaces of specimens treated with 5% BA showed higher contact angles than the surfaces of TB and BX treated specimens. Contact angle could not be measured with 5% BX treated pine specimens due to the initially high spreading rate of the drop on the surface (Figure 1c). Toker (2007) reported that borax treated wood exhibited greater hygroscopicity than boric acid treated wood. This is also in agreement with the previous findings on borax which is more hydrophilic than boric acid because of its high pH value of around 9.2 (Ramos et al., 2006). The solubility rate of TB in water at room temperature is more than three times greater than that of boric acid in water (Tomak et al., 2011a). Between the boron compounds, boric acid is hardly soluble in water at room temperature especially at high concentrations. The difference between the solubility

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of boron compounds in water might affect the wettability properties of the wood surface and therefore the spreading of the drop (Alade et al., 2021). Water repellency also reflects several wood-related factors, such as the nature of the bond between the preservative salt and the wood components, the crystalline or amorphous nature of the preservative deposit and the mechanical accumulation of preservative in certain parts of the wood structure (Rak, 1975).

The average change in volume of the sessile drop with respect to its initial volume on the treated specimen surfaces is presented in Figure 3. Figure 4 shows wetting tension of the specimens that gives an indication of the level of wettability (Kocaefe et al., 2008b). The surfaces of the control specimens showed the greatest change in droplet volume. Droplet volume decreased markedly during the experiment showing that water is absorbed by the wood. The drop was completely spread after 40 seconds, the total test period for the control pine specimens. Volume changes have a direct influence on the contact angle measurements (Kocaefe et al., 2008b). The quantity of water absorbed by wood, the spreading rate of the droplet and wetting tension were decreased on oily wood surfaces. Boron treated specimens showed higher wetting tensions than the oil treated specimens did and had lower wetting tension than the control specimens.

BA + waste oil treated specimens showed an average change in drop volume (%) of around 1-4% at the end of the test for pine and 1-3% for beech specimens, respectively. For BA + sunflower oil treated pine, this was found to be 2-3% and for beech specimens 2-4%. The change in drop volume (%) of TB + waste oil treated pine and beech was in the range 2-4% and 2-5%, respectively. It was found to be 3-6% for TB + sunflower oil treated pine and 2-3% for beech specimens. BX + waste oil treated specimens showed a change in drop volume (%) of around 3% for both pine and 1-5% for beech specimens. It was determined to be between 2-4% for BX + sunflower oil treated pine and 2-3% for beech specimens. Average contact angle of only oil treated specimens was found to be 2-5% for pine and 3% for beech depending on oil type. Kocaefe et al. (2008b) reported changes in the drop volume on heat-treated wood compared to untreated controls of around 13% and 45% after 30 seconds of measurement. In general, the change in the volume of the drop (%) increased slightly with increasing boron concentration for the combined treatment specimens in both pine and beech wood. The change in the wetting tension of 1% and 2% boron + oil treated specimens was negligible compared to only oil treated specimens. However, there was a sharp increase in the wetting tension of 5% TB and 5% BX pretreated and oil treated pine specimens. This is probably due to the high boron loading increasing the hygroscopicity of wood. Similar to the contact angle findings, in all the groups tested, waste oil treatment combined with the BA pretreatment seemed to result in the least change in drop volume. These specimens also showed better or similar wetting tension compared to the waste oil treated ones, depending upon the BA concentration. Wood surfaces treated with TB and BX were much more easily wetted than those treated with BA. As explained previously, this might be due to the different chemical composition and function of the chemicals. This is in agreement with a previous study dealing with the water repellency efficiency and water absorption of wood treated with a combination of boron and oils (Tomak et al., 2011b).



**Figure 3.** Change in volume of the drop relative to the initial volume (%) (a) for Scots pine specimens (b) for beech specimens.



**Figure 4.** Calculated wetting tension (mN/m) (a) for Scots pine specimens (b) for beech specimens.

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It was observed that the wetting tension of pine specimens was greater than that of beech specimens. Wettability of wood is influenced by many factors including macroscopic characteristics of wood such as porosity, moisture content, fiber orientation, etc. (Kocaefe et al., 2008b). The small difference in contact angles between the untreated softwoods and hardwoods is likely due to anatomical differences and the energetic properties of the two types of wood (Moura and Hernandez, 2005). Extractives in the wood species also play an important role on wettability and bonding (Roffael, 2016). Many wood properties depend on the density of the wood (Bozkurt et al., 1993). As the density of wood increases, its permeability will decrease, and there is a linear relationship between the permeability and uptake of chemicals in wood (Bozkurt et al., 1993). Scots pine wood has a higher permeability than beech wood; this may be the reason for the higher wettability of Scots pine. Surface roughness also affects the wetting characteristic of a solid (Sheng et al., 2007). A detailed investigation into the surface roughness of treated specimens could reveal the reasons for the different wetting tensions of these wood species.

# CONCLUSIONS

The results indicated that waste oil treatment with or without boron treatment caused a greater decrease in the wettability of wood than sunflower oil. High loadings of boron compounds, especially TB and BX, decreased the contact angle and therefore the quantity of water absorbed by the wood was increased. Wettability was decreased in specimens pretreated with boron and this confirmed that the hydrophobic surface was created by oil. On the other hand, wettability is a prerequisite for good adhesion, coating and painting and this attribute may be reduced by the less hydrophilic surfaces created after oil heat treatment. Treated wood is mainly used in outdoor applications where it is exposed to weathering factors such as ultraviolet (UV) irradiation and water. The greater wettability of weathered wood accelerates deterioration of wooden structures. Treatment with oils increases the hydrophobicity of wood surfaces and this may be an important improvement for durability in outdoor conditions.

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# **AUTHORSHIP CONTRIBUTION**

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