

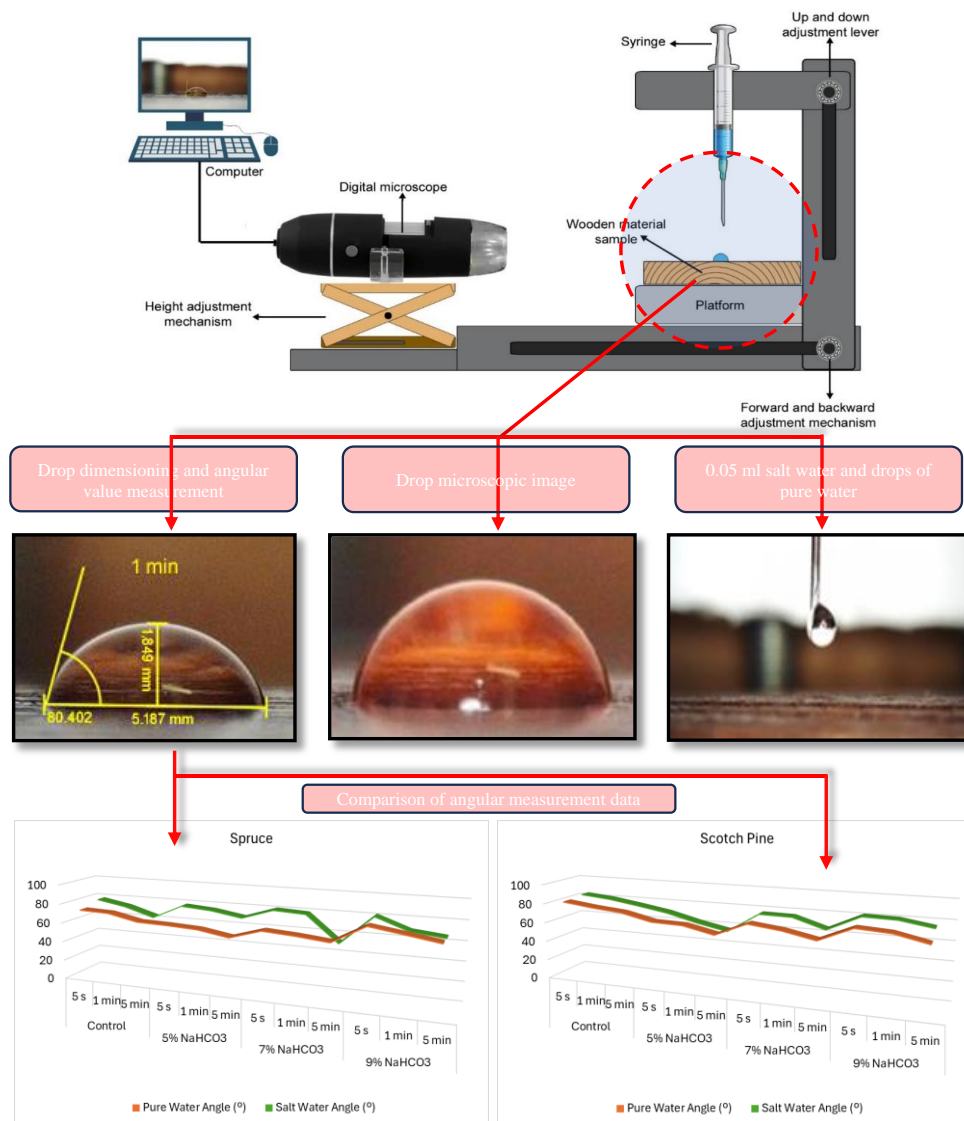
Analysis of Wettability and Contact Angle of Sodium Bicarbonate Impregnated Wooden Surfaces

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GRAPHICAL ABSTRACT



Analysis of Wettability and Contact Angle of Sodium Bicarbonate Impregnated Wooden Surfaces

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Scots pine and spruce wood samples were vacuum impregnated with 3, 5, and 9% sodium bicarbonate solution. Contact angle measurements were made by dropping 0.05 mL of pure water and 3% saltwater solution onto the material's surface. The surface roughness of each sample was measured, and that of Scots pine control samples was higher than that of spruce samples, but the surface roughness values of spruce wood were higher in the samples impregnated with sodium bicarbonate. When pure water or saltwater solution was dropped onto the samples, it was observed that as the waiting times increased, the contact angle value decreased, the droplet height decreased, and the droplet width increased. It was found that the contact angles were higher in the control samples of both tree species than in the samples of 5% and 7%. The contact angles of 9% impregnation solution, pure water, and salt water were higher than the control samples. As the solution ratio increased on the surfaces impregnated with sodium bicarbonate, the contact angle also increased, and the wettability behavior decreased accordingly. Sodium bicarbonate solution is effectively used in the impregnation process for pine and fir woods, making the materials more resistant to water under outdoor conditions. This solution significantly reduces the risk of deformation of wood by increasing the contact angle and reducing its water absorption properties.

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Keywords: Impregnation; Sodium bicarbonate; Wettability; Contact angle; Optimization

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INTRODUCTION

Wood is a natural building material that has been used in different application areas since ancient times because it is a renewable and sustainable material (Kilinçarslan and Türker 2020). However, the shrinkage, swelling, and deformation of the wood material that occurs because of nature and biological pests (insects and fungi) is one of the most common industry problems (Meena 2022).

The important difficulties regarding the use of wood materials produced in industry are listed as durability, sustainability, resistance to destruction by biotic organisms, and dimensional stability (Bekhta and Krystofiak 2016). In contrast, the wood used outdoors must be resistant to the climatic conditions of the environment and the destruction by biological organisms. In these cases, it can be said that the surface wettability of the wood is an effective attribute. The contact of wood with water is one of the main factors that initiate biological degradation. The general goal to increase the durability of the material is to increase hydrophobia or reduce hygroscopicity (Li *et al.* 2021).

Coating applications, such as varnish, are widely applied to wooden surfaces. The coating protects the wooden surface and gives it the desired appearance. The coating aims to give the wood better aesthetic value and serves to cover some of the weaknesses of the wood, both in terms of color and texture. The coating protects the wood from external conditions such as weather, temperature, sunlight, or wood-damaging organisms (Darmawan *et al.* 2020).

Wetting refers to macroscopic manifestations of molecular interaction at the interface between liquids and solids in direct contact (Szymczyk 2011). Wetting is controlled by the surface tension of the liquid and the substrate and encompasses the processes of adhesion, penetration, and spreading; each of these phenomena is a distinctly different type of wetting (Collett 1972). An obvious way to investigate the wettability of a wooden surface is to determine the contact angle of a liquid in contact with the wood material (Patton 1970). Wettability can be understood by measuring the contact angle between liquid molecules and the surface of a solid material. The surface tension is affected by many parameters such as the fluidity of liquids, the drying process, the natural aging of wood, and the defects inherent in wood (Qin *et al.* 2015).

Wettability can be measured by determining the contact angle between a liquid drop and the surface of the wood material. If the contact angle is below 90°, it means that the dropped liquid can effectively wet the wood surface. If the contact angle is greater than 90°, it means that the dropped liquid cannot wet the wood surface sufficiently (Gavrilovic-Grmusica *et al.* 2012). Contact angle values of a varnish formulation greater than 90° indicate that the varnish liquid will have difficulty wetting a surface (Bracco and Holst 2013).

The wettability of wood material surfaces has been extensively investigated since the 1960s (Jacob and Berg 1993; Walinder and Johansson 2001; Hubbe *et al.* 2015). The stationary drop (Gray 1962) and Wilhelmy plate (Wilhelmy 1863) are the two most widely used techniques for wettability measurements. It has been understood that the Wilhelmy plate method provides significantly more consistent, accurate, and repeatable measurement data than the stationary drop method in heterogeneous and hygroscopic materials such as wood (Gaonkar and Neuman 1984; Sedighi *et al.* 2013). It was found important that this method is simple and that it measures not only the contact angle of the dropped water on the wood surface but also the absorption of water. In this way, it is possible to determine the water permeability potential of wood (Vaziri *et al.* 2021).

The aim of this study was to measure the wettability of yellow pine and spruce wood used in outdoor applications after impregnation with 5%, 7%, and 9% sodium bicarbonate solutions.

EXPERIMENTAL

Materials

In the study, Scots pine (*Pinus sylvestris* L.) and oriental spruce (*Picea orientalis* L.), which are coniferous tree species widely used in wooden structures and obtained from Artvin Forest Management Directorate, were used. The diameter of trunk height at 1.30 m, crown circumference, and tree length were determined for the trees marked in the north direction. Then, 2-m-long logs were cut between 2 to 4 m in the direction of the trunk height from the root. These logs were sawn in a private enterprise in Trabzon Furniture industrial site, and radial and tangential sections were obtained. To prevent blue coloration due to blue-stain fungus in Eastern spruce and Scotch pine sample pieces, anti-blue solution

was applied to the cross-sections at a concentration of 2.5% and all pieces were stacked and left to dry naturally for 5 months.

Methods

Impregnation

First, a system was developed for contact angle measurement. The system consisted of software and hardware. With the help of a syringe, 0.05 mL of pure water and 3% concentrated salt water were dropped onto the wooden materials and the angle they made with the surface was determined at 5 s, 1 min, and 5 min intervals. Through determining the contact angle of the water drops with the surface, information about their wettability was obtained. Since it is known that salt water has a more aggressive effect than normal water, it was used to determine its effect on surface roughness. In this study, sodium bicarbonate (NaHCO_3) compound, which is harmless to humans and the environment, was used as an impregnating agent. The molecular mass of the sodium bicarbonate used is 84.01 g/mol, its density is 2.22 g/cm^3 at $20 \text{ }^\circ\text{C}$, and its solubility in water is approximately 95.5 g/L at $20 \text{ }^\circ\text{C}$. For the test, air-dried test specimens were impregnated with sodium bicarbonate solution prepared at concentrations of 5%, 7%, and 9% using vacuum pressure method according to ASTM D1413-99 (1999) standards as shown in Fig. 1.

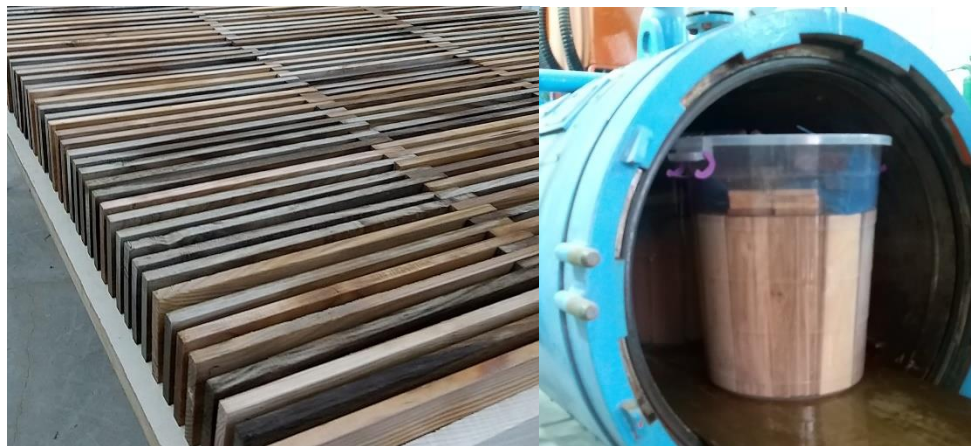


Fig. 1. Impregnated samples and vacuum boiler

After pre-vacuuming the wood samples at 750 mm Hg for 30 min, the impregnant material was taken into the cylinder and pressurized at 5 bar for 60 min.

Contact angle measurement

The contact angle of the water drop dropped on the wooden sample with the help of a syringe was measured with a digital microscope as shown in Fig. 2.

The wood samples were placed on a platform in the system, and approximately 0.05 mL of water or salt water was dropped onto the surface using a syringe. The dropping height of the water can be adjusted by sliding up and down with the device. The image of the water drop was taken with a digital microscope connected to the computer *via* USB. The back-and-forth adjustment mechanism at the bottom of the system and the optical zoom on the microscope were used to focus the image. In addition, a precise height adjustment mechanism under the microscope was used for the height of different materials. The images were processed and dimensioned on a computer using Celestron Digital Imager

HD software. In the study, 10x80x80 mm wooden test samples were prepared. 10 measurements were taken from different tangent section regions of each test sample and the average was taken.

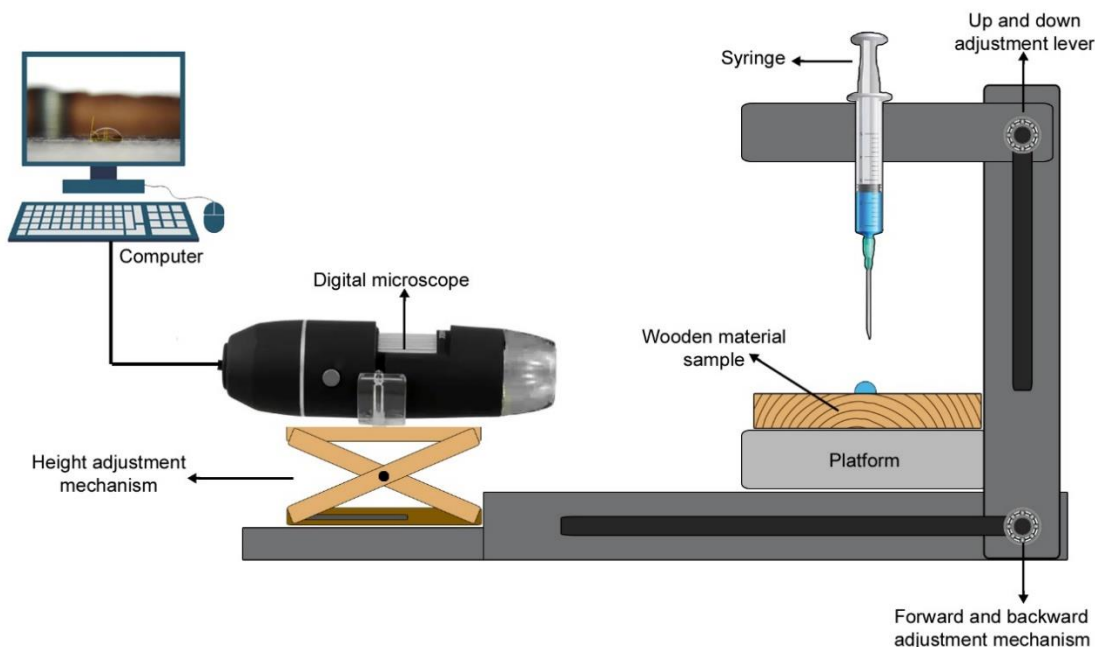


Fig. 2. The setup used for contact angle measurement

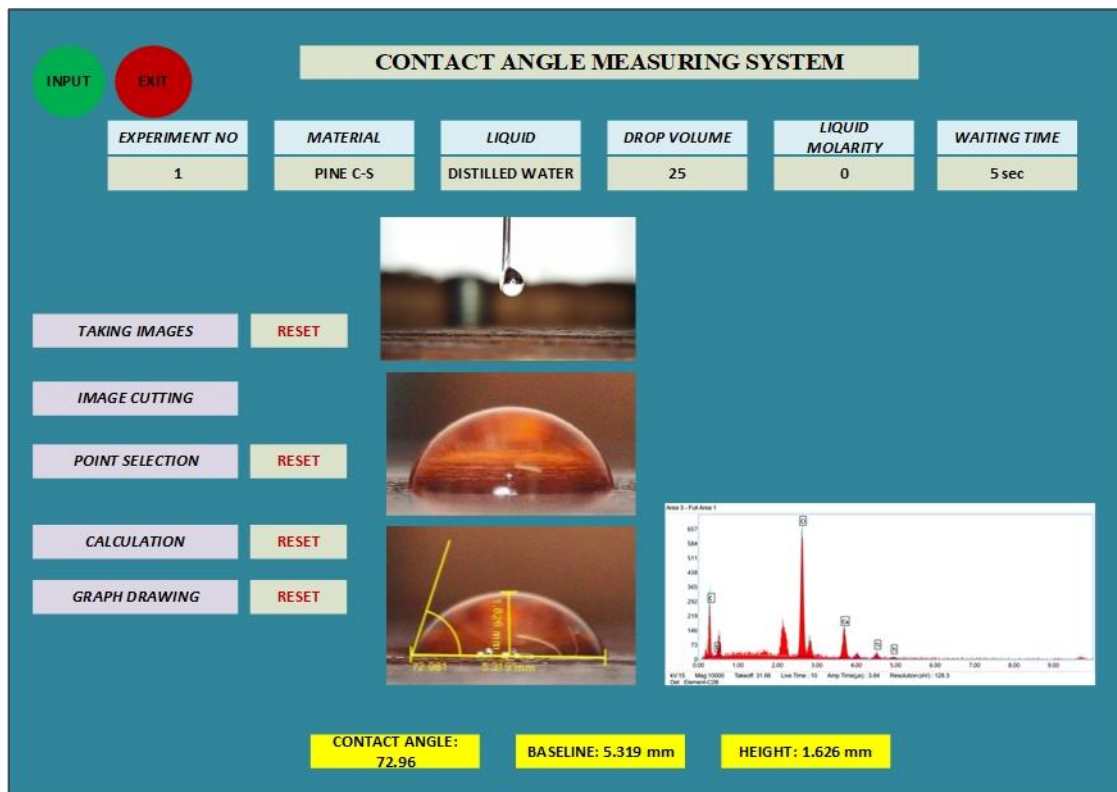


Fig. 3. MATLAB program and contact angle measurement interface

Using the interface prepared with the MATLAB program, the contact angle, the baseline of the drop, and the height of the drop were measured. With the interface, information about the measurement is stored in memory. The interface is shown in Fig. 3. The interface includes sections, such as the type of liquid used, the volume of the dropped liquid, the waiting time after the drop falls on the surface, image acquisition, point selection for measurement, calculation, and graphing from the measured values and displaying the results. The interface is flexible. The interface program can be developed when desired.

Wettability and contact angle

Wettability is defined in relation to the position of a liquid drop on a surface, with the drop taking a regular shape at a given angle θ to the surface. The interaction between liquid and solid was characterized by the cosine of the equilibrium contact angle θ for a given liquid. This equilibrium contact angle θ was used as an indicator of the wettability of the surface. Young's Eq. 1 describes the energy balance of the liquid contact angle on a surface (Young 1832),

$$\gamma_{sv} = \gamma_{sl} + \gamma_{lv} \cos \theta \quad (1)$$

where γ_{sv} denotes solid surface tension, γ_{sl} is the interfacial tension between solid and liquid, γ_{lv} is the liquid surface tension, and θ is the contact angle of the liquid at the solid-liquid-vapor boundary. Figure 4 shows a water drop and its contact angle with the surface.

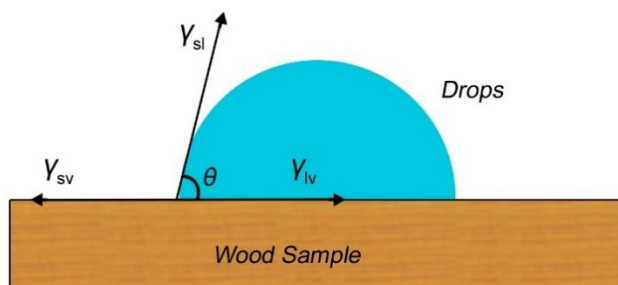


Fig. 4. Water drop and contact angle

Wettability refers to the determination of contact angles between a liquid and a solid that determines the degree of wetting. Low contact angles ($< 90^\circ$) represent good wettability and *vice versa*. Some factors, such as roughness of the surface structure, ambient conditions, time, temperature, and chemical reaction, can influence wettability behavior (Owens and Wendt 1969; Yuan and Lee 2013).

The wettability test was performed using a syringe and specially designed measuring equipment. Pure water and 3% brine were used in the experiments. Approximately 0.05 mL size drops were dropped onto the wooden surfaces. These drops were allowed to spread over the surface for 5 s. Digital microscope software was used to analyze the images and measure the contact angles. The drop base diameter, drop height, and contact angle measurements were performed with high precision using advanced microscope-based software.

Surface roughness

The Mitutoyo SJ-410 device, as shown in Fig. 5 was used for roughness measurement. The surface roughness test was performed according to the ISO 468 (1995) standard. The length of the surface pull is $L = 4.8$ mm, the speed is 0.5 mm/s, and $\lambda c = 0.8$.



Fig. 5. Surface roughness device

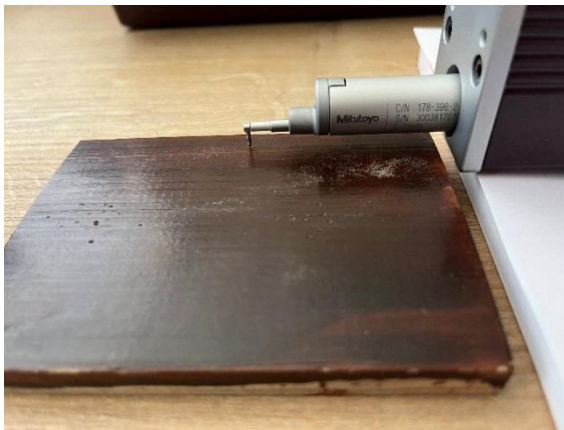


Fig. 6. Measured sample surface

The test specimens of height 10 mm were measured on a flat surface with the surface roughness device shown in Fig. 5. The measurements were made in the direction of the fibers of the wood material (along the annual rings) and on the smooth fibrous surfaces as shown in Fig. 6.

RESULTS AND DISCUSSION

Figure 7 shows the time-dependent contact angle change of 0.05 mL saline drop on pine control group samples at 5 s, 1 min, and 5 min. It is apparent that the width of the contact surface increased with the elapsed time and was inversely proportional to the drop height.

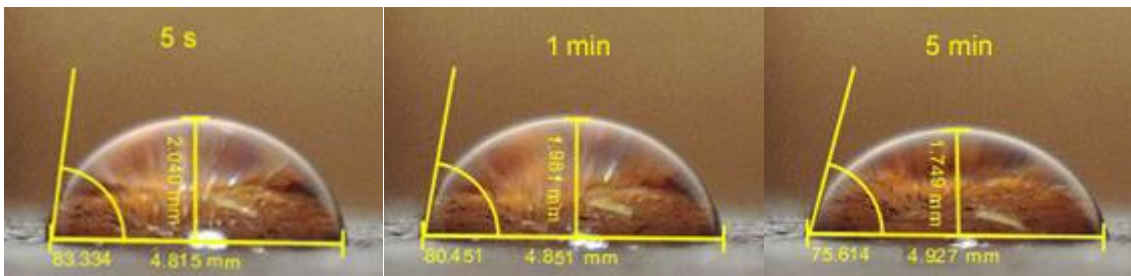


Fig. 7. Saline water surface contact angles of Scotch pine control group

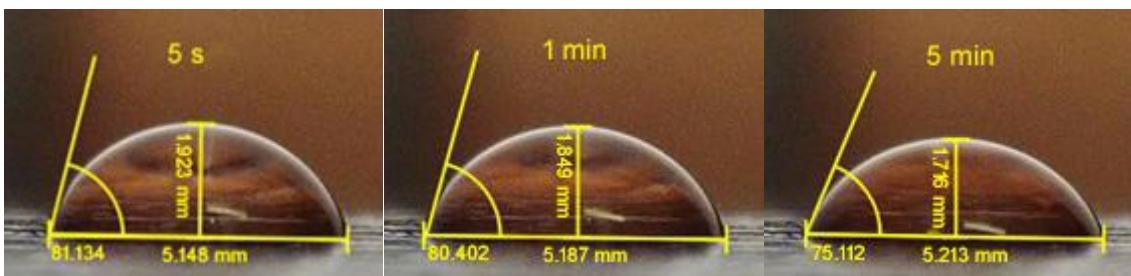


Fig. 8. Contact angles of scotch pine impregnated with 9% NaHCO_3 using brine-dropped surface

The time-dependent changes in the dimensions of the saline water samples dropped on the pine samples are shown in Fig. 8. As in the pure water samples, the contact angle

decreased in these samples as the contact surface increased and the drop height decreased with time. The contact angle was higher in the saline samples than in the pure water samples. Contact angles, contact surface widths, and drop heights measured after pure water and saline were dropped on the pine samples are given in Table 1.

Table 1. Contact Angle Values of Yellow Pine Samples

	Pure Water			Salt Water	
	NaHCO ₃	Duration	Angle (°)	Duration	Angle (°)
Scotch Pine	C	5 s	81.334	5 s	83.334
		1 min	77.451	1 min	80.451
		5 min	74.614	5 min	75.614
	5%	5 s	67.524	5 s	70.293
		1 min	66.669	1 min	62.289
		5 min	59.772	5 min	55.836
	7%	5 s	73.418	5 s	76.177
		1 min	69.557	1 min	75.676
		5 min	62.947	5 min	65.665
	9%	5 s	77.339	5 s	81.134
		1 min	75.323	1 min	80.402
		5 min	68.159	5 min	75.112

In the measurements made by dropping pure water on the Scotch pine samples, the highest contact angle value was measured in the sample impregnated with 9% NaHCO₃. As the solution ratio increased, the surface contact angle increased accordingly. In the measurements made by dropping saline solution, it was observed that the contact angles were higher than pure water.

Table 2. Contact Angle Values of Spruce Samples

	Pure Water			Salt Water	
	NaHCO ₃	Duration	Angle (°)	Duration	Angle (°)
Spruce	K	5 s	72.981	5 s	78.170
		1 min	71.885	1 min	72.610
		5 min	65.265	5 min	63.330
	5%	5 s	64.313	5 s	77.658
		1 min	62.643	1 min	75.286
		5 min	57.265	5 min	70.030
	7%	5 s	66.736	5 s	80.336
		1 min	64.497	1 min	78.633
		5 min	61.189	5 min	52.146
	9%	5 s	79.181	5 s	81.283
		1 min	74.150	1 min	69.432
		5 min	68.860	5 min	65.956

Contact angles, contact surface widths, and dropper heights measured after pure water and saltwater were dropped on spruce samples are given in Table 2. In the measurements made by dropping pure water on spruce samples, the highest contact angle value was measured in the sample impregnated with 9% NaHCO₃. As the solution ratio increased, the surface contact angle of the spruce specimens increased accordingly. In the measurements made by dropping saline solution on the samples, it was measured that the contact angles were higher than pure water.

Table 3 shows the surface roughness averages of spruce and yellow pine samples. Surface roughness was measured at the highest values in control samples in both wood species.

Table 3. Average Surface Roughness (R_a) of Scotch Pine and Spruce wood

	Scotch Pine	Spruce
Control	2.024 μm	1.740 μm
5% NaHCO ₃	1.106 μm	1.324 μm
7% NaHCO ₃	0.920 μm	1.193 μm
9% NaHCO ₃	0.630 μm	0.893 μm

The surface roughness values of the Scotch pine control samples were found to be higher than the corresponding values for spruce samples. It was determined that the surface roughness values decreased as the impregnation solution ratio increased in the samples impregnated with NaHCO₃. Although the surface roughness of Scotch pine control samples was higher than spruce, lower values were observed after impregnation.

CONCLUSIONS

In this study, contact angle measurements made by dropping pure water and saline water (containing NaHCO₃) on pine and spruce specimens were investigated. It was observed that when saline water was used on pine specimens, the contact angles were generally higher than pure water specimens. In particular, the contact angle reached the highest level in pine specimens impregnated with 9% NaHCO₃, indicating that the concentration of the impregnating solution increases the surface hydrophobicity. Furthermore, the contact surface was found to expand and the drop height decreased with time, indicating that the interaction of the impregnated samples with water changes with time.

Similar trends were observed on spruce samples. As the concentration of the impregnating solution increased, the contact angle increased and the spreading characteristics of the water drops on the surface changed. In particular, the contact angle was highest in spruce specimens impregnated with 9% NaHCO₃.

The results of the surface roughness tests showed that among the control samples taken from Scots pine (*Pinus sylvestris* L.) and spruce (*Picea orientalis* L.) wood, the measured roughness values of Scots pine samples were higher than those of spruce wood. However, when the samples were treated with NaHCO₃, the roughness value was higher in spruce samples as the solution ratio increased. In the Scotch pine samples, the surface roughness value was lower than spruce wood after impregnation. The reason why pine wood is better impregnated than spruce is that pine generally has lower density and larger

grain structures. These structural features make it easier for impregnation solutions to penetrate deeper into the pine tree, thus providing more effective protection.

In the surface roughness analyses, a general decrease in roughness values was observed in the impregnated samples compared to the control samples. This indicates that impregnation tends to optimize the surface morphology, and the concentration of the impregnating solution increases this effect.

When the wetting behaviour of the samples was examined, the contact angle values of the Scotch pine samples were higher than spruce. When pure water or saline solution was dropped on the samples, a decrease in the contact angle value, a decrease in droplet height, and an increase in droplet width was observed due to the increase in waiting times. This indicates that the droplet spreads to the surface over time and the effect of evaporation. It was determined that the contact angle value increased as the percentage ratio of NaHCO_3 solution applied to Scotch pine and spruce samples increased. In the measurements made with salt water, the contact angle value was higher than pure water.

In summary, it was observed that the impregnation properties of pine and spruce wood can vary significantly, depending on the type and concentration of the impregnating solution used. This study provides important information on how impregnation treatments can be optimised to improve the water resistance properties of wood materials.

Through determining the wettability behaviour of such materials with contact angle values, their applications can be better determined. This can ensure the long-lasting use of the materials.

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