

# Effect of Silane-siloxane Based Water-repellent Impregnant on the Dimensional Stability of Some Wood Species

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A silane-siloxane based water-repellent substance (S-WR) was used as an impregnation material for Scots pine (*Pinus sylvestris* L.), black pine (*Pinus nigra* Arnold), sessile oak (*Quercus petraea* Liebl.), and cedar (*Cedrus libani* A. Rich). In the impregnation process, the immersion method was carried out for various periods (30 min, 3 h, and 24 h). Following impregnation, physical properties of the wood (retention, contraction, expansion, water intake rate, specific gravity, etc.) were determined. The retention was highest in sessile oak wood (0.96%) and lowest in black pine wood (0.24%). The highest specific gravity value was found in stemless oak (0.86 g/cm<sup>3</sup>) impregnated for 24 h, and the lowest air-dry specific gravity value was found in Scots pine (0.40 g/cm<sup>3</sup>) impregnated for 30 minutes. Absolutely dry (0% humidity) specific gravity was highest in sessile oak for 24 h (0.83 g/cm<sup>3</sup>). Shrinkage was highest in the sessile oak control sample (13.5%). The expansion amount was highest in the cedar wood control sample (17.5%) and lowest in sessile oak wood (1.66%). The highest water uptake rate (SAO) was determined in Scots pine wood at 96 h (105%), and the lowest in sessile oak wood in 24 h (8.80%). In summary, silane-siloxane based water-repellent impregnation material was found to be effective in providing dimensional stability in wood.

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## INTRODUCTION

Wood is an indispensable material that human beings have used since early times due to its appearance, aesthetics, high availability, and ease of giving the desired form. Humankind has also used wood material while meeting one of their essential needs for shelter. There have been advancements of technology related to the strength properties of wood material, its protection, and various processing methods (Çağlayan 2020). There is a strong motivation to develop organic preservatives and methods that are suitable for the ecological structure, protective, reparative, strengthening and reusable at the same time (Bayraktar and Kesik 2022).

There are many ways to create a hydrophobic surface. In general, this can be done by changing the surface properties with a reactive chemical that reduces surface energy. According to the principles of interface coupling, the hydrophilic entities can be changed to hydrophobic ones by reducing the amount of hydroxyl groups on the surface. Those –



considerably lower than predicted theoretically, showing that little cell wall penetration had occurred. It is stated that this situation occurs with the formation of a silane bridge between wood components and polymeric binders. Özlusoğlu *et al.* (2018) investigated the surface properties of fiberboard by adding two types of silane (2-aminoethyl-3-aminopropyltrimethoxysilane and 3-aminopropyltriethoxysilane) at addition levels of 1.5%, 2.5%, and 3.5% as additional additives. According to the results obtained, it was determined that as the additive concentration increased, the color darkening and surface roughness of the plates increased and no resistance was provided against fungal rot.

By using silane-siloxane-based water repellent (S-WR), it is aimed to increase the service life of wood material, to gain economic gains, and to utilize an alternative protective material in our century where human/environmental health comes to the fore. In case of an increase in the physical properties of wood with the use of silane-siloxane products, it can be considered as an alternative protective chemical that will create areas where wood can be protected more effectively against factors such as moisture and decay.

## EXPERIMENTAL

### Materials

Scots pine (*Pinus sylvestris* L.), black pine (*Pinus nigra* Arnold), sessile oak (*Quercus petraea* Liebl.), and cedar (*Cedrus libani* A. Rich) woods were used. "Teknosil" brand silane-siloxane based water-repellent material of "Tekno Construction Chemicals" company, which is a commercial product, was used as impregnation material. The product code is CE-1504-2.

### Preparation of Experimental Samples

The test samples were prepared according to the principles of TS ISO 3129 (2021). Care has been taken to avoid defects cracks, knots, arcs, mold, rot, *etc.*, in all samples. Attention was also paid to the fact that the annual ring structure of the samples obtained from the sapwood part was radial to the surface (ISO 3129 2019). Samples with air-dry moisture were cut from the sapwood parts in 20 × 20 × 30 mm (tangential direction × radial direction × longitudinal) dimensions according to TS 345 (2012) and with five repetitions ( $n = 5$ ) for each test variable. Afterward, the samples were kept in a drying oven (oven) at  $103 \pm 2$  °C until they reached a constant weight, and then the exact dry weight (g) and dimensions (mm) of the samples were determined with an accuracy of 0.01.

### Impregnation

Teknosil brand silane-siloxane based water repellent (S-WR) was used as impregnation material. In the impregnation process, absolutely air-dry wood samples were impregnated with the immersion method (short term: 30 min, medium term: 3 h, long term: 24 h). The pH value was measured by cleaning the tip of the pH probe and immersing it in homogenized impregnation liquid. At the end of the impregnation process, the excess of the impregnation solution on the surface of the samples was removed with a paper towel and the test samples were immediately weighed in the wet state. Afterwards, the samples were kept in an oven at 50 °C until they reached a constant weight, and the full dry weight (g) and dimensions (mm) of the samples were re-determined with an accuracy of 0.01. The furnace temperature was kept at 50 °C to prevent the loss of S-WR in the impregnated samples.

### Specific Gravity (Absolutely Dry) and Retention

Determination of air dry and full dry specific gravity was made according to TS ISO 13061-1 (2012) and TS ISO 13061-2 (2021) standards. Retention amount of S-WR impregnation material on wood (remaining solid matter (%) amount based on dry wood weight) Eq. 1 (Yalınkılıç 1993),

$$\text{Retention (\%)} = (M_{0es} - M_{0e\ddot{o}}) / M_{0e\ddot{o}} \times 100 \quad (1)$$

where  $M_{0e\ddot{o}}$  and  $M_{0es}$  are the absolutely dry sample weight before and after impregnation (g), respectively.

### Dimensional Stability (Shrinking/Swelling) and Water Uptake Rate (SAO)

The samples' water uptake and dimensional stability (shrinking/swelling) tests were carried out according to the ISO 13061-15 (2017) standard. The samples, whose exact dry weight and dimensions were determined before, were kept in distilled water for 6, 24, 48, 72, and 96 h under laboratory conditions (Bozkurt *et al.* 1993; Akyürekli 2003). At the end of each holding period, the samples were removed from the water, dried with paper towels, and weighed immediately. The sizes of the samples, which were kept in water for 6, 24, 48, 72 and 96 hours, were determined. The water uptake rates (SAO) and dimensional stability (swelling/ shrinking) values (BD) of the samples in the tangential/radial direction were determined as follows,

$$\text{SAO (\%)} = (M_2 - M_1) / M_1 \times 100 \quad (2)$$

$$\text{BD (\%)} = (L_2 - L_1) / L_1 \times 100 \quad (3)$$

where  $M_2$  is the moist sample weight after soaking (g),  $M_1$  is the absolutely dry sample weight before soaking (g),  $L_2$  is the moist sample width-thickness after soaking (mm), and  $L_1$  is the absolutely dry sample width-thickness before soaking (mm).

### Statistical Analysis

Percent ratio and standard deviation calculations were made to determine the effects of variables such as wood type, water-repellent substance, soaking time, retention, specific gravity, and shrinking/swelling some physical properties of wood samples. In addition, the samples' average values of the physical properties were compared with Duncan tests at the wood type, water-repellent substance, soaking time, retention, specific gravity, and shrinking/swelling levels.

## RESULTS AND DISCUSSION

### Solution Properties and % Retention Amount

The relevant data before and after the impregnation are given in Table 1. No changes were observed in pH and density measurements before and after impregnation. The pH value of 7.4 indicates a near-neutral condition. A sufficiently low pH (acidic) can cause hydrolysis of wood. The pH stability showed that the S-WR solution did not cause significant chemical reactions/changes in the natural acidity of the wood. The temperature at which the impregnation was carried out was chosen as 22 °C. Higher temperatures generally speed up chemical reactions, including hydrolysis, which involves breaking down chemical bonds by water molecules. In the absence of any changes observed in pH

and density measurements before and after impregnation, it can be said that hydrolysis did not occur significantly, which indicates that the S-WR diffusion process does not damage the wood.

**Table 1.** Solution Properties

Solution (%)	Impregnation Material	Solvent Substance	Temp (°C)	pH		Density (g/mL)	
				BI	AI	BI	AI
100	S-WR	-	22	7.4	7.4	0.995	0.995

BI: Before impregnation, AI: After impregnation

Duncan test results for % retention amount are given in Table 2, and their change graph is given in Fig. 2. According to wood type and dipping parameters, the highest retention value was observed in oak wood at 30 minutes immersion time (0.96%), and the lowest retention value in black pine at 0.5, 3, and 24 h dipping time (0.29%). Change in retention amounts may be caused by the wood type, the anatomical structure of the wood, and the impregnation material. The retention amount increased as the immersion time increased in Scots pine wood. In black pine, the amount of retention remained constant during different immersion times. It provided retention of 0.29% regardless of whether the immersion lasted 30 min, 3 h, or 24 h. In sessile oak wood, the retention amount decreased as the immersion time increased. In this case, it was determined that sessile oak wood absorbed a significant amount of solution during the shorter immersion period in the first initial period, and there was no increase in retention as time increases. Cedar wood showed a different situation compared to other wood species and showed differences according to the immersion time.

**Table 2.** % Retention Amount

Impregnation Material	Wood Type	Immersion Duration (short-medium-long)	Retention (%)		
			mean	Stdev	HG
S-WR	Scots pine	30 min	0.42	1.68	D
		3 h	0.45	2.78	D
		24 h	0.61	3.67	C
	Black pine	30 min	0.29	2.91	E
		3 h	0.29	8.12	E
		24 h	0.29	5.46	E
	Sessile oak	30 min	<b>0.96</b>	6.47	A
		3 h	0.73	3.18	B
		24 h	0.64	1.09	C
	Cedar	30 min	0.72	2.73	B
		3 h	0.92	4.14	A
		24 h	0.43	1.66	D

HG: Homogeneous Groups ( $p < 0.05$  probability of mistake), st dev: standard deviation

The results obtained were also compatible with the literature information. Toker (2008) found the highest retention rate in Eastern beech wood with a 6% solution concentration impregnated with sodium perborate and the lowest retention rate in red pine wood with a 1% solution concentration impregnated with borax. Sarıca (2006) found the highest retention rate in eastern beech ( $29.6 \text{ kg/m}^3$ ) impregnated with borax and the lowest in sessile oak ( $3.1 \text{ kg/m}^3$ ) impregnated with boric acid. Atilgan and Peker (2012)

determined that beech wood was impregnated with 9% concentration (cement+borax), and the highest retention amount was  $42.4 \text{ kg/m}^3$ , while the lowest value was  $5.2 \text{ kg/m}^3$  in Scots pine (1% ammonium tetraflu borate).

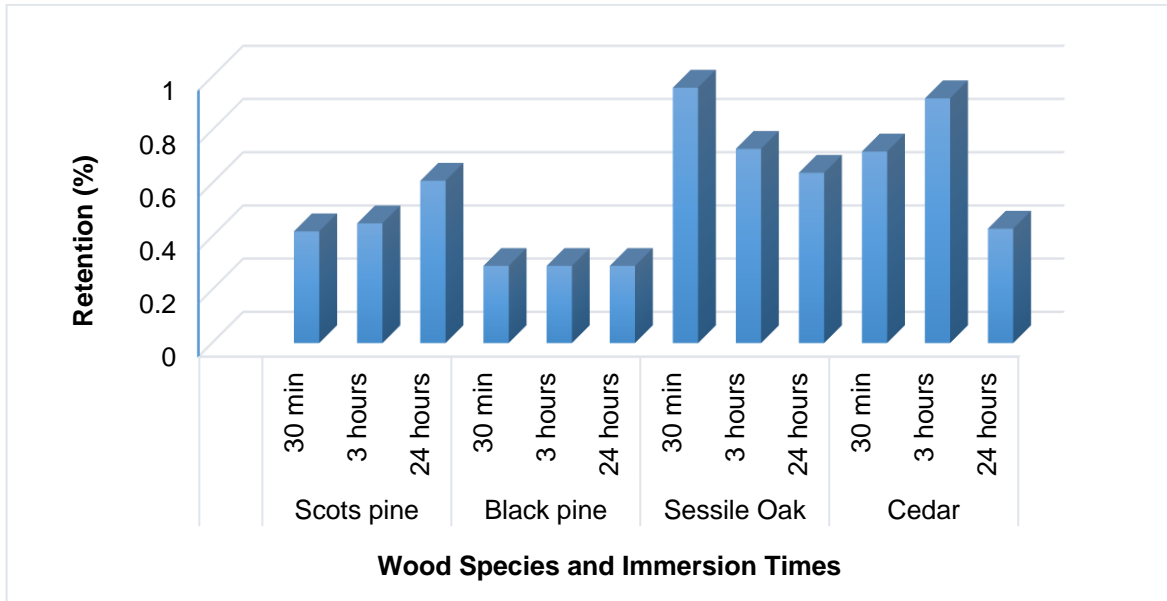


Fig. 2. % Retention values

### Specific Gravity Change ( $\text{g/cm}^3$ )

The specific gravity change results are given in Table 3, and the related change graph is given in Fig. 3. The highest value in air-dry specific gravity change was determined in sessile oak at 24-h dipping time ( $0.86 \text{ g/cm}^3$ ), and the lowest air-dry specific gravity change was determined in Scots pine at 30 min dipping time ( $0.40 \text{ g/cm}^3$ ).

Table 3. Air Dry and Absolutely Dry Specific Gravity ( $\text{g/cm}^3$ )

Wood Type	Immersion Duration (short-medium-long)	Air Dry Specific Gravity		Absolutely Dry Specific Gravity	
		Mean	HG	Mean	HG
Scots pine	Control (unimpregnated)	0.39	I	0.37	H
	30 min	0.40	H	0.37	H
	3 h	0.42	G	0.41	G
	24 h	0.44	F	0.40	G
Black pine	Control (unimpregnated)	0.44	F	0.43	F
	30 min	0.45	F	0.44	F
	3 h	0.43	G	0.41	G
	24 h	0.51	E	0.49	E
Sessile oak	Control (unimpregnated)	0.83	B	0.80	B
	30 min	0.85	A	0.82	A
	3 h	0.82	B	0.80	B
	24 h	<b>0.86</b>	A	<b>0.83</b>	A
Cedar	Control (unimpregnated)	0.56	D	0.55	C
	30 min	0.54	D	0.51	D
	3 h	0.59	C	0.56	C
	24 h	0.56	D	0.55	C

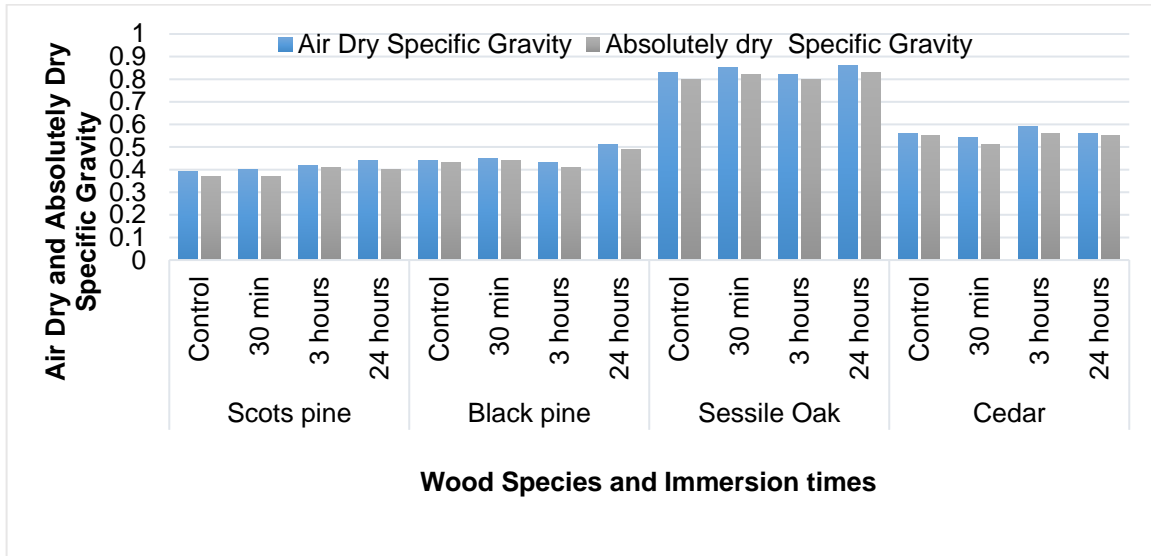


Fig. 3. Air and absolutely dry specific gravity change ( $\text{g}/\text{cm}^3$ )

The highest dry specific gravity change was detected in sessile oak wood in the 24 h ( $0.83 \text{ g}/\text{cm}^3$ ), and the lowest arid specific gravity change in Scots pine wood in the 30 min ( $0.40 \text{ g}/\text{cm}^3$ ). The specific gravity amounts after impregnation slightly increased for Scots pine compared to the control sample at all immersion times. Black pine showed changes in specific gravity amounts after being impregnated with S-WR. It has been observed that black pine impregnated with S-WR has a significant effect on the absolutely dry specific gravity value. In sessile oak, the specific gravity values showed minimum changes. There were slight changes in the amount of specific gravity after the impregnation of cedar wood. This may be caused by the anatomical structure of the wood, the wood type, the immersion time, and the impregnation method.

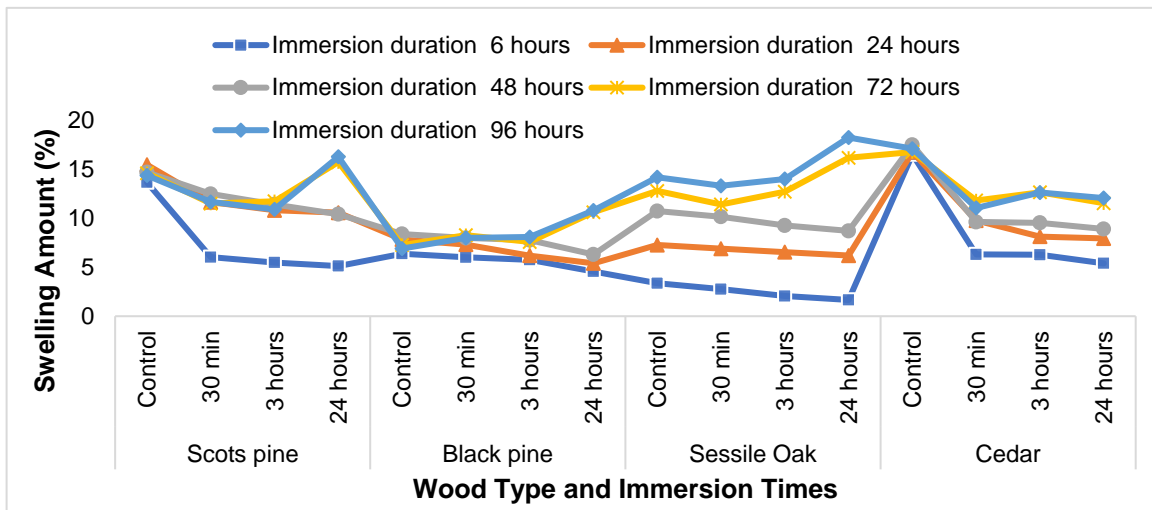
These results were similar to others in the literature. Rosenthal and Bues (2010) impregnated the ecologically accepted silicone into Scots pine wood and used it to provide dimensional stability and fire resistance. Kılıç (2012) impregnated beech and spruce wood with Dow Corning (R) Z-6341 Silane in his study, the specific gravity values did not change significantly compared to the control groups, it was determined as  $0.44 \text{ g}/\text{cm}^3$  in spruce control samples and  $0.43$  to  $0.56 \text{ g}/\text{cm}^3$  in impregnated. It is thought that this situation may be due to a possible polymerization on the surface of the test samples after impregnation.

### Swelling Change

The percent expansion variation results according to wood species and immersion times are given in Table 4, and the related variation graph is given in Fig. 4. While the amount of swelling tends to decrease as the immersion time increases in Scots pine wood, shorter immersion times positively affected the swelling. Black pine wood generally showed relatively low amounts of swelling. Sessile oak wood showed moderate expansion amounts, especially at longer immersion times. Generally, relatively high expansion (swelling) amounts were detected in cedar wood. Overall, the data show that S-WR impregnation had varying effects on different wood species swelling behavior. Scots pine and sessile oak woods exhibited notable decreases in expansion, showing that S-WR can effectively reduce swelling.

**Table 4.** Amount of Swelling (%)

Impregnation Material	Wood Type	Immersion Duration (short-medium-long)	Expansion Change (%)				
			6 h	24 h	48 h	72 h	96 h
S-WR	Scots pine	Control	13.66	15.46	14.69	14.60	14.37
		30 min	6.03	11.65	12.48	11.50	11.66
		3 h	5.48	10.81	11.41	11.74	10.90
		24 h	5.12	10.57	10.41	15.73	16.28
	Black pine	Control	6.38	7.83	8.39	7.26	6.91
		30 min	6.01	7.31	7.99	8.27	8.00
		3 h	5.76	6.18	7.77	7.58	8.086
		24 h	4.57	5.40	6.30	10.62	10.80
	Sessile oak	Control	3.37	7.27	10.73	12.79	14.19
		30 min	2.76	6.89	10.15	11.41	13.31
		3 h	2.06	6.54	9.26	12.71	14.01
		24 h	1.66	6.19	8.71	16.17	18.24
	Cedar	Control	16.52	16.75	17.49	16.76	17.10
		30 min	6.31	9.77	9.62	11.82	11.05
		3 h	6.27	8.13	9.53	12.65	12.63
		24 h	5.40	7.94	8.90	11.53	12.06



**Fig. 4.** Swelling amount (%) according to wood species and immersion times

The results obtained were compatible with the literature. Bak *et al.* (2023) impregnated European beech (*Fagus sylvatica* L.) and Scots pine (*Pinus sylvestris* L.) wood species with fluorinated silica nanoparticles. They investigated the wood's swelling, water uptake, and equilibrium moisture content. They reported that the presence of hydrophobic silica nanoparticles improves dimensional stability by permanently increasing the wood's hydrophobicity and the low weight percentage gain. Kim *et al.* (2010) investigated the effects of trialkoxysilane treatments on the fundamental properties, mechanical properties, and water uptake level of wood fiber-reinforced polipropilen composites by chemically modifying wood flour (WF), 3-aminopropyltriethoxysilane, 3-methacryloxypropyltri-methoxysilane, and vinyltrimethoxysilane. It was reported that silane treatments significantly improved the tensile, bending, impact and water absorption strengths of wood composites (Kim *et al.* 2010).

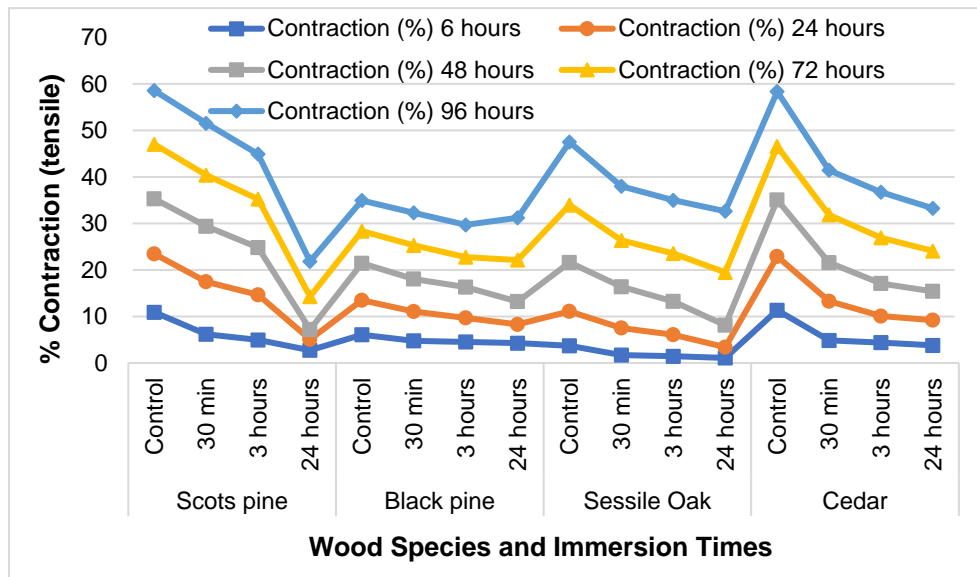


**Contraction (Shrinkage) Change**

The shrinkage change according to the wood species and dipping times are given in Table 5, and the change graph is given in Fig. 5. According to wood type and dipping times, the highest shrinkage was observed in the unimpregnated oak wood (control) sample (13.5%), and the lowest in the stemless oak wood impregnated samples by dipping for 24 hours (1.1%). S-WR material significantly affected the shrinkage of all wood species compared to the control samples group.

**Table 5.** Amount of Shrinkage (%)

Impregnation Material	Wood Type	Immersion Duration (short-medium-long)	Contraction (%)				
			6 h	24 h	48 h	72 h	96 h
S-WR	Scots pine	Control	10.91	12.55	11.837	11.74	11.54
		30 min	6.18	11.29	11.918	11.00	11.16
		3 h	4.96	9.70	10.144	10.44	9.67
		24 h	2.74	2.33	2.116	7.09	7.55
	Black pine	Control	6.08	7.41	7.943	6.92	6.58
		30 min	4.76	6.30	6.989	7.24	7.00
		3 h	4.53	5.15	6.637	6.45	6.92
		24 h	4.26	4.04	4.891	8.95	9.09
	Sessile oak	Control	3.70	7.39	10.52	12.35	13.54
		30 min	1.70	5.81	8.86	9.98	11.66
		3 h	1.45	4.62	7.16	10.33	11.44
		24 h	1.08	2.32	4.70	11.38	13.15
	Cedar	Control	11.34	11.55	12.17	11.51	11.80
		30 min	4.85	8.43	8.29	10.28	9.59
		3 h	4.38	5.70	7.00	9.845	9.809
		24 h	3.79	5.41	6.20	8.685	9.172



**Fig. 5.** Shrinkage amount (%) according to wood type and dipping times

Hochmańska *et al.* (2014), modified wood samples were impregnated with silane, and its effect on dimensional stability and decay properties was investigated. They reported that Corsican pine sapwood impregnated with two organo-alkoxysilanes showed a significant shrinking-reducing effect.

Mai *et al.* (2005), in a study conducted by tiny molecule silicon (polyallylsiloxane) material, was impregnated with Scot's pine test samples and stated that while dimensional stability increased, there was a decrease in water uptake rate.

### Percent Water Uptake (SAO)

The results of the water uptake rate (%) according to the wood species and immersion times are given in Table 6, and the change graph for them is given in Fig. 6. When evaluated according to wood type and dipping times, the highest water uptake rate was determined in Scots pine wood at 96 h (105%) and the lowest in S-WR impregnated sessile oak wood at 24 h (8.8%). All S-WR impregnated wood groups significantly affect the wood material in terms of water repellency.

Donath *et al.* (2006) treated various wood species with three types of silane solutions. They reported that while the fluoro-alkyl functional oligomeric silane was influential in the first period, the reduction in water intake was significantly reduced after a longer immersion time (24 h). They reported that the obstruction of the main penetration pathways, such as cells and ray tracheids, causes it.

Woźniak *et al.* (2018) vacuum-impregnated Scots pine wood with a propolis extract and two propolis-silane formulations. They reported that adding silane compounds to propolis extract reduced the water uptake of the treated wood and increased the contact angle value.

Szubert *et al.* (2019), the effectiveness of propyltriethoxysilane-based protective coatings in protecting the wood surface from the effects of water has been tested. A coating is produced that permanently increases the hydrophobicity of the wood due to the chemical bonds formed between the wood surface and the silane, and the fluorinated chains attached to the silicon atoms prevent the ingress of water and prevent the water from entering the wood surface. It has been reported to form an effective barrier limiting its effects.

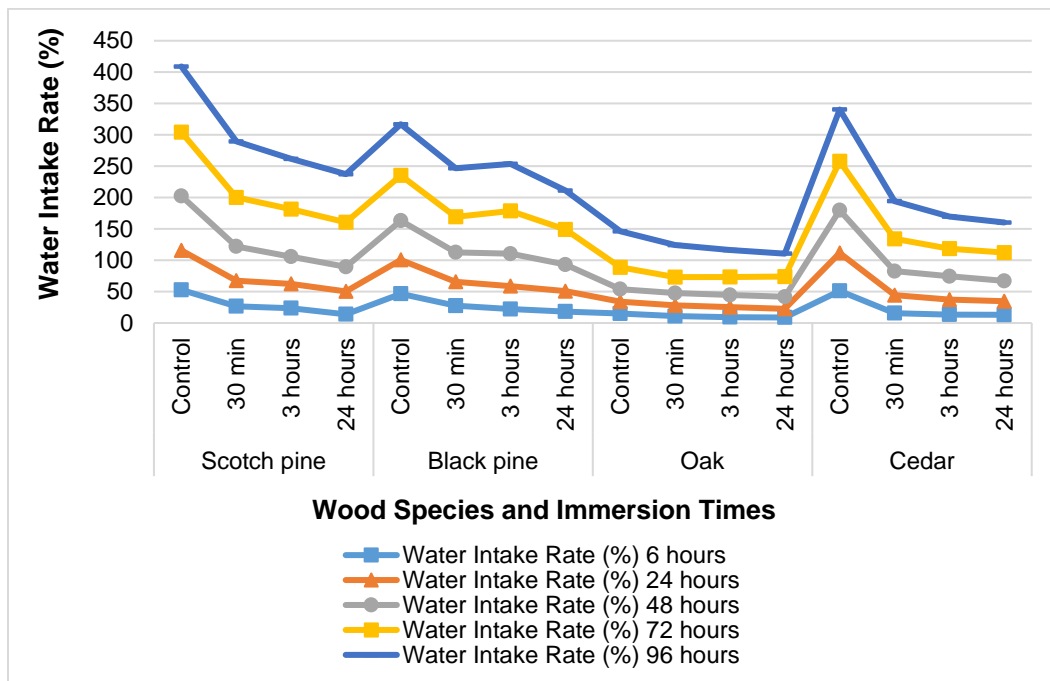
Broda *et al.* (2019) used in the archaeological artifact and subjected degraded sessile oak and intact oak wood to methyltrimethoxysilane pressure impregnation, where the silane modification increased the equilibrium moisture content of archaeological sessile oak samples from 23.7% to 19.4% for heartwood and 19.4% for sapwood, respectively. They reported that it decreased from 23.3 to 10.0%. When the results of surface area and porosity measurements were examined, it was reported that methyltrimethoxysilane helped preserve the wood's microstructure by deposition on the cell wall.

Lukowsky *et al.* (1997) reported that the water-repellent effectiveness of scots pine and beech specimens impregnated with silicone decreased over time.

In many countries, the central theme of general-purpose use is to protect wood textures with more natural materials (water absorption, rot, fungus, *etc.*), the high level of human/environmental health exhibitions, the long-lasting effects of wooden items, the reuse of objects in their historical storage (restoration *etc.*). Wood materials impregnated with non-toxic substances must be strongly assimilated into the ecological cycle. For this purpose, S-WR was used in this study. It can be used as a water-repellent preservative in wood restoration, wooden structures, wooden outdoor furniture. In this context, it is understood from the literature that the S-WR material has been used in many areas. These areas include natural stone, composite boards, concrete, and plastic cover materials.

**Table 6.** Water Intake vs. Time (%)

Impregnation Material	Wood Type	Immersion Duration (short-medium-long)	Water Intake Rate (%)				
			6 h	24 h	48 h	72 h	96 h
S-WR	Scots pine	Control	52.81	63.01	86.59	101.79	104.77
		30 min	26.65	40.80	54.63	78.16	89.25
		3 h	23.63	38.82	43.28	75.79	80.24
		24 h	13.93	36.52	39.15	71.00	76.65
	Black pine	Control	46.64	54.04	62.46	72.17	81.53
		30 min	27.54	38.16	46.91	56.66	77.45
		3 h	22.17	36.72	51.59	68.20	75.07
		24 h	18.22	32.52	42.38	56.01	62.10
	Sessile oak	Control	15.10	18.97	19.94	34.76	57.38
		30 min	10.99	17.15	19.66	25.39	51.11
		3 h	9.30	16.04	19.37	28.61	43.04
		24 h	8.80	13.879	19.19	32.12	36.62
	Cedar	Control	51.18	60.454	68.10	78.11	82.61
		30 min	15.67	28.728	38.28	51.53	60.03
		3 h	13.42	23.920	37.20	44.03	51.045
		24 h	13.10	21.613	32.44	45.15	47.874



**Fig. 6.** Water intake amounts (%) by wood species and immersion times

**CONCLUSIONS**

1. In this study, the use of a silane-siloxane based water-repellent substance (S-WR) as a water repellent in wood material was evaluated by various experiments. According to the obtained findings, it was determined that the treatment contributed to dimensional stability.
2. When evaluated in general, impregnation systems applied by dipping method provided

a viable retention of the S-WR.

3. The dimensional stability (shrinkage, swelling, dewatering) of the S-WR impregnated wood samples provided significant water repellency compared to the control samples (non-impregnated).
4. No significant performance was determined in the specific gravity (air dry/absolute dry) of the S-WR impregnated test samples.
5. The findings obtained with the water repellent property of S-WR in this study show that it can be used as an effective protector in outdoor furniture (camellia, pergola, wooden dustbin, benches, *etc.*), architectural structures, and in protecting historical wooden works against moisture.

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