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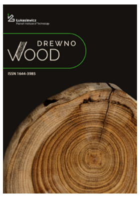

Effect of Liquid Nitrogen Application on Retention and Mechanical Properties of Oak Wood

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Wood is a natural material widely utilized in the forest industry and furniture sector, valued for its aesthetics and durability. Various methods have been developed to extend its service life and protect it against deterioration. In this study, liquid nitrogen treatment was applied to increase retention levels, and the effects of retention and impregnation agents on the mechanical properties of oak wood were examined. Oak (*Quercus robur*), a species known for its low permeability and difficulty in impregnation, was selected as a test sample. The samples were exposed to liquid nitrogen for 15 minutes, 90 minutes, and 6 hours, and subsequently impregnated with Tanalith-E, Imersol Aqua, Diammonium Sulphate, and Borax using brush application, short- and long-term immersion, and pressure methods. Retention levels were determined, and mechanical performance was assessed through compression parallel to the fibers and dynamic bending (impact) resistance tests. The maximum retention value was found to be 4.212 kg/m^3 in the long-term immersion method with Borax, and a 519% increase in retention rate was obtained. The results showed that liquid nitrogen application increased retention in oak wood, and due to this increase, changes in compressive strength and impact strength were observed parallel to the fibers.

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Introduction

Wood is a renewable and easily processable material that plays a vital role in the forest industry due to its favorable properties. However, the increasing consumption of wood, coupled with declining forest areas, necessitates the application of treatments such as drying, impregnation, and surface finishing to enhance its durability and extend its service life, as it is susceptible to environmental, chemical, and biological degradation (Higley & King, 1990; Özcan, 2011).

Wood preservation dates to the earliest times of human history. Historically, people protected wood from external influences by burning its surface or applying animal and vegetable oils, but with industrial and chemical advancements, these methods have evolved into the sophisticated preservation and impregnation techniques used today (Richardson, 2020; Lebow and Kirker, 2023). Among these methods, impregnation is widely employed to protect wood from environmental changes, fungi, insects, and marine organisms (Örs, Atar, & Demirci, 2005). Studies also highlight its role in

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enhancing fire resistance (Kesik et al., 2015; Özcan et al., 2016; Tomak et al., 2013). Impregnation significantly enhances wood's lifespan and resistance to environmental effects, thereby increasing its economic and aesthetic value (Kartal, 1998; Hill and Altgen, 2019; Sumarni and Nawawi, 2022).

Impregnation materials used in industry are expected to combine many features, such as being economical and easy to apply (Huo et al., 2016; Zhang et al., 2020). For successful impregnation, the impregnation material must adhere as deeply as possible within the material and penetrate the interior. Therefore, compatibility between the impregnation material and the tree's anatomy is sought (Rowell, 2021). Different anatomical structures of tree species, drying methods, moisture content, and other characteristics directly affect impregnation. Therefore, different problems can be encountered in the impregnation process performed on different tree species (Usta and Hale, 2022).

Liquid nitrogen pretreatment, especially for wood materials that are difficult to impregnate, has been among the preferred methods in recent years. With this method, it is observed that the retention rate increases due to microcracks formed in the wood structure. (Wang, Lin, & Xing, 2021). For the industrial use of liquid nitrogen application, Kurt et al. (2014) developed a system incorporating a liquid nitrogen tank into the impregnation process. They demonstrated that this system provided better adhesion in wood that was difficult to impregnate. Similarly, Yorur et al. (2017) reported that liquid nitrogen pretreatment had a positive effect on adhesion when applied after impregnation with Firetex.

This study selected oak wood (Quercus robur), a species widely used in the industry but known for its low permeability. Liquid nitrogen pretreatment was applied for different durations (15 minutes, 90 minutes, and 6 hours) before impregnation with Tanalith-E, Imersol Aqua, Diammonium Sulphate, and Borax. Impregnation was conducted using brush application, short- and long-term immersion, and pressure methods. The effects of liquid nitrogen pretreatment on retention, compression strength parallel to the fibers, and impact resistance were subsequently evaluated.

Materials and methods

1. Wood

In this study, the pedunculate oak (Quercus robur) species was selected as the test material because it is a material that is easily available and has widespread industrial use in the field of wood structure and furniture decoration in Europe and Turkey (Eaton & Hale, 2021; Akkemik & Yaman, 2024). The wood samples were obtained from the market using a random selection method. After air-drying under natural conditions, the materials were further dried until they reached equilibrium moisture content. Test specimens were then prepared from these materials according to the requirements of the subsequent experiments. The untreated samples served as the control group.

2. Impregnating agents and impregnation method

Tanalith-E, Imersol Aqua, Diammonium Sulphate and Borax were used as impregnation agents, applied via brush, short-term immersion, long-term immersion, and pressure methods

3. Liquid nitrogen

Liquid nitrogen, which makes up 78% of the atmosphere in gaseous form, is widely used industrially due to its cryogenic properties. It remains liquid below -195.79°C (Selçuk University Advanced Technology and Application Center, 2014).

4. Experiment preparation of samples

Test specimens were prepared from pedunculate oak according to TS 1476 standards. Defect-free, straight-grained, healthy wood without knots, resin pockets, or growth irregularities was selected. The samples were first treated with liquid nitrogen and subsequently conditioned in a climate chamber. After conditioning, the impregnation treatments were applied. Oven-dry density and moisture were determined per TS 2471 and TS 2472. Samples were conditioned at 20 ± 2°C and 65 ± 3% relative humidity until constant weight, then weighed with 0.001 g precision.

5. Liquid nitrogen processes

Samples were prepared in four groups: control, 15 minutes, 90 minutes, and 6 hours of liquid nitrogen application. The prepared samples were kept in liquid nitrogen for the specified periods. After the liquid nitrogen application, they were reweighed on an analytical balance with a sensitivity of 0.001 g and prepared for impregnation. The liquid nitrogen-treated samples were impregnated using brush application, short-term immersion (15 minutes), long-term immersion (90 minutes), and pressure (10-12 bar, ASTM D 1413-07, Full-Cell (Bethell). Tanalith-E, Imersol Aqua, Diammonium Sulphate, and Borax were used as impregnation agents for each group.

Table 1. Physical characteristics

The substance state	Gas
Intensity	0.001251 g/cm³
Liquid in the state density	0.808 g/cm³
Melting point	-210.00 °C
Boiling point	-195.79 °C
Melting heat	0.720 kJ/mol
Evaporation heat	5.57 kJ/mol
Heat capacity	29,124 (25 (°C) J/(mol K)

6. Determination of moisture content

The moisture content before and after treatment of the woods used in the preparation of the test samples was determined accordingly to TS 2471. Moisture content (r):

$$r = \frac{Mr - Mo}{Mo} \times 100 \tag{1}$$

where:
Mr: moist weight (g)
Mo: oven-dry weight (g)

7. Determination of density

The densities before and after treatment of the woods used in preparing the test samples were determined in accordance with TS 2472. Accordingly, density δ (g/cm³) was calculated with the following equation:

$$\delta = \frac{M}{V} \tag{2}$$

where:
M: oven-dry weight (g); V: volume (cm³)

8. Retention amount and retention rate

Retention amount (R, kg/m³) and retention percentage (R, %) for impregnation materials from the water-soluble salts group are calculated from the equations.

$$\mathrm{R} = \frac{\mathrm{G} \times \mathrm{C}}{\mathrm{V}} \times 10\ (\mathrm{kg/m^3}) \tag{3}$$

$$\mathrm{R}(\%) = \frac{\mathrm{m_{post}} - \mathrm{m_{pre}}}{\mathrm{m_{pre}}} \times 100 \tag{4}$$

where:
G: Amount of impregnation solution absorbed by the sample (g), $G = (T_2 - T_1)$, T_2: post-impregnation weight (g), T_1: pre-impregnation weight (g)
C: Concentration of the impregnant in the solution (%)
V: Sample volume (cm³)
m_{post}: Post-impregnation full dry weight of the sample (g)
m_{pre}: Pre-impregnation full dry weight of the sample (g)

Mechanical features

1. Strength parallel to fibers

The compressive strength parallel to the fibers was determined in accordance with TS 2595. The acclimatized specimens were first prepared with cross-sectional dimensions of 2 × 2 cm and a length of 3 cm, following the principles of TS 2474. After being removed from the acclimatization cabinet, the cross-sectional dimensions of the test specimens were measured with a digital calliper (precision ±0.01 mm). Subsequently, the specimens were placed in a universal testing machine with the fibers direction aligned parallel to the loading direction. The test was conducted at a loading speed of 6 mm/min to ensure crushing occurred within 30–60 seconds after loading (TS 2595, 1977) and calculated with the following equation:

$$\sigma_b = \mathrm{Fmax} / \mathrm{A}\ (\mathrm{N/mm^2}) \tag{5}$$

where:
F_{max}: Maximum force at the moment of fracture
A: Cross-sectional area of the sample (mm²)

After the test, the moisture content of the samples was determined according to TS 2471, and the compressive strength values of the samples deviating from 12% (σ_{b12})

$$\sigma_{b12} = \sigma_b\,[1\text{-}0.05\,(12\text{-}r)]\ \mathrm{N/mm^2} \tag{6}$$

An example of a compressive strength test parallel to the fibers in the universal testing machine is shown in Figure 1.

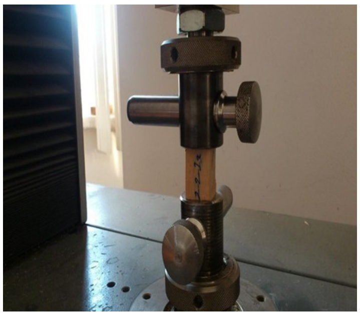

Fig. 1. Compressive strength parallel to the fibres on a universal testing machine

2. Dynamic bending (shock) resistance

Dynamic bending strength was determined in accordance with TS 2477. Before testing, the air-dried specimens' width and height were measured at the mid-point using a caliper (precision ±0.01 mm), and their cross-sectional areas were calculated. Control and treated specimens, prepared from pedunculate oak (20 × 20 × 320 mm), were subjected to liquid nitrogen treatment for varying durations and impregnated accordingly. Before testing, the specimens were conditioned at 20 ± 2 °C and 65 ± 3% relative humidity until they reached constant weight and a moisture content of 12%.

Dynamic bending resistance was calculated with a pendulum hammer tool. A 10 kg·m impact hammer dropped freely from a certain height spends some of its kinetic energy in the initial position to break the sample. Therefore, the difference between the height after breaking the sample and the initial height is the amount of work spent to break the sample. The work (w) expended at the moment of fracture is determined from the graduated dial on the tool. The dynamic bending resistance (σ_{DE}) is calculated with the following equation:

$$(\sigma_{DE}) = w / b.h \ (\mathrm{kg \cdot m / cm^2}) \tag{7}$$

where:
w: Work spent at the moment of breakage (kg·m)
b: Width of the sample (cm)
h: Thickness of sample (cm)

After the test, the humidity of the samples was determined according to TS 2471, and the dynamic bending strength values of the samples deviating from 12% ($\sigma_{DE12)}$:

$$\sigma_{DE12} = \sigma_{DE} \, [1 - 0.025 \, (12 - r)] \tag{8}$$

Results and discussion

Air-dry density data for oak wood impregnated with Tanalith-E, Borax, Diammonium Sulphate, and Imersol Aqua after liquid nitrogen exposure are presented in Table 2.

According to the table, the highest density value in oak wood was found in samples impregnated using Borax impregnation and pressure impregnation method, which were kept in nitrogen for 90 minutes, with 0.852 g/cm³. The lowest density value

Table 2. Statistical results regarding air-dry densities of stalked oak wood (g/cm³)

Impregnation Agent	Impregnation Method	Waiting Time 15 minutes	90 minutes	6 hours	Impregnation Agent	Impregnation Method	Waiting Time 15 minutes	90 minutes	6 hours
Tanalith-E	Brush	0.807	0.827	0.814	**Dia. Sulphate**	Brush	0.784	0.846	0.8
	Short term	0.844	0.79	0.846		Short term	0.838	0.812	0.798
	Long term	0.798	0.794	0.820		Long term	0.808	0.813	0.827
	Pressure	0.812	0.823	0.805		Pressure	0.784	0.782	0.816
Borax	Brush	0.832	0.845	0.817	**Imersol Aqua**	Brush	0.824	0.814	0.797
	Short term	0.802	0.819	0.814		Short term	0.772	0.810	0.790
	Long term	0.786	0.798	0.787		Long term	0.825	0.835	0.824
	Pressure	0.746	0.852	0.763		Pressure	0.830	0.804	0.795
Control					0.680				

Table 3. Oak wood kept in liquid nitrogen retention regarding the value's average values (kg/m^3)

Imp. Method	Impr.	Control	15 minutes	90 minutes	6 hours	Imp. Method	Impr.	Control	15 minutes	90 minutes	6 hours
Brush	Tanalith-E	0.683 (0.089)	1.347 (0.222)	3.131 (0.164)	2.443 (0.359)	**Long Term Imm.**	Tanalith-E	0.525 (0.182)	2.687 (0.554)	2.362 (0.318)	1.926 (0.671)
	Borax	0.652 (0.073)	1.668 (0.154)	3.447 (0.354)	2.423 (0.339)		Borax	1.306 (0.096)	3.265 (0.532)	4.212 (0.315)	2.810 (0.628)
	Dia. Sulphate	0.571 (0.135)	2.906 (0.295)	3.535 (0.204)	3.059 (0.631)		Dia. Sulphate	1.031 (0.190)	3.286 (0.699)	3.341 (0.250)	2.703 (0.348)
	Imersol Aqua	0.784 (0.118)	1.972 (0.177)	3.245 (0.095)	2.230 (0.504)		Imersol Aqua	1.108 (0.163)	2.711 (1.263)	4.131 (1.142)	3.280 (0.286)
Short Term Imm.	Tanalith-E	0.976 (0.096)	1.739 (0.240)	3.458 (0.186)	2.909 (0.493)	**Pressure**	Tanalith-E	0.597 (0.022)	0.665 (0.202)	2.854 (0.675)	2.168 (0.179)
	Borax	0.970 (0.185)	2.872 (0.523)	3.568 (0.156)	3.037 (0.662)		Borax	1.408 (0.150)	2.161 (0.587)	3.548 (1.091)	2.004 (0.842)
	Dia. Sulphate	0.678 (0.159)	3.170 (0.389)	3.307 (0.379)	2.215 (0.194)		Dia. Sulphate	0.509 (0.028)	1.762 (0.374)	2.710 (0.377)	1.989 (0.829)
	Imersol Aqua	0.964 (0.248)	2.579 (0.386)	3.894 (0.420)	3.262 (0.600)		Imersol Aqua	0.874 (0.124)	1.179 (0.632)	2.509 (0.280)	1.882 (0.545)

Values in parentheses give the standard deviation

Table 4. Multiple analyses of variance results for oak wood retention values

Sources of Variance	Sum of Squares	Degree of Freedom	Mean Squares	F Account	Importance Level
Corrected Model	347.981 ^{a}	63	5.524	26.132	0.000
Fixed Term	1602.669	1	1602.669	7582	0.000
A: Hold Time	227.887	3	75.962	359.387	0.000
B: Impregnation Method	28.103	3	9.368	44.32	0.000
C: Impregnation Agent	13.31	3	4.437	20.991	0.000
Interaction A*B	9.834	9	1.093	5.169	0.000
Interaction A*C	30.419	9	3.38	15.991	0.000
Interaction B*C	12.682	9	1.409	6.667	0.000
Interaction A*B*C	25.746	27	0.954	4.511	0.000
Mistake	54.11	256	0.211		
Total	2004.76	320			
Adjusted Total	402.09	319			
aR2= 0.865					

in oak wood was found in samples impregnated using the Borax impregnation and pressure impregnation method, which were kept in nitrogen for 15 minutes, with 0.746 g/cm^3.

The highest retention values were obtained after 90 minutes of liquid nitrogen application. These values were: Tanalith-E (3.458 kg/m^3, short-term immersion), Borax (4.212 kg/m^3, long-term immersion), Diammonium Sulphate (3.535 kg/m^3, brush), and Imersol Aqua (4.131 kg/m^3, long-term immersion). The statistical significance of the obtained values is given in Table 4.

According to the results of multiple variance analyses, impregnation type, duration, and impregnation agent were individually significant. Pairwise variations of these components and the simultaneous application of all three were found to have significant effects on oak wood retention. The results of the Duncan test, conducted to determine which

Table 5. Duncan test results for oak wood retention values at a 95% confidence interval

Holding Time in Liquid Nitrogen	**Average**	**Impregnation Method**	**Average**	**Impregnation Agent**	**Average**
Control	0.987 A	Pressure	1.801 A	Tanalith -E	1.904 A
15 minutes	2.113 B	Brush	2.131 B	Imersol Aqua	2.288 B
6 hours	2.521 C	Short term	2.475 C	Diammonium sulphate	2.298 B
90 minutes	3.328 D	Long term	2.543 C	Borax	2.459 C

Table 6. Average values of pressure parallel to the fibers of oak wood (N/mm^2)

Imp. Method	**Impr.**	**Control**	**15 minutes**	**90 minutes**	**6 hours**	**Imp. Method**	**Impr.**	**Control**	**15 minutes**	**90 minutes**	**6 hours**
Brush	Tanalith -E	84.708 (0.887)	81.612 (7.308)	86.304 (4.783)	83.290 (4.966)	Long Term Imm.	Tanalith -E	84.222 (5.863)	86.652 (3.846)	85.162 (1.338)	84.878 (3.250)
	Borax	82.962 (1.053)	82.266 (1.533)	85.022 (4.460)	80.848 (12.707)		Borax	86.304 (2.713)	78.354 (5.182)	79.926 (2.586)	85.980 (6.036)
	Dia. Sulphate	85.774 (2.936)	84.878 (7.272)	84.298 (10.005)	82.870 (5.262)		Dia. Sulphate	75.118 (14.07)	83.636 (2.922)	84.510 (2.047)	70.580 (14.631)
	Imersol Aqua	86.648 (4.579)	77.854 (5.677)	84.068 (6.506)	83.440 (4.097)		Imersol Aqua	87.756 (1.949)	85.756 (3.771)	82.070 (3.903)	70.078 (4.427)
Short Term Imm.	Tanalith -E	87.222 (1.429)	78.860 (9.250)	82.636 (6.377)	78.204 (7.561)	Pressure	Tanalith -E	93.550 (4.919)	88.500 (3.820)	82.426 (3.820)	78.064 (8.549)
	Borax	79.932 (9.682)	80.192 (5.653)	84.674 (6.052)	82.824 (2.460)		Borax	84.158 (4.161)	83.616 (3.586)	85.166 (2.785)	85.094 (1.522)
	Dia. Sulphate	80.470 (8.313)	81.694 (3.510)	83.170 (4.922)	80.394 (4.458)		Dia. Sulphate	85.820 (3.150)	85.082 (4.165)	87.678 (5.392)	80.232 (5.275)
	Imersol Aqua	88.660 (0.904)	80.418 (4.272)	87.930 (6.415)	79.820 (3.577)		Imersol Aqua	80.022 (4.502)	86.888 (2.565)	87.864 (4.739)	78.352 (4.046)

Values in parentheses give the standard deviation

Table 7. Results of multiple analyses of variance regarding the pressure values parallel to the fibers of oak wood

Sources of Variance	**Sum of Squares**	**Degree of Freedom**	**Mean Squares**	**F Account**	**Importance Level**
Corrected Model	5120.383 [a]	63	81.276	2.508	0
Fixed Term	2208994	1	2208994	68170	0
A: Hold Time	972.105	3	324.035	10	0
B: Impregnation Method	337.517	3	112.506	3.472	0.017
C: Impregnation Agent	145.903	3	48.634	1.501	0.215
Interaction A*B	419.049	9	46.561	1.437	0.172
Interaction A*C	789.477	9	87.72	2.707	0.005
Interaction B*C	519.441	9	57.716	1.781	0.072
Interaction A*B*C	1936.891	27	71.737	2.214	0.001
Mistake	8295.413	256	32.404		
Total	2222410	320			
Adjusted Total	13415.8	319			
aR2= 0.382					

treatments yielded significant differences, are presented in Table 5.

Duncan test results, the effects of pre-impregnation liquid nitrogen application, impregnation methods, and materials on oak wood retention values were examined separately. Liquid nitrogen application time was found to be significant at each stage. The highest average retention value was in 90 minutes of liquid nitrogen application. Short and long-term impregnation methods were found to be in the same range in impregnation methods. The highest average retention result was obtained in long-term immersion applications. While in impregnation materials Imersol Aqua and Diammonium sulphate were in the same group, the best average retention result was obtained in Borax.

The table shows the average pressure resistance values parallel to the fibers of oak wood for the application of liquid nitrogen before impregnation, impregnation methods, and impregnation materials. The highest-pressure resistance value parallel to the fibers for Tanalith -E was 93.55 N/mm^2 in samples that were not exposed to liquid nitrogen before impregnation and were impregnated with the pressure method. The highest value for Borax was 86.30 N/mm^2 in samples that were not exposed to liquid nitrogen before impregnation and were impregnated with the long-term immersion method. The highest value for Diammonium Sulphate was 87.67 N/mm^2 in samples that were exposed to liquid nitrogen for 90 minutes before impregnation and were impregnated with the pressure method. The highest compressive strength value parallel to the fibers for Imersol Aqua was 88.66 N/mm^2, which was observed in samples that were not exposed to liquid nitrogen before impregnation and were impregnated by the short-term immersion method.

According to the multiple variance analysis results, impregnation materials alone were not found to be significant on the compressive strength parallel to the fibers. Impregnation time and impregnation method were found to be significant alone. While the impregnation

Table 8. Duncan test results for the pressure values parallel to the fibers of oak wood

Holding Time in Liquid Nitrogen	Average	Impregnation Method	Average	Impregnation Agent	Average
Control	84.582 A	Short term	82.318 A	Imersol Aqua	82.976 A
15 minutes	82.891 A	Pressure	84.532 B	Borax	82.957 A
90 minutes	84.556 A	Long term	81.936 A	Tanalith -E	84.143 A
6 hours	80.309 B	Brush	83.552 B	Diammonium Sulphate	82.262 A

Table 9. Average values of dynamic bending (shock) resistance of oak wood (kg·m/cm^2)

Imp. Method	Impr.	Control	15 minutes	90 minutes	6 hours	Imp. Method	Impr.	Control	15 minutes	90 minutes	6 hours
Brush	Tanalith -E	3.300 (0.100)	3.760 (1.266)	3.660 (0.712)	3.440 (0.151)	**Long Term Imm.**	Tanalith -E	2.800 (0.100)	3.340 (0.288)	3.120 (0.576)	2.480 (1.135)
	Borax	2.620 (0.567)	3.180 (1.030)	4.080 (0.672)	2.600 (0.738)		Borax	2.960 (0.054)	3.000 (0.374)	3.320 (0.502)	2.900 (1.224)
	Dia. Sulphate	3.360 (0.409)	2.760 (0.371)	3.080 (0.258)	3.440 (0.151)		Dia. Sulphate	3.980 (0.083)	2.440 (0.658)	3.160 (0.409)	2.860 (0.766)
	Imersol Aqua	2.000 (0.100)	3.520 (0.672)	3.460 (0.288)	3.040 (0.801)		Imersol Aqua	3.800 (0.100)	2.360 (0.230)	3.160 (0.054)	3.400 (0.561)
Short Term Imm.	Tanalith -E	2.680 (0.083)	3.040 (0.270)	3.480 (0.622)	2.960 (0.507)	**Pressure**	Tanalith -E	2.280 (0.204)	3.140 (0.798)	2.620 (0.895)	3.000 (0.300)
	Borax	2.660 (0.134)	3.460 (0.746)	3.040 (0.952)	3.000 (0.300)		Borax	2.500 (0.100)	3.180 (0.563)	2.860 (0.773)	3.460 (0.350)
	Dia. Sulphate	1.720 (0.258)	2.720 (0.614)	2.940 (0.646)	3.360 (0.981)		Dia. Sulphate	2.460 (0.151)	2.700 (0.158)	3.320 (0.506)	2.880 (0.454)
	Imersol Aqua	2.100 (0.100)	3.020 (0.130)	3.640 (0.114)	2.320 (0.268)		Imersol Aqua	3.000 (0.100)	2.640 (0.151)	2.460 (0.350)	2.720 (0.712)

Values in parentheses give the standard deviation

Table 10. Dynamic bending resistance of oak wood results of multiple analysis of variance for the values

Sources of Variance	Sum of Squares	Degree of Freedom	Mean Squares	F Account	Importance Level
Corrected Model	79.359a	63	1.26	4.173	0
Fixed Term	2857.2	1	2857.2	9.465E3	0
A: Hold Time	9.319	3	3.106	10.291	0
B: Impregnation Method	3.978	3	1.326	4.393	0.005
C: Impregnation Agent	1.066	3	0.355	1.177	0.319
Interaction A*B	16.972	9	1.886	6.247	0
Interaction A*C	6.244	9	0.694	2.298	0.017
Interaction B*C	5.432	9	0.604	2	0.04
Interaction A*B*C	36.348	27	1.34	4.46	0
Mistake	77.276	256	0.302		
Total	3013.88	320			
Adjusted Total	156.635	319			

Table 11. Dynamic bending (shock) resistance of oak wood: Duncan test results for values

Holding Time in Liquid Nitrogen	Average	Impregnation Method	Average	Impregnation Agent	Average
Control	2.732 A	Pressure	2.826 A	Imersol Aqua	2.915 A
15 minutes	3.016 B	Short term	2.946 A	Diammonium Sulphate	2.948 A
6 hours	2.991 B	Long term	3.067 B	Tanalith -E	3.037 A
90 minutes	3.212 C	Brush	3.112 B	Borax	3.051 A

method and impregnation material pair were found to be insignificant among themselves, the other two-way and three-way interactions were determined to be significant. The results of the Duncan test conducted to determine in which applications the difference was significant are given in Table 8.

According to the Duncan test results, a significant difference was observed only when liquid nitrogen was applied for 6 hours. In the impregnation method, the short and long-term immersion methods were in the same group, with the highest average value found in the pressure method. No statistical difference was observed between the groups regarding impregnation materials.

Tanalith-E, the highest shock resistance value was 3.76 kg·m/cm^2 in samples that were exposed to liquid nitrogen for 15 minutes before impregnation and impregnated by brushing. For Borax, the highest shock resistance value was 4.08 kg·m/cm^2 in samples that were exposed to liquid nitrogen for 90 minutes before impregnation and impregnated by brushing. For Diammonium Sulphate, the highest shock resistance value of 3.98 kg·m/cm^2 was found in control samples prepared for the long-term immersion method. Similarly, the highest impact strength value for Imersol Aqua was found to be 3.80 kg·m/cm^2 in control samples prepared for the long-term immersion method.

According to the multiple variance analysis results, impregnation time and impregnation methods were found to be significant on shock resistance alone. The impregnation substance was found to be insignificant on shock resistance alone. Other binary and ternary interactions on shock resistance were found to be significant. The results of the Duncan test performed to determine in which applications the difference is significant are given below.

The Duncan test showed that 90 minutes of liquid nitrogen application provided the highest average shock resistance, with a statistically significant difference compared to the other durations. Among the impregnation methods, brush and long-term immersion were included in the same group. No significant difference was found between the impregnation methods.

Conclusions

To enhance wood material durability and service life, impregnation and surface treatments are commonly applied. Depending on the intended use, fire retardants or biocidal agents are employed. In impregnation treatments, the penetration depth and the amount of preservative retained in the wood are critical factors. In some species that are difficult to impregnate, the amount of retention may be insufficient, and even when impregnation is achieved, the preservative can be leached out over time. This results in wood becoming vulnerable to biotic deterioration.

As a novel approach to overcome these limitations, liquid nitrogen pretreatment was found to facilitate the penetration of preservatives into wood, leading to significant increases in retention. Consequently, the service life of the material can be improved.

In this study, applying liquid nitrogen to oak wood before impregnation resulted in retention increases across all methods tested. In the brushing method, the most significant increase was observed after 90 minutes of liquid nitrogen treatment compared with the control group: 358% for Tanalith-E, 428% for Borax, 519% for Diammonium Sulphate, and 313% for Imersol Aqua. In the short-term immersion method, the highest retention increase was obtained at 90 minutes, with 254% for Tanalith-E, 268% for Borax, and 303% for Imersol Aqua. In contrast, Diammonium Sulphate showed its maximum increase (387%) at 15 minutes. In the long-term immersion method, retention increases were 411% for Tanalith-E (90 min), 222% for Borax (15 min), 224% for Diammonium Sulphate (90 min), and 272% for Imersol Aqua (90 min). Under pressure treatment, retention increased by 378% for Tanalith-E (90 min), 151% for Borax (90 min), 432% for Diammonium Sulphate (15 min), and 187% for Imersol Aqua (90 min). These results demonstrate the effectiveness of liquid nitrogen pretreatment in enhancing retention.

When examining the effect of liquid nitrogen on mechanical properties, the compressive strength parallel to the fibers varied depending on the preservative used. The highest compressive strength (93.55 N/mm^2) was obtained in samples pressure-impregnated with Tanalith-E, while the lowest compressive strength (75.118 N/mm^2) was observed in samples treated with Tanalith-E.

Similarly, shock resistance was also affected. The highest shock resistance (4.08 kg·m/cm^2) was recorded in samples treated with Borax using the brush application method. The lowest value (1.720 kg·m/cm^2) was observed in samples treated with Diammonium Sulphate, also using the brush application method.

Overall, liquid nitrogen pretreatment significantly increased adhesion in all applications. While the effects on compressive strength and impact resistance vary depending on the preservative type and application method, the results clearly demonstrate that liquid nitrogen application is a promising method for improving the impregnation performance of oak. Changes in mechanical properties may be due to microcracks induced in the cell wall by liquid nitrogen (Wang, Lin, & Xing, 2021).

In a study on the cryogenic treatment of Uludağ fir wood, Kurt (2022) reported that pre-impregnation liquid nitrogen application enhanced retention by an average of 150–200%. This finding supports the retention increases observed in oak in the present study, though the greater gains in oak suggest species-specific anatomical differences. Similarly, Augustina et al. (2023) highlighted in their review of wood impregnation mechanisms that liquid nitrogen application improves retention across wood species without significant structural drawbacks.

These comparative data demonstrate that liquid nitrogen pretreatment enhances retention at varying rates across different wood species, underscoring its potential for broader industrial application as a pretreatment strategy.

Conflict of interest

The author(s) declare(s) that there is no conflict of interest concerning the publication of this article.

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