

Mechanical Properties of Red Pine (*Pinus brutia* Ten.) depending on Bay Leaf (*Laurus nobilis* L.) Extract Treatment Level and Time

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The rapid depletion of forest and water resources, drought, climate change, and the significant loss of freshwater resources, along with fires and wars, necessitate the creation of healthy materials and a hygienic structure, as well as the optimal use of wood. This study used various processes to obtain extracts from laurel leaf waste, which is known for its antioxidant/antibacterial properties. Subsequently, solutions of various concentrations (1%, 3%, and 5%) were prepared and the wood was impregnated using the immersion method (short, medium, and long-term). Tests were then conducted to determine the retention, specific density, bending strength, modulus of elasticity, compressive strength, and dynamic bending. The highest retention was determined at a 5% concentration for 6 h (2.50%), the highest air-dry specific density was determined at 5% for 6 h (0.68 g/cm³), and the highest bending strength was determined at 5% for 6 h (143 N/mm²). The extract prepared from ecological bay leaf plant waste with water creates a partially hygienic (antioxidant/antibacterial) framework for organic wood, benefiting human and environmental health.

DOI: 10.15376/biores.21.1.1747-1761

Keywords: Waste plant leaves; Aromatic plants; Extract; Wood technology; Antimicrobial wood surface

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INTRODUCTION

Wood is an important raw material that has been used and continues to be used in many areas of human existence, including shelter, food, fuel, tools, and more. It is distributed in diverse areas across the globe and is also a renewable resource. However, reckless felling and forest fires endanger the continuing existence of forests. All wood-based products must be utilized efficiently, their properties must be well understood, and their useful life must be extended. With advancements in technology and opportunities, diversification has increased, and the level of use has increased considerably. However, due to its organic structure, wood is susceptible to degradation by biotic and abiotic factors. This disadvantage can be mitigated through various protection methods and techniques. The diversity and continuity of risks necessitate chemical treatments (OGM 2025).

Studies on the effects of impregnated wood on biological durability have considered a range of different aspects, including the wood species. Natural or water-soluble impregnation materials have been used. Pressure is commonly applied to enhance impregnation. Both natural and treated wood samples have been evaluated with respect to combustion properties to show the effects of natural, oily, water-soluble, and organic

solvent-based impregnation materials. Both pressure-applied and non-pressure-applied methods were used as impregnation methods (Öztürk 2024). Yılmaz (2022) has shown that the extracts and essential oils of laurel and zahter plants, with their active compounds, exhibited antimicrobial activity against *S. typhimurium*, as a foodborne pathogen inoculated into chicken wings. Furthermore, this study evaluated the extracts and essential oils of zahter and bay laurel plants, and the antimicrobial activity of bay laurel essential oil was found to be more effective than other extracts and essential oils.

The widespread adoption of sustainable development has enabled humanity to better grasp the importance of ecology. However, this process has led to the emergence of flawed, albeit well-intentioned, environmental interventions. Past, uninformed practices, along with current, flawed approaches, threaten many wood species, disrupting the ecological restoration cycle and accelerating the rate of species extinction. Ecological restoration is a process that aims to restore ecosystems in degraded areas. The primary objectives in this process are the protection of existing species, the maintenance of biodiversity, and the restoration of the ecosystem to its former functional state. In every interaction with nature, preserving the natural environment and providing suitable habitats for all living things should be a priority (Gökkür and Şahin 2015). Impregnation applications and preferred methods are crucial for preserving the durability of all wood components and extending their lifespans. Impregnation processes carried out in accordance with relevant standards significantly extend the service life of wood by increasing its resistance to biological and environmental factors. Biotic and abiotic deterioration, which occurs over time in historical wooden structures, can be effectively addressed using appropriate impregnation techniques and on-site conservation interventions, thus ensuring both structural integrity and the sustainability of cultural heritage (Kartal 2016).

Several basic approaches have been developed for the maintenance and preservation of historic timber structures: renovation, restoration, impregnation, protection, and reconstruction (Lebow and Anthony 2012). The necessity of preserving a structure is often associated with its “authenticity.” This authenticity depends on the existence of information sources related to the structure. These sources encompass the building’s design, form, materials used, function, traditional practices, techniques, location and position, spiritual value, expressive power, and its development throughout history. The holistic structure created by all these elements allows for the multilayered definition of cultural heritage (Bayraktar and Kesik 2022).

The increasing prevalence of environmental pollution and diseases in recent years demonstrates the importance of environmental and human health. People are increasingly drawn to natural products. Therefore, products used in homes, such as furniture, the paper industry, park/garden furniture, the wooden toy industry, and others, are of great importance for a healthy life. Children grow up surrounded by toys from the first months of their lives, and it has been reported that impregnation of wooden toys and the use of herbal extracts create a high level of hygiene (Kızılcı 2025).

The main objective of this study was to optimize the use of bay leaf plant waste (with its medicinal and aromatic properties) in the country’s forest resources, to determine the adhesion level of various concentrations of plant extract impregnated onto wood material from a human/environmental health perspective, and to expand the uses of wood (red pine) by determining its various technological properties. This will also create a hygienic environment for the wood.

EXPERIMENTAL

Materials

Wood and plant

In the research, the wood of the coniferous tree species, red pine (*Pinus brutia* Ten.) was used. The extract from the waste laurel leaves (*Laurus nobilis* L.) waste were used. Both the wood and the waste laurel leaves were obtained from the city of Muğla in Turkey. Brutian pine (*P. brutia*) is a heat and drought-resistant coniferous tree commonly found in regions with a Mediterranean climate. It is a fast-growing species with good fire resistance. Long-lived trees can apparently live for a very long time (5,000+ years), but this seems to be limited by a size-growth interaction. Its average density is between (0.43 and 0.55 g/cm³), varying depending on habitat, altitude, etc. (Öktem 1987).

Sample preparation

Test samples were prepared from sturdy, knot-free, and smooth-fibered laths. For the tests, the laths were first conditioned to 20 ± °C at 65%±3% relative humidity according to TS ISO 3129 (2021) and TS 2470-2471 (1976). After conditioning using air-dry (12%) moisture content, they were brought to the desired dimensions. The standard TS 2472 (1976) was used for determining specific density, TS 2477 (1976) for dynamic bending resistance (shock), and TS 2478 (1976) for determining the modulus of elasticity in bending and ASTM E1131-08 (2010) was performed in thermogravimetric analyses (TGA).

Methods

Impregnation

The impregnation process used was a dipping method. Test samples were dried completely (0%) in an oven for 24 h at a temperature of 103±2 °C. Then they were immersed in a natural preservative derived from plant extracts for 30 min, 6 h, and 24 h. For each test (Ten samples in each tests), all test group samples, except the control group samples, were individually impregnated with a solution prepared from laurel plant extract by the dipping method (Var 1994).

Plant extract preparation

Bay leaf waste, a natural/ecological medicinal aromatic plant species, was ground into powder using a ring mill. Before use in preparing the solution, the resulting powder was passed through a sieve with a 63-micron mesh size to ensure homogeneity of particle size. Solutions were prepared from the sieved bay leaf powder in concentrations of 1%, 3%, and 5%, each in a volume of 100 mL. The weighed samples were transferred to 200 ml beakers and dissolved using a magnetic stirrer. Approximately 15 to 20 mL of 32% ultra-pure HCl and 10 mL of distilled water were added to the beakers for the dissolution process. The bay leaf waste was dissolved at a stirring speed of 500 rpm and a temperature of 200°C. Finally, the resulting solutions were diluted to a final volume of 100 mL using distilled water (Ceylan 2020).

Determination of extract retention in wood

The amount of material remaining in impregnated wood samples compared to completely dry wood (% retention) was calculated using the Eq. 1 (Baysal 1994),

$$\text{Retention (\%)} = \frac{M_{\text{oes}} - M_{\text{oeö}} \times C}{100} \quad (1)$$

where M_{oes} is the total dry weight of the test piece after impregnation (g), $M_{\text{oeö}}$ is the total dry weight of the test piece before impregnation (g), and C is the concentration

Thermogravimetric analysis

Thermogravimetric analysis (TGA) is a method that allows the precise measurement of mass loss in polymers and similar samples under a specific temperature increase. This technique is widely used, particularly in the study of thermal degradation processes of polymers and the kinetic events that occur during degradation. TGA measurements were conducted in accordance with the ASTM E1131-08 standard. Approximately 10 mg wood flour samples, which passed through a 40-mesh sieve but not through a 60-mesh sieve, were used in the experiments. The samples were heated from 25 to 600 °C in a nitrogen atmosphere with a flow rate of 57.10 °C/min. The gas flow rate was set at 50 mL/min throughout the experiment. Based on the data obtained, the mass loss at the highest temperature, the time intervals at which the most rapid mass loss occurred, and the fast pyrolysis temperature values were determined. These parameters were used to compare the thermal resistance of unimpregnated control samples and samples containing different concentrations of CaCO_3 (Tutuş *et al.* 2010).

Data Analysis

The SPSS statistical program (Long produced by SPSS Inc., acquired by IBM in 2009., version 26, Chicago, IL, USA) was used to analyze the data. The effects of wood type and the percentage change in plant extract concentration were analyzed to create homogeneity groups and apply simple analysis of variance. Ten samples were used in each experimental variation. Factors considered included the wood type x concentration x experimental groups, *etc.* The average, minimum value, and maximum value were determined in each experimental period. The confidence interval was taken as 95%. Homogeneity groups were created by performing Simple Variance Analysis.

RESULTS AND DISCUSSION

Solution Properties

The properties of the bay laurel plant extracts are given in Table 1.

Table 1. Properties of Solution

Plant Extract		Solvent	Temperature (°C)	pH		Density (g/mL)	
				BI	AI	BI	AI
Laurel Plant	1%	Water	22	5.90	5.90	0.918	0.916
	3%			5.88	5.87	0.921	0.921
	5%			5.82	5.80	0.928	0.927

BI: Before impregnation **AI:** After impregnation

No change in solution properties, pH, and density was detected.

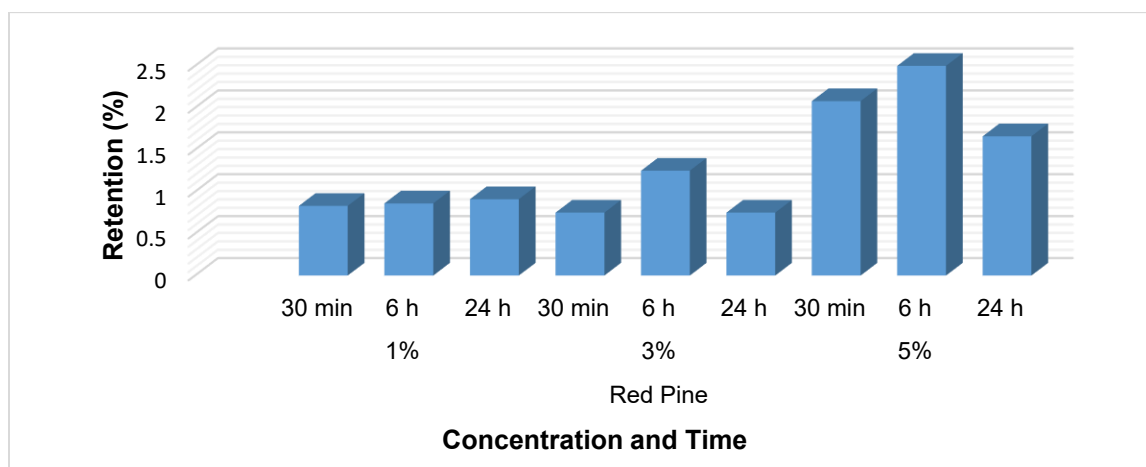
Retention

The Retention values are shown in Table 2 and the relevant graph in Fig. 1.

Table 2. Retention

Wood	Concentration	Immersion Time (minute/hours)	Retention (%)	HG
<i>Pinus brutia</i>	1%	30 min	0.83	G
		6 h	0.86	F
		24 h	0.91	E
	3%	30 min	0.75	H
		6 h	1.25	D
		24 h	0.75	H
	5%	30 min	2.08	B
		6 h	2.50	A
		24 h	1.66	C

HG: Homogeneity groups

**Fig. 1.** Retention of bay leaf oil based on mass

The highest retention was found in 5% extract (2.50%) at 6-h immersion and the lowest in 3% extract (0.75%) at 30-min and 24-h immersion.

Var and Kaplan (2019) found the highest retention value in red pine wood treated with geothermal waters as 2.77% in the SJ-5 40.9 °C treatment, while the lowest value was 2.56% in the SJ-1 23.0 °C treatment. Yılmaz (2022) applied vacuum impregnation process to wooden material obtained from sweetgum plant leaf extract (pure water / 1%,3%,5%) and reported that the highest percentage of retention was achieved at 5% solution concentration with 30 minutes of vacuum and 30 minutes of diffusion time (1.13%), and the lowest value was at 3% concentration (0.31%). Alkan (2025) impregnated mahogany and larex wood with mussel shell solution and the highest retention in mahogany wood was determined as 10.9% at 6% concentration and 24 h of immersion time; and the highest retention in larex wood was determined as 12.6% at 6% concentration and 24 h of immersion time. Bozkurt (2025) impregnated Scots pine and chestnut wood with moss - boric acid mixture and determined the highest retention in scots pine wood in the 24-h period (17.4%), and the highest retention in chestnut wood in the 24-h application (10.3%). As the concentration increases, the % adhesion increases, but the adhesion depending on the time is not directly proportional. This may be due to solution properties, wood type, anatomical structure and impregnation method.

Determination of Physical Properties

Air-dry and full-dry density values

The simple variance analysis and Duncan's test results for air-dry and fully dry density values are shown in Table 3 and the related graph in Fig. 2.

Table 3. Air-Dry and Full-Dry Density Values

Wood	Concentration	Immersion Time (min or h)	Air-dry Density (g/cm ³)	HG	Full-dry Density (g/cm ³)	HG
<i>Pinus brutia</i>	-	Control	0.53	B	0.63	D
	1%	30 min	0.68	A	0.65	C
		6 h	0.59	G	0.57	F
		24 h	0.65	C	0.67	A
	3%	30 min	0.56	I	0.55	G
		6 h	0.60	E	0.59	E
		24 h	0.56	I	0.53	H
	5%	30 min	0.57	H	0.55	G
		6 h	0.68	A	0.66	B
		24 h	0.61	D	0.60	E

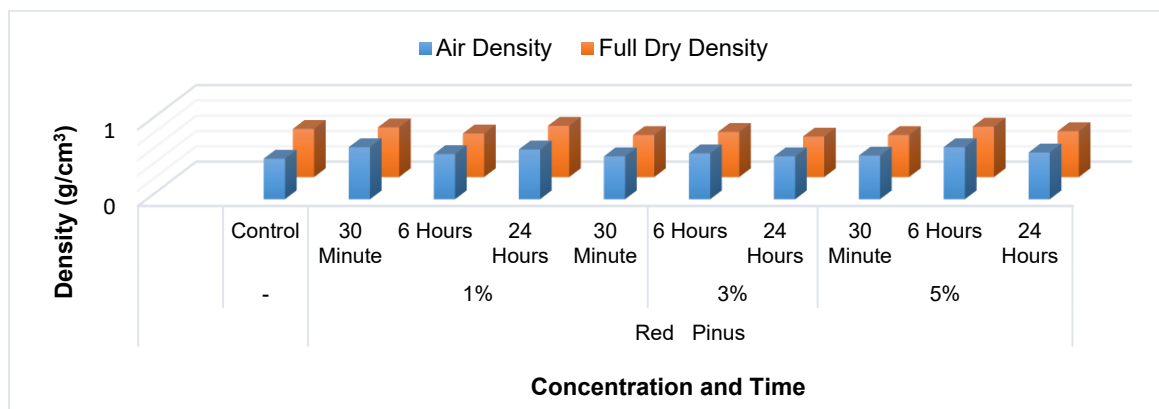


Fig. 2. Change of density values (air-dry and full-dry)

When the specific gravity data were evaluated, the highest value in terms of air-dry specific gravity was determined as 0.68 g/cm³ in the 30-min immersion period with 1% bay leaf extract, the lowest air-dry specific gravity was determined as 0.56 g/cm³ in the 24-h application of 3% extract; the highest value in full-dry specific gravity was determined as 0.67 g/cm³ in the 24-h application with 1% extract, and the lowest value was determined as 0.56 g/cm³ in the 24-h application with 3% extract.

Ölmez (2020) reported that tree species and impregnation materials showed different effects in terms of air-dry density values, and that in applications with plant extracts, the air-dry density of chestnut wood generally varied between 0.49 and 0.51 g/cm³, while these values varied between 0.42 and 0.48 g/cm³ in Scots pine wood. Yaşar *et al.* (2017) compared the effectiveness of plant-derived impregnation materials, such as acorn tannin and pine tannin, with chemical impregnation materials. Esen (2009) investigated the full-dry and air-dry density values of beech, pine, and sapele woods treated with different impregnation agents (boric acid, borax, tanalith-E, and imersol aqua); the air-dry density of beech wood was measured as 0.72 g/cm³, while the full-dry density was measured as 0.66 g/cm³ at the highest level.

Mechanical Tests

The bending strength results are given in Table 4 and the corresponding change graph is given in Fig. 3.

Table 4. Bending Strength

Wood	Concentration	Immersion Time (min or h)	Bending Strength (N/mm ²)	HG
		Control	103.83	H
<i>Pinus brutia</i>	1%	30 min	138.09	C
		6 h	89.55	I
		24 h	142.56	A
		30 min	130.00	D
	3%	6 h	121.17	F
		24 h	123.20	E
	5%	30 min	117.31	G
		6 h	143.24	A
		24 h	139.90	B

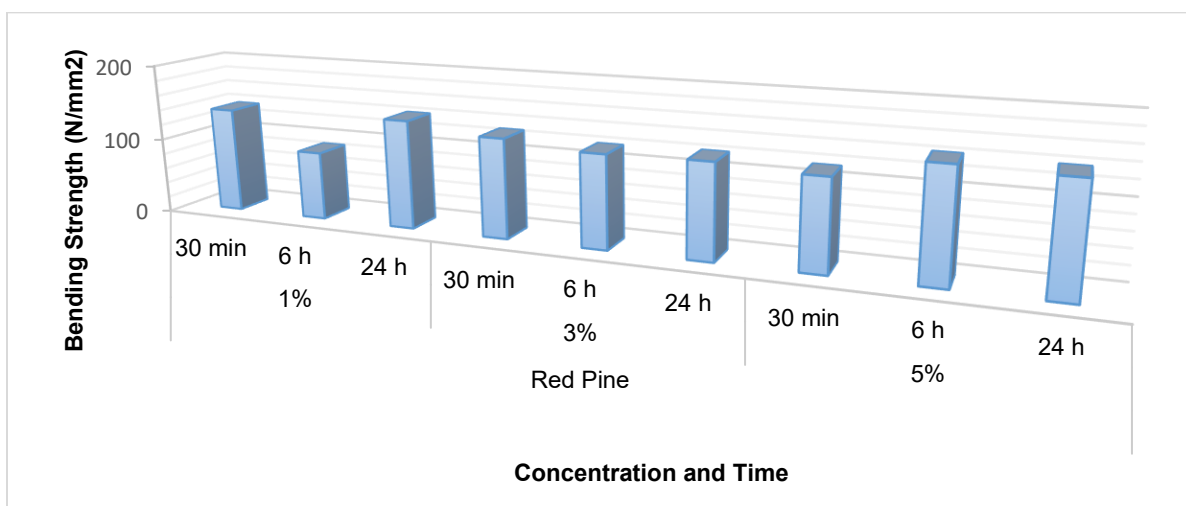


Fig. 3. Change of bending strength values

The highest bending strength value was determined at a 1% concentration after 24 h of immersion (144 N/mm²), while the lowest at a 1% concentration after 6 h (89.6 N/mm²). No significant increases were observed compared to the control sample. This suggests that the wood type, anatomical structure, impregnation material, and impregnation method all have an impact on bending strength. The importance of the anatomical characteristics of wood can be ranked as follows: veil/pores aspiration, permeability, sapwood heartwood, annual ring width, and springwood-summerwood ratio. In impregnation, the movement of liquids within the wood largely occurs through pores. Therefore, pores have a significant effect on the impregnability of the wood. Indeed, it has been reported that the flow rate of liquids within the wood is directly proportional to the 4th power of the pore opening radius (Yıldız 2003). The impregnability of wood is directly proportional to its permeability. Tree species are classified into four groups based on their permeability: easy, moderate, difficult, and very difficult. It has been reported that heartwood is more difficult to impregnate than sapwood due to aspiration and the accumulation of extractive substances on the pore membrane surfaces (Arsenault 1973).

Ulusoy and Peker (2020) determined that larex wood impregnated with 1% stone water solution gave the highest bending strength value (135 N/mm²). Efe and Çağatay (2011) studied the bending strength of solid wood materials and found that the highest bending strength of Scots pine wood was 104 N/mm² and the lowest was 75 N/mm². Ertürk (2011) impregnated some wood species and determined the bending strength using the chemicals as Imersol Aqua (98.2 N/mm²), Boric acid (95.6 N/mm²), Tanalith-E (94.7 N/mm²), and Borax (85.9 N/mm²).

The modulus of elasticity values and their related Duncan test results are shown in Table 5 and the related graph in Fig. 4.

Table 5. Modulus of Elasticity

Wood	Concentration	Immersion Time (min or h)	Modulus of Elasticity (N/mm ²)	HG
		Control	7560	F
<i>Pinus brutia</i>	1%	30 min	11709	C
		6 h	11950	C
		24 h	12102	B
		30 min	10630	D
	3%	6 h	13920	A
		24 h	11533	C
		30 min	10126	D
	5%	6 h	11877	C
		24 h	8388	E

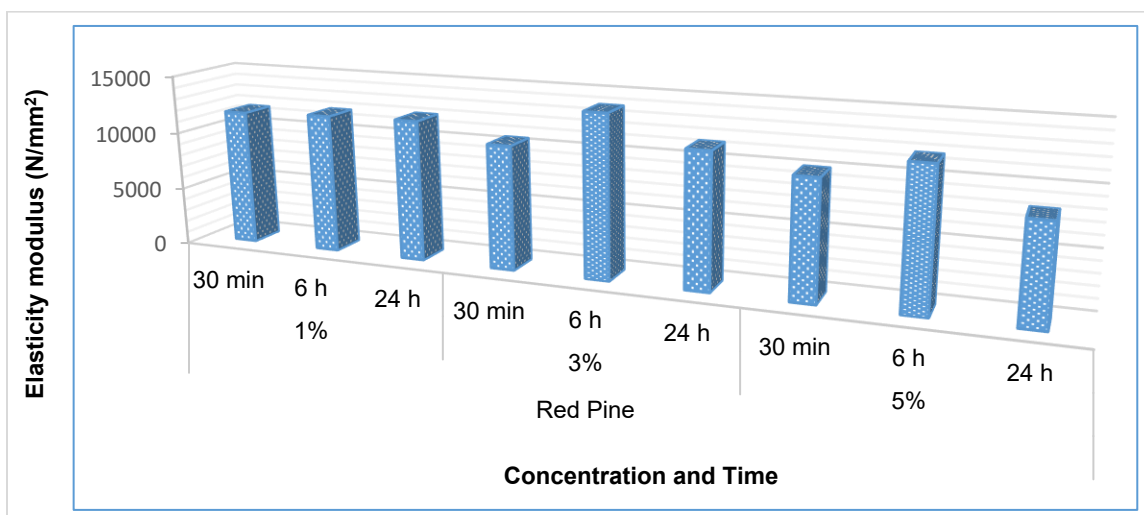


Fig. 4. Change of modulus of elasticity values

The highest elastic modulus value was determined at 6 h of immersion time for the 3% extract (13920 N/mm²), and the lowest at 24 h (8388 N/mm²) for the 5% extract. Significant increases were determined compared to the control sample.

Sefil (2010) showed that the type of wood, anatomical structure, impregnation material and impregnation method have an effect on the elastic modulus and this shows that different effects on the elastic behavior may occur depending on the concentration of the impregnation material. Toker (2009) impregnated *Pinus brutia* Ten. and Oriental beech (*Fagus orientalis* Lipsky.) samples with boric acid, borax, and sodium perborate, and reported that compressive strength, bending strength, and modulus of elasticity decreased

by 20% to 40% on average in both wood species as the solution concentration increased. Çakır (2012) chipped vineyard pruning residues and impregnated them with boron compounds (1% to 4%), and reported that impregnation with boron compounds and an increase in solution concentration in general caused decreases in the bending strength and modulus of elasticity of the test samples.

Dynamic Bending Strength

Duncan test results regarding dynamic bending strength values are given in Table 6 and the related graph is given in Fig. 5.

Table 6. Dynamic Bending Strength

Wood	Concentration	Immersion Time (min or h)	Dynamic Bending Strength (kg/cm ²)	HG
		Control	0.22	H
<i>Pinus brutia</i>	1%	30 min	0.20	I
		6 h	0.29	B
		24 h	0.24	F
	3%	30 min	0.23	G
		6 h	0.25	E
		24 h	0.21	I
	5%	30 min	0.27	D
		6 h	0.28	C
		24 h	0.32	A

HG: Homogeneity groups

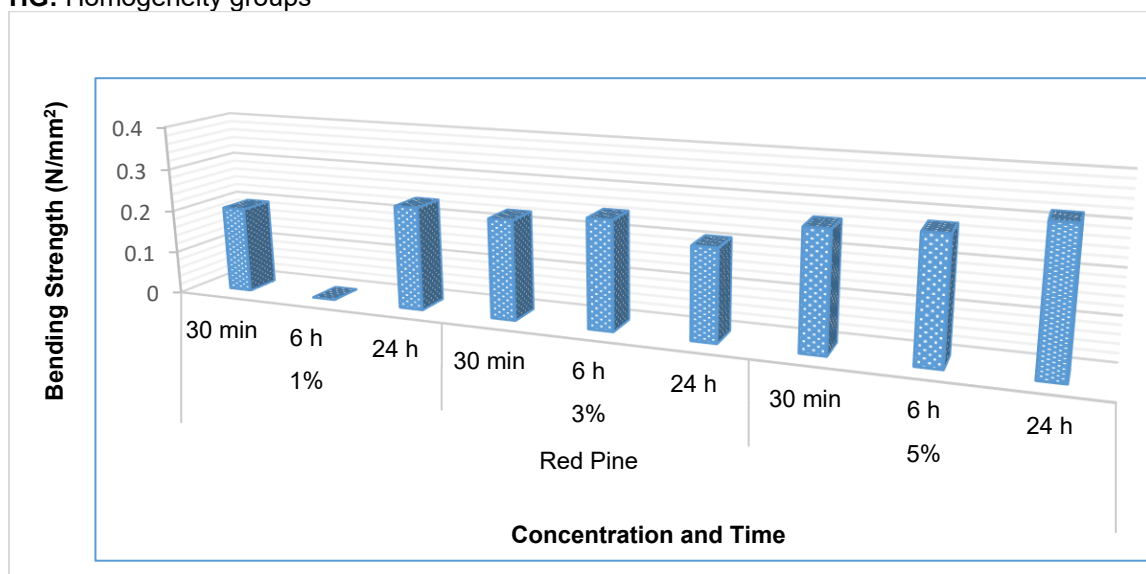


Fig. 5. Change of dynamic bending strength values

The highest dynamic bending strength value was determined at 5% concentration after 24 h of immersion (0.32 kg/cm²), and the lowest 1% concentration was determined at 30 min (0.20 kg/cm²). A significant difference was determined at a significance level of 0.05, considering homogeneity groups.

Yıldırım (2020) observed the change in dynamic bending strength to be positive compared to the control sample. The highest shock resistance was observed in spruce wood (0.63 N/mm²), and the lowest in 1% spruce + borax (0.24 N/mm²). Bektaş *et al.* (2008) reported that the dynamic bending strength of *Eucalyptus* (*E. grandis*) wood was 0.544

kg/cm², its full dry density value was 0.482 g/cm³, and that it was in the “good quality” wood group in terms of dynamic quality value.

Pressure Strength

Duncan test results regarding pressure strength values are given in Table 7 and the related graph is given in Fig. 6.

Table 7. Pressure Strength

Wood	Concentration	Immersion Time (min or h)	Pressure Strength (kg/cm ²)	HG
<i>Pinus brutia</i>	1%	Control	60.34	H
		30 min	54.26	I
		6 h	59.23	B
		24 h	55.33	F
	3%	30 min	60.10	G
		6 h	62.17	E
		24 h	66.58	I
	5%	30 min	51.83	D
		6 h	53.20	C
		24 h	60.34	A

HG: Homogeneity groups

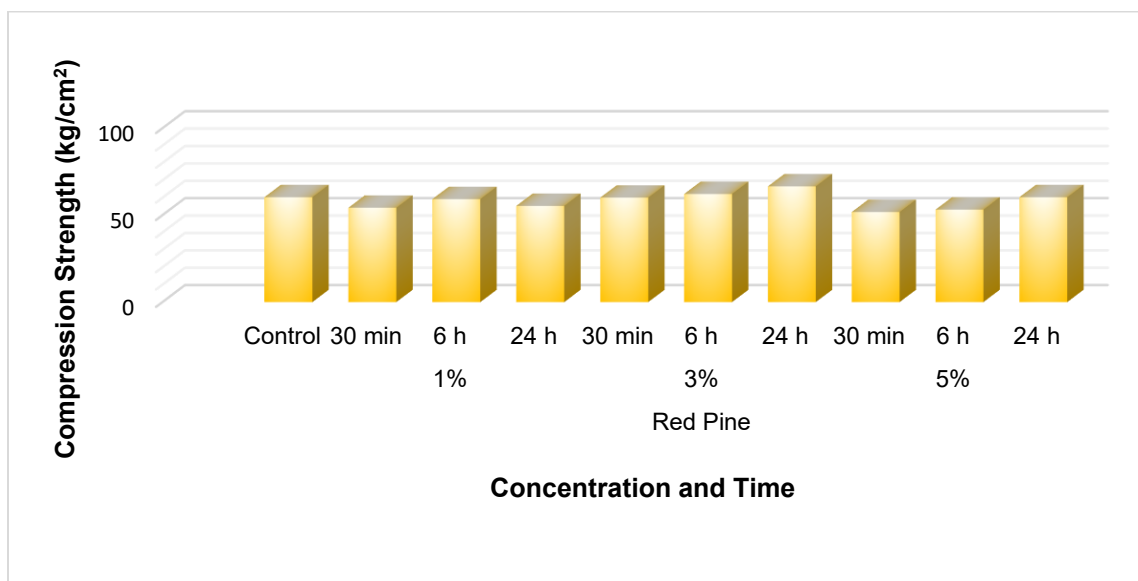


Fig. 6. Change of compression strength

The highest compression strength value was determined at 5% concentration for 24 h (60.24 N/mm²) immersion, and the lowest at 5% concentration for 30 min (51.83 N/mm²). Partial increases were detected compared to the control sample.

Çolak and Yıldız (2004) stated that when impregnation is applied at high temperatures and with high-content fluids, a decrease in compression strength may occur, particularly perpendicular to the fibers, while slight increases may occur parallel to the fibers due to material accumulation. Kartal and Kakitani (2002) demonstrated that changes in compression strength along the fiber direction in wood treated with natural and synthetic impregnates are related to the viscosity and diffusion depth of the impregnating fluid.

Thermogravimetric Analysis

The results of thermogravimetric analysis (TGA) of bay leaf extract impregnation on red pine wood at different concentrations are presented in Table 8.

Table 8. Thermogravimetric Analysis Results Based on Concentration

Concentration	Initial Temperature (°C)	End Point (°C)	Final Temperature (°C)	ΔY (%)	550 °C Amount Remaining (%)
Control	320.00	390.00	397.00	70.00	30.00
1%	315.00	385.00	395.00	68.00	32.00
3%	300.00	370.00	390.00	65.00	35.00
5%	290.00	360.00	382.00	62.00	38.00

Table 9. Thermogravimetric Analysis Results According to Concentration Properties

Bay Leaf Extract (%)	Immersion Time	Initial Temperature (T_{onset} °C)	Turning Point (T_{max} °C)	End Point (T_{end} °C)	ΔY (%)	550 °C Amount Remaining (%)
Control	–	260	425	550	84	16
1%	30 Min	260	424	553	83	17
1%	6 h	259	424	556	82	18
1%	24 h	258	423	558	81	19
3%	30 Min	259	424	555	82	18
3%	6 h	258	423	560	80	20
3%	24 h	257	422	565	78	22
5%	30 Min	258	423	558	80	20
5%	6 h	257	422	562	78	22
5%	24 h	256	421	568	76	24

ΔY (%) Total weight loss; **Initial Temperature** (°C) Temperature at which wood begins thermal decomposition (T_{onset})

Ending Point (°C): Temperature at which the most intense mass loss occurs (T_{max})

Final Decomposition Temperature (°C): Temperature at which thermal decomposition is largely complete (T_{end})

Residue at 550°C (%): Ash remaining after combustion (inorganic fraction - an indicator of thermal stability)

Compared to the control sample, positive combustion TGA results were observed at all concentrations. In particular, it was determined that the flame retardancy properties partially increased as the concentration increased. Impregnated with bay leaf waste extract rendered red pine wood more resistant to thermal degradation and may be expected to have a positive effect against fire effects.

Alkan (2025) conducted a TGA test on mahogany wood, demonstrating high thermal resistance even in the control group thanks to its dense and durable cell structure. Initial decomposition temperatures exceeded 320 °C, and this value gradually increased with the addition of mussel shells. As the impregnation rate increased, ΔY values decreased, while the remaining amount at 550 °C increased. This suggests that the CaCO_3 addition promotes charring behavior and leaves more inorganic residue after pyrolysis (Le Van and Winandy 1990). Wood is a flammable and combustible material. Flame-retardant impregnations decompose below the wood's decomposition temperature, rapidly converting cellulose into charcoal and water. This prevents the formation of volatile and

flammable substances that would form at higher temperatures, thus reducing the wood's flammability and preventing flames from spreading into the environment.

CONCLUSIONS

1. No change was detected in the properties, pH, and density values of the solution prepared from laurel plant extract.
2. The highest value in terms of air-dry specific density of the wood was determined as 0.68 g/cm³ in 30 min of immersion time with 1% bay leaf extract. The lowest air-dry specific density was measured as 0.56 g/cm³ in 24-h application of 3% extract.
3. When the bending strength results were evaluated; the highest bending strength value was determined as 144 N/mm² in 24 h of immersion time with 1% extract, the highest elasticity as 13900 N/mm² in 6 h of immersion time with 3% extract, and the lowest as 5% extract in 24 h (8390 N/mm²). The statistically significant increase in specific gravity, bending strength, and modulus of elasticity compared to the control sample showed the usability of this composition in a wide variety of wood products. If this natural material had shown a negative impact on technological properties according to statistical data, its application level would be very difficult.
4. Parameters such as compressive strength, bending strength, and shock resistance are of great importance, especially in earthquakes, fires, and natural disasters, in determining the durability of wood. Therefore, the data obtained can be preferred in all spaces/areas where wood is used, including children's toys. When the compressive strength results were evaluated, the highest compressive strength value was determined in the 5% extract at 30 min immersion time (66.6 N/mm²). No significant increases were observed compared to the control sample. It can be said that the increases and decreases in all technological properties are due to the wood type, anatomical structure, impregnation material, impregnation method, and solution concentration. The anatomical structure of the wood (cell structure, wood type, texture, *etc.*) played a positive/negative role on the obtained values.
5. The main objective of this study was to collect and reuse the waste remaining after the use of medicinal and aromatic plants, which have existed in nature since the beginning of humanity, and to integrate this material into the wood industry in relation to human/environmental health. Especially in this century where ecological balance is disrupted, the reuse of all natural resources (forests, plants, marine resources, *etc.*) in production and the creation of natural alternative resources against potential destruction over time, even if local, constitute another part of the study. The immersion method was preferred because it can be easily applied in all locations/areas. We can say that this organic solvent structure can be used in the restoration of historical wooden artifacts. Positive results have also been shown in TGA experiments.

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Article submitted: November 25, 2025; Peer review completed: December 20, 2025;
Revised version received: December 27, 2025; Accepted: December 28, 2025; Published:
January 9, 2026.

DOI: 10.15376/biores.21.1.1747-1761