



The orientation of growing in black pine wood, heat treatment and alpha-x chemical examination of the effects

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ABSTRACT: In this study, it was aimed to determine the mechanical properties of larch wood, growing direction, alpha x chemical and the effects of heat treatment. For this purpose, the black pine (*Pinus nigra*) used in the study was obtained from the Kicir Mountain warehouse in the Simav District of Kütahya Province. These samples taken were prepared for the experiment with dimensions of 20x20x30 mm and 20x20x300 mm. The samples were then impregnated with alpha x chemical and subjected to heat treatment. Pressure strength, bending strength, density and elasticity modulus tests were applied to the samples. As a result, it was found that the density and pressure values of the larches grown on the southern side were higher and the elasticity modulus values were lower compared to the larches on the northern side. When larch impregnation was applied, there was a decrease in density and bending values in both respects, while an increase in pressure values was observed. When heat treatment was applied to impregnated larches, an increase in bending and elasticity modulus values was observed in both respects, while a decrease in pressure values was observed.

Keywords: Black pine, growth orientation, alpha-x, immersion method

Karaçam odununda yetiştirme yönü, ısıtma işlem ve alfa-x kimyasalının etkilerinin incelenmesi

ÖZ: Bu çalışmada karaçam odununun mekanik özellikleri, yetiştirme yönü, alfa x kimyasalı ve ısıtma işleminin etkilerinin belirlenmesi amaçlanmıştır. Bu amaçla bu çalışmada kullanılan karaçam tomrukları Kütahya İlinin Simav İlçesine bağlı Kicir Dağı deposundan temin edilmiştir. Alınan bu örnekler 20x20x30 mm ve 20x20x300 mm ölçülerinde deney için hazırlanmıştır. Örnekler daha sonra alfa x kimyasalıyla empenye edilerek ısıtma işlemine tabi tutulmuştur. Örnekler basıncı direnci, eğilme direnci, yoğunluk ve elastikiyet modülü testleri uygulanmıştır. Sonuç olarak, güney bakıda yetişen karaçamların kuzey bakıdaki karaçamlara göre yoğunluk ve basınç değerlerinin daha fazla ve elastikiyet modülü değerlerinin daha az olduğu tespit edilmiştir. Karaçama empenye uygulandığında her iki bakıda yoğunluk ve eğilme değerlerinde düşüş görülürken basınç değerlerinde artış görülmüştür. Empenyeli karaçamlara ısıtma işlemi uygulandığında her iki bakıda eğilme ve elastikiyet modülü değerlerinde artış görülürken basınç değerlerinde düşüş görülmüştür.

Anahtar kelimeler: Karaçam, bakı, alfa-x, daldırma yöntemi

1 Introduction

The rapid development of the wood industry has contributed to a continuous decline in forest resources both globally and in Turkey. Wood is increasingly preferred due to its ease of processing, high mechanical strength, thermal and acoustic insulation properties, and aesthetic advantages compared to alternative materials (Özalp, 2003). To meet the growing demand and minimize forest depletion, it is crucial to use existing resources efficiently and conduct scientific studies to better characterize wood raw materials. Otherwise, dependence on imported wood products is expected to increase (Ulusoy et al., 2016).

One of the most effective methods to extend the service life of wood is impregnation. Impregnation involves introducing chemicals into the wood structure through various techniques. Pressure-vacuum impregnation, in particular, provides high-level protection against biological degradation factors such as saline water, soil, moisture, insects, and fungi, potentially increasing wood's service life up to tenfold (Bozkurt et al., 1993). Wood is also frequently exposed to elevated temperatures during service, which can induce physical and chemical changes depending on temperature, duration, pressure, and moisture content (Yılğör, 1999). Heat treatment offers benefits such as improved dimensional stability and biological strength; however, prolonged exposure to high temperatures may reduce mechanical properties due to material loss in cell wall components (Rusche, 1973).

Although wood is a renewable material with ease of processing and high mechanical performance relative to its density, its low biological strength and susceptibility to moisture-induced swelling and shrinkage limit its applications. Heat treatment is widely used to overcome these limitations. It can be applied using steam, oil, vacuum, or inert gas atmospheres and has been shown to improve both physical and mechanical properties. Numerous studies have investigated the effects of heat treatment and chemical impregnation on various wood species. For instance, Doruk et al. (2010) reported that bending strength was the most affected mechanical property in ash and black pine due to heat treatment, along with noticeable darkening of wood colour. Yıldız and Can (2012) observed that increasing temperature and treatment duration in black pine, poplar, spruce, and beech resulted in higher metal loss and corrosion values, while pH decreased, indicating increased acidity. Similarly, Beram (2015) reported reductions in density, volumetric shrinkage-swelling, compressive strength, bending strength, and modulus of elasticity in Iroko wood subjected to 190 °C for 120 minutes via ThermoWood treatment. Doğan (2018) observed decreases in density, bending, and compressive strength in black pine samples impregnated with borax and boric acid after heat treatment at different temperatures. Bal (2020) showed that heat treatment under different atmospheres reduced weight loss, equilibrium moisture content, swelling, and water absorption in black pine, with nitrogen atmosphere exhibiting lower weight loss. Canyılmaz and Yaşar (2022) also reported holocellulose degradation at higher temperatures in red pine subjected to heat treatment and thermomechanical compression, highlighting heat treatment as an effective modification method for both interior and exterior applications. Görgün and Ünsal (2024) investigated the effects of thermo-treatment (thermo-vacuum or Thermovuoto®) carried out in a vacuum environment on dimensional stability. Perçin 2025 showed that reinforced heat-treated LVL samples with glass fibers increased both air dry density and compressive strength parallel to the grain.

Phosphate-based compounds, such as Alpha-X, have recently gained attention as fire retardants. Studies indicate that Alpha-X can be effective up to 1500 °C and can be applied both as an additive and as a surface treatment (Özalp, 2022). Ayhan and Özalp (2019) showed

that Alpha-X lowered the initial weight loss temperature during combustion but increased the final combustion temperature and affected bending strength in Black pine and European beech. Additionally, Özalp (2022) reported that plywood panels containing poplar and beech, bonded with adhesives modified with 10–20% Alpha-X, exhibited reduced free formaldehyde content while experiencing a slight decrease in mechanical performance.

Ayhan and Özalp (2019) investigated the fire resistance of Scots pine (*Pinus sylvestris* Lipsky) and Oriental beech (*Fagus orientalis* Lipsky) woods impregnated with the fire-retardant chemical Alpha-X. The results indicated that the initial decomposition temperature was lowest in Scots pine treated with 20% Alpha-X (198 °C) and highest in untreated Scots pine (246 °C). The initial decomposition weight loss was lowest in untreated Scots pine (88.8%) and highest in Scots pine treated with 20% Alpha-X (94.3%). The final decomposition temperature was lowest in untreated beech (483 °C) and highest in both pine and beech samples treated with 10% and 20% Alpha-X (600 °C). Overall, the results demonstrated that Alpha-X effectively enhanced the fire resistance of wood, particularly at 10% and 20% concentrations, suggesting its potential use as a protective agent against combustion in wood materials.

The Alpha-X antifire is used as a complement to the polymer and is added to the production processes of various products. It is used in thermoplastic, paint, cable, synthetic, membrane, and wood-based boards. It is effective up to 1500 °C against flammability. Alfa-x was added to the adhesive in 0, 10, and 20 wt% ratios of the total amount of the solid content (Özalp, 2022).

Collectively, these studies demonstrate that impregnation and heat treatment improve the biological strength and dimensional stability of wood, although mechanical properties often decline. Therefore, combining chemical impregnation with moderate heat treatment may optimize performance by minimizing mechanical losses while enhancing durability and stability.

The aim of this study is to investigate the influence of heat treatment on the mechanical properties of black pine (*Pinus nigra*) wood sourced from southern and northern slopes, following impregnation with Alpha-X. After impregnation, the specimens were subjected to four distinct heat treatment regimes, and their bending and compressive strengths were determined using a universal testing machine (UTM).

2 Material and Methods

2.1 Material

In this study, heartwood samples of black pine (*Pinus nigra*) were collected from the northern and southern slopes of Kiçir Village, Simav District, Kütahya Province, Türkiye, at an altitude of 1540 m. All wood materials were obtained from the Kiçir Forest Depot (Figure 1). Naturally dried trees were harvested, and the logs were sawn at a local sawmill to produce the test samples.

2.2 Samples' Preparation

The test samples were prepared at the Workshop of the Department of Woodworking Industrial Engineering, Faculty of Technology, Dumlupınar University, Simav, Türkiye, following standard dimensions. Samples for the bending strength tests measured 20 × 20 × 300 mm, while those for the compression tests measured 20 × 20 × 30 mm. For each slope exposure, 30 samples were prepared for bending strength and 30 samples for compression

tests. Additionally, to determine the density of the wood, 20 samples per exposure were prepared, totalling 60 samples with dimensions of 20 × 20 × 30 mm (Figure 2).



Figure 1. Kicir forest depot

All samples were conditioned in a climate chamber for three weeks to reach equilibrium moisture content. 10 samples per exposure were designated as controls after conditioning. The remaining 20 samples per exposure were impregnated using a 10% Alfa-X solution in water (10% Alfa-X, 90% H₂O) via the dipping method in 10-liter tanks for 4 hours at room temperature. After the impregnation process the samples were drained at room temperature for 1 hour. From each exposure, 10 impregnated samples were returned to the climate chamber, while the remaining 10 samples underwent heat treatment at 250 °C for 4 hours.



Figure 2. Density, bending strength, and compression strength test samples

2.3 Experimental Method

After heat treatment, all samples were evaluated for bending and compression strength using a Universal Testing Machine (UTM). In accordance with the TS 2474 (1976) standard, the bending strength tests were conducted with a support span of 240 mm. The load was applied at a crosshead speed of 20 mm/min at the midpoint of the specimen to ensure a fracture occurred within 0.5–1.0 minutes. The bending strength test setup is illustrated in Figure 3-a.

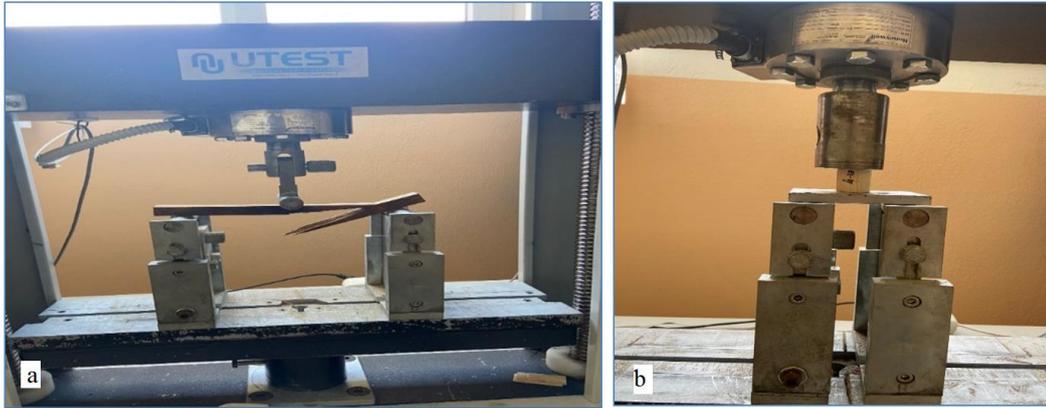


Figure 3. Bending strength (a) and compression strength test setup (b)

The bending strength (MOR) and the elasticity modulus (MOE) have been determined using the following equations:

$$MOR = \frac{3xF_{max} \times L}{2 \times b \times h^2} \quad (1)$$

In the given context, the variables are defined as follows: bending strength (N/mm^2), F_{max} represents the maximum load applied during testing, measured in N. L denotes the distance between the two supports, measured in mm. b represents the width of the test sample, also measured in mm. Lastly, h signifies the thickness of the test sample, measured in mm.

$$MOE = \frac{L_1^3 \times \Delta F}{4 \times b \times h^3 \times \Delta f} \quad (2)$$

In the given context, the variables are defined as follows: the elasticity modulus (N/mm^2), ΔF represents the load increment, measured in N. L denotes the distance between the two supports, measured in mm. Δf represents the deflection increment. b signifies the width of the test sample, measured in mm. Lastly, h signifies the thickness of the test sample, also measured in mm. In accordance with the TS 2595 (1976) standard, the compression strength test was conducted at a loading rate of 5 mm/min, ensuring that the test duration was completed within 0.5–1.0 minutes. The test continued until specimen failure, and the maximum force at the point of fracture was recorded for each sample. The compression strength test setup is illustrated in Figure 3-b. The compression strength (σ_b) of the grain test was conducted using an electromechanical universal testing machine (UTM) with a capacity of 10 kN. The following equations calculated the compression strength (σ_b) parallel to grain values.

$$\sigma_b = \frac{F_{max}}{a \times b} \quad (3)$$

In the given context, the variables are defined as follows: σ_b (N/mm^2) F_{max} represents the maximum load applied during testing, measured in N. The variable a denotes the width of the

samples, measured in mm. The variable represents the thickness of the samples, also measured in mm.

2.4 Data analyses

The results obtained after the experiments were subjected to a normality test at a 95% confidence level using the SPSS software, and subsequently compared through analysis of variance (ANOVA).

3 Results and Discussions

The physical and mechanical properties of black pine (*Pinus nigra*) wood samples were evaluated separately for north and south oriented sections. Measurements were conducted to determine the density, compressive strength, bending strength, and modulus of elasticity. For each treatment type, the arithmetic mean of ten test specimens was calculated to obtain representative values for these parameters. The summarized results of density, compressive strength, bending strength, and elasticity modulus are presented in Table 1.

Table 1. Density, compression strength, bending strength, and elasticity modulus results based on the type of process applied

Orientation	Process Applied	Statistical Value	Density (g/cm ³)	Compression Strength (N/mm ²)	Bending Strength (N/mm ²)	Elasticity Modulus (N/mm ²)
North	Unprocessed	Xmean	0.726	47.28	98.01	6531.69
		Xmin	0.685	44.96	89.07	6203.65
		Xmax	0.773	51.80	10.37	7262.64
		S	0.29	2.33	5.18	355.21
		V (%)	39.94	4.93	5.28	5.44
	Impregnation	Xmean	0.703	60.66	95.94	7970.16
		Xmin	0.659	53.32	88.51	7241.77
		Xmax	0.755	74.80	103.50	9073.50
		S	0.36	8.85	4.99	673.08
		V (%)	51.21	14.59	5.20	8.44
	Heat Treatment	Xmean	0.715	57.86	100.87	8265.37
		Xmin	0.631	57.43	88.05	6900.17
		Xmax	0.766	63.70	110.09	9999.41
		S	0.44	2.02	6.55	969.29
		V (%)	61.53	3.49	6.49	11.72
South	Unprocessed	Xmean	0.824	62.69	137.15	6039.22
		Xmin	0.756	56.41	118.42	5105.72
		Xmax	0.887	66.37	153.09	7206.59
		S	0.39	2.74	11.95	662.27
		V (%)	47.33	4.37	8.71	10.97
	Impregnation	Xmean	0.822	75.56	81.64	5962.93
		Xmin	0.759	63.70	51.72	5154.73
		Xmax	0.946	79.52	140.70	6906.53
		S	0.56	4.75	28.18	806.52
		V (%)	68.13	6.29	34.51	13.53
	Heat Treatment	Xmean	0.804	65.47	123.47	6010.46
		Xmin	0.765	54.73	111.27	5150.08
		Xmax	0.898	75.28	131.06	7998.45
		S	0.47	7.26	6.21	961.39
		V (%)	58.48	11.09	5.03	15.99

Note: S: the standard deviation; V (%): Coefficient of variation

3.1 Effect of orientation and treatment on density

According to the experimental results of the density change is given in Figure 5, and the mean and changes according to the applied process are given in Figure 6.

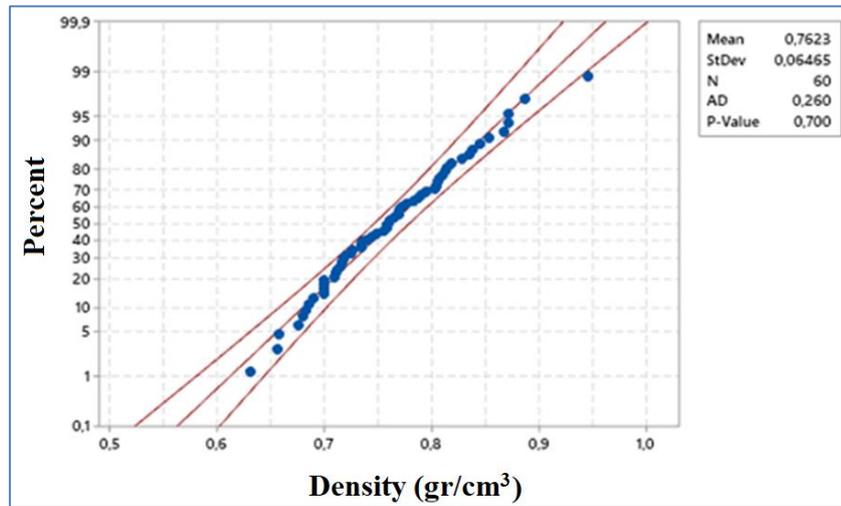


Figure 5. According to the applied process density change

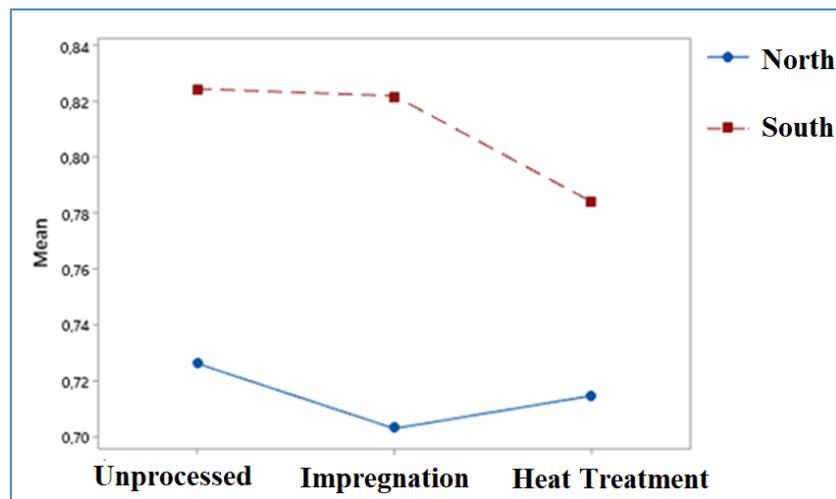


Figure 6. Density changes depending on the mean and applied process

The results presented in Table 1 indicate that the density of the black pine (*Pinus nigra*) wood obtained from the north-oriented samples was lower than that of the south-oriented ones. In other words, the density values increased proportionally toward the south direction. This phenomenon can be explained by the fact that the annual growth rings in south-oriented wood are narrower and denser than those in north-oriented wood, resulting in higher overall density. As illustrated in Figure 6, the density of untreated wood was higher compared to that of both impregnated and heat-treated specimens. This reduction in density following impregnation may be attributed to the dissolution or extraction of low-molecular-weight substances (extractives) during chemical treatment. Among the south-oriented specimens, the untreated wood exhibited the highest density value. The difference between untreated

densities of the north- and south-oriented samples was approximately 11.89%. After impregnation, a slight decrease in density (0.002) was observed in the south-oriented specimens, whereas a more pronounced reduction (0.023) occurred in the north-oriented ones. This difference is primarily associated with the initial density variation between the two orientations. Following heat treatment, the density of the south-oriented wood showed a notable decrease compared to the impregnated condition, while the opposite trend was observed in the north-oriented samples, where a remarkable increase in density was recorded after heat treatment relative to impregnation. In summary, the density of Black pine grown in the northern orientation tended to increase when heat treatment followed impregnation, whereas impregnation alone caused a reduction in density. Conversely, impregnation nor heat treatment contributed to an increase in density for the south-oriented Black pine; instead, both processes resulted in a measurable decrease. These findings suggest that the response of wood density to impregnation and thermal modification is significantly influenced by the growth orientation of the material.

3.2 Effect of orientation and treatment on compression strength

According to the experimental results of the compression strength change is given in Figure 7, and the mean and changes according to the applied process are given in Figure 8.

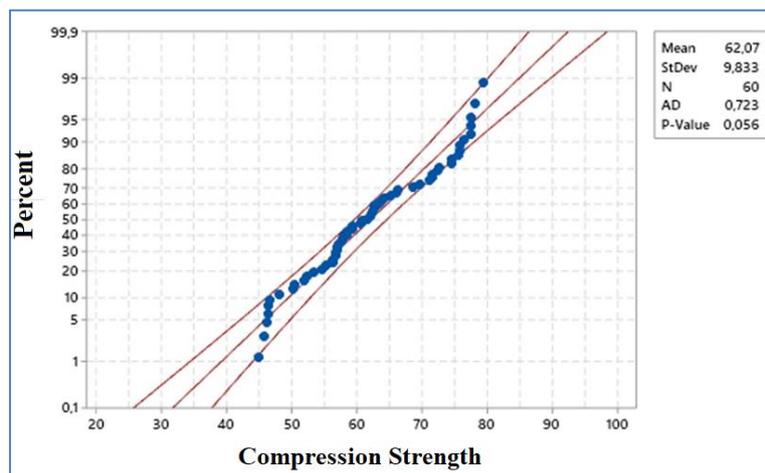


Figure 7. According to the applied process compression strength change

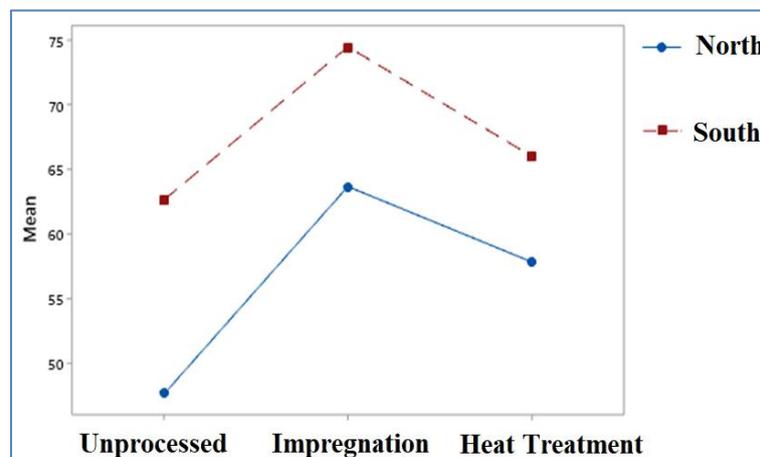


Figure 8. Compression strength changes depending on the mean and applied process

The compressive strength of the black pine (*Pinus nigra*) wood from the north-oriented samples was lower than that of the south-oriented samples, as shown in Table 1. In other words, the compressive strength increased proportionally toward the south direction. This variation can primarily be attributed to the difference in density between the two orientations. Furthermore, the results revealed that the compressive strength of untreated wood was considerably lower compared to those subjected to the Alpha-X impregnation and heat treatment processes. The highest compressive strength value was obtained after the Alpha-X treatment. According to Figure 8, the impregnation process led to the highest compressive strength in both the north- and south-oriented specimens. However, when heat treatment was applied following impregnation, a notable decrease in compressive strength was observed in both orientations. The difference between the untreated compressive strength values of the south- and north-oriented samples was approximately 24.58%. This difference may be associated with the higher lignin content in the south-oriented wood, which enhances its compressive load-bearing capacity. After impregnation, a significant increase in compressive strength was recorded in both orientations. Conversely, subsequent heat treatment caused a noticeable reduction in compressive strength compared to the impregnated state. Overall, the results indicate that impregnation treatment improves the compressive strength of Black pine grown in both north and south orientations, whereas the combination of impregnation and heat treatment tends to reduce it. These observations are consistent with the density variations discussed earlier, confirming the strong correlation between density and compressive behavior in thermally and chemically modified Black pine.

3.3 Effect of orientation and treatment on bending strength

According to the experimental results of the bending strength change is given in Figure 9, and the mean and changes according to the applied process are given in Figure 10. As illustrated in Figure 9, the bending strength data exhibited a normal distribution at a 95% confidence level. According to Table 1, the bending strength values of the black pine (*Pinus nigra*) obtained from the north-oriented specimens was lower than those from the south-oriented specimens. In other words, the bending strength increased proportionally toward the south direction. This difference may be attributed to the higher cellulose content observed in the south-oriented wood, which enhances the tensile and bending performance of the fibers.

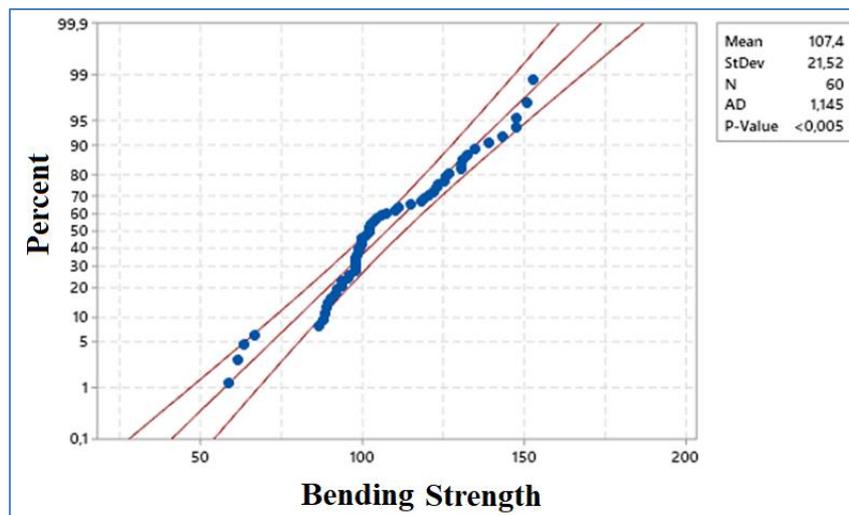


Figure 9. According to the applied process bending strength change

As shown in Figure 10, the bending strength of the chemically untreated but heat-treated south-oriented wood was higher than that of the north-oriented samples. In contrast, the lowest bending strength value was recorded in the north-oriented impregnated specimens. The highest bending strength was obtained in the untreated Black pine samples from the south orientation. The difference between the untreated bending strengths of the south- and north-oriented woods was approximately 28.5%. When impregnation treatment was applied, a slight decrease (2.07 MPa) in bending strength was observed in the north-oriented samples, while a more pronounced reduction (55.51 MPa) occurred in the south-oriented samples. Following heat treatment, however, the bending strength of the south-oriented wood increased notably compared to that of the impregnated condition. Overall, it was determined that impregnation treatment decreased the bending strength of the black pine grown in the southern orientation, whereas subsequent heat treatment of impregnated wood resulted in a noticeable improvement in bending strength. In summary, both impregnation and heat treatment did not enhance the bending performance of Black pine grown on the southern slope; instead, they tended to reduce it. These findings suggest that the effect of chemical and thermal modification on bending behaviour is highly dependent on the growth orientation of the wood material.

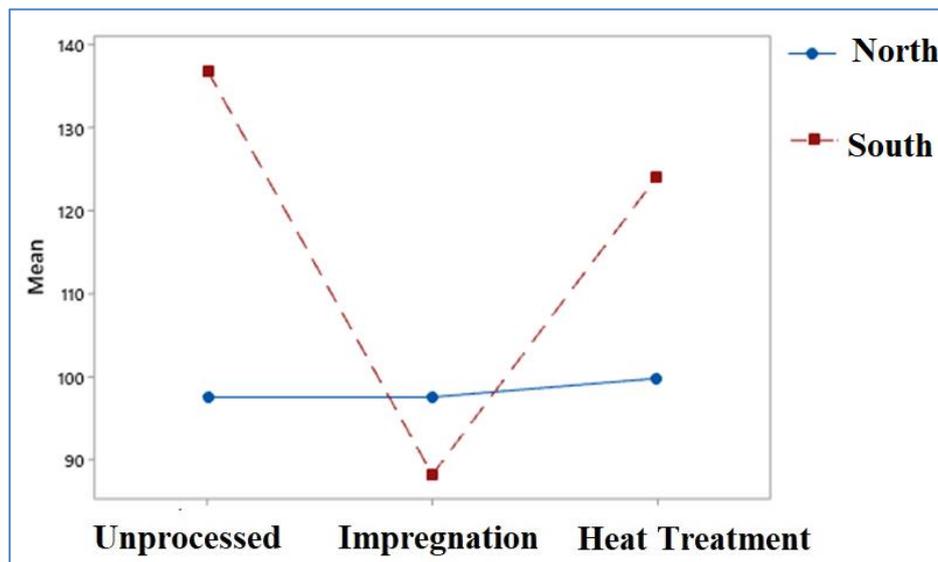


Figure 10. Bending strength changes depending on the mean and applied process

3.4 Effect of orientation and treatment on elasticity modulus

The change in bending strength according to the experimental results is given in Figure 1, and the mean and changes according to the applied process are given in Figure 12. It was observed that the modulus of elasticity (MOE) data exhibited a normal distribution at the 95% confidence level (Fig. 11). As presented in Table 1, the bending strength of black pine (*Pinus nigra*) wood obtained from the northern orientation was higher than that from the southern orientation, indicating that the MOE increased proportionally toward the north direction. Examination of Fig. 12 further revealed that the MOE values of untreated, impregnated, and heat-treated specimens from the northern direction were consistently higher than those from the southern direction. The lowest MOE value was recorded in impregnated samples from the southern direction, whereas the highest MOE value was observed in heat-treated samples from the northern direction. A comparison between untreated specimens showed approximately a 7.54% higher MOE in the north-oriented samples than in the south-oriented

ones. Following impregnation, a slight reduction (by 76.29 units) in MOE was observed in the southern direction, while a pronounced increase (by 1438.47 units) occurred in the northern direction. After heat treatment, the MOE in the north direction exhibited a substantial increase compared with the impregnated condition. Overall, the findings indicate that both impregnation and heat treatment enhance the elastic performance of Scots pine grown under northern exposure conditions. These treatments did not cause deterioration in stiffness; on the contrary, they contributed to a considerable improvement in the modulus of elasticity of wood originating from the northern slopes.

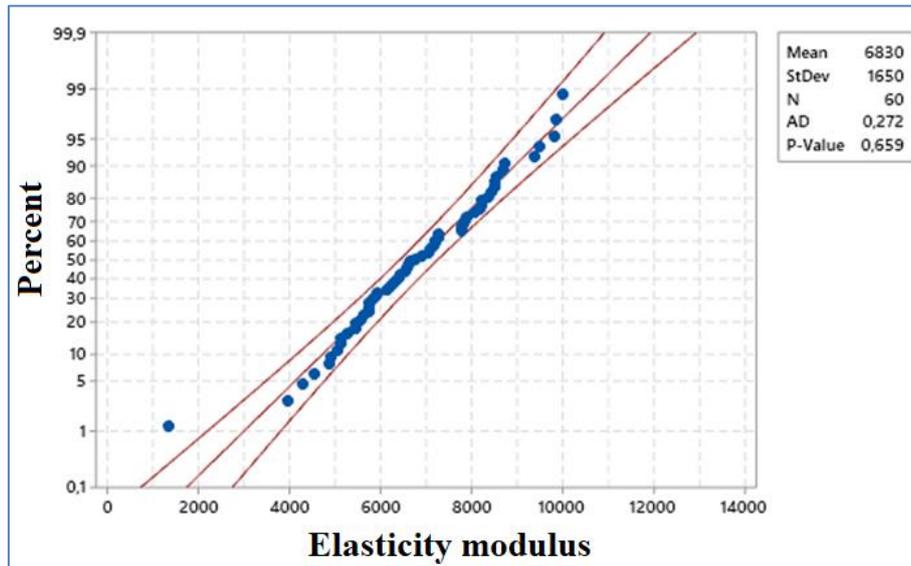


Figure 11. According to the applied process elasticity modulus change

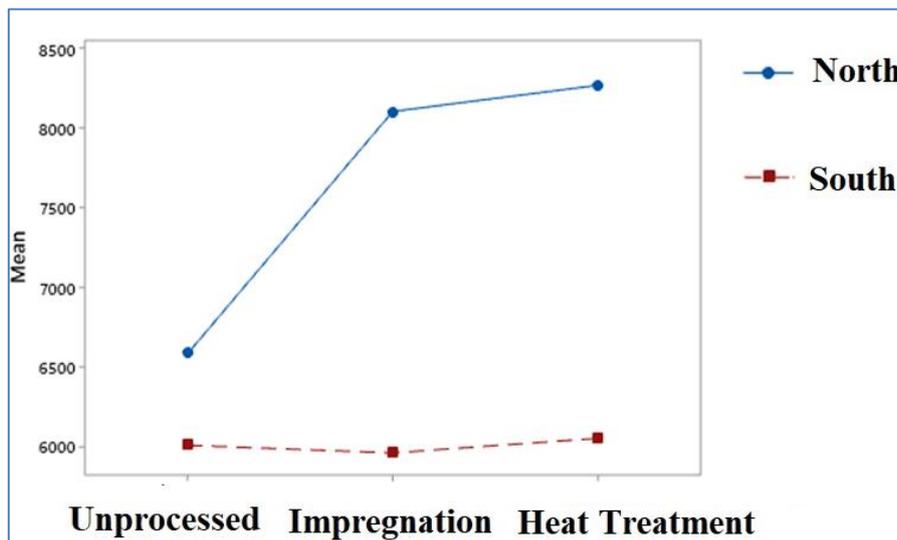


Figure 12. Elasticity modulus changes depending on the mean and applied process

4 Conclusion

In this study, titled “Investigation of the Effects of Growth Orientation, Thermal Treatment, and Alpha-X Chemical on black pine (*Pinus nigra*)”, the influence of orientation, thermal modification, and chemical impregnation on the physical and mechanical properties of the material was comprehensively examined.

- The results demonstrated that thermal treatment caused an approximate 10% decrease in the density of wood samples obtained from both the north and south orientations. While an increase in compressive strength was observed, mainly attributed to the lignin content, a noticeable reduction in bending strength occurred, which is influenced by cellulose.
- The thermal degradation sequence of the main wood polymers can explain this behaviour. During thermal treatment, cellulose degradation occurs earlier than that of lignin, leading to a decrease in bending strength. The observed increase in compressive strength, on the other hand, can be attributed to the fact that lignin had not yet reached its degradation temperature. Lignin begins to thermally decompose at relatively higher temperatures, typically between 350°C and 400°C, whereas cellulose decomposes at lower temperature ranges.
- In the part of the study focusing on the effect of the Alpha-X chemical, two experimental groups were prepared: one group of samples was impregnated with the Alpha-X chemical before thermal treatment, while the other group was thermally treated without chemical application. The findings revealed that the use of the Alpha-X chemical before thermal treatment negatively affected both the bending and compressive strength values of Black pine wood. This indicates that chemical impregnation before heat exposure may weaken the internal bonding structure within the wood cell wall.
- It is also noteworthy that all wood samples used in this study were obtained from trees harvested outside the vegetation (active growth) period. For future research, it is recommended that wood samples be collected from trees during the active vegetation period, allowing for further investigation into how seasonal growth conditions interact with chemical and thermal treatments. Such studies would provide a deeper understanding of how combined modification processes influence the physical, mechanical, and structural behaviour of the black pine wood.

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Author Contributions

Kemal Güler: Conceptualization, Data curation, Formal Analysis, Investigation, Methodology, Project administration, Resources, Supervision, Validation, Visualization, Writing- original draft, Writing-review & editing, **Murat Özalp** Conceptualization, Investigation, Methodology, Resources, Validation, Visualization, Writing-original draft. **Abdurrahman Karaman:** Formal Analysis, Writing-original draft,

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Conflict of Interest Statement

The authors declare no conflict of interest.

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