Effects of Thermal Bio-Oil Immersion Process on Antilarvicidal, Anisotropic, Mechanical, and Thermal Properties of Turkish Pine Wood

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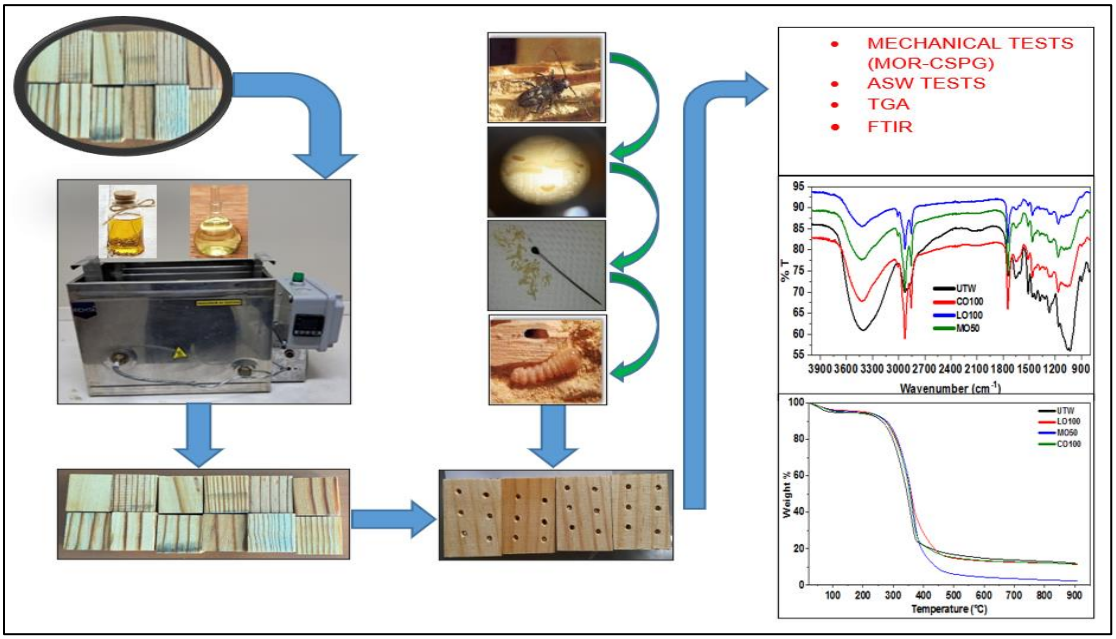
GRAPHICAL ABSTRACT



Effects of Thermal Bio-Oil Immersion Process on Antilarvicidal, Anisotropic, Mechanical, and Thermal Properties of Turkish Pine Wood

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Engineering properties of Turkish pine wood were evaluated to determine effects of a bio-oil thermal immersion process. Wood samples were impregnated with linseed oil, castor oil, and a mix of both oils. The investigation encompassed antilarvicidal activity, bending and compressive strength, anisotropic swelling, thermogravimetric analysis (TGA), and Fourier transform infrared (FTIR) spectroscopy. Linseed and castor oils demonstrated potent antilarvicidal properties, achieving 100% and 92% mortality rates, respectively. The mixed oil exhibited 79.2% mortality. The thermal immersion treatment significantly enhanced the bending and compressive strength, and linseed oil yielded the highest improvements at 28.7% and 77.0%, respectively. Anisotropic swelling notably decreased. The linseed oil showed the greatest reduction (7.1%) and the mixed oil the least (2.7%). TGA results indicated improved thermal stability, with weight losses of 1.7%, 2.0%, and 2.2% for linseed oil, castor oil, and mixed oil, respectively. FTIR analysis revealed new peak regions ranging from 1159 to 3398 cm^{-1} and suggested significant interactions between the bio-oils and the cell wall components, particularly for linseed oil. In conclusion, the thermal bio-oil immersion treatment effectively improved the selected properties of this wood.

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Keywords: Wood; Bio-oil; Thermal immersion; Degradation; Mechanical; Anisotropy; TGA; FTIR

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INTRODUCTION

The demand for wood materials is increasing due to their many superior properties. However, when such natural materials are used without protective treatment, their biological structure can degrade quickly under the influence of various harmful factors. This degradation can lead to some problems with the technological properties of wood during its use. One of these problems is the degradation of wood by insects. Wood is destroyed by adult insect larvae. This destruction manifests itself in the form of tunnels opened in the wood (Akkuş *et al.* 2022). Generally, wood is destroyed by insects as they acquire shelter, breeding grounds, or food supply. The most destruction occurs in wood at the place of end-use (Suprianto *et al.* 2023). Such damage can result in losses of considerable time, labor, and money. Wood produced from the sapwood part of tree trunks is heavily damaged by insects/insect larvae (Martín and López 2023). This is because in sapwood, the nutrients, moisture content and pH required for the growth of such organisms are more favorable, but there are few or no substances that are toxic to these organisms.

Moreover, this can limit many technological properties of wood (Var *et al*. 2021). Properties such as biological resistance, volumetric and dimensional change, and strength properties are closely related to the amount of moisture contained in the wood (Bozkurt and Erdin 1997).

Hydrophobic oils, in combination with coloring agents, increase the resistance of wood to harmful ultraviolet (UV) radiation, while their combination with anti-scuffing agents prevents fiber lifting on the wood surface (Tomak 2011). Such oily substances can protect wood against biotic pests by preventing the ingress of moisture into the wood, where fungi and microorganisms can thrive (Williams and Feist 1999). These oily substances, which can usually form weak Van der Waals bonds with cell wall substances in wood, are also applied by hot-cold (thermal bio-oil) immersion in an open boiler (Podgorski *et al.* 2008). In such thermal treatments, the woods are immersed in oil with a temperature ranging from 110 to 210 °C for a certain period of time and then again immersed in oil with a temperature ranging from 10 to 90 °C for a shorter period of time. Meanwhile, the pressure difference created by the temperature change allows the water-repellent oils to penetrate deeply into the wood (Berard *et al.* 2006). With this thermal treatment, the moisture needed for the growth of fungi and microorganisms in wood is reduced and nutrients are modified (Bozkurt and Erdin 1997). In this way, biological resistance is increased by preventing the destruction of wood by such biotic elements in environments with intense moisture/water vapor condensation (Tomak 2011). In addition, these oils provide a number of benefits, such as greater producibility, economy, and harmlessness for the natural environment (Temiz *et al*. 2008).

Hylotrupes bajulus (L.) is a significant wood pest worldwide, and primarily damages coniferous trees, especially pine species (Sen *et al*. 2017). According to Allison *et al*. (2004), volatile substances released by host plants attract *H. bajulus*, and the insects infest dry pine lumber and create tunnels in the wood, where their larvae grow, and cause substantial damage. This damage often compromises the structural integrity of wood, and results in serious financial losses in the processing and replacement of wood. Civelek and Çolak (2008) reported that adult *H. bajulus* typically enter the wood and lay their eggs in wounds on the wood's surface, and that the larvae hatching from these eggs damage the wood, but that impregnating the wood can effectively protect it from such infestations. A study by Vani *et al*. (2022) demonstrated that bioprotective agents were effective in preventing the growth and survival of *H. bajulus* larvae in treated wood. Furthermore, some studies investigating the efficacy of various bio-oils against wood-damaging organisms found that sapwood samples treated with bio-oils showed resistance to *H. bajulus* larvae (Barbero-López *et al*. 2021), and that the applications of bio-oil seriously reduced the survival rate of larvae (Urrutia *et al*. 2022).

Due to environmental and economic issues, the use of some vegetable oils as alternative hydrophobic impregnating agents has become an important issue. Among these, linseed oil and castor oil are among the well-known plant-based oils that are widely used as environmentally friendly impregnating agents. Several studies have investigated the effectiveness of plant-based hydrophobic oils (Temiz *et al.* 2010; Tomak 2012; Şimşek 2024; Zhang *et al*. 2024). Nevertheless, research on the engineering properties of wood materials treated with thermal bio-oil (hot-cold) immersion remains limited.

When thermal bio-oil immersion is applied to wood elements produced from easily impregnated wood species, the anisotropic behavior of these materials can be reduced, while their mechanical strengths, anti-termite efficiency and thermal stability can be increased. As a result, the improved biological strength and engineering properties can

minimize the problems related to the use of the material. Wood with these qualities can be used for a longer period than normal wood. Therefore, the biological strength and engineering properties of wood produced from Turkish pine stem wood can also be improved.

This paper investigated the effects of two different vegetable oils and a blend applied by the thermal bio-oil immersion technique on the biological strength and engineering properties of Turkish pine sapwood. The extent to which such an application affects selected performance criteria of wood materials produced from Turkish pine sapwood was the subject of this study. For this purpose, an experimental study was planned based on the treatment of small wood samples with different vegetable oils using the thermal bio-oil immersion method. In this context, several experimental investigations were carried out under laboratory conditions.

EXPERIMENTAL

Wood Specimens

Three different Turkish pine (*Pinus brutia* Ten.) logs were randomly selected from a sawmill (Özdamar Kerestecilik, Isparta, Türkiye). Small wood samples were prepared from the sapwood of the selected logs with the following dimensions for different tests: 20 mm radial (R) × 20 mm tangential (T) × 30 mm longitudinal (L) for antilarvicidal tests, 20 mm (R) × 20 mm (T) × 30 mm (L) for anisotropic tests, 20 mm (R) × 20 mm (T) × 300 mm (L) for modulus of rupture tests, and 20 mm (R) × 20 mm (T) × 100 mm (L) for compression strength parallel to grain tests. The samples were conditioned to dry air humidity in the laboratory before thermal bio-oil immersion, dried to oven dry weight at 103 ± 2 °C in an oven, cooled to normal room temperature in a desiccator (ISO 13061-1 2014), and labelled as test and control samples. At least ten wood specimens were used for mechanical strength (MOR, CSPG) and anisotropic swelling (ASW) tests, at least four specimens for the antilarvicidal activity (ALA) test, and at least three specimens each for TGA and FTIR tests. All sample sets were transferred and stored in ziplock plastic bags.

Plant Oils

In the study, three types of process oils were used as plant-based oil: two pure oils (castor oil, linseed oil) and one mixed oil. Castor oil (*Ricinus communis* L.) and linseed oil (*Oleum linii*) were obtained from an herbalist (Özkan Şifalı Ürünleri, Isparta, Türkiye). The mixed oil was obtained by mixing 50% castor oil + 50% linseed oil and prepared according to volume fraction. Castor oil (CO100), linseed oil (LO100), and mixed oil (MO50) were used at 100%, 100% and 50% purity, respectively. All treatment oils were divided into two sets of 5 liters each for the thermal bio-oil immersion process. The first set was labeled for hot bio-oil immersion treatment, while the second set was labeled for cold bio-oil immersion treatment. Each bio-oil immersion set was transferred and stored in a metal container with a lid.

Thermal Bio-Oil Immersion Process

Thermal bio-oil immersion was carried out as a two-stage process (hot oil immersion, cold oil immersion) according to TS 345 (2012). The dimensions and weights of the wood samples before treatment were measured and recorded. Firstly, hot oil immersion was carried out. In this step, the samples were placed in an open tank with oil

at room temperature (23 ± 2 °C) and the temperature was increased at a rate of 5 °C·min-1. When the target temperature (110 ± 2 °C) was reached, the samples were kept in this hot oil for 6 h and then removed. This was followed by immersion in cold oil. At this stage, the samples were removed from the hot oil and placed in a second open tank containing room temperature (cold) oil. The samples were kept there for 2 h and then removed from the cold oil. The gross dimensions and weights of the samples were then measured after the excess oil residue had been removed by applying 0.1 atm pressure. These measurements were recorded as post-treatment values. Finally, the samples were kept in an oven at 60 ± 2 °C for 24 h until constant weight, removed from the oven, cooled again to normal room temperature, and their net weights and dimensions were measured. These measurements were recorded as oven-dry data after treatment. All wood samples impregnated in this way were placed back into ziplock plastic bags and the selected test and analysis procedures were retained. The test group wood samples were not placed directly in hot oil to avoid cracks in the cross-sections. Control group wood samples were not subjected to thermal bio-oil immersion.

Antilarvicidal Activity (ALA) Test

House longhorn beetle [*Hylotrupes (H) bajulus* L.] larvae were used for the ALA test. Insect (larvae) tests were carried out in the Forest Biology and Wood Protection Technology laboratory at Düzce University. Insect tests were carried out according to the principles specified in EN 47 (2016). Four replicates were used for each process oil group and the control group (a total of 16 samples). Male and female adult *H. bajulus* beetles were mated. Experimental larvae were produced from the insect eggs obtained as a result of mating. This process was carried out under favorable climatic conditions. The newly hatched beetle larvae were placed in the wood samples. A total of 6 holes were drilled on the radial surfaces of the samples in two rows, three for each row. The beetle larvae were carefully placed in these holes with their heads downwards. The wood samples with larvae were kept in an air conditioning cabinet at 270 °C and 70% humidity during the experiment. Four weeks after the start of the experiment, one of the impregnated samples was opened, and the viability of the larvae was observed. Because live larvae were observed, the experiment was completed in 12 weeks. The experiment was considered successful when the proportion of live larvae in the control samples was above 70%.

Mechanical Tests

Modulus of rupture (MOR) and compression strength parallel to grain (CSPG) were tested to determine mechanical strength properties of the wood samples. MOR and CSPG were determined in accordance with ISO 13061-3 (2014) and ISO 13061-17 (2017). Ten replicates for each process oil group and the control group (a total of 80 samples) were used. Mechanical tests were performed with a Marestek (İstanbul, Türkiye) universal testing machine (4000 kPa, piston head speed 6 $mm \cdot min^{-1}$).

Anisotropic Swelling (ASW) Tests

Anisotropic swelling determines the in-use stability of a wood material. This parameter expresses the ratio of swelling in the tangential direction to swelling in the radial direction (Bozkurt *et al.* 1993). ASW was calculated according to Bozkurt *et al.* (1993). Ten replicates were used for each process oil group and the control group (a total of 40 samples). In the calculation, ASW in the longitudinal direction was not taken into account.

Thermogravimetric (TGA) Analysis

TGA analyses were performed using a Seiko SII TG/DTA 7200 model (Seiko, Chiba, Japan) thermogravimetric analyzer. This method is a technique that measures the weight change as a function of the temperature change in nitrogen at 0.1 mg resolution. For analyses, one wood sample was selected from each process oil group and the control group. The initial (original) weight of each sample was measured at approximately 10 mg in the TGA analysis. These samples were heated from 25 to 900 °C in a platinum pan at a rate of 10 ºC·min^{-1} and analyzed. During the test, the variation of sample mass with temperature and time was measured under controlled atmospheric conditions.

Fourier Transform İnfrared (FTIR) Analysis

FTIR analyses were performed with a Perkin Elmer Frontier FTIR spectrometer instrument. The chemical compositions of the wood samples were characterized with the FTIR device. FTIR spectra were recorded on the spectrometer instrument.

Statistical Analysis

One-way analysis of variance (ANOVA) and Duncan's multiple range test were used. ANOVA showed the statistical significance level of the effect of the thermal-bio-oil immersion process on tested properties of wood. The Duncan test was applied to compare the means values, and to allocate statistically significant differences between the means.

RESULTS AND DISCUSSION

ALA (Antilarvicidal Activity)

The insect test results of the wood samples are listed in Table 1.

Table 1. Insect Test Results against Larvae of *H. bajulus* in Test and Control

Name of Processing Oil	Wood Sample No	Number of Larvae			Rate of Larvae Mortality (%)
		Before Experiment	Alive After Experiment	Dead After Experiment	
LO100	1	6	0	6	100
	2	6	0	6	100
	3	6	0	6	100
	4	6	0	6	100
CO100	1	6	0	6	100
	2	6	1	5	83
	3	6	0	6	100
	4	6	1	5	83
MO50	1	6	2	4	66.7
	2	6	1	5	83
	3	6	2	4	67
	4	6	0	6	100
UTW (Control)	1	6	5	1	16.7
	2	6	5	1	16.7
	3	6	6	0	0
	4	6	6	0	0
LO100: Linseed oil (100%). CO100: Castor oil (100%). MO50: 50% Castor oil + 50% Linseed oil. UTW: Untreatedwood.					

According to these results, the average larval mortality rate of the control (untreatedwood, UTW) samples was approximately 8.4%. This result indicates that more than 92% of the larvae in the UTW survived and the larval test was valid. In 2 of the 4 UTW samples subjected to the larval test, only one newly hatched larva from each of the 6 eggs died. This means that almost all of the larvae placed in the UTW were alive and that these larvae were destroying the wood.

Figure 1 shows the larval mortality rates in impregnated and unimpregnated samples. Dead larvae were found in the majority of samples impregnated with LO100 and CO100. The larval mortality rate was 100% for LO100 and 92% for CO100. Thus, both treatment oils provided effective protection against *H. bajulus* larvae. When used as a mixture (MO50), CO100 and LO100 gave lower larval mortality and this MO50 mixture did not have a synergistic effect on larval mortality. Table 1 also shows that the survival rate of *H. bajulus* larvae was higher for MO50 than for the other two oils. For MO50, the larval mortality rate was 79.2%. These findings can be attributed to the fact that MO50 retention in the samples was too low to prevent larval development. Consequently, the MO50 treatment may not have been as toxic to *H. bajulus* larvae as the other treatments. It is clear that due to this low MO50 retention, the number of surviving larvae increased noticeably. This can be related to the improved biological resistance due to the increased amount of LO100 and CO100 in the wood.

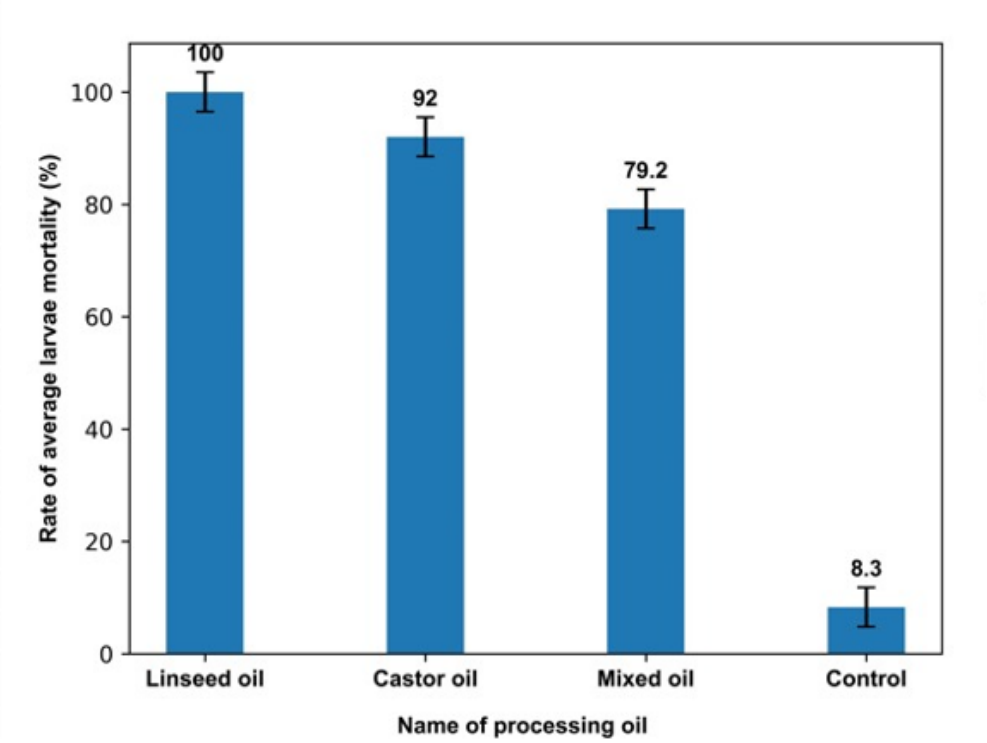

Fig. 1. Rates of average mortality of *H. bajulus* larvae in the treated wood and the control

The effectiveness of LO100 and CO100 treatments against *H. bajulus* larvae may be due to the fact that, at the end of the thermal bio-oil immersion, these two treatments reduce the nutrient and moisture content of the internal structure of the wood below the amount necessary for the survival of the insect larvae. This can be explained by the inhibition of moisture mobility in wood samples whose cell lumens and intercellular spaces are filled with oil (Rowell and Banks 1985).

At the end of the thermal LO100, CO100, and MO50 immersion treatments, all treatment oils formed a dry layer on the wood samples that did not feel greasy. This process may also have created an oily insulating layer in the wood, which reduced the volume of void space (air, oxygen) and moisture content. It has been noted that the oxygen content of the air within the spaces in the wood is in the range of 10 to 20%, keeping in mind that 20% oxygen is the usual condition of ambient air; and the decrease in void volume leads to a reduction in the oxygen content within of wood. Moreover, it is stated that the oxygen content of wood polymers (cellulose, hemicellulose, and lignin), as well as oxygen in wood extractives, is not included in the "10 to 20%". In impregnation processes, it has been reported that the retention of the impregnant in the wood reduces the void volume and the oxygen level, and consequently, prevents the development of these organisms (Bozkurt *et al.* 1993). This implies a reduction of oxygen within the wood, lubrication of surfaces and nutrients, and a change in pH. Bal (2015) reported that bio-oil treatment creates an oily layer on wood, which prevents contact with air and reduces oxygen penetration. Abnisa *et al*. (2013) found that the pH of bio-oils ranges from 2.1 to 4.6, and that bio-oils with low pH may degrade organics such as cellulose, hemicellulose, and proteins due to their high acidity. Bozkurt *et al.* (1993) stated that the optimal pH range for the development of harmful organisms in wood is between 4 and 6. These parameters are known to play a crucial role in the colonization, reproduction, and survival of organisms in wood. Such a treatment that restricts these parameters may have prevented the development of *H. bajulus* larvae, also leading to increased mortality. As a result, the improved biological resistance to *H. bajulus* larvae could have improved the ALA performance of Turkish pine wood. However, it is important to note that such oils, which are physically present within the wood and form a mechanical barrier, may be washed away over time. When such treatment oils are combined with fungicides or insecticides, the washout of biocides may also be reduced due to a decrease in the wood's water uptake rate. Therefore, the ALA performance of Turkish pine wood may be more permanent with synergistic effects.

Some researchers (Jebrane *et al.* 2017; Hosseinpourpia *et al.* 2020) reported that the oily layer formed on the surface of wood samples treated with linseed, canola, hemp, and tall oils makes colonization by biotic pests more difficult compared to untreated samples. Ghani (2021) found that sugar groups in the chemical structure of wood are more removed in wood impregnated with vegetable oils than in unimpregnated wood, and this sugar removal reduces the attack of biotic pests. Smith *et al.* (2003) reported that wood samples impregnated with canola oil exhibited better biological resistance to insect larvae. Nunes *et al.* (2006) suggested that this resistance may be related to a reduction in the wood's moisture content. It has been reported that wood samples impregnated with linseed oil provide good protection against biotic degradation, which is likely due to the formation of a film layer on the wood surface (Ahmed *et al.* 2008). Similarly, Temiz *et al.* (2013) found that bio-oil treatment significantly reduced the survival rate of *H. bajulus* larvae. In another study, it was established sapwood samples treated with different bio-oils showed resistance to *H. bajulus* larvae (Temiz *et al.* 2010). Thus, this study is consistent with the literature.

Mechanical Properties

The average MOR and CSGP results of the wood samples are given in Table 2. Figure 2 shows the MOR and CSGP change percentages of all samples. Percentages of change were calculated with respect to the control (UTW) samples. According to the results in Table 2, the MOR and CSPG values were 108.4 and 61.0 N/mm² for LO100, 103.8 and

59.7 N/mm² for CO100, 98.5 and 42.4 N/mm² for MO50, and 84.3 and 34.6 N/mm² for UTW samples, respectively. These results show that the best mechanical strength values were obtained for LO100 followed by CO100 and MO50. This means that for all treatment oils, the mechanical strength property was higher in impregnated samples than in unimpregnated samples. According to the findings in Fig. 2, thermal bio-oil immersion treatment improved the MOR of Turkish pine wood by 17.4 to 28.7% and CSPG by 22.9 to 77.0% more than normal wood. In this sense, the best MOR and CSPG results were realized in LO100 treatment by 28.7% and 77.0%, respectively. Moreover, LO100 treatment provided 22.2% and 65.3% better MOR recovery than CO100 and MO50, respectively. However, for the same treatment, CSPG recovery was 5.9% higher for CO100 and 236.4% higher for MO50. This means that LO100, when applied alone in the thermal bio-oil immersion process, significantly improved the MOR and CSPG of Turkish pine wood. It can be said that the strength properties were significantly improved when LO100 was used individually, but less so when mixed with CO100 at 50%. For MO50, there was a significant decrease in the resistance properties of the samples compared to LO100 and CO100, but a significant increase compared to UTW.

The improved strength values of the impregnated samples may be related to the weight increase due to the increased oil penetration in the samples at the end of the thermal bio-oil immersion. This weight increase may reduce the void volume in the wood and increase the amount of oil absorbed per unit volume. As a result, the density or specific mass of the samples increases. Thus, the thermal bio-oil immersion process increases the MOR and CSPG values of the samples, *i.e.*, it improves mechanical strength performance. The density increase can vary according to the density and viscosity values of the oils used in the impregnation process, and the density increases with the amount of absorption of oils with different density and viscosity (Var *et al.* 2021). Similar findings were reported in studies in which wood was treated with various vegetable oils, and density was reported to improve with increasing weight (Bazyar *et al.* 2010; Tomak 2011). Table 2 shows that after thermal bio-oil immersion, the MOR and CSPG values of the impregnated samples increased compared to the UTW samples. MOR value increased from 98.5 to 108.4 N/mm² and CSPG value increased from 34.6 to 61.0 N/mm². Therefore, the mechanical strength properties of impregnated samples improved between 17.5 and 28.7% for MOR and 22.9 to 77.0% for CSPG compared to UTW.

Gökmen (2017) reported that thermal tall oil treatment increased the mechanical strength values of wood. Van Eckeveld *et al.* (2011) revealed that increased density of linseed oil, wood oil, coconut oil and tall oil improved mechanical performance due to the fact that they form barriers and fill the gaps in the wood. Li *et al.* (2022) reported that linseed oil, tall oil, and canola oil had a good effect on the mechanical resistance of wood. Olsson *et al.* (2001) stated that there was a slight improvement in the mechanical properties of wood treated with linseed oil; this may be due to the hydrolytic resistance effect caused by the oil settling in the cell lumens. Lee *et al.* (2018) reported that the mechanical strength of wood slightly improved during thermal treatment with certain vegetable oils. They explained that the oxygen-free atmosphere created in the wood helped to enhance its mechanical properties, while the increased oil absorption further contributed to this improvement. Tomak *et al.* (2011) found that the oil-heat-treated samples exhibited higher CSPG compared to control samples due to the high oil retention. They emphasized that, at the end of the treatment, the oil filled the cell lumens and thickened the cell walls, thereby increasing the wood's strength. Similarly, Cheng *et al.* (2014) reported that after oil heat treatment, the fibers became thicker, and the wood's compressive strength increased due

to the high oil absorption. Bak and Nemeth (2012) concluded that the compressive strength increased by 15 to 25% for poplar wood and 5 to 15% for *Robinia* wood when treated with thermal oils. Boonstra *et al.* (2007) attributed this improvement to the increased densification of lignin during the treatment process. Megnis *et al.* (2002) suggested that the hydraulic effects of oil in the cell lumens may contribute to a slight increase in bending strength. Therefore, this study is consistent with the literature.

Table 2. MOR and CSPG Results of the Treated Wood and the Control

Name of Processing Oil	MOR (N/mm²)	CSGP (N/mm²)
LO100	108.39^{bc} (6.995)*	60.99^{c} (3.680)*
CO100	103.82^{b} (5.083)	59.66^{c} (3.231)
MO50	98.49^{b} (6.758)	42.35^{b} (2.617)
UTW (Control)	84.30^{a} (4.795)	34.64^{a} (2.550)

LO100: Linseed oil (100%). CO100: Castor oil (100%). MO50: 50% Castor oil + 50% Linseed oil. UTW: Untreatedwood. *: The difference between the numbers indicated by different letters within the same column is significant ($p \leq 0,05$; 95%). Numbers in parentheses are standard deviations.

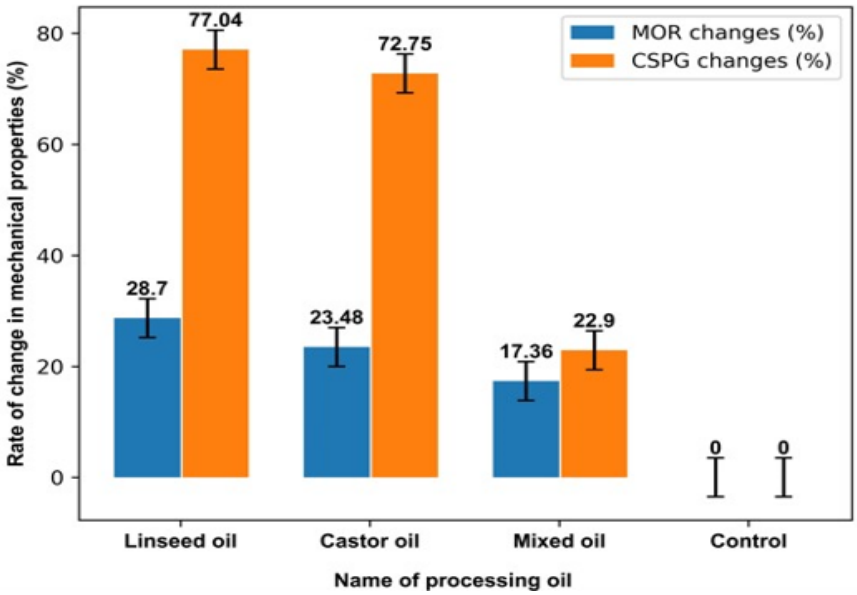

Fig. 2. MOR and CSPG changes of the treated wood and the control

Anisotropic Swelling Behavior

The ASW values of all samples are listed in Table 3. Figure 3 shows the ASW change percentages of the wood samples. Percentages of change were calculated according to UTW. The lowest ASW value was 1.12 for LO100. This was followed by CO100 with 1.14 and MO50 with 1.17. In terms of the effect on swelling anisotropy, there was no significant difference between the treatment oils. However, there was a difference between all three treatment oils and UTW in terms of swelling anisotropy. This difference, which was significant between UTW and LO100, was found to be insignificant between UTW and CO100 and MO50.

The ASW behavior of the treated samples decreased compared to the UTW samples, and this decrease was in the range 2.7 to 7.1%. In this sense, the highest ASW

reduction was 7.1% for LO100, while the lowest ASW reduction was 2.7% for MO50. Furthermore, the LO100 treatment significantly reduced the ASW behavior compared to the CO100 and MO50 treatments. The reduction was 26.2% higher for CO100 and 159.3% higher for MO50. Although CO100 and MO50 treatments reduced the ASW value at similar levels, this reduction was 105.5% more in CO100 treatment than MO50. The ASW values of the impregnated samples decreased compared to the UTW. According to this result, the treatment oils used in the study improved the ASW of Turkish pine wood.

The reduced swelling anisotropy may be related to the formation of an insulating layer against water uptake in the cell walls, whose inner and outer surfaces are coated with oil due to bio-oil retention. At the end of the impregnation process, ASW values may decrease due to this insulating layer formed in the samples. In other words, the decrease in the swelling anisotropy of the samples in Table 3 from 1.17 to 1.12 may be due to this water repellent layer formed on the cell walls. In other words, the ASW behavior of the treated samples in Fig. 3 decreased between 2.73 to 7.08% compared to the UTW. This result suggests that the swelling anisotropy is reduced due to the increased water-repellent insulating layer or decreased moisture diffusion in the wood. This can be explained by the formation of a layer in wood with a wood-water contact angle below 90°, which acts as a mechanical barrier and reduces water uptake (Temiz *et al.* 2008; Panov *et al.* 2010).

According to Bozkurt *et al.* (1993), the ASW of wood is closely related to the in-use stability of that material. Good in-use stability of wood depends on the decreasing swelling anisotropy of the material. The smaller (less) the ratio of swelling in the tangential direction to swelling in the radial direction, the better (less) the ASW behavior. Some researchers (Bozkurt and Erdin 1997; As *et al.* 2001) stated that ASW values range between 1.45 and 1.63. Bal (2015) reported that at the end of the thermal process in hot oil, the swelling anisotropy of the wood decreased, the contact with oxygen the wood was prevented, and this reduced swelling resulted in less stress on the wood under different climatic conditions. Dubey *et al*. (2011) described that there were no significant differences in swelling among the treatment oils, but that oil absorption did reduce swelling. In this regard, it was established that impregnated wood exhibited better performance (Okon *et al*. 2017; Lee *et al*. 2018). This means that the findings in Table 3 agree with the literature.

Table 3. ASW Behavior Results of the Treated Wood and the Control

Name of Processing Oil	ASW Behavior
LO100	1.12^{a} (0.086)*
CO100	1.14^{ab} (0.087)
MO50	1.17^{ab} (0.082)
UTW (Control)	1.20^{b} (0.072)

LO100: Linseed oil (100%). CO100: Castor oil (100%). MO50: 50% Castor oil + 50% Linseed oil. UTW: Untreatedwood. *: The difference between the numbers indicated by different letters within the same column is significant ($p \leq 0,05$; 95%). Numbers in parentheses are standard deviations.

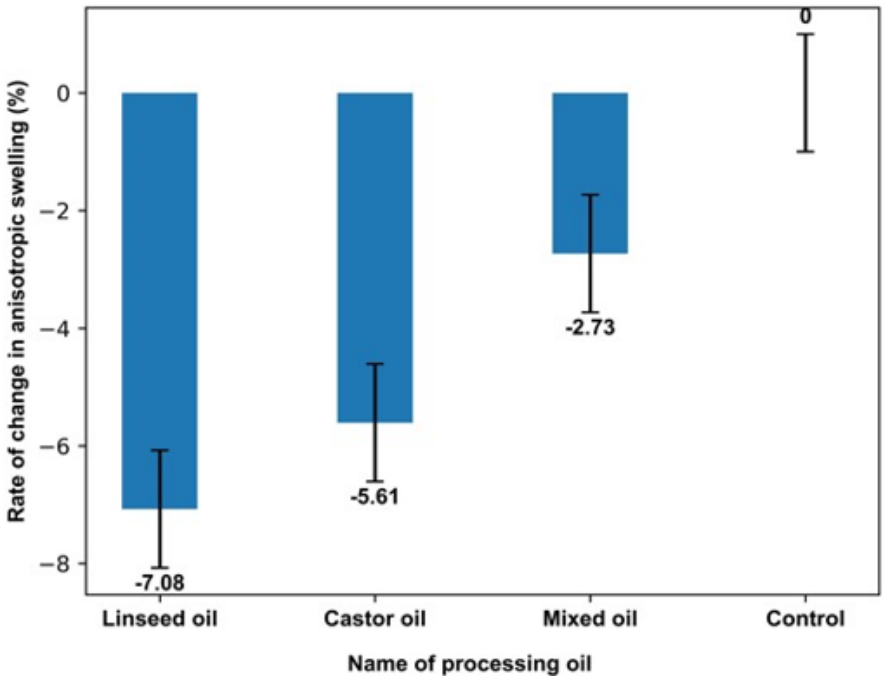

Fig. 3. ASW changes of the treated wood and the control

TGA (Thermogravimetric) Analysis

The results of the mass loss of the experimental wood samples after TGA are listed in Table 4. The thermal decomposition behaviour of these samples was comparatively investigated by TGA and DTG analyses. The obtained thermal decomposition curves/diagrams are shown in Fig. 5. Wood samples treated with LO100, CO100, and MO50 exhibited varying thermal stability but higher decomposition rates than the UTW sample. The impregnated samples showed an increase in thermal degradation temperatures and a decrease in weight loss compared to the UTW sample. The lowest weight loss of 86.3% was obtained for MO50. There was a 2.2% reduction in weight loss compared to the UTW sample. This reduction in weight loss was 1.7% for LO100 and 2.0% for CO100. Furthermore, when the thermal degradation parameters in Table 4 are analyzed, the temperature at which mass losses occurred at T10 wt% and T50 wt% increased continuously compared to the UTW sample. The temperatures increased from 262 °C to 272 °C, 274 °C, and 277 °C for T10 wt% and from 345 °C to 354 °C, 359 °C, and 357 °C for T50 wt%, respectively. Accordingly, it can be concluded that impregnated samples had higher degradation temperatures than unimpregnated samples. Based on this result, LO100, CO100, and MO50 treatment oils used in the study reduced the weight loss of Turkish pine wood after thermal bio-oil immersion. The decreases in weight loss and increases in thermal decomposition temperature may be due to the degradation of the chemicals contained in these oils at high temperatures. Several studies (Temiz *et al*. 2013; Ünlü 2013; Misni *et al*. 2019) reported that the treated samples exhibited higher initial decomposition temperatures compared to the control samples. Additionally, Misni *et al.* (2019) explained that TGA analysis demonstrated an improvement in the thermal stability of the bio-oil by the end of the process. Therefore, it is clear that these findings are compatible with the results reported in the literature.

Table 4. Weight Loss Results of Experimental Samples after TGA Analysis

Name of processing oil	$T10_{wt\%}$ (°C)	$T50_{wt\%}$ (°C)	DTG_{max} (°C)	Weight loss (%)
			Peak-1	
UTW (Control)	262	345	361.43	88.14
LO100	272	354	365.34	86.69
CO100	274	359	360.40	86.42
MO50	277	357	358.54	86.26

LO100: Linseed oil (100%). CO100: Castor oil (100%). MO50: 50% Castor oil + 50% Linseed oil. UTW: Untreatedwood. T10 wt%: Thermal decomposition temperature at 10% weight loss. T50 wt%: Thermal decomposition temperature at 50% weight loss

When the thermal decomposition curve in Fig. 3 is examined, the curved region between 25 and 100 °C indicates that the first mass losses due to thermal degradation in the analyzed wood samples occurred below 200 °C. This temperature corresponds to the removal of moisture and traces of inorganic compounds from the wood samples. These weight losses may be due to the evaporation of water/moisture and volatile organic compounds in the samples (Hadad and Goli 2018; Yücel *et al.* 2021; Zhang *et al.* 2024). In addition, these initial mass losses in wood samples may also be due to the presence of unstable groups. These mass losses at low temperatures may also be due to unstable alkyl groups increasing in the structure of CO100 oil in the mixture when LO100 is mixed with CO100 and used as MO50 (Mishra and Patel 2020). The curved region between 250 and 350 °C indicates the second decomposition step of thermal degradation in wood samples. In this region, it was determined that the molecules decomposed rapidly and the maximum mass loss was about 46% for UTW samples, while the minimum mass loss was about 34% for samples impregnated with LO100. The mass loss in the samples impregnated with CO100 was slightly higher than in the samples impregnated with MO50. The curved region between 350 and 500 °C indicates the decomposition of the main chemicals in the wood samples and the chemicals in the vegetable oils added to these samples. In this region, the highest mass loss occurred in MO50 with about 53%. The decomposition of ester bonds in the structure of linseed oil and castor oil may be due to dehydration, hydroxyl degradation, degradation of long alkyl chains, and polycondensation of alkyl groups, resulting in sharp decreases in mass loss in the indicated temperature range (Ganvit *et al.* 2024; Aparício *et al.* 2024). The samples impregnated with LO100 showed reductions in mass loss between 350 and 500 °C. These decreases can be explained by the destruction of sugar rings in the sample (Rashid *et al.* 2019). The curved region between 500 and 800 °C indicates that thermal decomposition in wood samples slows down. In this temperature range, the mass losses in the analyzed samples were found to be minimal.

When LO100 was mixed with CO100 and used as MO50, the increase in mass loss was higher for the samples impregnated with this MO50. Therefore, it can be said that the MO50 mixture used in the thermal bio-oil immersion process had no significant effect on the thermal decomposition behavior of the wood samples. These changes in the temperature ranges indicated in Fig. 5 are also confirmed by the DTG diagram.

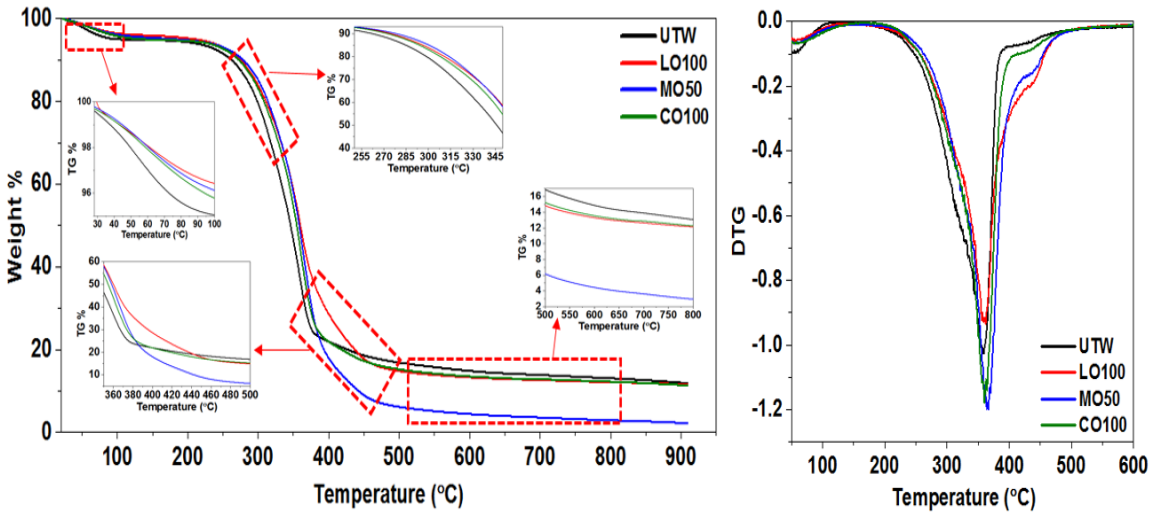

Fig. 4. The results of TGA and DTG analyzes of the experimental wood samples

FTIR (Fourier Transform Infrared) Analysis

The FTIR spectra of the experimental wood samples whose chemical compositions were characterized are shown in Fig. 5. The peak regions with bond structures according to functional groups are given in Table 5. The FTIR spectrum of the LO100-treated wood sample shows various peaks ranging from 1159 to 3398 cm^{-1}. Peaks between 3398 and 3425 cm^{-1} are also attributed to O-H hydrogen bonds present in the sample. There was a marked change in the peaks in this range. This change in the peaks can be attributed to the increase in the number of hydrogen bonds. The hydrogen bonds here may be phenol, alcohol or acidic, showing a polar structure. Peaks between 2927 and 2928 cm^{-1} and 2855 and 2856 cm^{-1} may be due to C-H resonance (Açıkgöz and Kaçkar 2003; Rashid *et al.* 2019; Safdar *et al.* 2019). The absorbances observed at 3010 cm^{-1} are attributed to the C-H strain symmetric vibration of cis-olefinic groups of flax oil (Çağlar 2011; Suri *et al.* 2020). The absorption peaks between 1640 and 1654 cm^{-1} may be due to the C=O asymmetric stretching vibration, which may be related to the bending mode of the absorbed water/rubber. This bond indicates the presence of ketone, aldehyde, or carboxylic acid. In addition, the carboxyl group is important for the binding of ions in the structure (Açıkgöz and Kaçkar 2003; Sun *et al.* 2018; Erkoç *et al.* 2021). The peaks at 1739 cm^{-1} and 1745 cm^{-1} are due to C=O stretching vibrations. Peaks between 1423 cm^{-1} and 1465 cm^{-1} are associated with symmetric COO-bond vibrations (Rashid *et al.* 2018; Hasanvand and Rafe 2018). Only for the untreated UTW sample, a distinct peak around 1375 cm^{-1} was observed due to CH_2 stretching vibrations (Bukhari *et al.* 2014). The 1509 and 1511 cm^{-1} peaks show C=O stretching vibrations (Hasanvand and Rafe 2018). The absorption bands located at 1159 and 1165 cm^{-1} correspond to the C-O-C glycoside bond (Prado *et al.* 2018). The interaction of this bond showed a significant increase in the LO100-treated samples compared to the UTW sample.

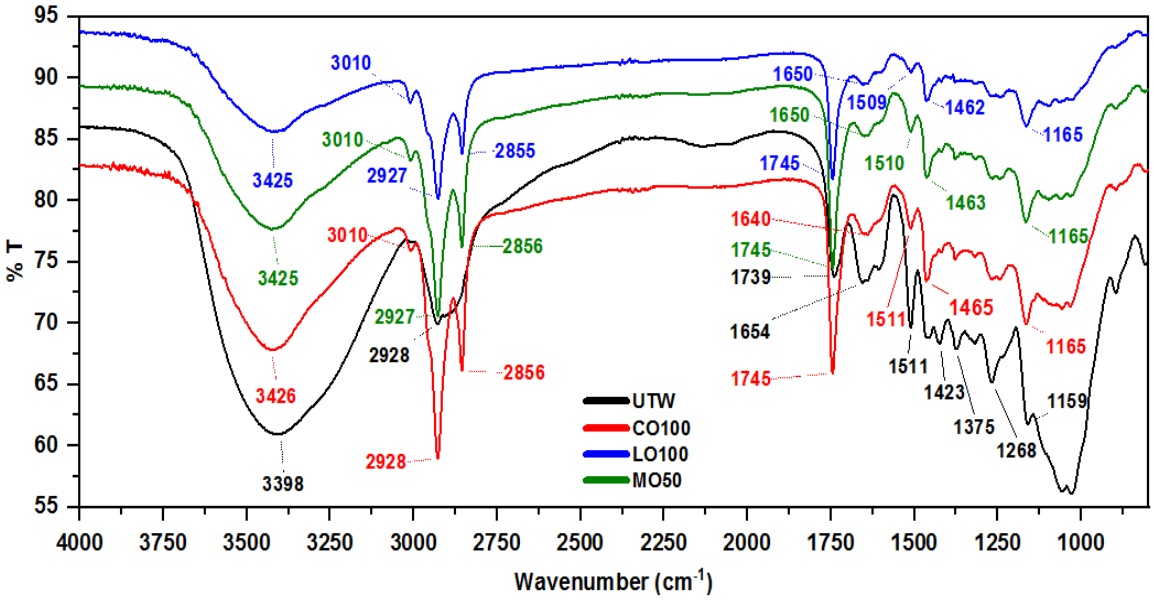

Fig. 5. FTIR spectra of the experimental wood samples

Table 5. Peaks in the FTIR Spectrum of the Experimental Wood Samples (Çağlar, 2011; Şimşek, 2024)

Band (cm^{-1})	Functional groups
3600 – 3200	-OH stretching
3006 – 3010	belonging to =CH group (LO100)
2950 – 2840	Symmetric and asymmetric -CH2 and CH3 stresses
1735 – 1750	belonging to the C=O group (ester)
1599 – 1580	belonging to the -C=C- group (aromatic or aliphatic)
1462 – 1385	belonging to the C-H group

CONCLUSIONS

1. In all analyses, the selected oil-treated wood properties for this study showed higher values, regardless of the type or ratio of bio-oil used.
2. Thermal bio-oil immersion produced a dry and non-greasy layer on the wood samples, and potentially it reduced the void volume and moisture content, while it limited the larvicide degradation of wood, and enhanced the selected engineering properties.
3. Specifically, pure linseed oil resulted in 100% larval mortality, with a modulus of rupture (MOR) of 108.4 N/mm², a compression strength parallel to the grain (CSPG) of 61.0 N/mm², an anisotropic swelling (ASW) value of 1.12, an 86.7% weight loss during thermal degradation, and a thermal degradation temperature of 365 °C.
4. Thermogravimetric analysis (TGA) demonstrated good thermal performance and stability against the thermal decomposition of cell wall materials in LO100-treated samples. Fourier transform infrared (FTIR) spectrocopy indicated an interaction

between the linseed oil components and the cell wall materials in LO100-treated samples. This interaction shows that the active linseed oil components were successfully placed into the cell wall materials without forming chemical bonds.

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