



**INTERNATIONAL FORESTRY SYMPOSIUM
IFS 2016**



PROCEEDINGS



DECEMBER 7-10, 2016 KASTAMONU UNIVERSITY, FACULTY OF FORESTRY, KASTAMONU, TURKEY



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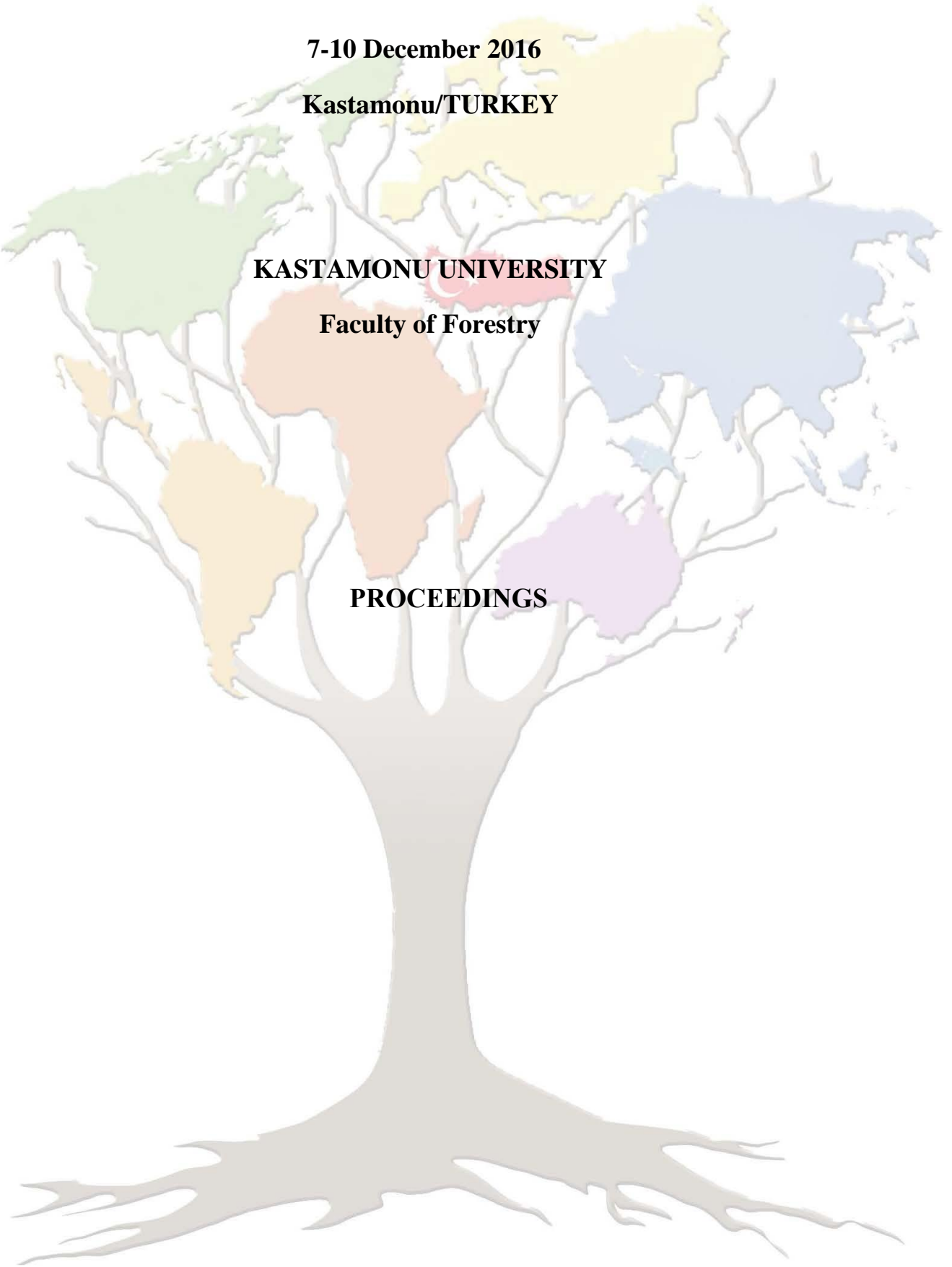
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Faculty of Forestry

PROCEEDINGS





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The Combined Effects of Copper and Oil Treatment on Wood Chemical Properties

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Abstract

Copper are among the most commonly used substances in impregnating industry due to its high toxicity against fungi at last 50 years. The performance of CCA (copper-chrome-arsenic) approved by all over the World because of its superior performance against wood-inhabiting fungi and insects, New and environmentally friendly materials have developed due to this substance banned. Among of developed impregnation materials, copper-azole is the most widely used, because it is highly effective against fungi and insects. But most important problems leach away copper from the wood. Various factors are affected on the copper material leaching from wood. Fixation time is most important effect of this situation. In this study, wood samples impregnated with 2.4% copper azole, after 24 hours and 5 days fixation time samples secondly exposed to tall oil and linseed oil. Chemical analysis of samples was carried out after conditioning time. In addition, the remaining amount of copper in the wood samples was measured by leaching test. According to copper leaching and FTIR-ATR results, combination tall oil and copper was the most resistant against copper leaching, and at 1508 cm⁻¹ and 1720 cm⁻¹ peak variation was observed.

Keywords: Copper azole, Tall oil, linseed oil, FTIR-ATR

Bakır ve Yağ Kombinasyonunun Odunun Kimyasal Özelliklerine Etkisi

Özet

Bakır, mantarlara karşı sahip olduğu yüksek zehirlilik etkisi nedeniyle son 50 yıldır emprenye endüstrisinde en fazla kullanılan maddeler arasında yer almaktadır. Mantar ve böceklere karşı performansı tüm dünya tarafından kabul edilen CCA (bakır-krom-arsenik) maddesinin yasaklanması, bu maddeye alternatif yeni ve çevre dostu maddelerin gelişmesine neden olmuştur. Geliştirilen maddeler arasında yaygın olarak kullanılan bakır-azol; mantar ve böceklere karşı son derece etkilidir. Fakat bakırın yıkanarak odundan uzaklaşması en önemli problemlerindendir. Bakır maddesinin odundan yıkanması üzerine çeşitli etmenler etki etmektedir. Bu etkilerin en önemlilerinden bir tanesi fiksasyon süresidir. Bu çalışmada bakır azol ile emprenye edilen örnekler 24 saat ve 5 gün sonra tall oil ve keten yağı ile ikinci bir emprenye işlemine tabi tutulmuşlardır. Kondisyonlama süresi sonrası örneklerin kimyasal analizleri gerçekleştirilmiştir. Ayrıca yıkanma sonrası odun örneklerinde kalan bakır miktarı ölçülmüştür. Elde edilen yıkanma ve FTIR-ATR sonuçlarına göre; Yağ ve bakır kombinasyonu yıkanmaya karşı direnç göstermiş ve 1508 cm⁻¹, 1720 cm⁻¹ piklerinde değişiklikler gözlenmiştir.

Anahtar kelimeler; Bakır azol, Tall yağı, Keten yağı, FTIR-ATR

Introduction

Wood preservation by combined processes including wood preservative treatment and subsequent impregnation with an hydrophobic product reduces the leaching of copper in use. H When hydrophobic agents are using for impregnation wood humidity being 12-20% and wood surface hydrophobic in order to reduce water uptake and decrease leaching of wood preservatives (Treu et al. 2011).

In wood protection, there is a trend towards using new-generation materials and methods by utilizing the recyclable materials and by-products of other industries. In recent years, the interest in using the natural and synthetic oils increased. In wood protection, various commercial or being-developed oils are utilized. The most important ones of these are linseed oil, tall oil, orange oil, soybean oil, and nut oil (Ozgenç, 2013). Tall oil is the byproduct

emerging as a result of paper production from needle-leaved trees. It consists of resin acids (40-55%), fatty acids (40-60%) and unsaponifiable matters (5-10%). In some of the studies, the tall oil has been shown to decrease the water intake and to prevent the fungal development. The large amount of oil required and the tendency of the oil to exude out of the treated wood have prevented extensive utilization of tall oil (Hyvönen et al., 2006; Koski, 2008; Lahtela, 2014; Temiz, 2008).

Linseed oil is made from the pressing of the dried ripe seeds of the flax plant which gives flaxseed oil. This oil is then exposed to a solvent extraction process to produce linseed oil. Linseed oil has some advantages such as water left on the surface may penetrate given enough time and oil finishes continue to protect as the Wood expands and contract. Linseed oil is generally composed of some oil acids. For example: α -linolenic acid (51.9-55.2%), palmitic acid (about 7%) and stearic acid (3.4-4.6%), oleic acid (18.5-22.6%), linoleic acid (14.2-17%) (URL- 2; URL-3). It is remarked in the research that flax oil is water repellent and enduring against UV rays (Ozgenç, 2013).

In a study of decreasing the copper washing, it has been reported that the hot oil procedure significantly decreased the copper washing and declined the water intake of wood samples during the weathering duration. Moreover, it has been emphasized that an aesthetic view was obtained by adding pigments into oil and the color change on wood surfaces was decreased (Treu et al., 2011).

In this study, the effects of fixation duration, tall oil and linseed oil on copper washing were examined. The amount of copper residuals on wood samples exposed to washing process was measured. Moreover, the effects of executed procedures on the chemical properties of wood were examined.

Materials and Methods

Scots pine (*Pinus sylvestris* L.) logs obtained from Blacksea region from Turkey. 30 mm diameter samples were used for XRF analysis. As you can see pictures of the samples used in

figure 1. The moisture content of samples was around 12%.

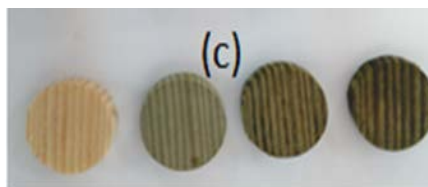


Figure 1. Photographs of treated samples (c) XRF samples

Copper-Azole was used as wood preservative at 2.4 % concentration alone as well as to the same ratio with water repellents. The major constituents of the copper azole are copper carbonate (20,5%), 2-aminoetanol (<20%), boric acid (<5%), tebuconazole (<0,5%), propiconazole (<0,5%), polyethyleneimine (<20%), organic acid (<5%), surfactant (<5%).

Linseed oil and tall oil was provided by the Izmir Altınyaz Company-Turkey and Çaycuma OYKA Paper Packing company-Turkey, respectively.

After the samples were prepared, they were conditioned for impregnation process for 2 weeks at 65% relative humidity and 25°C. The samples to be air-dried were impregnated at 30 minutes of 650 mmHg vacuum and 1 hour at 6bar pressure as predicted in ASTM D 1413-76 standard. The weights of samples before the impregnation ($M_{e\ddot{o}}$) were measured, and then their weights ($M_{e\ddot{s}}$) were measured by cleaning the solution residuals after the impregnation. The amounts of retention were determined by using the equation (1).

$$\text{Retention (kg m}^{-3}\text{)} = \frac{G \times C}{V} \times 10 \quad (1)$$

Where

G is amount of preservative solution absorbed by sample ($M_{e\ddot{s}} - M_{e\ddot{o}}$) (g), C is concentration of preservative solution (%) and V is sample volume (cm^3)

The weight percentage gain (WPG) which is indicative of net oil uptake was determined from the changes in the oven-dry weight after the treatment using equality 2.

$$WPG (\%) = \frac{W_a - W_b}{W_b} \times 100 \quad (2)$$

Where

W_b is the oven-dry weight of specimens before treatment (g), and W_a is the oven-dry weight of specimens after treatment (g).

Samples were divided into three test groups.

Test A; Examples in this group were impregnated with copper azole solution at 2.4% concentration.

Test B; The samples impregnated with copper azole solution were divided into 2 groups. First group of samples were kept at 50 °C for 25 hours for the completion of fixation process. Second group of samples, on the other hand, was kept for 5 days under room conditions (25°C and 65% relative humidity). After various fixation durations, the second impregnation procedures were executed with 50% tall oil (50TO) and linseed oil (50LO). Ethanol was used for preparing 50% tall oil and linseed oil.

Test C; Hot oil was used in this group. The samples impregnated with copper azole solution were kept at 50°C for 24 hours and 5 days under room conditions (25°C and %65 relative humidity) for completion of fixation process. At the ends of these durations, the samples were placed into oil heated to 80°C, and then kept for 4 hours. At the end of this period, after taking them out from the oil and cleaning the samples, they were weighed for determining the increase in weight (Equation 2).

In samples to be exposed to FTIR-ATR procedure, no washing was performed. Sample groups are included in the Table 1.

Fourier Transform Infrared Spectroscopy (FTIR-ATR) analysis has been used as a technique to obtain information about the structure of the wood constituents and chemical changes taking place in the wood impregnation process. FTIR spectra were obtained by a Shimadzu IRAffinity-1 equipped with a Single Reflection ATR pike MIRacle sampling accessory. Four accumulated spectra with a resolution of 4 cm^{-1} were obtained for wavenumbers from 1100 cm^{-1} -1800 cm^{-1} with 32 scans for each sample.

Tablo 1. Sample groups

Control	Non treated
Test A	Treated with copper azole
	CuA1d+50TO
Test B	CuA1d+50LO
	CuA5d+50TO
	CuA5d+50LO
	CuA1d+80TO
Test C	CuA1d+80LO
	CuA5d+80TO
	CuA5d+80LO

The measurements can be rapidly, sensitively, and reliably at low cost and without damaging the material via X-ray Fluorescence (XRF) technique. For this reason, it is widely used in scientific and technologic researches. The samples prepared were cut into 30 mm diameter and 5 mm thickness. These samples were kept in drying oven for 1 day at 60°C before starting the analyses and, in order to prevent any residual on the surface, they were air-dried with 1milibar air gun. In same day, the samples were analyzed using epsilon 5 model of XRF device and the device's own software. For each of the samples, the duration of total analysis was set at 25 minutes. Since the raw material was cellulosic material, H5C10O5 balance was selected and the amount of elements on surface was determined semi quantitatively. The measurements were executed under vacuum and He atmosphere conditions. Results were rapidly and reliably obtained at ppm-% concentration level without any damage.

Results and Discussion

Fourier Transform Infrared Spectroscopy (FTIR-ATR)

The FTIR spectra of treated and untreated samples are shown figure 2-3. Significant changes in the intensities of certain absorption bands are noticeable on the FTIR-ATR spectra between 1100 cm^{-1} and 1800 cm^{-1} . The assignments of characteristic absorption IR bands of Wood samples in the fingerprint region given in table 4.

Table 4. Assignments of absorption IR spectral bands in Wood

Wavenumber (cm ⁻¹)	Functionality	Vibrating type
1740-1720	C=O in unconjugated ketones, aldehydes and carboxyl	C=O stretching ¹
1660-1645	C=O in para-OH substituted aryl ketones, quinines	C=C stretching ³
1600	C=O in aromatic ring in lignin	Aromatic skeletal vibrations ²
1515-1500	aromatic ring	Benzene ring stretching vibrations ¹
1420	aromatic ring and CH	Benzene skeletal combined with C-H deformations ¹
1330-1240	CO in lignin and hemicellulose, and OH	C-O stretching and bending OH, antisymmetric stretching vibration of the acetyl ester groups ^{1,2}
1162-1086	C-O-C in cellulose	Antisym, Bridge oxygen stretching ²
1128	S-Syringyl lignin and C-O	C-H deformations in S lignin and C-O stretching ¹
1025-1035	C-O-C	Deformation ¹
897	Anti-symmetric out-of phase stretching in pyranose ring	Stretching in pyranose ring ¹

¹Ozgenç et al. (2013), ²Esteves et al. (2013),
³Temiz (2005)

In Table 4, the peaks show deviations because of the translocations of rings. In other words, the absorption peak at 1510 cm^{-1} is seen within the range of $1500\text{-}1515\text{ cm}^{-1}$. The similar results were obtained in our study.

Given Figure 2 and 3, it can be seen that the treatment with copper and oil led to changes in chemical structure of the wood. The peak at 1159 cm^{-1} changed after the treatment with copper and oils. These changes led to decrease in copper and significant increase in linseed oil. This increase can be attributed to ester bonds in linseed oil (Ozgenç et al., 2013; Weerd et al., 2005).

The peak at 1261 cm^{-1} shows the CO and OH groups in hemi-cellulose and lignin. A decrease was observed in samples impregnated with CuA

solution. This decrease was minimized by using tall oil and linseed oil. The changes in absorption peaks at $1230\text{-}1267\text{ cm}^{-1}$ are directly related with lignin delignification and modification of aromatic rings. Temiz et al., (2007) said that absorption peaks at around 1261 cm^{-1} for linseed oil and tall oil treatments did not change significantly.

The peak at 1508 cm^{-1} shows the aromatic ring peaks. In some of the studies, it has been shown that the peak at 1508 cm^{-1} disappeared or significantly decreased after the impregnation procedure (Temiz et al., 2007; Temiz, 2005; Salla et al., 2012). But, in another study, the increase in peak at 1508 cm^{-1} has been emphasized (Ozgenç et al., 2013).

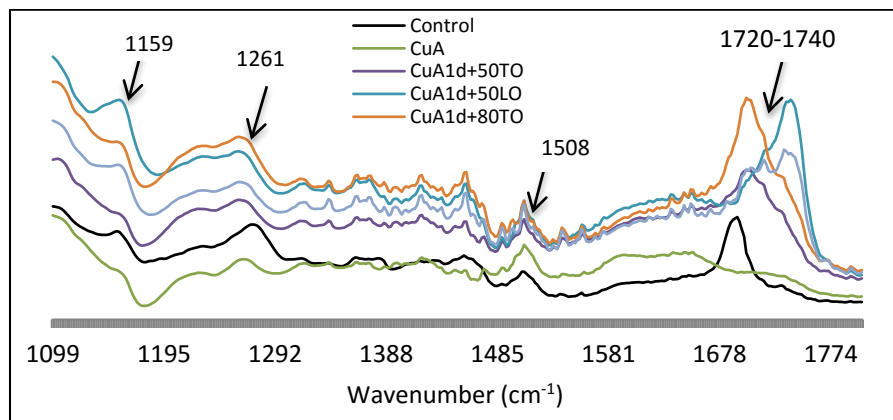


Figure 2. FTIR-ATR spectra of the control, CuA and 1 days fixation time test group

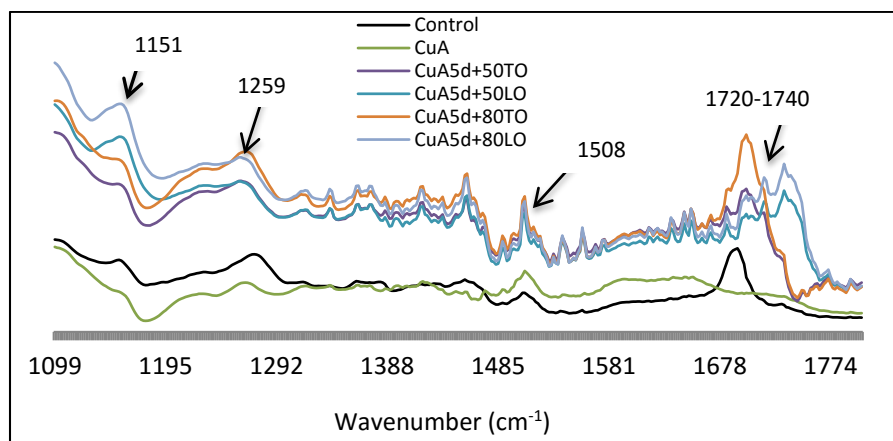


Figure 3. FTIR-ATR spectra of the control, CuA and 5 days fixation time test group

A decreased was found for the carbonyl absorption band at 1720-1740 cm^{-1} except for copper treatment. After the copper impregnation, this peak disappeared. Zhang and Kamdem (200a), in their study carried out by using copper ethanol amine, have reported that the peak at 1739 cm^{-1} decreased after the impregnation, and that this decrease was related with the anion of carboxyl acid. Moreover, it has been reported by Temiz (2005) that the fact that these peaks were not seen in the same region after the impregnation is caused from the effects of impregnation materials on translocation of aromatic rings and from the interaction between wood components and copper. But, an increase was observed in the samples exposed to secondary impregnation with linseed oil and tall oil. The increase in peak at 1720-1740 cm^{-1} during the treatment with oils shows similarities with literature (Temiz et al., 2007).

X-Işınları Floresans (XRF) Spektroskopisi
The lowest copper amount was determined from Cu5d+80TO treated wood. The highest copper amount was obtained from the samples treated with CuA. Significant level of washing (65%) was observed in Test A group samples. In their study, Kangsepp et al., (2011) have reported

that, after 14 days of washing, 23% copper washing occurred in samples impregnated with Tanalith-E.

The fixation duration is known to have effect on the wash of copper. In our study, except for CuA5d+50LO, the amount of copper washed decreased inversely proportional to the increase in duration. The oil treatment of samples kept in 80 °C for 5 days for fixation and the impregnation executed at 50% concentration in samples kept for 1 day for fixation produced effective results.

In both of 1 day of fixation and 5 days of fixation, the lowest WA values were observed in the samples impregnated with linseed oil. But, the level of copper washed is lower in samples treated with tall oil, except for CuA1d+50TO (1490 ppm). 65% washing was observed in Test A group samples. But, when the samples were treated with oil, the maximum washing was 50.76% (Table 4). In procedures, where the oils were used, the water intake of wood was decreased and the copper washing was declined.

Table 4. The percentage removed copper from CuA treated Wood samples

Variation	Retention (Kg m-3)	WPG (%)	Initial Cu amount (ppm)	Total Cu amount leached out (ppm)	Percentage of Cu leached out (%)
CuA	15.94	-	4860	1920	65.31
CuA1d+50TO	18.78	28.18	5260	1490	39.52
CuA1d+50LO	14.56	30.03	3490	570	19.52
CuA1d+80TO	14.47	16.30	3220	960	42.48
CuA1d+80LO	14.03	12.36	3750	1180	45.91
CuA5d+50TO	14.25	25.75	5040	600	13.51
CuA5d+50LO	16.28	35.28	5970	2010	50.76
CuA5d+80TO	14.38	16.88	5320	320	6.40
CuA5d+80LO	17.03	18.68	6310	700	12.48

In this study, after 24 hours and 5 days of fixation, the chemical analyses of the samples previously treated with tall oil flex oil were executed. The results obtained are summarized below.

1. According to FT-IR results, intensities of absorption band at 1508 cm⁻¹ (characteristic peak for lignin) were less increased by copper, linseed and tall oil treatments. A decreased was found for the carbonyl absorption band at 1720-1740 cm⁻¹ except for copper treatment. This peak disappeared after the copper impregnation. The increase in fixation duration caused alterations in chemical structure.

2. According to the study results, higher level of copper washing was observed when compared to copper azole impregnation. This rate decreased in parallel with the use of oils. The lowest level of copper washing was observed in Test C group.

3. Before using the samples impregnated with copper solutions, the completion of copper's fixation should be waited. In order to decrease the copper washing, the oiled heat-treatments are believed to be more efficient.

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