

Dyeing of Wooden Parts with Organic Dyes by Boiling Together Method

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Wood specimens were dyed by boiling them in the presence of walnut shells and dye to penetrate the dyestuff into the sample. Specimens prepared from Scots pine, oak, and beech woods were boiled separately in walnut shell, water, and NaOH environment, and the penetration of the dyestuff into the samples was ensured. Sodium hydroxide solution was preferred because it facilitates the dissolution of the hemicelluloses in the wood and the dyestuffs in the walnut shell. Alum was added in other samples to reveal the mordant effect on the adhesion of dyestuffs to wood. Thermogravimetric (TG) analysis revealed that wood degradation primarily occurs due to evaporation of free water at 50 to 120 °C, followed by lignin and cellulose breakdown across a wide temperature range of 160 to 600 °C. Treatment with NaOH and NaOH+alum notably reduced the peak temperatures in differential-thermogravimetric analysis by indicating an effect on lignin. This was supported by Fourier transform infrared analysis, particularly in the disappearance of carboxyl groups at 1710 cm⁻¹ and significant decreases in peak intensities at 1027, 1247, 1315, and 1501 cm⁻¹. Based on the findings, it was concluded that the dyed parts obtained can be used in the wooden toy industry.

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INTRODUCTION

Natural materials, such as wood, clay, stone, and leather, have had an important place in human life since the beginning of humankind. The use of these natural resources that were vital in the early ages has been reduced in recent times due to the over-consumption of synthetic products. The use of wood has started to regain its relevance in different fields because wood raw material is a sustainable natural resource and is more accessible to processing than metals and plastics. Wood products are subjected to many surface treatments for protection or appreciation, depending on the place of use. These are painting, upholstery, coating, varnishing, sandblasting, burning, inlay carving, printing, etc.

Natural dyes can be regarded as superior to synthetic dyes in that they do not cause poisoning, allergic, and carcinogenic reactions in the human body. In addition, they can be easily obtained from nature and are sustainable. However, some natural dyes show poor adhesion to the wood surface, depending on their activity. In this case, mordants are needed. The mordants can be organic or inorganic substances that increase the adhesion of the dyestuff. In cellulose-based materials, iron, and alkali group mordants are generally preferred.

Plant-based dyes are obtained spontaneously from nature or from cultivated dye plants. Vegetable dyes are used in textile, wood, and paper dyeing. Roots, bark, flowers, fruits, and seeds are used to produce dyestuffs from plants. A dyestuff obtained from elderberry (*Sambucus nigra* L.) seed has been reported to achieve a satisfactory level of coloration in the dyeing of bleached papers (Gencer and Can 2016). In wool carpet yarn dyeing with walnut fruit peel, it has been reported that different colors from dark cumin, dark brown to light red-brown, dark green to light green were obtained according to some other plant-based dyestuffs and the type of metallic mordant used (Kayabaşı *et al.* 2003). In a study to determine the dyestuffs used in the manuscripts of the 15th century, non-destructive analyses were conducted, and it was concluded that these papers were colored with plant-based compounds (Longoni *et al.* 2023). It was stated that dyestuffs obtained from onion peel and red pine bark can be used in paper dyeing, and aluminum can be used as a mordant in the retention of natural dyes (Keşmer *et al.* 2020a,b; Longoni *et al.* 2023). In paper dyeing with dyestuff obtained from Crimean pine bark, the gel-like layer in the leaves of aloe vera (*Aleo vera* L.) plant was used as a mordant, and it was stated that a brighter color was obtained with the increase in the amount of aloe vera in the colorimetric measurements (Keşmer *et al.* 2020a). In addition, the dyestuffs can also be obtained as by-products from the forest industry. In the studies conducted, it was stated that the bark left over from the industrial use of wild cherry and wild cranberry wood can be used in paper dyeing (Gencer *et al.* 2019a,b).

Various wood materials were dyed with the dyestuff obtained from the Boz-Pirnal oak (*Quercus aucheri*) extract. When the color change values (ΔE) after exposure to UV light were examined, it was stated that the applications with vinegar mordant provided as much resistance as the samples used with metal mordant (Atılğan *et al.* 2011). Natural dyestuff was obtained from the bark of the Indian sequoia (*Caesalpinia sappan*) tree, and it was stated that plastic-wood composite materials could be dyed with this natural dye (Hongriphan *et al.* 2011). It has been stated that it can be a furniture colorant and preservative with natural dyestuffs obtained from root dye plant (*Rubia tinctorum*), buckthorn (*Rhamnus petiolaris*), walnut pericarp (*Juglans regia* L.), gence (*Datisca cannabina* L.), henna (*Lawsonia inermis* L.), and safflower (*Carthamus tinctorius* L.) (Yeniocak *et al.* 2015). It has been stated that the dyestuff obtained by water distillation from onion skin and natural mordant obtained from willow branches by water fermentation can be used together in dyeing material obtained from Scots pine wood to a light color (Eser *et al.* 2013). The natural dyestuff obtained from tea (*Camellia sinensis*) wastes has an aesthetic appearance that can be used in surface treatments of solid wood material (Atılğan *et al.* 2011).

The natural dyestuff obtained from *Prunus persica* leaves by water extraction is used to dye cotton fabric and wool yarns, through metal mordants. The wool yarns' colorfastness was found to be better than cotton fabric's (Önal *et al.* 2021). Natural dyes also have antimicrobial properties. In a study, according to the antimicrobial activity test results, it was found that the mixture of walnut shell dye with copper sulfate was effective against *Escherichia coli* ATCC 25922 and *Staphylococcus aureus* ATCC 6538/P microorganisms (Yeniocak *et al.* 2015). It was determined that walnut shell dye used in wool fabric dyeing showed antibacterial properties against *S. aureus*, *E. coli*, and *P. aeruginosa*, and its effect decreased significantly after washing and especially after exposure to light (Ghaheh *et al.* 2012). The natural dyes, therefore, have hygienic properties.

Although there are not enough records about toys in historical documents, it is understood that the history of wooden toys is quite old. For example, the wooden toys made by imitating pig statues and various objects dating back to the 4th century BC in Cyprus can be shown as evidence (Sommer 2011). In the toy industry, the introduction of plastic and metal toys into the market with mass production has reduced the use of wooden toys, which were handmade until then. However, studies on the use of metal and plastic toys show some risks to children's health. For example, a study using synthetic saliva simulated sucking and chewing and tested metallic toys and jewelry, plastic toys, and painted or coated toys and found high total concentrations of As, Cd, Cu, Ni, Pb, and Sb in saliva (Guney *et al.* 2017). In a similar study, polybrominated diphenyl ethers (PBDEs) leaching from toys into saliva under simulated conditions was reported to increase with the extent of usage (Ionas *et al.* 2016). Today, thanks to conscious consumers, the demand for wooden toys is increasing daily due to the harmful effects of metal and plastic in the toy industry with the increasing demand for natural products in every field. As people become more aware of this issue, the interest in wooden toys will continue to increase. In the wooden toy industry, if the proper place of use is chosen, it is possible to use all kinds of wood species. For example, a study conducted on fruit trees stated that the trunk wood of pomegranate, olive, and loquat trees can be used in toy production (Topaloğlu and Ustaömer 2020). While wooden toys are used in children's education and development, pieces with different colors are needed.

For this reason, wooden toys need to be painted. In the painting of wooden toys, paints that are harmless to human health should be used. These dyes must be obtained from natural raw materials and organic dyestuffs.

Toys are indispensable for children from when they are born to adulthood. Especially in the oral period, when children tend to place every object they find in their mouth, it is vital that the objects they encounter do not harm their health. In this period, bringing the toy to the mouth is also a form of behavior in children's communication with toys and is inevitable. Therefore, health-conscious objects should be used. It is also essential to use natural dyes to color the wooden material used in toy production. In this way, children can contribute to their social and physical development as healthy individuals.

This study investigated the dyeing of wood pieces obtained from Scots pine, oak, and beech woods, which are widely distributed and preferred in the toy industry, using organic dyes. Attention was paid to the changing of the color of the dyes in response to ultraviolet radiation. In addition, surface contact angle, Fourier transform infrared (FTIR) spectroscopy, and thermogravimetric analysis (TGA) were performed, and the difference between control and treated samples were examined. Because the NaOH used in this study increases the dissolution of simple sugars and hemicelluloses, the hygroscopic properties of the material are also expected to increase, and consequently, the water absorption and swelling properties are expected to improve. Because the NaOH passes into the solution at the end of the process, it does not cause any harm to the use of wood.

EXPERIMENTAL

Materials

The sample surfaces should be sanded first before measuring the contact angle of the plates used. The test samples were cut in equal dimensions from Scots pine, oak, and

beech wood ($65 \times 150 \times 8 \text{ mm}^3$). Otherwise, the porous structure of the surface has a distorting effect on the measurements (Benkreif *et al.* 2021). For this reason, the sample surfaces were sanded with HHH number sandpaper. The staining process was carried out by boiling walnut shells from the Zonguldak region in water along with wood samples. The control samples were compared with painted samples from each wood species. Because the samples obtained from each wood species have a unique color, no comparison was made between the species in the control samples. In the boiling process, NaOH was used to dissolve the simple sugars in the wood and to dissolve the walnut shell to decompose the dyestuff. In this technique, the aim was to increase the dye adhesion while reducing the work of preparing the samples. The boiling process was carried out with distilled water in an electric heater under atmospheric conditions for 4 h, and boiled distilled water was added to replace the missing water and keep the water level constant. In addition, alum ($\text{KAl}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$) was added to the other sample group to increase the retention of the dyestuff. After 4 h, the samples were allowed to cool down, removed from the solution, and drained. The dried samples were kept under room conditions on a large surface protected from direct sunlight for 30 months. This period was chosen based on the estimated interest duration and the toy use probability. At the end of this period, the illuminated surface was labeled as ‘Clear surface,’ and the underlying surface was labeled ‘Closed surface,’ and $L^*a^*b^*$ values were measured. Sapwood samples were used, and all measurements were made from the same point. The CIELAB (Commission Internationale de l’Eclairage) system consists of three variables (Can and Sivrikaya 2019): L^* (light stability) and a^* and b^* chromatographic coordinates ($+a^*$ for red, $-a^*$ for green, $+b^*$ for yellow, and $-b^*$ for blue). In addition, ΔL^* , Δa^* , and Δb^* indicate the color changes occurring when comparing the initial color (i) and at different time intervals (f), while ΔE^* refers to the total color change. A low ΔE^* value indicates little color change, *i.e.* a stable color.

The DSA100 drop shape analyzer, version 1.92 from KRÜSS GmbH (Hamburg, Germany), was used to evaluate the contact angle. The liquid probe was delivered using a 50 μL dosing syringe with a 0.5-mm diameter needle at a height of 3 mm from the sample surface. The liquid probe was distilled water at 20 °C. For every sample, a standard drop volume of 2 μL was used. For 90 s, the drop form was examined every 4 s, yielding 24 readings at each time. Four locations per sample were used to assess contact angles, and each treatment included five duplicates.

Fourier transform infrared (FTIR) spectroscopy was used to investigate the chemical structure of WBHC. The sheets were ground into powder, and FTIR analyses were performed. The infrared spectra in the 400 to 4000 cm^{-1} region were determined using the JASCO 430 model.

Thermogravimetric analysis (TGA) was used to assess the thermal durability of each sample. The Perkin-Elmer TGA7 model apparatus was used to perform the TGA method, and its temperature range was 30 to 600 °C. It was heated at a rate of 20 °C per min in an argon gas environment.

RESULTS AND DISCUSSION

Color Analysis

‘Closed-surface’ and ‘Clear-surface’ lab values kept under room conditions for 30 months are given in Table 1.

Table 1. Lab Values of 'Closed-surface' and 'Clear-surface' Kept Under Room Conditions for 30 Months

Wood Species	Closed-surface			Clear-surface		
	L^*	a^*	b^*	L^*	a^*	b^*
PC	76.68	7.85	32.51	73.82	9.46	35.06
P1	33.64	6.97	12.37	32.98	7.05	11.85
P2	28.34	4.88	7.19	28.12	6.67	11.82
BC	63.56	12.85	22.01	61.75	11.79	21.87
B1	25.82	4.24	6.06	25.07	3.55	5.45
B2	25.61	4.41	6.43	25.02	2.73	4.31
OC	65.60	7.60	23.24	63.69	7.75	25.46
O1	27.92	4.06	6.87	27.51	3.95	6.48
O2	22.66	4.22	6.12	22.45	5.32	8.77

PC: Scots Pine (*Pinus sylvestris* L.) Control, P1: Scots pine NaOH, P2: Scots pine NaOH+Alum, BC: Beech (*Fagus orientalis* Lipsky) Control, B1: Beech NaOH, B2: Beech NaOH+Alum, OC: Oak (*Quercus robur* L.) Control, O1: Oak NaOH, O2: Oak NaOH+Alum

PC (Scots Pine)

It has been reported that there can be a significant color change in solid wood material even in short periods of 2 h, depending on the temperature in heat treatment applications (Sahin *et al.* 2020). The color changes according to the chromophore groups. It has been stated that the main components of the wood and the amount of extractive matter are effective in the color change (Pastore *et al.* 2004). In contrast, the paper could suppress some of the degradation pathways because of the polyphenols (Małachowska *et al.* 2021). However, exposure to light, temperature, pH, *etc.*, affects the natural red color's low stability and brightness (Harsito *et al.* 2021). This situation limits the usage of areas of natural dyes. In this study, the different $L^*a^*b^*$ values in the control samples (SPC, BC, and OC) may also be related to these reasons. Similarly, it has been reported that the color change is higher in control samples than in dyed samples in light aging tests of wood samples dyed with dyestuff obtained from root dye plant (*Rubia tinctorum* L.) in wood dyeing (Goktas *et al.* 2009).

Table 2. Color Change Values of Test Specimens

	Closed-surface				Clear-surface			
	ΔL^*	Δa^*	Δb^*	ΔE	ΔL^*	Δa^*	Δb^*	ΔE
P1	43.04	0.88	20.14	47.53	40.84	2.41	23.21	47.04
P2	48.34	2.97	25.32	54.65	45.70	2.79	23.24	51.35
B1	37.74	8.61	15.95	41.87	36.68	8.24	16.42	41.02
B2	37.95	8.44	15.58	41.88	36.73	9.06	17.56	41.71
O1	37.68	3.54	16.37	41.23	36.18	3.80	18.98	41.03
O2	42.94	3.38	17.12	46.35	41.24	2.43	16.69	44.56

The lowest ΔE value was obtained for "Closed-surface" in P1 variation and its value was measured as 47.04. This represents the point where the color change is least obvious. In this variation, ΔL^* , Δa^* and Δb^* values were lower compared to other variations, indicating less color change. These differences show the effect of surface type on color change. "Clear-surface" is generally associated with more noticeable color changes, while

“Closed-surface” may cause less obvious color changes. This is a result of how surfaces reflect light and affect color perception.

Contact Angle and Wettability

The contact angle is measured to get an idea of the wood samples’ water absorption and repellency. Figure 1 shows the contact angle of control samples and treated samples for the three wood species.

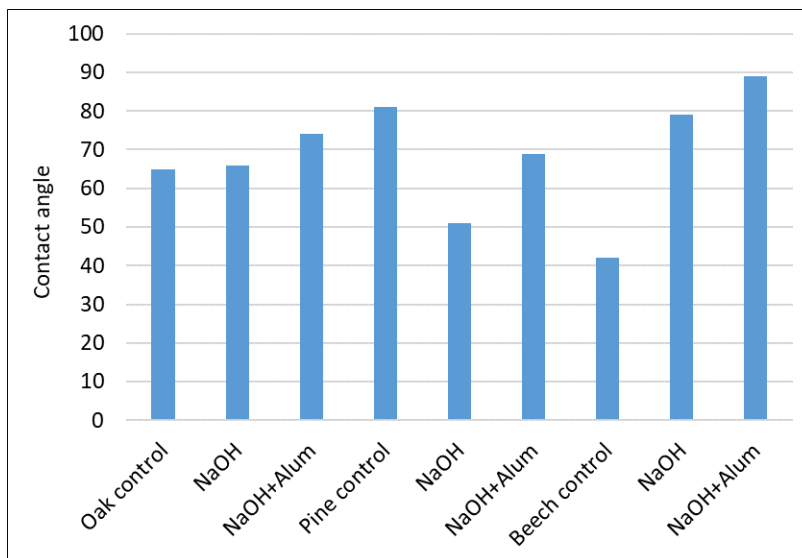


Fig. 1. Contact angle of control samples and treated samples for three tree species.

In this study, the contact angles of the Scots pine control samples (PC) was higher than the other samples. This is because the resins naturally found in Scots pine wood are hydrophobic. It has been reported that wood species with higher extractant content exhibit higher contact angles compared to wood species with lower extractant content (Papp and Csiha 2017). This observation has been confirmed in the present study. Treatment with NaOH resulted in a decrease in the contact angle with some resin dissolution. However, with the addition of alum, the contact angle increased in Scots pine and in all other samples. This treatment has increased the hydrophobic properties of all samples except the Scots pine control.

Thermal Analysis

Thermogravimetric analysis (TGA) has been used to ascertain the characteristics of wood thermal degradation. The evaporation of free water has been the primary cause of the weight loss of the samples, as shown in Figs. 2 to 4, from 50 to 120 °C. The breakdown of lignin and cellulose causes the thermal degradation of wood to occur throughout a broad temperature range from 160 to 600 °C. There was a single DTG peak at 364, 371, and 354 °C for pine, beech, and oak, respectively. The pine, beech, and oak control samples TGA curve showed the most weight reduction. In control woods, the DTG peaks were measured at 364, 371, and 354 °C, whereas the NaOH and NaOH+alum treatments, with the exception of beech, the peaks were somewhat lower at 326 and 334 °C for pine and oak, respectively. The findings demonstrated that NaOH and NaOH+alum treatments reduced the DTG peak temperature. Considering that NaOH and NaOH+alum treatment affects

lignin, these findings are consistent with other reports because cellulose has less heat stability than lignin (Lin *et al.* 2021).

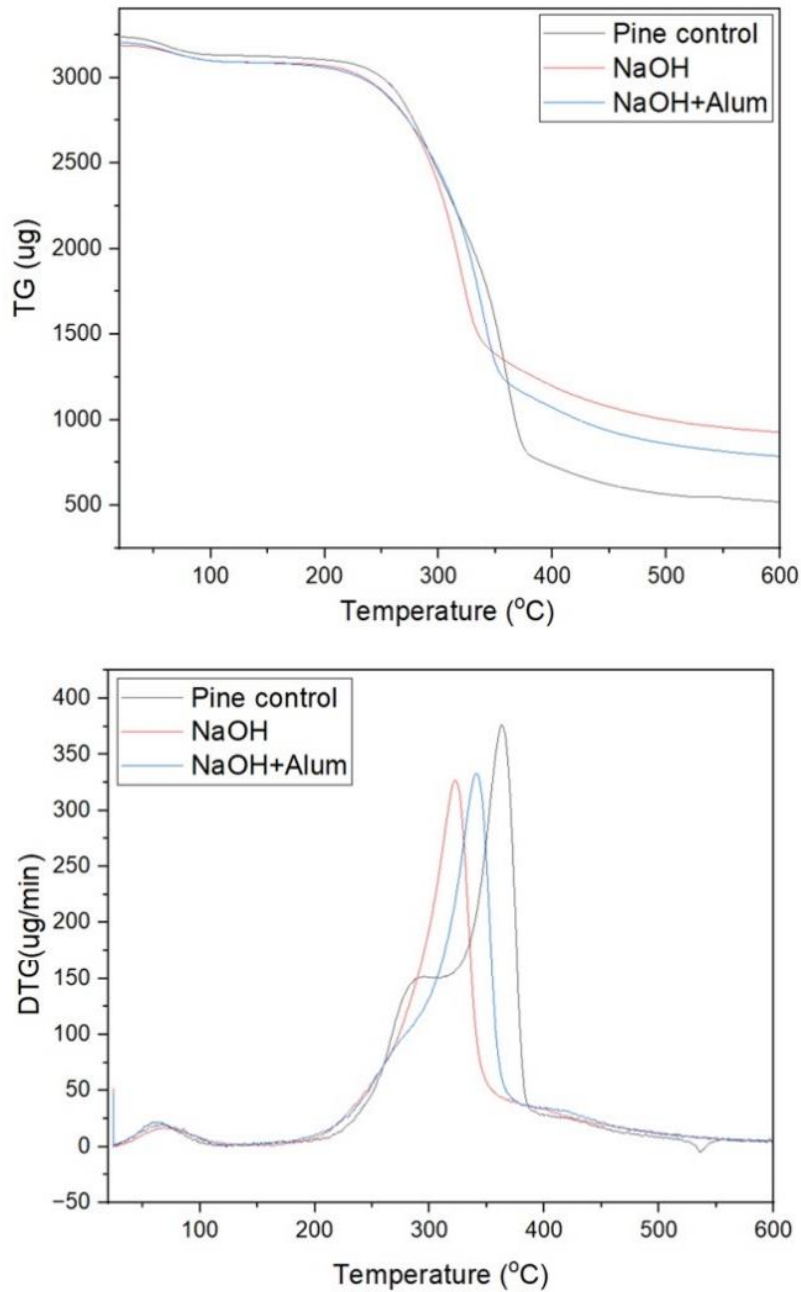


Fig. 2. TGA and DTG curves of Pine samples.

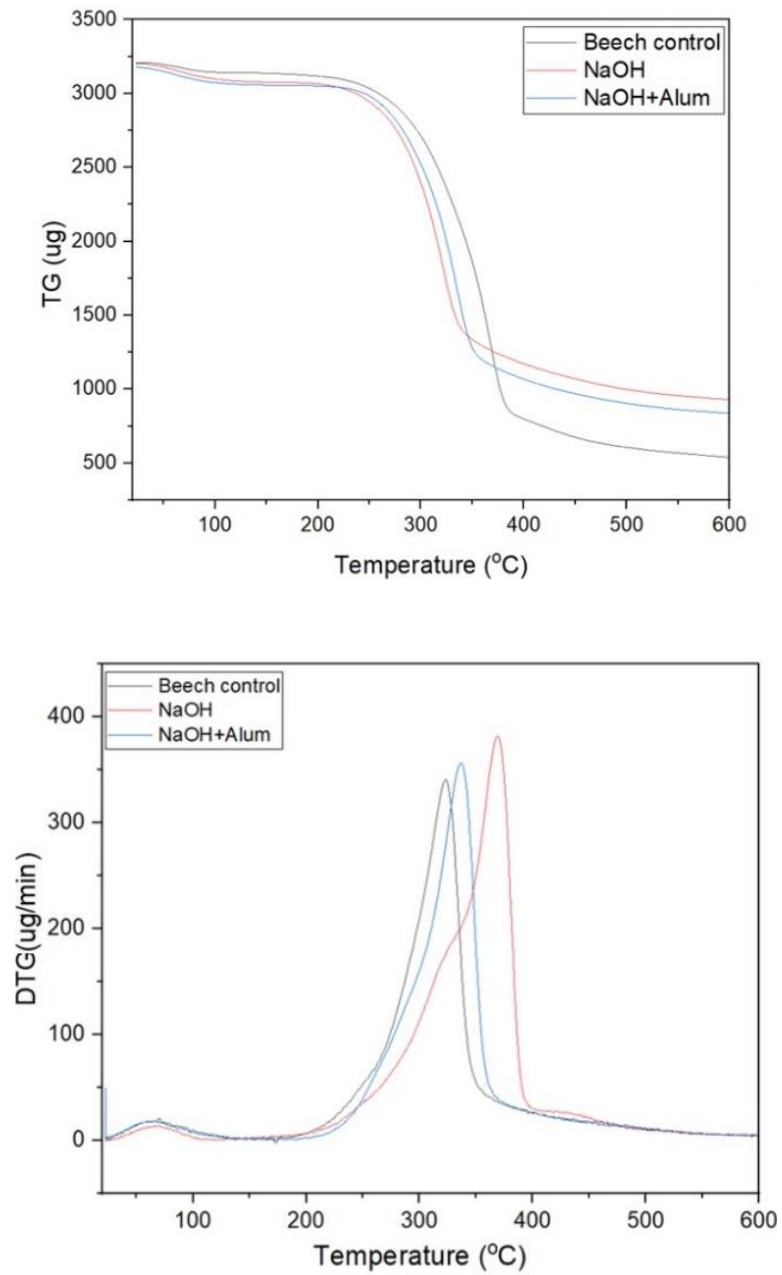


Fig. 3. TGA and DTG curves of beech samples

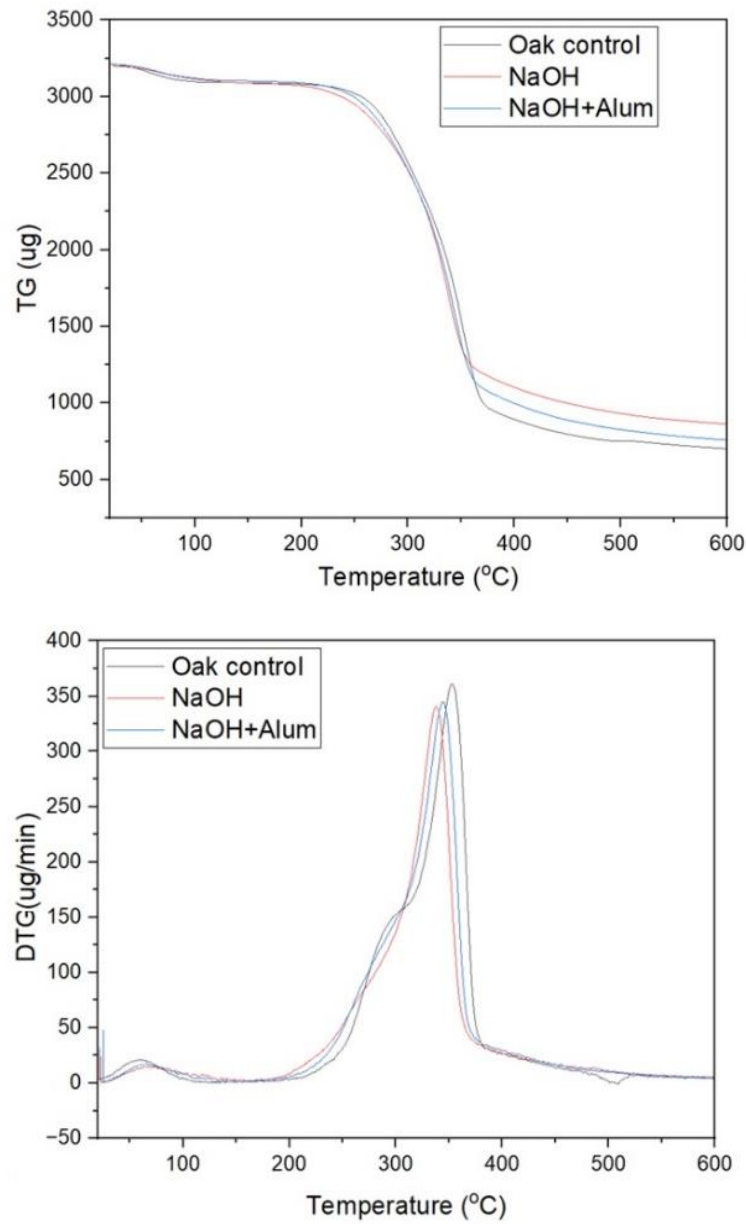


Fig. 4. TGA and DTG curves of oak samples

Infrared Spectrometry Results

The FTIR spectra of the wood samples before and after treatments are shown in Figs. 5 to 7. The large peak seen at 3342 cm^{-1} is attributed to the polysaccharides' and lignin's -O-H stretching vibrations, which are essential to the chemical structure of wood. It is noteworthy that following treatments with both NaOH and NaOH+alum, this peak became more intense. This implies that the interactions and hydrogen bonds inside the lignin structure are improved by these treatments. Furthermore, the -C-H bonds found in lignin and cellulose are responsible for the peaks that were identified at 2930 cm^{-1} , 2835 cm^{-1} , and 1425 cm^{-1} . These peaks offer important information about the chemical makeup of the wood samples. The peak at 1710 cm^{-1} , which represents the carboxyl groups in wood lignin, vanished following the NaOH treatment, which is an intriguing finding. This suggests that these carboxyl groups in the lignin structure are eliminated or undergo a change as a result of the NaOH treatment. Significant reductions in peak heights occur at 1027 , 1247 , 1315 , and 1501 cm^{-1} as a result of the NaOH treatment. These alterations point to probable changes in the cellulose and lignin contents of the wood samples due to differences in their chemical structure. It is clear from Figs. 5 through 7 that the cellulose components were most affected by the NaOH and NaOH+alum treatments. The relative increase in the peak intensity of lignin at 1510 cm^{-1} and the decrease in the hydroxyl stretching band around 3338 cm^{-1} provide support for this. These modifications show that the treatments break down the cellulose, which results in a lignin level that is comparatively higher.

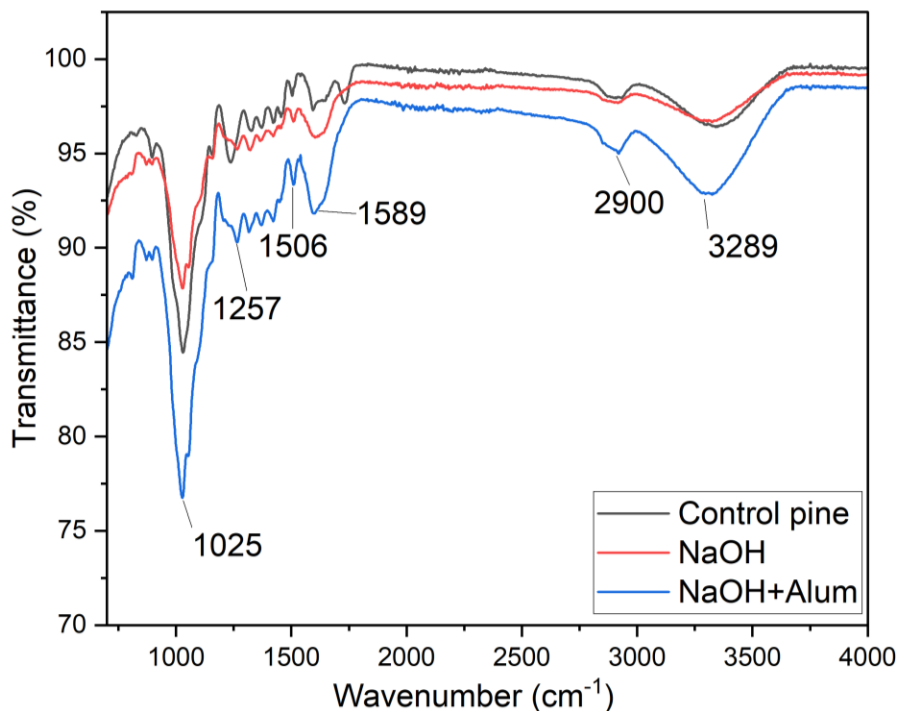


Fig. 5. FTIR curves of pine samples

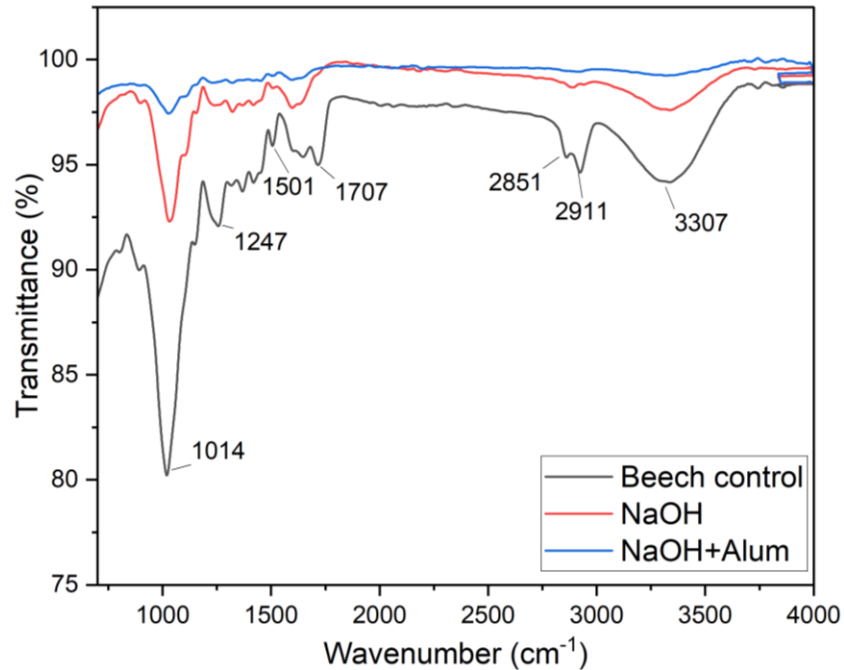


Fig. 6. FTIR curves of beech samples

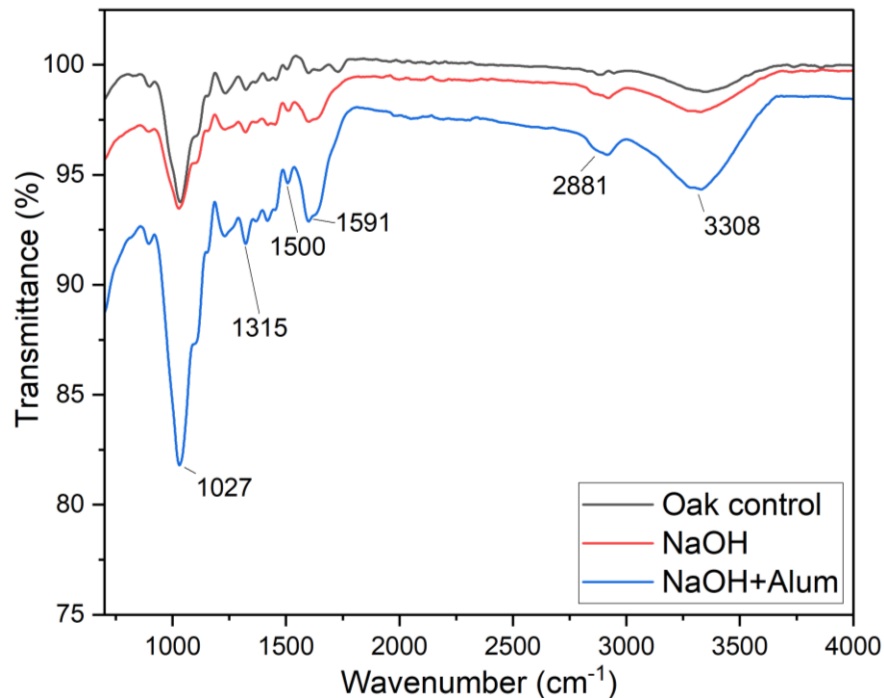


Fig. 7. FTIR curves of oak samples

All things considered, the FTIR measurements offer insightful information on the chemical changes that take place in the wood samples following the NaOH and NaOH+alum treatments. These results add to our understanding of the compositional variances and structural changes brought about by the treatments, and they are pertinent to the investigation into how various wood treatments affect the characteristics of the wood.

These changes may reflect transformations in wood's chemical structure and generally affect the cellulose and lignin contents. It is evident from Figs. 5 to 7 that the NaOH and NaOH+alum treatments mostly broke down cellulosic components, as shown by the relative rise in the lignin peak intensity at 1510 cm^{-1} and the decrease in the hydroxyl stretching band at around 3338 cm^{-1} .

CONCLUSIONS

1. Boiling wood material with the addition of dyestuff mordant for dyeing was found to be advantageous in terms of shortening the process time because more than one task was performed together. A disadvantage was the occurrence of cracking, warping, and some other wood defects in some parts during boiling. Therefore, the defective parts were excluded from the process. However, when considered in terms of production quality, it can be said that this situation, which is seen as a disadvantage, turns into an advantage. This is because the parts that come out of the boiling process intact will be resistant to such wood defects during use and will be able to be used for a long time.
2. When the wood species are grouped, the L value within the group was high in the undyed control samples and lowest in the samples with alum addition. This shows that the brightness decreased in all samples with the treatment. In all samples, L value was lower on surfaces exposed to light (Clear surface) than on surfaces avoided from light (Closed surface). Accordingly, the brightness values of all samples decreased with light intensity. The increase and decrease in a and b values varied with the change in wood species, indicating that there is no stability in blue and red color transformations.
3. Although the formation of wood defects in some parts may be seen as a disadvantage in painting with the boiling together method, when the defective parts are disposed of and the product is put on the market as a product, wood defects will not occur and there will be no quality problems because the work on the wood material is minimized.
4. Alum, which is used as a mordant against fading of organic paints, increases paint adhesion.
5. There will be a solution to an important problem in terms of turning walnut shells, which are still wasted, into a useful resource. The walnut shells left over from the production of dyestuff can be used as fuel or in soil improvement. For these reasons, it has a supportive feature for zero waste studies.
6. Most importantly, because dyeing is done with organic substances, it does not pose a risk to users.

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