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ORIGINAL ARTICLE

# Determination leaching of boron from Oriental beech wood coated with polyurethane/polyurea (PUU) hybrid and epoxy (EPR) resins

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**Abstract**

In this study, the leaching performance of boron compounds from Oriental beech (*Fagus orientalis* L.) wood coated with polyurethane/polyurea (PUU) hybrid resin and epoxy resin (EPR) was investigated. Leaching of boron test and SEM (Scanning Electron Microscopy) analyzes were applied to the prepared test specimens. According to the leaching of boron test results, specimens coated with polyurethane/polyurea (PUU) hybrid resin gave the most positive result against boron leaching. It was found that boric acid (BA) impregnated and epoxy resin (EPR) coated Oriental beech wood showed a smoother surface than other treatment groups.

**Keywords:** *boron compounds, epoxy resin, leaching of boron, Oriental beech, polyurethane/polyurea hybrid resin.*

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## 1. Introduction

Wood material has been among the most important building materials since ancient times due to its nature, structural features, and easy shaping. Among the most prominent features of wood being preferred in architecture; being light, resistant to physical and mechanical effects, low heat and sound insulation, aesthetic appearance, easy workability, ensuring color and design integrity, not having harmful effects on human health and the environment, being suitable for indoor and outdoor use. For these reasons, wood is preferred building materials[1-3]. There is increasing interest in using renewable natural resources in architecture. The main reason for this is the negative effects of industrial activities on the environment in recent years. Wood is an important natural material that is preferred in furniture production as well as in the building and construction industry. Wood is a desired building material in terms of its physical and mechanical properties. However, due to its organic structure, its chemical composition is cellulose, hemicellulose, lignin, resins, etc. consists of substances, it can decompose when exposed to environmental effects such as water, light, heat, fungi, bacteria, insects. Some inorganic materials are used to protect the wood. These substances are derivatives of copper, boron, and zinc. It also performs impregnation or coating processes by using organic compounds such as polymers, vegetable oils, essential oils, phenols, triazoles, benzoic acid derivatives, waxes, resins[4-9]. Structural changes in wood occur in connection with various mechanical and physical properties of wood. The permeability of wood should be improved in order to increase the amount of retention and to make a good impregnation process. The amount of retention can be increased by pre-treating wood biologically, chemically, physically, or mechanically. Wood that is not impregnated or coated begins to deteriorate chemically. The mechanical properties of chemically degraded wood generally continue to deteriorate. For example, alkali can cause cell collapse and increase wood density. The mechanical properties of wood, which is damaged by acids, are partially reduced not only in the wet state but also in the dry state of wood[10]. As concerns about the quality and durability of buildings increase, solutions are needed to increase the likelihood that structures can maintain their physical and mechanical integrity. It is even more important to preserve the wooden material in historical buildings[11]. Notable among the most observed anomalies in wooden parts of buildings are those caused by xylotrophic organisms such as rot fungi and subterranean termites, as well as dry wood termites and woodworms. The first two of these anomalies occur in timber with high moisture content and the second two occur in dry timber. Keeping timber in a good preservation condition and curing infection or infestation requires the use of chemical compounds. These chemical protection methods include impregnation, varnish coating, and paints[12]. One of the good ways to protect forests and ensure sustainability is the treating of wood. The use of treated wood in buildings also helps homeowners save money[13,14]. Until the early 1990s, the use of active substances such as pentachlorophenol, copper, tin or lindane, and CCA (chromated copper arsenate) compounds applied in organic solvents (LOSP) were common as wood preservatives[12-14]. Some of these formulations can be very effective at prolonging the life of the wood. However, the health hazard to workers and the risk of environmental impact on soil and landscape must be seriously considered[13,14]. Therefore, severe restrictions have been placed on the use of many of the above-mentioned constituents in the last few decades in Europe[15] and, for instance, in the USA[16] where CCA has been phased out from all new housing uses from the beginning of 2004[16]. With the increase in industrialization and technological developments, functional material options that are environmentally friendly, non-toxic, resistant to fire and flame, and widely used in architecture are also increasing. Wood material meets most of these characteristics. However, wood material, as mentioned above, is adversely affected by the damages in the outdoor environment due to its organic structure[17]. These problems can be partially overcome by modification or impregnation of the wood[18]. The use of boron compounds instead of using impregnation materials that have harmful effects on the environment can be an important step for a solution to the problems. Boron compounds protect the wood material against fungi, fire, and pests. However, it is necessary to increase the retention amount of boron minerals and to fix the boron compounds by decreasing the leaching amount. Attempts have been made to improve the leaching performance of boron compounds by using water repellents, monomers, and polymer systems[19]. Lloyd et al.[20] reported that the addition of polyols to borate solutions highly decreased boron leaching from wood by borate/polyole chalate complexion. Yalinkilic[21] investigated leaching characteristics of borates from sugi wood impregnated with boric acid-vinyl monomer combination treatment. Results indicated that sugi wood impregnated with boric acid ready lost its boron content in early leaching cycles, whereas vinyl monomer treatment resulted in five times lesser boron release into the water. Baysal and Yalinkilic[22] studied a natural wood polymer composite that was obtained under the catalytic effect of borates by using furfuryl alcohol. Furfuryl alcohol and borates were mixed at different ratios before treatment. When borates alone are used, they were leached from wood after cyclic leaching periods. However, this was not encountered with the mixture of furfuryl alcohol and borates. Baysal et al.[23] studied boron leaching from Douglas wood impregnated with borates and secondary some water repellent chemicals. Results indicated that secondary treatments of wood with the water repellent chemicals following borate impregnation reduced the leaching of chemicals from wood in water[24].

Among the construction materials which are used in architecture, wood holds a special place because of its impressive range of attractive properties, including low thermal extension, low density, and high enough mechanical strength. However, wood material has a leaching issue after impregnation with boron compounds. This study was carried out to measure the leaching of boron from Oriental beech (*Fagus orientalis* L.) wood, which is widely preferred in the architecture and furniture industry. In this context, experimental specimens impregnated with boron minerals, which have fire retardant properties, and coated with polyurethane/polyurea (PUU) hybrid resin and epoxy resin (EPR) were used.

## 2. Materials and Methods

### 2.1 Materials

Oriental beech (*Fagus orientalis* L.) wood was collected from Muğla province in Turkey. Wood specimens were prepared in 20 mm × 20 mm × 20 mm (radial, tangential, and longitudinal) dimensions. $H_3BO_3$ (Boric acid; CAS: 10043-35-3) and $Na_2B_4O_7.10H_2O$ (Borax; CAS 1303-96-4) were purchased from Merck Chemicals company and were used without any purification. Epoxy (sikafloor®-156) and polyurethane/polyurea hybrid resin (sikalastic®-851 R) resins were purchased from Sika company in Turkey. The microstructures of the specimens were obtained by using a Jeol JSM 7600F scanning electron microscope (SEM).

### 2.2 Impregnation process

3% aqueous solutions of boric acid (BA), borax (BX), and, a mixture of boric acid and borax (1:1; weight:weight) (BA+BX) were prepared to impregnate the Oriental beech wood specimens. The impregnation of the Oriental beech wood specimens was carried out according to ASTM D 1413-07[25]. In this study, the specimens were prepared for the impregnation process were first subjected to pre-vacuum for 30 minutes and then to the impregnation process under 1.01 bar pressure for 30 minutes in the impregnation device. Then the borates retention of Oriental beech was calculated from the following Equation 1:

$$R = \frac{G.C}{V} X10^3 \left( kg / m^3 \right) \quad (1)$$

where:

$G = T_2 - T_1$;

$T_2$ = Specimen weight after impregnation (gr);

$T_1$ = Specimen weight before impregnation (gr);

V = Specimen volume ($cm^3$);

C = Solution concentration (%).

### 2.3 Coating process

In this study, epoxy and polyurethane\polyurea hybrid resins were used for the surface coatings. Firstly, the impregnated test specimens were primed with epoxy resin (Sikafloor®-156) to prepare EPR coatings, afterwards, they were coated with polyurethane/polyurea hybrid resin (Sikalastic®-851 R) to obtain the PUU coatings. PUU coatings should be noticed that they were primed with epoxy resin (Sikafloor®-156) before coating with polyurea/polyurethane hybrid resin (Sikalastic®-851 R). Coating processes are depicted in Figure 1.

### 2.3.1 Preparation of epoxy coatings (EPR)

Sikafloor®-156 was used to prepare epoxy resin (EPR) coatings. It is composed of two parts, an epoxy part (A) and a hardener part (B). Sikafloor®-156 resin has some advantages such as low viscosity and being solvent-free[26]. The mixtures were prepared by mixing the epoxy and hardener with a 3:1 ratio of epoxy to hardener as follows the manufacturer's instructions. Then they were applied to the wood specimens which are impregnated with boron compounds (Figure 1).

The possible cure reaction between an epoxy part and a hardener part is given in Figure 2. The hardeners are known as the chemicals which are converted epoxy resin to thermosets, have usually bear active hydrogen attached to an electronegative atom such as N, O, or S. The curing reaction is a ring-opening reaction between the oxirane ring and a nucleophile (Figure 2). The ring-opening reaction occurs via nucleophilic attack by the hardener to the oxirane ring, then a second reaction follows until the remaining active hydrogens attached to the hardeners are fully reacted[27, 28].

### 2.3.2 Preparation of polyurea/polyurethane coatings (PUU)

Sikalastic®-851 R was used for PUU coatings. PUU coated by using Sikalastic®-851 R resin were made after test specimens were primed with epoxy resin (Sikafloor®-156). Sikalastic®-851 R is known as a two-component, elastic, crack-bridging, rapid-curing modified polyurethane/polyurea hybrid resin. Component A consists of isocyanate derivative and component B consists of polyol/amine derivative[28,29]. The coatings were professionally made by the manufacturer firm. Two layers were applied to the floor using special polyurea coating machines (GAMA G-30 H) at the consumption of 1.7-2.2 kg per $m^2$ and the second layer application was started within a maximum of 6 hours after the first layer application. A possible reaction, between polyurea/polyurethane and epoxy moieties which forms an epoxy-urea bond, and displays the new interfacial chemical reaction between Sikafloor®-156 and Sikalastic®-851 R, is illustrated in Figure 3[28,29].

## 2.4 Leaching test procedure

Wood specimens impregnated with boron compounds then coated with epoxy and polyurea/polyurethane hybrid resins were subjected to leaching of boron test. A leaching test was performed regarding the Yeniocak and Kahveci[30] study. First, the maximum wavelengths of the impregnation materials were determined by UV spectrophotometer to be a reference point for absorbance in evaluating the leaching results in the study. Three wood specimens from the same group were placed in the erlenmeyer with 250 ml of distilled water. The leaching properties of boron were determined by taking the erlenmeyer in a shaking water bath at 120 rpm shaking speed at different time intervals, different pH, and different temperatures. The tests were carried out at 10 °C, 22 °C, 40 °C temperature, in pH 7. The leaching of boron was measured at 5, 15, 30, 45, 60, 75, 90, and 120 minutes and the amount of substance (absorbance) transferred to the leaching water was measured in the UV spectrophotometer device, regarded maximum wavelengths values. Biochrom Libra S70 model, UV-Visible Spectrophotometer was used in

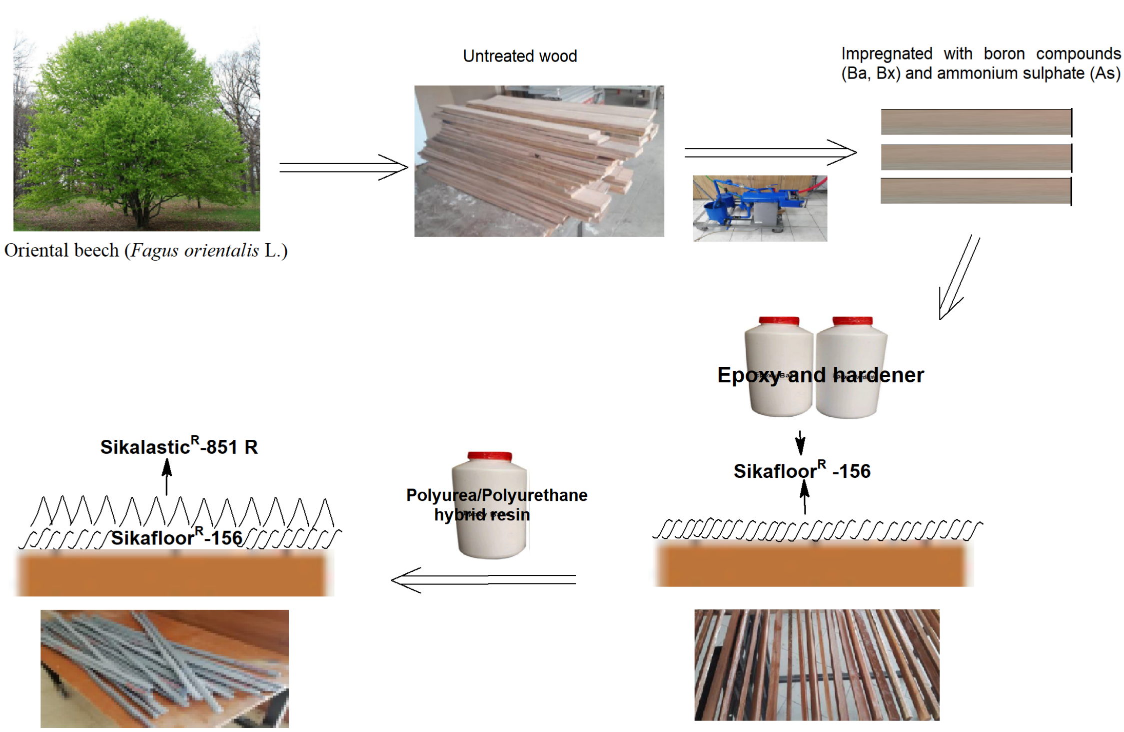


**Figure 1.** Preparation of epoxy coatings (EPR) and polyurea/polyurethane coatings (PUU).

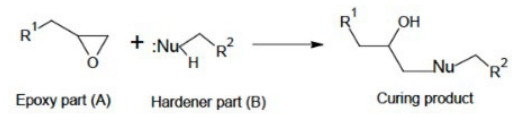


**Figure 2.** Possible curing reaction between epoxy part and hardener part for EPR coatings.

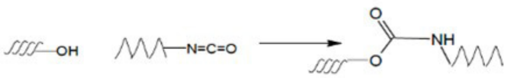

**Figure 3.** The reaction between epoxy and polyurea/polyurethane moieties.

the tests. The UV/VIS Spectrophotometer had a dual-beam in the wavelength range of 190-1100nm.

### *2.5 SEM (scanning electron microscope) analyzes*

Specimens impregnated with boron compounds and coated with EPR and PU (control, EPR, BA+EPR, BX+EPR, and ((BX+BA)+EPR) were coated with gold for surface morphology. The specimens were then examined with a scanning electron microscope (Jeol JSM 7600F). In this study, SEM analyzes could not be performed on PUU-coated specimens due to imaging problems of the scanning electron microscope device.

## 3. Results and Discussions

### *3.1 Leaching test*

The maximum wavelengths of solutions are given in Table 1. Maximum wavelengths were measured in nanometers in a UV spectrophotometer device, taking pure water as a reference. The maximum wavelength indicates the maximum amount of substance that can pass into the pure water during leaching. These obtained data were used as a reference in determining the amount of substance transferred to the water in the leaching test.

Retention is a factor in determining the total amount of solution added to the wood material in the impregnation process. Table 2 shows the retention values of boron compounds. The highest retention value was obtained as 17.23 kg/m$^3$ in BA+PUU applied group, while the minimum retention value was measured as 14.29 kg/m$^3$ in the ((BX+BA)+PUU) applied group.

Results of Oriental beech wood specimens coated with PUU and EPR and leaching of boron compounds under conditions of pH: 7, 10 °C, 22 °C, and 40 °C temperatures and 120 rpm in shaking speed were given in Table 3 in the unit of absorbance (abs).

In Table 3, after a temperature of 10 ˚C and 120 minutes, there was no leaching of boron observed in the group covered by PUU. On the other hand, low leaching of boron was observed in specimens impregnated with BX, BA, and BX+BA and coated with EPR. Here, minimum leaching of boron rate of 0.38 abs was observed in the wood specimens impregnated with BX+BA and coated with EPR, while the

**Table 1.** Maximum wavelength values of solutions.

| Impregnation group | Max. wavelength (nm) |
|---|---|
| BA | 201 |
| BX | 195 |
| BX+BA | 192 |

BA: Boric acid; BX: Borax.

**Table 2.** Retention values of boron compunds.

| Impregnation group | Retention (kg/m$^3$) |
|---|---|
| | Mean |
| BA+PUU | 17.23 |
| BX+PUU | 15.53 |
| BA+ EPR | 17.08 |
| BX+EPR | 16.52 |
| ((BX+BA)+PUU) | 14.29 |
| ((BX+BA)+EPR) | 15.30 |

Note: PUU: Polyurethane/Polyurea hybrid resin (Sikalastic®-851 R); EPR: Epoxy resin (Sikafloor®-156); BA: Boric acid; BX: Borax.

maximum 0.67 abs was measured in the wood specimens impregnated with BA and coated with EPR. After a temperature of 22 ˚C and 120 minutes, no leaching of boron in all groups was covered by PUU. Besides, low leaching of boron was observed in wood specimens impregnated with BX, BA, and BX+BA and coated with EPR. While the minimum leaching of boron with 0.92 abs was observed in the wood specimens impregnated with BA and coated with EPR, maximum leaching of boron was measured in wood specimens impregnated with BX + BA and coated with EPR as 1.18 abs. After a temperature of 40 °C and 120 minutes, no leaching of boron was observed in all experimental specimens impregnated and coated with PUU at 40 °C temperature, 120 rpm shaking speed, and after 120 minutes. Low leaching of boron was observed in wood specimens impregnated with BX, BA, and BX+BA and coated with EPR. The minimum leaching of boron rate was observed in wood specimens impregnated with BA and coated with EPR as 0.48 abs and maximum leaching of boron was measured in wood specimens impregnated with BX and coated with EPR as 1.27 abs. In this study; by applying polyurethane (PUU) and epoxy (EPR) resins to wood specimens subjected to the impregnation process, they were subjected to leaching of boron tests and it was aimed to prevent the water from physically dissolving and losing the preservative material by reaching the impregnation materials. When the results obtained were evaluated in general, it was observed that leaching of boron was completely prevented in all groups in which PUU was applied. On the other hand, it was observed that particular proportions of leaching of boron occurred in the epoxy resin applied groups. The factor of temperature plays an important role in the leaching of boron performance. Leaching of boron was observed at least at 10 °C, while the maximum leaching of boron was monitored at 40 °C (except for the BA+EPR group). The temperature rise may cause the water to overcome the resin layer physically bonded to the wood surface, causing the preservative to dissolve. In this case, it can be said that the groups coated with PUU are more resistant to the temperature increase than the EPR applied group.

### 3.2 Scanning electron microscope (SEM) analysis

The SEM images of untreated (control) wood specimens and coated wood specimens of Oriental beech (EPR, BA+EPR, BX+EPR, and (BX+BA) +EPR) are given in Figure 4 (Magnification are 50, 100, 250 and 500 times and bar is 100 µm). In this study, SEM analyzes could not be performed on specimens coated with PUU, due to problems in taking images in the scanning electron microscope device.

When the SEM images of the control specimen in Figure 4 are examined, a macroscopically smooth surface can be seen, even if there is a microscopically rough surface at x500 magnification.

When the SEM images of epoxy-coated specimens are examined in Figure 5, layered and rough surfaces can be seen easily. Since epoxy resin was cured with its hardener on the wood surface as was illustrated in Figure 2, it provides layered surfaces. While pure epoxy resin exhibited smooth

**Table 3.** Leaching of boron compounds from Oriental beech wood coated with PUU and EPR.

| Leaching temperatures | Impregnation group | Leaching time | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | | 5 min | 15 min | 30 min | 45 min | 60 min | 90 min | 120 min |
| Leaching at 10 °C temperature | ((BX+BA)+PUU) | - | - | - | - | - | - | - |
| | BA+PUU | - | - | - | - | - | - | - |
| | BX+PUU | - | - | - | - | - | - | - |
| | BX+EPR | 0.21 | 0.23 | 0.26 | 0.30 | 0.35 | 0.41 | 0.48 |
| | BA+EPR | 0.23 | 0.29 | 0.36 | 0.44 | 0.50 | 0.59 | 0.67 |
| | ((BX+BA)+EPR) | 0.01 | 0.04 | 0.11 | 0.18 | 0.05 | 0.30 | 0.38 |
| Leaching at 22 °C temperature | ((BX+BA)+PUU) | - | - | - | - | - | - | - |
| | BA+PUU | - | - | - | - | - | - | - |
| | BX+PUU | - | - | - | - | - | - | - |
| | BX+EPR | 0.38 | 0.04 | 0.49 | 0.60 | 0.71 | 0.86 | 1.02 |
| | BA+EPR | 0.17 | 0.14 | 0.34 | 0.48 | 0.59 | 0.73 | 0.92 |
| | ((BX+BA)+EPR) | 0.82 | 1.05 | 1.18 | 1.11 | 1.14 | 1.14 | 1.18 |
| Leaching at 40 °C temperature | ((BX+BA)+PUU) | - | - | - | - | - | - | - |
| | BA+PUU | - | - | - | - | - | - | - |
| | BX+PUU | - | - | - | - | - | - | - |
| | BX+EPR | 0.57 | 0.72 | 1.02 | 1.11 | 1.26 | 1.26 | 1.27 |
| | BA+EPR | 0.01 | 0.09 | 0.17 | 0.24 | 0.30 | 0.38 | 0.48 |
| | ((BX+BA)+EPR) | 0.49 | 0.82 | 1.05 | 0.98 | 1.09 | 1.00 | 0.99 |

Note: PUU: Polyurethane/Polyurea hybrid resin (Sikalastic®-851 R); EPR: Epoxy resin (Sikafloor®-156); BA: Boric acid; BX: Borax.

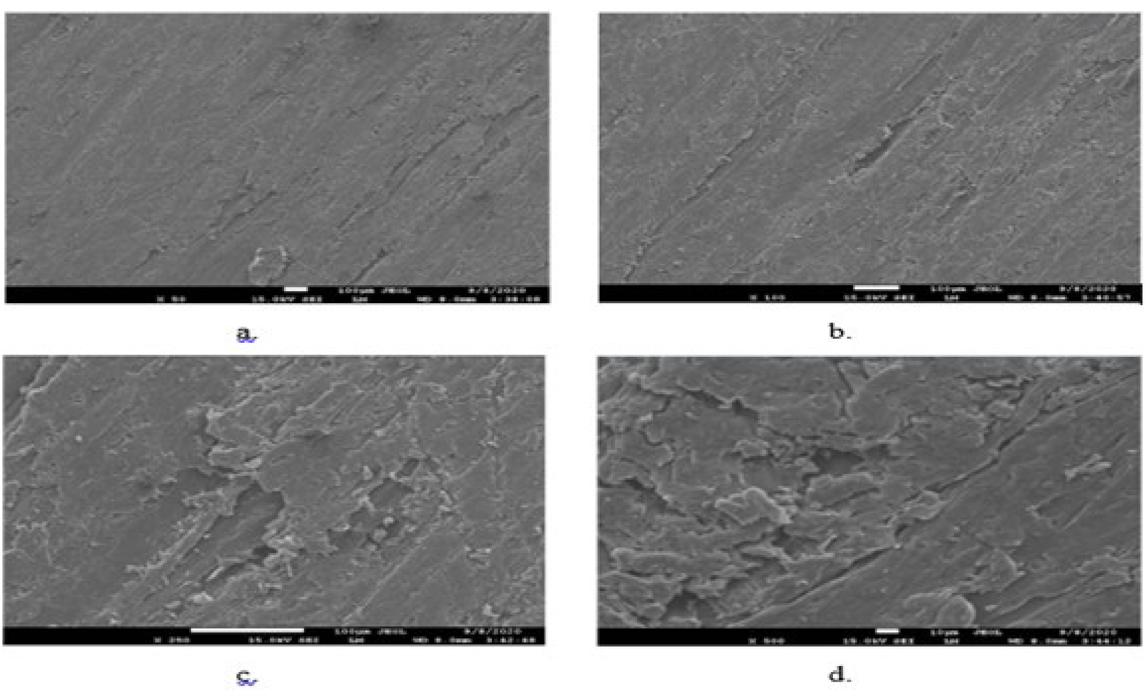

**Figure 4.** Magnified SEM images of untreated wood specimens. (a) 50x, bar 100 µm; (b) 100x, bar 100 µm; (c) 250x, bar 100 µm; (d) 500x, bar 100 µm.

surfaces at 50x magnification in the literature, EPR exhibited rough surfaces in this study. It is expected that EPR reduces surface roughness. The low viscosity of the EPR or the insufficient amount of application to the surface may be the main reasons for the rough surfaces in the Figure 5.

The SEM images impregnated with BX and coated with EPR displayed that the specimens covered the micropores and cell walls (Figure 6). It was observed for BX impregnated and EPR coated wood specimens had more layered, hollow, and rough surfaces compared to the impregnated with BA and coated with EPR (Figure 7). The reason could be that the presence of empty cells and these empty cells create aspiration of these passages after the polymerization or drying process of the specimens[31]. Besides this, the cause

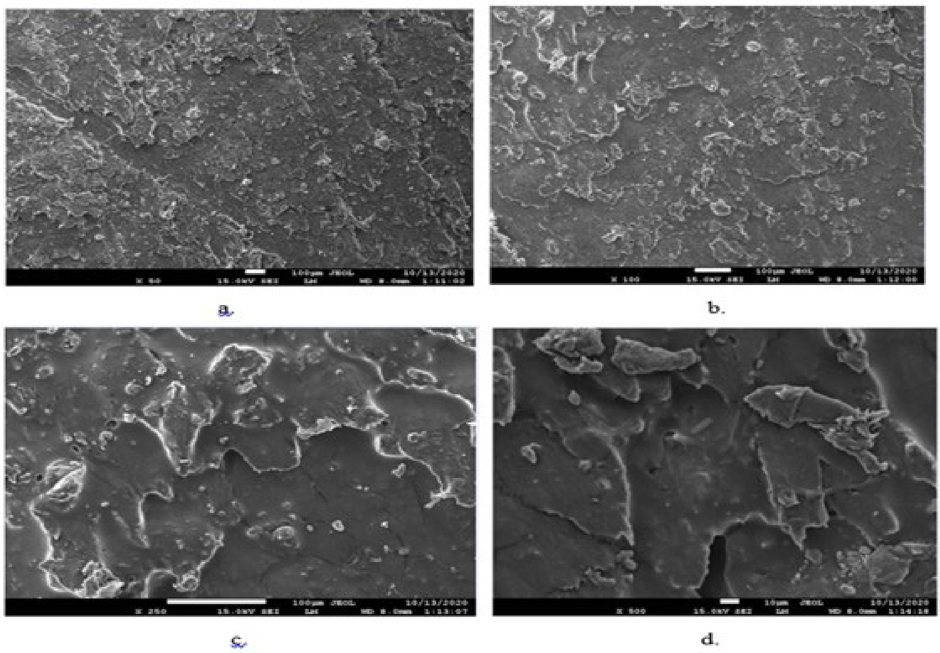


**Figure 5.** Magnified SEM images of epoxy coated wood specimens (EPR). (a) 50x, bar 100 μm; (b) 100x, bar 100 μm; (c) 250x, bar 100 μm; (d) 500x, bar 100 μm.

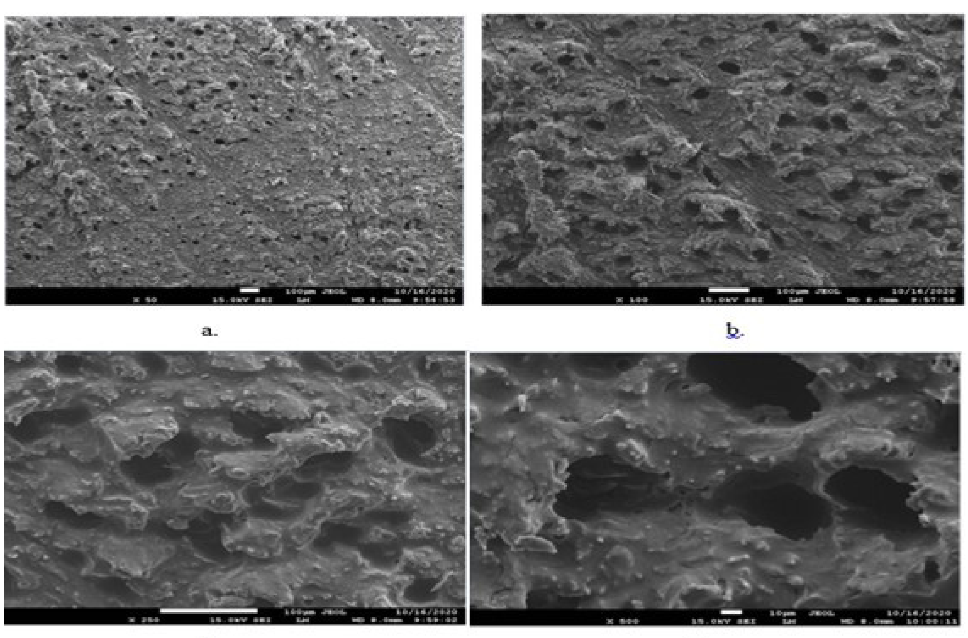


**Figure 6.** Magnified SEM images of impregnated with borax and coated with epoxy resin wood specimens (BX+EPR). (a) 50x, bar 100 μm; (b) 100x, bar 100 μm; (c) 250x, bar 100 μm; (d) 500x, bar 100 μm.

of the bulky distribution of borax in some places within the wood cells could be the other reason. When the SEM images of impregnated with BA+BX and coated with EPR are examined (Figure 8), It can be seen on the pictures that the mixture of BA+BX covers the micropores and cell walls. Especially at x50 magnification, it is seen that some cell voids are formed as a result of impregnation with BA+BX on the wood surface. It can be said that this is caused by the bulky distribution of boric acid and borax at some points. It can also be seen that at x250 magnification the borax and boric acid polymer chains interact and form an interface. While the layered and rough part could be borax in the upper part of the image, the polymer with a flatter and smoother appearance in the lower part could be boric acid. There is no clear phase separation between them. On the other hand, SEM photographs show some broken layers. These broken layers are indicative of penetration or copolymerization in

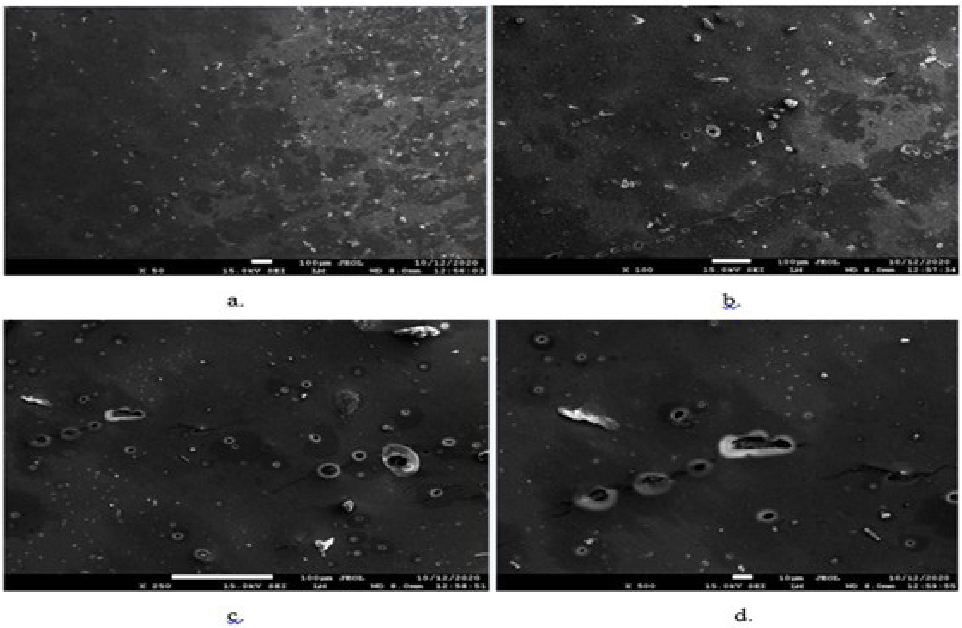


**Figure 7.** Magnified SEM images of impregnated with boric acid and coated with epoxy resin wood specimens (BA+EPR). (a) 50x, bar 100 µm; (b) 100x, bar 100 µm; (c) 250x, bar 100 µm; (d) 500x, bar 100 µm.

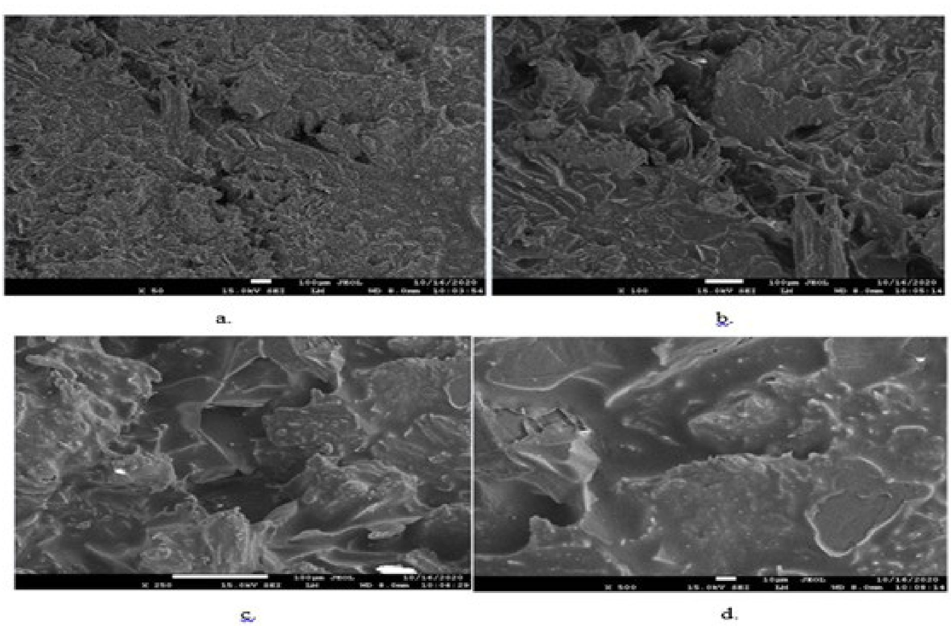


**Figure 8.** Magnified SEM images of impregnated with boric+borax mixture and coated with epoxy resin wood specimens ((BA+BX)+EPR). (a) 50x, bar 100 µm; (b) 100x, bar 100 µm; (c) 250x, bar 100 µm; (d) 500x, bar 100 µm.

the cell walls. This could be due to the preservatives in the cell wall interacting with the hydroxyl groups.

## 4. Conclusions

In this study, Oriental beech wood was impregnated with boron compounds and then was coated with polyurethane/polyurea (PUU) hybrid resin and epoxy resin (EPR).

According to the leaching of boron test results, the coating with polyurethane/polyurea (PUU) hybrid resin gave the most positive results against leaching of boron after impregnation at 10 °C, 22 °C, and 40 °C. Results showed that temperature plays an important role in the leaching of boron characteristics. The lower temperature values resulted in lower leaching of boron rates. When the SEM images of boron compounds impregnated and EPR coated Oriental beech wood are examined, it is seen that the impregnation material covers the cell spaces and cell walls. SEM results showed that the micropores on the BA impregnated Oriental beech wood specimens are less than the other treatment groups.

## 5. Acknowledgements

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