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ORIGINAL ARTICLE

Ecological structure: production of organic impregnation material from mussel shell and combustion

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Abstract

In the research, sea mussel shell (*Chamelea gallina*) powders were impregnated on the samples of Eastern spruce (*Picea orientalis* (L.) Link.) and Anatolian chestnut (*Castanea sativa Mill.*) by dipping method at different concentrations (1%, 5%, 10%, 15%). To investigate the level of use in the wood industry and especially its effects against fire; adhesion, thermogravimetric analysis (TGA), limiting oxygen index (LOI) test measurements were carried out. According to the TGA results, while the residue quantity in the spruce wood sample was the highest at 5%, the residue amount in the chestnut wood sample was the highest at 15%. With increasing amounts of mussel shell powder, the limiting oxygen index values in both wood species samples increased. As a result, it was discovered that impregnating wood samples with mussel shell powder improved the wood's fire resistance.

Keywords: *ecosystem, thermogravimetric TGA analysis, LOI analysis, mussel shell, human/environmental health.*

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1. Introduction

The ecosystem's health, the acquisition and development of new organic preservatives/top surface treatment materials in the wood industry, and the creation of novel impregnation technologies are all critical for humanity's future. Wood can provide valuable services in a variety of sectors when used properly. However, Unfavorable usage environments and fire, can easily cause the wood to deteriorate. As a result, fire retardant treatment of wood is vital process. The proper application of fire retardant chemicals protects the wood from burning and, as a result, increases the wood's service life. The use of fire retardant chemicals does not guarantee that the wood will be fully non-flammable. However, they can make it harder for the wood to ignite and delay the quick spread of fire once it has begun[1,2].

The transport of moisture away from the wood is influenced by permeabiliy or moisture ingress into the wood under the charred layer affects its movement. The intensity of the fire has an impact on the rate of carbonization. The rate of carbonization lowers as the temperature to which the wood is exposed rises[3]. Pabeliña et al.[4] It has been reported that the pyrolysis of cellulose and its interaction with oxygen causes wood combustion and the process of pyrolysis starts with a rise in temperature.

Wood is an essential raw material that has been utilized by humans in a variety of industries since humankind existence. Without the use of chemicals, wood can be made to be durable with some precautions. Chemical operations are also necessary due to the diversity and continuity of risks. Impregnated wood has a significant role in the construction industry due to its economics, aesthetics, and appearance, in addition to its resistance to environmental factors. It has been reported that synthetic/chemical materials in the living environment pose major risks to human and environmental health, thus the creation of organic wood preservatives has become a requirement[5,6].

In the field of impregnation, new approaches are being developed. To provide fluid fluidity and increase retention rate depending on the impregnation process; drying, steaming, engraving, and vacuum pressure application are among the techniques employed in addition to biological, chemical, mechanical, and physical processes. The effectiveness of fire retardant chemicals on wood has been studied in various academic researches. Because of their environmental friendliness, low cost, and fire retardant efficacy, nitrogen and phosphorus-containing compounds are known to be essential criteria in the usage of fire retardant chemicals[7]. The wood was impregnated with boric acid, a borax combination, and a variety of natural tanning chemicals; and it is resulted that natural tanning materials produce positive results in terms of combustion parameters[8]. Carbonization occurs between 500 and 800 °C, and wood components disintegrate into hemicellulose, cellulose, and lignin, respectively[9]. Stone water has been shown to be effective in preventing fire effects in the wood sector in a variety of experiments[10,11].

In this respect, natural mussel shells are grinded and prepared in different concentrations, and chestnut and spruce woods are impregnated by dipping method; physical properties were determined by performing adhesion level/ thermogravimetric (TGA) analysis and Limit Oxygen Index (LOI) tests. In terms of environmental health, it is aimed that this natural preservative will be applied in the wood industry as an impregnation substance and a fire retarder.

2. Materials and Methods

2.1 Material

Within the study's scope, chestnut (*Castanea sativa Mill.*) and spruce (*Picea orientalis* (L.) Link) woods were preferred. As a natural impregnation medium, a solution made from mussel shell was employed. The mussel shell samples were pulverized in a ring mill and then passed through a 63 micron screen to guarantee particle size uniformity before being used to make the solution. From the sieved mussel powder samples, 100 mL of 1 percent, 3 percent, 5 percent, 10 percent, and 15 percent solution were prepared using weights of 1, 3, 5, 10, and 15 g, respectively. Weighed samples were placed in a 200 mL beaker on a magnetic stirrer sequentially. To dissolve the mussel powder, the beaker was filled with 15-20 mL ultrapure 32 percent HCl and 10 mL distilled distilled water. The shell samples were dissolved at 500 rpm and 200 °C temperatures. Then, the solution obtained from the dissolved shell samples was completed to a final volume of 100 mL with the help of distilled distilled water.

2.2 Preparation of experimental samples

For TG Analysis, both wood types were taken to represent the entire main mass, that is, the entire material from which the sample was taken, the materials that remained on the 40 and 60 mesh (250 1847 micron) sieves were ground in a Willey mill and placed in jars[9]. For LOI analysis, wood samples were prepared as 100x10x10 mm.

2.3 Impregnation process

The immersion (medium-term immersion) approach is preferred for impregnation. Ten test samples were processed for each TGA and LOI test. The completely dried samples were kept in the solution for 12 hours and re-weighed, and the measurement was carried out again at 103±2 °C after 24 hours[12].

2.4 Thermogravimetric analysis (TGA)

Thermogravimetric analysis is an analysis method in which the weight loss of polymeric or other samples at a certain temperature rise rate is precisely detected. The thermal deterioration of polymeric materials and the characterization of kinetic events happening during the degradation are the two primary applications of this approach[13]. TGA analysis was carried out according to ASTM E1131-08[14] that has conditions; approximately 10 mg of wood flour passing through 40 mesh, not passing through 60 mesh, 50 mL/min, under nitrogen gas at a flow rate of 57, 10°C/min, with a temperature rise rate of 25 °C to 600 °C. The % weight loss in the sample at the highest temperature point, the time period with the maximum amount of instant weight loss, and the rapid pyrolysis temperature point values were all analyzed as a consequence of the experiment. In addition, the temperature-dependent weight loss curves were graphically obtained.

2.5 Limit oxygen index test (LOI)

All The oxygen index test is applied to assess the flammability of combustible materials. This value represents the quantity of oxygen that must be present in the atmosphere for combustion to proceed; it also determines how the combustion event occurs, as well as the amount of carbon in post-combustion residues and decomposable organic compounds ASTM 2863-09[15]. For the LOI test, 1%, 3%, 5% and 10% impregnated wood material groups were contructed.

3. Results and Discussions

3.1 Solution properties

The solution properties are given in Table 1.

Before and after impregnation, the pH/density did not change. It has been reported in the literature that the acidic/ basic structure has a positive/negative effect on the anatomical and technological properties of the wood.

3.2 Retention (%)

% Retention results are presented in Table 2.

Chestnut wood has the highest retention value (1.88%) and spruce wood has the lowest retention value (0.22%).

3.3 Thermogravimetric (TGA) analysis

3.3.1 Chestnut wood thermogravimetric (TGA) analysis results

Table 3 shows the findings of a thermogravimetric (TGA) examination of chestnut wood, whereas Figure 1, 2, 3 shows the corresponding graphics.

Table 1. Solution properties.

Concentration and Material		Solvent	Temperature (ºC)	pH		Density (g/ml)	
				BI	AI	BI	AI
1%	Mussel shell	(CaCO3) HCI	22ºC	1,38	1,38	0,988	0,988
5%				1,08	1,08	1,006	1,006
10%				0,88	0,88	1,010	1,010
15%				1,00	1,00	0,999	0,999

BI: Before impregnation; **AI**: After impregnation.

When the table and graph were analyzed, it was discovered that a peak at 80°C was generated by the elimination of moisture from the material, and that the primary degradation peak was about 335-342°C. The thermal resistance of the control sample is generated by the degradation of lower hemicelluloses and some extractives; we can conclude that the thermal resistance of this peak value is formed by the degradation of lower hemicelluloses and some extractives. With the amount of additive supplied to the material, the peak height reduced and the decomposition temperature of the readily pyrolysis sample components decreased. The turning point temperature increased from 335°C to 340°C when the amount of additive was raised in the sample compared to the control sample; this could be owing to the amount of additive added.

Table 2. % Retention and duncan test results.

Wood type	Concentration	% Retention	HG
Chestnut wood	%1	0.89	D
	%5	1.61	C
	%10	1.88	A
	% 15	1.70	B
Spruce Wood	%1	0.22	H
	%5	0.31	G
	%10	0.88	E
	%15	0.64	F

HG: Homegeneity groups.

3.3.2 Spruce Wood Thermogravimetric (TGA) analysis results

Table 4 shows the results of the thermogravimetric (TGA) study of spruce wood, with accompanying visuals in Figures 4, 5, 6.

According to the results obtained; with the increased amount of chemicals, the turning point temperature was 388.95 °C, and even with the addition of 1% chemical, the turning point temperature reached 377.63 °C. However, as can be seen in the table and graph, it is seen that rates above 5% do not have a positive effect. It can be concluded that the most suitable ratios for spruce samples are 1% and 5%. The amount of residues at 550°C rose as expected with the addition of the chemical. It's worth noting that impregnating spruce wood is anatomically challenging. When compared to the literature, it has been discovered that identical results have been found we can say that this is due to the anatomical structure of the wood, wood moisture, wood dimensions and wood type.

3.4 LOI analysis

Table 5 shows the results of the limit oxygen index test (LOI), and Figure 7 shows the corresponding graphic.

In comparison to the control samples, the limiting oxygen index percentages increased as the solution % increased in both wood types. The results demonstrate that the highest LOI percentage (26%) of the 5 percent and 10% values of chestnut wood samples was identified; the results show

Table 3. Results of thermogravimetric analysis (TGA) of Chestnut Wood.

Concentration	Initial Temperature (°C)	Turning point (°C)	Final (°C) Temperature	Delta Y (%)	Residual amount at 550 °C (%)
Control	304.00	335.18	350.15	58.41	25.18
%1	309.45	338.64	351.25	58.00	25.50
%5	305.91	341.91	354.03	55.84	27.11
%10	306.15	342.34	356.59	54.66	26.16
%15	302.81	340.61	358.80	53.31	27.01

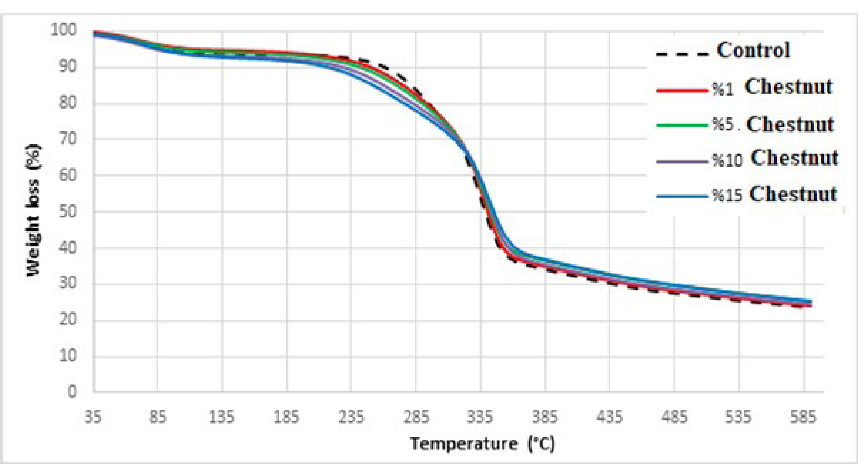

Figure 1. Chestnut wood TGA spectra.

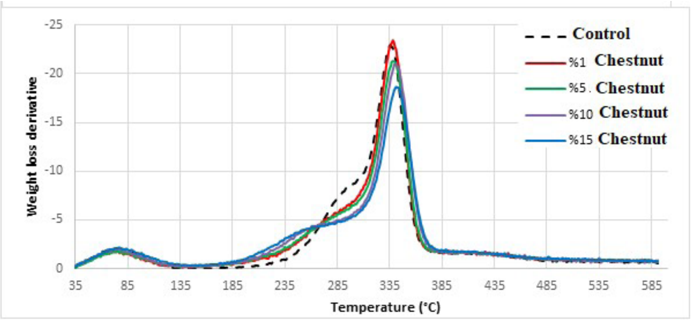

Figure 2. TGA spectrum derivatives of chestnut wood.

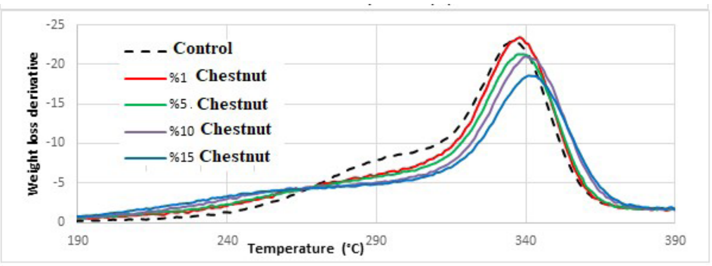

Figure 3. TGA Weight loss derivatives of chestnut wood.

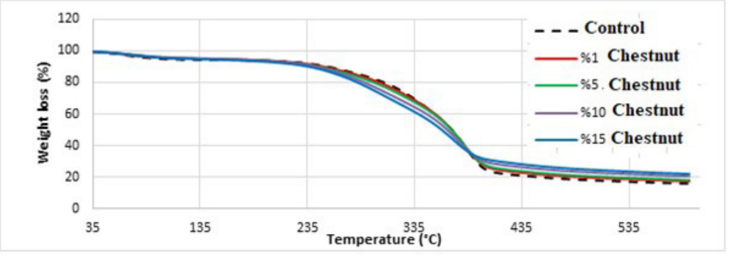

Figure 4. Spruce wood TGA spectra (%).

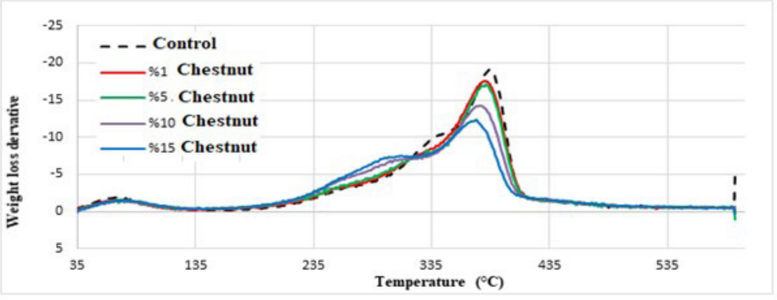

Figure 5. Spruce wood TGA spectra (%).

Table 4. Spruce wood thermogravimetric (TGA) analysis results.

Concentration	Initial Temperature (°C)	Turning point (°C)	(°C) Final Temperature	Delta Y (%)	Residual amount 550 °C (%)
Control	338.56	388.95	397.91	72.29	17.23
%1	323.01	377.63	398.32	71.23	18.43
%5	326.71	376.87	396.06	69.36	19.33
%10	307.04	373.60	397.64	68.10	21.58
%15	294.99	342.56	381.27	62.77	23.36

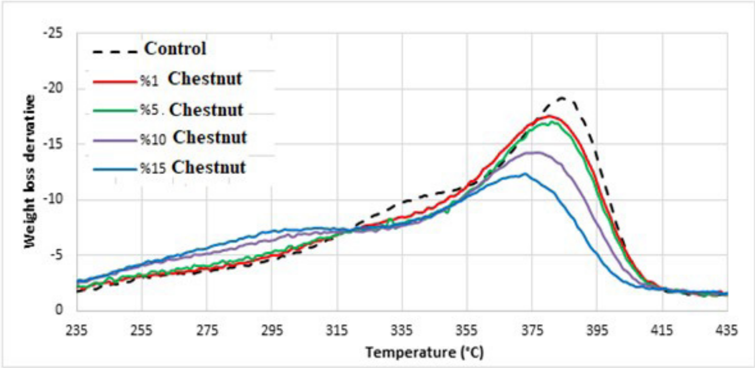

Figure 6. Spruce wood weight loss derivative.

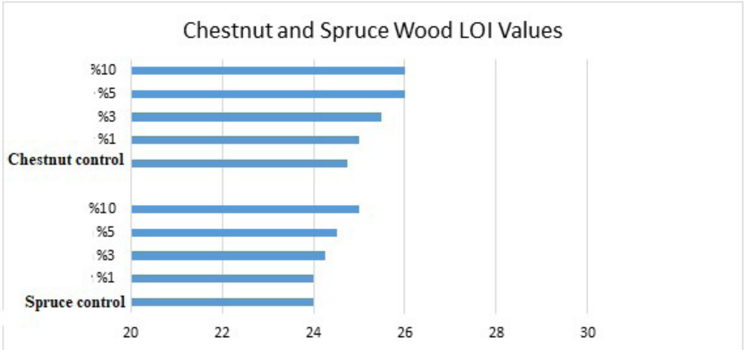

Figure 7. Chestnut and spruce wood samples LOI values.

Table 5. Spruce and chestnut wood LOI results.

Wood type	Solvent Concentration	Limiting Oxygen index (%)
Spruce wood	Kontrol	24.00
	%1	24.00
	%3	24.25
	%5	24.50
	%10	25.00
chestnut wood	Kontrol	24.75
	%1	25.00
	%3	25.50
	%5	26.00
	%10	26.00

parallelism with retention values. It has been resulted that increasing the amount of substance penetrating the wood material raises the wood's fire resistance as well as the oxygen percentage, which is required by another method.

4. Conclusions

The mussel shells ($CaCO_3$), which are found inert in nature and can be used sparingly (poultry feed, etc.), are pulverized and treated with HCl acid, then, the impregnation material is formed by concentration with water at appropriate percentages in the research.

Örs and Keskin[16] Wood's resistance to acids at low concentrations is higher than its resistance to bases. This is due to the fact that cellulose and lignin are acid resistant, whereas hemicellulose and lignin are strong base resistant. 2% solutions of HCl and NaOH and other acids and bases do not cause any significant deterioration in wood at room temperature. However, the degradation rate increases as the concentration rises and the temperature and duration of action rise. Many tree species' resistance drops by 50-75 percent at a concentration of 10% and a temperature of +50 °C. In this respect, bases are more destructive. Coniferous woods are more resistant to chemicals than leafy woods because they contain less hemicellulose. HCl, when applied at low rates, appears to be a favorable choice in terms of wood's acid resistance, according to the research.

Thermogravimetric analysis (TGA) and oxygen limit index (LOI) analyses were performed after impregnation of the produced impregnation material on spruce and chestnut woods. It was determined that the LOI analysis result percentages increased in the same period in parallel with the concentrations of the impregnation material. Furthermore, based on the findings of TGA analysis, it was found that impregnation material with a 5% solution would enough, particularly for spruce wood and the highest result was obtained with a 10% solution on chestnut wood. In both wood types, the amount of residue increased in parallel with the amount of solution. Based on these findings, the ideal proportions for spruce and chestnut are 5% solution combination for spruce and 10% solution mixture for chestnut, taking into account time, labor, and cost.

Hirata et al.[17] The thermal breakdown of cellulose, hemicellulose, and lignin in wood occurs at various temperatures, according to thermogravimetric analysis (TGA) research.

Uysal[18] When wood is heated from room temperature to 100 °C, essentially no chemical reactions occur, and only the moisture and essential oils evaporate in the wood. It has been reported that when the temperature is significantly raised, such as to 200 °C, chemical bonds in wood weaken and degrade as a result of dehydration processes, and above 250 °C, all wood components are exposed to thermal deterioration.

Rowell and Dietenberger[19] The majority of the carbohydrate components were destroyed between 300 and 375 °C, while lignin remained. Hill[20] Ranking of wood components for thermal resistance were given as at low temperatures; hemicellulose < lignin < cellulose, while at high temperatures; hemicellulose < cellulose < lignin. It has been reported that the thermal degradation of hemicelluloses starts at 180-200 °C, and the thermal degradation of cellulose starts at 210-220 °C and reaches the highest level at 270-280 °C and is completed between 300 °C and 340 °C. Thermal degradation was shown to occur between 294-388 °C for spruce wood and 302-342 °C for chestnut wood in the research. The hemicellulose and lignin content of spruce wood is higher than chestnut wood, this situation plays an active role in thermal deterioration at high temperature values.

Goldstein[21] Methanol, acetic acid, furan, and furfural are released during rapid pyrolysis when hemicellulose is degraded, levoglucosan (1,6-anhydro-beta-D glucopyranose) is released when cellulose is degraded, and phenols are released when lignin is degraded. Rapid pyrolysis produces aromatic moieties such as xylenols, guaiacols, cresols, and catasols. It was determined that charcoal remained after carbonization between 400 and 500 °C and the removal of combustible gases. Rowell et al.[22] It has been discovered that the rate of lignin coal generation during burning is higher than that of cellulose and hemicellulose, indicating that lignin has a more heat-resistant structure and a high char formation rate. It is concluded that it reduces the generation of flammable gas, hence preventing further thermal deterioration of the wood.

Akgün[23] The cell wall does not contain extractive chemicals; instead, they are present in the cell gaps. Coniferous woods typically contain high levels of resinous compounds (oil and resin), resulting in high ether extract rates. The amount of this resinous material in spruce is typically less than 1%, although the resin rate in pines has been found to be between 2 and 6 percent. Although the impregnation process is particularly tough in the study, the low amount of resin in spruce wood is one of the reasons why it is preferred over coniferous plants.

Peker and Atılgan[24] It is resulted that; when wood is ignited, it has been discovered that the rate of flame propagation on its surface is substantially governed by the wood's heat conduction and heat capacity, with an inverse relationship between wood density and flame propagation speed. Wang et al.[25] The accelerated decomposition is is owing to the fact that fire retardant bonds like P-O-C (Phosphorus-Carbon) are substantially less stable than C-C bonds in the control sample, and they decompose around 180 °C. Basak et al.[26] This early breakdown reduces dangerous flammable gases generation while also promoting the formation of carbonized coal. Tutuş et al.[9] The thermal degradation of wood and wood components happens between 300 and 500 °C, according to research. The number of residues at 550 °C increased in parallel with the amount of retention in our study in both tree species following TG analysis.

Kartal and Imamura[5] Since the impregnation material that we use has not been tested before, the most effective mixture can be determined by preparing mixtures at certain percentages with other tried (boron, borax, etc.) Even without the use of various chemicals, wood can become durable with some precautions. Nonetheless, chemical activities are required due to the variety and continuity of dangers. Kartal[6] With its economy, aesthetics, and appearance, as well as its resistance to factors, impregnated wood (biotic/abiotic, etc.) plays a significant role in the building industry. According to Özdemir[27], the usage of synthetic/chemical materials in his environment creates major hazards to human and environmental health, the creation of organic wood preservatives is necessary. Finding and developing wood organic preservatives is extremely significant in this study.

In the field of impregnation, new approaches are being developed. To ensure fluid fluidity and boost the retention (retention) rate, methods such as drying, heating, vacuum, and pressure application are utilized in addition to biological, chemical, mechanical, and physical processes, depending on the impregnation procedure. The effectiveness of fire retardant chemicals on wood has been studied in a number of scholarly research. Environmental friendliness, low cost and fire retardant performance are important parameters in the use of nitrogen and phosphorus-containing chemicals as fire retardant chemicals[28]. In line with the literature, it is critical for the industry to provide ecologically acceptable mussel shells, which we have tested in combustion experiments and shown to be effective as a fire retardant, and which are plentiful in nature and also was not completely evaluated before our research.

Peker and Atılgan[29] impregnated with waste tea plant extract, Iroko wood had the lowest percent retention rate (1.58 percent), beech wood had the highest percent retention rate (6.75 percent), iroko had the lowest overall retention at (31,27 kg /m^3), and they determined that the highest total retention value in beech wood (100.65 kg/m3). Flynn[30] The differences in retention rates may have been caused by the wood species, the anatomical structures of the trees, thus the physical properties, the impregnation process and the solution and it was determined that many factors related to the anatomical structure such as heartwood, sapwood, spring wood, summer wood, density, sapwood, tracheid and resin affect the permeability. Both TGA and LOI data indicate favorable results in terms of combustion retardation as the quantity of retention rises, according to the research. When the variations in retention are compared to spruce and chestnut trees, as stated in the literature, spruce wood impregnation is difficult due to its anatomical structure.

Özdemir et al.[31] Polymer chemical structures have a considerable impact on LOI values. LOI values can be determined by the number of oxidizable atomic or molecular groups of polymers. The greater the hydrogen-to-carbon ratio, which impacts the materials' flammability, the more flammable they are. A high LOI rating for any substance suggests that it will be difficult to ignite in the atmosphere. As a result, materials with a LOI value of less than 25% may readily burn in air, whereas those with a LOI value of more than 25% have reported that they are self-extinguishing in air. In our research, we discovered that, as the solution percentages increase, the LOI values in both tree species increased. As a result, the amount of CO_2 emitted into the atmosphere by $CaCO_3$ during burning leaves the environment without oxygen. Therefore, the LOI values increased.

Göker and Ayrılmış[32] when wood is subjected to high temperatures, its structure deteriorates, forming an insulating layer (the carbonized section) that prevents oxygen from entering the burning material and slows further degradation. The cross-sectional dimensions of structural wood pieces determine their load-bearing capability. Thefore, the carbonization rate of the cross-section is a significant determinant for the resistance of structural wood parts, among other things. It prevents the rapid chemical degradation of the material and the diffusion of oxygen from the charred surface into the interior. The fire resistance of structural wood elements is intimately connected to the rate of carbonization of wood and wood-based materials. The carbonization and pyrolysis (heat decomposition) layer impregnated results can be seen in the TG analysis tables of spruce and chestnut wood samples in our experiments. Compared to the control groups, the initial temperatures decreased, that is, it charred earlier and formed the pyrolysis layer, therefore, as mentioned in the literature, it is possible to prevent oxygen from entering the part whose structure is deteriorated. As a consequence, the LOI results also support this. In other words, TG Analysis increased the LOI (Limit Oxygen Index) value due to the fact that the decrease in the initial temperatures, this causes early carbonization and pyrolysis, and the lack of oxygen in the environment.

Çavdar[33] investigated the effects of copper-based and water-based wood preservatives on LOI values on fir wood. CbWPs and new generation wood preservatives have a partial fire retardant effect on wood other than CCB. It was concluded that concentrations of CCB treatment greater than 3% may have a potential fire retardant. Based on our LOI results, it is an example of the literature that refers to the increase in LOI values as the solution percentage increases.

In general, it is known that the mechanical and physical resistance of wood decreases with impregnation. In the percentages of this combination that we developed in another investigation, it is suggested that several mechanical and physical resistance capabilities should be assessed in the same wood species. The physical and mechanical resistance properties of the boards, particularly the adhesion resistance, can be investigated by using the same impregnation material for wooden boards and coatings, in addition to TGA and LOI tests on wooden boards, because we know that impregnation materials have a negative effect on adhesion resistance. After impregnation, the surface gloss and surface treatments of the wood material can be examined. The wood samples we utilized in our studies did not change color after being impregnated. Therefore, they can be utilized in the construction of wood materials (wooden door and window joinery, roofing materials, building materials, toy industry, playgrounds, collective living spaces, wooden structures, insulation boards, furniture) especially for indoors. In nano-technology with different methods other than forest products; in industries such as the textile industry, impregnated products can be considered for also military textile products, especially with fire protection.

As a result, a raw material (mussel shell), which has never been tried before and is abundant in nature, and it was applied for the first time as imperagnated material, a solution was prepared (as a result of melting the ground mussel shell powders with HCl) and its effect on the burning property of wood was evaluated; TGA analysis and LOI analysis showed positive results as flame retardant. When compared to any interior wood preservative on the market, the raw material utilized is far more cost-effective. Furthermore, delivering a raw material that has never been analyzed and left to the ecology as inert in nature will contribute significantly to the country's economy.

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