

Ecological Approach to the Restoration and Preservation of Historical Wood Material: Natural Mussel Shell Impregnation and Combustion (TGA) Analysis

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The goal of this work was to evaluate the fire-protection attributes after treating wood with crude acidic carbonate solutions from a natural source. A broader aim of this project was to find ways to increase the period of usefulness of wooden objects, thus contributing to a sustainable society. In this context, samples of scotch pine (*Pinus sylvestris* L.) wood with insecticides, fungi, insects+fungi, samples were taken, and sea mussel (*Chamelea gallina*) powders were impregnated at different solution concentrations (1%, 3%, 5%) according to ASTM D 1413 76 principles. Thermogravimetric analyses (TGA) were carried out. Although there were no significant changes in the initial temperature, the turning point temperature, or the final temperature values compared to the control groups, the percent weight loss and percent residue amount increased in all the impregnated group periods. Although there was little change in some groups due to the heterogeneity and anatomical structure of the wood, the percentage of residue decreased as the percentage of weight loss increased. Compared to the control sample, the second highest adhesion was observed in 3% cork pine wood (0.81%), weight loss (65.7%), and the amount of residue was 22.0%. Based on the TGA results, mussel shell was found to delay the combustion.

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INTRODUCTION

Understanding the structure of wood is crucial for the care of each wooden artifact. Wooden objects provide details about human existence and culture and are deemed worthy of preservation for the future (Altun 2021). Wooden works with a moisture level of more than 20% show signs of fungal production; this is more common in wooden structures, especially in outdoor conditions. In addition to these problems, willful mistreatment and neglect result in the majority of the destruction of timber cultural property objects. The degree of deterioration may vary depending on the factors of deterioration, tree species, and environmental conditions. Inorganic information, the species of wood, age and environmental circumstances, amount of degradation, and deterioration status are all determined using the methodologies employed for wooden historical objects (Dubey *et al.* 2012; Kantoğlu 2019). The future of humanity depends critically on the health of the ecosystem, the discovery and development of new organic preservatives and top surface treatment materials for the wood industry, and the choice of new impregnation techniques.

Wood may provide vital functions in a variety of sectors when used properly. However, unfavorable usage environments and fire can easily damage the wood. Therefore, treating wood with fire retardants is an essential step. The proper use of fire-retardant chemicals prevents the wood from burning and so indirectly increases the service life of the wood. The wood substance cannot be totally protected from flammability by fire retardant additives. However, they can make it difficult for the wood to ignite and delay the rapid spread of fire after the combustion has started.

In 2022 the author was part of a team project “Ecological structure: production of organic impregnation material from mussel shell and combustion.” Positive results were observed in that study for fire retardancy and inhibition of combustion. Therefore, it is aimed to increase the usage area of the white mussel shell, which is inert in nature, in the wood sector (Tan *et al.* 2022). Humans have used wood in a wide variety of ways throughout history (Candan *et al.* 2012). It is feasible to manufacture wood construction materials such as laminated timber (glulam), cross-laminated wood (CLT), and wood concrete composite (WCC) that is resistant to fungal and insect degradation. Additionally, safety precautions against burning must be performed (Ayrılmış *et al.* 2009). The use of wood items made for outdoor applications that have been impregnated with fire retardants, such as building siding and roof components, is rising. Depending on the environment and the intensity of the wood’s heat exposure, additional wood-related elements that are related to its qualities also influence the ignition temperature of the wood. These variables include, in general, the type of wood, its density, the amount of moisture, the thickness and surface area of the substance, surface absorption, and pyrolysis temperature (White and Dietenberger 1999; Reh *et al.* 1993). Pabelina *et al.* (2012) reported that the pyrolysis of cellulose and its reaction with oxygen cause the burning of wood, and that pyrolysis starts with an increase in temperature. Gao *et al.* (2004) reported that a good flame-retardant impregnation agent initiates the decomposition process at temperatures lower than 300 °C, increases the formation of more pyrolysis and reduces the emission of combustible gas. Wang *et al.* (2010) found that fast pyrolysis of wood material raises the combustion temperature compared to slow pyrolysis, which results in less carbonization and higher combustible gas generation. In slow pyrolysis, there is more carbonization and less flammable gas output. The majority of combustible gases are released from burning wood when cellulose is burned, whereas lignin is burned to generate embers (Özdemir 2020). Alternative techniques for impregnation are being developed. Depending on the impregnation procedure, techniques including drying, steaming, cutting, and vacuum pressure are also used, along with biological, chemical, mechanical, and physical processes, to assure fluid fluidity and increase the retention rate. To find out how well fire-retardant chemicals work on wood, numerous academic investigations have been carried out. In order to employ chemicals containing nitrogen and phosphorus as fire retardant chemicals, it is crucial that they be affordable, environmentally friendly, and effective at putting out fires.

The study's objective is to use historical wooden architectural works, restorations, indoor and outdoor spaces, a variety of geographical conditions, biotic and abiotic conditions, and providing fire retardation for all wood-related structures and works in these and other areas where human and environmental health are prioritized. Particularly, both repair and reuse (reuse) of wood products with fungus, insects, damage, *etc.* will be offered, giving this scenario more context in the current eco-system, where the world's forest resources are depleting for a variety of causes and the economy is becoming more crucial. By figuring out the sources of degradation, structural damages, their causes, and the special

performance qualities of the new materials to be employed in the protective activity, it will significantly improve the protection of wood against the impacts of fire. The continuity of climate change in the ecological structure, the increase in temperatures, and the use of natural (organic) materials rather than the use of synthetic / chemical structures in the fight against them have come to the fore. The aim of the study was to impregnate the solutions prepared from mussel shell (1%, 3%, 5%) on insect, fungus, insect+fungus damaged scotch pine wood and perform both adhesion (%) and thermogravimetric analysis (TGA) analyzes on the damaged wood.

EXPERIMENTAL

Wood Material or Plant Type Material

Within the scope of the study, undamaged, insect-damaged, fungus-damaged, insect+fungus-damaged structures of Scots pine wood (*Pinus sylvestris* L.) were preferred. After grinding the mussel shell (CaCO_3) as a natural impregnation material, it was dissolved with hydrochloric acid at different solution concentrations (1, 3, 5%). In this way, the impregnation material prepared with mussel shell flour was applied by mixing it with distilled water in equal proportions. The solutions can be referred to as “stone water”.

Preparation of Test Samples

Samples were ground in a Wiley mill, and particles remaining on the 40 and 60 mesh (250 to 1847 μm) sieves were placed in containers (Tutuş *et al.* 2010). Samples representing the entire main mass were taken for the TGA. Experimental sample groups were chosen as 10, and a total of 100 experimental samples were used.

Impregnation Process

The test samples were impregnated for 30 min under a pre-vacuum corresponding to 70 cm Hg pressure ASTM D 1413-76 (1986), after which the samples were allowed to diffuse in the solution at atmospheric pressure for the following 30 min.

Thermogravimetric Analysis

According to ASTM E1131-08 (2014), TGA was conducted with approximately 10 mg of wood flour, which does not pass through a 60-mesh sieve, passing through a 40-mesh sieve, under nitrogen gas at a flow rate of 50 mL/min. The temperature was increased from 25 to 700 °C with a temperature increase rate of 10 °C/min. As a consequence of the experiment, the sample's weight loss percentage at the greatest temperature point, the timeframe during which the most weight was lost instantly, and the temperatures at which fast pyrolysis occurred were all evaluated.

RESULTS AND DISCUSSION

Solution Properties

The solution properties are given in Table 1. Before and after impregnation, the pH or density remained unchanged. The anatomical and technological qualities of wood were positively and negatively impacted by the acidic/basic structure of wood, according to the literature.

Table 1. Solution Properties

Solution (%)	Impregnation Material	Solvent Material	Temperature (°C)	pH		Density	
				BI	AI	BI	AI
1%	Mussel shell	Distilled water	22 °C	1.38	1.38	0.988	0.988
3%				1.26	1.26	0.998	0.998
5%				1.08	1.08	1.006	1.006

BI: Before impregnation **AI:** After impregnation

As shown in the table, there were no detectable changes in pH values and densities of solutions measured before and after impregnation. This may be due to working with fresh solution in each impregnation.

Retention (%)

The retention (net dry impregnation amount / %) values are given in Table 2, and the graph of change is given in Fig. 1. The highest amount of adhesion was observed in 3% solution in insect-degraded Scots pine wood (0.87%), and the lowest in 5% solution with insect- and fungal-degraded wood.

Table 2. Amount of Retention (Remaining Amount of Solid Matter in Proportion to the Dry Wood Weight)

Group	Insect/fungal Wood	Concentration	Retention Rate (%)	HG
1	Control (Non-destructive)	-		
2	Insect Degraded	1%	-	-
3	Fungal Degraded		0.65	D
4	Insect + Fungal Degraded		0.60	F
5	Insect Degraded	3%	0.51	G
6	Fungal Degraded		0.87	A
7	Insect + Fungal Degraded		0.81	C
8	Insect Degraded	5%	0.86	B
9	Fungal Degraded		0.53	G
10	Insect + Fungal Degraded		0.64	E

HG: Homogeneous groups; the bolded values are high values

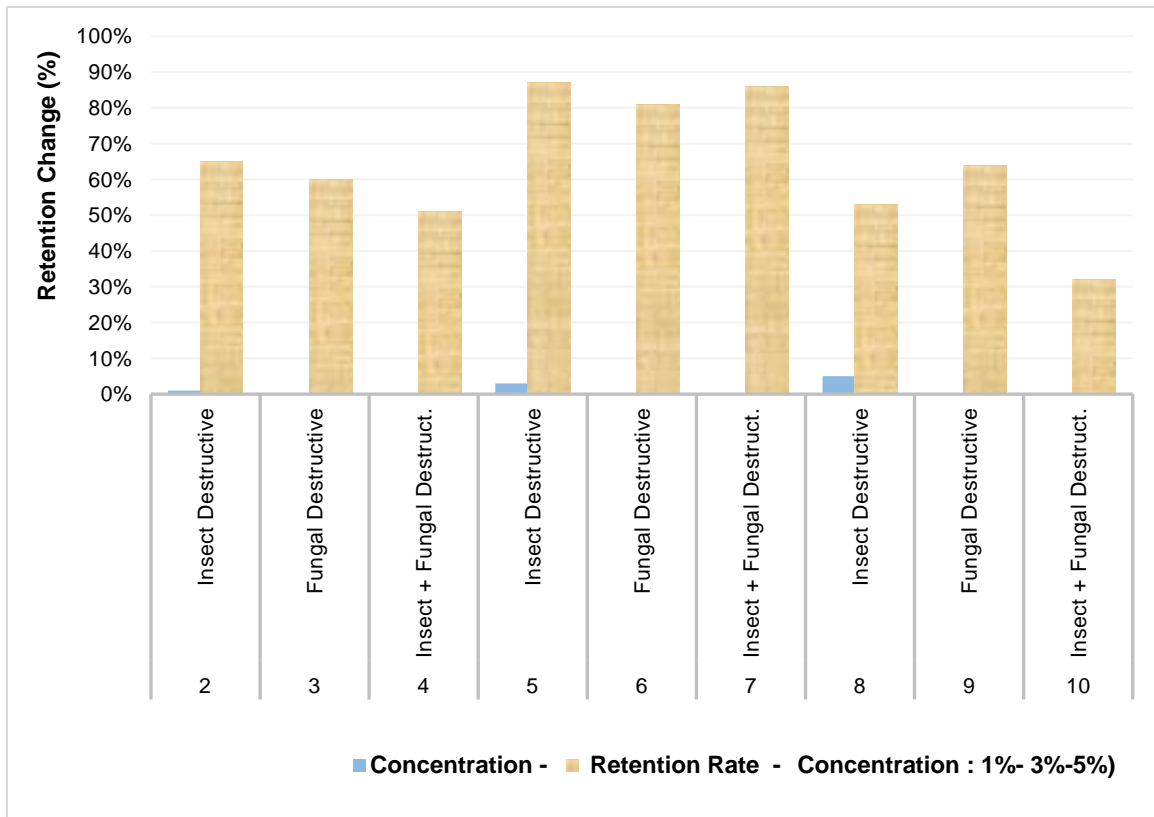


Fig. 1. Retention change (%)

TGA of (Insect, Fungal, Insect+Fungal) Degraded Scots Pine Wood (1%)

The TGA results of the samples impregnated with 1% mussel shell solution of scotch pine wood are given in Table 3, and the graphics related to them are given in Figs. 2,3, and 4.

Table 3. TGA of Scots Pine Wood (Insect /Fungal / Insect+Fungal Destructive)1%

Concentration	Initial Temperature (°C)	Turning point (°C)	Final Temperature (°C)	Delta Y (%)	Residual amount at 600 °C (%)
Control	324.3	382.5	400.6	72.2	18.2
1% CaCO ₃ (Insect degraded)	319.9	377.5	398.6	67.8	19.5
1% CaCO ₃ (Fungal Degraded)	321.4	377.3	397.4	69.8	20.0
%1 CaCO ₃ (Insect+Fungal Degraded)	311.6	373.65	397.9	66.8	20.3

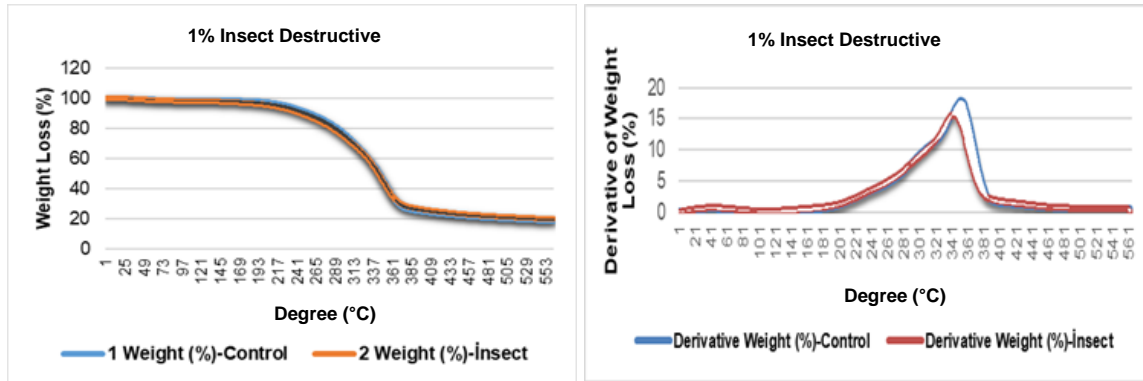


Fig. 2. TGA spectra (Weight Loss and Derivatives (Insect Degraded))

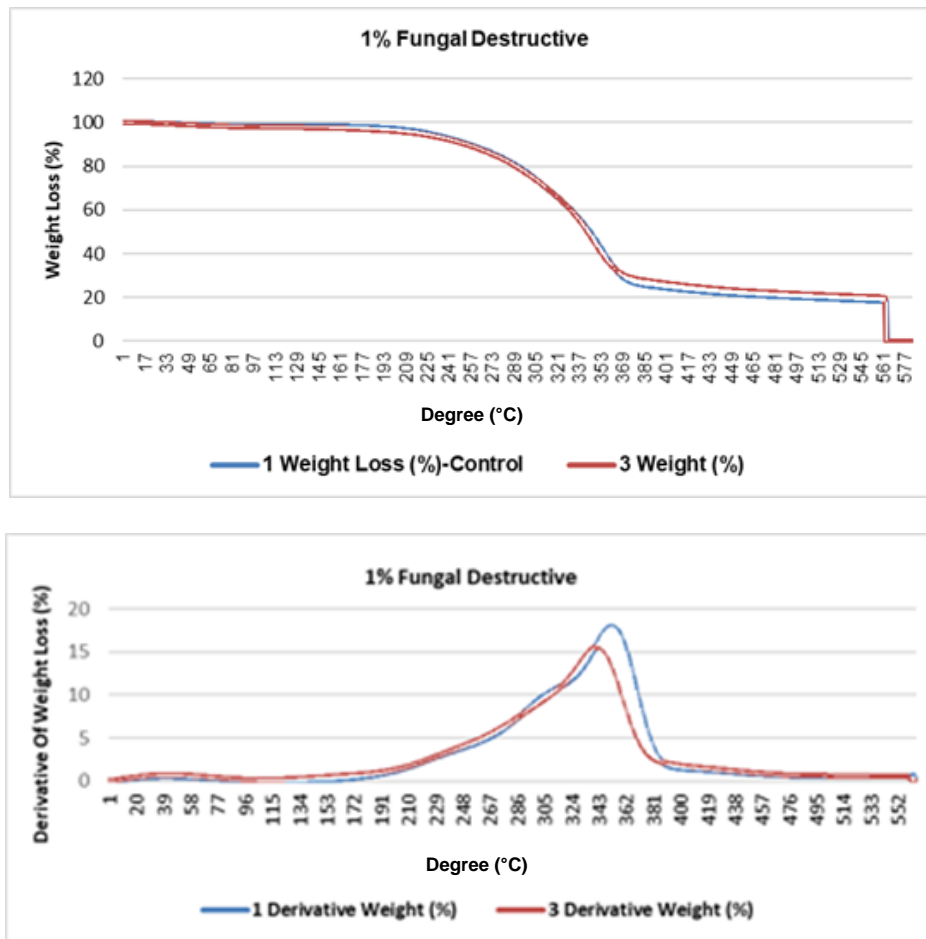


Fig. 3. TGA spectra Weight Loss and Derivatives (Fungal Degraded)

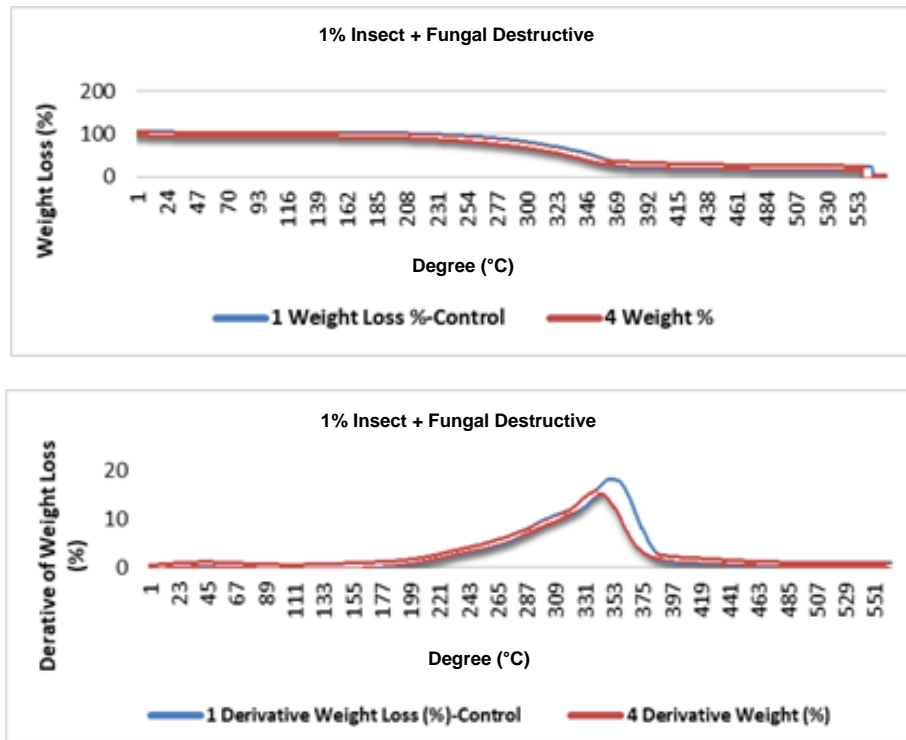


Fig. 4. TGA spectra Weight Loss and Derivatives (Insect+Fungal Degraded)

When the table and graphs are examined, % weight loss highest value (72.2%), lowest % weight loss 1% solution (insect+fungal) was damaged (66.8%) in the control group in the (TGA) process applied up to 600 °c temperature.. The highest amount of residue was (18.2%) in the control group at 600 °C, and the lowest residue amount was determined to be (20.3%) in the 1% CaCO₃ (insect+fungi).

TGA of (Insect, Fungal, Insect+Fungal) Degraded Scots Pine Wood (3 %)

The TGA results of the samples impregnated with 3% mussel shell solution of scotch pine wood are given in Table 4, and the related graphics are given in Figs. 5, 6 ,and 7.

Table 4. TGA of Scots Pine Wood (Insect, Fungal, Insect+Fungal Degraded) 3%

Concentration	Initial Temperature (°C)	Turning point (°C)	Final Temperature (°C)	Delta Y (%)	Residual amount at 600 °C (%)
Control	324.3	382.5	400.6	72.2	18.2
3% CaCO ₃ (Insect Degraded)	314.4	374.7	395.9	64.8	21.4
3% CaCO ₃ (Fungal Degraded)	315.9	372.1	394.1	65.5	21.9
3% CaCO ₃ (Insect+Fungal Degraded)	307.9	373.2	397.8	66.7	21.0

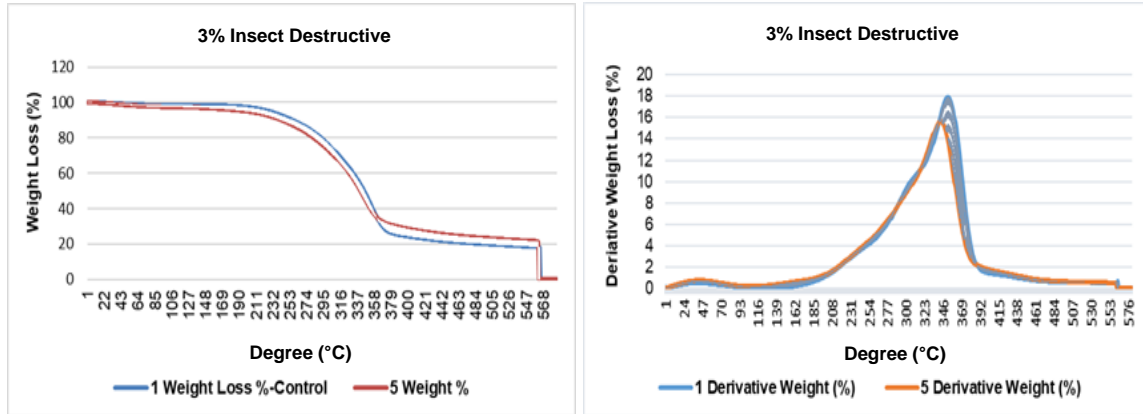


Fig. 5. TGA spectra Weight Loss and Derivatives (Insect Degraded)

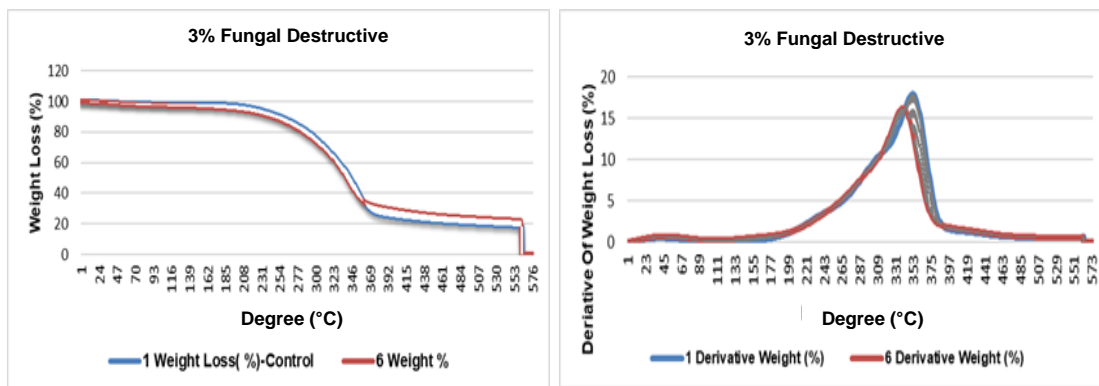


Fig. 6. TGA spectra Weight Loss and Derivatives (Fungal Degraded)

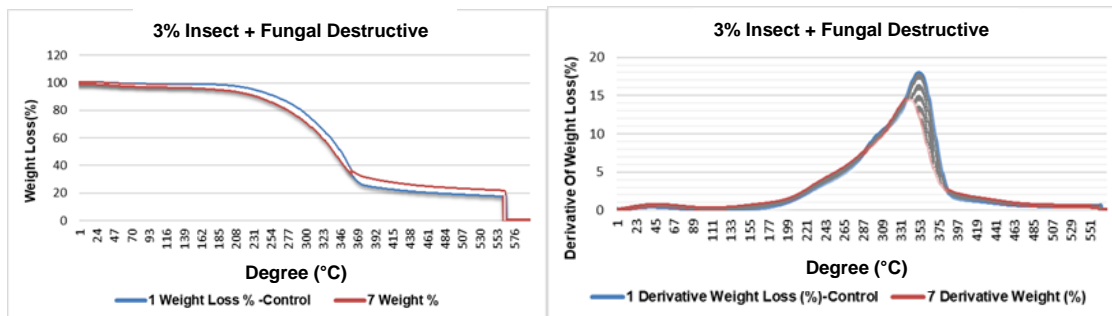


Fig. 7. TGA spectra Weight Loss and Derivatives (Insect+Fungal Degraded)

Table 4 and the graphs indicate in the highest % weight loss control group (72.2%) in the (TGA) process applied up to 600 °C temperature degree, the lowest % weight loss was determined as (66.8%) in the 1% solution (insect+fungal) group. The highest amount of residue was (18.2%) in the control group, at 600 °C, and the lowest residue amount was (21.9%) in the 3% CaCO₃ (with mushrooms) group.

TGA of (Insect, Fungal, Insect+Fungal) Degraded Scotch Pine Wood (5%)

The TGA results of the samples impregnated with 5% mussel shell solution of scotch pine wood are given in Table 5, and the related graphics are given in Figs. 8, 9 and 10.

Table 5. TGA of Scots Pine Wood (Insect, Fungal, Insect+Fungal Degraded) 5%

Concentration	Initial Temperature (°C)	Turning point (°C)	Final Temperature (°C)	Delta Y (%)	Residual amount at 600 °C (%)
Control	324.3	382.5	400.6	72.2	18.2
5% CaCO ₃ (Insect Degraded)	308.3	371.5	393.1	63.4	22.3
5% CaCO ₃ (Fungal Degraded)	314.8	374.1	395.4	65.8	20.8
5% CaCO ₃ (Insect+Fungal Degraded)	301.6	369.6	398.8	62.	22.4

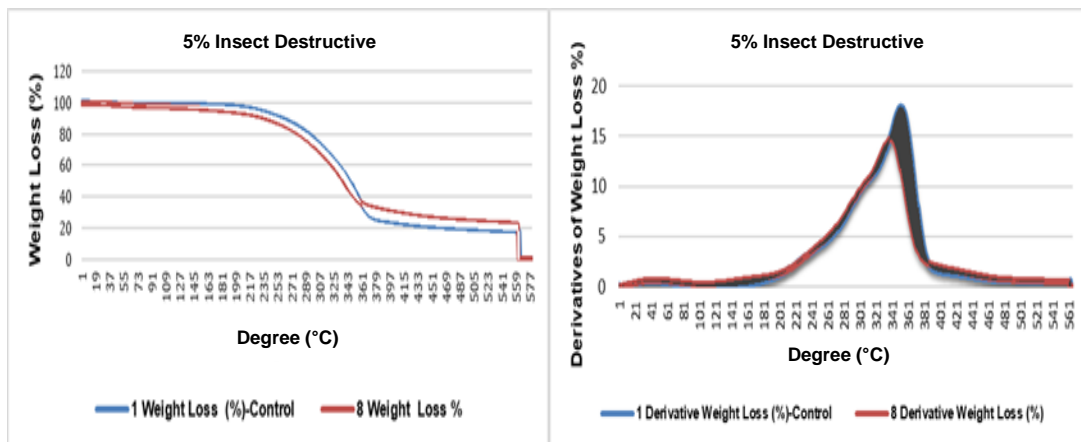


Fig. 8. TGA spectra Weight Loss and Derivatives (Insect Degraded)

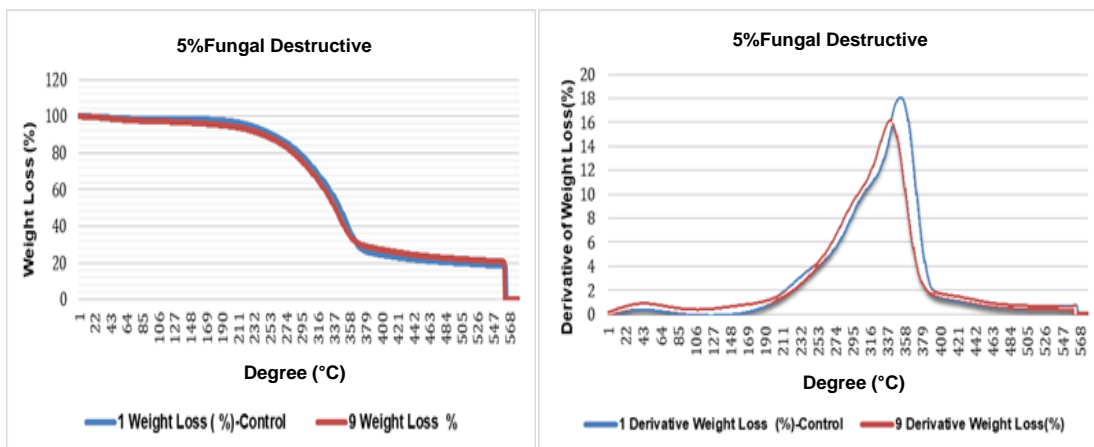


Fig. 9. TGA spectra Weight Loss and Derivatives (Fungal Degraded)

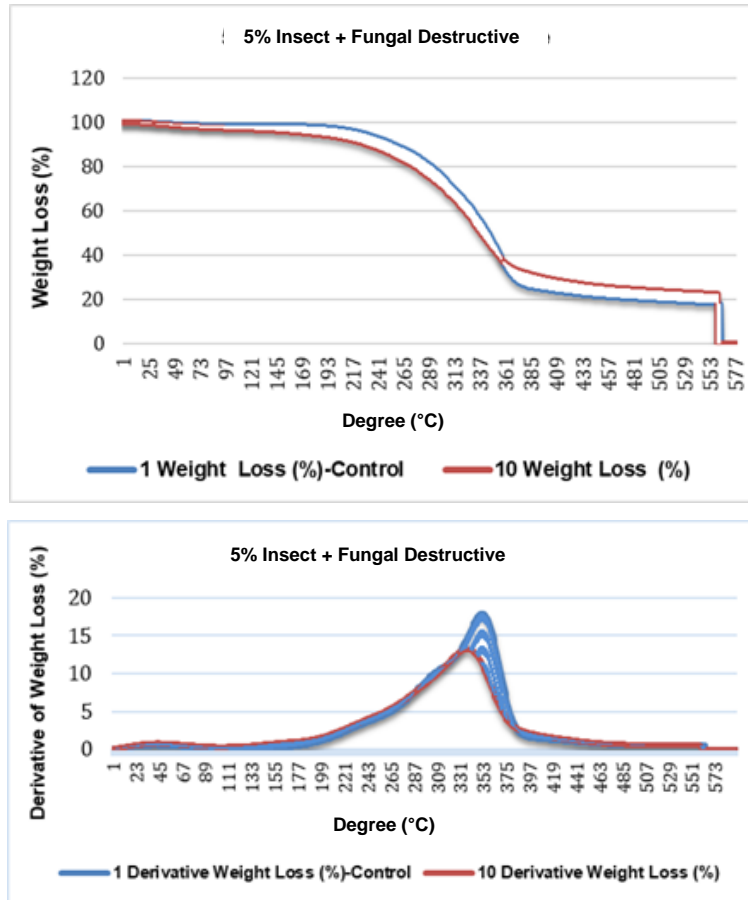


Fig. 10. TGA spectra Weight Loss and Derivatives (Insect + Fungal Degraded)

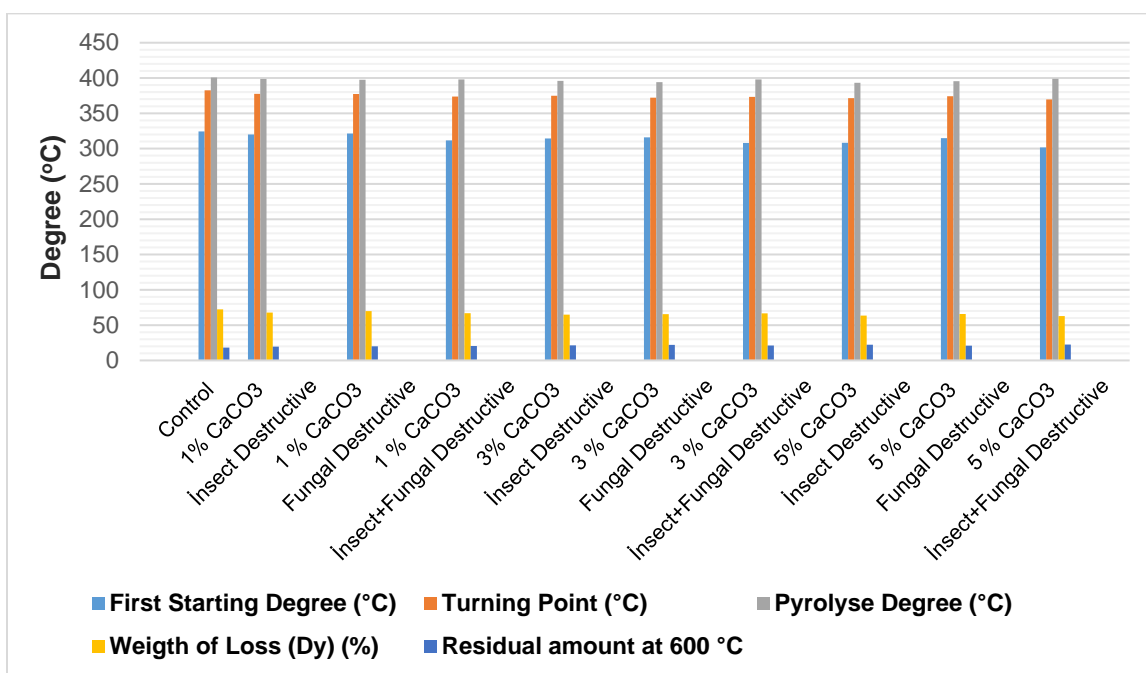
When the table and graphs are examined, in the highest % weight loss control group (72.2%) in the (TGA) process applied up to 600 °C temperature degree, the lowest % weight loss was determined as (62 %) in the 5 % solution (insect+fungal) group, and the lowest residue amount was (22.4%) in the 5% CaCO₃ (insect+fungi) group.

TGA Analysis at All Solution Concentrations

TGA change characteristics of Scotch pine wood by all solution concentrations are given in Table 6 and the change graph for them is given in Fig. 11.

Table 6. Thermogravimetric Analysis Results of Scotch Pine Wood by Concentration Groups (1%, 3%, 5%)

Impregnation/ Concentration	First Starting Degree (°C)	Turning Point (°C)	Pyrolyse Degree (°C)	Weight Loss (% Delta Y)	Residual amount at 600 °C (%)
Control	324.3	382.5	400.6	72.2	18.2
1% CaCO ₃ Insect Degraded	319.9	377.5	398.6	67.8	19.5
1% CaCO ₃ Fungal Degraded	321.4	377.3	397.4	69.8	20.0
1% CaCO ₃ Insect+Fungal Degraded	311.6	373.6	397.9	66.8	20.3
3% CaCO ₃ Insect Degraded	314.4	374.7	395.9	64.8	21.4
3% CaCO ₃ Fungal Degraded	315.9	372.1	394.1	65.5	21.9
3% CaCO ₃ Insect+Fungal Degraded	307.9	373.2	397.8	66.7	21.0
5% CaCO ₃ Insect Degraded	308.3	371.5	393.1	63.4	22.3
5% CaCO ₃ Fungal Degraded	314.8	374.1	395.4	65.8	20.8
5% CaCO ₃ Insect+Fungal Degraded	301.6	369.6	398.8	62.9	22.4

**Fig. 11.** TGA Change Graph by Scots Pine Wood Concentration Groups (1%, 3%, 5%)

All thermogravimetric analyzes were completed up to a temperature of 600 °C. When the percentage of residue at the final temperature is examined in Table 6, the lowest value was 18.2% in the control group and the highest value. It is seen that 5% CaCO₃ impregnated mushroom+insect is 22.4%. Parallel to this, it is seen from the same table that the residue amount of the 5% impregnated mushroom+insect group has the lowest weight loss percentage of 62.4%. Mussel shells (CaCO₃), which are inert in nature and have few uses (poultry feed, *etc.*) were applied as impregnation material that was prepared by concentrating mussel shells (CaCO₃) with water at specific ratios after being pulverized and treated with HCL acid. Compared with bases, wood is more resistant to acids at low concentrations. This is due to the fact that cellulose and lignin are resistant to acids, whereas hemicellulose and lignin are resistant to strong bases. At room temperature, wood does not significantly deteriorate when exposed to solutions of HCL, NaOH, and other acids and bases in concentrations of 2%. However, higher concentration, increased temperature, and prolonged action all cause degradation. Many tree species' resistance drops by 50 to 75% at +50 °C and 10% concentration. In this respect, bases are more destructive. Coniferous woods (softwoods) are more resistant to chemicals than deciduous woods (hardwoods) because they contain less hemicellulose. Based on the the literature, a good option for increasing the acid resistance of wood is HCL, which is applied at modest levels (Hirata *et al.* 1991).

Tongtong *et al.* (2022) reported that TGA-FTIR and SEM analysis demonstrated that the thermal stability and flame retardancy of the impregnated wood samples were improved by generating non-flammable gases and forming an insulating char layer synergistically. From a practical standpoint, this work has significant engineering application value for the development of value-added wood products with high-efficiency fire safety and dimensional stability from relatively low-cost wood. Rowell and Le Van (2005) found that the majority of the carbohydrate components were broken down, although lignin was left behind between 300 and 375 °C. Hill (2006) stated that “Thermal resistance ranking of wood components at low temperatures; as hemicellulose < lignin < cellulose, while at high temperatures as hemicellulose < cellulose < lignin”. Hemicelluloses begins to degrade thermally between 180 and 200 °C, whereas cellulose begins to degrade thermally between 210 and 220 °C, achieves its peak level between 270 and 280 °C, and is finally degraded between 300 and 340 °C. According to the study, the thermal deterioration of spruce and chestnut wood occurred at temperatures between 294 and 388 °C and 302 and 342 °C, respectively. The fact that the hemicellulose and lignin content of spruce wood is higher than that of chestnut wood plays an active role in thermal deterioration at high temperatures.

Vargun *et al.* (2019) reported that based on the TGA curve for untreated beech wood, weight loss takes place at three distinct steps. The impregnation of inorganic salts resulted in higher char yields and additional thermal degradation steps were identified. The highest char yield (80%) was obtained from (NH₄)₂ HPO₄ -K₂ HPO₄ mixture impregnated sample. Salt mixtures containing phosphates ((NH₄)₂ HPO₄ and K₂ HPO₄) have remarkable effects on thermal stability of beech wood, whereas NH₄ Cl salt lowered the decomposition temperatures and char yield. Wang *et al.* (2004) found that the bonds in fire retardants such as P-O-C (phosphorus-carbon), which typically breakdown at 180 °C, are substantially less stable than the C-C bonds in the control sample, which is the cause of the faster degradation. Tutuş *et al.* (2010) discovered that wood and wood-related components thermally decomposed between 300 and 500 °C. In the present study, after TGA analysis in both tree species, the amount of residues at 600 °C increased parallel to

the amount of retention. According to the study, TGA data show improved fire retardancy as the amount of retention increases.

Dönmez (2014) investigated the effects of copper-based and water-based wood preservatives on LOI values on fir wood. It has been discovered that CbWPs and new generation wood preservatives have a partial fire retardant impact on wood other than CCB, leading researchers to the conclusion that CCB treatment concentrations larger than 3 percent may have a possible fire retardant effect. Again, based on the present LOI results, it is an example of the literature that refers to the increase in LOI values as the solution percentage increases. Since the impregnation material used in this work has not been tried before, preparing certain percentages of mixtures with other tried (boron, borax, *etc.*) substances with most effective mixture can be determined by TGA and LOI analysis. In keeping with the literature, it is crucial for the industry to introduce environmentally friendly mussel shells, which are abundant in nature and cannot be fully assessed but which we have tested, particularly in combustion tests, and which have shown promise as a fire retardant. Yang *et al.* (2016) found that the effectiveness of fire retardant chemicals on wood has been the subject of numerous academic investigations. When using chemicals containing nitrogen and phosphorus as fire retardants, it is crucial to consider their environmental friendliness, low cost, and fire retardant effectiveness. Tan *et al.* (2022) TGA increased the LOI value as a result of the early carbonization and pyrolysis that the decrease in starting temperatures induced, which led to a lack of oxygen in the atmosphere.

CONCLUSIONS

1. Impregnation causes a reduction in the mechanical and physical resistance of wood. Some mechanical and physical resistance features in the same tree species can be attributed to the percentages of this mixture that were prepared in a different investigation.
2. After impregnation, the wood material's surface treatments and appearance attributes can be examined. The wood samples used in this research don't change color after being impregnated; thus they can be applied to make indoor-specific wood products like wooden door and window joinery, roofing materials, building materials, toys, playgrounds, communal living spaces, and furniture.
3. By evaluating a raw material (mussel shell) that has never been tried before and abundantly found in nature (as a result of melting the ground mussel shell powders with HCL), a solution is prepared. Regarding the burning property, which is one of the negative aspects of wood according to its intended use, positive results were obtained as a flame retardant retardant after TGA analysis.

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