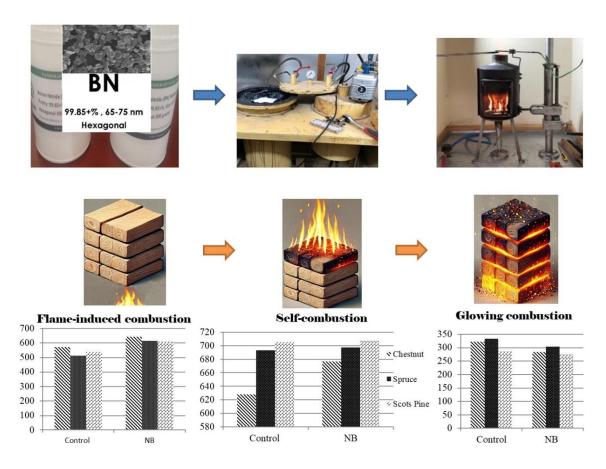
Effect of Nano Particulate Preservation Materials on the Combustion Temperatures of the Wood

Sekip Sadiye Yasar, a Eyup Cicek, b and Mehmet Yasar a,*

* Corresponding author: myasar@aku.edu.tr

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GRAPHICAL ABSTRACT



Effect of Nano Particulate Preservation Materials on the Combustion Temperatures of the Wood

Sekip Sadiye Yasar, Eyup Cicek, and Mehmet Yasar a,*

It is acknowledged that boron, ammonium, and nitrogenous compounds, which are used today as fire retardants, cause an increase in the hygroscopicity of wood materials. In this study, Scots pine (*Pinus sylvestris* L.), Anatolian chestnut (*Castanea sativa* Mill.), and Eastern spruce (*Picea orientalis* Link) woods were impregnated with 1.5% nano-sized hexagonal boron nitride (NB) according to ASTM D1413-76 (1976) standards. Flame-induced combustion (FIC), self-combustion (SC), and glowing combustion (GC) temperatures were determined. The highest retention amount was measured in spruce and the lowest was in chestnut among the wood samples taken for testing and measurement. When compared with the control samples, NB application caused an increase in SC and FIC temperatures (at higher rates). According to the glowing combustion temperature control samples, an increase was observed in chestnut and spruce and a decrease was observed in Scots pine among the samples applied with NB.

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Keywords: Wood; Nano hexagonal boron nitride; Combustion temperatures; Flame-induced combustion; Impregnation

Contact information: a: Afyon Kocatepe University, Department of Industrial Design, Afyonkarahisar, 03400, Turkey; b: Gumushane University, Department of Environmental and Forest Sciences, Gumushane, Turkey; * Corresponding author: myasar@aku.edu.tr

INTRODUCTION

Wooden material is an engineering and construction resource that has been in use since the earliest times in human history. Although the variety of materials increases in parallel with technological developments, wood is still widely preferred due to its superior features such as being light, easy to process, its aesthetic appearance, being renewable and environmentally friendly, having heat-sound insulation, and its high resistance to collapse in fire and earthquake (Kurtoglu 2000). In addition to all these positive characteristics, wood material can be negatively affected by variables including termites, bacteria, fungi, insects, sunlight, seasonal changes, rain, frost, humidity, chemicals, and fire, which can damage its physical and chemical structure. Studies are being conducted to increase the resistance of wood to the combustion and factors in the usage area (Rowell and Dietenberger 2005; Esteves and Pereira 2009; Percin *et al.* 2015; Fidan *et al.* 2016b; Reinprecht 2016; Yasar *et al.* 2016; Sandberg *et al.* 2017; Godovčin *et al.* 2022).

Wood preservatives can increase the service life of the wood material by protecting it from fire, mechanical wear, weather conditions, biological pests, and physical and chemical degradation. Creosote, pentachlorophenol, chromium-copper-arsenic (CCA), acid-copper-chromium (ACC), alkaline-copper-quat (ACQ), copper-chromium-boron (CCB), copper-chromium-phosphate (CCP), and copper -azole compounds are widely used wood preservatives. It is of great importance that today's preservatives are resistant to

environmental conditions, can bond well to wood materials, have a good level of effectiveness, and do not harm the environment and are not harmful to human health. The Environmental Protection Agency (EPA) stated that it is appropriate to make label changes by taking mitigating measures for arsenic, pentachlorophenol, and creosote (Archer and Lebow 2006; EPA 2006; Ibach 2013; Mai and Militz 2023).

In recent years, new "wood modification" methods have been used to adjust the properties of the wood material and increase their service life in parallel with the acceleration of technological developments. Thermal (Thermowood, Platowood), chemical (acetylation, furfurylation, resin impregnation), coating with layer-forming agents, and impregnation applications are used as common modification methods (Yildiz *et al.* 2006; Xing and Li 2014; Reinprecht, 2016; Sandberg *et al.* 2017; Mai and Militz 2023). Ammonium, nitrogen, sulfur, silica, and boron compounds, which are widely utilized today to delay the combustion of wood materials, prevent thermal deterioration by preventing the production of flammable gases with the help of a charcoal layer that they form (Park *et al.* 2017; Deng *et al.* 2022). Studies have been conducted to prevent these compounds from scouring onto wood. Nanotechnology is a branch of science that studies substances in the size range of 1 to 100 nm. In parallel with technological developments, nanotechnology has found use in many sectors (Siegel 1999).

In this study, the combustion properties of wood material were examined using hexagonal boron nitride (NB), which is one of the nano-sized substances that is thought to penetrate deeply into the wood. For this purpose, Anatolian chestnut (*Castanea sativa* Mill.), Scots pine (*Pinus sylvestris* L.), and eastern spruce (*Picea orientalis* Link) woods were impregnated with 1.5% nano NB according to ASTM D1413-76 (1976) standards. Flame-induced combustion (FIC), self-combustion (SC), and glowing combustion (GC) temperatures were examined according to ASTM E160-50 (1975) standards.

EXPERIMENTAL

The wood materials used in the research were obtained from Scots pine (*Pinus sylvestris* L.) and eastern spruce (*Picea orientalis* Link) trees, which are among the coniferous trees, and Anatolian chestnut (*Castanea sativa* Mill.), which were randomly selected from first class timber. Samples were chosen carefully in accordance with TS 2470 (1976) standards from normal and regularly grown wood material pieces that did not contain resin and knots and had regular fibers.

The nano-sized hexagonal boron nitride used in the study was 99.85% pure with dimensions between 65 and 75 nm (Nanografi 2024). Hexagonal boron nitride is a material with low density and low thermal expansion and better thermal conductivity than steel (Haubner *et al.* 2002; Ebin 2007; Oz 2016). It has a wide application area in the fields of chemistry, metallurgy, high temperature technology, electrotechnics, and electronics. Hexagonal boron nitride can be used pyrolytically as a powder coating and as a preservative for composite materials in sprayed, hot-pressed, dense forms (Paine and Narula 1990; Lelonis *et al.* 2003).

Randomly selected timber was subjected to climatization at 20 ± 2 °C and $65 \pm 3\%$ relative humidity until a constant moisture content of 12% was achieved before the rough cutting. Test pieces were prepared in dimensions of 13 x 13 x 76 mm³ (radial x tangential x length) according to ASTM E160–50 (1975) standards. Nano hexagonal boron nitride was penetrated wood pieces with 1.5% concentration with the vacuum-pressure method according to ASTM D1413-76 (1976) principles. In impregnation application, a pre-

vacuum of 600 mm Hg was applied first to the test pieces for 120 min, and then a pressure of 7 atmospheres was applied for 240 min.

The impregnated sample pieces were kept in the conditioning room at 20 ± 2 °C and relative humidity of $65 \pm 3\%$ until they reached a constant weight so that the solvent would evaporate. To measure the retention rate of NB during the impregnation process, the samples were weighed before and after the impregnation process.

In each combustion period, 24 pieces were stacked on a wire stand, and a total of 72 pieces were used in 3 replicates for each test. A total of 432 pieces were combusted for each wood group (3), including control and NB application (2).

Samples were prepared from the sapwood part of randomly selected first class material that had uniform fiber, did not have knots or cracks, and had no color and density difference, and the annual rings were upright to surface ISO 3129 (2019). The retention amount of the nano hexagonal boron nitride used in the study was calculated as in Eq. 1 after the impregnation application was conducted and the solvent material evaporated,

$$R = \left[\frac{G \times C}{V} \right] \times 10 \left(\frac{kg}{m^3} \right) \tag{1}$$

where G is T_2 - T_1 , T_1 is the weight of the test sample before impregnation (g), T_2 is the weight of the test sample after impregnation (g), V is the sample volume (cm³), and C is the solution concentration (1.5 %).

To determine the weight loss during combustion, each experiment group was weighed with a precision scale and placed on a wire stand. The weight losses of the woods used in the study during combustion were obtained for chestnut (88.3%), Scots pine (87%), and spruce (86.8%), respectively. According to the application type, 87.5% was obtained in control samples and 87.2% in NB. After the impregnation application, measurements were recorded on the combustion test device shown in Fig. 1, in accordance with ASTM E160–50 (1975) principles, to measure the combustion properties of the samples.



Fig. 1. Combustion test device

While the samples were being placed in the combustion device, the Meeker burner located under the combustion device was positioned according to the center of the flame output. The combustion experiment was started by setting the gas pressure to $0.5~{\rm kg/cm^2}$ and the flame mechanism to $25 \pm 1.3~{\rm cm}$ before placing the stack.

Stages in combustion:

- "Flame-induced combustion (FIC)" for 3 min upon starting the flame source,
- "Self-combustion (SC)" by turning off the flame source and self-combustion process

• After self-combustion ends, "glowing combustion (GC)".

To monitor the combustion stages, temperature changes were measured with the thermometer in the combustion mechanism for every 15 s in flame-induced combustion and 30 s in other combustion stages. At the end of the combustion analyses, combustion temperatures (°C) were determined.

The software MS Excel 2010 (Microsoft Corp., Redmond, WA, USA) was used for data evaluation and MSTAT-C 2.1 statistical program (Michigan State University, East Lansing, MI, USA) was utilized for statistical analysis, and multiple analyses of variance (ANOVA) were conducted between all groups. If the factor effects were significant with a margin of error of $p \leq 0.05$, comparisons were made using the Duncan test. Variance analysis was conducted using SPSS 20.0 (2011) statistical program (Sun Microsystems, Inc., Network Circle, Santa Clara, CA, USA) package software to determine whether the changes in the combustion temperature data measured at the end of the experiment were significant. Mean values were compared using the least significant difference (LSD) test.

RESULTS AND DISCUSSION

The retention values of the nano-sized hexagonal boron nitride used in the study for the wood materials are shown in Table 1.

Table 1. Retention Amount (kg/m³)

Wood Type	R (kg/m³)
Anatolian Chestnut	45.7
Scots Pine	70.5
Eastern Spruce	49.4

The highest retention amount was determined in Scots pine, then in Eastern spruce, and the lowest was in Anatolian chestnut. Parallel values were obtained in retention rate values (Bozkurt *et al.* 1993).

The results of multiple variance analysis regarding the effect of wood species and application type on temperature values in flame-induced combustion are given in Table 2.

Table 2. Variance Analysis Belonging to FIC Process

Source	Degrees of Freedom	Sum of Squares	Mean Squares	F- value	Sig.
Wood Type (A)	2	6874.009	3437.005	0.676	0.527
Application (B)	1	30370.718	30370.718	5.974	0.031*
Interaction (AB)	2	1060.314	530.157	0.104	0.902
Error	12	61004.087	5083.674		
Total	18	6173718.472			

^{*:} Statistically significant difference α ≤ 0.05

As seen in Table 3, the one-way effect of the application type on flame-induced combustion temperature values was found statistically significant, and other interactions were found insignificant. Duncan test was conducted to determine among which groups the difference was significant (Table 3).

Table 3. Temperature Values (°C) in FIC Process According to Application Type

Application	Maana	95% Confidence Interval		
Application	Application Means		Upper Limit	
Control	540	488	591.6	
NB	622	570.2	673.8	

Std. Error:: 23.767

In flame-induced combustion, the highest temperatures by the application type were determined in the samples where NB was applied (622 °C). The average, maximum, and minimum values for flame-induced combustion are given in Table 4.

Table 4. Average Temperature Values in FIC Process According to Wood Type and Application Type

Parar	neters	Average Temperature Values in FIC (°C)	
Wood Type	Application	Means	Std. Error
Chestnut	Control	572.4	6.26
Chestriat	NB	643.5	1.90
0	Control	510.5	157.65
Spruce	NB	614.4	12.59
Scots Pine	Control	536.6	73.37
	NB	608.1	7.99

In average values, the highest temperatures were obtained in Chestnut+NB (643.5 °C) and the lowest was in spruce control samples (510.5 °C). The graph of temperature changes in flame-induced combustion is shown in Fig. 2.

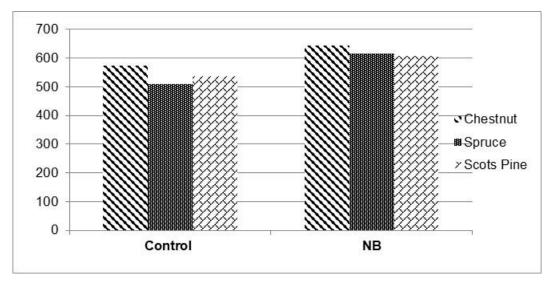


Fig. 2. Average temperature values (°C) in flame-induced combustion

In flame-induced combustion, the combustion degrees in the NB applied samples were measured with a maximum increase of 20.3% in spruce, 13.3% in Scots pine, and 12.4% in chestnut when compared with the control samples. The results of multiple

variance analysis regarding the effect of wood type and application type on temperature values in self-combustion are given in Table 5.

Table 5. Variance Analysis of the Temperature Values in SC Process

Source	Degrees of Freedom	Sum of Squares	Mean Squares	F- value	Sig.
Wood Type (A)	2	9705.337	4852.669	8.375	0.005*
Application (B)	1	1525.452	1525.452	2.633	0.131
Interaction (AB)	2	2087.171	1043.586	1.801	0.207
Error	12	6953.151	5083.674		
Total	18	8461677.058			

^{*:} Statistically significant difference α ≤ 0.05

As observed in Table 5, the one-way effect of wood type on temperature values in self-combustion was found to be statistically significant, and other interactions were found to be insignificant. Duncan test was performed to determine among which groups the difference was significant (Table 6).

Table 6. Average Temperature Values in SC Process According to Wood Type

Mood Turns	Maana	95% Confidence Interval		
Wood Type	Means	Lower Limit	Upper Limit	
Chestnut	652.6	631.2	674	
Spruce	695.4	674	716.9	
Scots Pine	706.4	685	727.8	

In self-combustion, the highest temperatures were obtained in Scots pine (706.4 °C) and the lowest was in chestnut (652.6 °C). The average, maximum and minimum values for self-combustion are given in Table 7.

Table 7. Average Temperature Values in SC Process by Wood Type and Application Type

Parameters		Average Temperature Values in SC (°C)		
Wood Type	Application	Means	Std. Error	
Chestnut	Control	628.2	10.61	
Criestriut	NB	677	8.32	
C	Control	693.3	32.94	
Spruce	NB	697.6	14.76	
Scots Pine	Control	705.3	43.84	
	NB	707.5	8.34	

Std. Error: 13.898

The highest average temperatures were obtained in Scots pine + NB (707.5 $^{\circ}$ C) and the lowest was in chestnut control samples (628.2 $^{\circ}$ C). Temperature changes during self-combustion are shown in Fig. 3.

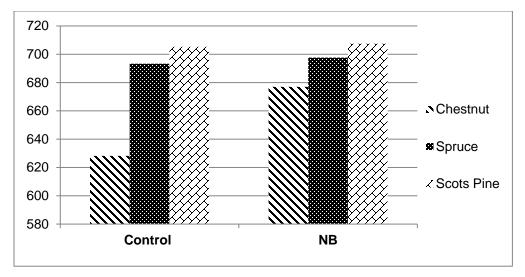


Fig. 3. Average temperature values in self-combustion (°C)

When compared with the control samples, the highest increase in self-combustion was measured in chestnut with 7.8%, spruce with 0.6%, and the lowest in was in Scots pine with 0.3% among the samples impregnated with NB. The results of multiple variance analysis for the effect of wood species and application type on temperature values in glowing combustion are given in Table 8.

Table 8. Variance Analysis of Temperature Values in GC Process

Source	Degrees of Freedom	Sum of Squares	Mean Squares	F- value	Sig.
Wood Type (A)	2	4566.800	2283.400	1.680	0.227
Application (B)	1	3040.418	3040.418	2.237	0.161
Interaction (AB)	2	522.240	261.120	0.192	0.828
Error	12	16308.628	1359.052		
Total	18	1644076.434			

^{*:} Statistically significant difference α ≤ 0.05

Table 9. Average Temperature Values in GC Process by Wood Type and Application Type

Pa	rameters	Average Temperatu	ure Values in GC (°C)
Wood Type	Application	Means	Std. Error
Observat	Control	321.3	15.43
Chestnut	NB	283.3	9.45
0	Control	332.3	74.92
Spruce	NB	304.1	27.92
Onata Dina	Control	285.3	35.28
Scots Pine	NB	273.5	13.80

As can be observed from Table 8, all interactions in glowing combustion temperature values were found to be insignificant. Maximum and minimum values for glowing combustion are given in Table 9.

The highest average temperatures were obtained in the spruce control samples (332.3 °C), and the lowest was in the Scots pine NB samples (273.50 °C). The application of NB, which showed high temperatures in the FIC and SC stages, may have caused cooling in the GC (Ors *et al.* 1999; Temiz *et al.* 2008; Fidan *et al.* 2016a; Gan *et al.* 2020). Temperature changes during glowing combustion are shown in Fig. 4.

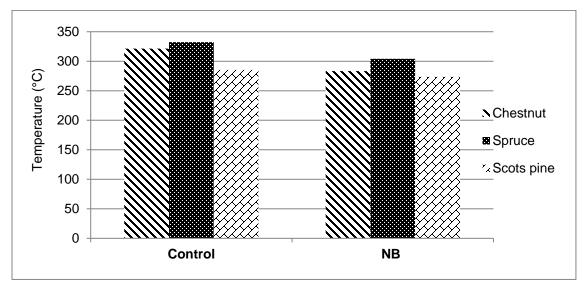


Fig. 4. Average temperature values in glowing combustion (°C)

For the control samples, the glowing combustion temperature showed a decrease of 11.8% in chestnut, 8.5% in spruce, and 4.1% in Scots pine samples to which NB was applied.

CONCLUSIONS

The study aimed to determine the combustion properties of wood species impregnated with nano hexagonal boron nitride (NB). The selected species were Scots pine (*Pinus sylvestris* L.), Eastern spruce (*Picea orientalis* Link), and Anatolian chestnut (*Castanea sativa* Mill.).

- 1. The application of NB significantly increased the flame-induced combustion (FIC) temperatures across all wood species. The highest FIC temperature recorded was in Anatolian chestnut, which was likely due to its high extractive content.
- 2. Scots pine exhibited the highest self-combustion (SC) temperatures, which may be attributed to its resin content. NB-treated samples showed increased SC temperatures compared to control samples, with chestnut showing the most significant increase.
- 3. The NB treatment resulted in lower glowing combustion (GC) temperatures across all wood species, suggesting that the higher temperatures in the FIC and SC stages led to a cooling effect during GC. However, it led to reduced GC temperatures, which could be beneficial in certain fire retardant applications. Future studies should explore the long-term effects of NB treatment and its implications for various industrial applications.

In conclusion, nano hexagonal boron nitride impregnation improved the fire resistance of wood materials, making it a promising treatment for enhancing the safety and durability of wood used in construction and other industries. Further research is needed to fully understand its potential and optimize its application methods.

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