Improved Performance of Brutian Pine Wood *via* Impregnation with Nanoclay

Abdullah Beram *

This study aimed to evaluate the effects of brutian pine (*Pinus brutia* Ten.) wood impregnation with nanoclay in different concentrations on the chemical and thermal changes, and their impact on the physical and mechanical properties. The samples were impregnated with nanoclay in concentrations of 1%, 3%, and 5%, for 2, 24, and 48 h. Fourier transform infrared spectroscopic analysis revealed a shift in the typical peaks of cellulose, hemicellulose, and lignin depending on the impregnation time and nanoclay ratio. Thermogravimetric analysis and the limiting oxygen index indicated that the samples impregnated with clay solutions exhibited higher thermal stability than the unimpregnated wood. Impregnation with nanoclay caused a decrease water absorption and thickness swelling of the sample groups. The modulus of elasticity, modulus of rupture, and compression strength strength values of samples were improved *via* impregnation. In conclusion, the applied nanoclay are suitable substances to increase the durability and thermal stablity of brutian pine wood.

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*Contact information: Pamukkale University, Faculty of Architecture and Design, Department of Industrial Design, 20160, Denizli, Türkiye; *Corresponding author: abdullahberam@pau.edu.tr*

INTRODUCTION

Wood is a vital substance that is naturally cellular, composite, and of botanical origin with distinct chemical and structural properties; it is utilized all over the world (Shi *et al*. 2007; Qu *et al*. 2011). Because it is a unique renewable and ecologically beneficial natural resource, wood is one of the most extensively used materials in construction, furniture, tools, and building supplies (Yildiz *et al*. 2006; Rowell and Dietenberger 2012). In addition to its renewability, as a biological material, wood has the disadvantage of being degraded by biological agents such as fungi (Meyer *et al*. 2014; Marais *et al*. 2022). Moreover, one of the most severe problems with wood that results in significant loss of life and property is its flammability (Gaan and Sun 2007; Lu *et al*. 2021).

In recent years, many wood modification methods have been applied to minimize the negative properties of wood, to promote awareness of its potential applications, and to develop products. These modifications include acetylation (Rowell 2012; Bi *et al*. 2021), furfurylation (Lande *et al*. 2008; Gerardin 2016; Li *et al*. 2019), heat treatment (Hill 2006; Kubovsky *et al*. 2020; Cao *et al*. 2022), NaOH treatment (Meng *et al*. 2019; Beram and Yasar 2020; Kaya *et al*. 2021; Beram 2022), impregnation (Var *et al*. 2002; Aziz *et al*. 2020; Kaya 2023), and treatments with other chemicals (Yaşar *et al*. 2010).

One of the most widely used commercial treatments for wood products is impregnation. The technique, which involves injecting various chemicals into the wood's structure, has been shown to enhance the wood's hydrophobicity and tolerance to

biological organisms, fire, and weathering. The impregnation utilizes an array of salts, oils, chemical mixtures, natural solutions, and nanoclays (Megnis *et al*. 2002; Var *et al*. 2015; Kaya 2023).

Nanoclays have a wide range of industrial applications today and are economically beneficial. The mineral family to which clay belongs includes hydrous silicates. Wood-based composites and wood materials frequently contain nano-clays to enhance their fungal, chemical, physical, and mechanical properties (Fabiyi *et al*. 2011; Bari *et al*. 2015; Han *et al*. 2016; Karkoodi *et al*. 2020; Kaya 2022).

Turkey's most prevalent and widely spread coniferous species is the brutian pine (*Pinus brutia* Ten.), occupying over 5.8 million ha (GDF 2021). It is the leading species in Turkish wood production, with 5 million m^3 used for industrial wood and 11 million m^3 for fuelwood (Guler and Yasar 2020; Pekgozlu *et al.* 2020). This pine's wood is used to produce construction materials, packing cases, agricultural tools, various poles, and paper production. Additionally, it is a key source of raw material for producing particle board and fiberboard. The bark, stump, and roots of this species are also used for a variety of purposes (Tutus *et al.* 2012; As *et al.* 2001; Guler and Yasar 2020; Pekgozlu *et al.* 2020).

In this study, an impregnation process was applied on brutian pine (*Pinus brutia* Ten.) wood with nanoclay prepared in different concentrations (1%, 3%, and 5%) for different durations (2, 24, and 48 h). The chemical and thermal changes caused by the impregnation process in the samples were evaluated by comparison with the control samples. The effects of impregnation at different durations and concentrations on the physical and mechanical properties of the samples were investigated.

EXPERIMENTAL

Materials

The material of brutian pine used in the study was obtained from Denizli Candan Timber Company, Türkiye. The material was prepared in accordance with the measurements used in TS 2470 (1976) and TS 53 (1981) to determine the physical and mechanical properties. Samples were selected from the parts of the wood material, which has regular fibers without knots and cracks.

The nanoclay (kaolinite) was purchased from Sigma-Aldrich, US. Clay is a layered silicate material that is frequently encountered in nature. Kaolinite is a well-known example of a layered substance or clay (Deng *et al*. 2017). Pure kaolinite has the chemical composition aluminum silicate hydrate ($Al_2O_3.2SiO_2.2H_2O$). It was obtained from the kaolin fields located in the Düvertepe region of Sındırgı district of Balıkesir province (Türkiye). Product standards vary depending on the industrial properties of kaolinite. These changes occur in terms of mineralogical composition, chemical composition, grain size distribution and various rheological properties. Generally, the particle sizes of kaolinite are +10 µm 2-20(%), -2 µm 35-70(%) and -1 µm 0-2(%), and the surface area is 13-22 m^2/g (Oteyaka *et al.* 2009; MRE 2023).

Impregnation Method

Wood specimens were prepared with kaolin suspensions at 1%, 3%, and 5% (w/v) at room temperature. Samples were impregnated with these suspensions in medium-term (2 h) and long-term (24 and 48 h) dipping method according to ASTM D1413-99 (2005) standard principles. The experimental design of the samples is given in Table 1.

Table 1. Experimental Design

Sample Type	Concentration of Suspension (%)	Impregnation Time (h)
PB0	-	-
PB12	1	2
PB124	1	24
PB148	1	48
PB32	3	2
PB324	3	24
PB348	3	48
PB52	5	2
PB524	5	24
PB548	5	48

Preparation of Samples and Determination of Properties

The samples were prepared using sapwood lumber. Afterwards, the samples were conditioned in an air-conditioning room for 48 h at 20 ± 2 °C with relative humidity 65 ± 5% until air-dried to a moisture content of 12%. Density (*D*), thickness swelling (TS), and water absorption (WA) experiments were performed with samples of 20 x 20 x 30 mm^3 volume. The density of samples (TS 2472 1976), dimensional change (TS 4084 1983), and water uptake (TS EN 317 1999) were determined as per respective standards. For each treated wood sample the weight percent gain (WPG) was computed. All weight measurements were made when the objects were oven-dry prior to and following impregnation. The determination of mechanical properties followed pertinent criteria. The modulus of rupture (MOR) per TS 2474 (1976), modulus of elasticity (MOE) per TS 2478 (1976), and the compression strength parallel to gain per TS 2595 (1977) were also determined. Shimadzu AGS-X (10 kN) universal testing machine (Shimadzu Corp., Kyoto, Japan) was used for the mechanical tests.

Preparation of Chemical Test Samples and Application of Experiments

Samples were ground in the 40- to 100-mesh range for use in Fourier transform infrared (FTIR) spectroscopy and thermogravimetric analyses (TGA). Afterwards, pellets obtained by pressing at 602 N/mm^2 10 mg of wood flour with KBr at a ratio of 1:100 (w/w) for each sample group were used for FTIR. The FTIR spectra were analyzed in the Perkin Elmer BX FTIR spectrometer instrument (PerkinElmer U.S. LLC, Shelton, CT, USA) in the wavenumber range of 4000 to 400 cm^{-1} with a resolution of 4 cm^{-1} at ambient temperature. Thermogravimetric analysis (TGA) was conducted with an Exstar SII TG DTA 7200 (Exstar, SII NanoTechnology Inc., Tokyo, Japan) to examine the thermal stability of samples under nitrogen gas at a heating rate of 10 °C/min from 25 to 600 °C with an approximately 5 mg sample. The limiting oxygen index (LOI) test of wood samples was performed on a flammability tester (S.C. Dey Co., Kolkata, India) according to the ASTM D-2863 (2006) method. The LOI is the minimum amount of oxygen required to cause ignition. The sample placed vertically in the LOI apparatus was exposed to combustion for at least 30 s. The ratio of nitrogen and oxygen was recorded during the combustion process.

Statistical Analysis

Statistical analysis was performed on the study's findings using Minitab 16 software. The data were subjected to an analysis of variance (ANOVA). A Duncan test was used to identify the various groups in cases where the ANOVA test revealed statistical differences *via* SPSS® 20.0 for Windows® (IBM Corp., Armonk, NY, USA).

RESULTS AND DISCUSSION

The physical properties of the impregnated and unimpregnated brutian pine samples are shown in Table 2. A statistical difference was found between the physical properties of the test samples from the control and impregnated samples according to the ANOVA test. According to the Duncan test, 4 homogenous groups were formed in each of D_0, WA -2, WA -24, TS-2, and TS-24-h data and 5 homogenous groups were formed in each of WPG data.

Table 2. D_0, WA, TS, and WPG Properties of Impregnated and Unimpregnated Wood

Sample Type	D_0 (g/cm^3)	WA-2 h	WA-24 h	TS-2 h	TS-24 h	WPG (%)
PB0	0.49 (0.09)[1] a[2]	32.67 (3.04) a	58.42 (5.34) a	13.51 (1.05) a	19.77 (2.41) a	-
PB12	0.55 (0.11) b	26.11 (2.32) b	49.82 (6.27) b	11.85 (1.28) b	17.31 (2.01) b	0.46 (0.08) a
PB124	0.58 (0.13) b	24.97 (3.13) b	46.77 (5.31) b	10.66 (0.88) b	16.55 (1.82) b	0.54 (0.12) b
PB148	0.59 (0.14) bc	23.03(1.80) b	43.18 (3.68) b	10.03 (0.97) bc	15.81 (1.44) b	0.67 (0.15) b
PB32	0.57 (0.08) b	21.52 (2.45) c	37.93 (2.15) c	10.23 (1.83) b	15.93 (1.63) bc	1.58 (0.28) c
PB324	0.60 (0.12) c	20.17(3.68) c	35.74 (3.75) c	9.38 (1.41) c	15.12 (1.38) b	2.09 (0.32) d
PB348	0.62 (0.13) c	19.21 (1.45) c	32.85 (3.19) c	8.71 (0.69) c	14.29 (2.02) c	2.21 (0.44) d
PB52	0.59 (0.12) bc	17.68(1.64) d	32.26 (2.81) cd	8.87 (0.92) cd	14.05 (1.60) c	2.13 (0.53) d
PB524	0.64 (0.10) d	16.33 (2.08) d	30.05 (3.66) d	8.12 (0.77) cd	13.37 (1.12) d	3.06 (0.62) e
PB548	0.67 (0.15) d	15.17(1.53) d	28.62 (3.68) d	7.43 (1.05) d	12.26 (0.99) d	3.57 (0.79) e

[1]: Standard deviation, [2]: Groups defined by different letters in each column according to the Duncan test (for D_0, WA (2 and 24 h), TS (2 and 24 h), and WPG $p < 0.01$); N=40

In the applied impregnation method, it was observed that the initial density (D_0) and WPG increased as the nanoclay ratio increased. When the 1%, 3%, and 5% nanoclay was added, respectively, the D_0 increased between 12.2% and 36.7%, the WPG increased between 0.46% and 3.57%. Impregnating with nanoclay progressively decreased water absorption and thickness swelling. WA-2, WA-24, TS-2, and TS-24 values decreased as the nanoclay ratio and residence time increased. When the 1%, 3%, and 5% nanoclay was added, respectively, the WA-2 decreased between 20% and 63.5%, WA-24 values between 14.7% and 51%, TS-2 values between 12.2% and 45%, and TS-24 values between 12.4%

and 37.9%. These obtained data suggest along with the previous studies that the nanoclay material made the wood material more stable and its physical properties improved (Hetzer and De Kee 2008; Alamri and Low 2013; Kaya 2022).

The mechanical properties of the impregnated and unimpregnated brutian pine samples are shown in Table 3. A statistical difference was found between the mechanical properties of the test samples from the control and impregnated samples according to the ANOVA test. According to the Duncan test, 4 homogenous groups were formed in each of MOR, MOE, and CS data.

The addition of nanoclay increased the modulus of rupture (MOR), modulus of elasticity (MOE), and compression strength (CS) of impregnated woods. When the 1%, 3%, and 5% nanoclay was added, respectively, the MOR increased between 4.6% and 30.2%, MOE values between 2.3% and 17.7%, CS values between 5.5% and 33.1% and they reached maximum at 48 h clay impregnation time. The changes in the chemical composition of the impregnated brutian pine woods increased the MOE, MOR, and CS values of the samples as the concentration of the nanoclay impregnate and impregnating time increased (Islam *et al*. 2012; Ghofrani *et al*. 2015; Holy *et al*. 2022). The increased interactions between clay and the other constituents in the composites were supported by the FTIR analysis of composites containing nanoclay (Hristov and Vasileva 2004; Hetler and De Kee 2008; Mandal *et al*. 2020).

The LOI values of the sample groups are summarized in Table 3. The values were between 24.8% and 42.2%. It has been determined that the highest fireproof sample group with a LOI of 42.2% is obtained with PB548, which increases fire resistance 70% compared to PB0. In wood impregnated with nanoclay, the LOI value increased as the nanoclay ratio increased. The flame slowing effect seems to originate from the ability of the clay to contribute to char formation. This resultant coating of coal created an insulating layer that slowed down the flow of gases that fed the flame and resisted heat transfer (Powell and Beall 2006; Mandal *et al*. 2020; Kaya 2022).

Table 3. MOR, MOE, CS, and LOI Properties of Impregnated and Non-impregnated Wood

Sample Type	MOR (N/mm^2)	MOE (N/mm^2)	CS (N/mm^2)	LOI (%)
PB0	86.23 (4.55)[1] a[2]	8786 (267) a	56.62 (3.59) a	24.75
PB12	90.27 (6.28) b	8996 (338) b	59.77 (4.31) b	26.30
PB124	91.84 (3.62) b	9053 (311) b	61.62 (2.23) b	28.65
PB148	94.65 (2.78) c	9132 (326) b	63.44 (3.66) b	29.20
PB32	91.88 (4.92) b	9277 (388) bc	66.23 (3.45) c	31.10
PB324	95.41 (5.68) c	9318 (224) c	67.96 (2.78) c	33.88
PB348	97.12 (2.36) cd	9449 (157) c	69.55 (3.11) c	34.55
PB52	105.59 (7.22) d	9867 (208) d	72.83 (2.08) d	38.78
PB524	106.94 (8.81) d	9938 (166) d	73.76 (2.35) d	40.45
PB548	112.33 (10.24) d	10348 (196) d	75.29 (1.98) d	42.15
1: Standard deviation, 2: Groups defined by different letters in each column according to the Duncan test (for MOR, MOE, CS, and LOI $p < 0.05$); N=30				

In the impregnated sample groups, FTIR was utilized to determine the functional groups and chemical interactions of materials on each other. The FTIR spectrum revealed a shift in the typical peaks of cellulose, hemicellulose, and lignin depending on the impregnation time and nanoclay additive ratio. The FTIR spectra of the nanoclay

impregnation and control brutian pine and poplar samples were recorded in the range of 4000 to 400 cm^{-1} waves (Fig. 1).

The band at 3450 cm^{-1} reduced the number of OH groups, and the nano-treatment reduced the number of OH groups in comparison to the control group. As a result, there was a decrease in the peak. The findings demonstrated a substantial connection between the nanoclay and the wood's chemical components. Looking at the peaks of FTIR spectroscopy, cellulose, hemicellulose, and lignin were changed by the processing (Wada 1967; Poletto *et al*. 2013). When compared to the control group, the peak created in the 2910 cm^{-1} band was lower. The asymmetric stretching of the C-H methyl and methylene groups is responsible for this peak (Bellamy 1966; Pandey and Pitman 2003; Tatzber *et al*. 2007; Papadopoulos and Mantanis 2011; Beram and Yasar 2018). When compared to the control group, there was an increase in the peak in the 1450 cm^{-1} band. A typical peak for lignin components is symmetrical stretching vibrations of C=O and -COO in aromatic rings. Panday (2005). It has been proposed that the peak of the peaks in the 1060 cm^{-1} range was reduced when compared to the control group, and that the CH breakdown in polysaccharides and the change in this peak were indicators of the change in the hydrophilic characteristic of the cellulosic material (Can and Sivrikaya 2017). Meanwhile, the change indicates the actions of the functional groups in the nanomaterial minerals added to the wood (Chang and Chang 2001; Pandey and Vuorinen 2008; Pandey *et al*. 2010; Dubey *et al*. 2012). For montmorillonite nanoclay, the characteristic peak at 1060 cm^{-1} is caused by Si-O-Si stretching and the out-of-plane Si-O-Si strain mode (Elsacker *et al*. 2022). There is an increase in the 873 cm^{-1} band when compared to the control group, and this band is attributable to the Si-O-Al stretching mode for montmorillonite nanoclay (Moser *et al*. 2017; Jones *et al*. 2018). The peak bands can be seen at 430 cm^{-1} (Abidin *et al*. 2007). As a result, FTIR spectroscopy reveals its presence in clay and nanoclay (Wada *et al*. 1988). The FTIR results were consistent with the results of the analysis of TG/DTG and compressive strength results.

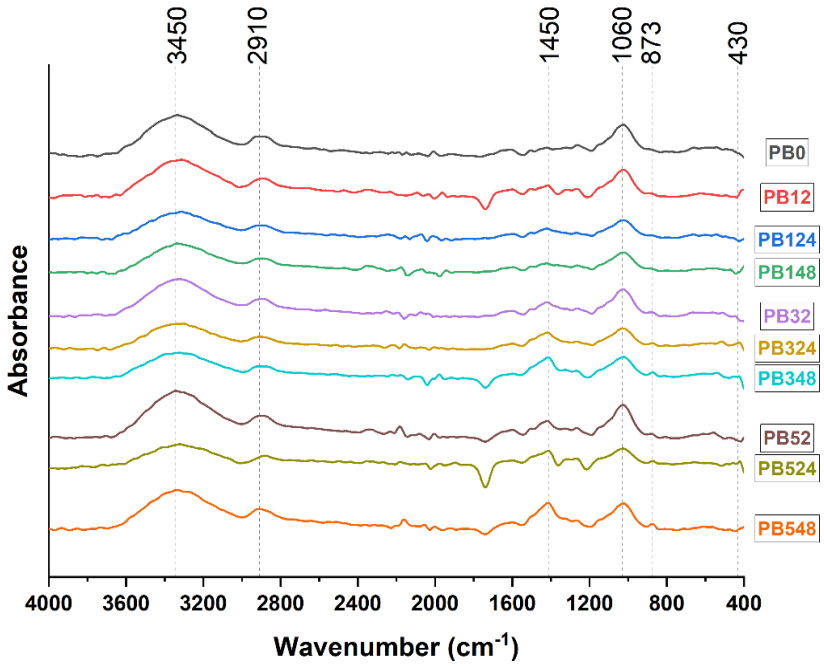

Fig. 1. FTIR spectra of the impregnated and unimpregnated brutian pine samples

Both TGA and DTG (differential thermogravimetric analysis) thermograms of the impregnated and non-impregnated brutian pine samples are plotted in Figs. 2 and 3.

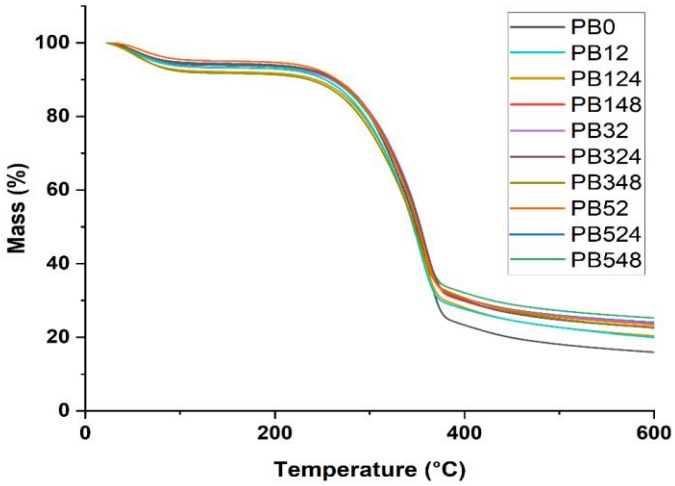

Fig. 2. TGA thermograms of the impregnated and non-impregnated brutian pine

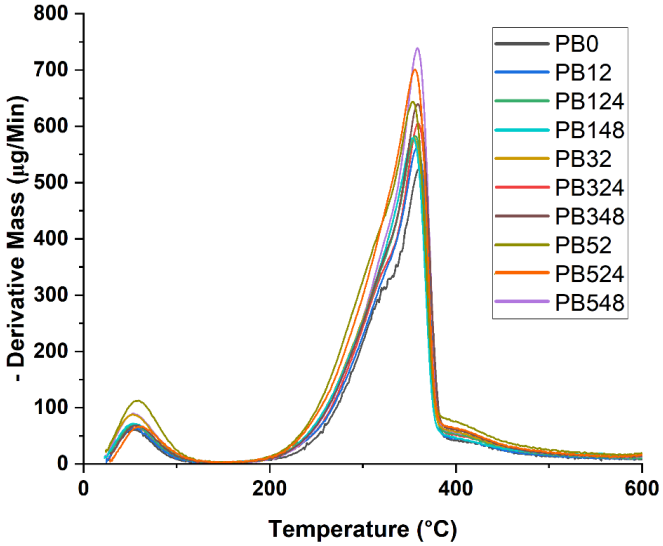

Fig. 3. DTG thermograms of the impregnated and non-impregnated brutian pine samples

The initial decomposition temperature (T_0), maximum degradation temperature (T_{max}), final temperature (T_f), and residual weight (RW, %) for woods with and without nanoclay are summarized in Table 4. The initial degradation temperature of the samples was 150 °C in the impregnate and non-impregnated brutian pine samples. Water and some extractives in the samples were removed until that temperature (Thurner and Mann 1981). After 150 °C, the decomposition process continued until 552 °C in the control sample, between 554 to 562 °C in the samples impregnated with 1% nanoclay, between 560 and 565 °C in the samples impregnated with 3% nanoclay, and between 566 and 574 °C in the samples impregnated with 5% nanoclay. The highest final temperature was determined in the PB548 sample with 574 °C. From 150 to 552 to 574 °C, hemicellulose, the remaining extractives, lignin, and cellulose were decomposed (Thunder and Mann 1981; Meszaros *et*

al. 2007). As the nanoclay ratio and impregnation application duration increased, the residue weight increased. The residual amount of the RW at 600 °C in the samples was 16.0% in control sample (PB0), between 19.2 and 23.2% in the samples impregnated with 1% nanoclay, between 23.9% and 24.7% in the samples impregnated with 3% nanoclay, and between 24.9 and 26.6% in the samples impregnated with 5% nanoclay. The TGA study results showed that as the concentration of nanoclay increased, the heat resistance of the fibers gradually increased.

Table 4. Thermal Degradation Temperatures and Residue Weight of Brutian Pine

Sample Type	T_0 (°C)	T_{max} (°C)	T_f (°C)	RW at 600 °C (%)
PB0	150	358	552	15.96
PB12	150	352	554	19.21
PB124	150	354	557	19.86
PB148	150	356	562	23.22
PB32	150	357	560	23.94
PB324	150	358	563	24.55
PB348	150	359	565	24.73
PB52	150	353	566	24.91
PB524	150	357	571	25.25
PB548	150	359	574	26.60

T_0: Initial decomposition temperature, T_{max}: maximum degradation temperature, T_f: Final temperature

CONCLUSIONS

In this study, brutian pine woods were impregnated with 1%, 3%, and 5% nanoclay solutions for 2, 24, and 48 h. At the conclusion of this procedure, the chemical and thermal changes in the wood material were identified, and their influence on the physical and mechanical properties were assessed.

1. Increasing the nano clay content increased the WPG ratio and thus reduced the values of water absorption (WA) and thickness swelling (TS). The findings indicated that increasing nanoclay rates enhanced performance indicators. Wood pores were filled with these nanoclay particles. Thus, water was prevented from penetrating deeper sections of the wood through capillary action, which resulted in an increase in hydrophobic characteristics of the wood.
2. Moreover, mechanical properties, such as modulus of rupture (MOR), modulus of elasticity (MOE), and compression strength (CS), were also observed to increase in the impregnated sample groups as the nanoclay ratio increased compared to the control groups. Thus, it has been revealed that nanoclay positively affects the mechanical strength properties of brutian pine wood.
3. The results of the Fourier transform infrared (FTIR) analysis showed that the nanoclay penetrated the wood material by impregnation. Compared to the control group samples, it was observed that the thermal endurance of the impregnated samples increased as the nano clay incorporation rate increased.

4. Nanoclay impregnation increased the thermal stability qualities of wood samples, according to thermogravimetric analysis (TGA) and limiting oxygen index (LOI) studies. During the LOI test, nanoclay compounds demonstrated char formation. Thermal stability performance of samples increases with the high ration of nanoclay.
5. According to the findings, nanoclay has a great deal of potential for usage as a pine wood preservative. The experimental results indicated that properties of wood materials could be significantly improved with ideal application methods and an appropriate combination of the nanoclay type.

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