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Effect of thermal modification on mechanical, physical and biological properties of *Picea abies* (L.) H.Karst.

Supram Mitra, Sauradipta Ganguly* and Ajmal Samani

Wood Preservation Discipline, Forest Product Division, Forest Research Institute, Dehradun 248006, India

*Corresponding author: sauradipta.ganguly@fridu.edu.in

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Abstract

Background: Wood modification by heat treatment has become more commercially important over the years. Thermal modification is predominant among all modifications in the market. Heat treatment alters structural compositions of the chief polymeric constituents of wood (cellulose, hemicellulose and lignin) and significantly improves its performance in service.

Methods: In the present study, thermal modification of *Picea abies* was attempted at temperatures of 180°C, 210°C and 240°C to examine changes in mechanical strength, dimensional stability, aesthetic appearance of wood and resistance to contamination by staining fungi.

Results: Samples treated at 180°C showed a 0.62% reduction in MOE in comparison to untreated wood sample controls. 2.85% and 9.88% reductions were observed in samples treated at 210°C and 240°C respectively. Similar trends were observed for MOR values with samples treated at 180°C exhibiting a reduction of 4.6% in MOR values, and 22.99% and 39.09% reductions were observed in samples treated at 210°C and 240°C compared to the controls. Heat treatment also imparted dimensional stability to the wood species chosen for the study which is evident from ASE (Anti swelling efficiency) values of 34.52 %, 45.44% and 63.26% treated at 180°C, 210°C and 240°C respectively. The overall colour change (ΔE*) and bio resistance to staining fungi increased gradually with increasing treatment temperature. Samples treated at 240°C showed the highest resistance whereas the lowest resistance was exhibited by controls.

Conclusions: The study established the efficacy of thermal modification as an alternative to conventional treatment methods for improving properties of Norway spruce wood for specific end uses.

Keywords: ASE; colour change; MOE; MOR; thermal modification

Introduction

Wood is a renewable and economical asset and has been utilized in both indoor and outdoor applications for thousands of years. As a biological material, dimensional instability under changing moisture conditions and biodegradability are the primary hindrances to use of wood. Wood has been a favored material for development because of its availability, ease of working and other properties like high specific strength, insulation, and processing, however, its degradation due to fire, biological organisms, water and light is the main concern in its extensive utilization.

Preservative treatment with chemicals is a viable solution and can improve the service life of wood, but the chemicals used for preservation are often highly toxic and this has led to preservatives being banned in several parts of the world. Therefore, wood manufacturers are looking for alternative ways to increase the service life of the wood. Thermal modification (TM) results in more acceptance of wood as a construction material by altering its properties and reducing the shortcomings associated with it in use. TM improves wood qualities and renders it fit for several applications by increasing its biological resistance, enhancing dimensional stability and decreasing its susceptibility to moisture in the air (Hygroscopicity) (Alen et al. 2002; Hom et al. 2020). Heat applied to wood reduces the amorphous cellulose and increases the crystallinity in cellulose

which in return reduces the hygroscopicity of modified wood (Sahin 2008). In addition, TM of softwoods at temperatures below 200°C minimizes the production of wood dust during manufacturing processes (Ockajova et al. 2020). The degree to which wood properties alter depends on tree species and the process of modification. In general, applied temperature, duration of treatment, wood moisture content and oxygen level are the most critical factors (Esteves & Pereira 2009). Typically, thermal modification makes only a small change in modulus of elasticity (MOE), but greatly decreases modulus of rupture (MOR). Several studies have shown that the MOR and the MOE of wood change when it is thermally modified, depending on the temperature and the duration of thermal treatment i.e., the higher the temperature during the treatment, the higher the loss in strength (Militz 2002).

India's rapidly growing furniture manufacturing industries compete with those in China and Vietnam and are expanding their networks into lucrative markets in Europe and North America. Large companies as well as many small-scale enterprises export timber products. They also fulfil India's timber demands of growing urban populations. Despite enormous and varied forest resources, India is a timber-deficient country. Over-exploitation, illegal felling and lack of proper forest management have led to a partial ban on logging in natural forests and wood-based industries are now forced to import significant quantities of both hardwood and softwood to meet market demands (Ganguly et al. 2018).

The consumption of industrial wood greatly increased during the last decade. India over has become a major importer of raw timber, pulpwood and allied products which amounted to an estimated $2 billion in the past decade (Sood 2019) and consequently, the import policy of wood and wood products in the country has been liberalized to support local needs and facilitate conservation of natural forests. The durability of several wood species of non-indigenous origin became questionable due to changes in silvicultural and pre-harvesting operations worldwide. Another reason for poor durability could be the climatic conditions in service (Tripathi et al. 2022). Preliminary trials (Tripathi 2012) have established that timbers from Malaysia categorized as nondurable based on graveyard tests which indicated that 80% of the wood was categorized as moderately durable and nondurable, but durable when they are determined by EN 350-1 (1994). One of the most limiting factors for commercial utilization of such wood is its low resistance to termites and fungi which shorten the service life of structures or even leads to structural failure. It is therefore essential to know the durability and specifications of each imported and indigenous species to optimize costs involved in construction and processing.

Norway spruce (*Picea abies* (L.) H. Karst.) is one of the most imported coniferous species to India and is suitable for a variety of end uses. However, not much data is available on the species' performance in tropical climatic conditions of India. Wood in service is often exposed to harsh climates and thus, despite being a suitable building material, it has certain constraints that restrict its extensive use in outdoor conditions (Ganguly et al. 2021; Tripathi et al. 2022). Hence research on imported species is needed before it can be recommended to users. The research described here was designed to contribute knowledge about Norway spruce wood to be used in the commercial sector, to bridge the gap between supply and demand of woody biomass in India.

Methods

Sample preparation

Seasoned planks of *Picea abies* (L.) H.Karst. were procured through local vendors. The study was executed at the Wood Preservation Discipline of Forest Research Institute, Dehradun, India. The planks were converted into desired dimensions for performing physical, biological and mechanical tests. Relatively straight grained samples free from any visual defects were selected from the same part of each board for a variety of tests. Samples contained mixed portions of sap and heartwood. The dimensions of the samples prepared for conducting various tests were as follows:

(i) Static bending test: 30 cm x 2 cm x 2 cm
(ii) Dimensional stability test: 2 cm x 2 cm x 2 cm
(iii) Study of colour parameters: 15 cm x 10 cm x 1.5 cm
(iv) Bio resistance test: 10 cm x 2.5 cm x 0.6 cm

Treatment details

Treatment details are provided in Table 1.

TABLE 1: Number of samples and replications used in different experiments.

Sl. no	Name of test	Number of treatments	Duration of treatment (h)	Number of replicates used per treatment
1	Static bending test	4 (Control, T1=180°C, T2=210°C and T3=240°C)	3	6
2	Dimensional stability test	4 (Control, T1=180°C, T2=210°C and T3=240°C)	3	8
3	Colour of thermally modified wood	4 (Control, T1=180°C, T2=210°C and T3=240°C)	3	120
4	Bio resistance test on thermally modified wood	4 (Control, T1=180°C, T2=210°C and T3=240°C)	3	6

Thermal modification
Thermal modification of wood samples was done in a hot-air oven (Thermotech, PID-91S) at varying temperatures. The temperatures used in these experiments were 180° C, 210° C and 240° C. All the samples of each treatment were heated for three hours except for the untreated controls. The samples were placed inside the oven, and then the desired temperature was set on the oven controls. When the temperature reached the desired level, three hours of time was counted. Tests were performed after thermal modification to determine changes in properties.

Static bending test
Static bending tests were conducted on the Universal testing machine in the laboratory of Timber Mechanics discipline of FRI, Dehradun according to IS: 1708 (Part 5) - 1986. The test specimens were placed on a rig so that the load was applied through a loading block to the tangential surface. Specimens were supported on the rig in such a way that they could bend. Load was applied continuously on the samples and deflections of the neutral plane at the center of each length were measured at 10 kg intervals of load. Load-deflection curves were drawn. Formulae used were:

(i) Modulus of elasticity (MOE) = $PL^3 / 4\Delta bh^3$ (kg/cm^2)

(ii) Equivalent fibre stress at maximum load (modulus of rupture or MOR) = $3P'L/2\ bh^2$ (kg/cm^2)

Where P = load in kg at the limit of proportionality which shall be taken as the point in load-deflection curve above which the graph deviates from the straight line; L = span of the test specimen in cm; b = breadth of the test specimen in cm; h = depth of the test specimen in cm; P' = maximum load in kg; Δ = deflection in cm at the limit of proportionality.

Dimensional stability
Dimensional stability was determined by comparing the total volumetric swelling (VS) of thermally modified samples with control samples. Initial dimensions and weight were recorded for both treated and control samples. Swelling was determined by a water immersion test. All the samples were submerged in distilled water and were kept under vacuum for 30 minutes and allowed to soak for 24 h (Samani et al. 2021). Weights and dimensions of saturated samples were recorded.

(i) The swelling coefficient was calculated as:

$$S\ (\%) = ((V_s - V_i)/V_i) \times 100$$

Where V_s =Volume of soaked sample and V_i = Initial volume (before water soaking)

(ii) Anti-swelling efficiency (ASE) was calculated as:

$$ASE\ (\%) = ((S_u - S_m)/S_u) \times 100$$

Where S_u and S_m are swelling coefficient of unmodified and modified samples respectively.

(iii) Water exclusion efficiency (WEE) % was calculated as:

$$WEE\ (\%) = ((W_c - W_t)/W_c) \times 100$$

Where W_c = Mass of water absorbed by control samples (g) and W_t = Mass of water absorbed by treated samples (g).

Colour variables of thermally modified wood samples
Colour was measured using the CIE lab system (spectral photometer colour reader CR-10). Wood samples heat-treated at different temperatures were used to estimate changes in color. Color profiling was done on the same samples at the same spot before and after heat treatment and data were compared.

The colour coordinates were as follows: lightness L* (varying from 0 for black to 100 for white), redness a* (varying from negative values for green to positive values for red on the green-red axis), and yellowness b* (varying from negative values for blue to positive values for yellow on the blue-yellow axis). For all wood samples, these values were measured at different places. For each test sample, colour measurements were performed at 20 points (Rectangular areas of about 3-inch x 1.2 inch), and an average value was calculated.

Colour differences between treated and control samples were calculated as:

$$E = [\Delta L^*2 + \Delta a^*2 + \Delta b^*2]1/2$$

Where ΔL*, Δa*, and Δb* are differences of the initial and final values (before and after heat treatment at different temperatures) of the L*, a*, and b* parameters, respectively. A low E value corresponds to a low colour difference.

Bio resistance of thermally modified wood
Selection of test fungi:
Aspergillus niger stain fungus was chosen due to its rapid growth potential.

Preparation of test culture and introduction in the test samples
Aspergillus niger was sub-cultured in sterilized petri-plates using nutrient agar media and incubated with biochemical oxygen demand (BOD) for two weeks. The fungus was inoculated in test samples by dipping. The fungal spore was collected from the petri plates using distilled water within a laminar air flow to prevent contamination. Distilled water containing spores was collected in a 200 mL beaker. Spores were mixed in distilled water using a magnetic stirrer for 15 minutes. A filter paper was laid on the surface of sterilized boxes to hold moisture within them. Wooden samples were dipped inside the beaker containing spore solution and kept inside the boxes. Boxes containing test blocks were incubated for four weeks. During the incubation period the samples were sprayed with spore suspensions at intervals of 8 days.

Assessment of damage
The samples were assessed visually after air drying at the end of exposure period of four weeks. The area covered by fungus was expressed as a percentage of the total surface area. The samples were rated for damage based on the following numerical scale (Jain et al. 1991).

Condition of sample rating scale

0 = No stain; samples free from stain

0.5 = Trace stain; surface area covered less than 5% of total surface.

1.0 = Light stain; 5 to 15% of the total surface area covered by stain.

3.0 = Moderate stain; 15 to 45% of the total surface area covered by stain.

5.0 = Heavy stain; more than 45% of the total surface area covered by stain.

Statistical analysis
IBM® SPSS® Version 28 software was used for statistical analysis. For analyzing data, means of different treatments were computed. One-way ANOVA was applied to treatment means. Tukey's post hoc analysis at α=0.05 was used to determine whether treatment means are significantly different.

Results and Discussion

Static bending test

Modulus of elasticity and modulus of rupture
The MOE and MOR test results are presented in Table 2. Temperature affected MOR significantly (P<0.05) but not MOE, which corroborates findings of several previous researchers (Korkut et al. 2008). MOE decreased with increasing treatment temperature. Although the rate of decrease was negligible for the first two treatments (T1 and T2). Small fluctuations in MOE in heat treated spruce were reported by previous researchers (Kim et al. 1998).

According to (Boonstra et al. 2007), heat treating Norway spruce resulted in a 17% increase in MOE. However, in the present study no statistically significant changes in MOE were observed. It is also important to mention that in some other studies (Inoue et al. 1993); (Kocaefe et al. 2008) thermal modification resulted in significant reductions in MOEs of many species. Degradation in hemicellulose of wood cell wall disturbs the load sharing capacity of the lignin-hemicellulose matrix (Hillis 1984), results in the increase of crystalline cellulose may contribute to changes in MOE observed in other studies.

MOR reduced with increasing temperature. Samples treated at 180°C showed a 4.6% reduction in MOR compared to control samples, while 22.99% and 39.09% reductions were observed in samples heat treated at 210°C and 240°C respectively. The loss in static bending properties can be attributed to the degraded cell wall components of wood. Results are in line with other studies (Boonstra et al. 2007; Silva et al. 2015) and (Percin et al. 2016) where reduction of strength with increasing treatment temperature was reported due to degradation of hemicelluloses. Decreases in MOR values can be correlated with the rate of thermal degradation and losses of substance after heat treatments at increasing temperature (Korkut et al. 2008). The decrease in strength can primarily be attributed to degradation of wood polymers under the application of heat (Kotilainen 2000); (Wikberg & Maunu 2004) with hemicellulose being the most affected component.

Dimensional stability test

Amount of water absorbed by modified and unmodified samples (g)
Water absorption values of thermally modified *Picea abies* are presented in Figure 1 and Table 3. Treatment temperatures significantly decreased water absorption (P<0.05). Percentage reductions of water absorbed for different treatments compared to control samples were 17.05%, 24.51% and 37.33% at 180°C, 210°C and 240°C respectively.

(i) Swelling coefficient and ASE (%)
Treatment temperature significantly affected swelling coefficient (Table 4) and ASE values (P<0.05). Swelling coefficient was highest for untreated samples and decreased with modification temperature. Samples treated at 240°C had the lowest value, which is desirable for dimensional stability. Percentage reduction of swelling coefficient values for different treatments compared to control samples were 34.51%, 45.43% and 63.28% and the ASE values were 34.52 %, 45.44% and 63.26% at 180°C, 210°C and 240°C respectively, Figure 2. These

TABLE 2: MOE and MOR of thermally modified *Picea abies*

Treatment	MOE (N/mm²)	MOR (N/mm²)
Control	15060.48 (±1558.44)[a]	74.68 (±3.18)[a]
T1 (180 °C)	14966.93 (±742.65)[a]	71.24 (±2.79)[a]
T2 (210°C)	14631.19 (±1123.89)[a]	57.51 (±3.62)[b]
T3 (240°C)	13573.07 (±1081.05)[a]	45.49 (±2.79)[b]

Note: The body of the table shows the average values ± 95% confidence interval of 6 replicates per treatment. Equal letters in columns indicate no significant difference between treatments, and different letters indicates significant difference between treatments according to Tukey's post hoc analysis (**α** = 0.05).

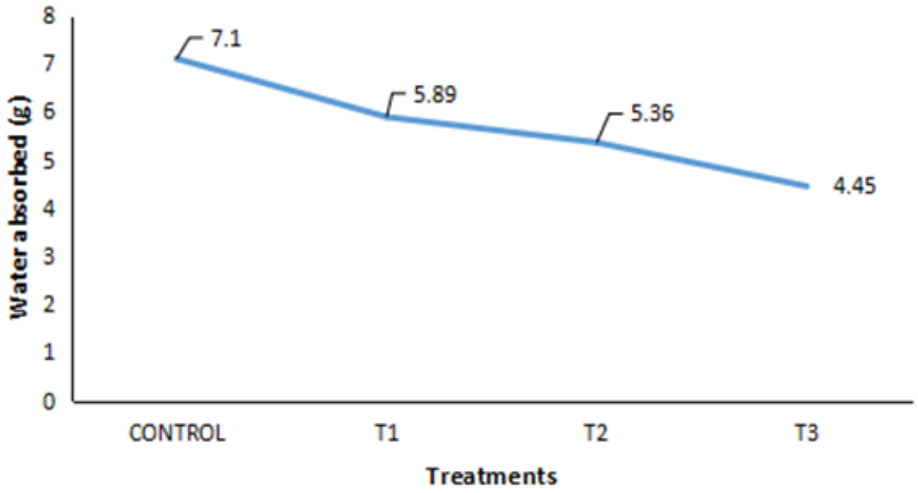


FIGURE 1: Amount of water absorbed by wood samples of different treatments.

TABLE 3: Amount of water absorbed by modified and unmodified samples.

Treatment	Amount of water absorbed (g)
Control	7.10 (±0.33) [a]
T1 (180°C)	5.89 (±0.20) [b]
T2 (210° C)	5.36 (±0.22) [b]
T3 (240°C)	4.45 (±0.10) [c]

Note: The body of the table shows the average values ± 95% confidence interval of 6 replicates per treatment. Equal letters in columns indicate no significant difference between treatments, and different letters indicates significant difference between treatments according to Tukey's post hoc analysis (**α** = 0.05).

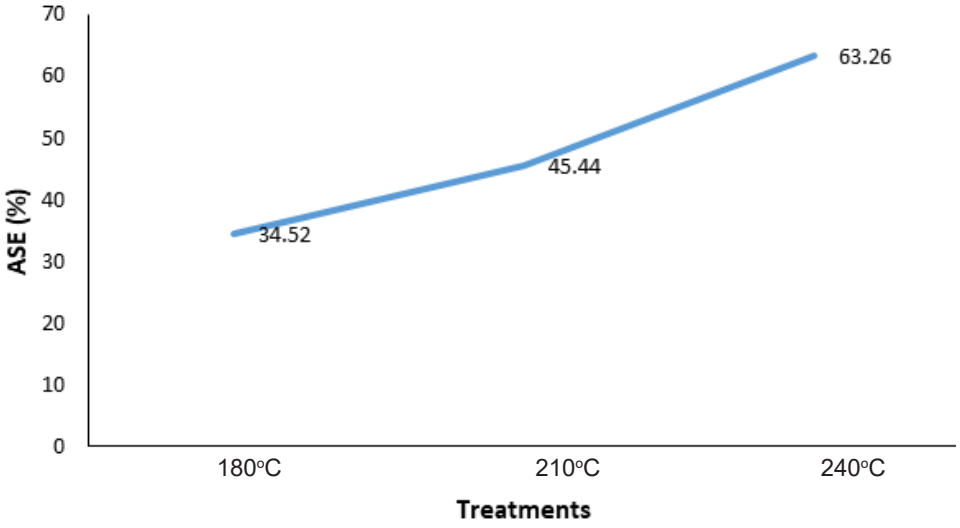
TABLE 4: Swelling coefficient and ASE of modified wood samples

Treatment	Swelling coefficient (%)	Anti-swelling efficiency (ASE) (%)
Control	18.49 (± 0.42) [a]	-
T1 (180 °C)	12.11 (±0.34) [b]	34.52 (±1.75)[a]
T2 (210°C)	10.09 (±0.20) [c]	45.44 (±0.81)[b]
T3 (240°C)	6.79 (±0.36) [d]	63.26 (±2.47)[c]

Note: The body of the table shows the average values ± 95% confidence interval of 6 replicates per treatment. Equal letters in columns indicate no significant difference between treatments, and different letters indicates significant difference between treatments according to Tukey's post hoc analysis (**α** = 0.05).

results were like ASE values obtained for other thermally modified softwoods (Esteves et al. 2009); (Romagnoli et al. 2015). ASE improvements were due to changes in the chemical structure of wood according to the treatment temperature used which made them less hygroscopic in nature (Dirol & Guyonnet 1993) reducing the amount of hydroxyl groups caused mainly by the autocatalytic reactions of the constituents of the cell wall of wood.

FIGURE 2: Behaviour of ASE % in *Picea abies* wood with increase in modification temperature.



(ii) Water exclusion efficiency (%)

Water exclusion efficiency (WEE) values obtained indicate that treatment temperature significantly affected dimensional stability of the wood (Table 5; Figure 3). The water exclusion efficiency (%) values were 17.09%, 24.63% and 37.73% at 180°C, 210°C and 240°C respectively. Increase in the amount of crystalline cellulose may improve dimensional stability as hydroxyl groups are not easily accessible to the water molecules (Kocaefe et. al. 2008).

Rowell and Banks (1985) highlighted that hemicelluloses, non-crystalline cellulose, surfaces of crystalline cellulose and to a small degree lignin are responsible for hygroscopicity of wood. Of these, the hemicellulose is the most hygroscopic and least thermally stable. So, it can be fairly concluded that improved dimensional stability and water repellency in heat treated wood can primarily be attributed to decomposition of hemicelluloses at higher temperatures (Seborg et al. 1953; Hillis 1984; Evans 2003).

Colour of thermally modified wood samples

The colour values are reported in Table 6. Change in Lightness (ΔL^*) was calculated using initial and final L^* values for each sample. At higher temperatures, ΔL^* values increased, showing statistically significant differences ($p \leq 0.05$) between treatments. These results are comparable to those obtained for other thermally modified pine woods (Barcík et al. 2015).

The overall colour change (ΔE^*) increased gradually and samples became darker with treatment temperature, Figure 4. The resulting colour difference was statistically significant ($P<0.05$). On the one hand, the reported colour changes are probably related to the intensity of the treatment, and on the other hand, they might be related to the formation of chromogenic products due to the thermal degradation of some cell wall components, which are mainly connected to the lignin present in the wood (González-Peña et al. 2009). Colour is an important property for consumers, as the decorative look of wood is a determining factor in the selection of the wood or wood products (Esteves & Pereira 2009).

TABLE 5: Water exclusion efficiency (%) of thermally modified *Picea abies.*

Treatment	Water excluding efficiency (%)
T1 (180 °C)	17.09 (±1.15)[a]
T2 (210°C)	24.63 (±1.98)[b]
T3 (240°C)	37.31 (±1.82)[c]

Note: The body of the table shows the average values ± 95% confidence interval of 6 replicates per treatment. Equal letters in columns indicate no significant difference between treatments, and different letters indicates significant difference between treatments according to Tukey's post hoc analysis (**α** = 0.05).

TABLE 6: ΔL and ΔE of thermally modified *Picea abies*

Treatments	ΔL	ΔE
T1 (180°C)	1.27 (±0.49) [a]	2.84 (±0.54) [a]
T2 (210°C)	13.80 (±0.29) [b]	15.52 (±0.34) [b]
T3 (240°C)	35.84 (±0.56) [c]	36.74 (±0.5) [c]

Note: The body of the table shows the average values ± 95% confidence interval of 6 replicates per treatment. Equal letters in columns indicate no significant difference between treatments, and different letters indicates significant difference between treatments according to Tukey's post hoc analysis (**α** = 0.05).

FIGURE 3: Increasing trend of water excluding efficiency with increase in treatment temperature.

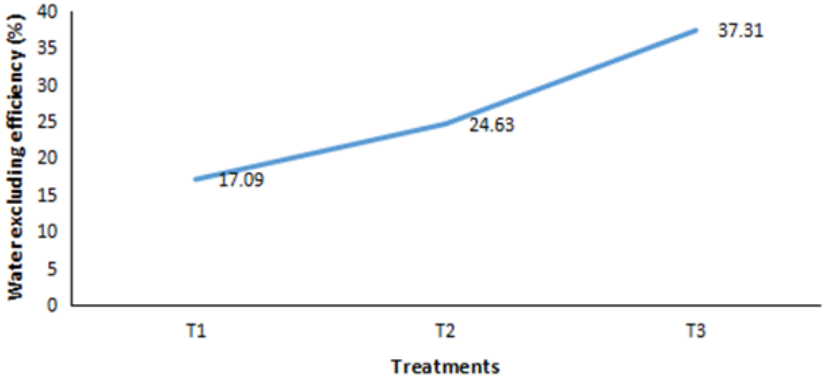
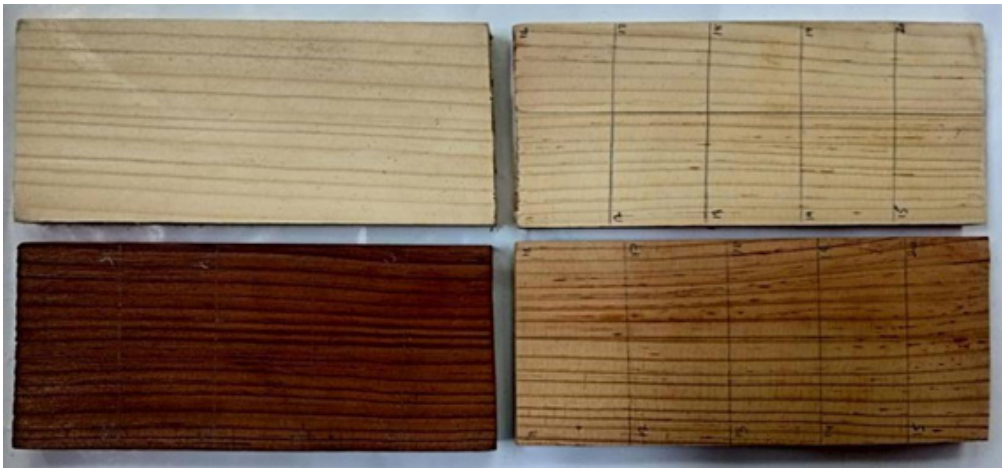


TABLE 7: Effect of stain fungus on thermally modified *Picea abies*

Treatment	Area covered (cm²)	Area covered (%)	Numerical rating	Condition of sample
Control	7.16 (±0.42)	47.75 (±2.79)[c]	5	Heavy stain
T1 (180°C)	5.06 (±0.32)	33.75 (±2.14)[b]	3	Moderate stain
T2 (210°C)	3.15 (±0.25)	21 (±1.7)[a]	3	Moderate stain
T3 (240°C)	1.98 (±0.15)	13.21 (±0.99)[a]	1	Light stain

Note: The body of the table shows the average values ± 95% confidence interval of 6 replicates per treatment. Equal letters in columns indicate no significant difference between treatments, and different letters indicates significant difference between treatments according to Tukey's post hoc analysis (**α** = 0.05).

FIGURE 4: Change in colour of wood after exposing to different temperature



Bio resistance of thermally modified wood

Areas covered by sap stain fungus on treated and untreated samples of thermally modified *Picea abies* are presented in Table 7. Treatment temperature significantly increased ($p \leq 0.05$) resistance to sapstain fungus.

Stain fungus infested 47.75% of the area on the untreated samples. Infestation was 33.75%, 21% and 13.21% at 180°C, 210°C and 240°C respectively. At the end of the exposure period, the samples heat treated at 240°C showed light stain, moderate stain was observed in the samples heat treated at 180°C and 210°C and heavy stain was observed in controls; Figure 5.

Reduction in the fungal degradation was probably due to alteration of the chemical compositions which made it unpalatable to the decaying agents. After thermal modification timber also became dimensionally stable and the pH of wood also changed. All these factors can contribute to the improved bio-resistance of thermally modified wood (Boonstra et al. 2007).

Conclusions

Thermal modification improved the dimensional stability of *Picea abies* (L.) H.Karst wood. Samples treated at 240°C became more dimensionally stable than the untreated controls and samples treated at lower temperatures.

At higher temperatures wood samples became darker in colour and the overall colour change value (ΔE*) increased gradually with an increase in the treatment temperature. The main changes were increased saturation and darkening, reddening, and yellowing effects.

Thermal modification did not significantly affect MOE whereas MOR values exhibited a steep reduction with increasing treatment temperature.

Competing interests

The authors hereby declare that there is no conflict of interest associated with this manuscript to the best of their knowledge.

Authors' contributions

AS: funding, conceptualisation and laboratory resources; SG: conceptualisation, draft preparation, data analysis, satistical analysis, supervision correspondence, editing; SM: conceptualisation, execution, draft preparation, editing, analysis.

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FIGURE 5: Samples before (a) and after (b) fungus infestation

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