

Behavioral Change in Physical, Anatomical, and Mechanical Characteristics of Thermally Treated *Pinus roxburghii* Wood

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Thermal treatment of pine wood was carried out at 80, 120, 160, and 200 °C for 2, 4, and 6 hours. The highest mean values were, for specific gravity (0.492), moisture content (29.1%), and maximum moisture content (191%), whereas the lowest mean values were for specific gravity (0.418), moisture content (1.20%), and maximum moisture content (127%). The maximum mean values were for shrinkage in longitudinal plane (0.42%), radial plane (4.63%), volumetric shrinkage (9.28%), and maximum mean value tangential plane (3.67%). The minimum mean values were for shrinkage in longitudinal plane (0.04%), radial plane (2.22%), tangential plane (1.55%), and volumetric shrinkage (4.88%). Maximum mean values were for swelling in longitudinal (0.41%), radial (5.22%), and tangential plane (3.15%) and maximum mean volumetric swelling (7.71%), while minimum mean values were for swelling in longitudinal plane (0.08%), radial plane (2.26%), and tangential plane (1.29%) and minimum mean volumetric swelling (3.15%). The highest mean values were for tensile strength (57.3 MPa) and compression parallel to the grain (50.3 MPa), the maximum mean value of bending strength (84 MPa) and compression strength perpendicular to grain (27 MPa), whereas the lowest values were for tensile strength (42.7 MPa), bending strength (7.33 MPa), compression parallel to the grain (7.33 MPa) and compression strength perpendicular to grain (12.3 MPa).

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INTRODUCTION

Wood has been used in both indoor and outdoor applications since early times. It has been receiving increased attention due to its renewable and sustainable nature. Wood quality is determined by anatomical, chemical, mechanical, and physical properties that individually or in combination, have a positive influence on different wood products. Physical properties of wood are vital parameters in the use of wood for different applications. Wood anatomy has a great role in timber identification and also in assessing the properties and uses of different wood products. Several studies have highlighted the importance of the wood anatomy in relation to wood properties and uses. Specific gravity is a very important parameter to determine wood quality, pulp yield, and strength of wood (Elliott 1970; Panshin and DeZeeuw 1970; Horn 1974). Mechanical properties are usually the most important wood quality indicator for structural applications (Bodig and Jayne,

1982). The mechanical properties of wood indicate its strength properties and behavior for specific applications (Kretschmann and Green 2000).

Thermal modification enables the upgrading of less durable softwood and hardwood species into wood products of more constant quality. It has potential to improve the dimensional stability and biodurability of timber and to reduce the equilibrium moisture content of wood (Jämsä and Viitaniemi 2001; Gosselink *et al.* 2004; Metsä-Kortelainen *et al.* 2006). In addition, it can enhance its dimensional stability (Kollmann and Schneider 1963; Boonstra *et al.* 1998; Bekhta and Niemz 2003; Epmeier *et al.* 2004; Wang and Cooper 2005) and durability (Bourgois *et al.* 1998; Tjeerdsma *et al.* 1998; Militz 2002). The most useful way to increase the utilization of heat-treated timber is to expand the knowledge of strength properties. However, the major disadvantage of this method is the reduction of mechanical strength, which has been reported by many researchers (Epmeier *et al.* 2004; Unsal and Ayrilmis 2005; Korkut 2008; Kubojima *et al.* 2008). Lignin, which holds the adjacent wood cell together, is the least heat sensitive component of the wood. Therefore, heat-treated wood invariably has a higher percentage of lignin than normal wood (Vukas *et al.* 2010).

Pinus roxburghii Sargent, which belongs to the family Pinaceae, commonly known as chir pine, extends from northern Pakistan, across northern India and Nepal to Bhutan (Wu *et al.* 1999; Arya *et al.* 2000). Its range extends longitudinally from 71° to 93°E and latitudinal distribution varies from 26° to 36°N (Sharma 2002). Chir pine holds immense economic and ecological importance in northern India, where it has been extensively cultivated for its timber and resin. However, the quality of chir pine timber is substantially influenced by spiral grain and local environmental conditions, which mold the tree's growth patterns. Moreover, chir pine serves as an affordable source of raw material for various industries, including paint, soap, cosmetics, and paper production. Pine needles from this species find application as bed material in solid-state fermentation for lactic acid production (Ghosh and Ghosh 2011). The significance of chir pine extends beyond industrial utility; its timber is highly sought after for furniture and handicraft construction, making a significant contribution to the annual income of local communities. Additionally, the mature stems of chir pine find purpose in the manufacture of traditional agricultural tools such as wooden ploughs, soil equalizers, and handles for various implements (Singh, 2014).

This study delves into the intricate relationship between wood properties, especially mechanical strength, and the thermal treatment of chir pine, shedding light on a critical research gap in the field. By comprehensively examining the effects of thermal treatment on chir pine timber, the goal of this work was to provide valuable insights into maximizing the utility of this resource while addressing the challenge of strength reduction associated with thermal modification. In doing so, this research seeks to contribute to the sustainable and efficient utilization of chir pine and, by extension, promote the broader understanding of wood modification techniques for enhancing wood quality and durability.

EXPERIMENTAL

Sample Preparation

A wooden log from a single trunk of the species was collected from the Solan market, Himachal Pradesh, India. The log was converted into the following dimensions for conducting the study:

- i) 20 mm x 20 mm x 20 mm (for moisture content and specific gravity of wood)
- ii) 20 mm x 20 mm x 20 mm (for anatomical studies)
- iii) 300 mm x 10 mm x 10 mm (for tensile strength)
- iv) 300 mm x 20 mm x 20 mm (for static bending)
- v) 50 mm x 20 mm x 20 mm (for compression parallel to the grain)
- vi) 50 mm x 20 mm x 20 mm (for compression perpendicular to the grain)

Thermal Treatment of Samples

The thermal treatment of wood, often referred to as wood heat treatment or wood modification, is a process that alters the properties of wood by subjecting it to elevated temperatures in a controlled environment. This treatment is primarily aimed at improving the durability, stability, and other desirable characteristics of wood for various applications. In the current study, a stability oven was used for thermal treatment.

Pine proper sized wood samples were modified according to the following procedure in the stability oven:

Step 1: Measuring the green weight of samples

Step 2: Oven-drying of samples at $25\pm 2^\circ\text{C}$ in order to evaporate water from the samples until constant weight observed

Step 3: Oven-heating of samples at the target temperatures ($80\pm 2^\circ\text{C}$, $120\pm 2^\circ\text{C}$, $160\pm 2^\circ\text{C}$, and $200\pm 2^\circ\text{C}$) for assumed time (2, 4 and 6 h).

Step 4: Placing samples in a desiccator in order to cool down the samples to room temperature and constant weight without access of moisture.

A total of 39 specimens were tested per test and 3 replications were taken per treatment.

Measurement of Weight and Dimensions

The dimensions for larger wood samples were measured with a scale and that of smaller ones with Digital Caliper, whereas weights (g) were recorded on an electronic balance.

Physical Characteristics

Specific Gravity

Specific gravity of the wood samples was determined by the Maximum Moisture Content method (Smith 1954). The wood samples were submerged in water until saturation. The weight of the samples at this point was recorded as weight at maximum moisture content level. These samples were then oven dried at $105\pm 2^\circ\text{C}$ until a constant weight was attained. The specific gravity was calculated as follows,

$$\text{Specific gravity} = \frac{1}{\frac{M_m - M_o}{M_o} + \frac{1}{GS}} \quad (1)$$

where M_m is the fresh or green weight of the sample having maximum moisture (g), M_o is the oven-dried constant weight of the sample (g), and GS is the average intrinsic density of wood substance (for which the pore volume is not regarded as part of the wood), a constant, having a value of 1.53.

Moisture content (%)

The weight of the wood samples was recorded immediately after conversion. After initial weighing, the samples were oven dried at 105 ± 2 °C until constant weight. The moisture content (%) of the samples was calculated by using the formula given by Desch and Dinwoodie (1996),

$$\text{Moisture content (\%)} = \frac{M_{ini} - M_{od}}{M_{od}} \times 100 \quad (2)$$

where M_{ini} is the initial weight of wood samples (g) and M_{od} is the oven dried weight of wood sample (g).

Maximum moisture content (MMC) %

Maximum Moisture Content (MMC) of wood samples was determined by the procedure prescribed as per the Indian Standard (IS: 1708 - BIS, 1986). The wood samples were submerged in distilled water for 7 days to ensure complete saturation. The saturated samples were taken out and weighed. These samples were then dried first in air and then at 105 ± 2 °C until constant weight values were obtained. The Maximum Moisture Content (%) was calculated by the following formula,

$$\text{Maximum moisture content (MMC)\%} = \frac{M_m - M_0}{M_0} \quad (3)$$

where M_m is the saturated weight of wood samples (g), and M_0 is the oven dried weight of wood sample (g).

Swelling (%)

Swelling of wood in three different planes viz., longitudinal, radial, and tangential, was calculated as follows,

$$\text{Swelling (\%)} = \frac{l_2 - l_1}{l_1} \times 100 \quad (4)$$

where l_1 is the length of wood sample (oven dry) in a plane before treatment (cm) and l_2 is the length of wood sample in a plane with treatment (cm).

The volumetric swelling coefficient (%) was calculated as follows,

$$S (\%) = \frac{V_2 - V_1}{V_1} \times 100 \quad (5)$$

where S is the volumetric swelling coefficient, V_1 is the wood volume of oven-dried wood sample before treatment (cc), and V_2 is the wood volume with treatment (cc).

Shrinkage (%)

The shrinkage of wood in three different planes, viz., longitudinal, radial, and tangential, was calculated as,

$$\text{Shrinkage (\%)} = \frac{l_2 - l_1}{l_1} \times 100 \quad (6)$$

where l_1 is the length of wood sample (oven dried) in a plane after treatment (cm) and l_2 is the length (wet) of wood sample in a plane with treatment (cm).

The volumetric shrinkage coefficient (%) was calculated as follows,

$$S = \frac{V_2 - V_1}{V_1} \times 100 \quad (7)$$

where S is the volumetric shrinkage coefficient, V_1 is the wood volume of oven-dried sample after treatment (cc), and V_2 is the wood volume (wet) with treatment (cc).

Texture

Texture means the relative size as well as the amount of variation in size of the wood cells. It depends upon the size of the cells and the distribution and proportion of the various types of cells. The texture of teak was determined and described based upon the range of tangential diameters of vessels, under four main classes (Peng *et al.* 1988):

- (a) Very fine: Mean tangential diameter of vessels <100 μm
- (b) Fine: Mean tangential diameter of vessels 100 to 200 μm
- (c) Medium: Mean tangential diameter of vessels 200 to 300 μm
- (d) Coarse: Mean tangential diameter of vessels >300 μm

The texture classification in coniferous woods was done on the basis of (I.S: 399-BIS, 1986) Indian Standard Classification of Timbers and their Zonal Classification.

Table 1. Texture Classification

	Tracheid Diameter (μm)	Texture
Very small	< 25	Very fine
Small	25-35	Fine
Medium	35-50	Medium
Large	50-60	Coarse
Very large	>60	Very coarse

Note: Values from Meier (2014)

Colour

Colour was assessed in each wood sample using the Royal Horticultural Society (RHS) Chart. Wood samples were matched with different colours in the chart and the colour Codes were recorded.

Anatomical Characteristics

Tracheid dimensions

Tracheid length was determined by macerating the wood shavings in Jeffery's solution, *i.e.* 10% chromic acid and 10% nitric acid, for 48 hours (Pandey *et al.* 1968). Thereafter, the shavings were thoroughly washed, stained with safranin, and teased with the help of needle in 10% glycerine prior to mounting on slides. Straight and complete tracheids were selected and measured under a Stereo Microscope equipped with a 10X eyepiece. 5 measurements of tracheids were made in each slide (03 Slides per treatment) using an ocular micrometer fitted to the eyepiece of a microscope at 10X magnification and standardized with the help of a stage micrometer.

Tracheid length (mm)

The lengths (mm) of the tracheids were observed from the macerated wood samples by using ocular and stage micrometer of ERMA (Tokyo, Japan) make.

Tracheid diameter (μm)

The average diameter (μm) of the tracheids was measured from macerated wood samples by measuring mid diameter of tracheids with the help of an ocular and stage micrometer of ERMA (Tokyo, Japan) make.

Mechanical Characteristics

The mechanical wood properties were tested as per the procedure followed for testing in Universal Testing Machine (Model:UTN-10). The mechanical test of wood samples was determined by the procedure prescribed by Indian Standard (IS: 1708-BIS 1986).

Tensile strength (kN/mm²)

The standard size of the wood specimens taken for conducting this test was 300×10×10 mm. The computer generated data and graphs were obtained with the Universal Testing Machine (Model: UTN-10) to obtain the values of maximum load, maximum displacement and breaking pattern for all the species. Proper care was taken such that each specimen faced similar type of test measures.

Bending strength (kN/mm²)

The standard size of the wood specimens taken for this test was 300×20×20 mm. Proper care was taken such that each specimen faced similar type of test measures. The data generated in computer were used for further analysis and comparison.

Compression strength

The compression strength parallel to the grain (kN/mm²) was measured in the direction along the grain, and the data were generated using the Universal Testing Machine (Model:UTN-10). The standard size of specimens for this compression test was 50×20×20 mm.

The compression strength perpendicular to the grain (kN/mm²) was determined for specimen size of 50×20×20 mm. The measurement was taken across the direction of the grain for carrying out this test. The data recorded were used for further analysis and comparison.

Data Analysis

The experiments were laid out in the completely randomized block design (CRBD) factorial. In total, three replicates for each treatment were taken.

Analysis of Variance (ANOVA)

The analysis of variance table was set up as shown in Table 2.

Table 2. Analysis of Variance

Source of Variation	Degree of Freedom	Mean sum of squares	Variance Ratio
Replication	(r-1)	M _r	M _t /M _e
Treatment	(t-1)	M _t	
Error	(r-1)(t-1)	M _e	
Total	(r x t)-1	M _T	

Note: r is the number of replications, t is the number of treatments, M_r is the mean sum of squares due to replication, M_t is the mean sum of squares due to treatment, and M_e is the mean sum of squares due to error.

The critical difference (CD) was calculated as follows,

$$CD = SE(d) \times t_{0.05}$$

where, SE (d) is the standard error of difference, which was calculated as,

$$SE(d) = \sqrt{2Me/r}$$

and $t_{0.05}$ is the value of t at 95% level of confidence.

RESULTS AND DISCUSSION

Extractives are located mainly in the cell lumen, and fill they fill vacant spaces in the wood. In the course of thermal modification, the extractives content is decreased, and the effect becomes more pronounced with increase in temperature. The result is an increase the porosity and thereby a decrease in specific gravity. Even the lignin deposition on the cell wall influences the specific gravity of the wood (Zobel and Talbert 1984). Figure 1 reveals significant difference in the specific gravity of wood at different temperature ranges at the 95% level of confidence. Among different temperatures, the maximum mean value of specific gravity (0.492) was recorded at 80 °C, which was statistically at par with mean control (0.487) and 120 °C treatment (0.469). The minimum mean value for specific gravity (0.418) was noticed at 160 °C, which was statistically at par with the value obtained at 200 °C (0.419). Non-significant differences were observed for all the temperatures and durations, which ranged from 0.450 to 0.465. The interactions between temperatures and durations were found to be significant, and the maximum value of 0.493 at 80 °C for 6 h was recorded, which was statistically at par with the specific gravity values of 0.473 at 120 °C for 4 h, 0.487 at control and 0.490 at 80 °C for 2 h, whereas minimum value of 0.360 was recorded at 160 °C for 2 h.

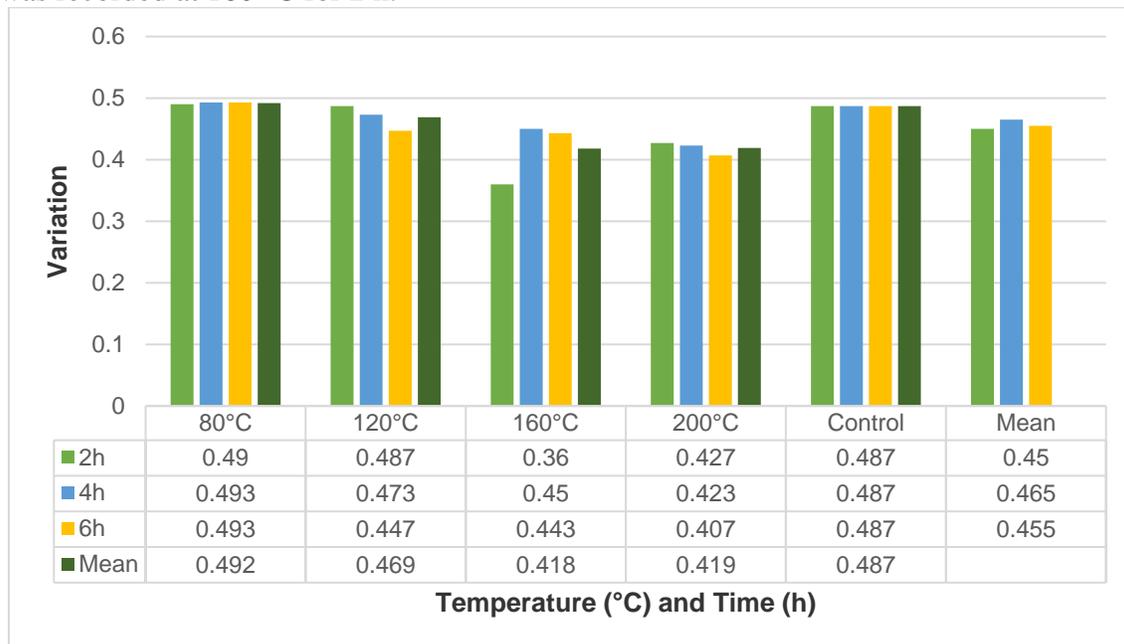


Fig. 1. Specific gravity of thermally treated *Pinus roxburghii* wood

The present results are in line with the previous study (Cademartori *et al.* 2014) on rose gum and Sydney blue gum, where the specific gravity was found to decrease with increase in temperature. Similar findings have been reported on *Luehea divaricata* hardwood. For example, the specific gravity was reported to decrease with an increase in temperature duration (Schneid *et al.* 2014).

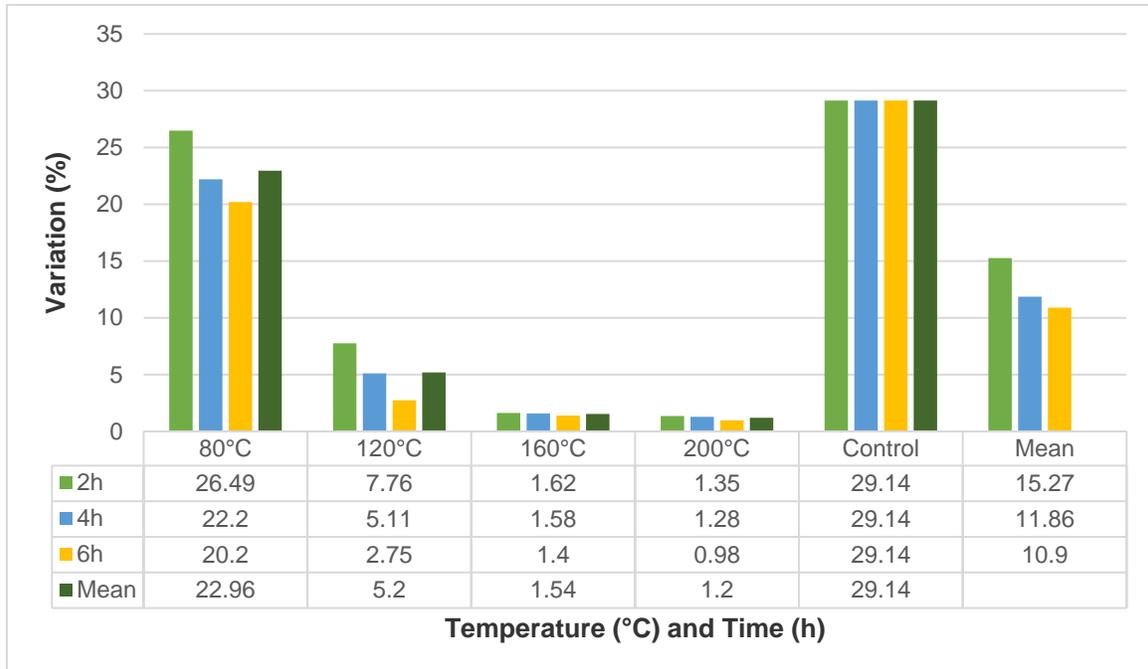


Fig. 2. Moisture content (%) of thermally treated *Pinus roxburghii* wood

The presence of moisture in wood makes it dimensionally unstable. This results in many problems in the utilization for different applications, as it has pronounced effect on its strength properties. The statistical analysis of the data related to moisture content of thermally treated wood samples are presented in Fig. 2, which shows significant differences for all the temperatures at the 95% level of confidence. The maximum mean value of moisture content of 29.1% was observed in the control, whereas minimum mean value of moisture content (1.2%) was recorded at 200 °C, which was statistically at par with the value obtained (1.5%) at 160 °C. The data recorded was also found to be significant for all the time durations. The highest value (15.3%) was recorded at 2 h and lowest (10.90%) at 6 h. The interactions between temperatures and durations were also found to be significant. The highest moisture content of 29.1% was observed in the control, while the lowest value of moisture content (0.98%) was noticed at 200 °C for 6 h, which was statistically at par with moisture content value (1.35%) at 200 °C for 2 h, (1.28%) at 200 °C for 4 h, (1.62%) at 160 °C for 2 h, (1.58%) at 160 °C for 4 h, and (1.40%) at 160 °C for 6 h.

According to the present study, it could be stated that the drying effect of thermal modification was important. The results obtained in this study were in accordance with the previous study by Candan *et al.* (2013). In that work, thermal compression technique on poplar wood was applied and it was found that with increase in temperature the moisture content decreased. Esteves *et al.* (2007) also found that the equilibrium moisture content decreased by 61% in steam-heated eucalyptus wood. Similarly, Icel *et al.* (2015) also reported that the heat-treated samples of *Picea abies* and *Pinus sylvestris* had almost half the equilibrium moisture content then the control. Unsal and Candan (2008) compared the final moisture content of thermally compressed and untreated pine wood board and found decreased moisture content in thermally compressed pine wood board.

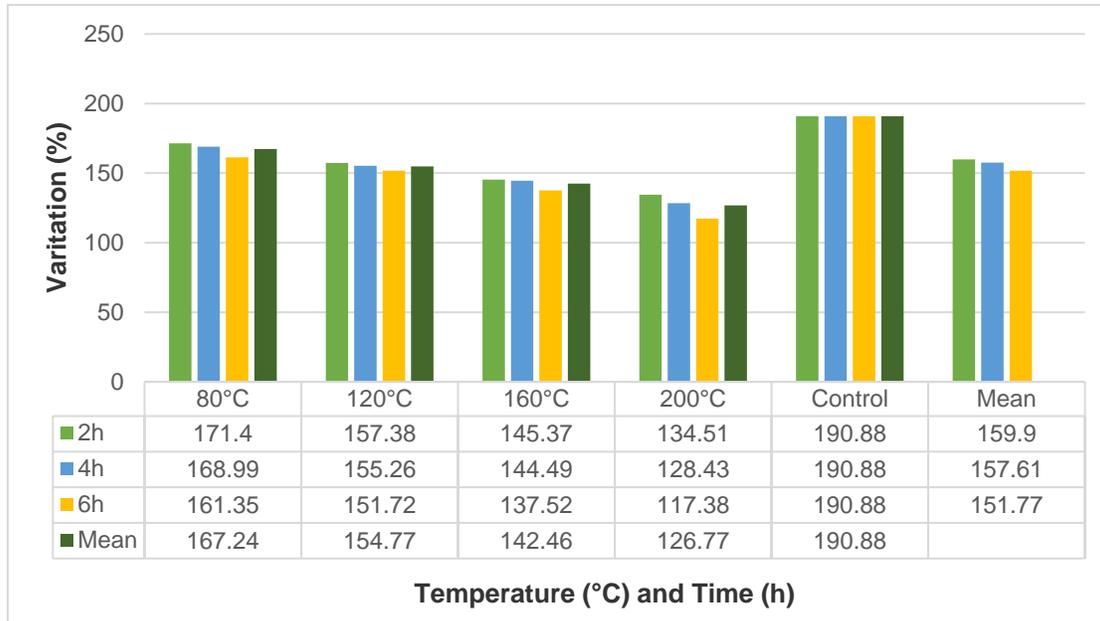


Fig. 3. Maximum moisture content (%) of thermally treated *Pinus roxburghii* wood

There were significant differences in maximum moisture content of thermally treated wood among different treatments at the 95% level of confidence (Fig. 3). The maximum moisture content was highest (191%) in control and lowest (127%) obtained at 200 °C. The results were significant for different time durations. The highest value of 160% was recorded at 2 h, which was statistically at par with 158% observed at 4 h and lowest value (152%) at 6 h. The combination of temperatures and durations were not significant, and the values ranged from 117 to 191%. The change in equilibrium moisture content could be due to decrease in OH⁻ groups, cleavage of the chains, and loss of substance at high temperatures (Akyildiz and Ates 2008). The present study is in line with the previous study of Cademartori *et al.* (2014), where it was observed that with increase in temperature the equilibrium moisture content decreases, and the highest equilibrium moisture content was reported in control samples of two eucalyptus wood (rose gum and Sydney blue gum).

Figure 4 reveals non-significant differences in tracheid length (mm) of thermally treated wood among different temperatures at 95% level of confidence. For different temperatures, the tracheid length was 3.57 to 3.72 mm. In time durations, tracheid length ranged from 3.64 to 3.71 mm. The interactions between temperatures and durations were not significant; tracheid length ranged from 3.53 to 3.83 mm. Batista *et al.* (2015) concluded that there was no effect of temperature on the dimension of fibers, vessel diameter, and ray parenchyma height of *Eucalyptus grandis* at 140, 160, and 180 °C. Andersson *et al.* (2005) observed the same for thermally treated *Pinus sylvestris*. Hietala *et al.* (2002) observed no change in the anatomical microstructure of treated and untreated Scots pine. Similar results were observed in the present study.

There was no significant difference in tracheid diameter of wood among different temperatures at the 95% level of confidence. The tracheid diameter with different temperatures ranged from 48.4 to 49.5 µm. The data on tracheid diameters at different time durations were also found to differ by non-significant amounts, and values were between 48.8 and 49.1 µm. The interactions between temperature and durations were not significant. The value of tracheid diameter recorded ranged between 48.1 and 49.5 µm.

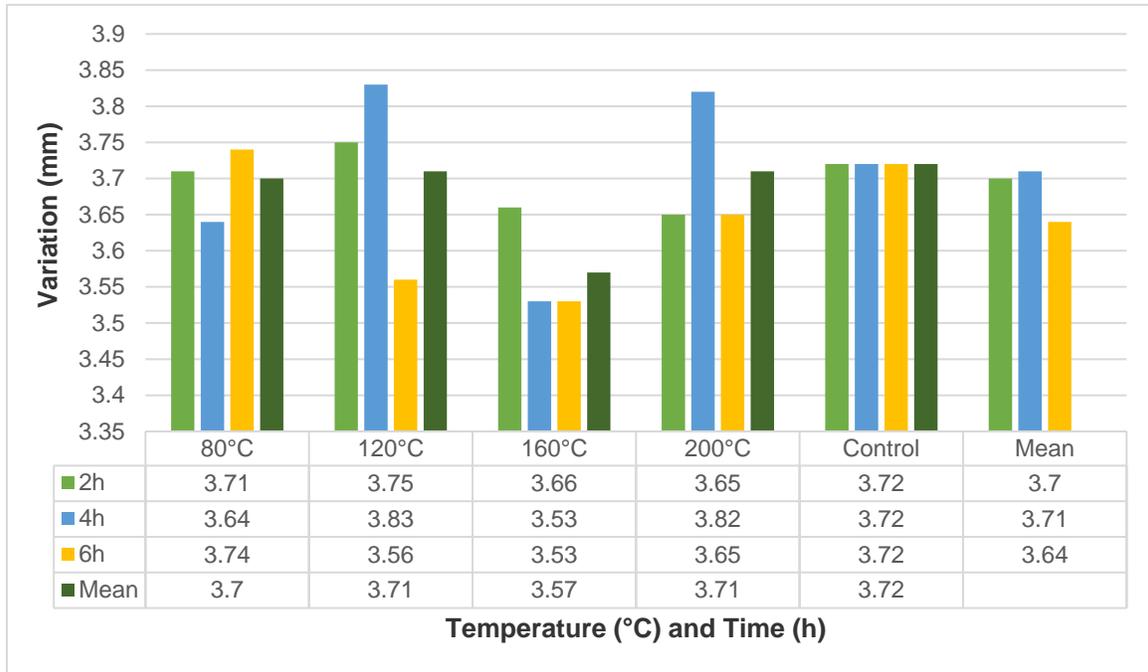


Fig. 4. Tracheid length (mm) of thermally treated *Pinus roxburghii* (mm) wood

The variation in tracheid diameter, which may be due to variable cellular and genetic characteristics only, can be found at the individual tree level, within individuals of a population, and amongst sites/populations of a species. Such variability has already been reported by Zhang and Morgenstern (1995) in *Picea mariana*.

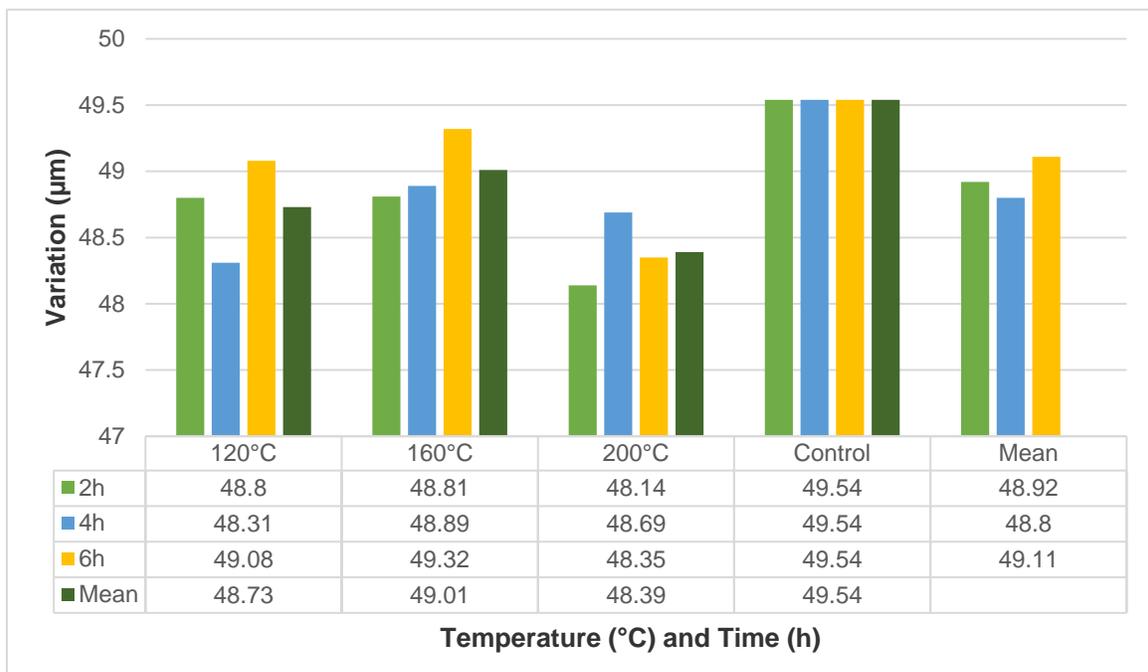


Fig. 5. Tracheid diameter (µm) of thermally treated *Pinus roxburghii* (µm) wood

Sykes *et al.* (2006) reported that fibre length is genetically controlled and is not influenced by environmental fluctuations. But the environment in combination with genetic variability also plays an important role in the alteration of fibre length, as has been observed by Tesfaye *et al.* (2007). Mburu *et al.* (2008) noted that the vessels, fibers, parenchyma, and rays of *Grevillea robusta* were not affected significantly by thermal treatment. The findings of the present study are similar to previous results.

The observations related to the texture of different heat-treated wood as presented in Table 3 revealed non-significant variation based on mean tracheid diameter. The medium texture was observed in all replicates of all treatments. Texture of the wood depends upon the size of the cells and the distribution as well as the proportion (Mahmood and Athar 1997). The coniferous woods are mainly composed of tracheids, and these are almost similar in structure. Variations in their size occur mainly due to seasonal growth only. In the present study the temperature did not showed any significant effect in tracheids length and diameter. Chauhan (2013) reported the similar texture for *Pinus roxburghii* wood.

Table 3. Texture of Thermally Treated *Pinus roxburghii* Wood

Temperature (°C)	Time		
	2 h	4 h	6 h
80 °C	Medium	Medium	Medium
120 °C	Medium	Medium	Medium
160 °C	Medium	Medium	Medium
200 °C	Medium	Medium	Medium
Control	Medium	Medium	Medium

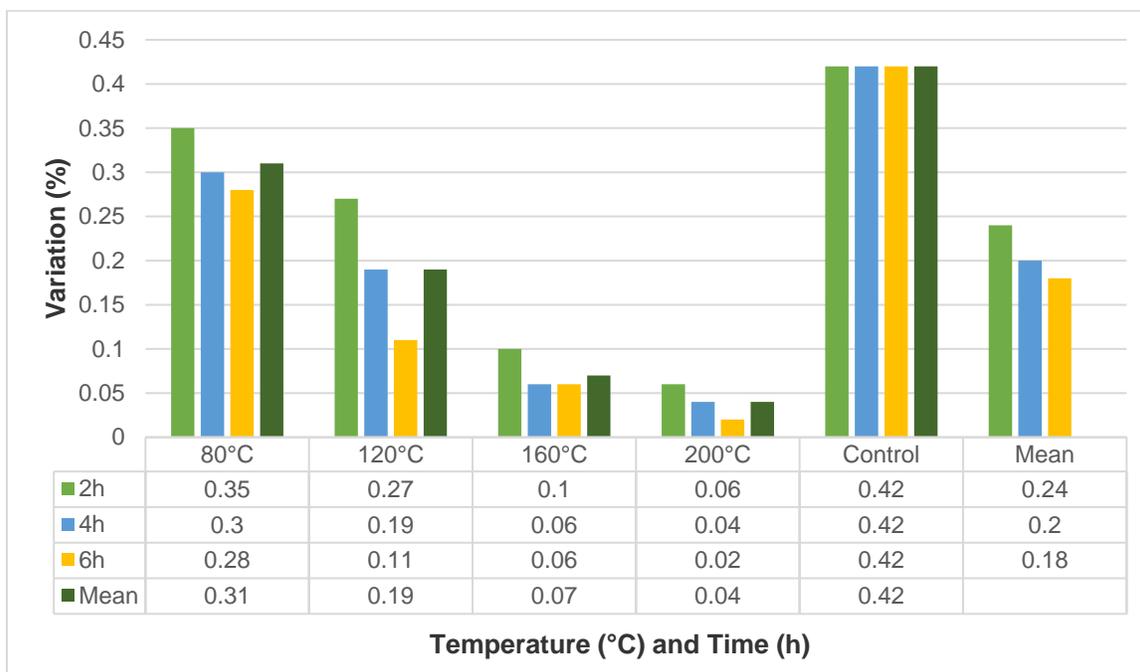
Table 4 presents the variation in colour of thermally treated wood samples. The wood treated at different time durations showed seven different colours. The wood samples treated at 80 °C for 2, 4, and 6 h showed pale yellow colour with a hue of 161 C. Pale yellow colour (162 D) was noticed at 120 °C for 2 h, whereas light yellow colour (162 C) was observed at 120 °C for 4 h and 6 h. Wood samples treated at 160 °C for 2 h and 4 h showed moderate yellowish pink colour with the hue 173 D at 160 °C for 6 h, while it showed moderate orange colour (173 C). Greyish reddish orange colour (174 C) 200 °C for 2 h and (177 C) at 200 °C for 4 h was observed and moderate reddish brown (177 A) observed at 200 °C for 6 h. The wood samples of the control had pale yellow colour with the hue 161 C. Colour change of wood during heating can be attributed to many complex chemical reactions *viz.*, oxidation, reduction, hydrolysis, formation of aldehydes and phenolic compounds.

Sundqvist (2004) reported that in the colour formation of wood, oxidative changes were more predominant than hydrolysis. Oxidation of polysaccharides and lignin may produce phenolic compounds that can induce colour change (Fengel and Wegener 1989). McDonald *et al.* (2000) revealed that when wood is heated, aldehydes and phenols are formed from degraded carbohydrates, which may result in the formation of coloured compounds after chemical reactions. On other hand, Jimenez *et al.* (2011) reported that as the treatment temperature and duration increases from 160 °C (30 min) to 220 °C (120 min), the colour change intensified from yellowish brown to chocolate brown in Malapapaya wood.

Table 4. Colour Variation in Thermally Treated *Pinus roxburghii* Wood

Temperature	Time		
	2 h	4 h	6 h
80 °C	161 C (Pale yellow)	161 C (Pale yellow)	161 C (Pale yellow)
120 °C	162 D (Pale yellow)	162 C (Light yellow)	162 C (Light yellow)
160 °C	173 D (Moderate yellowish pink)	173 D (Moderate yellowish pink)	173 C (Moderate orange)
200 °C	174 C (Greyish reddish orange)	177 C (Greyish reddish orange)	177 A (Moderate reddish brown)
Control	161 C (Pale yellow)	161 C (Pale yellow)	161 C (Pale yellow)

Figure 6 shows significant differences at different temperatures and non-significant differences in time durations and also in combination of temperatures and durations in shrinkage of wood in longitudinal directions. A critical scrutiny of results obtained at different temperatures revealed maximum mean value of shrinkage (0.42%) in control, which was statistically at par with the shrinkage value (0.31%) at 80 °C. The minimum value of shrinkage (0.04%) was observed at 200 °C, which was statistically at par with 160 °C (0.07%). In time durations the values of shrinkage ranged between 0.18 to 0.24%. Among interactions the range of shrinkage values varied between 0.02% and 0.42%.

**Fig. 6.** Shrinkage (%) in longitudinal plane of thermally treated *Pinus roxburghii* wood

The availability and accessibility of free hydroxyl groups of wood carbohydrates play an important role in the process of water absorption and desorption. The heat treatment decreases the hydroxyl group in wood (Kol 2010); it was also reported that the swelling and shrinkage in all directions were decreased for both pine and fir species with the

ThermoWood process. The shrinkage in longitudinal plane decreased significantly with increase in temperature in both species under consideration. The same decrease in the shrinkage with increase in the temperature has also been observed by Akyildiz *et al.* (2009) in Anatolian black pine wood. In the present study, similar results have been observed for *Pinus roxburghii*, where the shrinkage decreased with increase in treatment temperature.

Analysis of variance reflected significant differences at different temperatures for shrinkage of wood in the radial plane (Fig. 7). The data revealed a maximum mean value (4.6%) in the control which was statistically at par with shrinkage value of 3.5% at 120 °C and 3.8% at 80 °C. The minimum mean value of (2.2%) observed for 200 °C, was found to be at par with 3.1% at 160 °C. Results obtained for time durations were found to be not significant. The value for shrinkage in radial plane lay between 3.31 and 3.59%. The interaction between temperature and time duration were also found to be not significant at the 95% level of confidence, and the values ranged from 1.9 to 4.6%. These results are in line with Kol (2010); *i.e.* the shrinkage in the radial plane decreases with increase in temperature in both pine and fir species. In a previous study (Icel *et al.* 2015) reported that heat-treated wood of *Picea abies* and *Pinus sylvestris* became dimensionally more stable as compared to the controls. Significant decreases in shrinkage values of heat-treated wood of Douglas fir and Corsican pine were observed, as compared to untreated wood sample (Romagnoli *et al.* 2015). In the present study, similar results were observed for treated and untreated samples of *Pinus roxburghii*.

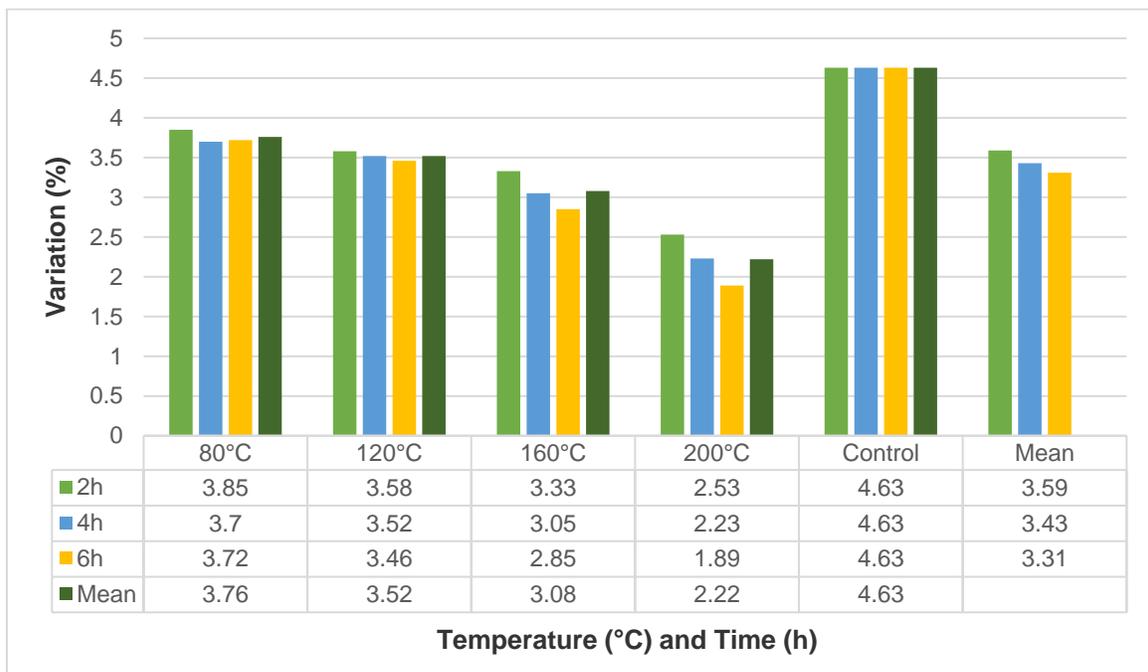


Fig. 7. Shrinkage (%) in radial plane of thermally treated *Pinus roxburghii* wood

A critical scrutiny of the data (Fig. 8) revealed statistically significant differences for shrinkage of wood in the tangential plane with temperature treatment. The maximum mean shrinkage value (3.7%) was found at 120 °C, which was statistically at par with (3.4%) at 80 °C and (3.6%) in control. The minimum mean shrinkage value (1.6%) was observed at 200 °C. The duration of temperature and the interaction between temperature and time durations was found to be not significant. The value of shrinkage among duration

ranged from 2.9 to 3.2%. The value of shrinkage in tangential plane ranged from 1.4 to 3.7% in the case of interaction between temperatures and durations. The earlier studies conducted by Kol (2010) and Akyildiz *et al.* (2009) revealed that the wood became more dimensionally stable in all direction with increase in temperature. The comparative decrease could be seen in untreated and thermally treated wood of pine, fir, and black pine wood species. Antons *et al.* (2018) concluded that the temperature treatment of birch and pine wood resulted in the improvement of wood hydrophobicity. The findings of the present study are in line with above results obtained by different workers.

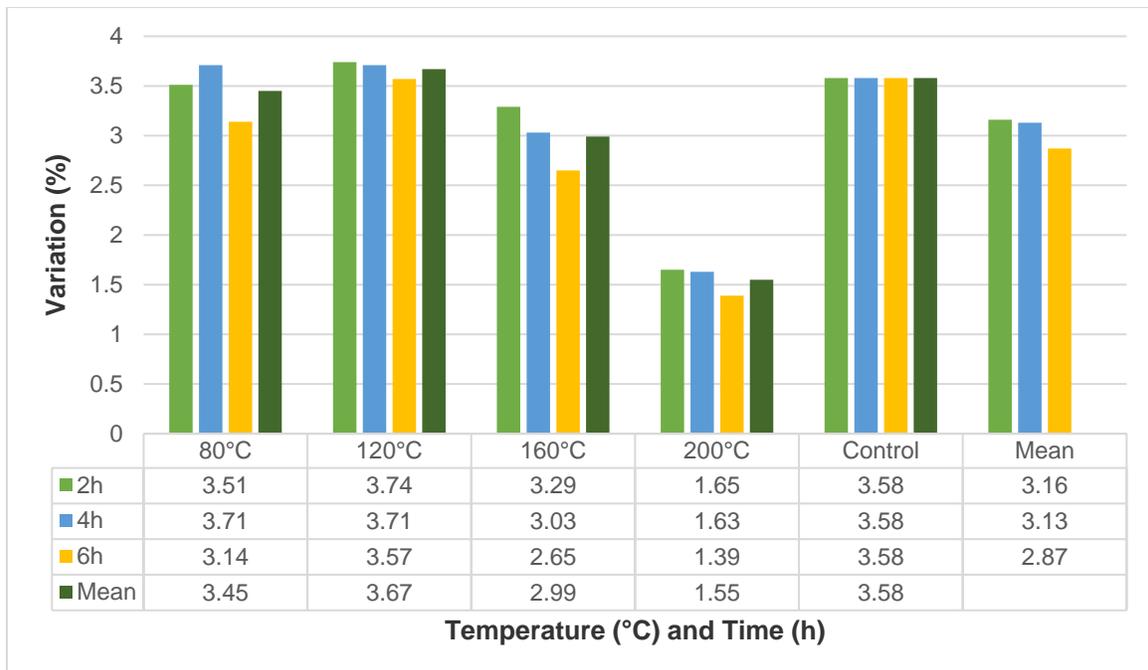


Fig. 8. Shrinkage (%) in tangential plane of thermally treated *Pinus roxburghii* wood

Figure 9 shows statistically significant values for volumetric shrinkage coefficient in different temperature ranges at the 95% level of confidence. The highest mean value 9.3% was recorded for the control, which was statistically at par with 80 °C (8.75%), and the lowest mean value (4.9%) was observed at 200 °C. The duration of treatment and the interaction between temperatures and durations was found to be not significant. The value of volumetric shrinkage among duration ranged from 7.2 to 7.6%. Interactions between temperatures and durations were also found to be not significant and values of volumetric shrinkage varied from 4.5% to 9.3%. A similar decrease was observed for volumetric swelling and shrinkage with thermal treatment.

According to Viitaniemi (1997), reductions in shrinkage and swelling (30 to 80%) for spruce, pine, and birch wood were found at temperatures between 185 and 250 °C. Akyildiz *et al.* (2009) also concluded that the value of volumetric shrinkage decreases with increase in the heat treatment in black pine wood at 130, 180 and 230 °C for 2 and 8 h. Kol (2010) also reported that the values of shrinkage in all planes decreased with increasing temperature in both treated pine and fir wood at 190 and 212 °C for 2 h. In the present study, the values of volumetric shrinkage also decreased with an increase in temperature, and results were in line with the results of above studies.

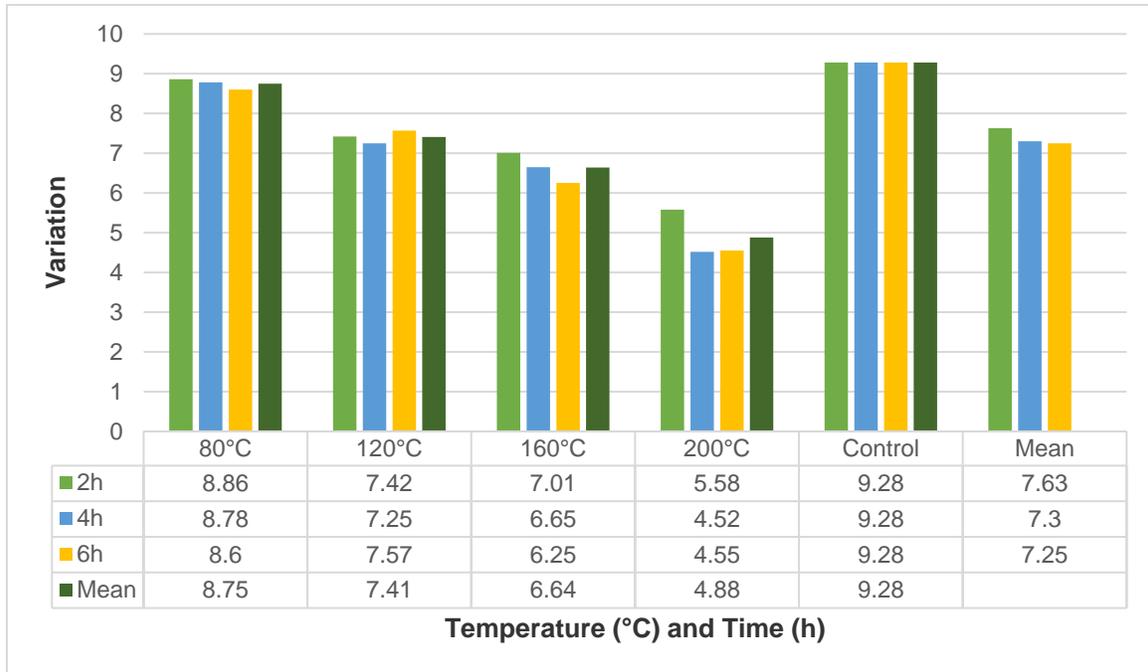


Fig. 9. Volumetric shrinkage coefficient in of thermally treated *Pinus roxburghii* wood

Figure 10 depicted significant differences in swelling of wood in the longitudinal plane at different temperatures and time durations. Among different temperatures, the maximum swelling of 0.41% was recorded in the control and the minimum mean swelling of 0.08% was observed at 200 °C. Relative to durations, maximum swelling of (0.27%) was recorded at 2 h, which was statistically at par with 4 h (0.25%), and the minimum swelling of 0.22% was noticed at 6 h duration.

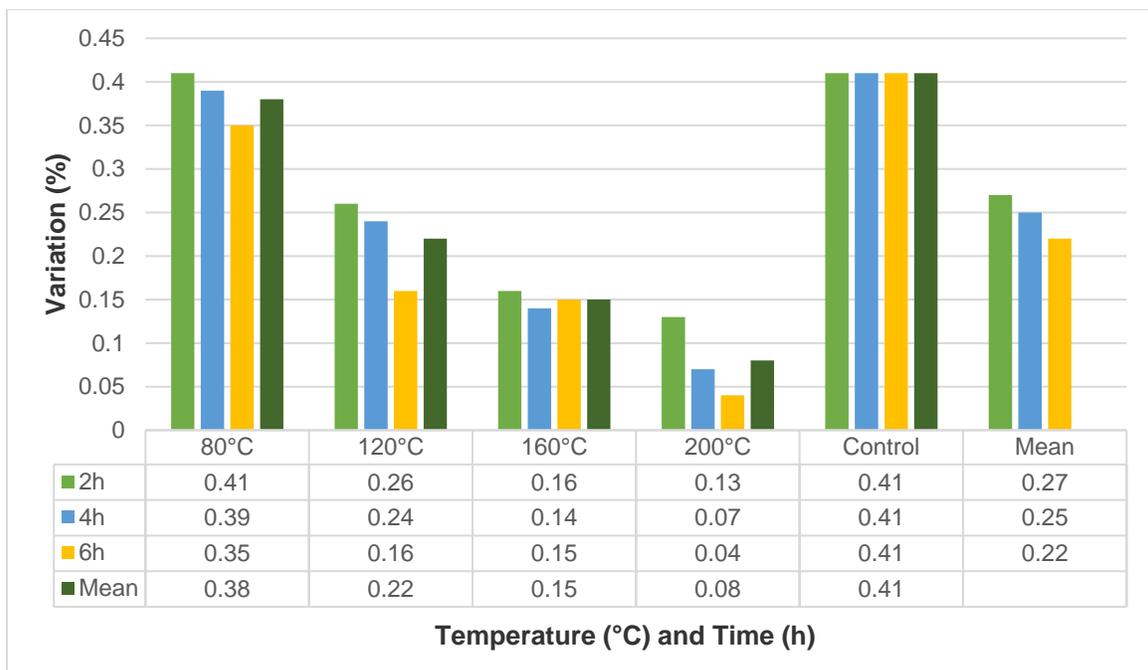


Fig. 10. Swelling (%) in longitudinal plane of thermally treated *Pinus roxburghii* wood

The combined effect of temperatures and durations were also found to be significant. The maximum value of 0.41% was found at 80 °C for 2 h, and the control was statistically at par with (0.39 %) at 80 °C for 4 h. The minimum value (0.04%) was recorded at 200 °C at 6 h, which was statistically at par with (0.07%) at 200 °C at 4 h. The dimensional stability is determined mainly by the number of OH⁻ free groups on the amorphous polysaccharides present in the wood structure and wood permeability (Ding *et al.* 2011). Taghiyari *et al.* (2011) also observed that the permeability of beech wood drastically decreased after exposure to hydrothermal and hygrothermal conditions due to the deposition of extractives on vessel performance and blocking of fluid transfer through the pores of wood by cell walls. Kol (2010) also observed the similar decrease in swelling in longitudinal plane of heat-treated Turkish pine and fir. The swelling in longitudinal plane of untreated wood was observed as 0.18%, whereas in heat-treated wood it was recorded as 0.07%. Similarly, in the present study the swelling in heat-treated wood was less than in untreated sample in longitudinal plane.

Figure 11 shows statistically significant results for swelling of thermally treated wood in the radial plane at different temperatures and time durations. The highest mean value of (5.2%) swelling was recorded in control and was found to be at par with the value of 4.9% at 80 °C. The lowest mean value (2.3%) was recorded at 200 °C. The results were also found to be significant for the durations, whereas the highest value (4.2%) was noticed at 2 h, which was at par with the value of 4.0% at 4 h. The minimum value (3.8%) was observed at 6 h.

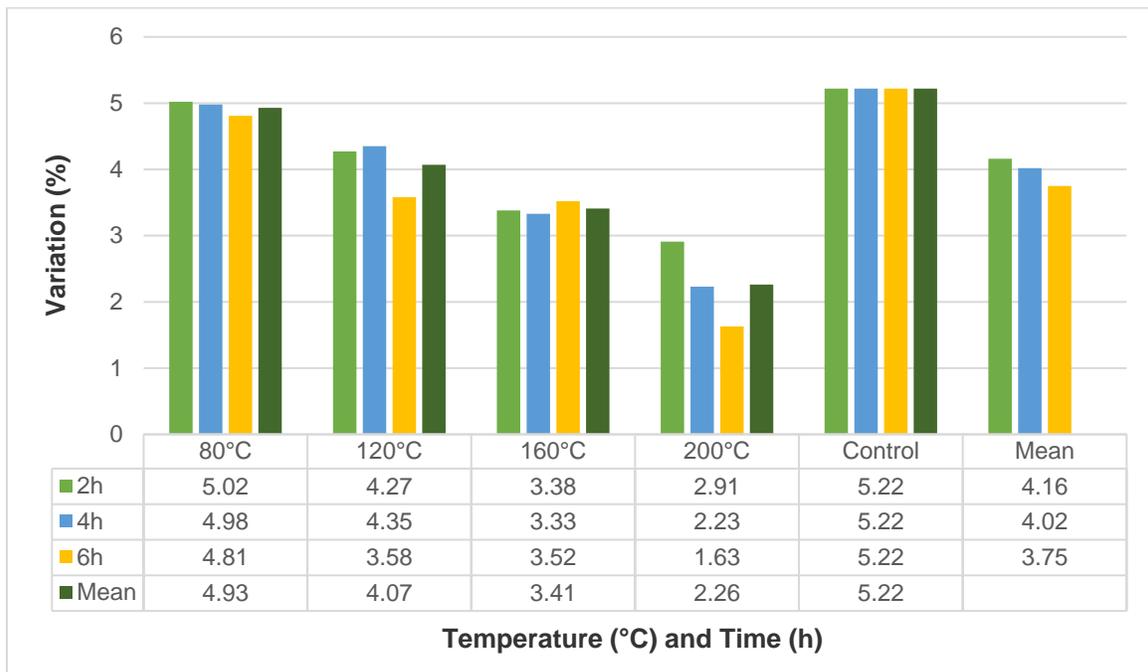


Fig. 11. Swelling (%) in radial plane of thermally treated *Pinus roxburghii* wood

The interaction between temperature and duration was found to be not significant, and the value of swelling ranged from 1.6 to 5.2%. The shrinkage and swelling behavior of heat-treated black pine wood was found to be affected positively due to an increase in lignin ratio and the undamaged carbohydrates with crystalline structure (Akyildiz *et al.* 2009). Those authors also reported the reduction of swelling in radial plane with an increase

in temperature. Jimenez *et al.* (2011) also revealed that the control samples of Malapapaya swelled by 2.9%, while the heat-treated sample of Malapapaya swelled by 0.45% (220 °C for 120 min). The results revealed the reduction in radial swelling of the treated samples by 84.8% to 16.6%. The results of present investigation also revealed the reduction in radial swelling of the treated sample.

The swelling of thermally treated wood in tangential plane showed significant variation with respect to temperature (Fig. 12). The highest mean swelling of 3.2% was noticed in the control, which was at par with 3.02% (80 °C), and the minimum mean value (1.29%) was recorded at (200 °C). Non-significant variations were noticed among the durations and ranged between 2.3% and 2.5%. The combinations of temperatures and durations were also found to be not significant, and values of swelling ranged from 1.1% to 3.2%. With the increase in temperature and duration treatments, the percentage of tangential swelling decreased. Jimenez *et al.* (2011) also reported the significant decrease in the tangential swelling of treated sample as compared to a control, and the maximum tangential swelling was observed in the control (4.4%) and minimum at 220 °C (0.6%). Cademartori *et al.* (2014) also reported linear swelling in tangential direction for both rose gum and Sydney blue gum woods and showed that the highest reduction in the tangential section was 70% at 240 °C. Akyildiz *et al.* (2009) also reported that the swelling in tangential direction decreased by increasing temperature and time duration in Anatolian black pine wood at 130, 180 and 230 °C for 2 and 8 h (Dubey *et al.* 2011). They studied the effect of heat treatment with oil on the swelling of *Pinus radiata* wood and reported a similar decrease in tangential swelling with increase in time durations. The highest value of swelling in radial plane was observed in untreated sample, whereas the lowest value was recorded at 210 °C.

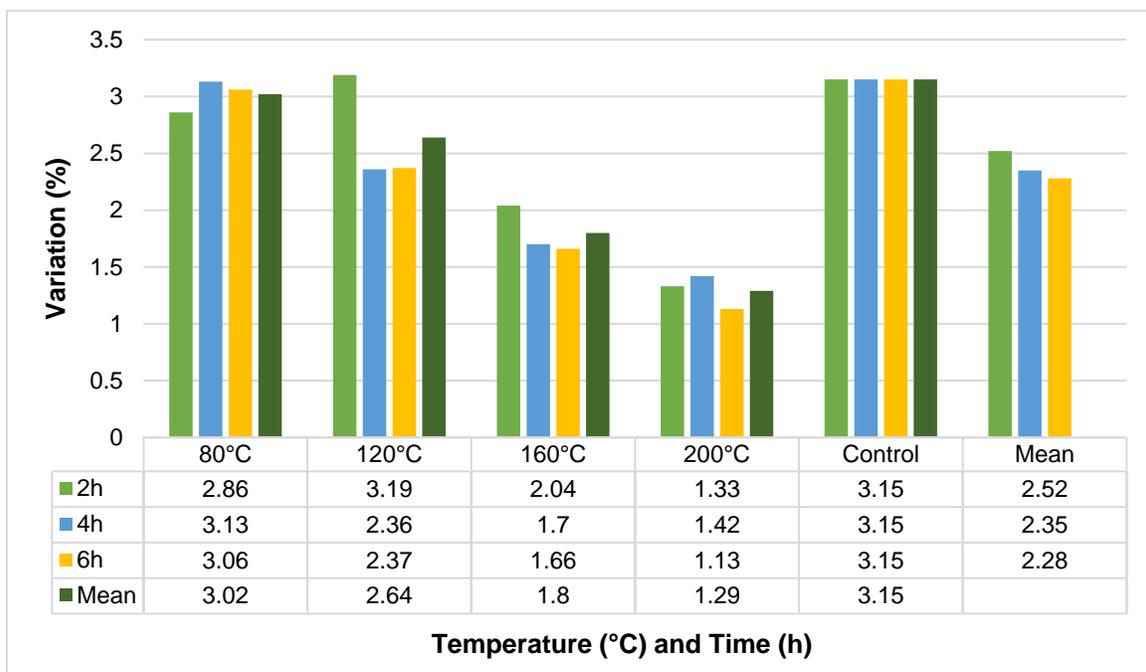


Fig. 12. Swelling (%) in tangential plane of thermally treated *Pinus roxburghii* wood

The data on volumetric swelling coefficient of wood in different temperature were observed to be statistically significant (Fig. 13). The highest mean volumetric swelling coefficient of (7.7%) was recorded at 120 °C, which was statistically at par with control (7.4%) and at 80 °C (7.2%). The lowest mean value (3.2%) was observed at 200 °C, which was statistically at par with the value (4.6%) at 160 °C. The durations and combinations of temperatures and durations were found to be not significant. The value of volumetric swelling coefficient of wood at different time durations ranged from 5.7 to 6.6%, whereas the values for interaction between temperatures and durations were found to be between 2.9 and 11.2%. Jimenez *et al.* (2011) reported the reduction in the water absorption capacity of thermally treated samples of Malapapaya wood at 220 °C at 2 h. This shows a substantial reduction in hygroscopicity of the treated materials after 24 h treatment. Cademartori *et al.* (2014) has also observed that with an increase in the temperature, the value of volumetric swelling decreases significantly, for both *Eucalyptus* species, *i.e.* approximately 64% (rose gum) and 65% (Sydney blue gum). This may be due to homogeneous degradation of the amorphous material in hemicelluloses. Kol (2010) also reported the reduction in the volumetric swelling of heat-treated wood. Mohebbi and Sanaei (2005) reported that the value of volumetric swelling in thermally treated beach wood was less than untreated wood. Similar results are observed in the current study on thermally treated *Pinus roxburghii* wood.

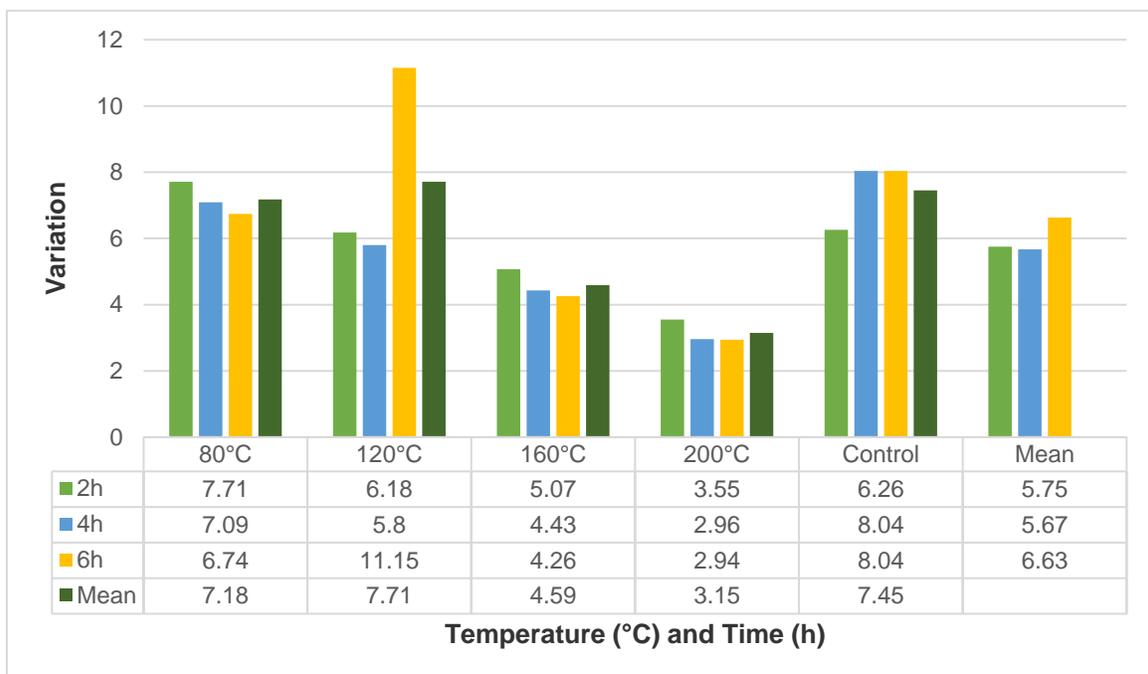


Fig. 13. Volumetric swelling coefficient of thermally treated *Pinus roxburghii* wood

The critical analysis of the data on tensile strength presented in Fig. 14 reflected significant differences at different temperatures at the 95% level of confidence. The mean tensile strength (57.3 MPa) was recorded to be the highest at 120 °C which was statistically at par with the control (54 MPa) at 160 °C (48.7 MPa) and at 200 °C (46.7 MPa). The lowest mean value of 42.7 MPa was noticed at 80 °C and was statistically at par with 200 °C (46.7 MPa) and at 160 °C (48.7 MPa). Among the durations, data were found to be not significant and ranged between 47 and 54.2 MPa. The interactions between temperatures

and durations were also found to be not significant; the observation recorded for tensile strength ranged from 33 to 62 MPa. Boonstra *et al.* (2007) revealed a clear effect on mechanical properties of thermally treated softwood species, where tensile strength parallel to grain has shown decrease in value as compared to a control after heat treatment. Changes in main components of wood results in the effect of heat treatment on mechanical properties. The decrease in the quantity of extractives, degradation of hemicelluloses and amorphous region of cellulose leads to the decrease in the tensile strength. The impact strength of Scots pine (56%), Radiata pine (80%), and Norway spruce (79%) has shown a decrease in tensile strength after heat treatment. Mainly cellulose has been primarily responsible for the tensile strength of wood (Stamm 1964; Kollman 1968). Heat treatment results in a small but remarkable degradation of amorphous cellulose polymer (Boonstra and Tjeerdsma 2006). This could be the reason for the observed decrease in tensile strength. The decreasing trend was also observed in the present study.

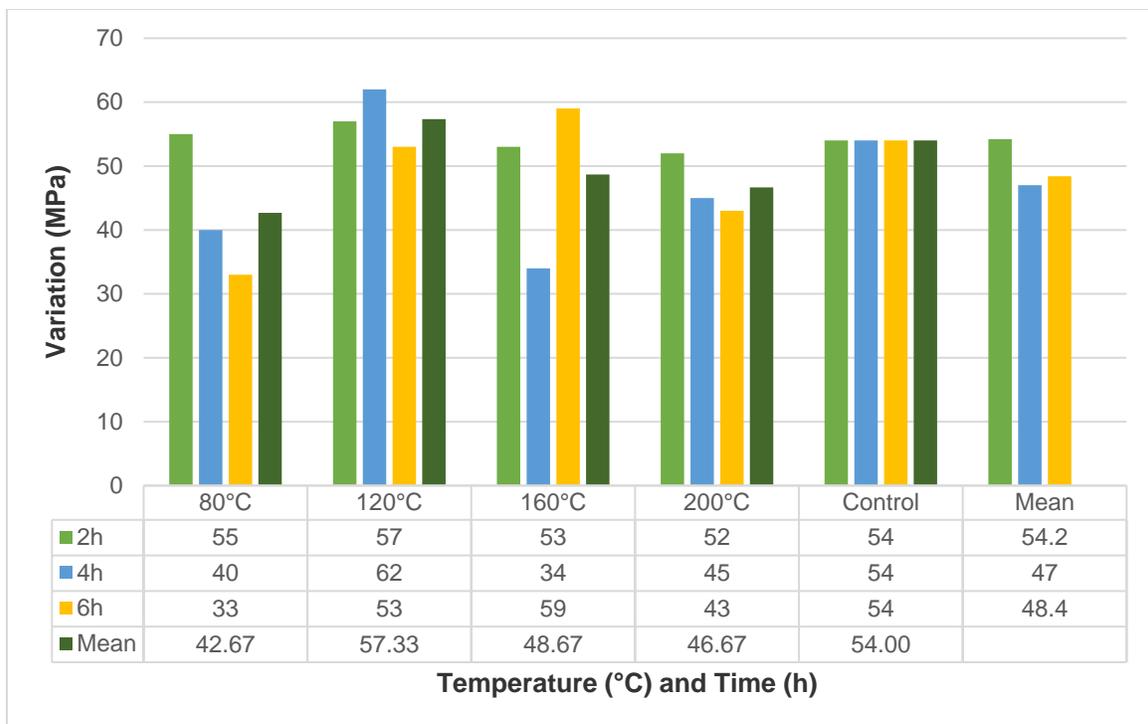


Fig. 14. Tensile strength (MPa) of thermally treated *Pinus roxburghii* wood

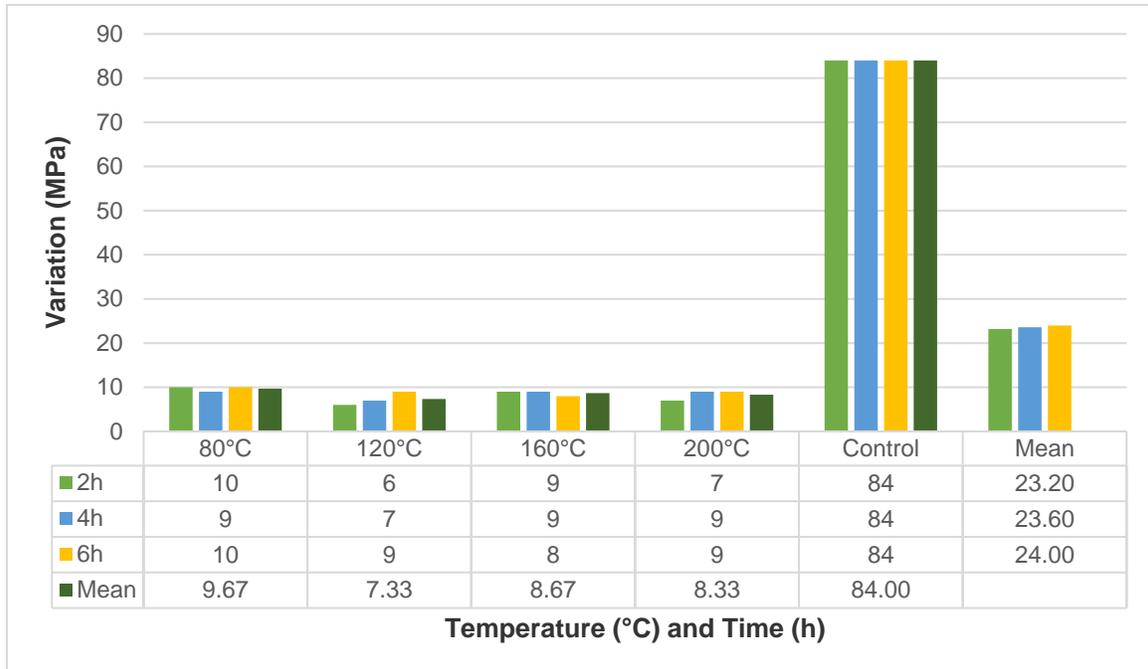


Fig. 15. Bending strength (MPa) of thermally treated *Pinus roxburghii* wood

The critical analysis of data on bending strength of thermally treated wood as presented in Fig. 15 showed significant variation among different temperatures at the 95% level of confidence. The maximum mean bending strength (84 MPa) was recorded in the control and minimum (7.33 MPa) at 120 °C, which differed significantly for all other values. Among the durations, non-significant results were found and the values ranged between 23.2 and 24 MPa. The combinations of temperatures and durations were also found to be non-significant and lay between 7.3 and 84 MPa. A relatively large decrease of the bending strength of radiata pine after heat treatment has been observed by Boonstra *et al.* (2007), which might be related to the occurrence of relative large amount of juvenile wood in pine. The chemical composition of juvenile wood differs from mature wood with higher hemicelluloses and lignin content. According to Davis and Thompson (1964), degradation of hemicelluloses is mainly responsible for a decrease of toughness. Also, in the present study, degradation of hemicelluloses took place, which may lead to a decrease in the tensile strength of *Pinus roxburghii*.

Figure 16 shows significant variation among different temperatures on compression parallel to grain at the 95% level of confidence. For different temperatures, the maximum mean compression parallel to grain was recorded as 50.3 MPa at 120 °C, which was at par with 46.7 MPa (160 °C) and 42 MPa (200 °C). The minimum mean value (17.3 MPa) was observed at 80 °C, which was at par with 19 MPa (control).

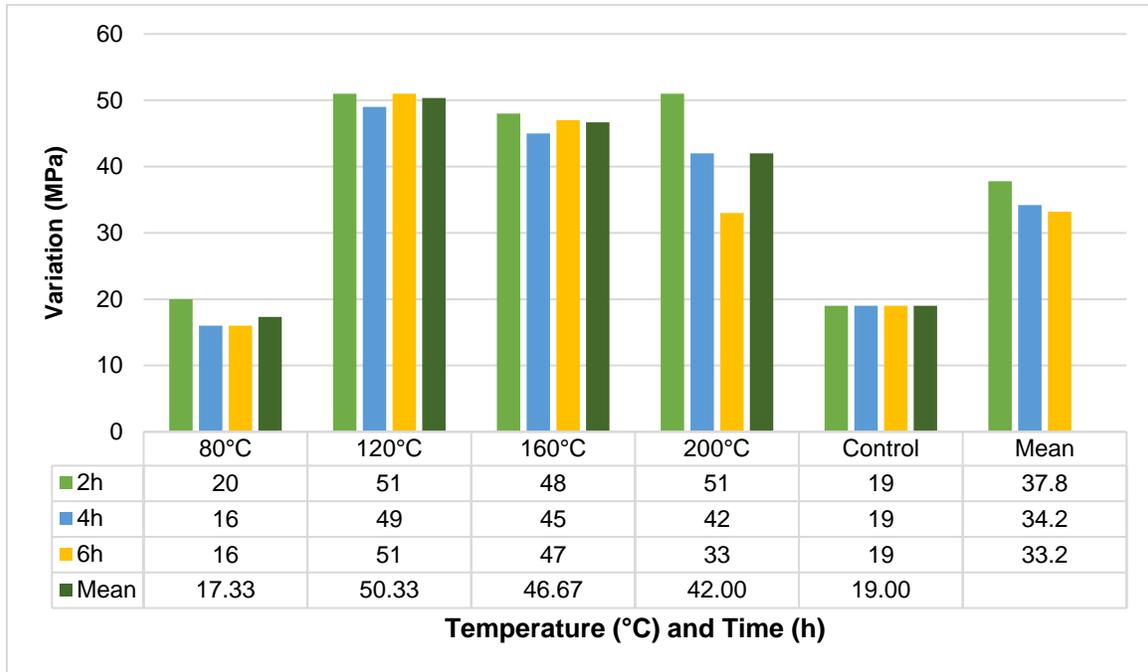


Fig. 16. Compression (MPa) parallel to the grain of thermally treated *Pinus roxburghii* wood

Among the time durations, the non-significant values were observed in the range between 33.2 and 37.8 MPa. The interactions between temperatures and durations were also found to be not significant, and data ranged between 17.3 and 50.3 MPa. The cellular orientation perpendicular to the grain makes the wood weaker in compression, as cell layers in this direction may have variable cell types with weaker linkages. Such type of results have been observed in the earlier study by Boonstra *et al.* (2007) at 165 °C and 185 °C for 30, 45, 60, and 90 minutes time duration. The compressive strength parallel to grain was increased (28%) significantly after heat treatment. The increase of the compressive strength in longitudinal direction was due to a lower amount of bonded water in heat-treated wood; however, it is expected that the amount of bound water must be higher to affect the strength properties. Also in the present study the compression strength parallel to the grain was observed to be more than that of the control.

The critical analysis of data on compression perpendicular to the grain is presented in Fig. 17, which shows significant variation among different temperatures at 95% level of confidence. The highest value compression perpendicular to grain (27 MPa) was recorded in the control, where the minimum mean value (12.3 MPa) was recorded at 160 °C, which was statistically at par with 16.3 MPa (80 °C) and 13 MPa (200 °C). Among durations, the data was recorded to be not significant and ranged between 17.2 and 18.4 MPa. The combinations of temperatures and durations were also found to be not significant. The values ranged between 12.3 and 27 MPa. The compressive strength and hardness perpendicular to the grain was much lower than parallel to the grain. Different types of bonds were present in different directions. Strong and stiff bonds were oriented along the chain axis, whereas weak and soft secondary bonds were acting in the transverse direction. The orientation of the polymer molecules in wood, such as micro fibril angle of crystalline cellulose and rather angular structure of lignin polymer network, are thought to be the main cause for this anisotropic difference (Winandy and Rowell 1984). An earlier study by Boonstra *et al.* (2007) also reported that the compression perpendicular to the grain had

significant decrease with control sample and was less than compression parallel to grain at 165 and 185 °C.

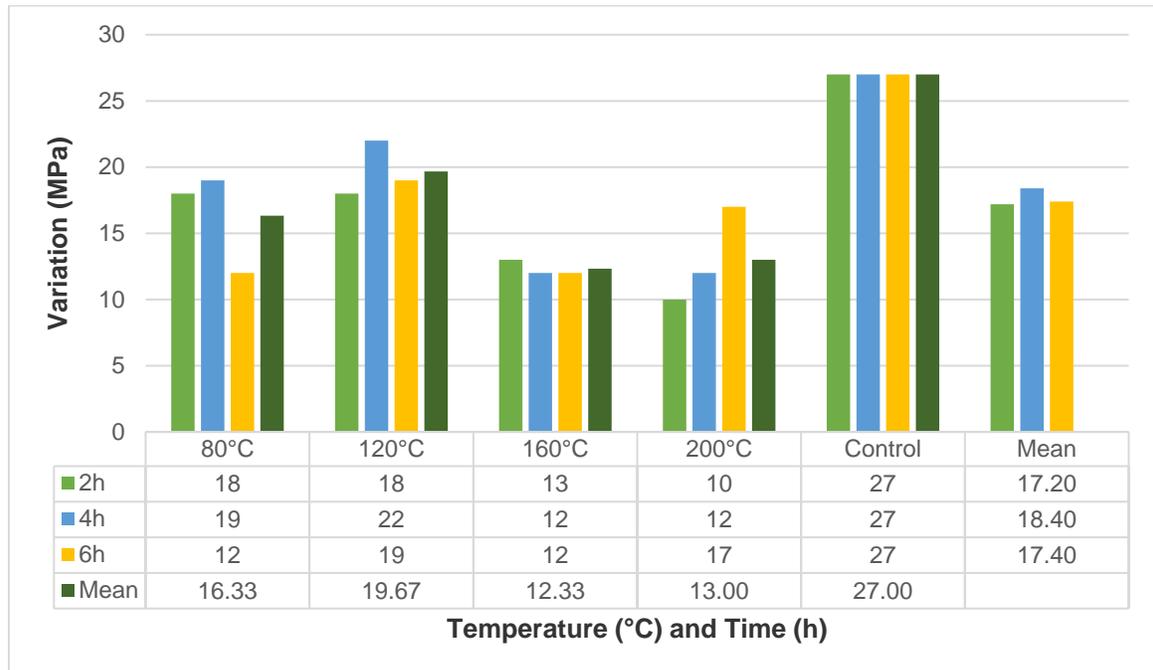


Fig. 17. Compression perpendicular to the grain (MPa) of thermally treated of *Pinus roxburghii* wood

CONCLUSIONS

1. Temperature of heat-treatment exhibited an inverse relationship with the resulting specific gravity, moisture content, and maximum moisture content.
2. Tracheid length and diameter remained unaffected by thermal modification, indicating no significant alterations in these wood properties.
3. All wood samples, whether thermally treated or untreated, displayed a consistent medium texture.
4. As temperature increased, the color of the wood deepened, resulting in a darker hue.
5. Thermal treatment led to a reduction in shrinkage and swelling across all dimensions, enhancing dimensional stability compared to the control samples.
6. The highest tensile strength was achieved at 120 °C, while the control samples exhibited the highest bending strength among all treatments.
7. In thermally treated wood, compression parallel to the grain was more pronounced compared to the control samples, whereas compression perpendicular to the grain was less prominent in thermally treated wood as opposed to the control samples.

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