Mechanical properties changes in fir wood (abies sp.), linden wood (tilia sp.), and beech wood (fagus sp.) subjected to various thermal modification process conditions

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Abstract. Wood is one of the most important construction materials in Europe and its use in building applications has increased in the recent decades. To enable even more extensive and reliable use of wood, this article aimed to determine the effect of thermal modification on mechanical properties of fir wood (lat. Abies sp.), linden wood (lat. Tilia sp.), and beech wood (lat. Fagus sp.). The thermal modification was conducted in a laboratory oven at five different temperatures of 170, 180, 195, 210, 220 °C and processed with a different maximum duration of the process of 78, 120, 180, 240, 276 minutes. Mechanical properties of treated wood have shown statistically insignificant fluctuations at lower temperatures compared to control samples. On the other hand, raising the temperature to 210 °C significantly affected the strength of all the species. The results revealed that thermal modification at high temperatures and longer exposure causes a decrease in the maximum force of the three wood species.

1. Introduction

Wood is one of the earliest construction materials. The structural use of wood and wood-based composites continues to increase steadily. In fact, new wood-based materials continue to develop and are being successfully introduced into the engineering and construction marketplace [1,2]. Wood is an important natural resource used in many applications, from construction to furniture and different domestic or industrial objects, tools, and artworks. Despite its properties, wood presents some limitations in exterior environment use, such as dimensional instability, susceptibility to fungal decay or weathering, etc. [3]. The main disadvantages of wood, such as poor dimensional stability and biological degradation or deterioration, are mainly due to the nature of the main polymers of the cell wall of wood, especially due to the abundance of free hydroxyl groups (OH), as reported in previous studies [4,5,6]. Though thermal modification has been successful in improving dimensional stability and fungal attack resistance, as stated in the research [7,8,9], it reduces the mechanical properties of wood. The researchers reported that thermal modification of wood at different temperatures and duration decreased the equilibrium moisture content (EMC), increased dimensional stability [10,11], mass loss (ML) [12,13], biological durability [14], and decreased some mechanical properties, as well as the wettability of wood, although in some studies these latter two parameters increased [15]. The diversity of wood species in combination with different thermal processes, cause changes in the physical properties, such as ML, anti-swelling efficiency, and EMC, and produce a varied range of comparable
values justifying the success of the process. ML is a determinative factor of the result of heat treatment, i.e. the greater the ML, the more severe the effects on the physical and mechanical properties. In a similar research [16] authors reported that there is a significant relationship between ML and compression strength of wood, whereas [17] observed that there is a significant relationship between ML and equilibrium moisture content of wood [18]. During previous research, a test for optimization of bending and tensile force parameters was performed. The procedure resulted in mathematical models and optimal parameters of the influence of the bending and tensile force process to the maximum fracture force [19, 20, 21]. In line of the above, for the present work, the objectives are to determine the mechanical properties of Abies sp., Tilia sp., and Fagus sp. wood under various temperatures and process durations to determine the mechanical properties of thermally modified samples using a series of standardized four-point static bending and tensile tests on the maximum force.

2. Materials and methods

2.1. Materials
The experiment used unmodified samples of beech wood (lat. Fagus sp.), linden wood (lat. Tilia sp.) and fir wood (lat. Abies sp.) for bending and elongation, whose measured average bulk density was 0.366 g/cm$^3$ for fir, 0.472 g/cm$^3$ for linden, and 0.675 g/cm$^3$ for beech samples. In the case of modified samples, the average bulk density was 0.353 g/m$^3$ for fir samples, 0.455 g/m$^3$ for linden samples and 0.655 g/m$^3$ for beech samples. The thermal modification process was performed on rectangular four-point bending samples (Figure 1) with dimensions of 380 x 50 x 20 (mm) and elongation samples with dimensions of 162 x 22 x 18 (mm).

|                | 180 °C/ 120 min | 210 °C/ 120 min | 180 °C/ 240 min | 210 °C/ 240 min | 195 °C/ 180 min |
|----------------|----------------|----------------|----------------|----------------|----------------|
| Fir - Control  | ![](image1)     | ![](image2)     | ![](image3)     | ![](image4)     | ![](image5)     |
| Lime - Control| 195 °C/ 180 min | 170 °C/ 180 min | 220 °C/ 180 min | 195 °C/ 78 min  | 195 °C/ 276 min |
| Beech - Control| 180 °C/ 120 min | 210 °C/ 120 min | 180 °C/ 240 min | 210 °C/ 240 min | 195 °C/ 180 min |

![Figure 1](image6). Thermally modified samples of fir, linden, and beech wood during different processing processes

2.2. Thermal Treatment
Thermal modification of wood is a process in which the molecular structure of cell wall polymers (cellulose, hemicellulose and lignin) are changed. This can lead to crosslinking, reduction of OH groups and (unwanted) chain cleavage. The reduction of available OH groups leads to limited interaction with water in relation to untreated wood. The structure of the cell wall of wood mainly consists of cellulose, hemicellulose and lignin. All of these components contain free hydroxyl groups. These hydroxyl groups play a key role in the interaction between water and wood. At the same time, these groups present the most reactive space in wood. If wood is exposed to humid conditions, water molecules accumulate between the wood polymers and form hydrogen bonds between the hydroxyl groups and the individual water molecules. This water needs space between the components of the cell wall, which leads to
swelling of the wooden piece, and fungi and insects infestation (Figure 2). The wood industry is continuously developing advanced processes, materials and solutions to meet the requirements and increase market competitiveness. One such treatment that occurs is thermal modification of wood.

Thermal modification of wood is performed under controlled conditions at high temperatures. In the process of modification, only heat, water vapor and fresh water are used, without the application of any chemical preparations or additives, which makes it completely environmentally friendly. It is performed at temperatures between 160 °C and 260 °C, in specially designed chambers equipped with a fully automated process control system. The use of high temperature, pressure and water vapor significantly increases the quality of wood and its physical properties. The most important parameters on which the properties of thermal modification of wood depend are the following: type of heating medium, duration of the process, final temperature, wood moisture before temperature, and type of wood. Prior to the thermal modification (TM) process, all samples in this research were marked according to the experimental plan and oven-dried in the drying chamber of the manufacturer “Kambič”. The thermal modification was performed according to a commercial process (Silvapro®, Silvaprodukt, Ljubljana, Slovenia), with an initial vacuum in the first step of the treatment [22,23]. The samples were heated to maximum temperatures ranging between 170 ºC - 220 ºC and treated with a different maximum duration of the TM process in different time intervals of 78, 120, 180, 240, 276 minutes depending on the wood species.

Figure 3 shows four parameters in the heat treatment process of beech wood samples (lat. Fagus sp.), linden wood samples (lat. Tilia sp.), and fir wood samples (lat. Abies sp.), where the marked curves of temperature and pressure are present in the diagrams.
2.3. Bending and tensile testing procedures

Solid wood for various construction applications must be sorted by its strength before exploitation. To make the process as economical as possible, the measurement usually focuses on the most important mechanical properties: density, modulus of elasticity (MOE), and bending and tensile strength. Testing in this paper was carried out in accordance with standard EN 408 (2012) [24]. The tests of the maximum force ($F_{\text{max}}$) on bending and elongation of five samples per cycle of thermal modification were performed. The samples were always positioned in such a way that they were subjected to maximum load. The test samples, having a minimum length of approximately 19 times the depth of the section, were simply supported and symmetrically loaded in bending at two points over a span of approximately 18 times the depth. The maximum force was measured within the loading points which is shown in Figure 4 a.

![Mechanical tests: a) bending test: 4-point bending procedure, b) tensile test c) dimensions of samples of rectangular cross-section b/h [24]](image)

The tensile test in parallel with the fibers was carried out by EN 408 + A1, where the wood of full cross-section of length 9 is the thickness of the tested samples (Figure 4 b). As mentioned above, the experiments used testing machine type SIL-50KNAG, manufactured by SHIMADZU, in which a working cylinder is located in the upper part of the testing machine. The working cylinder moves down (bending) and up (elongation) depending on the test, while the lower head is stationary. Then, through a computer connected to the SIL-50KNAG type testing machine, the bending is measured at four points so that the values of the force achieved can be read in the diagram.

3. Results and discussion

3.1. Mass loss (ML) of thermally modified samples

The results have shown a growing trend of ML with increasing thermal modification temperature from 170 °C to 210 °C in bending and tensile samples (Figure 5). The lowest ML were recorded for linden bending and tensile samples at a temperature of 170 °C and duration of 180 minutes while the highest ML were recorded in bending samples of linden wood at a temperature of 220 °C and duration of 180 minutes and tensile samples of beech wood at a temperature of 210 °C for 240 minutes. This indicates that degradation of cell-wall polymers was very low at this temperature. It is expected that mostly hemicellulose and lignin were degraded at this temperature leading to the ML as cellulose tends to have higher resistance to thermal degradation in comparison to hemicellulose and lignin. [25,26,29]. It is noticeable from the diagrams (Figure 5 a and b) that the ML increased with increasing temperature and treatment time. This paper determined that the weight loss was inversely proportional to the increase in
temperature and length of thermal treatment which is in line with the findings of previous researchers [13].

**Figure 5.** Mass loss in fir, linden and beech wood heat-treated from 170 to 210 °C: a) samples for four-point bending; b) tensile samples

### 3.2. Bending strength
The average maximum four-point bending strength (Fmax) of thermally modified wood showed statistically insignificant fluctuations at low temperatures and shorter duration of the thermal modification process compared to unmodified samples. This is considered to be corroborating evidence of low cell wall polymer degradation at 210 °C/120 minutes for thermally modified fir wood. On the other hand, the increase in temperature and duration time had a significant impact on all properties. There was a significant decrease in maximum force (Fmax), indicating degradation in cell wall polymers, as can be seen in Figures 6 and 7. Thermal modification in this study was performed at five different temperatures and process durations causing a glass transition of lignin in certain wood species, as noted through research in [11,28]. Therefore, as a binder, lignin crosslinking, formation of new bonds...
and lignin binding points with cellulose and hemicelluloses, which have been thermally developed, can occur [29, 30]. Also for cellulose, the crystallinity remains intact initially but with prolonged exposure the heat degradation becomes dominant and results in strength loss.

**Figure 6.** Average value of maximum bending strength of thermally modified and unmodified wood

Due to the crosslinking of lignin and binding to cellulose, there is a partial increase in the $F_{\text{max}}$ value in fir wood at a temperature of 210 ºC for 120 minutes. Although the increase was not statistically significant, there was still an increase of 2.18 % which can be seen in Figures 6 and 7. Crosslinking of lignin has put thermal degradation in the foreground, causing small increases in mechanical properties in fir wood at a temperature of 210 ºC during 120 minutes. This indicates that the degradation of the cell wall polymers was very small at this temperature and time duration. At this temperature, hemicellulose and lignin decomposed, whereas cellulose had a higher resistance to thermal decomposition compared to hemicellulose and lignin.

The $F_{\text{max}}$ ratio decreases (Figures 6 and 7) in linden wood at a temperature of 195 ºC for a process duration of 180 minutes, where there was a slight decrease of 3.35 %. Similar decreases in Fmax were recorded in cycles in the same tree species where at a temperature of 170 ºC for 180 minutes there was a decrease of 11.88 %, while at temperature of 195 ºC for 78 minutes the decrease was 9.51 %. The largest reductions in bending $F_{\text{max}}$ of 51.71 % were recorded in linden wood at a maximum temperature of 220 ºC for 180 minutes. There were also larger reductions at 195 ºC for 276 minutes where the reduction was 34.68 % compared to unmodified linden samples.

The value of bending $F_{\text{max}}$ of unmodified and modified beech samples is shown in Figure 6. The data show a large decrease of $F_{\text{max}}$ in thermally modified beech wood, especially at a temperature of 210 ºC for 240 minutes with $F_{\text{max}}$ decrease of 63.12 %, 26.35 % decrease at a temperature of 210 ºC for 120 minutes, 28.97 % decrease at 180 ºC for 260 minutes, and about 45.34 % decrease at a temperature of 195 ºC for 180 minutes. It can be noticed is that at lower temperatures and time duration there were smaller reductions, which was about 14.53 % at a temperature of 180 ºC for 120 minutes compared to unmodified samples.

When it comes to this research, it is important to emphasize that hemicellulose is less thermally stable than cellulose. The data on the exact degrees of temperature for the beginning of the degradation process are different. Various thermal analyzes of wood show that there is a broad endotherm in the range of 90 to 150 ºC which is associated with the evaporation of retained water in the cell wall. The analysis shows that in harder wood species there is a greater loss of bending Fmax than in soft wood species, when monitoring the process of thermal modification under the same conditions, as can be seen in Figure 7 and this is in conformity with the findings of several researchers as mentioned in [13], Hardwood is less thermally stable than softwood, which can be attributed to differences in the content
and composition of hemicellulose. Pentosans, which are found in larger quantities in hardwood hemicellulose, are more susceptible to thermal destruction than hexosans. In addition, hardwood generally has a higher proportion of hemicellulose which has a higher content of acetyl groups compared to softer species.

![Figure 7: Percentage reduction of maximum bending strength](image)

The results have shown a growing trend of decreasing $F_{\text{max}}$ as the temperature of thermal modification increased. As previously described, the largest losses of maximum force were recorded in fir wood at a temperature of 210 °C for 240 minutes, in linden wood at 220 °C for 180 minutes and beech wood at 210 °C for 240 minutes. The increase in $F_{\text{max}}$ occurred in fir wood at a temperature of 180 °C for 240 minutes.

3.3. Tensile strength

The mean values of tensile $F_{\text{max}}$ before and after thermal modification of wood are shown in Figure 8. It is visible that there was a decrease in mechanical tensile properties at elevated temperatures and duration in all cycles. In linden wood, there was the smallest reduction in $F_{\text{max}}$ of 3.4 %, where the temperature was 195 °C for 180 minutes.

The temperature of 180 °C/120 minutes has shown the smallest decrease of tensile $F_{\text{max}}$ in fir wood, which was about 23.67 %. With increasing temperature and process duration compared to unmodified wood, a relatively lower $F_{\text{max}}$ was obtained, as seen in Figure 9. The largest decrease in $F_{\text{max}}$ of modified samples compared to unmodified fir wood samples (69.31 %) was recorded at a maximum temperature of 210 °C for 120 minutes. In addition, there was a significant reduction of tensile strength $F_{\text{max}}$ by about 57.18 % at a temperature of 180 °C and duration of 240 minutes. Thus, it can be said that with increasing temperature and duration of the process, there is a decrease of tensile strength $F_{\text{max}}$ of fir wood, which is best seen in Figure 9.

Diagram 8 shows the results of $F_{\text{max}}$ of thermally treated linden wood. It is important to emphasize that the thermal modification carried out at five different levels clearly affects the tensile strength of linden specimens, which is significantly reduced by about 59.56 % at 220 oC for 180 minutes, 54.87 % at 195 °C for 276 minutes, while in the smallest decrease of 3.42 % was recorded at a temperature of 195 °C for 180 minutes.

Heat treatment of beech wood at 180 °C for 120 minutes resulted in $F_{\text{max}}$ reduction of 53.39 % (Figure 9). Increasing the effective processing temperature to 210 °C for 120 minutes caused a decrease in tensile $F_{\text{max}}$ of 48.92 %. In addition, by increasing the process duration while maintaining the temperature at 180 °C for 240 minutes the reduction was as much as 62.16 %, and at 210 °C for
240 minutes it resulted in an even greater reduction of the maximum tensile force by 77.60%, compared to unmodified samples.

![Tensile strength](image.png)

**Figure 8.** Maximum tensile strength of thermally modified and unmodified wood

The results have shown, as with the bending samples, that the lowest tensile $F_{\text{max}}$ losses were in the fir wood samples that were thermally modified at 180 °C for 120 minutes where the tensile $F_{\text{max}}$ was reduced by about 23%, and in the linden wood samples that were modified at 195 °C for 180 minutes. The largest reductions in tensile $F_{\text{max}}$ were recorded at temperatures of 210 °C for 240 minutes in fir and beech wood, and at a temperature of 220 °C for 180 minutes in linden wood, where the reduction was 59.56%.

![Tensile strength loss](image.png)

**Figure 9.** Percentage of reduction of maximum tensile strength

Figure 9 shows the percentage decrease in tensile $F_{\text{max}}$ of the modified samples for different temperatures, durations, and densities. Based on the analysis of the results, it is clear that there is a decrease in $F_{\text{max}}$ when processing wood at elevated temperatures. In addition, the loss of tensile strength depends a lot on the maximum duration of the thermal modification process.

4. Conclusion

Prior to testing the thermally modified wood, it is recommended to perform an analysis of the most influential process parameters (temperature, pressure, process duration, geometry, wood type, etc.).
Every situation is different and the values of the influential parameters are constantly changing. Therefore, making general statements about the mechanical strength of thermally modified wood is not a wise approach. Although, perhaps with a few minor exceptions, the basic knowledge needed to make solid, durable elements still exists.

The possibility of improving the mechanical properties of thermally modified wood is advancing each day. Technology is constantly changing, and a byproduct of seemingly unrelated fields can have a significant effect on solving apparently impossible problems. The development of wood modification and other wood processing processes could influence production methods which, in turn, would result in better dependence on mechanical properties and extension of the life of thermally modified wood.

Based on the analysis of the bending and tensile strength of all materials tested in the experiment, it can be concluded that the duration of the thermal modification process at maximum temperature, wood type and maximum achieved heat treatment temperature are very important parameters that affect the retention of mechanical properties in thermal modification of wood.

Experimental research of the bending and tensile strength determined a higher load, i.e. a higher value of the fracture force at lower temperatures and a shorter duration of the processing. That is, by increasing the temperature and duration of thermal modification, there is a significant reduction in the fracture force of the treated samples, especially in deciduous species. Therefore, heat-treated wood can be used with appropriate heat treatment time and temperature without any loss in strength values in areas where wood elements such as parquet, wooden facades, decorative purposes and where dimensional stability is important.

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