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Changes in chemical and optical properties of silver fir (*Abies alba* L.) wood due to thermal treatment

Viera Kučerová[1], Rastislav Lagaňa[2*] and Tatiana Hýrošová[3]

Abstract

Alterations in the chemical composition of silver fir wood (*Abies alba* L.) during thermal treatment were investigated. The mass loss, extractives, saccharides, and lignin were determined. In both untreated and heat-treated wood, extractives were determined according to ASTM D1107-96, lignin content according to ASTM D1106-96, and saccharides by high-performance liquid chromatography. During the thermal treatment, mass loss and the chemical composition of the wood were found to change. The extractives content decreased up to 200 °C, and then increased at higher temperatures. The mass loss increased with increasing temperature and time of thermal treatment. A relative increase in lignin content and a decrease in saccharides were observed. The color of the wood was stable at temperatures up to 150 °C when treated up to 1 h. A change in color significantly depends on thermal energy intake above that level. Saccharides, color coordinates L^* and b^*, lignin, mass loss, temperature, and color change ΔE^* strongly vary together. These investigated properties of fir wood set out the boundaries for high-temperature treatment and show potential for utilization of this species.

Keywords: Thermal treatment, Mass loss, Lignin, Saccharides, Extractives, Silver fir wood, Color

Introduction

Wood is a complex polymeric material constituted mainly of cellulose, hemicelluloses, and lignin, with a minor proportion of extractives. Heat treatment is now a well-known and commercial wood modification process that improves wood properties, changing low value species into higher value materials. The most commonly known processes are rectified wood in France, thermowood in Finland, Plato wood in Holland, and oil heat treatment in Germany. All these treatments decrease the equilibrium moisture content and increase the dimensional stability of the wood [1–4].

The thermal modification of wood is defined as the application of heat to wood in order to bring about a desired improvement in the performance of the material. The thermal modification is invariably performed between the temperatures of 180 °C and 260 °C, with temperatures lower than 140 °C resulting in only slight changes in the material properties and higher temperatures resulting in unacceptable degradation to the substrate [5]. All obtained changes are achieved through the heat treatment process, without any added chemicals. Thus, heat-treated wood has been considered to be an ecological alternative to impregnated wood material [6]. A review on the heat treatment of wood, with descriptions of the major publications in this field, has been published by Esteves and Pereira [7]. Heat treatment causes a chemical change in the wood, and results from the chemical composition of treated wood have been reported by Tumen et al. [8], Zawadzki et al. [9], de Moura [10], Brito et al. [11], Kačíková et al. [12], Barański et al. [13], Kučerová et al. [14], and Tolvaj et al. [15]. The chemical modification starts with the degradation of hemicelluloses by deacetylation, followed by depolymerization, catalyzed by the released acetic acid, giving origin to some low mass extractable compounds [16–18]. At the same time, carbohydrate dehydration decreases the

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*Correspondence: lagana@tuzvo.sk
[2] Department of Wood Science, Technical University in Zvolen, T. G. Masaryka 24, 960 53 Zvolen, Slovak Republic
Full list of author information is available at the end of the article

overall content of hydroxyl groups [19] and leads to the formation of aldehydes, such as furfural and hydroxymethylfurfural from pentoses and hexoses, respectively [20]. Although cellulose is more resistant than hemicelluloses, degradation of the amorphous cellulose does occur, causing an increase in cellulose crystallinity [21]. Lignin is affected by the cleavage of β-*O*-4 linkages and, in softwood lignin, there is also a reduction in methoxyl content, leading to a more condensed structure [20, 22, 23].

In relation to extractives, the most volatile compounds leave the wood, while the others are degraded. Almost all of the original extractives disappear, and new compounds are formed, including monosaccharides and their dehydration products, as well as syringaldehyde, syringic acid, and sinapaldehyde as the most prominent compounds.

Wood color is an oft-used property for the identification of thermal treatment effects. Several studies related to wood color changes under high temperatures have been performed. With increased temperature and length of treatment, wood becomes darker [1, 14, 24–29]. Color changes in thermally-treated wood are linked to the degradation of hemicelluloses and cellulose depolymerization [12], color changes in sap and extractives [30], loss of bending strength [1], and wood species [31]. The color changes are visible in the relatively small mass loss of wood [24].

Decision making for recycling thermally modified wood after its lifespan is based on the chemical composition of treated wood products. The products could be reuse in bioethanol production if a content of carbohydrates is sufficient or in pulp and paper industry that aims for cellulose. Both compounds degrade and a level of degradation depends on an intensity of the treatment. Secondary utilization of thermally modified wood as wood fuel to substitute firewood is another environment friendly option [32].

In this study, we determined the effects of thermal treatments on the mass loss, extractives, saccharides, lignin and color, and focused on possibility to predict chemical changes of fir using relatively easy measured surface color. Indirect and fast determination of chemical components of processed wood could help in decision making for further recycle processing in bioethanol or energy industry.

Materials and methods

Materials

Silver fir (*Abies alba* L.) wood was obtained from the Zvolen region of the Slovak Republic. The fir tree was 59 years old, and the diameter at breast height was 262 mm. From the butt end of the trunk, a radial board was cut and used for the preparation of 10 mm × 10 mm × 150 mm (radial × tangential × longitudinal) samples. Small dimension of samples leads to small temperature differences in the volume of samples during a thermal treatment. The samples were conditioned in a chamber at 20 ± 2 °C and 65 ± 5% relative humidity to a moisture content of about 12%. A total of seven groups of 10 samples each were heat-treated under defined conditions, and a reference group of 10 samples was left without treatment (20 °C).

Methods

Thermal treatment

Heat treatment of the samples was carried out in a preheated Binder ED 53 (Tuttlingen, Germany) laboratory heating oven at 100, 150, 200, 220, 240, 260, and 280 °C for 1, 3, and 5 h under atmospheric pressure in the presence of air. The wide temperature range and time of treatment were chosen in order to cover common temperature treatments before starting ignition of fir wood. The length of time of reheating the oven to the required temperature after inserting the samples was 5 min. After the treatment, each group of samples was removed from the oven and cooled down in a desiccator, and then the mass loss was determined.

Chemical composition

The samples were mechanically disintegrated to sawdust. Fractions of 0.5 to 1.0 mm were extracted in a Soxhlet apparatus, with a mixture of ethanol and toluene, according to American Standard Test Method (ASTM) D1107-96 [33]. The lignin content was determined according to ASTM D1106-96 [34].

Determination of the saccharides content was carried out according to the National Renewable Energy Laboratory [35]. The analysis was performed with an Agilent 1200 high-performance liquid chromatograph (HPLC; Agilent Technologies, CA, USA) with an Aminex HPX-87P column (BioRad, USA) at a temperature of 80 °C, and a mobile phase flow rate of 0.6 ml/min. Hydrolysis was performed on two samples of each treated group for each set of conditions, and HPLC analysis was performed twice per sample.

All measurements were carried out four times. The data are presented as percentages of the oven-dry weight of unextracted wood.

Color changes

The color of the treated and untreated samples was determined after conditioning at a temperature of 20 °C and relative humidity of 65%. Prior to the measurements, a BYK Spectro-Guide Sphere spectrophotometer (Geretsried, Germany) was calibrated using a white color standard. The color of the wood was expressed in the CieLab coordinate system that expresses three color space coordinates—L^* (lightness), a^* (green to red component), and b^* (blue to yellow component). An average of eight readings for each sample was used to define the sample color. A change in color ΔE^* was defined as the space distance between the average color coordinates of 10 untreated samples and the color coordinates of thermally-treated ones [36].

Statistical analyses

For all parameters, multiple comparisons were first subjected to an analysis of variance (ANOVA), and significant differences between average values of control and treated samples were determined using Duncan's multiple range test at a p-value of 0.05.

Multivariate associations among 10 wood traits were analyzed using principal component analysis (PCA; Statistica 12.0) to describe the patterns of covariation among the examined traits.

Results and discussion

Changes in chemical properties

In natural silver fir wood, the average results of the wood components were: 1.86% (standard deviation SD = 0.03) extractives; 31.08% (SD = 0.34) lignin; and 64.42% (SD = 0.04) saccharides. The content of holocellulose (saccharides), lignin, and extractives in the wood depends on various factors, such as age, growth conditions, the season of timber harvesting, etc. According to previous authors, the proportion of wood components in silver fir wood are: extractives 0.88%; cellulose 40.69%; lignin 29.51%; holocellulose 75.88% [37]; extractives 1.84%; holocellulose 75.35%; cellulose 49.21%; and lignin 27.89% [8]. Chen et al. [38] reported the following values in Chinese fir wood: holocellulose 67.3%; cellulose 46.7%; hemicelluloses 20.6%; and lignin 33.6%.

The thermal treatment of the wood was carried out at 100, 150, 200, 220, 240, 260, and 280 °C; 280 °C was the highest temperature that could be used for thermal treatment. The most significant factor that influenced the thermally-treated wood was the changes in its chemical composition. After thermal treatment of the silver fir wood, the mass loss and chemical composition of the wood were distinctly changed. The percentage content of mass loss increased with increasing temperature and length of thermal treatment (Fig. 1). Mass loss of major wood compounds took place predominantly above 200 °C at all three lengths of time, the highest being recorded at 280 °C and 5 h (73.29%). The change in mass loss is one of the most important features in the thermal treatment of wood, and is commonly referred to as an indicator of quality [7].

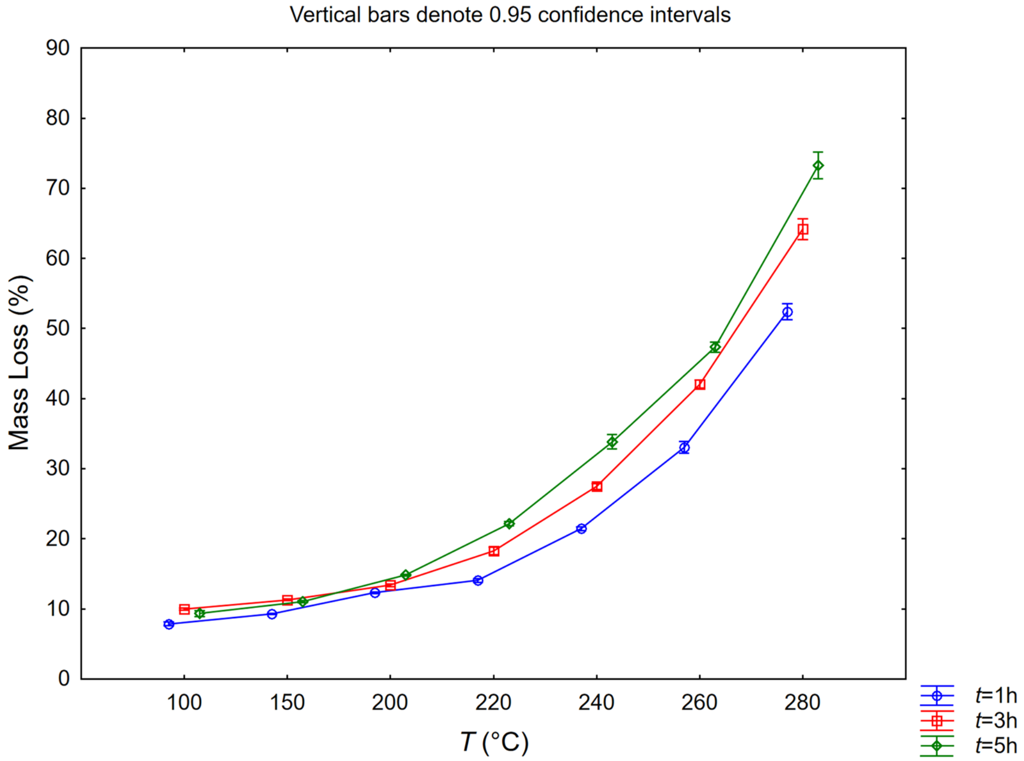

Fig. 1 Influence of treatment temperature and time on the mass loss content in fir wood

As showed in Fig. 2, the extractives content decreased when the temperature changed from 20 to 150 °C. This change was probably caused by both the volatilization of different extractives from the wood due to the thermal treatment, while others were probably degraded. At higher temperatures, the total content of extractives increased, reaching a maximal value of 3.11% at 240 °C and 1 h; after that, the extractives content decreased. Similar results have been reported for thermally-treated wood by Esteves et al. [3, 39]. The extractives content can vary as a function of wood species [11, 40] and the utilized treatment process. According to Boonstra et al. [41] and Windeisen et al. [42], the thermal modification of wood causes additional transformations in lignin and extractives.

The saccharide content of the treated samples changed slightly when the treatment temperature was below 200 °C, compared to that of untreated wood. Maximum values were recorded when samples were thermally-treated at 200 °C for 1 h. Above 200 °C, the amount of saccharide decreased as the treatment temperature increased. This indicates that saccharides rapidly degrade with increasing temperature and time (Fig. 3). A temperature range of 150 °C to 200 °C led to the initial degradation of hemicelluloses.

The total saccharide content of wood comprises cellulose and hemicellulose portions. The cellulose portion is the most resistant to thermal degradation of holocellulose. The least stable components of the wood at high temperatures are the hemicelluloses. These are much more susceptible to thermal degradation than the other polymeric wood constituents due to their branched, amorphous structure with their different and substituted monomeric units [42].

According to de Moura et al. [10], all monosaccharides, with the exception of glucose, are significantly reduced in wood samples after thermal treatment at a maximum temperature of 200 °C due to their resulting degradation and volatilization. After thermal treatment, the carbohydrate portion of the wood is almost exclusively composed of glucose and xylose.

In a study by Zhang et al. [2], the content of the chemical components was shown to also change after thermal treatment. Holocellulose and α-cellulose decreased significantly with increasing temperature and duration when samples were heat-treated above 160 °C, and this also strongly correlated with mass loss. This mass loss was mainly due to the degradation of hemicelluloses and cellulose.

The change in lignin content has been recognized as being strongly dependent on temperature and length of treatment, and on the characteristics of the wood species [5]. The lignin content (Fig. 4) of untreated wood

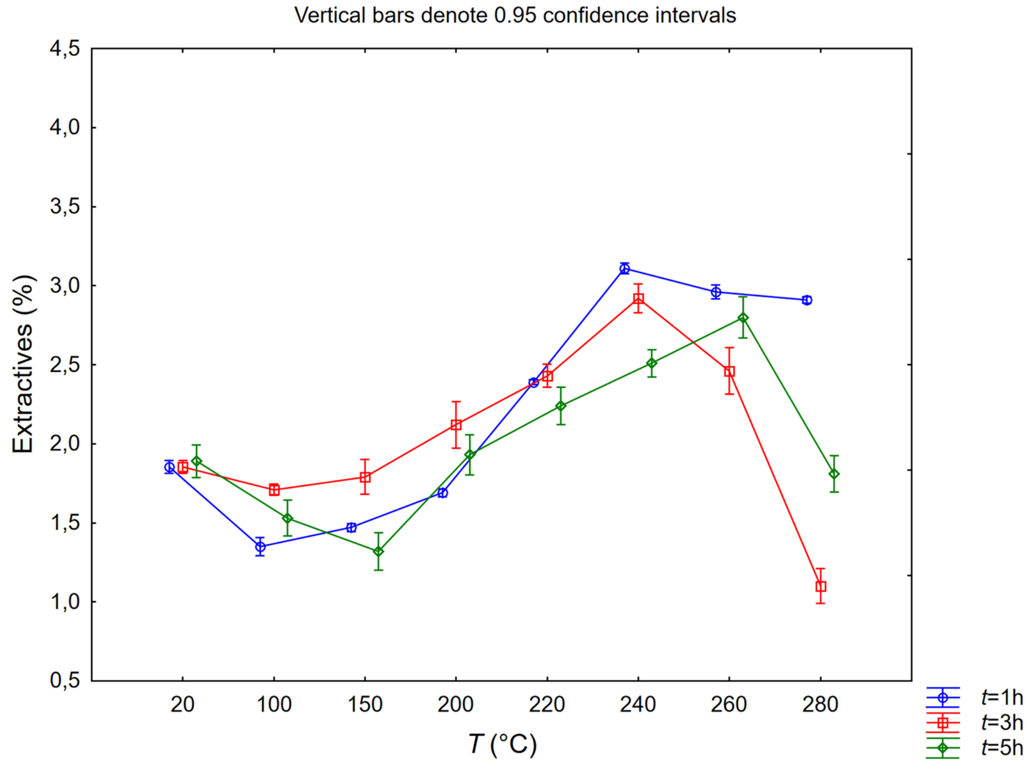

Fig. 2 Influence of treatment temperature and time on the extractives content in fir wood

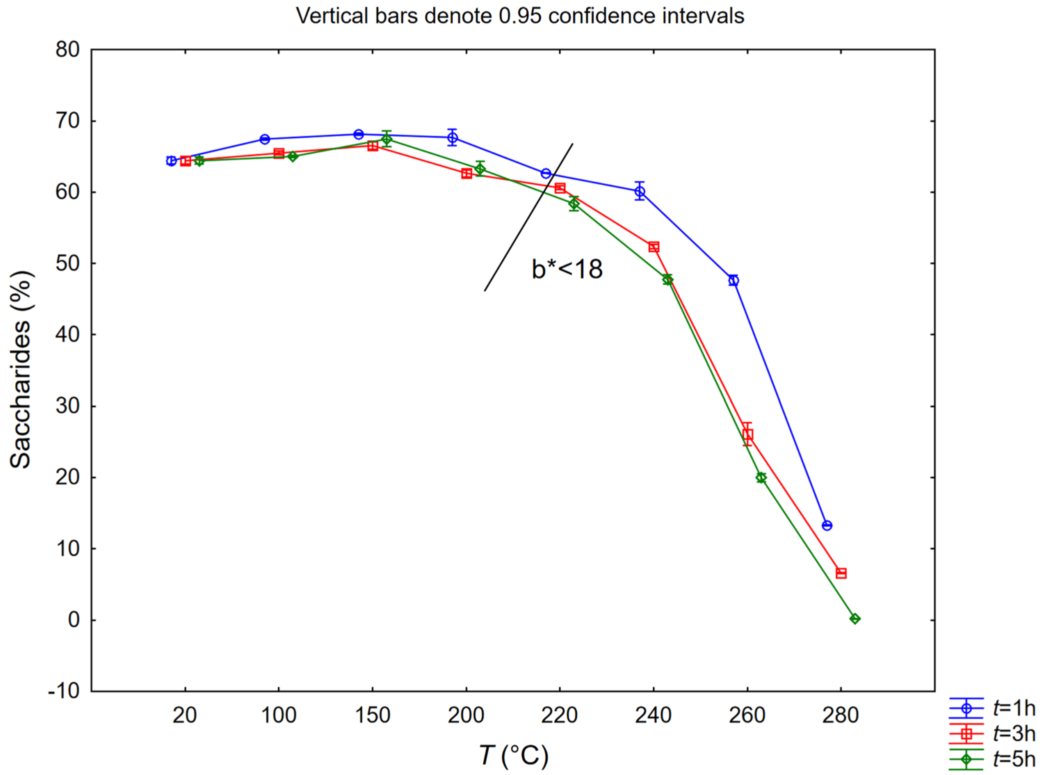

Fig. 3 Influence of treatment temperature and time on the saccharides content in fir wood

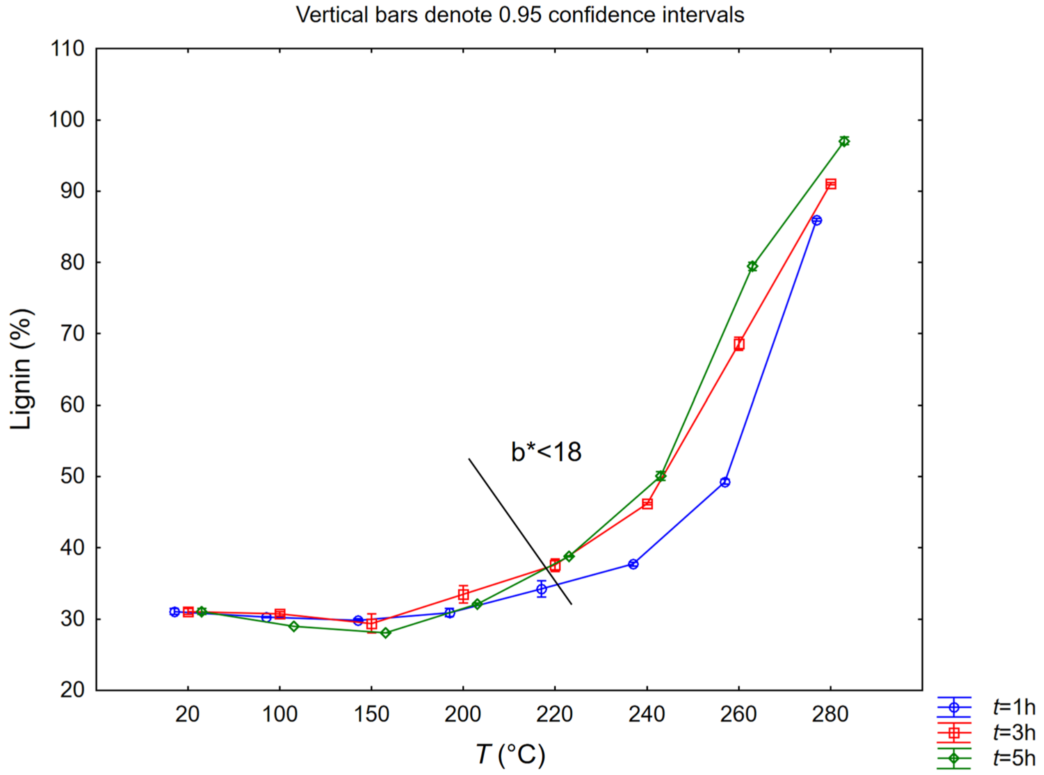

Fig. 4 Influence of treatment temperature and time on the lignin content in fir wood

was determined to be 31.08%. At all treatment temperatures and times, the acid insoluble lignin content slightly decreased between 20 and 150 °C; that can be attributed to degradation reactions. At 150 °C, the lignin content started to increase. Its content was highest (97.03%) at 280 °C and 5 h. It is generally accepted that the increase in lignin content in thermally-treated wood is due to the loss of polysaccharide material occurring during heating; however, as suggested by Esteves and Pereira [7], such an increase cannot be considered to be constituted of pure lignin since polycondensation reactions occur in the cell walls of other wood components, with consequent polymerization, thereby increasing the apparent lignin content.

Color changes

The results of the color change analysis are given in Figs. 5 and 6. These confirm the strong influence of temperature and treatment time on color changes [1]. Color change ΔE^* was significantly depended upon both temperature ($F_{6,170} = 22{,}583$, $p < 0.0001$, where F is value of Fisher–Snedecor distribution, the first index 6 is a degree of freedom and the second index 170 is a number of observations) and length of treatment ($F_{2,170} = 597$, $p < 0.0001$). Lightness changes (ΔL^*) mainly contributed to the color change, and thus the factor effects were similar (temperature $F_{6,170} = 18{,}210$, $p < 0.0001$, length of treatment $F_{2,170} = 460$, $p < 0.0001$). The color coordinates a^* and b^* alternated, initially both increasing up to 200 °C, but above 200 °C, the a^* coordinate decreased to initial values and the b^* coordinate decreased even further, approaching zero. A similar alternation of the a^* and b^* coordinates has been observed in other coniferous wood species, such as spruce [43], pine [31, 44], and cypress [25]. Again both factors—temperature and length of treatment—were significant. For a^*, a temperature factor showed $F_{6,170} = 1159$, $p < 0.0001$, and length of treatment $F_{2,170} = 24$, $p < 0.0001$. For b^*, it was $F_{6,170} = 2611$, $p < 0.0001$ for temperature and $F_{2,170} = 187$, $p < 0.0001$ for length of treatment, respectively.

Fig. 5 Influence of treatment temperature on the color of fir wood

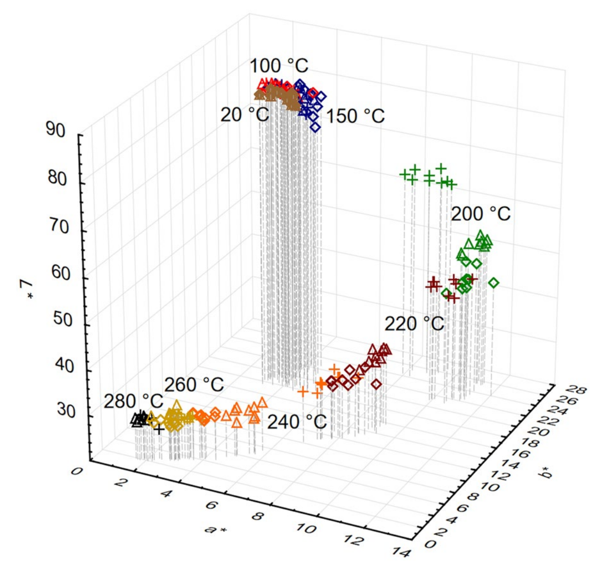

Fig. 6 Color coordinates of high-temperature treated and untreated fir wood. Each color represents a specific temperature of treatment. Signs +, Δ and ◇ stand for 1-h, 3-h and 5-h treatment, respectively

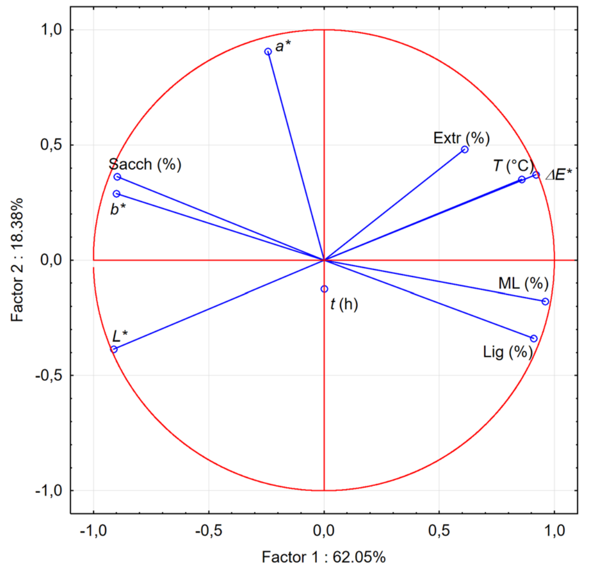

Fig. 7 Projection of variables on the factor-plane. *T*, temperature; *t*, time; *Extr*, extractives; ML, mass loss; Lig, lignin; Sacch, total saccharides, color coordinates *L**, *b**, *a**; Δ*E**, color change

The trend of the color changes in color space followed a specific path (Fig. 6). A short, high-temperature treatment produced the same color as a long, low-temperature treatment. For example, one can compare 1 h of treatment at 220 °C with 5 h of treatment at 200 °C, or similarly, 240 °C with 220 °C. The color is identical when those two cases are compared using Duncan's test (for ΔE^*, $p = 0.0610$ or $p = 0.3309$, respectively). Despite a significant temperature factor, the Duncan's test probabilities of equal groups at a significance level of $\alpha = 0.05$ reveal boundaries of color unaffected at lower-temperature treatments. Those were 1 h at 100 °C for changes in the b^* coordinate ($p = 0.1624$), 1 h at 150 °C for ΔE^* and ΔL^* ($p = 0.1495$ and $p = 0.1408$, respectively), and 3 h at 150 °C for the a^* coordinate ($p = 0.5337$). This observation suggests that thermal energy intake is a significant contributor to color change that could be modeled by a time–temperature superposition [26].

Principal component analysis

To analyze the interrelationships among the variables, we used PCA analysis. Its aim is to reduce the number of interrelated variables and explain them in terms of a smaller number of variables—principal components—with a minimum loss of information. A visualization of the results is illustrated in the factor score plot (Fig. 7).

The first component (factor 1), explaining 62.05% of the total variance in the observed variables, is strongly and negatively correlated with saccharide content, L^*, and b^*, and strongly and positively correlated with lignin, mass loss, temperature, and ΔE^*. Additionally, the PCA detected a very strong correlation between lignin and mass loss, and temperature and ΔE^*, respectively. Based on the magnitude of the factor coordinates for the variables in the analysis, the first factor can be labeled as changes in the chemical components of the wood.

The second component (factor 2) explained 18.38% of the total variance in the observed variables, and showed a positive correlation with a^*. The second factor can be labeled as the time axis.

The cases score plot (Fig. 8) shows the factor coordinates for all observations. One result that is apparent in this plot pertains to the clustering of the 20, 100, and 150 °C temperatures (in 1, 3, and 5 h), where the changes in chemical structure are insignificant.

The high temperatures plotted on the positive (right) side of this dimension indicate the significant effect of these temperatures on lignin, mass loss, and the values of ΔE^* (positively); saccharides content and the values of b^* and L^* (negatively).

The color coordinate b^* could be used as a predictive value of saccharides and lignin content in thermally-treated silver fir wood. Data of thermally-treated spruce wood [43] showed that once b^* value decreased bellow the value of untreated wood, saccharides content started decreased. We observed an exponential drop of saccharides starting when b^* value get below the value of untreated wood ($b^* < 18$) which occurred after a 1-h treatment at 220 °C (compare Figs. 3 and 6). High saccharide content gives feasibility for ethanol production

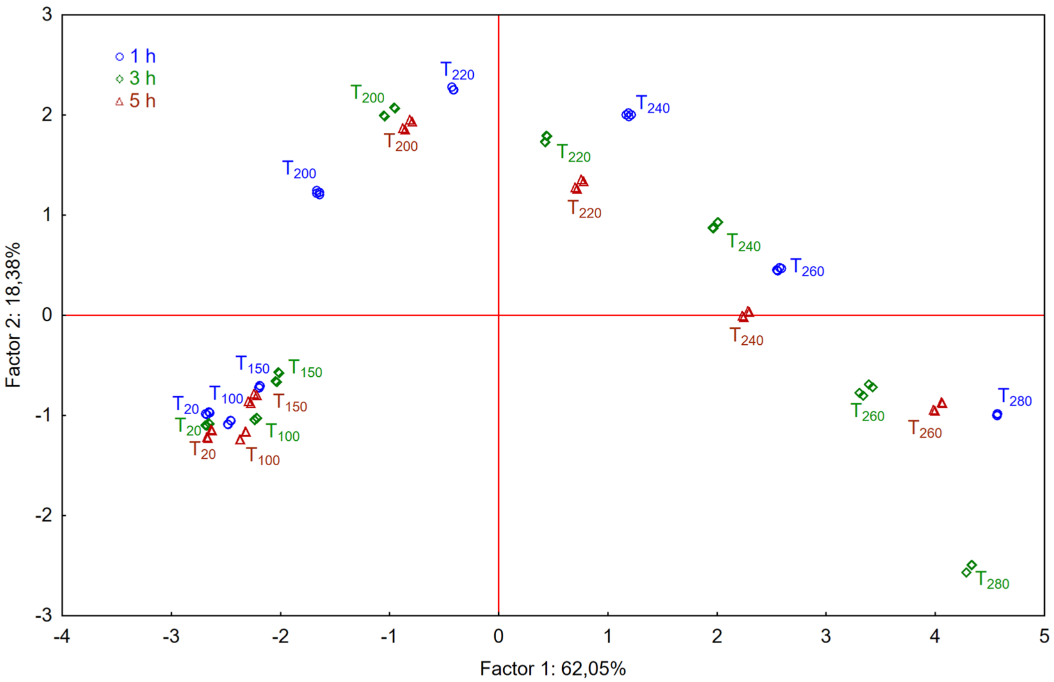

Fig. 8 Projection of cases on the factor-plane. T_{20}, 20 °C; T_{100}, 100 °C; etc.

and limiting b^* value can be used for efficient reuse of thermally-treated fir wood.

The relative lignin content exponentially increased with a level of treatment. Since lignin is known for a higher heating capacity compared to the cellulose and hemicelluloses, combustion of high lignin content wood will be more suitable. Torrefaction of oak and pine wood above 220 °C increased energy content [45]. They find significant correlation of heating value and mass loss which points to our correlation of lignin and mass loss. Similar increase in caloric value above 240 °C was reported for larch wood [46]. Likewise in saccharides, the same color value b^* could be used as a boundary for relative growth of lignin predicting heat capacity (Fig. 4).

Thus we can conclude that b^* value could be used for decision making of secondary utilization of thermally-treated fir wood if we could assume that color of inner part of wood does not change in time of use. It should be noted that the statement cannot be generalized to other thermally-treated wood species, such as oak, due to different color change processes [43, 47].

Conclusions

The effects of temperature and time led to changes in the proportion of chemical components and mass loss in thermally-treated silver fir (*Abies alba* L.) wood. The percentage mass loss increased with increasing temperature and length of thermal treatment. The extractives content decreased when temperature increased from 20 to 150 °C, and then increased. It is thus assumed that a proportion of the substances extracted from the samples treated above 200 °C were not constituted by the original extractives of the untreated wood. The saccharides content decreased, and the relative lignin content increased, with increasing temperature and length of thermal treatment. After thermal treatment above 200 °C, hemicelluloses were completely or partially eliminated, and this was recorded in a reduction in the saccharides content.

Color changes after thermal treatment of fir wood were confirmed to be highly significant. It followed the same path in the color space coordinate system, with a short, high-temperature treatment showing the same color as a long, low-temperature treatment. Thermal energy intake during treatment is a main factor in changing the color, and could be used in the modeling of color changes. The color of fir wood is relatively stable at 1 h of treatment at 150 °C.

PCA analysis showed that saccharides, L^* and b^*, lignin, mass loss, temperature, and ΔE^* strongly covary. If one of these properties changes, the others will follow. The color coordinate b^* is suitable for prediction of saccharides, lignin, and mass loss and could be potentially used for decision making in the process of secondary utilization of thermally modified silver fir wood. Change of extractives is potentially described by color change ΔE^*.

Changes in the properties of thermally-treated fir wood point to a treatment limit of 1 h at 200 °C. A longer duration of the thermal treatment, or higher temperatures, leads to undesirable changes in its properties.

Abbreviations

a^*: color coordinate of CIELAB color space representing a green to red component; b^*: color coordinate of CIELAB color space representing a blue to yellow component; $F_{\mathrm{dof},N}$: value of Fisher–Snedecor distribution, dof is a degree of freedom and N is a sample size; ΔE^*: change in color in CIELAB color space defined as the space distance between the average color coordinates of 10 untreated samples and the color coordinates of thermally-treated ones; L^*: color coordinate of CIELAB color space representing the lightness; p: probability value of receiving/rejecting a null hypothesis; SD: standard deviation of a sample; t: time of thermal treatment in hours; T: treatment temperature in °C; α: significance level ($\alpha = 0.05$).

Authors' contributions

VK conceived of and designed the experimental design and setup. VK carried out and interpreted the chemical analysis. RL carried out and interpreted the color analysis. TH carried out and interpreted the PCA analysis. VK and RL wrote the initial version of the paper, and edited it through to the final version. All authors read and approved the final manuscript.

Author details

[1] Department of Chemistry and Chemical Technologies, Technical University in Zvolen, T. G. Masaryka 24, 960 53 Zvolen, Slovak Republic. [2] Department of Wood Science, Technical University in Zvolen, T. G. Masaryka 24, 960 53 Zvolen, Slovak Republic. [3] Department of Mathematics and Descriptive Geometry, Technical University in Zvolen, T. G. Masaryka 24, 960 53 Zvolen, Slovak Republic.

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Competing interests

The authors declare that they have no competing interests.

Availability of data and materials

The datasets used and/or analyzed during the current study are available from the corresponding author on reasonable request.

Consent for publication

Not applicable for this study.

Ethics approval and consent to participate

This article does not contain any studies with human participants or animals performed by any of the authors.

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