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PERMANENT CHANGES TO STRUCTURE AND PROPERTIES OF BEECH AND ASH WOOD AFTER ITS HYDROTHERMAL PLASTICIZATION PART I. CHANGES IN SELECTED PROPERTIES

Jozef Kúdela

Faculty of Wood Sciences and Technology
Technical University in Zvolen

SYNOPSIS. The paper was aimed at study of permanent changes in beech and ash wood after its hydrothermal plasticization over a temperature range from 100 to 160°C. Changes in selected physical and mechanical properties were observed.

The results showed that conspicuous changes in properties occurred after plasticization at temperatures exceeding 140°C. It could also be observed that relations have been found between the changes in physical and mechanical properties of wood chemical structure and properties.

KEY WORDS: hydrothermal plasticization, beech wood, ash wood, shrinkage, density, strength, colour

INTRODUCTION

A range of technological procedures in wood processing (bending, pressing, peeling, preparing veneers, etc.) requires to modify physical and mechanical properties of wood. The required temporary changes in wood properties can be obtained with suitably chosen type of plasticization of the material. The oldest, but also presently most frequently used plasticization process is hydrothermal plasticization – where the main wood plasticizers are water and heat in their mutual interaction. In process of hydrothermal plasticization (in the following only plasticization), the interaction among wood, water and heat can induce to wood structure not only transient but also permanent changes, entailing also permanent changes in wood physical and mechanical properties (Kúdela 2005).

The extent of these changes depends on the plasticizer parameters and on plasticization time. In addition, Zemiar and Gáborík (1996) and Makovíny (2006) also pointed out an important influence of the plasticization type (steaming, boiling, microwave heating, etc.). The last, but not least is timber species and

its properties. Dealing with the required temporary changes, we must also take into account the possibility of unfavourable permanent changes to wood structure and also properties. These changes may not be negligible, especially in case of temperatures exceeding 100°C (Melcer et al. 1989, Čunderlík et al. 1995, Kúdela and Laurová 2006, Pervan et al. 2006, Sehlstendt-Persson et al. 2006).

The objective of this paper is to summarize our study of permanent changes of selected properties of beech and ash wood, plasticized in water bath at temperature ranging 100-160°C. The two wood species were chosen as the ones that are most frequently used as suitable to be shaped, primarily by bending.

METHODS

Changes in ash (*Fraxinus exelsior* L.) and beech (*Fagus sylvatica* L.) wood properties induced by plasticization were studied on test specimens 20 × 20 × 30 mm (R × T × L) in size. The beech specimens were cut from ten logs, the ash ones from three logs. The specimens were firstly seasoned to 12% moisture content, and then to 6% moisture content. Eventually, they were oven-dried at 103 ±2°C to the zero moisture content. The dried specimens were weighed with a precision of 0.001 g and measured in all the three anatomical directions with a precision of 0.01 mm. At this stage, their oven-dry density (ρ_0) was determined. Subsequently, the specimens were immersed in water again and fully saturated. Then, they were placed in stainless-steel sterilisation containers, ten specimens in each (Fig. 1a): The containers were filled with distilled water in amounts resulting in wood/water weight proportions of 1:4. In case of beech wood, in each set each cut was represented by one specimen. In case of ash wood, in each set each cut was represented by three specimens. The first four series of autoclaves were immersed in an oil bath at temperature of 100°C (Fig. 1b). Two series were heated at this temperature for one hour, the other two, for two hours. This process of plasticization was replicated at temperatures of 120, 140 and 160°C.

After plasticization and oven-drying the specimens to the zero moisture content, the following parameters were measured: weight loss, maximum shrinkage in the individual anatomic directions, volumetric shrinkage and density in the oven-dry state (ρ_0) and colour change. From mechanical properties, compression strength parallel to grain was determined. The strength was quantified at moisture content MC > FSP and at zero moisture content.

Shrinkage and density were determined according to the Standards STN 49 0128 and 49 0108, strength limit in compression parallel to grain according to the Standard STN 49 0110.

The change in wood colour was measured with a spectrometer MINOLTA CM 2600d. A standard measuring screen with a hole 6 mm in diameter was used. The irradiation system was adjusted to the measurement regimen scanning also dispersed components (SCI). Measurements covered the range of wavelength 360-

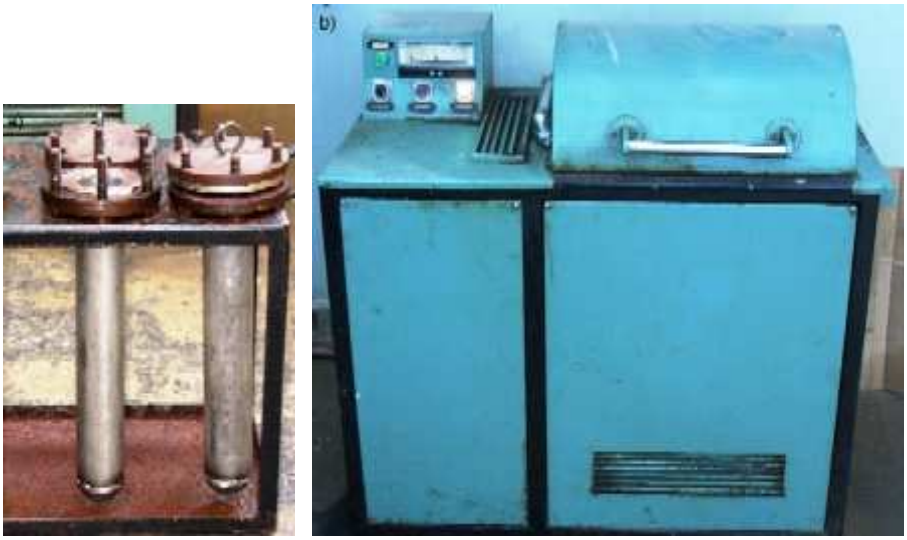

Fig. 1. Stainless-steel sterilising containers (a) and equipment in that plasticization took place (b)

-740 nm, with a precision of 10 nm. The colour coordinates were presented in the CIELAB system (Kubovský et al. 2008).

For each plasticization regimen, hydrolysate was sampled for chemical analysis tackled in the paper by Laurová and Kačík (2009).

RESULTS AND DISCUSSION

The experimental results show that in case of temperatures exceeding 100°C, permanent changes in wood properties should be assumed. It has been found that plasticization of both beech and ash wood at temperatures exceeding 100°C is connected with negligible weight losses only, which have never exceeded a limit of 2% (Fig. 2). As we can see in Figure 2, at temperatures exceeding 100°C, the weight loss in the plasticized wood is more pronounced. The weight loss after one-hour plasticization running at 160°C was 6-8%. The loss after two hours at the same temperature was 13-16%. The weight loss in case of beech and ash wood was very similar – both in terms of quality and quantity. A significant difference was only observed after two hours of plasticization running at 160°C – when the weight loss in ash wood was higher by 3% compared to the beech wood.

This weight loss is caused on one hand by the temperature, and on the other hand by the used plasticizer turning to acid in the process of plasticization. The nascent acids, identified in the condensate (Laurová and Kačík 2009), promote the degradation impact of the plasticizer on wood. This fact was reflected in higher wood mass losses. Similar changes in wood weight connected with plasticization process were also reported by Fengel and Wegener (1989).

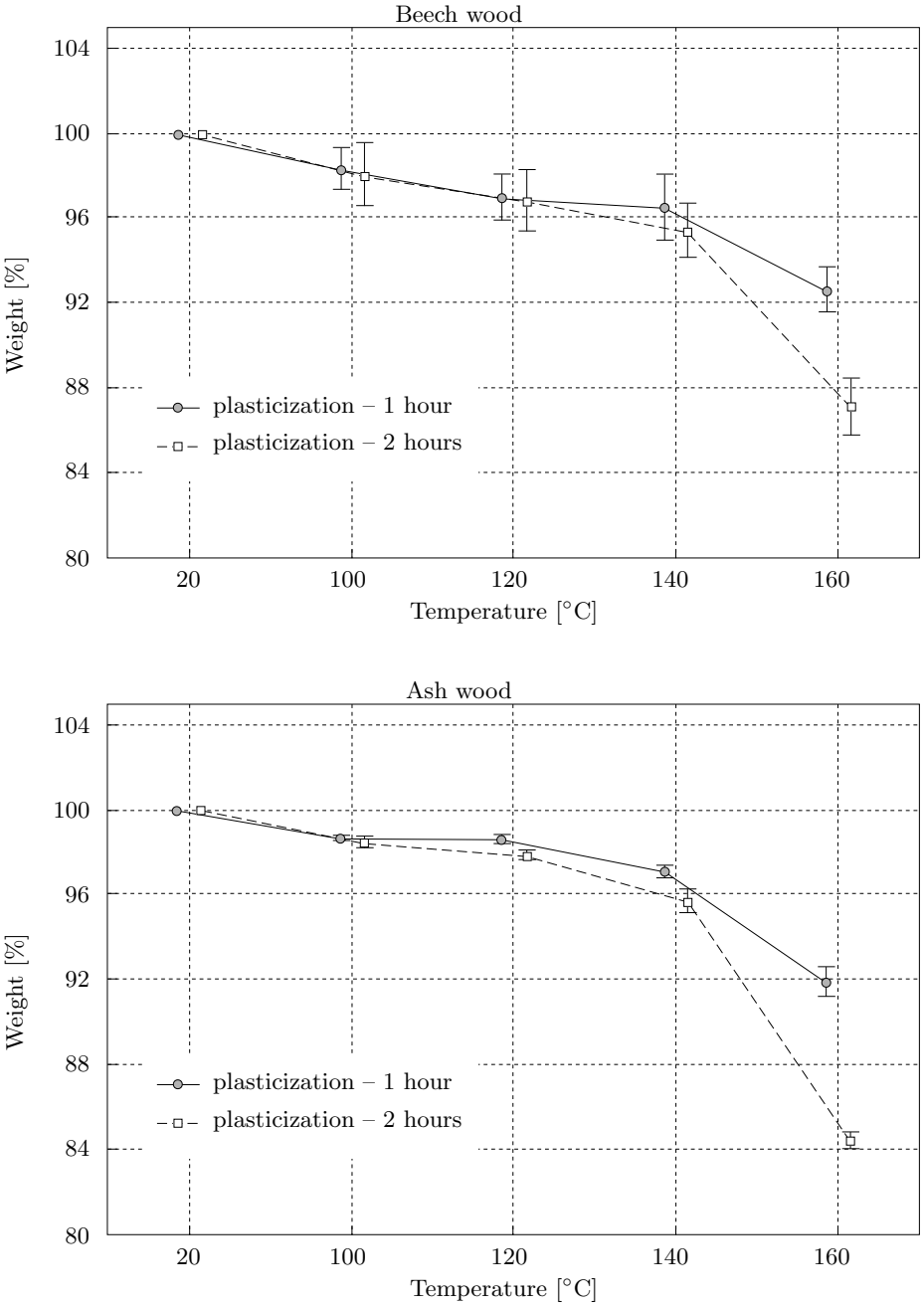

Fig. 2. Influence of temperature of plasticization medium on beech and ash wood weight loss

Experimental results have also confirmed that there were no statistically significant differences in maximum shrinkage values in the individual anatomical directions as well as in maximum volumetric shrinkage, either in beech or ash wood, between the wood plasticized at 100°C (in several cases up to 120°C) and the non-plasticized wood. However, at plasticization temperatures exceeding 100-120°C, there was evident an exponential increase in values of maximum shrinkage with increasing temperature for both wood species. More conspicuous changes were observed in case of ash wood. After one hour of plasticization at 160°C, the shrinkage in the individual anatomical directions as well as the volumetric shrinkage was more than two times higher. After two hours at the same temperature, the shrinkage of plasticized ash wood was even three times higher than the shrinkage of non-plasticized wood of the same species (Fig. 3).

The plasticization time has also been recognised as an important factor with the influence necessary to consider already since 120°C. The experimental results have confirmed that the increase in beech and ash wood shrinkage is linearly dependent on the weight loss. It means that the increase in wood shrinkage after its plasticization depends on the amount of degraded compounds of the lignin-saccharides matrix.

The change in oven-dry density (ρ_0) of the two wood species depends on temperature change after one and two hours of plasticization – as illustrated in Figure 4. As can be seen in Figure 4, there is no significant change in beech wood density, dependent on temperature of plasticization. In case of ash wood, the initial trend is moderately decreasing (up to 100-120°C), followed by an increase. The change in density is in a close correlation with the weight change and the change in volumetric shrinkage.

The differences in colour change between plasticized and non-plasticized wood were also observed where we studied colour patterns within the given plasticization regimens (Fig. 5). The wood lightness L* was decreasing with increasing plasticization temperature and time. The ash wood was lighter, the change in its lightness, however, was more pronounced than in beech. At temperature of 160°C, the lightness of beech and ash wood was the same. In both wood species, also the colour hue was changing – with increasing temperature, also the coordinates a* and b* changed. Up to 100°C, there were found no significant changes in the coordinate a* in case of beech wood, in case of ash wood, a moderate decrease was observed. With further temperature increase, a* increased in both wood species. The trend of coordinate b* was decreasing with increasing temperature over the whole temperature range studied. In such a way, wood was turning dark-brown with increasing plasticization time and temperature. The same results were obtained by Tolvaj and Faix (1996), Tolvaj et al. (2002), Pervan et al. (2006).

Studying changes in mechanical properties we observed that, apart from the temperature of plasticization medium and the time of plasticization process, the changes in wood properties were also influenced by the wood moisture content at which the properties were measured (Fig. 6). In case of specimens with moisture content above the FSP, the compression strength parallel to grain decreased over the whole temperature range. At 160°C, after one-hour lasting plasticization, the strength of beech wood decreased on average by 56%, and ash wood strength

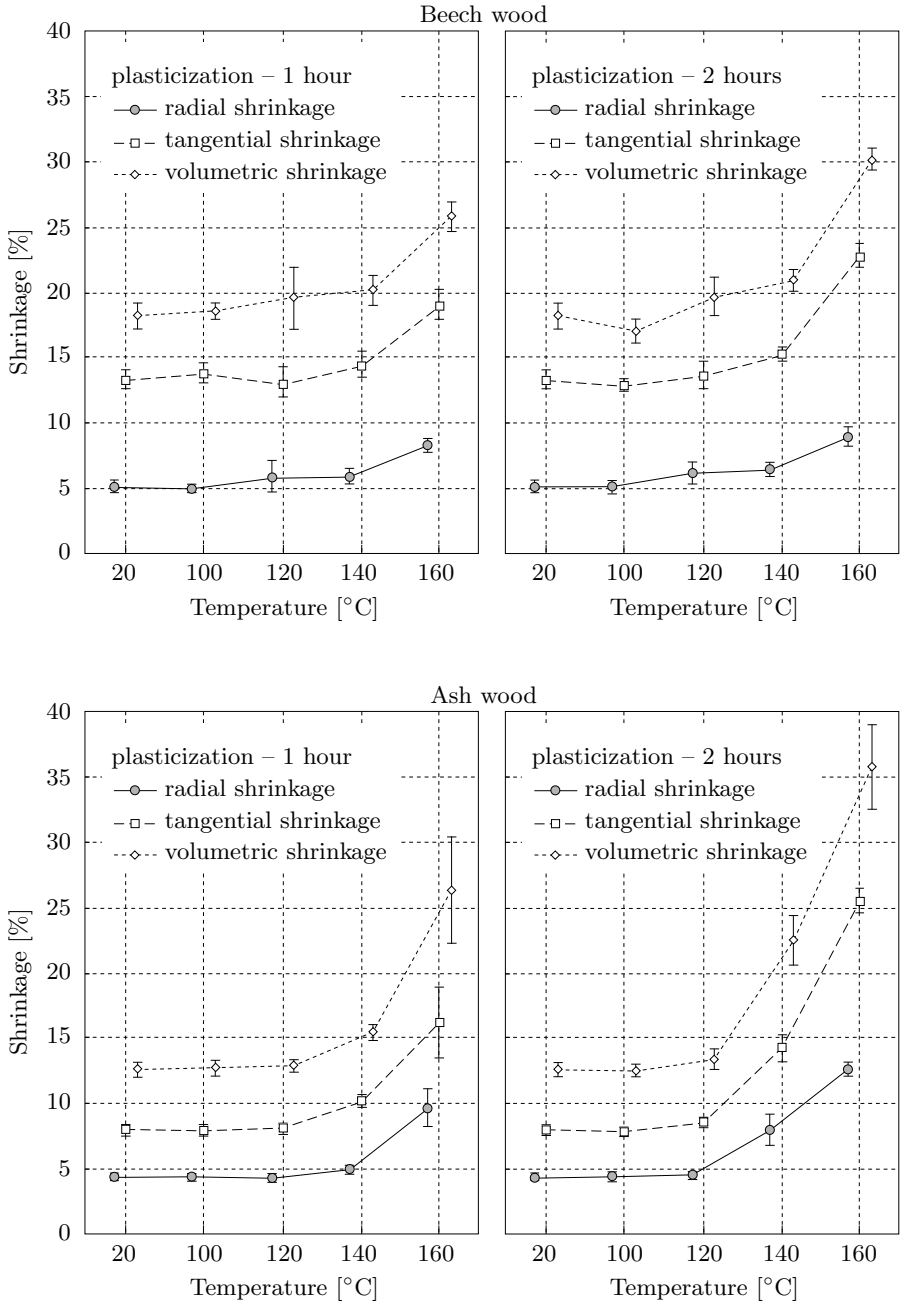

Fig. 3. Influence of temperature of plasticization medium on beech and ash wood shrinkage

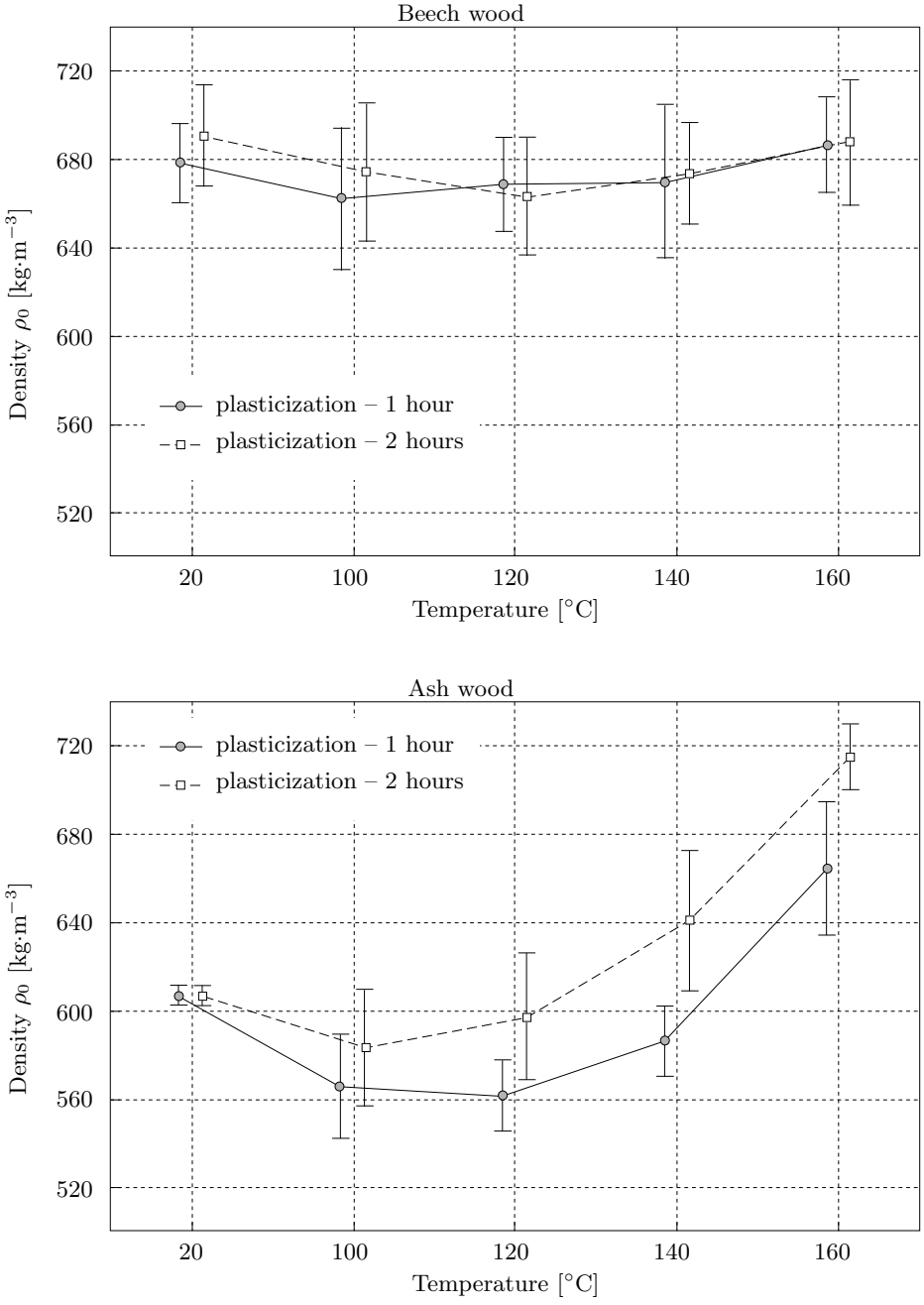

Fig. 4. Influence of temperature of plasticization medium on beech and ash wood density

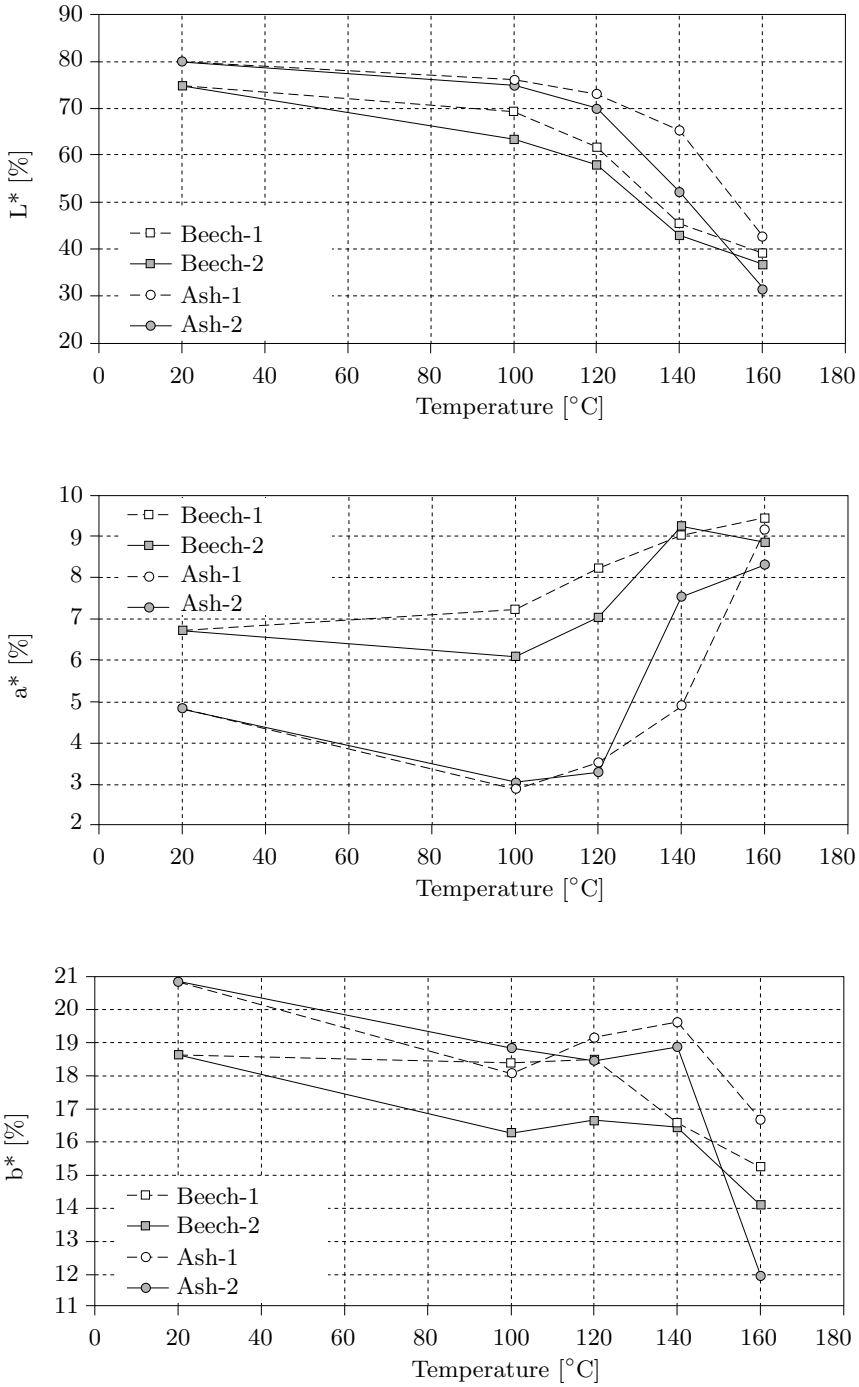

Fig. 5. Influence of temperature of plasticization medium on beech and ash wood colour change

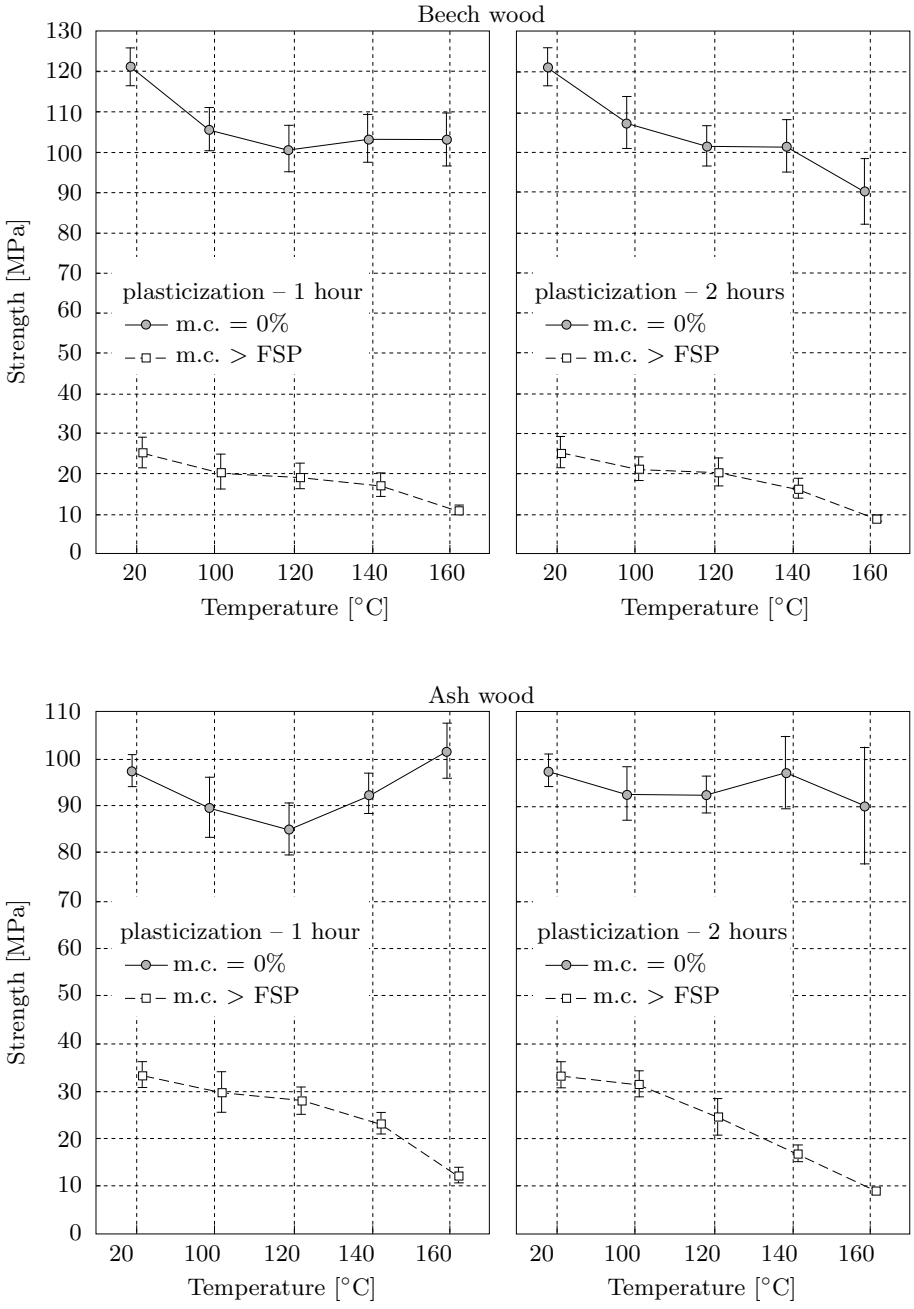

Fig. 6. Influence of temperature of plasticization medium on beech and ash wood strength parallel to grain

by 63%. After two hours, at the same temperature, the decrease in beech wood strength was about 65%, in ash it was about 72%.

In case of specimens dried to the zero moisture content, the influence of plasticization was found much less pronounced than in wet specimens. The drop in strength of beech wood plasticized for one hour at 160°C represented only 16%. After two hours at the same temperature, the change was more remarkable (26%). Similar qualitative changes in permanent strength of plasticized beech wood were also observed by Čunderlík et al. (1995).

In case of ash wood with zero moisture content, no unambiguous influence of plasticization was evident after one-hour plasticization. The negative influence of plasticization was partially outweighed with higher density of the plasticized specimens after their drying. The influence of plasticization was not only reflected in the strength value but also in the way of specimens' distortion. The distortion modes (primarily at 160°C) suggest a considerably degraded middle lamella.

Pronounced changes in the studied physical and mechanical properties of beech and ash wood are initiated by a temperature of 100°C or 120°C, and they are correlated with chemical changes described in the paper by Laurová and Kačík (2009).

CONCLUSIONS

Hydrothermal plasticization of beech and ash wood in water bath heated above 100°C induces wood mass loss caused by gradual removal of chemical components from the lignin-saccharide matrix. This is not only reflected in momentous but also permanent changes in wood physical and mechanical properties. Wood shrinkage increases, mechanical properties decrease, changes in wood colour are present, too. These changes in wood properties are more remarkable as temperature and length of plasticization time increase.

Since hydrothermal plasticization of wood is frequently carried out at higher temperatures (above 100°C), apart from instantaneous changes, also permanent changes in wood properties are necessary to investigate.

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Author's address:
Prof. Dr. Jozef Kúdela
Faculty of Wood Sciences and Technology
Technical University in Zvolen
T.G. Masaryka 24
960 53 Zvolen
Slovak Republic
kudela@vsld.tuzvo.sk