

INFLUENCE OF AN AGE AND DAMAGE OF THE OAK WOOD ON ITS FIRE RISK

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ABSTRACT

The aim of this work is to study the effects of different ages and damage of the oak wood (*Quercus petraea* Mattusch) in relation to its flash point temperature and ignition temperature, as well as on determination of the ratio of changes in extractives, cellulose and lignin, in the heat loaded samples of 5 - years old oak wood, 160 - years old oak wood, and also 160 - years old oak wood degraded by wood-destroying insects and fungi.

The results of the analyses showed that the flash point temperatures were in the range of 357.52°C - 360.63°C. The ignition temperatures were at interval of 398.93°C - 414.92°C. The time to reach the ignition point was at the interval between 344 and 365 seconds. Under the thermal loading of oak wood, there comes to the significant changes, especially in the surface layers. These are, in addition to colour changes, demonstrated the chemical changes of the main components of wood and extractives. Increasing the temperature, there increases also the content of the substances extractable with ethanol and toluene. Increase in their amount is mainly due to the lignin, but partially also due to the polysaccharides decomposition products.

KEYWORDS: *Quercus petraea* Mattusch, fire risk, cellulose, lignin, extractives.

INTRODUCTION

Wood was the traditional building material in Slovakia in the past. For its excellent design features, workability and easiness to splice, it served for the construction of residential and farm buildings. The windows, doors, ceilings, roofs, and also covering, were made from wood.

The homeland of the wood constructions is the northern and central part of Slovakia (geomorphologically rugged, richly forested territory), in particular. The typical localities are in the Kysuce, Orava, Liptov, Horehronie, Spis regions. There, the whole villages made of wood were known (Thurzo 2010).

Many of these wooden buildings, despite the fact that wood as an organic material, were subjected to various insults over the time, but they were preserved until this time and has become a part of the cultural heritage. According to Reinprecht (2009), in order to preserve this cultural heritage for future generations, there is an absolute need for adequate care of monuments made from wood, especially the objects included in the list of cultural monuments (wooden houses, wooden churches, palaces, castles, historical ceilings, roofs, doors, windows, staircases and other parts of buildings with folk architecture).

Wood is a material characterized by a relatively inhomogeneous anisotropic structure, consisting of a macromolecular substances complex (cellulose, hemicelluloses, lignin and extractives). It has sufficient strength, flexibility, good thermal-isolation and acoustic properties. Its surface is often treated in an appropriate manner by applying various coatings.

The most serious weaknesses of wood is the little resistance to attacks of biotic pests (fungi, mould, rot) and its easy flammability. For a correct and complete understanding of the fire protection of wood, it is important to know the mechanism of the thermal energy (fire) action on the wood surface, and the changes occurring in the wood, as well as the impacts of the combustion processes. For this reason, the great importance is given to the research in the framework of which is necessary to follow, in addition to the mechanical (Krakovský and Král 2004), physical and structural changes (Korytářová and Osvald 2000), also the chemical changes in the elementary building components of wood, which take place in the thermal loading (Kačíková et al. 2006, Chrebet et al. 2013, Martinka et al. 2014), and thus to contribute to addressing the issue of wood fire protection.

In terms of fire investigation, the number of wood buildings fires is constantly growing. Therefore, the demands to know more about the fire behaviour of wood are still increasing, too. It is not just because of finding the exact place of fire initiation, the initiator respectively, but also to determine the time of fire initiation. For this reason, also the presented paper discusses the degradation of wood and determination of fire characteristics, which are the basic data needed to correctly determine the cause of fire.

Combustion of wood is in general a very complex physical-chemical process, in which comes to the thermal break down of wood particular components and to the change in its chemical composition. The least resistant to thermal decomposition are the wood polyoses (hemicellulose) (Kačíková et al. 2006, Wendeise et al. 2009), which are broken down in the temperature range of 170-240°C. The most durable component of wood is the lignin. Active decomposition of lignin takes place at temperatures of 300-400°C. The cellulose is much more resistant to heat than the hemicellulose. Up to the temperature of 250°C, the process of its decomposition is slow. More intense thermal decomposition occurs in the temperature range of 250-350°C (Shafizaden 1984, Reinprecht 1996, Chrebet et al. 2012). The extractives contents in the wood depends largely on the sampling site, conditions for growth of the tree, weather conditions, season and the way of storage of the specimens. During the wood thermal loading, the part of extractives, e.g. terpenes, disappears. The degradation of the main components of wood gives rise to a new, less volatile, low molecular weight compounds, which may be extracted by polar or non-polar solvents.

When heating the wood, there is formed the acetic acid, by removal of the acetylene groups from the hemicelluloses, which catalyses the hydrolysis of hemicelluloses to soluble sugars. The acetic acid also results in the depolymerisation of cellulose into the amorphous areas, while reducing its chains (ThermoWood® Handbook 2003).

The changes in the chemical composition and structure of the wood particular components were observed in aging (Dadashian and Wilding 2001, Zachar et al. 2014), oxidation (Hon 1989), hydrolysis (Laurová and Kúdela 2008), and thermal degradation (Lee et al. 1988, Kačíková 2007, Windeisen, Wegener 2009, Rousset et al. 2009).

The aim of this paper is to determine the impact of the age of oak wood on its flash point temperature and ignition temperature, which belong among the basic fire characteristics, as well as on determination of the ratio of changes in extractives, cellulose and lignin, in such heat loaded specimens, prepared from 5 - years old oak wood, 160 - years old oak wood, and also 160 - years old oak wood degraded by wood-destroying insects and fungi.

MATERIAL AND METHODS

Material

Oak tree (*Quercus petraea* Mattusch) is one of the most valuable species in Slovakia. Oak wood quality may vary by the habitat. It is believed that the oak trees with thick, deeply furrowed bark, have less quality wood. Wood is a circular-porous, with significant ray cells, with distinct brown grain. It has a coarse fibre, is hard, heavy, rich in tannins. When stored in water, it is durable and is valued for its excellent technical properties (Kyzlík et al. 1966).

The specimens of oak wood themselves were made from new wood (5 - years old tree), old wood (160 - years old tree) and old wood affected with the wood decaying insects and fungi, with dimensions of 20 x 20 x 10 mm and weight of $3.0 \text{ g} \pm 0.2 \text{ g}$, in accordance with the STN ISO 871 standard. The moisture content of the specimens tested was set to 9%. The specimens / samples were conditioned at $23^\circ\text{C} \pm 2^\circ\text{C}$ and relative humidity of $50\% \pm 5\%$ for 40 hours, in accordance to ISO 291 standard. The measurement procedure for ignition temperature analysis corresponds with the methodology introduced in the STN ISO 871 standard.

Determination of the flash point and ignition temperature of the oak wood

The test is based on heating the material in the heating chamber - in an oven at various temperatures. When the hot-air furnace is opened, the released gases are ignited and the flash point temperature is determined. Ignition temperature is determined the same way as the flash point temperature, but without the use of igniting flame. The course of the temperature in the furnace was measured with thermocouples (Type K) with a diameter of 0.5 mm. To record the temperatures, there was the ALMEMO 2290-8 device used. For weighing the specimens the Sartorius Basic plus electronic scale was used.

Removing the toluene extractive using the ethanol extraction

Into the toluene ethanol mixture come the substances with medium polarity, such as the low molecular weight phenols, aromatic aldehydes, lignols, terpenoids and some vegetable dyes and to a lesser extent also the lipids, monosaccharides, sugar alcohols, cyclitols and esters.

For the quantitative determination, according to the ASTM Standard D 1107-96, there were used the wood chips with weight of 2 g (0.425 to 0.250 mm fraction). These wood chips were extracted using the azeotropic mixture, containing the toluene and ethanol, at a ratio of 427 ml of toluene and 1000 ml of ethanol. The advantage of the azeotropic mixture is the fact, that the mixture is binding also the moisture present in the wood chips. The wood chips extraction takes 6-8 hours and if necessary, it can also be extended (Kačík et al. 1999).

Determination of Seifert's cellulose

Seifert (1956, 1960) developed a rapid method for the determination of cellulose based on the action of acetyl acetone. The resulting cellulose is of high purity, and compared to the cellulose determined by the Kuschner and Hoffer method, it contains less hemicellulose and lignin and has higher values of PPS. Another advantage of this process is the short duration of the analysis –the analysis itself run less than one hour.

For the determination was used 0.5 g of extracted wood chips, to which 6 ml of acetylacetone, 2 ml of dioxane and 1.5 ml of concentrated hydrochloric acid (with consistency of 1.19 g.ml⁻¹) were added. The mixture was heated on a boiling water bath for 30 minutes in a round bottom flask with a volume of 250 ml, after which there was added 40 ml of methanol into the mixture and further was transferred to G2 frit. The mixture was successively washed with 100 ml of methanol, 40 ml of hot water, 40 ml of dioxane and 40 ml of methanol. The obtained cellulose was dried to constant weight at temperature of 105 ± 2°C (Kačík et al. 2007).

Determination of lignin content

The analysis was performed in accordance to the T222 NREL standard. Into the flask of drop shape (volume of 50 ml) was inserted the sample (weight of 150 mg), which had been dried in a vacuum oven at 50°C, and then we added 1 ml of cooled 72% H₂SO₄. The sample was placed in a water bath at temperature of 30 ° C for 1 hour, to be tempered. Then we added 42 ml of distilled water to the sample, the flask was tightly sealed with a ground glass stopper and the sample was hydrolysed for 1 hour at 120 ° C in a thermostat (oven). The bank was immersed in glycerine bath in the hydrolysis. After the hydrolysis, the sample was cooled, quantitatively transferred to a volumetric flask (50 ml) and filled up with distilled water to the mark. The whole amount of sample was filtered through a previously dried and weighed S4 glass filter. The filter was collected lignin, which we washed with 500 ml of hot distilled water until the neutrality was reached and then we placed the air-dry samples in the oven with temperature of 105°C and dried for 2 hours. The filter with lignin, after the cooling in a desiccator, was weighed and the lignin content in the sample was determined by the differences in the weight.

RESULTS AND DISCUSSION

Resulting values of selected fire parameters of oak wood

The results of the analyses showed, that the flash point temperatures were in the range of 357.52°C for samples of oak 160 - years old and affected by wood decaying insects and fungi, up to 360.63°C in case of 160 - years old sample of oak wood, see Tab. 1. The flash point temperature of 5 - years old oak tree was fixed to 357.89°C. With regard to the time, in which the flash point temperatures were reached, the samples parameter values are comparable. They moved in the range of 370-416 seconds. Ignition temperature was recorded at intervals of 398.93°C for samples of 160 - years old wood, 399.56°C for the sample of 5 - years old wood and 414.92°C for 160 years old sample of oak wood affected by wood decaying insects and fungi. The value of time interval was between 344 and 365 seconds. The individual values of the flash point temperature and of ignition temperature can be seen in Tab. 1. In this case, it is necessary to take into account the different densities of individual samples after conditioning, which showed a significant decrease in bulk density, depending on the age and damage caused by the wood decaying insects and fungi.

Tab. 1: Resulting values of oak specimen flash point and ignition temperature and time.

Wood specimen	Bulk mass after conditioning (kg.m ⁻³)	Flash point temperature (°C)	Time to flash point (s)	Ignition temperature (°C)	Time to ignition (s)
Oak – young wood (5 - years old)	828	357.89	370	399.56	364
Oak – old wood (160 - years old)	770	360.63	416	398.93	365
Oak – damaged old wood (160 - years old)	586	357.52	396	414.92	344

*The measurements were repeated 10 times.

Our results are comparable also with results of other authors. Chrebet et al. (2011), in their work introduced the flash point temperature of European beech wood samples at interval starting from 400°C and the time to the flash point of 300 - 420°C seconds. The time to reach the flash point was of 235 seconds. This issue was also addressed in the work of Delichatsios et al. (2003), who reached the ignition temperature of about 478°C when used the pine wood. Also Hagen et al. (2009) reached the ignition temperature of the samples Norway spruce, untreated with retardant, of 487.9°C. The flash point temperature of oak samples introduced Zachar et al. (2010), in their previous work. They obtained very comparable values of flash point temperature: 420° C for the time interval of 455 seconds. Martinka et al. (2011) in their work considered the induction period of oak samples in the interval of 450-550°C. They described the full time course of the temperatures. The values measured are significantly higher, which could be a result of different moistures of samples, as well as the surface finish quality. The ignition temperature and assessment of wood-based materials addressed also Galla, Ivaničová (2013). In their work, they evaluated particle materials and OSB boards. When considering the rate of heat release, similar results have been measured in work Zachar et al. (2014), confirming the fact, the higher the age of the wood, the lower the measured heat release rate over the time interval of 30 minutes.

Resulting chemical changes

Under the thermal loading of oak wood, there comes to the significant changes, especially in the surface layers. These are in addition to colour changes, demonstrated in the chemical changes of the main components of wood and extractives (Kačík et al. 2006). Increasing the temperature, there also the content of the substances extractable with ethanol and toluene increases. The increase in their amount is mainly due to the lignin, but partially also polysaccharides decomposition products (Tab. 2).

The increased yields of cellulose in the thermally degraded sample, in comparison to the original sample (Tab. 2), is related to changes in the structure of cellulose (Inari et al. 2007, Kato and Cameron 1999). It is likely, that the increase in the content of the cellulose is provided only relative in terms of the degradation of less stable hemicelluloses. The cellulose is thermally more labile than lignin.

Tab. 2: Resulting values of extractives, cellulose and lignin.

Wood specimen	Extractives share (%)	Cellulose share (%)	Lignin share (%)
Oak – young wood (5 - years old) thermally unloaded	3.22	38.73	24.86
Oak – young wood (5 - years old) at flash point temperature of 357.89 °C	12.94	56.36	31.56
Oak – young wood (5 - years old) at ignition temperature of 399.56 °C	5.93	86.30	76.52
Oak – old wood (160 - years old) thermally unloaded	5.90	37.48	24.86
Oak – old wood (160 - years old) at flash point temperature of 360.63 °C	13.20	61.75	39.17
Oak – old wood (160 - years old) at ignition temperature of 398.93 °C	11.16	73.52	56.72
Oak – damaged old wood (160 - years old) thermally unloaded	4.86	37.00	24.28
Oak – damaged old wood (160 - years old) at flash point temperature of 357.52 °C	9.78	56.70	38.09
Oak – damaged old wood (160 - years old) at ignition temperature of 414.92 °C	7.62	70.93	54.15

Almost all the original extractives are removed in the wood during the heating, and there occurs formation of new compounds from the degradation of lignin and polysaccharides. Those are the monosaccharides and their dehydration products (e.g. arabinose, xylose, galactose, glucuronic acid, etc.), and the degradation products of lignin (e.g. vanillin, syringaldehyde, syringic acid, sinapylaldehyd, etc.). In our work, there can be seen higher amount of extractives in the wood affected by wood decaying fungi and insects and also in old wood. According to Esteves et al. (2008), during the thermal loading of wood, first comes to the increase in extractives, later the amount decreases. Increase of extractives in thermally treated wood has been found in other works (Kačík et al. 2006, Kačíková et al. 2008, Nuopponen et al. 2004, Windeisen and Wegener 2009), too.

The results of determination the polysaccharide component of wood - cellulose, aromatic component - lignin and extractives (Tab. 2) showed that the increase of temperature leads to the significant degradation of polysaccharides, mainly the hemicelluloses. The amount of lignin in the char layer is increased two- to threefold. Similar trends in yields have also been observed by other authors (Fengel and Przyklenk 1970, Reinprecht 1999, Alen et al. 2002, Nuopponen et al. 2004, Kačík et al. 2007). One reason can be found in the condensing reactions with the products of degradation of the carbohydrates and also in the lignin auto-condensation (Kačík et al. 2006, 2007). When comparing lignin individual samples to each other, it can be said, that after the thermal degradation, the lowest yields were achieved by old wood affected by wood decaying fungi and insects. Wood-destroying fungi and insects contain enzymes which cause degradation of the main components of wood. Compared with polysaccharides, lignin is thermally the most stable main component of the wood however its change depends on the method of thermal loading and the origin of the sample. Slow heating at atmospheric pressure creates the conditions for growth, and the condensation reactions and the yields of volatile products are relatively low (Elder 1984, Blazej and Košík 1985, Cart 1985, Kačík et al. 2007).

The results obtained by thermal degradation of wood under different conditions (old, new, damaged wood) showed changes in yields of cellulose. Some authors found the increase in the yields (Kačík et al. 2006, Bubeníková et al. 2006), others on the contrary, observed the opposite trend (Fengel 1967, Kačíková 2004). The reasons for the different findings may be just different experimental conditions of wood heating.

CONCLUSIONS

In the paper, there were described the methodological procedures and results of the analyses focusing the effects of different ages and damage of the oak wood (*Quercus petraea* Mattusch) on the flash point temperature and ignition temperature determination, and determination of the ratio of changes in extractives, cellulose and lignin under the thermal loading. In the analyses, samples of 5 years old oak wood, 160 years old oak wood, and also 160 years old oak wood degraded by wood-destroying insects and fungi were applied. In this research, there were obtained results comparable with several authors dealing with this issue.

The flash point temperatures were found in the range of 357.52°C up to 360.63°C. The ignition temperatures were found at interval of 398.93°C up to 414.92°C. The time to reach the ignition point was at the interval between 344 and 365 seconds. Under the thermal loading of oak wood, there comes to the significant changes, especially in the surface layers. These are in addition to colour changes, demonstrated in the chemical changes of the main components of wood and extractives. The results of determination the polysaccharide component of wood - cellulose, aromatic component - lignin and extractives showed that the increase of temperature leads to significant degradation of polysaccharides, mainly hemicelluloses. The amount of lignin in the char layer is increased two- to threefold.

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