ORIGINAL ARTICLE

Thermal characterization of wood of nine European tree species: thermogravimetry and differential scanning calorimetry in an air atmosphere

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Abstract

This paper presents a thermal characterization of hardwoods of Hungarian origin. The study covers the wood of the following tree species: sessile oak (*Quercus petraea*), beech (*Fagus sylvatica*), hornbeam (*Carpinus betulus*), black locust (*Robinia pseudoacacia*), lime (*Tilia platyphyllos*), ash (*Fraxinus ornus*), maple (*Acer campestre*), alder (*Alnus glutinosa*), and aspen (*Populus tremula*). Wood samples were subjected to differential scanning calorimetry and thermogravimetry in an air atmosphere at different heating rates of 10, 20, and 40 °C·min^{-1}. The activation energy was calculated using the Flynn–Wall–Ozawa method. Thermogravimetric analysis revealed that tree species differed considerably in the mass loss and the temperature of the maximum mass loss rate related to the thermal degradation of the main wood components and the oxidation of char mainly derived from lignin. Beech, hornbeam and ash were characterized by a high mass loss attributed to decomposition of hemicelluloses. The mass loss assigned to the thermal degradation of cellulose varied from 35.41% (ash wood) to 53.65% (alder wood). Sessile oak wood showed the greatest mass loss and the highest temperature of thermoxidation of residual lignin and char. Calorimetrically measured values of the exothermic effect of the wood thermal degradation were in the range 7.93–9.65 kJ g^{-1}. The average values of activation energy for the thermal degradation of carbohydrates and the thermoxidation of residual lignin and char were found to be $176.0 \div 197.3$ kJ mol^{-1} and $196.4 \div 319.5$ kJ mol^{-1}, respectively.

1 Introduction

With rising environmental problems, wood remains a unique renewable and environmentally friendly natural resource produced in significant amounts worldwide. The physical and mechanical properties of wood, its aesthetically pleasing characteristics, and its low cost are reasons for a growing interest in the use of wood for construction, building, decoration, and furniture (Tomak et al. 2012; Jiang et al. 2015) as well as for heating (Narron et al. 2020; Korshunov et. al. 2020). The properties of wood very often need to be modified in order to work with the material that is most suitable for a particular application. Thermo-hydro and thermo-hydro-mechanical modifications of wood are common, for example drying, steaming, and heat treatment (Sandberg et al. 2013). It is practically important to understand the thermal behavior of wood before it can be used as a building material, as reinforcement in polymer composites or as fuel (Yao et al. 2008; Poletto et al. 2012a; Poletto 2016b; TranVan et al. 2014; Todaro et al. 2018).

The methods of thermal analysis such as thermogravimetry (TG) and differential scanning calorimetry (DSC) are widely applied to study the thermal behavior of wood and lignocellulosic materials in inert and oxidative atmospheres. It is known that the thermal degradation of wood is determined by the thermal decomposition of its main components, i.e. hemicelluloses, cellulose, and lignin (Grønli et al. 2002; Poletto et al. 2010; Sebio-Puñal et al.

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2012). The proportions of these components in wood vary among species. In general, hardwoods have a higher content of hemicelluloses (25–35%) and less lignin (18–30%) as compared to softwoods (hemicelluloses 20–25%, lignin 26–34%). Cellulose content ranges between 35 and 50% for both hardwoods and softwoods. In hardwoods, pentosans are the main hemicelluloses, whereas hexosans are prevalent in softwoods (Azarov et al. 2010; Rowell et al. 2012). Wood shows different degradation profiles depending on the variation of chemical composition (Sebio-Puñal et al. 2012). TG and DSC allow to distinguish the temperature intervals of the thermal degradation of wood components and provide information about mass loss, the maximum mass loss rate, and reaction heat.

Among wood components, hemicelluloses have the lowest thermal stability. These polysaccharides are easily hydrolyzed in the temperature range of 225–325 °C due to the presence of acetyl groups. Cellulose decomposes in the temperature range of 305–375 °C (Shen et al. 2009; Poletto et al. 2010; Sebio-Puñal et al. 2012). Lignin decomposes slowly in a wide temperature range because various oxygen functional groups in its structure have different thermal stability, their cleavage occurs at different temperatures. The decomposition of the polymer structure in lignin starts at 200–275 °C but the main process occurs around 400 °C (Bartkowiak and Zakrzewski 2004; Brebu and Vasile 2010). Thus, the intervals of thermal degradation of wood components partly overlap (Órfão et al. 1999; Safi et al. 2004).

Numerous studies have focused on the pyrolysis of wood and lignocellulosic fibers, and the thermal decomposition kinetics under an inert atmosphere due to pyrolysis plays a major role in the production of fuels, wood-based carbonized materials, and polymer composite processing (Ota and Mozammel 2003; Grioui et al. 2006; D'Almeida et al. 2008; Yao et al. 2008; Poletto et al. 2010, 2012b; Slopiecka et al. 2012; Monteiro et al. 2012; Zakikhani et al. 2016; Sharma and Mohanty 2021). Analysis of wood degradation under oxidative conditions is related to the design and development of combustion and gasification systems (Branca and Blasi 2004). Combustion of lignocellulosic materials involves solid pyrolysis and combustion of pyrolysis products (Janković et al. 2020). It is known that the presence of oxygen enhances the thermal degradation of the materials at low temperatures, causes the appearance of gas-phase reactions between the volatile released and oxygen, and promotes the combustion of the char generated in the early stages of the solid degradation (Bilbao et al. 1997). Analysis of the literature shows that either the wood thermal degradation of various deciduous species in an oxidative atmosphere or kinetics of wood combustion has been described in several studies (Fang et al. 2006; Franceschi et al. 2008; Brostow et al. 2009; Budrugeac and Emandi 2010; Emandi et al. 2011; Garcia-Maraver et al. 2013; TranVan et al. 2014;

Loskutov et al. 2015). It was demonstrated that the methods of thermal analysis can be applied to the assessment of wood composition and the holocellulose/lignin ratio as well as to the evaluation of the combustion properties in terms of the amount of energy released and the thermal stability of wood. Brostow et al. (2009) stated that the species of the wood had an effect on the combustibility of the sample, but no connection was found between the wood density and the parameters characterizing the burning process. Fang et al. (2006) studied the influence of oxygen concentration on kinetics of wood combustion. It was found that the activation energy increased linearly with concentration of oxygen at the first stage. At the second stage of combustion, the activation energy varied linearly only when the concentration of oxygen was below 65%. So far, as we know, only a few studies provide both the thermal degradation characteristics of wood and the kinetic parameters of combustion process simultaneously for some hardwoods (Liu et al. 2002; Shen et al. 2009; Maryandyshev et al. 2015). Therefore, in the present work, the experiments on the wood samples were carried out in an air atmosphere. The objective was to study the wood thermoxidative degradation and its kinetics of nine deciduous species used in the timber industry in Europe. The data obtained from the thermal analysis of wood of the given species in an oxidative atmosphere will enhance our knowledge of wood heat resistance and contribute to the development of different combustion applications.

2 Materials and methods

The samples from nine hardwood species were studied, namely sessile oak (*Quercus petraea*) (1), beech (*Fagus sylvatica*) (2), hornbeam (*Carpinus betulus*) (3), black locust (*Robinia pseudoacacia*) (4), lime (*Tilia platyphyllos*) (5), ash (*Fraxinus ornus*) (6), maple (*Acer campestre*) (7), alder (*Alnus glutinosa*) (8), and aspen (*Populus tremula*) (9) of West Hungarian origin. These tree species have been widely used in the European wood industry. Wood samples were collected from the forests or the sawmills of Sopron region. A full disk (20 mm thick) was cut from the tree at breast height. The wood disks were stored for two months in a normal climate room to reach homogenous moisture content on the full cross section. The disks were sawn carefully with a fine-tooth metal saw on the diameter line of the disk to represent the full cross section of the tree (Fig. 1). The fine sawdust was used for the thermal analysis. The particle size was $50 \div 80$ meshes.

Thermogravimetry was carried out on a TG 209 F1 thermal analyzer (Netzsch, Germany) in an air atmosphere with 20 cm^3/min flow, from 25 up to 700 °C. Approximately 10 mg of powdered samples were placed into Al_2O_3 crucible and heated at different rates of 10, 20, 40 °C/min. Differential scanning
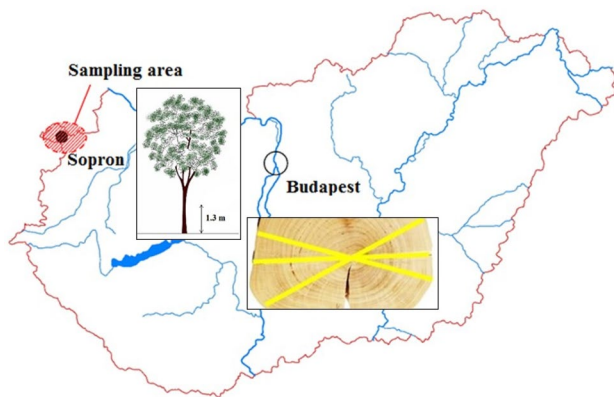
Fig. 1 Scheme of sampling and sample preparation

calorimetry was performed on a DSC 204 F1 thermal analyzer (Netzsch, Germany) in an air atmosphere with 40 cm³/min flow, from 25 up to 600 °C. Approximately 1 mg of powder was placed in an alumina crucible with a pierced lid and heated at a rate of 10 °C/min. The DSC measurement was repeated in triplicates for each specimen.

The activation energy (E_a) is one of the important kinetic parameters for a reaction. The activation energy of the thermal decomposition of wood was calculated based on thermogravimetric data using the isoconversional method of Flynn–Wall–Ozawa (FWO). According to the FWO theory, an equation for calculating E_a looks like (Mamleev et al. 2004):

$$\ln(\beta) \cong ln\left(\frac{AE_a}{RF(\alpha)}\right) - 5.3305 - 1.052 \times \frac{E_a}{RT}$$

where α is the degree of conversion: $\alpha = \frac{m_0 - m}{m - m_f}$; m_0 is the initial sample mass in the TG experiment; m is the current sample mass at temperature T; m_f is the final sample mass after thermal degradation; β is the heating rate; R is the gas constant; A is the pre-exponential factor; $F(a)$ is the function of mathematical representation of the FWO kinetic model.

The FWO method is based on the assumption that the reaction rate for a constant degree of conversion depends only on the temperature. The activation energy of the thermal decomposition of a sample is determined by the value of the angle of the straight line slope constructed in coordinates $\ln\beta - \frac{1}{T}$ for a number of α values. Knowledge of the reaction mechanism (order) is not required.

3 Results and discussion

The TG/DTG profiles obtained from the thermal degradation of hardwoods in an air atmosphere are shown in Fig. 2. During thermal degradation in air, mass loss occurs continuously until the mass becomes almost constant and the DTG

peaks give a clear indication of the number of mass loss stages (Safi et al. 2004).

The mass loss in the TG curves below 180 °C (not shown in Fig. 2) attributed to the evaporation of water and volatile compounds of wood (Kumar et al. 2008) varied from 3.5% (lime) to 6.8% (aspen). In the range 180–700 °C, deciduous species revealed three peaks (shoulders) in the DTG (derivative thermogravimetric) curves related to three consistent stages of mass loss which represent the thermal degradation of the main wood components and char combustion (Liu et al. 2002; Gao et al. 2004a, b; Safi et al. 2004; Fang et al. 2006; Shen et al. 2009; López-González et al. 2013; Protásio et al. 2017; Janković et al. 2020). Analyzing the TG and DTG curves, it can be noticed that the thermal degradation of wood completely finished at 500 °C for all tree species studied.

The first stage is assigned to decomposition or dehydration of hemicelluloses in the range 185–303 °C (Gao et al. 2004a) and can be seen as a peak or shoulder in the DTG curves for all deciduous species studied. Up to 300 °C, the material does not undergo significant oxidation, and reactions typical for pyrolysis occur (Órfão et al. 1999). The peak at 283–289 °C was observed for beech, hornbeam, and ash. For other tree species, the decomposition of hemicelluloses was identified only as a shoulder. This can be explained by different amounts of hemicelluloses. The tree species namely beech, hornbeam, and ash had the high loss of mass (33.91–35.94%) at this stage. Alder, aspen, black locust, and sessile oak showed only 20.23–24.33% (Table 1). The rest revealed intermediate values. The present findings are consistent with the literature data. Shen et al. (2009) reported a shoulder-peak at around 280 °C attributed to thermal decomposition of hemicelluloses of deciduous species (aspen, oak, birch) in an oxidative atmosphere. Emandi et al. (2011) investigated the wood composition by thermogravimetry in static air and observed the maximum mass loss rate of hemicelluloses at a temperature of 288–297 °C for lime, hornbeam, maple, beech, and poplar.

The second stage is due to the dehydration and combustion of cellulose up to 380 °C. At this stage, the less-stable aliphatic groups are mainly broken down through hemolytic cleavage of C–C and C-H bonds, resulting in a highly condensed and cross-linked carbonaceous material (Gao et al. 2004a). The degradation of cellulose was observed as a well defined peak in DTG curves for all tree species studied. Sessile oak had the lowest temperature of the DTG peak, while lime, alder, and aspen showed the highest one. By defining the thermal stability as the DTG peak temperature it is suggested that cellulose component of sessile oak wood is the least thermally stable among hardwoods. The mass loss at this stage varied from 35.41% (ash) to 53.65% (alder). The maximum mass loss rate was significantly higher (18.68–19.65% min⁻¹) for
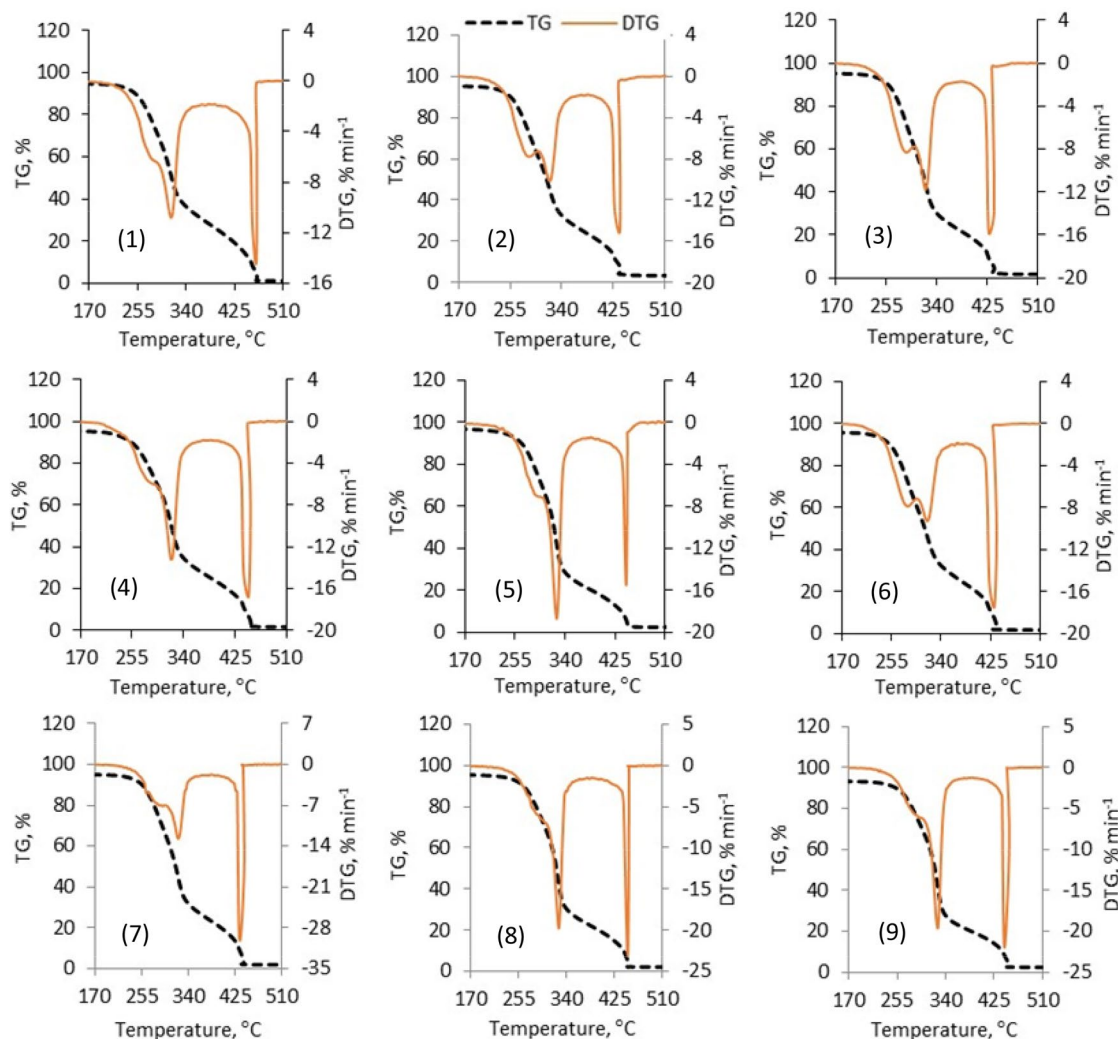
Fig. 2 TG/DTG curves of wood samples at a heating rate of 10 °C/min in air atmosphere: sessile oak (1), beech (2), hornbeam (3), black locust (4), lime (5), ash (6), maple (7), alder (8), aspen (9)

lime, alder, and aspen which had a greater mass loss of cellulose and the least (9.28% min^{-1}) for ash. The difference in the thermal stability of cellulose in wood of the tree species studied may be related to its crystallinity index, crystallite size and degree of polymerization (Poletto et al. 2012a). The cellulose thermal stability may also be influenced by extractives. It was demonstrated that a higher content of extractives accelerates the degradation process and promotes an increase in the conversion values at lower temperatures reducing the wood thermal stability (Poletto 2016a).

In general, the deciduous species showed a total mass loss of carbohydrates (hemicelluloses and cellulose) from 67.72% (sessile oak) to 75.88% (lime). The present results are similar to those obtained by thermogravimetry in air for maple, beech, hornbeam of Romanian origin (Emandi et al. 2011).

The third stage is due to degradation of residual lignin and oxidation of char formed previously. Lignin is the main contributor at this stage. Lignin is responsible for the formation of char due to its higher thermal stability compared to cellulose and hemicelluloses (Protásio et al. 2017). According to Órfão et al. (1999) and Yang et al. (2007), char yield was 37–46% for lignin, 20% for hemicelluloses and 5% for cellulose. A very narrow peak in the DTG curves and a high mass loss rate reflect a rapid combustion of char. Sessile oak showed the highest mass loss (25.69%), aspen and lime the lowest ones (17.54 and 17.69%, respectively). The DTG peak temperature was the highest (462 °C) for sessile oak indicating the greatest lignin thermal stability, followed by alder, black locust and aspen, lime, maple, beech, hornbeam, and ash. The maximum mass loss rate was the lowest for sessile oak and the highest for alder. It indicates that more thermally stable lignin of sessile oak wood decomposes more

Table 1 Parameters of wood thermal degradation obtained by TG

Tree species	Tpeak, °C % min⁻¹	Mass loss, %	Tpeak, °C % min⁻¹	Mass loss, %	Total mass loss of carbo hydrates, %	Tpeak, °C % min⁻¹	Mass loss, %	Residue at 700 °C, %
Sessile oak	Shoulder at 283	24.33	*314* 10.82	43.39	67.72	*462* 14.80	25.69	1.27
Beech	*286* 7.79	33.94	*320* 10.19	37.18	71.12	*433* 15.90	20.72	3.35
Hornbeam	*289* 8.32	33.91	*322* 11.76	39.55	73.46	*432* 17.11	19.87	1.92
Black locust	Shoulder at 292	24.10	*320* 13.33	46.46	70.56	*446* 17.54	23.49	1.71
Lime	Shoulder at 297	27.76	*326* 18.68	48.12	75.88	*443* 15.72	17.69	2.71
Ash	*283* 7.86	35.94	*317* 9.28	35.41	71.35	*429* 17.89	22.24	1.82
Maple	Shoulder at 296	28.20	*322* 12.41	42.69	70.89	*437* 17.52	21.56	1.68
Alder	Shoulder at 295	20.23	*326* 19.34	53.65	73.88	*447* 23.48	19.63	1.79
Aspen	Shoulder at 295	22.21	*326* 19.65	50.92	73.13	*446* 21.25	17.54	2.19

Tpeak temperature of the peak in the DTG curve, %min⁻¹ maximum mass loss rate

slowly as compared to other hardwoods studied. Shen et al. (2009) also observed that oak had the highest temperature of DTG peak (450 °C) compared to other deciduous species at a heating rate of 10 K/min in an oxidative atmosphere. Emandi et al. (2011) reported the temperature of the DTG peak assigned to lignin thermoxidation at 473.8 °C for oak (*Quercus robur*).

The amount of residual at 700 °C (ash) reflects the inorganic content and varied from 1.27% (sessile oak) to 3.35% (beech). The present results are consistent with literature data. Brostow et al. (2009) studied combustion properties of woods and reported ash content of 0.6–2.7% obtained in TGA experiments. Liu et al. (2002) found that the ash yield at 750 °C from wood degradation was in the range 1.0–6.3%.

The DSC curves show the heat flow during thermal degradation of wood samples (Fig. 3). Below 150 °C, an endoderm at 49–63 °C mainly due to the removal of moisture was observed for all wood samples studied (not shown in Fig. 3). With increasing temperature above 150 °C, the processes become exothermic with energy release from the devotalization and combustion (Maryandyshev et al. 2015). The DSC curves of tree species exhibited two exothermic peaks (exotherms) at 333.6–341.6 °C and 447.8–481.9 °C, which can be attributed to thermal degradation of cellulose and thermoxidation of residual lignin and char, respectively. The present findings related to the temperatures of two exotherms are in accordance with literature data reported for the combustion of woods (Tsujiyama and Miyamori 2000; Maryandyshev et al. 2015). Besides the first exothermic peak of cellulose degradation,

there is a slight shoulder located at the lower temperature region in the DSC curves, which may be assigned to the decomposition of hemicelluloses. DSC peaks correspond to two main peaks in DTG curves of wood samples but occur at higher temperatures due to differences in processes of the heat and mass transfer. Two sharp and closely spaced exothermic peaks indicate a large rate of heat release during the oxidative degradation of wood (Gao et al. 2004b).

The tree species revealed some differences in peak intensities in the DSC curves. According to the literature, the height and area of DSC peaks reflect the various content and nature of cellulose and lignin in woods (Reh et al. 1986; Franceschi et al. 2008). It can be noticed that the temperature of the second exothermic peak varied significantly among hardwoods. The sessile oak wood was characterized by the highest peak temperature at which heat release occurred during the thermoxidation of residual lignin and char formed previously. This fact confirms the thermogravimetry data.

The values of exothermic effect of wood thermal degradation obtained by DSC are given in Table 2. The amount of heat released was the highest (9.65 kJ g⁻¹) for sessile oak and the lowest (7.93 kJ g⁻¹) for aspen. The present results are similar to the reported values of 8.60 kJ g⁻¹ obtained for maple (*Acer saccharum*) but higher than 6.83 kJ g⁻¹ found for oak (*Quercus rubra*) by Brostow et al. (2009). A positive correlation was found between the value of the exothermic effect and the mass loss of samples at the third stage assigned to thermoxidation of lignin and char (r = 0.95 at p < 0.05).

Fig. 3 DSC curves of wood samples at a heating rate of 10 °C/min in air atmosphere: sessile oak (1), beech (2), hornbeam (3), black locust (4), lime (5), ash (6), maple (7), alder (8), aspen (9)
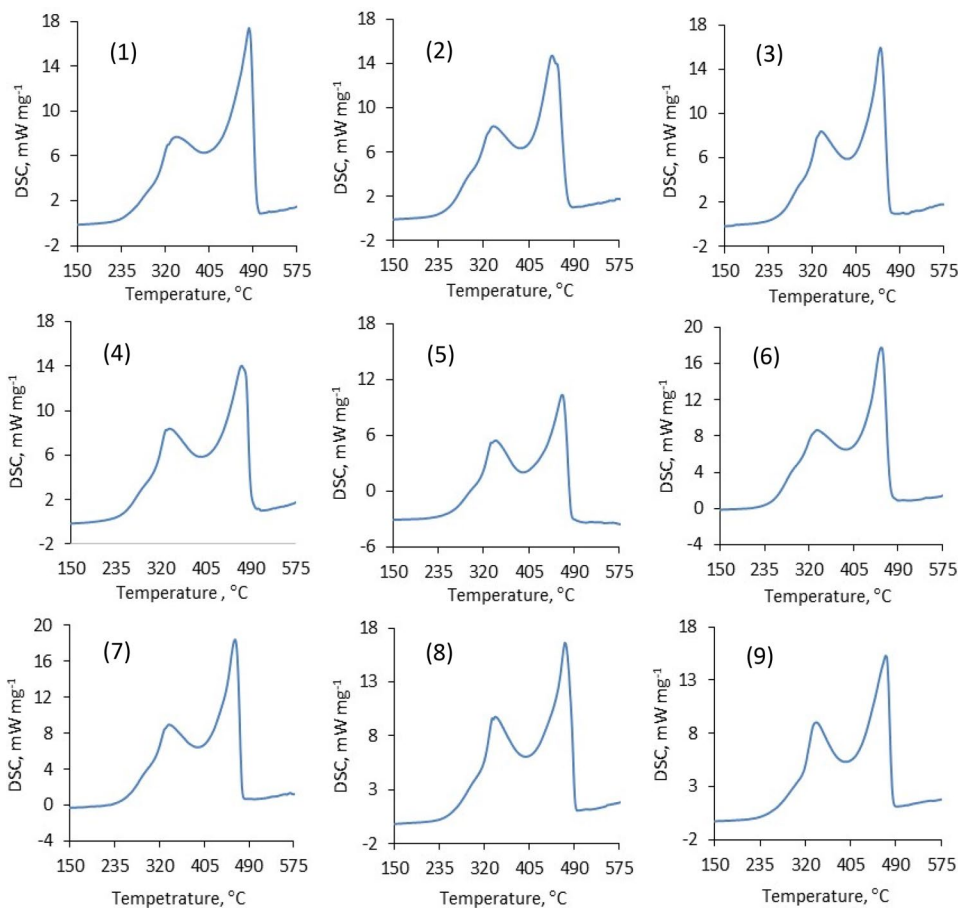
Table 2 Peak temperatures and exothermic effect of wood thermal degradation obtained from the DSC curves

Tree species	Peak, T °C	Peak, T °C	Q, kJ g^{-1}
Sessile oak	340.9 ± 0.4	481.9 ± 0.8	9.65 ± 0.14
Beech	337.8 ± 0.7	447.8 ± 1.6	8.92 ± 0.86
Hornbeam	335.3 ± 1.6	453.8 ± 1.9	8.62 ± 0.66
Black locust	333.6 ± 1.7	471.4 ± 2.0	9.50 ± 1.27
Lime	340.1 ± 2.1	463.8 ± 3.2	8.37 ± 0.58
Ash	334.6 ± 1.3	455.0 ± 3.3	9.05 ± 0.84
Maple	337.3 ± 0.6	460.2 ± 2.9	8.79 ± 0.73
Alder	339.2 ± 0.9	470.3 ± 3.0	8.82 ± 0.49
Aspen	341.6 ± 1.0	470.7 ± 2.5	7.93 ± 0.29

Data presented as a mean and standard deviation

Fig. 4 FWO plot of ash wood for given conversion values

The FWO method is the most suitable method to determine the kinetics of the thermal decomposition of wood and other materials (Mamleev et al. 2004; Muralidhara and Sreenivasan 2010; Slopiecka et al. 2012; Baroni et al. 2016; Abdelouahed et al. 2017; Sharma and Diwan 2017; Sobek and Werle 2020; Janković et al. 2020). The mechan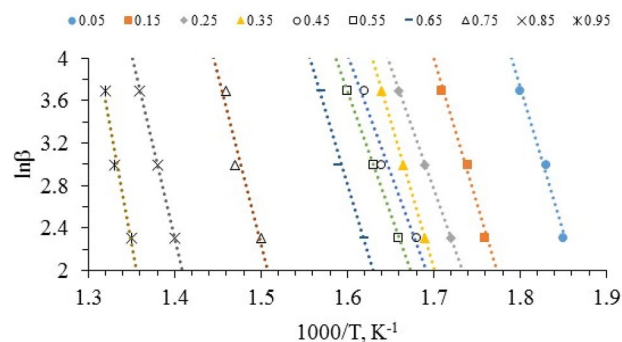isms and kinetics of thermal decomposition of wood in non-isothermal conditions have been described in detail by Poletto et al. (2012b).

Figures 4 and 5 show the building FWO graphs for the thermal degradation of ash wood (as an example) and the dependence of activation energy on the conversion rate of wood substance for all tree species studied.

The average value of activation energy of thermal decomposition of holocellulose (hemicelluloses, cellulose) for all tree species at $0.05 \le \alpha < 0.50$ was 186.90 kJ mol^{-1}, the standard deviation was 7.55 kJ mol^{-1}. The coefficient of
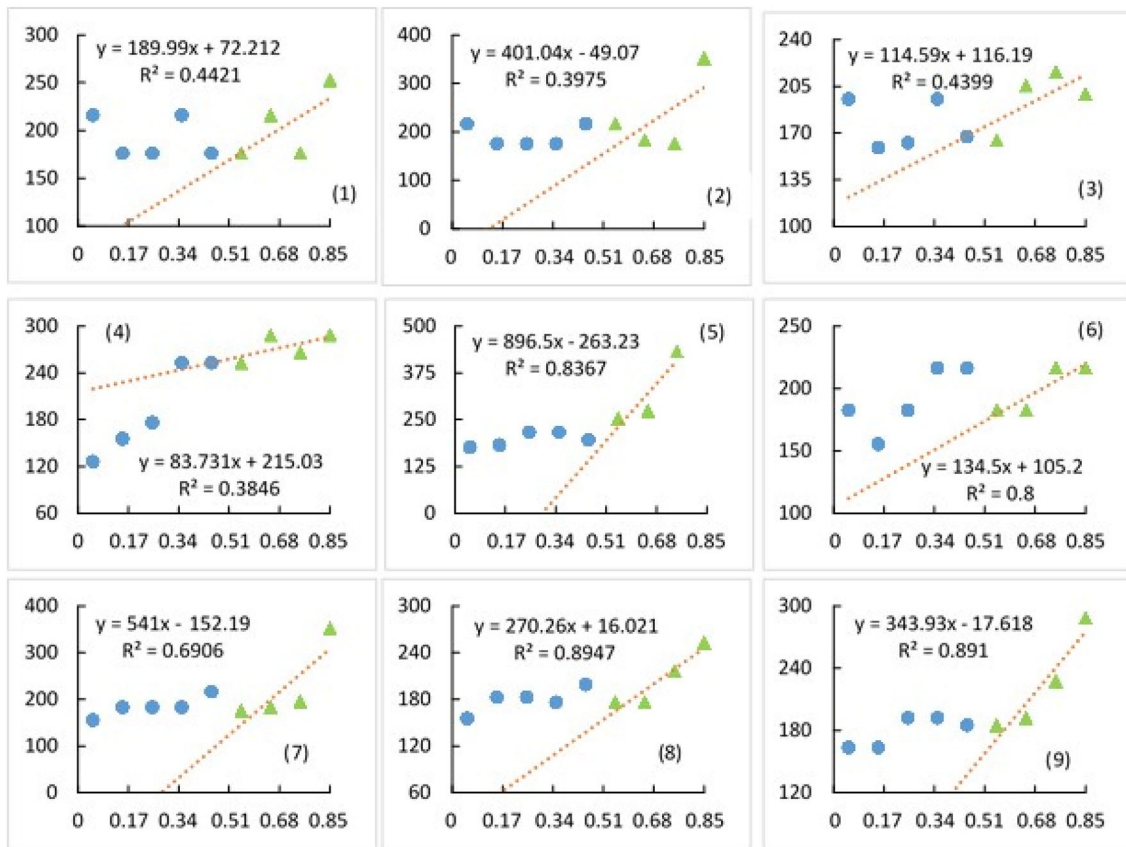
Fig. 5 Curves of activation energy (E_a) versus conversion degree (α) obtained by FWO method: sessile oak (1), beech (2), hornbeam (3), black locust (4), lime (5), ash (6), maple(7), alder (8), aspen (9); X-axis is the conversion degree (α); Y-axis is the activation energy (E_a, kJ mol^{-1}); black circle: predominant change in the degree of holocellulose conversion, black triangle: change in the degree of residual lignin and formed char conversion

variation was 4.04% (Fig. 6). The values E_a obtained in this study are in agreement with results of López et al. (2013) and Maryandyshev et al. (2015) who applied FOW isoconversional model to evaluate kinetic parameters for the combustion of lignocellulosic biomass and woods.

According to the differences between the minimum and the maximum of E_a at $0.05 \leq \alpha < 0.50$, tree species can be divided into two groups: (1) poplar, oak, beech, lime, hornbeam, and alder ($29 \div 44$ kJ mol^{-1}); (2) black locust, ash, maple ($60 \div 127$ kJ mol^{-1}) (Fig. 5). It indicates that black locust, ash, maple have more heterogeneous composition and structure of carbohydrate complex of wood.

At the stage of residual lignin and char thermoxidation ($E_{a(average)}$ 231.17 kJ mol^{-1}, standard deviation 40.71 kJ mol^{-1}; $0.45 < \alpha \leq 0.85$), the coefficient of variation of the average activation energy among all tree species was significantly higher (17.61%) than the one for the thermal degradation of holocellulose. It can be noticed from Fig. 5 that value of activation energy has tendency to increase at the end of thermoxidation. It was found that E_a correlated well with the degree of conversion in this range of
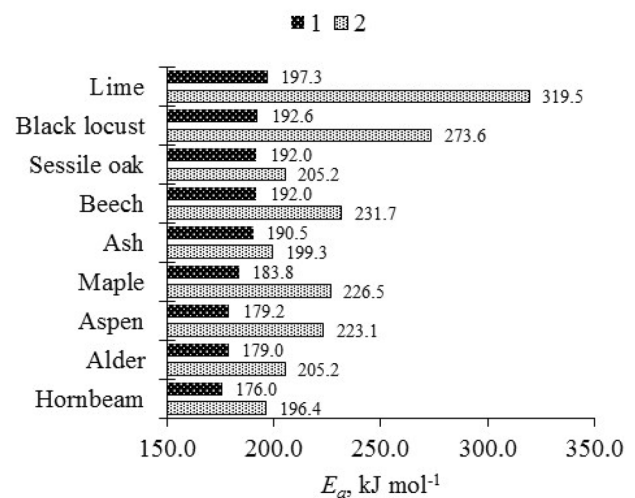

Fig. 6 Average values of activation energy for the thermal decomposition of carbohydrate complex (1) and thermoxidation of residual lignin and char (2)

the thermal degradation for some wood specimens (lime, ash, maple, alder, and aspen). This can be explained by the increasing density of molecular packing of the decomposing sample residue.

4 Conclusion

In this study, thermogravimetry and differential scanning calorimetry were used to determine the parameters and kinetics of the thermal degradation of nine hardwoods in an oxidative (air) atmosphere. The results showed that the differences found in the temperature of DTG/DSC peaks, the mass loss, and the maximum mass loss rate of main wood components (hemicelluloses, cellulose, lignin) as well as the values of exothermic effect and activation energy allow to differentiate tree species in terms of wood thermal stability. The obtained results can be used to develop modes of wood drying, thermal modification, and pellet manufacturing.

Declarations

Conflict of interest The authors declare that there is no conflict of interest.

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