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# Chemical and Physical Properties of Pine Wood during Pyrolysis

## Kemijska i fizikalna svojstva borovine tijekom pirolize

Original scientific paper • Izvorni znanstveni rad

Received – prispljelo: 17. 3. 2016.

Accepted – prihvaćeno: 15. 11. 2016.

UDK: 630\*812.26; 630\*813.4; 674.032.475.4

doi:10.5552/drind.2017.1617

**ABSTRACT** • This paper examines the influence of temperature on several physical properties of wood. The results demonstrated that devolatilization during pyrolysis greatly affects the inner structure of wood, followed by changes in volume, mass, density and shape. The research showed that a decrease in wood density caused a significant increase in permeability. Data on pyrolytic gases, char composition and calorific value measurements of these products are also presented. The results showed that the lower heating value (LHV) of pyrolytic gas can reach 12 MJ/Nm<sup>3</sup> at 600 °C and the higher heating value (HHV) of char can reach 34 MJ/kg for 700 °C. The analysis of the calorific values of pyrolytic gases and char showed that the thermal conversion of biomass can lead to the generation of good quality gaseous and solid fuels.

**Key words:** pyrolysis, pine wood, wood permeability, wood density, pyrolysis gas, thermogravimetric analysis

**SAŽETAK** • U radu se razmatra utjecaj temperature na neka fizikalna svojstva drva. Rezultati su pokazali da isplinjavanje tijekom pirolize uvelike utječe na unutarnju strukturu drva te na promjene njegova volumena, mase, gustoće i oblika. Analizom rezultata istraživanja može se zaključiti da smanjenje gustoće drva uzrokuje znatno povećanje propusnosti drva. U radu se prikazuju i podaci o pirolitičkim plinovima, sastavu ugljena i mjerenu njihovih kalorijskih vrijednosti. Rezultati su pokazali da donja ogrjevna vrijednost (LHV) pirolitičkog plina može dosegnuti 12 MJ/Nm<sup>3</sup> pri 600 °C, a gornja se ogrjevna vrijednost (HHV) ugljena kreće i do 34 MJ/kg pri 700 °C. Analiza kalorijskih vrijednosti pirolitičkih plinova i ugljena pokazala je da se toplinskom pretvorbom biomase mogu proizvesti kvalitetna plinovita i kruta goriva.

**Ključne riječi:** piroliza, drvo bora, propusnost drva, gustoća drva, pirolitički plin, termogravimetrijska analiza

### 1 INTRODUCTION

#### 1. UVOD

Biomass, such as pine wood, is a very common and generally accessible source of renewable energy. Traditionally, biomass has been utilized by direct combustion; however, the development of a biomass thermal conversion technology represents a promising alternative for energy production. Detailed reviews of other renewable energy solutions, such as hydrogenation,

pyrolysis and gasification, can be found in Demirbas (2001) and McKendry (2002). Biomass processing is already a very popular topic in global research; many studies with different types of wood can be found in the literature (Qiang *et al.*, 2011; Gupta *et al.*, 2002; Bridgwater and Peacocke, 2000; Park *et al.*, 2010, Bellais *et al.*, 2003; Luo and Resende, 2014, Kim *et al.*, 2014). In addition, different temperature ranges and pyrolysis conditions have been studied. For example, Amutio *et al.* (2012) tested pinewood sawdust flash pyrolysis (con-

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tinuous mode) in a pilot plant with a conical spouted bed reactor in the narrow range from 400 °C to 600 °C. Martinez *et al.* (2014) tested co-pyrolysis of biomass with waste tires from 150 °C to 700 °C. Further, the influence of various heating rates during pyrolysis on the shape of pine wood (degree of shrinkage or swelling) was investigated by Patanottai *et al.* (2015).

During the pyrolysis process, the physical properties of wood change significantly. Changes of wood components, such as lignin, after thermal treatment were tested by Windeisen *et al.* (2008). Analysis of the effect of temperature on basic physical properties of wood, such as elementary composition, density, mass, specific heat and the calorific values of pyrolysis products, are numerous (Pattanotai *et al.*, 2014; Neves *et al.*, 2011; Kardaś *et al.*, 2014, Kluska, 2015).

Permeability and density of wood also change significantly. Measurements of permeability for different types of wood are available, but only for wood at room temperature (Tanaka *et al.*, 2015; Redman *et al.*, 2012; Jinman *et al.*, 1991). Therefore, the permeability of wood, as a function of pyrolysis temperature, is not yet well understood. Similarly, analyses of the change in wood density during pyrolysis can be found in the reports of Brewer (2014) and Sommerville (2015). Since the numerical prediction of the pyrolysis process has also been intensively studied (Peters and Bruch, 2003), it is important to determine how the above parameters change with temperature for setting the boundaries of simulations and successfully predicting the outcome of the process. The change in the physical properties of wood during pyrolysis has an influence on the specific heat of wood (Gupta *et al.*, 2002; Gronli, 1996; Harada *et al.*, 1998).

This work thoroughly describes one type of biomass (pine wood) and provides detailed information on the pyrolysis process and the characteristics of its gaseous and solid products.

## 2 MATERIALS AND METHODS

### 2. MATERIJALI I METODE

#### 2.1 Wood samples

##### 2.1. Uzorci drva

Samples used in this study were prepared from Scots pine (*Pinus sylvestris*), which is a very common type of wood in Poland. Although different analysis methods and equipment required different size and mass of wood samples, each one of them originated from the same cylindrical wood bar of 16 mm in diameter. In order to prepare the samples for elementary analysis, pine wood was ground in a knife mill and then in a centrifugal mill equipped with a sieve of 0.2 mm mesh diameter.

#### 2.2 Pyrolysis

##### 2.2. Piroliza

The high temperature reactor (Figure 1 Pyrolysis test stand) heats the core of the device to 900 °C and maintains that temperature to enable the thermal conversion of the biomass and waste materials.

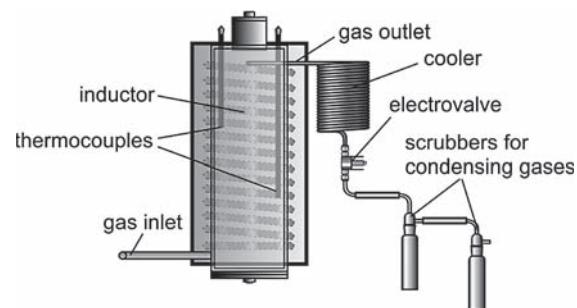


Figure 1 Pyrolysis test stand

Slika 1. Uredaj za pirolizu

The chamber is cylindrical with a 98 mm inner diameter. The active portion of the chamber, enveloped with a heating induction coil and thermal insulation, is located in the center of the device. It is equipped with ungrounded thermocouples fixed at 1/3 and 2/3 of the total height of the cylinder. Such an arrangement enables measuring and recording temperatures inside the reactor and inside the bed of the tested feed in real time during the experiment. The device was also equipped with a blow-through electrovalve to let in the process gas (argon) as well as a bleeder electrovalve to remove it. Argon is used in order to remove the air from the device before the process. The reactor works in a constant sweep of gas mode. The experiment was conducted on a group of 20 samples, from 20 °C to 900 °C.

#### 2.3 Permeability measurement

##### 2.3. Mjerenje propusnosti

###### 2.3.1 Test stand

###### 2.3.1. Ispitni uređaj

The test stand for measuring permeability consisted of the following devices: air compressor, differential pressure converter, steel container equipped with interchangeable steel sleeve (containing the biomass) and a bubble velocity meter (Figure 2 Permeability test stand). The upper part of the container is attached with screws and can be removed to place the interchangeable steel sleeve inside the container between two circular seals. This type of construction allows for the quick and easy replacement of biomass samples.

###### 2.3.2 Sample preparation

###### 2.3.2. Priprema uzoraka

At the beginning of the sample preparation process, each sample was of a cylindrical shape with a 16 mm diameter and was approximately 6 cm in height. A group of three samples was inserted into the stove and

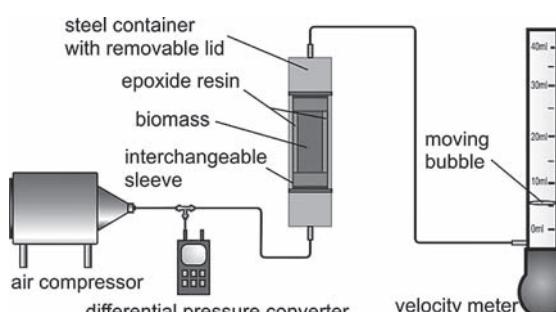


Figure 2 Permeability test stand

Slika 2. Ispitni uredaj za mjerjenje propusnosti

heated to a given temperature in an oxygen-free atmosphere (the temperature was different for each group, from 50 °C up to 350 °C, at 50 °C intervals). After heating, the samples were removed from the stove and allowed to cool to room temperature. Each sample was inserted into the steel sleeve and fixed to it with epoxide resin (the samples were covered with varnish prior to this procedure to prevent the epoxide resin from percolating into the wood).

Dimensions of the samples varied as a result of the pyrolytic decomposition. Using a milling machine, each sample was leveled and adjusted so that each measurement was conducted on a 30 mm tall biomass cylinder. Different diameters (due to shrinkage during pyrolysis) were taken into consideration in the calculations.

### 2.3.3 Methodology and measurements

#### 2.3.3. Metodologija i mjerena

The method is based on blowing air (under pressure) into the container along the axial direction of the wood cylinder (which also is the direction of the wood grain) and measuring the air velocity after it crosses the wood cylinder.

The measurement of the wood permeability to air was indirect. It was calculated from Darcy's law:

$$K = \frac{u \cdot \mu \cdot l}{\Delta P} \quad (1)$$

Where  $\Delta P$  is pressure difference,  $K$  permeability,  $\mu$  is dynamic viscosity of the air,  $l$  is length (cylinder height), and  $u$  is velocity of the air flowing through porous media (in this case wood). The pressure difference was measured during the flow of the compressed air using a differential pressure converter (Figure 2 Permeability test stand). The velocity of the flow through wood cannot be measured directly; therefore, it was calculated based on the Law of Continuity, by measuring the time that it takes for a bubble to pass through the given volume of the velocity meter (constant and equal to 40 cm<sup>3</sup>). For each measurement, the pressure value was set independently to obtain a measurable bubble velocity.

## 3 RESULTS AND DISCUSSION

### 3. REZULTATI I RASPRAVA

#### 3.1 Wood sample elementary analysis

##### 3.1. Elementarna analiza uzorka drva

The results of the technical and elementary analyses of pine wood are shown in Table 1 Technical and elementary analysis of biomass. The moisture content was determined using a MAC (Radwag) moisture balance and an elementary analysis was performed using a Thermo Scientific CHNS-O Flash 2000 elementary analyzer. The calorific value and calculation of the heating value were determined using a KL-11 calorimeter. The presented results agree with other published data (Neves *et al.*, 2011; Alauddin *et al.*, 2010; Yıldız *et al.*, 2015).

#### 3.2 TGA analysis of pine wood

##### 3.2. TGA analiza borovine

To analyze the thermal conversion rate and the temperatures at which they occur, a thermogravimetric

**Table 1** Technical and elementary analysis of biomass  
**Tablica 1.** Tehnička i elementarna analiza biomase

<b>Heating value, MJ/kg</b> <i>Ogrjevna vrijednost, MJ/kg</i>	19.59
<b>Technical / tehnička analiza</b>	%
Moisture / sadržaj vode	8.40
Volatiles / hlapljivi spojevi	67.90
Char / ugljen	21.40
Ash / pepeo	2.30
<b>Elementary / elementarna analiza</b>	%
C	45.00
H	6.40
O	47.30
N	1.30

analysis of the biomass samples was conducted. The results of the thermal decomposition of the biomass presented in this study were obtained by using the TA Instruments SDTQ600 thermogravimeter at different heating rates of 5 °C/min, 10 °C/min and 15 °C/min. The mean mass of the sample was 10 mg.

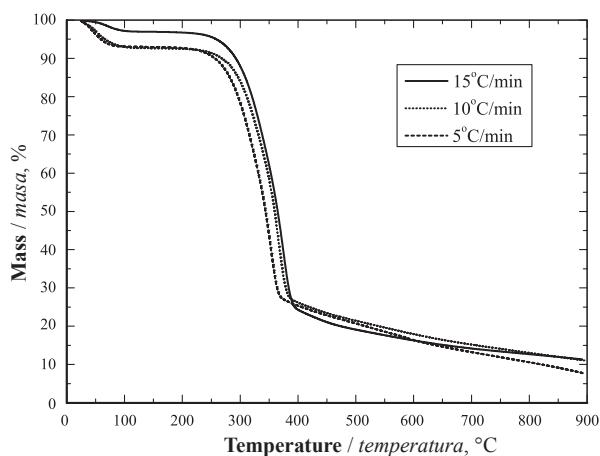
The beginning of the devolatilization process occurs at 180 °C to 190 °C (Figure 3). The highest intensity devolatilization occurs at different temperatures for different heating rates (345 °C for the heating rate of 5 °C/min, 367 °C for 10 °C/min and 373 °C for 15 °C/min). Moreover, increase of the heating rate moves the thermogravimetric curve in the direction of higher temperatures. The obtained data is in good accordance with the literature (Alvarez *et al.*, 2015).

#### 3.3 Pyrolytic gas analysis

##### 3.3. Analiza pirolitičkih plinova

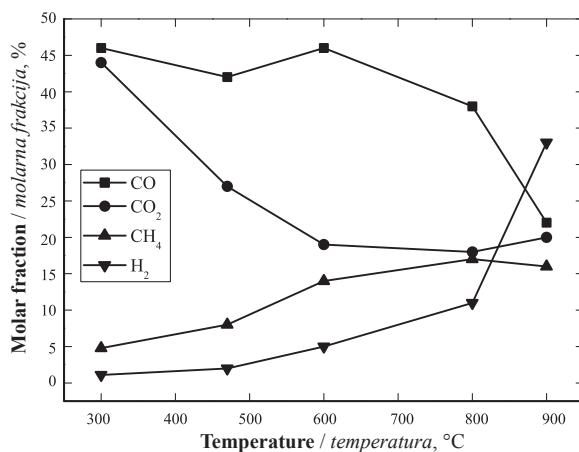
The pyrolytic gas analysis was conducted on SRI Instruments 310 gas chromatograph with ShinCarbon ST 80-100 packed column, thermal conductivity detector (TCD) and argon as a carrying medium.

The results of the pine wood pyrolysis (Figure 4) showed that, with an increase of the temperature to 600 °C, the CO content in the pyrolysis gases maintained a more or less constant value of 45 %, but decreased to 20 % at 900 °C. The content of CO<sub>2</sub> in the pyrolysis gas mixture decreased from 44 % at 300 °C to 27 % at 470 °C. After reaching 900 °C, the CO<sub>2</sub> content decreased



**Figure 3** TGA analysis of pine wood

**Slika 3.** TGA analiza borovine

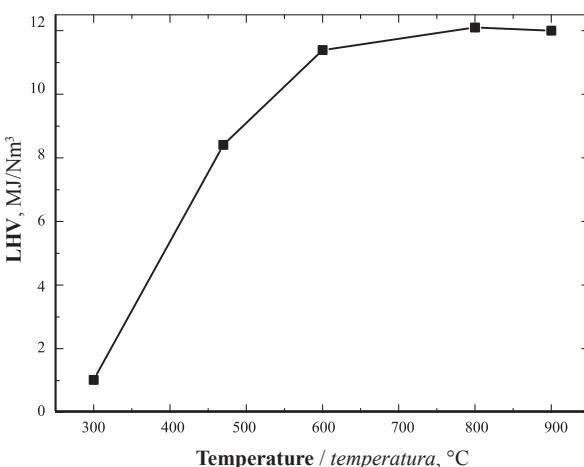


**Figure 4** Influence of temperature on gas composition during pine wood pyrolysis

**Slika 4.** Utjecaj temperature na sastav plina tijekom pirolize borovine

even further to 20 %. The gas composition at 600 °C was very similar to the composition obtained by Amutto *et al.* (2012). Also, the results in the range of temperatures from 600 °C to 900 °C are in agreement with the work of Ningbo *et al.* (2015) as well as Song *et al.* (2014). We observed that high temperatures (above 600 °C) caused an intensive hydrogen release. In the final stage of the process, (900 °C), the content of H<sub>2</sub> was equal to 33 %. Additionally, the results showed that, with increasing temperatures (up to 700 °C), the methane content reached 16 % and then maintained a value of 15 %. Based on the analysis of the experimental results, it can be concluded that high temperatures move the pyrolysis process towards a gas composition with a high hydrogen content. The results also showed a high carbon oxide content in the range of temperatures from 400 °C to 800 °C (37 % to 45 %).

The results of the analysis of gas composition as a function of temperature in the pyrolysis process showed that the LHV depends on the temperature. The LHV of the pyrolytic gas mixture reached 8 MJ/Nm<sup>3</sup> at 500 °C and then increased to 12 MJ/Nm<sup>3</sup> (Figure 5 Lower heating value of pyrolytic gases as a function of tempera-



**Figure 5** Lower heating value of pyrolytic gases as a function of temperature

**Slika 5.** Donja ogrjevna vrijednost pirolitičkih plinova u ovisnosti o temperaturi

ture5). These results are similar to other published data (Bridgwater and Peacocke, 2000; Neves *et al.*, 2011; Fabgemi *et al.*, 2011). At the lower range of temperatures, the LHV of the gases was determined by the carbon monoxide content (up to 45 % at 600 °C). The higher LHVs at higher temperatures are related to the high content of hydrogen and methane in the mixture.

### 3.4 Cylindrical chip mass, volume and density changes during pyrolysis

#### 3.4. Promjene mase, obujma i gustoće cilindričnih uzoraka borovine tijekom pirolize

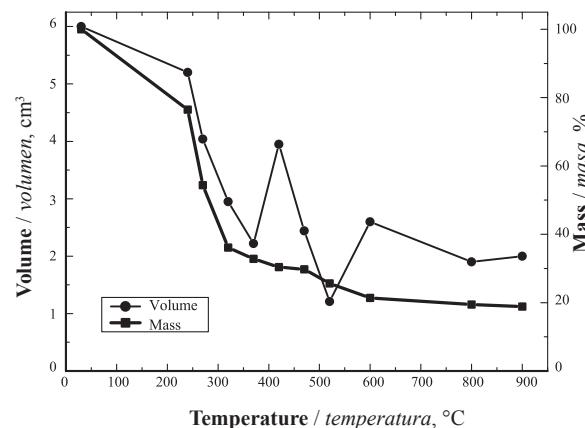
The results show a general decrease of the volume of wood cylinders with increasing temperatures (Figure 6). After decreasing to 2.22 cm<sup>3</sup> at 370 °C, an increase in the volume to 3.95 cm<sup>3</sup> at 420 °C is observed, followed by another decrease to 0.12 cm<sup>3</sup> at 520 °C. For higher temperatures, the volume decreases slowly from 2.6 cm<sup>3</sup> at 600 °C to 2 cm<sup>3</sup> at 900 °C. These fluctuations of volume are the result of difference in the radial shrinkage and swelling of the samples for these temperatures caused by the internal pressure during pyrolysis (Pattanotai *et al.*, 2014; Park *et al.*, 2010). The internal pressure in the cylindrical wood samples during pyrolysis at a high heating rate is higher than in the low rate of heating and helps to resist wood shrinkage in the radial direction (Pattanotai *et al.*, 2014). In the case of wood particles, the internal pressure produced is an important factor for the pyrolysis process. The pressure gradient causes the release of volatile components outside the particles. Moreover, high internal pressure can also partially crack the particles.

The largest loss of mass and volume, as a function of pyrolysis temperature, was observed in the range of temperatures from 240 °C to 370 °C (Figure 6). The final mass at 900 °C was 19 % of the initial mass of the pine wood cylinder samples, and the final volume at the same temperature was 30 %.

### 3.5 Elementary composition and LHV of char

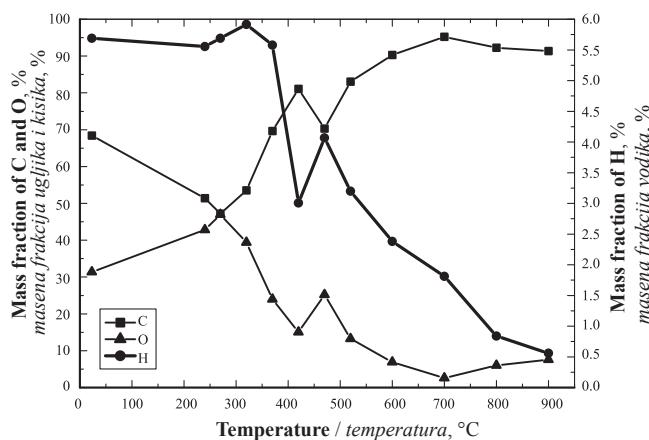
#### 3.5. Elementarni sastav i donja ogrjevna vrijednost ugljena

The measurements were conducted in a CHNS-O Flash 2000 elementary analyzer, while the calorific



**Figure 6** Pine wood mass and volume dependence on pyrolysis temperature

**Slika 6.** Ovisnost mase i obujma uzorka borovine o temperaturi pirolize



**Figure 7** The influence of pyrolysis temperature on char composition

**Slika 7.** Utjecaj temperature pirolize na sastav ugljena

value and calculation of the heating value were determined using a KL-11 calorimeter.

Figure 7 shows how the char composition changes as a function of pyrolysis temperature. The carbon mass fraction increases with temperature, while the oxygen and hydrogen mass fractions decrease. The largest increase in carbon content, along with the largest decrease in oxygen content, occurred for the temperature range of 270 °C to 400 °C. This is because the wood devolatilization is the most intense within this range (Figure 3). Fluctuations of elementary composition of char (Figure 7) in the range of temperatures from 400 °C to 500 °C can be caused by irregular heating rate of particles during pyrolysis of packed bed. The bed consisted of 20 cylindrical samples. The heat could propagate with different rate depending on the sample location in the bed. Thus, at the temperature range representing the most intensive devolatilization (Figure 3), different rates of heating have a significant importance and may affect the elementary composition of char.

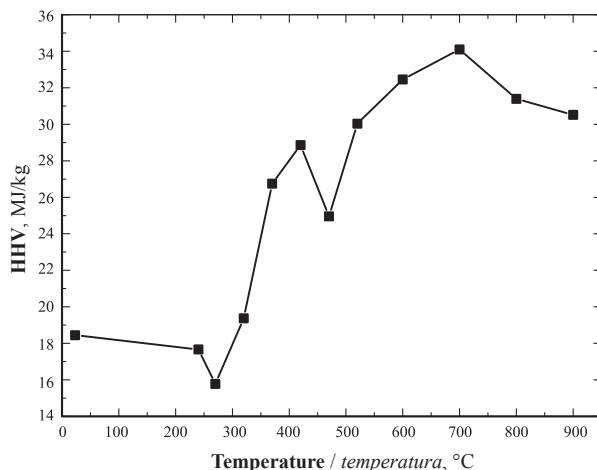
Due to wood volatiles leaving the solid particles, the mass fractions of carbon and oxygen significantly

changed. In contrast, the hydrogen mass fraction, although small (5.5 % - 6 %), was relatively constant at lower temperatures and began to decrease at temperatures higher than 370 °C, down to 0.5 % at 900 °C. The obtained results agree with other published data (Neves *et al.*, 2011; Fabgemi *et al.*, 2011, Özbay *et al.*, 2015).

Figure 8 shows the dependence of the HHV of char on pyrolysis temperature. As a natural result of the rapid carbon and oxygen content changes from 270 °C to 400 °C, the HHV of the char also rapidly changes at these temperatures. The initial HHV of char was 18.2 MJ/kg, and after the devolatilization process this value increased by nearly 60 % (up to 29 MJ/kg). The small decreases of HHV at 470 °C and from 700 °C to 900 °C are caused by the decreases of mass fraction of C and increases of mass fraction of O at the same temperatures (Figure 7). The char obtained during the pyrolysis process can increase in the HHV by a total of 87 % (at 700 °C) compared to the initial value. The obtained results are in a good accordance with the model (Figure 8) proposed by Channiwala *et al.*, (2002) as well as with other literature (Zhengang and Guanghua, 2015).

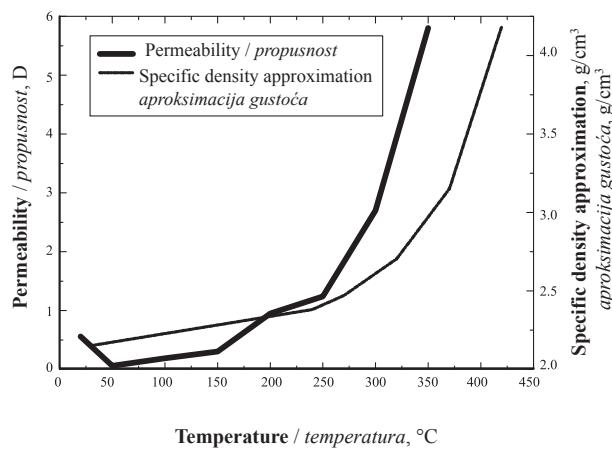
### 3.6 Permeability to air 3.6. Propusnost zraka

The permeability measurements were performed at room temperature for samples obtained at different pyrolysis temperatures (from 22 °C up to 350 °C) along the axial direction of the wood cylinder. Samples obtained at temperatures higher than 350 °C were cracked or too fragile to perform measurements of permeability. The pyrolysis caused irreversible changes in the wood structure and even after cooling the wood to room temperature, the permeability to air was different than before the process. The dependence of the wood permeability on the pyrolysis temperature is complex. From 50 °C to 150 °C, the permeability was smaller than for wood at room temperature. One possible explanation for this is that the wood resin blocks the pores at that range of temperatures, making it difficult for air to pass through (Figure 10). The inner structure of pine wood contains many long resin conduits along the trunk and branches, which are also connected with transverse conduits. The main component of the resin is a class of naturally occurring organic chemicals called terpenoids (among other compounds such as stilbenes, lignans, extractives). The melting point of these substances occurs at temperatures from 100 °C to 130 °C, and the vaporization temperatures are from 156 °C to 224 °C (Connelly, 1991). These values suggest that from 50 °C to 150 °C the resin movement caused by the heating process blocks the pores, and when the wood is cooled to room temperature the pores remained blocked (thus a decrease in permeability is observed). From 200 °C to 350 °C a large increase (up to ten times the initial value) in permeability is observed. This is caused by the loss of wood mass and by the increased size of its pores as a result of devolatilization. Since the resin components begin to evaporate at 156 °C, this may also contribute to an increase in permeability. Using the data presented in Figure 6, an approximation curve of wood



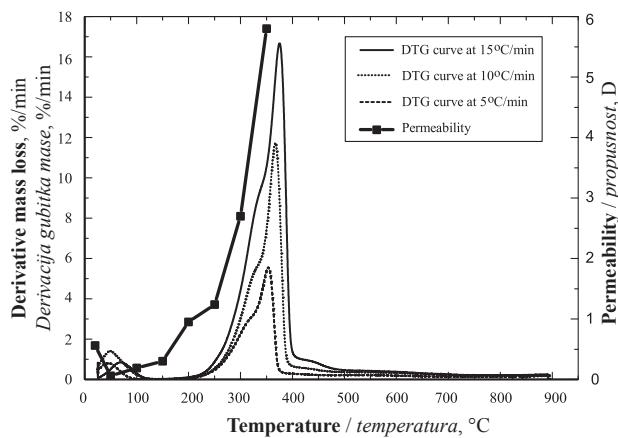
**Figure 8** The influence of pyrolysis temperature on char higher heating value

**Slika 8.** Utjecaj temperature pirolize na gornju ogrjevnu vrijednost ugljena



**Figure 9** Characteristics of wood permeability and reverse approximation of density

Slika 9. Obilježja propusnosti drva i aproksimacija gustoće drva



**Figure 10** DTG and permeability curves for pine wood thermal decomposition

Slika 10. DTG i krivulje propusnosti za toplinsku razgradnju borovine

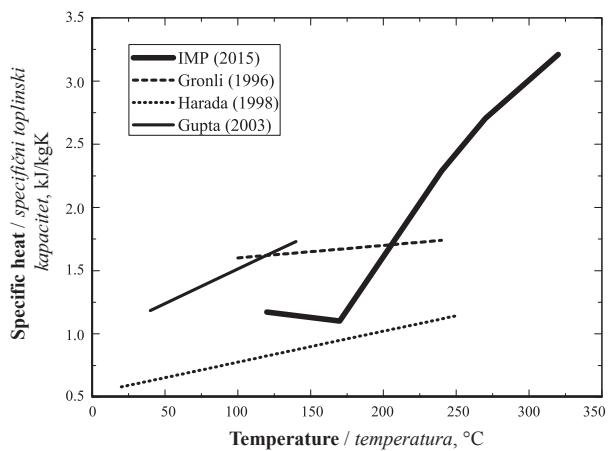
apparent density can be created, which can be further used to calculate reversed approximation curve. Figure 9 also shows that the permeability of wood is in accordance with the reversed approximation of apparent density in the range of temperatures from 25 °C to 350 °C. This means that the decrease of wood density may cause an increase in permeability.

Figure 10 shows DTG curves for thermal decomposition of pine wood at heating rates of 5, 10 and 15 °C/min as well as permeability curve. Dramatic increase in permeability occurs for the same range of temperatures (starting at 250 °C) as the most intensive degradation kinetics. The DTG curve for 15 °C/min is in good accordance with literature data (Alvarez *et al.*, 2015).

### 3.7 Specific heat

#### 3.7. Specifična toplina

The measurements of the specific heat of wood were conducted in a two-stage process. During the first stage, small wood samples were treated with temperatures from 70 °C to 320 °C inside a TA Instruments Q600 Thermogravimeter. The samples were heated up to the specified temperature at a rate of 20 °C/min in a nitrogen atmosphere and maintained at that tempera-



**Figure 11** The influence of temperature on wood specific heat

Slika 11. Utjecaj temperature na specifičnu toplinu drva

ture for 2 hours to reduce the moisture content. The second stage consisted of measuring the heat capacity of wood using a TA Instruments Q2000 Differential Scanning Calorimeter (DSC).

Each sample was closed inside a hermetic sample cell and heated to a temperature five degrees lower than corresponding temperature of that sample during the previous TGA pre-heating to avoid any chemical decomposition during measurement. The process was conducted with a heating rate of 5 °C/min in a nitrogen atmosphere. The average mass of wood samples during the DSC measurements was 3.35 mg.

The results are presented in Figure 11. From 120 °C to 170 °C, the specific heat was relatively constant at 1200 kJ/kgK, and for higher temperatures it rapidly increased to 3250 kJ/kgK. Gronli (1996) studied the specific heat of wood in the narrow range from 100 °C to 250 °C and Gupta *et al.* (2003) studied the specific heat of wood from 40 °C to 150 °C. The results of Gronli and Gupta show values 40 % - 50 % higher compared to the results obtained by the Institute of Fluid Flow Machinery PAS (IMP in Figure 11). Harada *et al.* (1998) tested the specific heat of wood from 20 °C to 250 °C and reported lower values. The specific heat of wood increases with temperature and the values obtained at the IMP are in agreement with the available literature.

## 4 CONCLUSION

### 4. ZAKLJUČAK

This paper provides information on pyrolytic gas properties as well as physical properties of pine wood such as permeability, density and specific heat and their interrelation over a wide range of temperatures. Pyrolysis causes a significant reduction in wood density, due to mass and volume changes. It was demonstrated that when the density of wood decreases, an increase in its permeability to gases is observed for the same range of temperatures. Permeability generally increases with temperature; however, from 50 °C to 150 °C, it reaches values lower than at room temperature. The density and volume of wood decreases with temperature, but from 350 °C to 500 °C some fluctuations were observed, es-

pecially in density. The LHV of pyrolytic gases reached 12 MJ/Nm<sup>3</sup> at 600 °C, while the HHV of char reached a maximum value of 34 MJ/kg at 700 °C. A large increase in the HHV of char, which was observed from 250 °C to 300 °C, is the result of the large increase in carbon content for the same range of temperatures. Thermal conversion of biomass can lead to the generation of good quality gaseous and solid fuels.

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