

Original scientific paper

Received: 24.04.2024

Accepted: 10.12.2024

UDC: 662.63.04:620.925

LIGNOCELLULOSE COMPOSITION, PROXIMATE ANALYSIS, AND HEAT VALUE OF CERTAIN FOREST AND ENERGY CROP BIOMASSES AND THEIR POTENTIAL AS RAW MATERIALS FOR THE PRODUCTION OF SOLID BIOFUELS

Božidar Matin¹, Ana Matin², Ivan Brandi², Alen urovi¹, Josip Ištvanj¹, Alan Antonovi¹

¹*University of Zagreb, Faculty of Forestry and Wood Technology, Zagreb, Croatia,
e-mail: bmatin@sumfak.hr; adurovic@sumfak.hr; jistvanic@sumfak.hr aantonovic@sumfak.hr;*

²*University of Zagreb, Faculty of Agriculture, Zagreb, Croatia,
e-mail: amatin@agr.hr; ibrandic@agr.hr*

ABSTRACT

Biomass as a raw material is available in large quantities and is inexpensive. Its renewable energy can reduce greenhouse gas emissions, and it depends primarily on the composition of lignocellulose, especially the lignin content. Forest biomass of oak and beech, as well as biomass of the energy crops switchgrass (*Panicum virgatum* L.) and *Miscanthus x giganteus*, were used for this study. The aim of the study was to determine the lignocellulosic composition (proportions of cellulose, lignin, and hemicellulose), proximate analysis (proportions of moisture, ash, coke, fixed carbon, and volatiles), and calorific value of the studied biomasses, as well as to examine the possibility of their use as raw materials for the production of solid biofuels. The research showed that both forest biomass and biomass of energy crops have favourable values of the studied parameters, which is best reflected in the excellent calorific value, ranging from 17.0 to 18.5 MJ kg⁻¹. It was also found that the studied samples are ideal raw materials for the production of solid biofuel with lignin content between 20.0 and 30.0%.

Keywords: forest biomass, energy crops, lignocellulose, proximate analysis, calorific value, solid biofuel

1. INTRODUCTION

The burning of fossil fuels releases large amounts of CO₂ and other greenhouse gases (GHGs) into the atmosphere. These excessive GHG emissions lead to adverse climate changes that have recently manifested themselves in the form of droughts, rainfall accompanied by heavy storms, floods, etc., as well as the depletion of non-renewable energy sources (McKendry, 2002).

In the production of primary energy in 2020, fossil fuels had a share of 80% (oil 29.0%, coal 27.0%, natural gas 24.0%), while the share of renewable energy was 15.0% (WBA, 2022). All this drew the attention of mankind to the use of lignocellulosic biomass, which could replace fossil fuels thanks to its energy potential (Bilandžija et al., 2016).

Lignocellulosic biomass, as a source of organic material in which solar energy is stored through the process of photosynthesis, is becoming an increasingly important component of society and the economy itself. Biomass releases stored energy when the bonds between molecules are broken, whether through a decomposition, digestion, or combustion process. Burning biomass also releases certain amounts of CO₂ into the atmosphere; however, replanting plants ensures that these amounts are absorbed through their new growth cycle (McKendry, 2002).

In 2020, the global biomass supply was 57.5 exajoules (10¹⁸ joules), with 86.0% of the total supply coming from solid biomass such as wood chips, wood pellets, and other traditional biomass

sources; 7.0% from liquid biofuels; 3.0% from municipal waste; 2.0% from industrial waste; and 2.0% from biogas (WBA, 2022).

For agricultural biomass, which includes energy crops, there is almost no systematic data on available quantities. The amount of available agricultural biomass is mainly determined by empirical models through various deductions from total agricultural production, rarely taking into account variables that occur due to varietal differences, agrotechnical measures, or agroclimatic conditions, and the calculation is also affected by the lack of data on the method of collection and use of agricultural residues (Camia et al., 2018).

In recent decades, research has focused on fuel properties and the technology of processing, refining, and using biomass for energy production (Saleem, 2022). Biomass-derived renewable energy, i.e., biofuel, can be divided into primary and secondary energy. Primary fuels include firewood, wood chips, wood pellets, and post-harvest residues, which are mainly used in heating systems (Rodionova et al., 2017), while secondary biofuels include solid (coal), liquid (biodiesel, bioethanol), and gaseous (hydrogen, biogas) fuels as derivatives of the primary fuels. Liquid and gaseous biofuels can also be produced by processing biomass and used as an energy source in certain industrial processes or transportation (Doshi et al., 2016). Secondary biofuels are divided into four generations: the first and second generations are based on primary biomass sources (Azad et al., 2015), the third on microalgae (Chew et al., 2018), and the fourth on genetically modified microalgae (Zhu et al., 2017). It has become clear that the first two generations will not be sufficient to meet growing energy demand, and therefore more work will be needed to develop third and fourth generation biofuels (Osman et al., 2021).

Recently, lignocellulosic biomass of forest or agricultural origin has gained importance as a feedstock for energy production (Arteaga-Perez et al., 2015). Lignocellulosic biomass is a valuable renewable feedstock source that can be used directly or indirectly to produce bioproducts through chemical, physical, enzymatic, or microbial processes in the energy and industrial sectors (heat and/or electricity) and in the chemical industry (chemicals, adhesives) (Guo, Sun, Grebner, 2010; Rodriguez and Espinosa, 2021).

Forest biomass remains the most important feedstock source for solid biofuel production. However, new European Union legislation to prevent excessive deforestation and use of forest biomass requires a change in biomass sources, which is expected to increase the share of biomass use from agriculture and the agri-food industry (Grzybek, 2008; Budzy ski, Szczukowski, Tworkowski, 2009). The agricultural sector contributes about 10.0% of the global biomass supply, but there is significant potential for increasing its contribution. For this reason, agriculture could be a key sector for bioenergy use in the future, especially for energy crops (WBA, 2022).

Various studies show that energy crops are an environmental and economic option for sustainable energy production. Energy crops can be annual or perennial and grown on poorer quality soils, tolerate moisture deficiencies well, and can provide high biomass yields. These energy crops include switchgrass and *Miscanthus x giganteus*, which have already been researched (Koçar and Civas, 2013; Bilandžija et al., 2014). However, biomass as a fuel also has disadvantages such as irregular shape, large volume, low bulk density (up to 100 kg/m³ in agriculture, up to 200 kg/m³ in forestry), uneven combustion due to different moisture content, and lower calorific value compared to fossil fuels (Mitchell et al., 2007; Vassilev, Vassileva, Vassilev, 2015). The composition of the biomass itself can vary due to several variables, such as the type or part of the plant, the ability to absorb nutrients from soil, water, and sunlight during growth and their accumulation in plant tissues, the amount of artificial fertilisers and preservatives used, agroecological growing conditions, the timing and method of harvesting, the collection method and conditions during transportation and storage, differences in ash content, and the combination of different types of biomass (Vassilev et al., 2010).

It is necessary to know the physicochemical properties as well as the methods for analytical characterisation of these properties because they can significantly affect the conversion process as well as the supply chain network for raw materials and transportation (Cai et al., 2017). In addition, physicochemical properties serve as protection against infestation by pathogens and pests (Bhuiyan et al., 2009). The chemical composition of biomass is determined by several main components, namely polymers: cellulose, lignin, and hemicellulose (Williams, Emerson, Tumuluru, 2017), which provide structure and strength to plants (Sanderson, 2011), and their proportion and structure depend on the type of plant cell wall (Barakat, de Vries, Rouau, 2013). Cellulose and hemicellulose, together with

lignin, account for more than 90.0% of the composition of lignocellulosic biomass, and biomass with higher lignin content is more suitable for conversion into solid biofuels because it increases the calorific value (Li et al., 2003; Chen and Dixon, 2007). This is because lignin has an upper heating value (HHV) between 22.2 and 28.5 MJ kg⁻¹ (Demirbas, 2017), while the heating value of cellulose and hemicellulose is slightly lower at 18.6 MJ kg⁻¹ (Demirbas, 2001).

Proximate analysis includes non-combustible materials that do not ignite under normal conditions, even when exposed to elevated temperatures, and includes moisture, ash, coke, solid carbon, and volatiles.

Calorific value is a basic parameter for calculating the energy and potential of biomass, as well as a basic parameter for classifying the quality of the biofuel itself. The heating value is divided into the higher heating value (HHV) and the lower heating value (LHV). The HHV is the heat released during the combustion of the fuel, additionally utilising the condensation heat of the water vapour from the flue gases, i.e., it is the highest possible energy that can be obtained during the combustion of a fuel. The LHV is the heat released by the combustion process of the fuel without additional use of the condensation heat of the water vapour, and therefore the LHV is always lower than the HHV. For biomass, the difference between these values is about 7.0% on average (Lewandowski et al., 2003; Garcia et al., 2012).

The objective of this work was to determine the lignocellulosic composition, proximate analysis, and heating value of two forest species (oak and beech) and two agricultural energy crops (switchgrass and *Miscanthus x giganteus*). In addition, the possibility of their use for the production of solid biofuels such as pellets and briquettes was determined, depending to a large extent on the lignocellulosic composition itself and on the energy potential of each of the above biomasses.

2. MATERIALS AND METHODS

2.1 Raw material collection and sampling

Two agricultural energy crops (switchgrass and *Miscanthus*) were collected at the Šašínovec Experimental Station of the Faculty of Agriculture, University of Zagreb (N 45° 85' 01", E 16° 17' 67"). The sampled switchgrass and *Miscanthus* biomass was harvested in March 2022. Two forest species (oak and beech) were collected from company Bjelin Spa va d.o.o. in the town of Vinkovci, Croatia.

Chemical analyses were performed in the laboratory of the University of Zagreb, Faculty of Forestry and Wood Technology, and in the laboratory of the University of Zagreb, Faculty of Agriculture, according to standard methods. Prior to the chemical analyses, the biomass was dried for 48 hours using a laboratory dryer UF 160 Memmert (Mettler-Toledo GmbH, Germany) at a temperature of 60 °C in order to achieve equilibrium in the material so that the comparison of the samples could be performed under identical operating conditions.

2.2. Methods

After drying, the biomass was comminuted with a laboratory mill SM400 Retsch (Retsch GmbH, Germany) using a sieve with round openings 4.0 mm Retsch (HRN EN ISO 14780:2017) and then further comminuted with a rotating hammer mill SR300 Retsch (Retsch GmbH, Germany) using a sieve with trapezoidal openings 2.0 mm Retsch (HRN EN ISO 14780:2017). Each sample was analysed at least three times to ensure reproducibility of the analyses.

2.2.1 Lignocellulosic composition

In the analysis of lignocellulosic composition, the percentage of accessory substances (TAPPI 204 cm-2007) was determined using the Soxhlet R108S BEH Rotest extraction instrument (Behr, Labor-Technik GmbH, Germany), cellulose (a mixture of HNO₃ and CH₂OH) by boiling in a Hydro H9V Lauda water bath (Lauda GmbH, Germany), and lignin (TAPPI T 222-2002) by boiling on a magnetic stirrer with an IKA C-MAG HS 7 heater (IKA®-Werke GmbH & Co. KG Germany), while the percentage of hemicellulose was determined by calculation.

2.2.2 Proximate analysis

For proximate analysis, samples were characterised using standard methods: moisture content (HRN EN 18134-2:2017) was determined in a laboratory dryer UF 160 Memmert (MettlerGmbH, Germany), while ash content (HRN EN ISO 18122:2015), coke (HRN EN ISO 18123:2015), fixed carbon (HRN EN ISO 18123:2015), and volatile matter (HRN EN ISO 18123:2015) were determined using a Nabertherm L9/11/B170 muffle furnace (Nabertherm GmbH, Germany).

2.2.3 Heating value

HHV was determined according to the method (HRN EN ISO 18125:2017) using an IKA C 6000 adiabatic calorimeter (IKA® -Werke GmbH & Co. KG, Germany), while LHV was determined by calculation.

2.2.4 Statistical analysis

After analysis, results were evaluated using PROC MIXED from the SAS software package (SAS Institute Inc., SAS 9.1.2 Help and Documentation, Cary, NC: SAS Institute Inc., 2002-2004, Raleigh, NC, USA, North Carolina State College).

3. RESULTS AND DISCUSSION

3.1 Laboratory tests

The lignocellulose composition results are shown in Table 1, while proximate analysis results are shown in Table 2. Lignocellulosic composition and proximate analysis are considered some of the most important parameters in evaluating biomass as more or less suitable feedstock for use in direct combustion systems. Moisture content (MC), ash content (AC), and volatile matter (VM) are undesirable components of biomass, while higher fixed carbon content (FC) and coke content (CK) and higher heating values are desirable and improve the quality of biomass in direct combustion systems.

3.2 Lignocellulosic composition

Lignocellulosic biomass has cellulose content between 30.0 and 50.0%, 10.0 to 30.0% lignin, and 20.0 to 35.0% hemicellulose, while ash, oils, and proteins make up the rest (Saha, 2005). Forest biomass contains on average between 50.0 and 55.0% cellulose, 20.0 to 30.0% lignin, and between 15.0 and 30.0% hemicellulose (Antonovi , 2004). Agricultural biomass has an average cellulose content between 40.0 and 50.0%, 10.0 to 30.0% lignin, and between 20.0 and 30.0% hemicellulose (Malherbe and Cloete, 2002; Kumar et al., 2009; Iqbal et al., 2011). Comparing the average percentages of lignocellulosic composition of the studied forest species and agricultural energy crops listed in Table 1, it can be concluded that the obtained values are in agreement with the above studies of the mentioned authors.

Table 1: Lignocellulosic composition

Sample	Cellulose (%)	Lignin (%)	Hemicellulose (%)	Extractives (%)
Oak	50.39b±0.09	26.02b±0.29	21.98ab±0.29	1.59ab±0.36
Beech	45.78a±0.10	25.50b±0.22	23.61b±0.01	1.34a±0.29
Switchgrass	46.42a±0.52	21.34a±0.61	20.55a±0.05	3.81b±0.10
Miscanthus	47.85b±0.55	21.61a±0.42	22.89b±0.19	1.22a±0.06
Significance	***	***	***	***

Values in the column with the same letter are not statistically significantly different with $p < 0.05$. Statistical difference: *** $p < 0.001$, NS—not significant.

3.3 Proximate analysis

All examined proximate parameters showed a significant statistical difference. As shown in Table 2, MC of the studied samples ranged from 9.09% for oak to 14.02% for Miscanthus. AC was also lowest in oak at 0.35%, while the highest AC of 3.74% was recorded in switchgrass samples. Switchgrass and oak had the highest contents of CK at 14.79% and 14.29%, respectively. The highest content of FC (13.01%) was recorded in oak, while beech and Miscanthus had the lowest FC content with 8.91% and 9.01%, respectively. The highest VM content was found in Miscanthus (85.44%) and Beech (83.19%), and the lowest in Oak (79.99%).

Table 2: Proximate analysis

Sample	MC (%)	AC (%)	CK (%)	FC (%)	VM (%)
Oak	9.09a±0.04	0.35a±0.02	14.29b±0.29	13.01ab±0.26	79.99a±0.26
Beech	11.89b±0.10	0.55a±0.01	10.19a±0.09	8.91a±0.70	83.19b±0.56
Switchgrass	10.98a±0.22	3.74b±0.08	14.79b±0.67	10.29b±0.63	80.89a±0.72
Miscanthus	14.02b±0.18	1.73ab±0.26	13.87b±0.84	9.01a± 0.79	85.44ab±0.85
Significance	***	***	***	***	***

Values in the column with the same letter are not statistically significantly different with $p < 0.05$. Statistical difference: *** $p < 0.001$, NS—not significant.

Noncombustible components of biomass include MC, AC, and FC. MC in biomass can vary widely and is an undesirable component of any fuel (Oberberger and Thek, 2004). For example, Carvalho et al. (2017) reported a MC of 9.7% for oak, which is almost identical to the result of this study. In general, biomass AC can contain between 1.0 and 40.0% (forest less than 0.5%, agriculture up to 25.0%). Feedstocks with a higher AC have a lower calorific value, so a lower percentage is desirable, and biomass with a higher AC significantly reduces the efficiency of the combustion system (Vo a et al., 2021).

Telmo, Lousada, and Moreira (2010) and Ulusal et al. (2021) reported for hardwood AC up to 0.8%, VM up to 85.9%, and FC up to 16.4%, so the AC, VM, and FC of the studied oak and beech were below the literature limits. In the literature, MC (spring harvest) for switchgrass varies from 6.0 to 9.1% (Sadaka et al., 2014; Tumuluru, 2015), while for Miscanthus (spring harvest) it ranges from 7.5 to 11.5% (McKendry, 2002; Garcia et al., 2012); therefore, MC for the studied switchgrass is below the literature limits, while it is much higher for Miscanthus. In the literature, Clarke and Preto (2011) reported AC of 5.7% for switchgrass, Sadaka et al. (2014) reported FC of 23.1%, and Kumar and Ghosh (2018) reported VM of 83.2%, so it can be concluded that AC, VM, and FC of the studied switchgrass are below the literature values. For Miscanthus, Bilandžija et al. (2018) reported AC of 1.78% in the literature; for FC, Jackson et al. (2016) reported 14.49%, and for VM, Bilandžija et al. (2017) reported 86.52%. The results of the study show that for the studied Miscanthus, only the percentage of VM was above the limits of the literature reports, while the percentages of AC and FC were below.

3.4 Heat value

In this type of research, calorific value is one of the most important parameters because it represents the energy value that can be obtained by burning certain biomass or biofuels (Garcia et al., 2012). Of the heating values, the HHV and LHV have been studied, and the differences that occur are mainly the result of unequal cell structure and different MC and AC (Lewandowski et al., 2003). For example, forest biomass has a slightly higher calorific value than agricultural biomass, so that the calorific value of forest biomass ranges from 18.0 to 23.0 MJ kg⁻¹, while most agricultural biomass has a calorific value between 15.5 and 19.5 MJ kg⁻¹ (Cai et al., 2017). As shown in Table 3. Switchgrass had the highest HHV and LHV, while beech had the lowest HHV, and oak had the lowest

LHV of all samples studied. However, when all results are considered, it can be seen that the values of HHV and LHV were nearly identical for all samples.

Table 3: Heat values

Sample	HHV (MJ kg ⁻¹)	LHV (MJ kg ⁻¹)
Oak	18.34a±0.02	17.10a±0.03
Beech	18.09a±0.11	17.23a±0.15
Switchgrass	18.50a±0,29	17.39a±0,27
Miscanthus	18.41a±0,25	17.19a±0,31
Significance	NS	NS

Values in the column with the same letter are not statistically significantly different with $p < 0.05$. Statistical difference: *** $p < 0.001$, NS—not significant.

Comparing the average values of the determined HHV and LHV of the investigated forest and energy crops presented in Table 3, it can be concluded that the values determined in this study are within the ranges reported by the mentioned authors in the literature.

3.5 Biomass potential for the production of solid biofuels

The study shows that the lignocellulosic composition of forest species and agricultural energy crops is within the average limits for each biomass. Lignocellulosic biomass, which has higher lignin content, is more suitable for conversion of feedstocks into solid biofuels through the pressing process, which can later be used for heat or power generation through direct combustion. It is obvious that the studied forest species have higher lignin content than energy crops, but their slightly higher content of cellulose and hemicellulose affects the calorific value, so it is almost the same for all studied biomasses. Studies have shown that there is a significant relationship between the lignin content and the HHV and LHV of biomass (Demirbas, 2001; Demirbas, 2004).

Conversion to a solid fuel by pressing can significantly improve the above disadvantages of biomass (Barry et al., 2022). Biomass pressing depends mainly on the system used and the working conditions (mill, pressure, temperature), as well as on the physicochemical properties of the feedstock (MC, AC, lignocellulosic composition, heating value). When biomass is pressed by the thermoplastic process of pelleting and briquetting, the biomass particles are compressed into compact pellets and briquettes under the action of high pressure and temperature, which increases the low bulk density of biomass to almost 1300 kg/m³ (Mani, Tabil, Sokhansanj, 2004; Tumuluru et al., 2011). In the pressing process, moisture plays a crucial role and is one of the most important parameters for the durability of pellets and briquettes, together with the lignin content. For efficient pressing, biomass moisture content should be between 5.0 and 20.0%, while ash content should be less than 4.0%, as higher content causes lower HHV (Maia et al., 2014; Mopoung and Udeye, 2017).

It is estimated that about 44.3 million tonnes of pellets were produced worldwide in 2021. Most of the pellets available on the market are produced exclusively from wood biomass, but there are also pellets produced from post-harvest residues of primary agricultural production. Pellets produced in this way are not widely used due to the higher AC (3.0-5.0%), which requires more frequent maintenance of combustion equipment at the user's site, so they are increasingly used by industry as a biofuel for heating or electricity generation. The calorific value of agropellets ranges from 12.0 to 18.0 MJ kg⁻¹, while AC is above 1.0%. However, the solution to this problem can be seen in the combination of the right ratio of the different biomasses. Biomass that produces a smaller AC can be combined with one that produces a larger one; i.e., by mixing biomass, MC, and AC, the calorific value can be adjusted to obtain a suitable product that meets all quality parameters (Smaga et al., 2018).

Comparing all the research results with the cited literature, it can be concluded that the two studied forest species and two agricultural energy crops can indeed be used as feedstock for solid biofuel production through the compression process.

4. CONCLUSION

Based on this study, it was found that all studied parameters were within the limits or showed a slight deviation from the listed literature data and from the standard for solid biofuels. Forest species had slightly higher lignin content, while energy crops had significantly higher ash content. The calorific value was higher for energy crops due to a slightly higher content of cellulose and hemicellulose in forest species, and as mentioned earlier, cellulose and hemicellulose have a lower calorific value than lignin. Certain deviations of the determined parameters from the literature data can be attributed to the influence of different cultivation sites, agroecological conditions, and other factors. When analysing the possibility of using the biomass of the investigated forest species and agricultural energy crops as raw material for the production of solid biofuels such as pellets and briquettes, it was found that all parameters meet the physicochemical properties that the biomass must have during the pressing process.

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The Authors' Address:

Božidar Matin MSc, University of Zagreb Faculty of Forestry and Wood Technology, Svetošimunska cesta 23, Zagreb, Croatia,

Associate Professor PhD Ana Matin, University of Zagreb Faculty of Agriculture, Svetošimunska cesta 25, Zagreb, Croatia,

Ivan Brandi MSc, University of Zagreb Faculty of Agriculture, Svetošimunska cesta 25, Zagreb, Croatia,

Alen urovi MSc, University of Zagreb Faculty of Forestry and Wood Technology, Svetošimunska cesta 23, Zagreb, Croatia,

Associate Professor PhD Josip Ištvan , University of Zagreb Faculty of Forestry and Wood Technology, Svetošimunska cesta 23, Zagreb, Croatia.

Professor PhD Alan Antonovi , University of Zagreb Faculty of Forestry and Wood Technology, Svetošimunska cesta 23, Zagreb, Croatia.