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Preliminary communication

Microscale Combustion Calorimetry as a New Method of Composition and Heating Value Determination of Miscanthus - An Early View

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Abstract: The perennial biomass crop Miscanthus combines key attributes achieving high energy output/input ratios in a wide range of climatic conditions and it is an important feedstock for bio-mass-to-energy. New Miscanthus hybrids, with better yield resilience and scalability, that are expected to boost supply from marginal lands in Europe, were grown in Croatia and the composition of ripe biomass samples from a spring harvest were analysed by a new microscale combustion calorimetry (MCC) method that determines the heat release rate (HRR). The same samples were also analysed by standard chemical methods: structural analysis, and bomb calorimetry. Higher heating values (HHVs) measured by bomb calorimetry ranged from 17.76 MJ/kg to 18.10 MJ/kg. Microscale combustion calorimetry developed to quantify flammability indices such as HRR was strongly correlated with HHV, which could allow differentiation between new hybrids faster and more efficiently. Obtaining such energy properties data from an early stage of development is necessary for the detection of genotypes with high biomass-to-bioenergy potential for intensive monitoring in the coming period.

Keywords: energy properties; microscale combustion calorimetry; Miscanthus; novel hybrids

1 INTRODUCTION

Biomass is the only renewable energy source with relevant industrial use in EU (93% of the RES used in the EU industry was solid biomass, 3% municipal waste and 2% biogas) [1]. Those industries that are coproducing solid biomass residues have already implemented biomass-to-bioenergy utilization projects, while those with no biomass residues are facing economic challenges for further biomass use [1]. This potential shortage of raw material could be mitigated by a careful selection and expanded cultivation and production of energy crops. Energy crop should contain more potential energy per mass unit than what is used for its cultivation, harvesting, transportation, and processing; that is, it should be more energy-efficient (output : input energy ratio) [2]. The efficiency of the biomass used in such a manner depends on the whole life cycle. Still, here we focus on variation in biomass composition and its impact on energetic efficiency. Solid fuel qualities for new and fossil biomass (coal) are often determined by structural (lignocellulosic) polysaccharide composition and the heating value [3, 4].

Currently, the cultivation of energy crops includes short-rotation coppice (SRC) cultivation in forestry, and perennial lignocellulosic grasses, such as *Miscanthus*, switchgrass, and reed canary grass in agriculture. Perennial lignocellulosic grasses are characterized by a high potential of biomass yield with low input requirements and high efficiency of resource usage. Moreover, in comparison with annual energy crops, perennial lignocellulosic energy grasses display better ecological and environmental performance [5]. It is estimated [6] that more than 53000 ha of land are used for the cultivation of perennial lignocellulosic energy grasses (117 401 ha for energy crops in total) in the European Union (EU28). According to the same source, almost half of that land is used for the cultivation of *Miscanthus*.

Miscanthus is a rhizomatous C4 grass originating from South East Asia [7, 8]. It is known for its high performance compared to low demands and can be harvested annually over a period of 20 years and more [7-9]. Furthermore, *Miscanthus* can be cultivated on marginal land; thus, there is no conflict with the production of food and feed [9]. The main hybrid cultivated in Europe is *Miscanthus* x *giganteus*. However, due to the enhancement of stress tolerances, new hybrids are being developed. Namely, during drought periods, *M. xgiganteus* fails to regulate the rate of water loss making it susceptible to death and yield loss in severe and prolonged droughts, which are more likely with climate change [10]. In addition, improvement of drought tolerance in *M. x giganteus* through breeding is impossible due to its sterility. Possible increase of drought tolerance in *M. x giganteus* through breeding is a selection of drought tolerant parent species (*M. sinensis* and *M. sacchariflorus*). Currently, there is no commercially available *Miscanthus* hybrid which fully corresponds to different supply chains and end-uses of biomass (e.g. direct combustion, anaerobic digestion or phenols production) [9].

In terms of combustion, biomass conversion energy efficiency is a crucial trait and it is determined by its energy properties. Among them, a higher heating value (HHV) is the most common assessment to describe gross compositional properties impacting biomass-to-energy conversion [11]. It defines the energy content of biomass representing the amount of heat energy released during the complete combustion of a mass unit of biomass. It is used for numerical simulations of thermal systems to assess the quality of combustion [12]. However, this process is rather time consuming and material inefficient, so the development of novel, more efficient analytical methods is needed to identify both positive and negative compositional traits for robust selection of promising hybrids.

Microscale combustion calorimetry (MCC) was originally developed by the U.S. Federal Aviation Administration (FAA) to assess the fire safety of aircraft materials. It is a fast flammability test that uses mg-sized samples for characterization of the ignitability of different materials, their heat release, and thermal behaviour during the combustion itself [25].

The objective of this research was to valorise the energy potential of four new *Miscanthus* hybrids, including commercial *M. x giganteus*. *M. x giganteus* was used as a control, as it has been intensively investigated in different studies throughout the previous years. Furthermore, the higher heating value data, as a property inevitable for the modelling of biomass-to-bioenergy efficiency, were correlated with heat release rates (HRRs), in order to screen the possibility of using faster and more efficient method for *Miscanthus* composition and heating value determination.

2 MATERIALS AND METHODS
2.1 Materials

In this study, the biomass of 4 new *M.sacchariflorus x M.sacchariflorus* (GRC 11-15) hybrids, and *M. x giganteus* (GRC 9) as a control hybrid was used. New hybrids have been specifically selected from several European breeding programs due to promising traits directly related to energy conversion efficiency and are currently in the phase of testing and research. Biomass was harvested at the University of Zagreb Faculty of Agriculture experimental station Šašinovec (45°50'59.3"N, 16°11'26.2"E), Croatia, in March 2019, at the end of the 1st vegetation year. Before the analyses, biomass was dried, chopped, and prepared according to the EN ISO 14780:2017 method.

2.2 Methods

Determination of lignin, cellulose, and hemicellulose content was conducted by the modified standard method ISO 5351-1:2002.

Higher heating value (HHV) (EN 14918:2010) was determined in an adiabatic bomb calorimeter IKA C200, by determination of the heat energy released from a combusted sample.

For thermal characterization of Miscanthus samples, so-called microscale combustion calorimetry was conducted using an MCC-2 calorimeter (The Govmark Organization Inc., UK) according to the ASTM D7309-2007 method. Prepared samples, weighing approximately 5 mg, were heated to 70 °C using a linear heating rate of 1 °C/s in a nitrogen stream with a flow rate of 80 cm^3/min. Thermal decomposition products were mixed with an oxygen stream of 20 cm^3/min to simulate the combustion processes of the condensed and gas phases (Fig. 1).

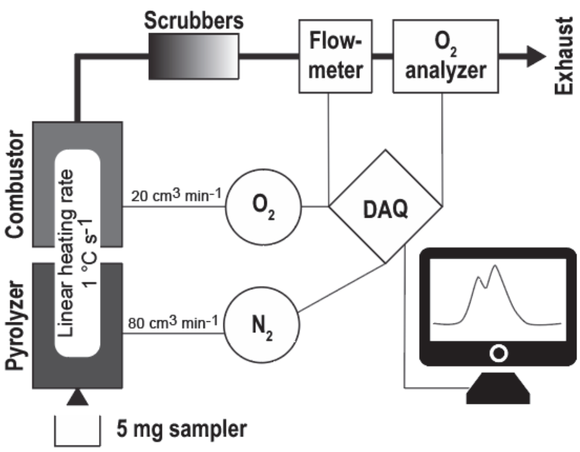

Figure 1 Microscale combustion calorimetry (MCC) scheme

Statistical analysis of gathered data was performed using one-way ANOVA with Tukey's multiple comparison test ($p < 0.005$). Also, Pearson correlation coefficient method was conducted. Pearson correlation coefficient (r) is a measure of the linear association of two variables, which varies from –1 to +1. A p-value level of 0.05 ($p < 0.05$) was used to determine statistically significant correlation coefficients.

Statistical analysis and graphical representation were done using R (R Development Core Team, 2008), R Studio (RStudio, Inc., 2018) and "multcompview", "lsmeans", "multcomp", "devtools", "ggpubr" and "ggplot2" packages.

3 RESULTS

Structural composition is a parameter that significantly affects HHV of biomass. Cellulose content of biomass is undesirable in larger proportions, as well as hemicellulose, if the biomass is going to be used for thermochemical conversions, such as direct combustion. The main disadvantage of cellulose and hemicellulose is that both contain large contents of oxygen, thus reduce the heating value of biomass when used as fuel. On the other hand, the lignin content of biomass positively impacts the heating value [13-15]. Tab. 1 shows structural properties of new Miscanthus hybrids biomass, as well as associated heating values.

Table 1 Structural polysaccharide composition of different Miscanthus hybrids (oven dry weight basis) with associated heating values. Statistically significant differences (at $p < 0.05$) are shown by different superscript letters in the vertical columns ($n = 4$)

Hybrid ID	Lignin / %	Cellulose / %	Hemicellulose / %	HHV / MJ/kg
GRC 9	12.79^{a}	42.54^{a}	40.04^{e}	17.94^{b}
GRC 11	15.57^{b}	44.68^{c}	35.06^{d}	17.76^{a}
GRC 13	17.34^{d}	43.23^{b}	33.39^{c}	18.01^{bc}
GRC 14	16.63^{c}	46.01^{d}	32.36^{b}	18.10^{d}
GRC 15	17.93^{e}	46.73^{e}	29.39^{a}	18.02^{c}

In Tab. 1, it is shown that lignin content ranges from 12.79% to 17.93%. Biomass with high shares of lignin is considered desirable for the process of direct combustion. Average levels of the biomass structural composition are 10.00% to 28.39% of lignin, 40.00% to 60.00% of cellulose, and 15.30% to 40.00% of hemicellulose [4, 12]. Cellulose content ranged from 42.54% to 46.73%, which is equal to the average literature level. High shares of cellulose are related to a high content of oxygen in biomass, which has a negative impact on heating value but promotes bioethanol production efficiency. Hemicellulose ranged from 29.39% to 40.04%, which is the most significant variability between hybrids in terms of the structural properties, but in accordance with average levels found in lignocellulosic biomass. Lignocellulosic composition of various agricultural straws (wheat, rice, corn, barley, rye, and oat) contained 13% to 25% of lignin, 30% to 39% of cellulose, and 22% to 27% of hemicellulose [17], while *A. donax* biomass contained 36.14% of cellulose, 32.09% of hemicellulose, and 10.66% of lignin [18].

HHV is probably the most critical parameter in such research. It represents the amount of energy that can be obtained by burning a certain amount of biomass [19]. Thus, it is of the highest interest for industry users, as it mostly defines the energy balance and output. Its value is strongly associated with the elemental composition [20-22]. HHV of the five analyzed *Miscanthus* hybrids (Tab. 1) ranged from 17.76 MJ/kg (GRC 11) to 18.10 MJ/kg (GRC

14). In previous researches, HHV of *Miscanthus* biomass ranged from 17.52 MJ/kg to 19.36 MJ/kg [4, 23]. Wood pellets (19.80 MJ/kg) and sunflower husk pellets (20.10%) had somewhat higher HHV than *Miscanthus* hybrids, while forest chips (14.80 MJ/kg), straw (16.00 MJ/kg) and hay (15.60 MJ/kg) briquettes had considerably lower value [24]. Samples of softwood (18.21 MJ/kg), hardwood (18.01 MJ/kg) and wheat straw (17.24 MJ/kg) pellets had similar heating values with *Miscanthus* biomass [25]. By comparing these results with examined hybrids, no significant deviations from the obtained values was noticed.

The following parameters are the usual output of the MCC: heat release capacity (HRC), peak heat release rate (PHRR), peak heat release temperature (TPHRR), total heat release (THR) and heat release rate (HRR). Heat release rate is usually presented as a temperature-dependent curve (Fig. 2); however, in order to correlate it with HHV, the area under the curve (AUC) was calculated. Heat release rate of investigated hybrids, in relation to the increase of temperature, is shown in Fig. 2.

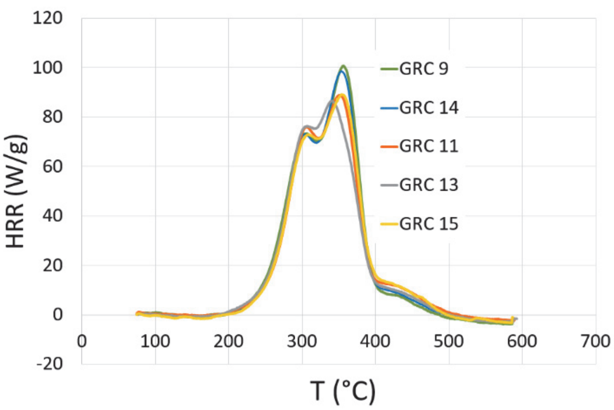

Figure 2 Heat release rate (HRR) of new *Miscanthus* hybrids biomass

Fig. 2 shows the dynamics and behaviour of investigated biomass during the combustion and the increase in temperature. To uniformly capture the initial temperature and avoid data impurities, a value of 2 W/g heat release was taken as a combustion starting point. All investigated hybrids had initial combustion temperature in the relatively close range from 205.41 °C (GRC 13) to 220.22 °C (GRC 15). Compared to the combustion of cotton, the initial combustion temperature of *Miscanthus* is significantly lower. Cotton combusts at 300 °C. Along with cotton, starch [26] and cardboard [27] samples as well started with combustion at around 300 °C. This could be explained by different structural composition of raw materials, since *Miscanthus* has only 42.54% - 46.73% of cellulose [28].

Peak heat release values of investigated hybrids could be divided in two groups in terms of heat release. Temperatures at which the peak heat is being released, occur within a rather narrow range from 339.88 °C (GRC 13) to 356.48 °C (GRC 9). The first group had lower heat release values; GRC 13 released 86.46 W/g, GRC 11 88.97 W/g and GRC 15 89.23 W/g. The second group emitted something more heat per mass unit, respectively GRC 14 (98.61 W/g) and GRC 9 (100.85 W/g). After the peak heating rate, all investigated hybrids started to extinguish rapidly until temperature reached around 400 °C, following the smouldering until the temperature reached around 500 °C.

Another thing presented in Fig. 2 is shouldering, where all hybrids showed a mild drop in the heat release after the temperature exceeded 300 °C, followed by continued increase at the temperature around 330 °C. Physically, this corresponds to the surface of a burning sample when the temperature gradient extends into the sample and fuel is generated simultaneously at every depth [13]. Second shoulder, in terms of the temperature, corresponds with thermal degradation of cardboard [27]. A cardboard used in that study contained 59.7% of cellulose and 14.2% of lignin, which is more comparable to the structure of investigated hybrids. Another research that supports those findings [14] showed that combustion of cotton is initiated at the temperature around 300 °C, and it reaches its peak at the HHR of around 140 W/g, while xylan and lignin started with the combustion at the temperatures in a range similar to the *Miscanthus* biomass. Throughout the combustion of that sample, a decrease in xylan HRR was present at a temperature of around 315 °C, while lignin and cellulose did not reach their peak; thus, shouldering appeared.

In order to understand the importance of the MCC output data, in terms of the energy valorisation of biomass, the area under the HRR curve was correlated with HHV to obtain the outlook of the specific relationship (Fig. 3).

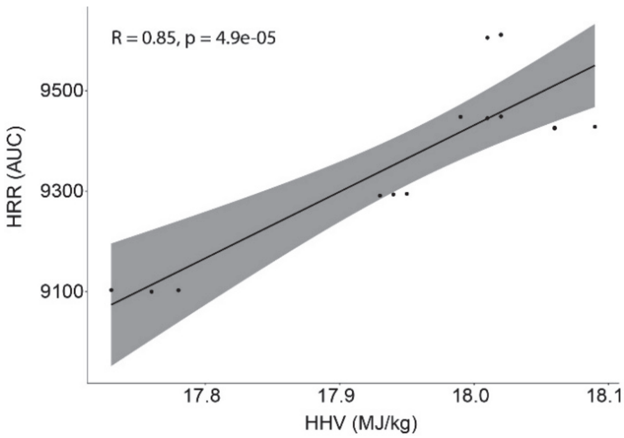

Figure 3 Correlation between higher heating value (HHV) and heat release rate (HRR)

With the correlation coefficient of 0.85 ($p < 0.001$), the relationship between HHV and HRR is negligible. As HVV represents the amount of energy that can be obtained from a certain amount of fuel, and HRR heat released from a certain amount of fuel throughout the combustion process, a significant correlation was expected.

MCC is a time- and material-efficient standardized method that uses milligram-sized. Bomb calorimetry completely combusts around 1.0 g of sample for up to 25 minutes. MCC analysis takes 5 minutes and outputs various detailed characteristics of fuel thermal and degradational behavior, at different temperatures. Beside HHV assumptions and calculations, structural composition of samples could be recognized prior to polysaccharides analysis.

4 CONCLUSION

In contrast to cooler regions in northern Europe, the warm summer climate in Croatia promotes rapid physiological maturation with prolific rhizome growth even in the first year after planting. This study which reports on the biomass quality in the first harvest after planting will be extended in the second and third years to detect if stand age influences biomass composition under Croatian climatic conditions. If compositional changes are not detected between first year and subsequent harvests, or if the ranking for the different hybrids is consistent, this "early view" of compositional traits could be used to accelerate breeding through faster selection of promising hybrids than is currently possible in cooler regions.

Microscale combustion calorimetry (MCC) was used as a novel method for the flammability analysis of the biomass. Since no study, where the MCC was used for valorization of energy properties of the *Miscanthus* biomass, was conducted so far, a strong correlation that was found between the MCC's main parameter (HRR) and HHV, could provide the basis for future research related to HHV. MCC decodes thermal properties evaluating several relevant outputs that could be used for either pre-selection of fuels, or detailed characterization of novel fuels such as *Miscanthus* biomass from new hybrids.

A more detailed study of the *Miscanthus* biomass combustion behaviour could provide a new approach to the structural analysis of biomass since the relationship was found to be relevant.

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