

The Impact of Harvest Timing on Properties of *Miscanthus x Giganteus* Biomass as a CO₂ Neutral Energy Source

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ABSTRACT

The European Commission, through its Directive 2009/28/EC, decided to extensively support the consumption and production of »green« energy from renewable energy sources, primarily because of diminishing supplies and constant rise in fossil fuels prices and critical necessity to mitigate their negative influence on the environment. The alleviation of a part of these problems can be achieved by growing and use for energy purposes of biomass from *Miscanthus x giganteus* grass. Given the fact that these cultures can be harvested in the whole period from the month of November until the beginning of new vegetative cycle, the objective of this paper is to determine the ultimate properties (C, H, O, S, volatile matter) and proximate properties (moisture, fixed carbon, coke, ash, N), content of minor (Cd, Cr, Co, Cu, Fe, Pb, Mn, Ni, Zn) and major elements (Ca, K, Na, Mg) as well as high heating value of biomass *M. x giganteus* from the first year of cultivation from three different locations in the Republic of Croatia (Donja Bistra, Medvednica and Donji Lapac), harvested in three different times (November 2011, February and March 2012). The conducted investigations confirmed the potential of *M. x giganteus* crop grown in the fields in Croatia as a high-quality and environmental friendly feedstock for direct production of electricity and heat.

Key words: *Miscanthus x giganteus*, harvest times, ultimate and proximate properties, minor and major elements

Introduction

Due to increasing world population and economic development, the energy needs are incessantly growing. Enormous quantities of natural resources are used daily with effect of deteriorating the environment. The use of non-renewable fossil fuels, greenhouse gas emissions and presence of toxic substances in atmosphere are rising, thus undermining the environment and human health¹. With the aim to increase the share of renewable energy and mitigate climate changes through reduction of GHG emissions, based on set guidelines deriving from the Kyoto Protocol, the European Commission has drawn up the Energy Strategy for Europe until 2020. The strategy laid down three main pillars: sustainability (environmental protection), competitiveness (cost-effectiveness), and security of supply (technical and technological devel-

opment), also known as 3x20. Practically, this should lead to GHG reduction by 20%, reduction in total energy consumption by 20% and increase in renewable energy sources by 20%, taking into account that 10% of biofuels must be used in the transport sector². Accordingly, it can be said that the European Union's pursue of energy independence and volatility of fossil fuels' prices are the main drives behind production and use of new, clean CO₂-neutral fuels³. The estimates show that the production of renewable energy will be based, in addition to solar and wind energy, on the existing and newly-produced biomass from ligno-cellulose energy crops. The surfaces under these crops are constantly rising in global terms. Since there is a strong connection between using fossil energy sources and global environmental warming, en-

ergy crops are one of possible factors of reducing the consumption of non-renewable energy sources and make an increasingly large part of the strategy aimed at GHG emission reduction⁴.

The selection of energy crops for production of »green energy« is based on conservation of relatively high net fossil energy during cultivation and on the possibility of reducing GHG emissions by unit of biomass and agricultural surface⁵. If we add exceptional capacity of disease and pest resistance and possibility of economical use of nutrients from soil⁶, then *Miscanthus x giganteus*, perennial energy grass, appears to be an adequate crop which meets all requirements. The main characteristic of *M x giganteus*, is that they enable multi-year cultivation (15–20 years and more) large scale biomass production (starting from the third year of cultivation it amounts to average 15–25 tone dry matter/ha) of energy valuable biomass (17.05 to 19.21 MJ/kg annually). Due to remarkable ability to translocate minerals and nutrients from above-ground parts in to rhizome at the end of the vegetation period and re-translocation from rhizome back to above-ground plant at the beginning of the vegetative process, *M. x giganteus* is remarkably efficient in using nutrients, especially nitrogen. At the same time this reduces nitrate leaching due to reduced application of fertilizers^{7–9}. In addition to this, the environmental aspect of *M. x giganteus* is also enhanced by the fact that herbicides, as well as most of other agro-engineering measures are to be applied only in the first and possibly second year from establishing crop¹⁰, which directly contributes to GHG emission reduction compared to conventional agricultural crops.

Given its exploitability for energy purpose, *M. x giganteus* is presently being used mostly for co-firing with fossil solid fuels and for direct combustion in electricity and/or heat generation¹¹, mostly in form of wood chips, pellets/briquettes and bales. Combustion is defined as a chemical reaction occurring between a fuel and an oxidizing agent producing CO₂ and steam. Compared to fossil fuels, biomass gives cleaner combustion products. Also, throughout its cultivation cycle, by reduced treatment and nurture of the crops and by sequestration process (plant/soil), approximately as much of CO₂ is reduced as is released during its energy use. Therefore, it can be concluded that energy production from this type of second-generation biofuel is almost CO₂-neutral¹². The energy utilization of this crop significantly enables to preserve fossil energy sources, e.g., 20 tons of *M. x giganteus* biomass is an equivalent to 12 tons of hard coal¹³, while 30 tons of this biomass is equivalent to 12,000 litres of fuel oil¹⁴. Moreover,^{11,13,15} mention the possibility of reducing CO₂ emission by 75–93%, if gas, fuel oil, brown coal and electricity are replaced with *M. x giganteus* biomass.

Given the fact that harvesting can be carried out from November (after first frosts) all the way through the beginning of new vegetative cycle (March, April), in each climate region an optimal harvest timing can be chosen taking into consideration the present moisture content

and energy properties of the crop^{16,17}. Early harvesting maximizes energy yield per hectare while late harvest will lower it. The investigations in Germany show that energy yields of *M. x giganteus* harvested in December are between 187 and 528 GJ ha⁻¹. With late harvest energy yield drops by 12–15% between December and February, and by additional 13% between February and March. Postponing harvest from autumn to spring (March, April) results in lower biomass yield in the year following, but it gives better combustion properties due to lower contents of moisture, ash, chlorine, nitrogen and sulphur^{18,19}. The losses occurring in winter time may lead to lowering biomass yield by 15–35%, but drying in the field lowers moisture content in the crop itself, which has a positive effect combustion performance of the feedstock^{20,21}.

Out of 2.150,000 ha of potentially arable land in Croatia as much as 974,080 ha not in use, including over 600,000 ha of lower quality land which is inadequate for intensive crop production³. Based on this situation and on the investigations carried out by²², it can be concluded that *M. x giganteus* can be grown on a significant portion of the unused surfaces, where the energy crop cultivation will not compete for land against food production.

The aim of this paper is to determine ultimate (C, H, O, S, volatile matter) and proximate properties (moisture, fixed carbon, coke, ash, N), levels of minor elements (Cd, Cr, Co, Cu, Fe, Pb, Mn, Ni, Zn) and major elements (Ca, K, Na, Mg), and high heating values of biomass of *M. x giganteus* in the first cultivation year, grown in three locations (Donja Bistra, Medvednica and Donji Lapac), harvested at three different times (November 2011, February and March 2012).

Material and Methods

The harvested *M. x giganteus* biomass, which is used as a raw material in this investigation, was grown in three locations, Donja Bistra (N 45° 55' 06,2'', E 15° 50' 32,5'', 144 m above sea level), Medvednica (N 45° 55' 37,2'', E 15° 58' 24,4'', 650 m asl) and Donji Lapac (N 44° 32' 03,8''; E 15° 58' 31,1''; 565 m asl), on lower quality soils, in a regime of low agricultural investments. The trial was set at a random block arrangement pattern (3 harvest times in 6 replications). The size of the main plot was 20 x 3 m, with a 2-meter gap between main plots. The harvest of the analysed samples was carried out in the year of establishing plantation, at three points: at the end of vegetative cycle, immediately after appearance of first strong frosts (November 2011), and prior to beginning of the new vegetation (February and March 2012). A sub-sample of about 1,000 grams of cut biomass was taken from each main plot and put to drying for 48 hours at 60 °C in order to make the samples from different locations comparable. After sampling and drying, the investigated biomass was ground in a laboratory grinder (IKA Analysentechnik GmbH, Germany). In order to obtain homogenous samples for further research, after grinding and drying, the samples were separated. The sub-sam-

ples from each of the main plots were mixed and then separated in a separator with two separations, each containing eight sub-separations. In all samples the readings were taken for moisture content (CEN/TS 14774-2:2009), volatile matter (CEN/TS 15148:2009), fixed carbon and ash (CEN/TS 15148:2009). The contents of carbon, hydrogen, nitrogen and sulphur were also determined by standard methods (HRN EN 15104:2011; HRN EN 15289:2011; HRN EN 14918:2010), as well as high heating value (HRN EN 14918:2010). The content of all minor and major elements was determined by atomic absorption spectrometry (CEN/TS 15290:2006, CEN/TS 15297:2006; Determination of minor elements, 2006). All data obtained in this way were analysed according to the GLM procedure in the SAS system package version 8.00²³.

Results and Discussion

This investigation looks at proximate substances (moisture, ash, fixed carbon – Cfix and nitrogen), ultimate ones (carbon, sulphur, hydrogen, oxygen, volatile matter – VM) and high heating value (HHV) of biomass from *M. x giganteus*. The authors^{24–26} place the components analysed here among the most important chemical properties of biomass in dry processes of its transformation, which are tightly connected with the quality of biomass as an energy source. Accordingly, Tables 1 and 2 show the contents of analysed parameters for specific cutting times, i.e., the average of the three locations of biomass cultivation (Donja Bistra, Medvednica and Donji Lapac). The values obtained were compared to²⁷ norm for solid fuels, and to the relevant literature because this norm does not define all values observed in this investigation.

The average moisture content in the samples was 28.59%. However, the comparison of moisture contents with regard to harvest times shows a significant moisture reduction. Moisture content of the second harvest was 60.55% lower compared to the first one while in the third harvest it was lower by as much as 48.69%. That delayed harvest results in lowering percentage moisture content was corroborated by the investigations carried out by^{16,21,28}. High moisture content lowers heating value of biomass and leads to an overall drop in combustion efficiency²⁹. Thus, a lower content of moisture is a desirable feature. The obtained values confirm the thesis that there are economic reasons for late harvest, when mois-

ture content is below 30% because of lower costs of drying the material (if necessary)¹⁸. Therefore, it can be said that harvesting in February or March would be the most acceptable, regarding the percentage share of moisture in biomass. Ash is an undesirable element in biomass because of its catalytic impact on thermal decomposition, i.e., higher ash concentration results in higher concentration of coal and gases²⁹. Ash in biomass has a relatively low melting point, and ash melting during the thermal process creates »slag«, whose settlement in furnaces or boilers lowers energy transmission and overall combustion efficiency²⁹. In the National plan for scientific research and technological innovations in Spain³⁰, the reasons for varying amounts of ash in biomass are found in different contents of mineral nutrients in soil, ash is made of (Si, Al, Ti, Fe, Ca, Mg, Ca, K, S and P), and climate conditions (plants grown in semiarid and arid areas contain even higher amounts of silicium and aluminium, which are main components of ash). Also,³¹ asserts that ash composition in biomass depends on sort of plant, part of plant (leaf, stalk, bark), availability of nutrients and quality of soil. The found ash contents are 4.84% on the average, and different harvest times point at significant difference in relation to the investigated parameter. Because²⁷ norm for solid biofuels sets out limit values of percentage ash content in range from 1% to 6% it can be concluded that all presented values are in accordance with this norm. However, it is also important to notice that continuous reduction in ash content was observed. Namely, compared to the first harvest, where ash content was 5.07%, postponing harvest to the second and third date caused the lowering of ash content by 3.54% and 10.45% respectively. The average coke content is 16.51%, and is somewhat lower than values presented by³², who have determined its value in different types of biomass to be in a range from 16.72% to 21.79%. Postponing harvest to the second date caused reduction of coke content by 3.54%, while in the third-date harvest this value was reduced by 8.54% in relation to the first harvest. Fixed carbon is 11.62% on the average, which is somewhat lower from the results obtained by³³ who found a 14% fixed carbon content, while it is in accordance with the investigations performed by³⁴ who found a fixed carbon content of 11.40%. Fixed carbon content in the biomass harvested at the second harvest date was reduced by 2.34%, and 6.36% in the third harvest in relation to the first one. Also, since the amounts of nitrogen

TABLE 1
CONTENTS OF PROXIMATE SUBSTANCES (DRY MATTER), IN THREE DIFFERENT HARVEST TIMES

Harvest time	Moisture, %	Ash, %	Coke, %	Cfix, %	N, %
November 2011	53.36a±3.10	5.07a±0.54	17.21a±3.49	11.97a±3.23	1.02a±0.13
February 2012	21.05b±2.20	4.91ab±0.71	16.60a±2.49	11.69a±2.68	0.72b±0.07
March 2012	11.37c±1.94	4.54b±0.40	15.74b±1.95	11.21a±1.82	0.67b±0.06
Significance	<0.0001***	<0.0025**	0.2743 NS	0.5677 NS	<0.0001***

***p=0.001, ** p=0.01, * p=0.05, NS – non-significant, Cfix – fixed carbon

TABLE 2
CONTENTS OF ULTIMATE SUBSTANCES (DRY MATTER) AND HIGH HEATING VALUES, AT THREE HARVEST TIMES

Harvest time	C, %	S, %	H, %	O, %	VM, %	HHV, (MJ/kg)
November 2011	48.31b±0.80	0.09a±0.03	4.16b±0.10	46.39a±0.95	78.63a±3.89	18.79a±0.17
February 2012	49.28a±0.55	0.05c±0.002	3.98c±0.10	45.64b±0.52	77.88a±2.61	18.41b±0.27
March 2012	48.62b±0.18	0.08b±0.008	4.27a±0.15	46.34a±0.28	77.32a±1.81	18.26b±0.32
Significance	<0.0001***	<0.0001***	<0.0001***	0.0014 **	0.0654 NS	<0.0001***

***p=0.001, ** p=0.01, * p=0.05, NS – non-significant, VM – volatile matter, HHV – high heating value

and sulphur is a cause of undesirable emissions (of NO_x and SO₂) during biomass combustion³⁴, it is desirable that biomass has as low nitrogen concentration as possible. The average amount of nitrogen at all of different harvest times was 0.80%. Compared to the early harvest, percentage share in the second harvest was 29.41% lower, while in the third harvest it was 34.31% lower. As the norm²⁷ sets values from 0.1 to 1.5%, it can be said that late harvests significantly affect environmental value of biomass of *M. x giganteus*. Table 2 presents the variations in combustible substances contents in *M. x giganteus*, depending on harvest times.

From the obtained results (Table 2), it can be observed that carbon content did not vary significantly as result of planting location and harvest time; its average value was 48.73%. The analysed values are in full accordance with²⁷ values set between 46% and 52%. This is in accordance with²⁶, who claim that the expected carbon content in biomass is between 47% and 54%. Good carbon content is highly important, because carbon is identified as one of the most critical elements in the combustion process³⁵. In relation to the earliest harvest, percentage share of carbon increased by 1.99%, while in the third harvest it dropped to 0.64%. Sulphur has the lowest content, but from the environmental point of view it is, along with nitrogen, the most crucial element. Data for different harvest times showed differences in sulphur content; its average value was 0.07%. Accordingly, significant drop in sulphur content was observed in biomass harvested at the second date from the early harvest (44,45%). However, postponed harvest to the third date resulted in increasing sulphur content by 11.12%. So, it can be concluded that the second harvest was the most acceptable regarding the percentage share of sulphur in the investigated raw material. Since the applicable norm sets maximum allowed limit for sulphur at 0.6%, the analysed biomass of *M. x giganteus* was proved to be an ecologically acceptable fuel. Also, average hydrogen value was 4.13%, but significant differences with regard to harvest dates can be observed. Hydrogen content dropped in the second harvest by 4.33% from the early harvest, but in the third harvest it increased by 2.64% from the second. Since norm and quote values of 5% to 6.5% and 5.7% respectively as satisfactory, it can be noted that hydrogen content is lowered in the investigated biomass^{27,36}. Reduced hydrogen content may represent a problem because hydrogen, together with carbon, is defined as a very important element determining energy features of

solid biofuels³⁵. In general, biomass has high content of volatile matter, with values of about 75%, but they can grow up to 90%, pending on raw material²⁵. The analysed volatile matter in different harvest times is in accordance with the literature references^{33,34,37} and its average value is 76.61%, so that it falls in a range from 65% to 85%³⁸. Postponing harvest to the second date resulted in lowering hydrogen content by 1.62% in relation to the first harvest, while in biomass harvested at the third date it was reduced by 0.11%. High heating value, as one of the basic parameters of energy value of a fuel, appeared to be higher than in the investigations by^{37,34}, who quote values of 17.77 MJ/kg and 18.07 MJ/kg respectively; its average was 18.48 MJ/kg. The reason behind this difference in high heating values is mainly related to varying content of cell wall and water and ash concentration²⁹. Different harvest dates notably determined the heating value: in late harvests a gradual reduction in this parameter was recorded. In the second harvest, high heating value dropped by 2.02% and in the third one by 2.82%, all in relation to the first harvest. However, regardless of the harvest dates, the analysed high heating values are still within the limits of²⁷ norm (17.00 MJ/kg – 20.00 MJ/kg).

In order to obtain a complete insight in to energy and environmental features of the investigated raw material, the analyses of minor and major elements were carried out (Table 3). It is commonly accepted that the concentration and behaviour of elements such as Ca, K, Na, Si and specially heavy metals (or, more precisely, trace elements) are mostly responsible for many technological and environmental problems connected to biomass processing³³. Increased contents of some of these elements, particularly the toxic ones, may represent a problem when it comes to ash disposal in environmentally acceptable way following the biomass combustion process.

From data in Table 2 it was determined that amounts of chrome, lead, nickel and cobalt in the investigated biomass were below sensitivity levels. The comparison of these values with those set out in²⁷ for: copper, (1 mg/kg – 6 mg/kg), magnesium (300 mg/kg – 900 mg/kg), iron (40 mg/kg – 400 mg/kg), cadmium (0.4 mg/kg – 8 mg/kg) and sodium (20 mg/kg – 100 mg/kg), shows that these elements are fully in the range set by the norm, regardless of the harvest date. As in the case of combustible and non-combustible substances, the analysis of these elements showed some changes in the biomass composition in different harvest times. Compared to the early har-

TABLE 3
MINOR AND MAJOR ELEMENTS, IN THREE HARVEST TIMES

Analyses mg/kg	Harvest time			Significance
	November 2011	February 2012	March 2012	
Cu	5.56a±1.01	5.78a±1.28	4.47b±1.41	***
Mn	215.35a±119.35	168.17ab±81.34	147.08b±76.02	***
Cd	1.62b±0.63	2.78ab±3.86	4.32a±5.68	NS
Zn	46.00a±6.24	48.02a±9.62	48.81a±10.56	NS
Fe	284.63a±174.12	323.48a±115.00	319.27±113.32	NS
Na	61.87a±18.94	67.48a±18.40	63.94a±18.12	NS
K	946.9a±58.99	926.66a±75.67	892.95a±48.39	***
Mg	409.02a±24.42	401.14a±41.69	330.76b±22.20	***
Ca	2415.4a±666.71	1836.5a±283.51	907.0c±282.87	***
Cr	Below sensitivity level			–
Pb	Below sensitivity level			–
Ni	Below sensitivity level			–
Co	Below sensitivity level			–

***p=0.001, ** p=0.01, * p=0.05, NS-non-significant

vest, the amounts of magnesium, copper, potassium, manganese, and calcium in the investigated biomass were gradually reduced, with average respective values of 380.00 mg/kg, 5.27 mg/kg, 922.00 mg/kg, 176.86 mg/kg, and 1719.63 mg/kg. Therefore, in the second harvest the amounts of these elements were reduced by 1.93%; 3.96%; 2.14%; 21.91%; 23.97% respectively, while the third harvest showed larger reduction, by 24.27%; 19.6%; 5.7%; 31.7%; 62.45% respectively. However, as mentioned before, the amounts of magnesium and copper are within the norm, while certain divergences from the limit values were identified in the amounts of potassium, manganese, and calcium, 1,000 mg/kg – 11,000 mg/kg, 10 mg/kg – 100 mg/kg, 300 mg/kg – 900 mg/kg respectively. In contrast to these elements, the amounts of cadmium, iron, zinc and sodium were increasing with postponing the harvest, and average values for these elements were 2.90 mg/kg, 309.12 mg/kg, 47.61 mg/kg, and 59.38 mg/kg respectively. Thus, in the second harvest these elements increased by 71.60%, 12.86%, 4.39%, 9.07% respectively, and in the third harvest by 166.66%, 11.39%, 6.11%, 3.35% respectively compared to the first harvest. The comparison of the analysed values with relevant values set out in the applicable norm shows full accordance in the amounts of cadmium and iron, but also some discrepancies in the amounts of zinc and sodium. Namely, according to²⁷ norm the values for zinc and sodium are 3 mg/kg – 30 mg/kg and 20 mg/kg – 100 mg/kg respectively. The investigations conducted by³⁹ on pelleted biomass of *M. x giganteus* already indicated that divergences in amounts of specific elements can be expected. They quote the amounts of calcium (3400 mg/kg), sodium (280 mg/kg) and zinc (150 mg/kg). As it is demonstrated, the analysed values from the investigations by also diverge from the norm³⁹. Given the fact that biomass composition mostly depends on by soil properties and climate factors of the cultivation area,

it should be noted that norm²⁷ is based on scientific data obtained in the north European regions (Sweden, Finland, Denmark, The Netherlands, and Germany).

Conclusion

Following the investigations of biomass of *Miscanthus x giganteus* grass, which was grown in three locations (Donja Bistra, Medvednica, Donji Lapac) and with regard to three different harvest dates (1 November 2011; 2 February; 3 March 2012), it results that: the analysis of chemical composition of biomass of *M. x giganteus* grass showed certain variations in amounts of organic chemical compounds, carbon, hydrogen, oxygen, and sulphur as well as in content of water, ashes, coke, volatile matter, fixed carbon, minor and major elements, with regard to harvest dates. Postponing harvest for later dates (February, March 2012) resulted in reduction of the amounts of moisture, ash, nitrogen and sulphur, which directly upgraded the quality of biomass but it also caused the lowering of fixed carbon and high heating value. Late harvesting also brought about reduction of the amounts of magnesium, copper, potassium, manganese and calcium, while the same plant-growing measure resulted in increasing content of cadmium, iron, zinc and sodium. The amounts of chrome, lead, nickel, and cobalt in the investigated biomass were below sensitivity level. Except for hydrogen, all analysed data for ultimate and proximate substances, as well as minor and major elements, are in accordance or diverge only minimally from CEN/TS 14961 (2005) norm for solid biofuels and/or relevant literature references. The conducted investigations corroborated the potential of *M. x giganteus* cultivated in Croatia as highly valuable energy crop and environmentally acceptable raw material for direct production of electricity and/or heat.

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UTJECAJ ROKA ŽETVE NA SVOJSTVA BIOMASE TRAVE *MISCANTHUS X GIGANTEUS* KAO CO₂ NEUTRALNOG ENERGENTA

SAŽETAK

Europska komisija je kroz Direktivu 2009/28/EC odlučila intenzivno poticati potrošnju ali i proizvodnju »zelene« energije iz obnovljivih izvora i to prvenstveno uslijed smanjenja zaliha i konstantnog rasta cijena fosilnih goriva kao i neophodne potrebe za ublažavanjem posljedica njihovog negativnog utjecaja na okoliš. Ublažavanje jednog dijela navedenih problema može se postići uzgojem i energetskim iskorištenjem biomase trave *Miscanthus x giganteus*. Obzirom da se žetva navedene kulture može provesti od studenog pa sve do početka novog ciklusa vegetacije, cilj ovog rada je utvrditi goriva (C, H, O, S, hlapive tvari) i negoriva (vlaga, fiksirani ugljik, koks, pepeo, N) svojstva, udio mikro (Cd, Cr, Co, Cu, Fe, Pb, Mn, Ni, Zn) i makro elemenata (Ca, K, Na, Mg) te gornju ogrjevnu vrijednost biomase iz prve godine uzgoja trave *M. x giganteus* s 3 različite lokacije na području Republike Hrvatske (Donja Bistra, Medvednica i Donji Lapac), požete u 3 roka žetve (studen 2011, veljača i ožujak 2012). Provedena istraživanja su potvrdila potencijal trave *M. x giganteus* uzgojene u Republici Hrvatskoj kao energetski visokokvalitetne i ekološki prihvatljive sirovine u neposrednoj proizvodnji električne i/ili toplinske energije.