Desalination and Water Treatment
www.deswater.com

doi: 10.5004/dwt.2020.25438

189 (2020) 38–44
June

Management of sewage sludge ash (SSA) in the cultivation of *Miscanthus giganteus*

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Received 12 June 2019; Accepted 4 January 2020

ABSTRACT

The paper presents the results of the pot experiment using sewage sludge ash (SSA) at the growth of *Miscanthus giganteus*. The waste used in the tests, constituting the substitute of phosphorus fertilizer, was obtained in the wastewater treatment Plant in Szczecin. This experiment covers three variants of fertilizing with the dust-gravel in doses of phosphorus. The purpose of the research is to develop an effective method of managing the dust-gravel caused in the process of thermal disposal of municipal sewage sludge for the growth of the energetic plants. Laboratory studies covered a conduct of marking the macro-components' content and heavy metals contained in SSA in the soil material and crops of test plant. During the pot experiment, an average sample of the soil was gathered in which a dry mass was marked, pH, phosphorus, and total potassium dissolved and digestible, calcium, and heavy metals. In addition, the analysis of the physical-chemical composition was conducted, gross calorific value was marked and net calorific value of the crops collected. Outcomes of the research indicate for the possibility to use the SSA, that is, as the phosphorus fertilizer for the growth of *M. giganteus*. Favorable influence of the increasing dose of the phosphorus on the energetic value of the test plant was observed. Increase of the content of potassium was marked on the fertilized facilities with the largest dose of SSA. The content of heavy metals marked in the straw of *M. giganteus* corresponded to the standards for industrial plants.

Keywords: Sewage sludge ash; Phosphorus recovery; Sewage sludge; Cultivation; Energy crop

1. Introduction

In 2012, the municipal wastewater treatment plants in Poland generated 533,338 Mg dry mass of sewage sludge, transforming 11%. With an increasing amount of sewage sludge generated in 2017 (584,454 Mg dry mass), a percentage of their thermal management amounted to 18% increased [1].

"National Program of the Waste Management 2014" forecasted reaching 60% of the share of the thermal transformation of waste deposits till 2018 [2]. Ongoing "National Program of the Waste Management 2022" forecasts an annual increase of the deposit's amount by 2–3% in recalculation into dry matter. As in the preceding program, one of the main goals of the management of municipal sewage sludge is to increase the amount of wastage subjected to thermal transformation which also constitutes the main practice followed by the members of the European Union [3]. Simultaneously, the resolution stresses seeking to the maximization of usage's degree of the biogenic substances contained in the deposits [4]. The recommendations issued on 1st of March 2017 The Baltic Marine Environment Protection Commission (HELCOM) regarding the reprocessing of the sewage sludge also indicates for the recirculation of the biogenic substances contained therein, especially phosphorus. Therefore, it is crucial to conduct research in the scope, leading to halt of the circulation of the phosphorus in the nature, in accordance with an idea of the closed-circuit economy [5].

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Adopted National plan of waste economy 2022, as well as other provisions regarding the management of the sewage sludge, are based on the Union directives which include, first of all: Directive of the Council 86/278/EEC of 12th of June 1986, Directive of the Council 91/271/EEC of 21st of May 1991 and the Directive of the European Parliament and the Council 2008/98/EC of the 19th of November 2008 on waste [3,6].

Thermal transformation of waste process, similarly as in case of combustion of conventional fuels, causes the creation of dusts and gravels which constitute by-products [7]. Depending on the chemical composition of the waste combusted, their classification takes places in accordance with the applicable catalogue of waste, constituting the attachment to the regulation of the Minister of Environment of 9th of December 2014 on catalogue of waste [8]. By-products of sewage sludge's combustion are characterized by the high content of the biogenic substances: calcium, magnesium, and most importantly, phosphorus, which presence is linked with the course of the removal process from the system of waste treatment [9]. It allows to use the waste of that type in the fertilization process of plants as a substitute for the phosphorus fertilizer [10–13]. Content of the phosphorus in the waste created after the combustion of sewage sludges, according to literature data falls between 5.1% and 14.9% d.m. [14–24].

Phosphorus yielding functions are a derivative of many physiological processes that cause specific yielding effects. Good nutrition with phosphorus has a beneficial effect on the physiological state of the plant, including root growth, ion uptake, increases the content of stalk building compounds and shortens the maturing time of the plant. Irrespective of the chemical form, phosphorus compounds in the soil form fractions: soluble, labile, and stable. The soluble fraction includes inorganic phosphorus compounds. Depending on the pH of the soil solution, phosphorus compounds in the form of H_3PO_4, $H_2PO_4^-$, and HPO_4^{2-} dominate. Plants mainly take up $H_2PO_4^-$ ions that occur in the pH range from 4 to 8 [25,26].

In order to preserve soil properties and increase their productivity, it is essential to use energy plant fertilization. Using sewage sludge ash (SSA) as a fertilizer ensures soil protection, the maintenance of soil fertilization, biomass production for energy purposes. Simultaneously it allows for residual products from sewage sludge treatment processes to be developed and reuse [13,27,28].

The aim of the study was to determine the possibility of using SSA as a plant growing aid and soil quality improvement agent.

2. Materials and methods

The pot experiment was conducted in a random system between 2011 and 2013. The superficial layer of the incomplete, brown-russet soil, created from clay sand slightly dusty, with an average underlining with light clay were used for the experiment purposes. Soil order according to the United States Department of Agriculture Soil Taxonomy Classification—Inceptisols. Prior to the experiment, the soil pH was slightly acidic (5.8 pH in 1 mol KCl dm^{-3}), the total carbon and nitrogen content were, respectively, 8.59 and 0.84 g kg^{-1}. Used soil was obtained from the Agricultural Experiment Station in Lipnik near Stargard (Poland).

The dust-gravel was used as the substitute of the phosphorus fertilizer created as a result of the combustion of the municipal sewage sludge in mechanical grate furnace in the wastewater treatment Plant "Pomorzany" in Szczecin (Poland). For the first 2 y of the experiment, the material collected in 2011 was being used, whereas in the last year the dust-gravel was collected again at the beginning of 2013. Due to the variety of the waste fractions, it was sieved each time to the fraction with a grin diameter of 2 mm.

Wastage collected in 2011 from the combustion of the sewage sludge's content of the dissolvable phosphorus was marked in the strong mineral acids in the amount 93.13 g kg^{-1} P. The dust-gravel obtained 2 y later contained 71.06 g kg^{-1} P. An average total content of potassium, calcium, and magnesium in the materials used amounted to, respectively, 46.1 ± 3.5 g kg^{-1} K, 65.95 ± 3.75 g kg^{-1} Ca, and 42.7 ± 4 g kg^{-1} Mg (Fig. 1). In the materials examined, there were 1.758 ± 0.046 g of heavy metals marked in total (Table 1).

The test plant was *Miscanthus giganteus*, which is characterized by a fast pace of growth and very intensive and effective photosynthesis. In order to limit the influence of atmospheric conditions, the experiment was conducted under the foil roofing. Humidity of the soil material was maintained on the level of 60% of full water capacity. Average monthly air temperatures in periods from May to October were, respectively, 15.6 in 2011, 16.2 in 2012, and 17.8 in 2013.

Before planting the following fertilization was applied: N = 36 kg, P_2O_5 = 60 kg (26.4 kg P), K_2O = 120 kg (98.4 kg K) ha^{-1}. The experiment included the addition of three variants of the fertilization with ash in doses: P1 = 0.15 kg P_2O_5 (66 g P), P2 = 0.30 kg P_2O_5 (132 g P), and P3 = 0.45 kg P_2O_5 (198 g P), in recalculation into 9 kg of soil in the pot (respectively: 50, 100 *i*, 150 kg $P_2O_5 \cdot ha^{-1}$) and two variants of the mineral fertilization (NK, NPK). Additionally, the test plant was undergone through mineral fertilization each year in the form of ammonium nitrate (34% N), monocalcium phosphate (40% P_2O_5 and potassium soil (60% K_2O).

The plant material for research was collected in January, after the growing season. The recommended dates for harvesting energy crops are January and February, to provide an optimal balance between maximizing biomass yield while minimizing nutrient offtake and moisture content [29]. The entire aboveground parts of plants were harvested by hand and dried at 60° to air-dry matter. The presented test results are given on a dry mass.

Laboratory studies covered determination of microcomponents (nitrogen, phosphorus, potassium, calcium, and sulfur), the content of heavy metals (such as cadmium, copper, iron, manganese, nickel, lead, and zinc) contained in the dust after combustion of sewage sludge, in the soil material, and crops of test plants. Average soil samples were being created by means of an annual collection of individual samples from four repetitions of each pot. Dry mass was determined in these samples, pH, phosphorus and total potassium, dissolved and digestible, calcium, and heavy metals. The analysis of the physical-chemical composition was conducted (i.e., dry mass and total contents of nitrogen, phosphorus, potassium, calcium, and sulfur) and the gross calorific value

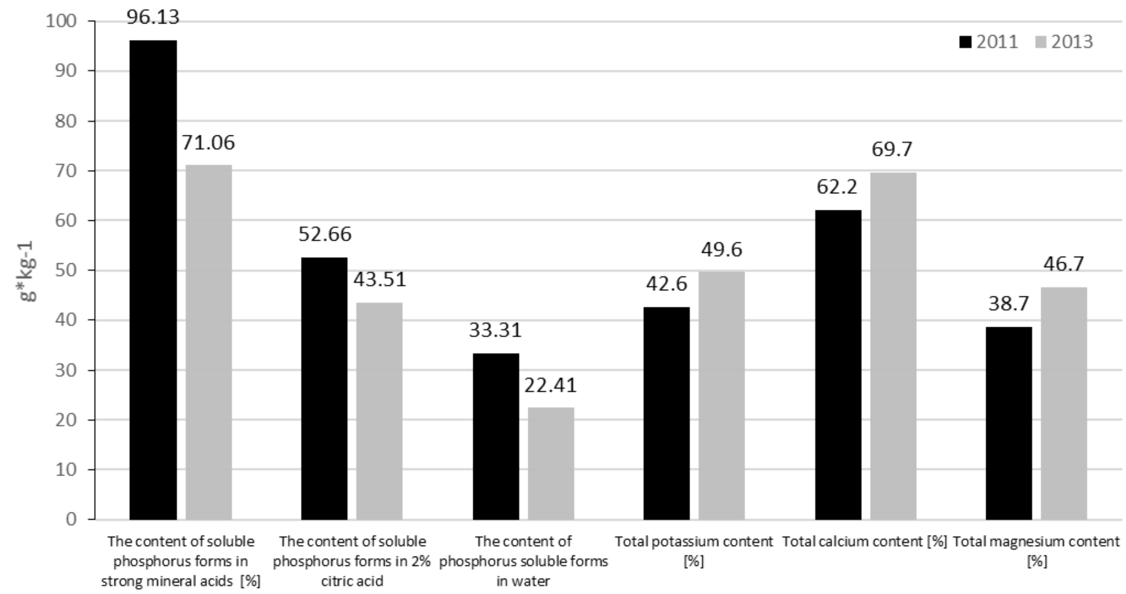

Fig. 1. Chemical composition of sewage sludge ash.

Table 1
Total average content of heavy metals in sewage sludge ash

	Total content (mg kg^{-1} d.m.)					
	Cd	Cu	Mn	Ni	Pb	Zn
2011	4.16	476.2	411.4	74.70	93.00	745.1
2013	2.39	310.3	476.9	18.00	84.30	820.2
Average	3.28	393.3	444.2	46.35	88.65	782.7
Standard deviation	0.88	82.95	32.75	28.35	4.350	37.55

and net calorific value of the crops collected were marked. Medium-size objects samples of dried straw were created. Subsequently, the content of dry mass was determined with the oven-drying method (at 105°C), the total content of nitrogen and S—on an CNS analyzer, phosphorus—with the Barton method. Potassium, calcium, and magnesium were determined with the atomic absorption spectrometer method after prior mineralization of samples in a mixture (3:1) of nitric (V) and chloric(VII) acids.

Statistical studies of the results of test plants were conducted in accordance with statistical programs: FR–Analwar–4.3 and Statistica 10. Student's *t*-distribution was made to compare the results of the research. Data were analyzed at the 0.05 confidence limit.

3. Results and discussion

Obtained amount of the crops with a list of average values for particular years and methods of fertilization is presented in Table 2. In natural conditions, *M. giganteus* may reach the length up to 3 m and its effectiveness from the hectare fluctuates between 15 and 25 t of a dry mass [30]. For the most effective growth, *M. giganteus* requires the climatic regions with an annual sum of rainfalls of 600–1,000 mm and

Table 2
Yielding of the *M. giganteus* [amount of the dry mass of straw (g) gathered from a pot]

Objects	Year				
	2011	2012	2013	$\sum$	$\bar{x}$
NK	20.1	125.0	161.7	306.83	102.28
NPK	19.6	104.0	169.0	292.58	97.53
NK+P1	18.8	134.0	166.5	319.25	106.42
NK + P2	16.8	114.0	121.9	252.70	84.23
NK + P3	20.6	136.0	121.2	277.78	92.59
Average	19.2	123.0	148.1	289.83	96.75
$LSD_{0.05}$	n.s.	n.s.	n.s.	n.s.	n.s.

n.s. = not significant

an average air temperature of 8°C [31,32]. This plant should be sown in the slightly acid soil (pH 5.5–7.5) with the level of groundwater between 50 and 100 cm. *M. giganteus* collects the nutritious substances from a variety of sources, including natural fertilizers, municipal sewage sludge, and compost prepared from these sediments and other waste [32–34]. According to Carver [31], *M. giganteus* requires following fertilization: N = 100, P = 25–40, K = 100–160 kg ha^{-1}.

An average crop of *M. giganteus* collected from one pot in 2011 amounted to 19.2 g d.m. Height of crops in next years was from six to eight time higher than in first year. Throughout the first 2 y, the experiment indicates the most favorable crops of the facilities fertilized with the largest dose of dust (NK + P3). However, in 2013 the same sample was characterized by the smaller plant growth and amounted only to 121.2 g d.m. A similar amount (121.9 g d.m.) of a straw was obtained also from the fertilized items NK + P2. In the last year of the experiment, the largest crops were collected from the fertilized pots NPK (169.0 g d.m.), NK+P1 (166.5 g d.m.), and NK (161.7 g d.m.). By analyzing the average values for each variant of the fertilization, the largest crops (106.42 g d.m.) were observed in the facilities fertilized with dust in the amount of 0.15 kg P_2O_5 per pot (NK+P1). There was 319.25 g d.m. of straw in total collected from them. With addition of the second dose of dust (NK+P2) the smallest crop was obtained from the pot—252.70 g of the dry mass of straw of *M. giganteus* in total. As a result of the ash fertilization applied, the non-significant impact of this waste on the yield increase compared to the control (NPK) was observed.

The results of the average content of nitrogen, phosphorus, potassium, calcium, magnesium, and sulfur marked throughout 3 y of tests in the straw of *M. giganteus*, fertilized with the nitrogen and potassium (NK), mineral fertilizer (NPK), and increasing doses of dust-gravel, were presented in Table 3.

By analyzing the concentration of nitrogen in the straw of the test plant, no positive influence of the dust-gravel fertilization was stated after the combustion of the sewage sludge. The largest concentration of the element in the amount of 5.54 g kg^{-1} d.m. took place on the items fertilized with mineral fertilizer.

In comparison with the items fertilized with NK and NPK, an addition of the waste constituting the substitute of the phosphorus fertilizer, contributed to the increase of the contents of the phosphorus and potassium. The concentration of the phosphorus increased by 107.8% in the items fertilized with NK+P2, whereas the potassium—by 103.5% in the items with addition of NK+P3. However, in the case of calcium contents—an addition of dust-gravel contributed to lowering the concentration of the element. According to Lewandowski et al. [29], *Miscanthus* biomass after mineral fertilization had low concentrations of chloride (0.3–2.1 g kg^{-1}), nitrogen (0.9–3.4 g kg^{-1}), and potassium (3.7–11.2 g kg^{-1}). Christian et al. [35] research report the amount of N fertilizer did not affect phosphorus and potassium content in the established crop at harvest. The results of these studies indicate that SEM fertilization allows for the increase of the content of individual elements.

In the case of the contents of magnesium, addition of the first and second dose of waste after combusting the sewage sludge caused the lowering of its concentration in comparison with the control facilities. However, a maximum amount of the element (1.35 g kg^{-1} d.m.) was marked in the items fertilized with the third, largest dose of dust-gravel.

An average concentration of the sulfur in the straw of *M. giganteus* amounted to 1.34 g kg^{-1} d.m. Addition of dust-gravel affected the content of the element—in comparison with the fertilized item NPK, in the facility of NK+P1 and NK+P3 marked an increase of the concentration of sulfur, respectively, by 120.7% and 118.2%.

The least concentration of macro-components was characterized by the items fertilized with the first dose of dust-gravel (NK+P1)—a sum of the macro-components marked amounted to 18.13 g kg^{-1} d.m. Maximum amount of potassium, calcium, magnesium, and sulfur was marked in the straw of *M. giganteus* fertilized with the third dose of waste (NK+P3). However, the largest summary contents of the analyzed macro-components were marked in the items fertilized with the mineral fertilizer (20.26 g kg^{-1} d.m.).

In the fourth year of a field experiment conducted by Kalembasa and Malinowska [36] the average content of macro-components in the biomass of *Miscanthus sacchariflorus* was marked: 0.056 g P kg^{-1}, 1.62 g K kg^{-1}, 4.65 g Ca kg^{-1}, 0.480 g Mg kg^{-1}, and 0.422 g S kg^{-1}.

Average content of heavy metals from the 3 y experiment, marked in the straw of the test plant, fertilized with different variants of the fertilizers, was listed in Table 4.

In comparison with the control facilities (NK and NPK) in the fertilized pots with the largest dose of waste after combustion of the municipal sewage sludge, an increase of the content of cadmium, iron, and magnesium was observed by 102% and manganese—by 182%. In the samples fertilized by NK+P2, a 128% increase of copper was marked. *M. giganteus* fertilized with the first dose of dust-gravel was characterized with the smallest concentration of cadmium (1.087 mg kg^{-1} d.m.), copper (5.779 mg kg^{-1} d.m.), iron (170.64 mg kg^{-1} d.m.), and zinc (38.06 mg kg^{-1} d.m.). Research conducted by Antonkiewicz et al. [37] indicates a reduced uptake of macronutrients with lower doses of sewage sludge.

A concentration of nickel and lead was reduced with an addition of the wastage being the substitute of the phosphorus fertilizer. In the items fertilized with nitrogen and potassium, the following was marked 1.679 mg Ni kg^{-1} s.m and 4.208 mg Pb kg^{-1} d.m. The contents of nickel marked in the straw of *M. giganteus* amounted to 1.271 mg Ni kg^{-1} d.m. (NK+P2), whereas the concentration of the lead in the items fertilized with NK+P3 was as twice lower as control item NK (2.460 mg kg^{-1} d.m.). Manganese, iron, and zinc content was characterized by a high value of standard deviation (2.515–20.40) which indicates a high variability of the occurrence of these elements in the fertilizers used. A low standard deviation indicates the dose of SSA used did not have a significant effect on the content of macro-components

Table 3
Average content of macro-components determined in the straw of the *M. giganteus* (mg kg^{-1} d.m.)

Component	NK	NPK	NK+P1	NK+P2	NK+P3	Mean	Standard deviation
Nitrogen	4.47	5.54	4.31	4.57	4.40	4.66	0.502
Phosphorus	1.94	1.92	1.95	2.07	1.98	1.97	0.059
Potassium	5.18	5.22	4.59	4.81	5.36	5.03	0.320
Calcium	5.48	5.05	4.63	5.07	5.40	5.13	0.337
Magnesium	1.30	1.32	1.19	1.07	1.35	1.25	0.115
Sulfur	1.26	1.21	1.46	1.35	1.43	1.34	0.107

Table 4
The average content of heavy metals determined in the straw of the *M. giganteus* (mg kg^{-1} d.m.)

Component	NK	NPK	NK+P1	NK+P2	NK+P3	Mean	Standard deviation
Cd	1.191	1.186	1.087	1.129	1.203	1.159	0.049
Cu	7.015	6.042	5.779	7.761	5.869	6.493	0.864
Fe	187.4	190.4	170.6	176.3	190.6	183.1	9.090
Mn	54.53	61.78	91.96	93.33	99.07	80.13	20.40
Ni	1.679	1.513	1.527	1.271	1.430	1.484	0.149
Pb	4.208	3.699	3.636	3.878	2.460	3.576	0.662
Zn	41.83	44.66	38.06	40.63	43.16	41.67	2.515

and trace elements. Likewise in studies conducted by Wierzbowska et al. [38] there was no significant effect of the dose of heavy metal ash content. Results of an experiment conducted by Fernando et al. [39] indicate to low tolerance of *Miscanthus* to Cd, Cu, and Hg contamination of soils.

Miscanthus is used for phytoremediation of contaminated sites in Central and Eastern Europe, as well as in the central US. According to Pidlisnyuk et al. [40] *Miscanthus* has the potential to stabilize and possibly remove and phytoextraction heavy metals from contaminated soils, slowly over time while being grown for its energy value. In some researchers reported biomass growth was higher in the presence of contamination. The level of contaminant substances taken up by aerial biomass growth is small and biomass can be used for energy production [40].

Table 5
Energy value of the *M. giganteus* straw [MJ kg^{-1} d.m.]

Objects	Gross calorific value			Net calorific value		
	2011	2012	2013	2011	2012	2013
NK	16.45	16.44	16.83	15.25	15.29	15.67
NPK	16.40	17.40	16.99	15.21	16.17	15.83
NK+P1	16.58	18.51	17.09	15.38	17.28	15.93
NK+P2	16.36	17.16	17.05	15.17	15.94	15.89
NK+P3	16.38	17.38	17.10	15.18	16.17	15.94
Annual average	16.43	17.38	17.01	15.24	16.17	15.85
Total average	16.94			15.75		

Table 5 and Fig. 2 present the gross calorific value and the net calorific value marked in the straw of *M. giganteus* in particular years of duration of the pot experiment.

The largest value of gross calorific value occurred within items fertilized with the first dose of dust-gravel and amounted to an average 17.39 MJ kg^{-1} d.m. of straw. Also, in the case of the net calorific value, the most energetic straw was collected from the pots fertilized with NK+P1 (16.20 MJ kg^{-1} d.m.). Similarly, as in Kołodziej et al. [34] experiment, the net calorific value of *M. giganteus* biomass (after fertilization of sewage sludge) was characterized by relatively high net calorific value (16.0–16.8 MJ kg^{-1} d.m.). In other variants of fertilization, the beneficial influence of addition of phosphorus was observed on the increase (on average by 103%) of the net calorific value and gross calorific value in comparison to the item controlled fertilized with nitrogen and potassium.

The least energetic crops of the *M. giganteus* were collected in the first year of the duration of the experiment whereas the largest ones in 2012—when the increase of gross calorific value was recorded and the net calorific value 106%. An average (3 y) net calorific value and the gross calorific value of the analyzed plant amounted to, respectively, 15.75 and 16.94 MJ kg^{-1} d.m.

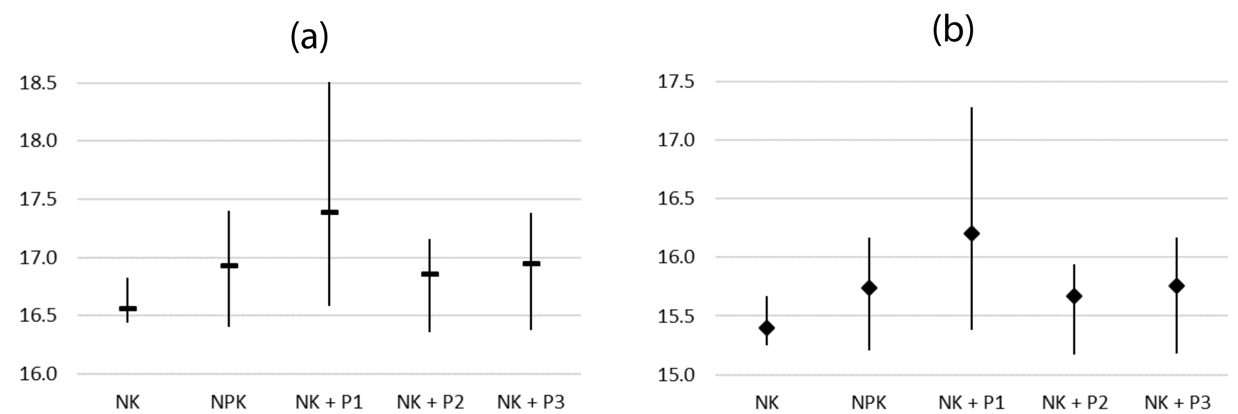

Fig. 2. Minimum, maximum and average gross calorific value (a) and net calorific value (b) of the *M. giganteus*, determined throughout 3 y of the experiment (MJ kg^{-1} d.m.).

4. Conclusions

The results of the conducted pot experiment indicate the possibility of using the dust-gravels created in the process of combustion of municipal sewage sludge as the phosphorus fertilizer for the growth of the *M. giganteus* as the energetic plant. The waste obtained from the combustion of municipal waste in a dry mass contained on average: 8.4% phosphorus, 4.6% potassium, 6.6% calcium, and 4.3% magnesium (Fig. 1).

The physical-chemical analysis of crops collected throughout 3 y of the examination did not show any positive influence of fertilization with dust on the concentration of nitrogen in the straw of *M. giganteus*, and a significant increase of the concentrations of the phosphorus in comparison with the control item. In the items fertilized with the largest dose of the dust, the increase of the contents of potassium was marked by 103.5% in comparison with the items fertilized with nitrogen and potassium. There was no inhibition of crop yield due to the concentration of determined heavy metals. Heavy metals marked in the straw of the test plant with the total amount of 1.758 ± 0.046 mg kg^{-1} d.m., corresponded to the standards for the industrial plants.

The tests conducted show the beneficial influence of the application of the fertilizer in the form of dust-gravel on the energetic values of the *M. giganteus*. Maximum values of the gross calorific value and net calorific value were marked in the crops of 2012 and, respectively, amounted to 18.51 and 17.28 MJ kg^{-1} d.m. of straw.

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