CONTENTS OF HEAVY METALS IN PLANTS
AND SOIL FERTILIZATION OF ASH
FROM SEWAGE SLUDGE COMBUSTION

Abstract: In the thesis assessed was fertilizing value of ashes from municipal sewage sludge combustion in grate furnace in Pomorzany Sewage Treatment Plant in Szczecin. The ash was treated as a substitute of phosphorus from phosphatic fertilizer because general content of this component exceeded 20 % of P2O5. In 2011–2012 a field experiment was conducted on a light soil. The scheme of the experiment included five objects of fertilizing corn for seeds and spring rape with mineral fertilizers (NK, NPK) and mineral fertilizers together with increasing doses of ash from municipal sewage sludge combustion (NK + P1, NK + P2, NK + P3). Substitution of phosphorus in a dose of NPK from superphosphate enriched with phosphorus from ash (P1, P2, P3) from municipal sewage sludge combustion did not cause any differences in the volume of corn seeds or corn stove or rapeseed straw, and in the object NK + P3 received were considerately richer crops with spring rape seeds than from any other objects. Ash caused an increase in content of cadmium and nickel in seeds and straw, as for lead only in corn seeds, content of cadmium and lead in spring rape seeds and cadmium in straw. After two years of applying nitrogen and potassium as mineral fertilizers and ashes from municipal sewage sludge combustion, in comparison with exclusive fertilizing corn and spring rape with mineral fertilizers, an increase of lead in general soil content was observed, and general content of cadmium and nickel as well as their dissoluble form in 1 molHCl · dm−2 in soil from all objects was similar.

Keywords: municipal sewage sludge, ash from sludge, Cd, Ni, Pb, plants, soil

Introduction

The intention to implement a prohibition to store municipal sewage sludge at municipal landfill site forces to search for a new solutions to manage it, taking into

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consideration the following aspects: ecological, technical and economic. Bien et al [1] state that sludge, after having been processed, should come back to the environment, mostly because its agricultural use is recommended to small and medium treatment plants, and for big treatment plants other solutions should be searched for. Agricultural use of municipal sewage sludge can still have a major meaning when it comes to light soils [2] and with fertilizing energy plants [3].

According to Pajak [4] strategy of sewage sludge management with thermal ways, acknowledged by big and medium national agglomerations, is fully legitimate. According to the author, Switzerland is an example confirming this direction because 97% of sludge there is processed thermally, and it is not used agriculturally at all. In Germany ca 20% is managed agriculturally, yet simultaneously ca 50% is processed thermally, at the same time in Poland only 1% of sewage mass was a subject to such a process.

Thermal processing of sewage sludge by combustion is not neutral for the environment because during combustion gasses are produced which get contaminated with speck, carbon monoxide, nitrogen oxide, sulphur oxide as well as atypical mineral acids, heavy metals and hydrocarbons [5]. Sewage sludge combustion generates a new kind of waste – ash – which needs to be managed. From Kosior-Kazubek and Karwowska’s [6] researches it results that ash from sewage sludge combustion can be used as an active component substituting a part of cement in mortars.

Dependent on chemical composition of sludge, as well as technical solutions in its thermal utilisation, ash with different content of macro- and microelements, including heavy metals, is received. The thesis touches upon an attempt to assess fertilizing value of ash from sewage sludge combustion in municipal grate furnace in “Pomorzany” Treatment Plant in Szczecin. Ash was treated as a substitute of phosphorus from phosphatic fertilizer because the general content of this component exceeded 20% of P2O5. Moreover, phosphorus among basic macroelements (N, P and K) is the most expensive one and its alternative sources should be searched for.

**Material and methods**

In the time period of 2011–2012 in Agricultural Experimental Station in Lipnik, which is located near Stargard Szczeciński, tests with the use of ash from municipal sewage sludge combustion for fertilizing plants were conducted. Field experiment was performed on rusty soil, incomplete, received from clayey sand which is light and dusty, planted on average depth with light clay (valuation class IVb, good rye complex – 5, soil category – light soil). Before the experiment started soil pH was slightly acidic (5.8 pH in 1 molKCl • dm–3), total content of carbon amounted to 8.59 g • kg–1, nitrogen 0.84 g • kg–1, easily assimilated form was high for phosphorus, and low for potassium and magnesium.

Total content of trace elements in soil amounted to: 254.7 mgMn, 62.31 mgZn, 6.439 mgCu, 8.320 mgNi, 0.554 mgCd and 5.215 mgPb • kg–1 d.m.

Scheme of tests included five forms (in 4 repetitions) of fertilizing with mineral fertilizers and ash from municipal sewage sludge combustion: NK, NPK, NK + P1 (P1 – 1 ash dose), NK + P2 (P2 – 2 ash dose), NK + P3 (P3 – 3 ash dose) corn for seeds.
and spring rape. Pre-sowing mineral fertilizing with nitrogen under both plants was used as ammonium sulphate (20 % of N), and topdressing fertilizing with ammonium sulphate (34 % of N). Phosphorus (enriched superphosphate 40 % of P₂O₅) and potassium (potassium salt 60 % of K₂O) and in mineral fertilizers were used yearly only for pre-sowing fertilizing. Ash from municipal sewage sludge combustion, which constitutes a substitute for phosphatic fertilizer, comes from “Pomorzany” Sewage Treatment Plant in Szczecin. In both years of research, the same ash was applied for pre-sowing fertilizing and its chemical composition was shown in Table 1.

### Table 1

<table>
<thead>
<tr>
<th>Content of forms soluble in strong mineral acids in 2% citric acid in water</th>
<th>Total content of</th>
<th>K</th>
<th>Ca</th>
<th>Mg</th>
<th>Cd</th>
<th>Cu</th>
<th>Mn</th>
<th>Ni</th>
<th>Pb</th>
<th>Zn</th>
</tr>
</thead>
<tbody>
<tr>
<td>[g kg⁻¹]</td>
<td>[mg kg⁻¹]</td>
<td>% P₂O₅</td>
<td>g kg⁻¹</td>
<td>g kg⁻¹</td>
<td>g kg⁻¹</td>
<td>g kg⁻¹</td>
<td>g kg⁻¹</td>
<td>g kg⁻¹</td>
<td>g kg⁻¹</td>
<td>g kg⁻¹</td>
</tr>
<tr>
<td>22.05</td>
<td>12.08</td>
<td>7.64</td>
<td>42.6</td>
<td>62.2</td>
<td>38.7</td>
<td>4.16</td>
<td>476.2</td>
<td>411.4</td>
<td>74.7</td>
<td>93.0</td>
</tr>
</tbody>
</table>

Experiment plants in both years were corn and spring rape, which in the first year was cultivated on soil on which winter wheat had been cultivated before, and in the second year spring rape was cultivated after corn, and then after spring rape was corn and after corn spring rape again. Corn for seeds (varieties DKC 3016 – FAO 240) was sown in the first year on 28.04., and in the second on 05.05. in the number of 75 thousand seeds per ha. Under corn pre-sowing fertilizers were used as following: 40 kg N, 50 kg P₂O₅, 110 kg K₂O · ha⁻¹, and with further doses of ash applied was 50, 100 and 150 kg of P₂O₅ · ha⁻¹. For topdressing fertilizing (in 6–7 leaf stage) 40 kg N · ha⁻¹ was used. During vegetation, corn was two times protected against pests and one time weeds were destroyed. Harvesting of all corn plants was done manually (18.10.2011 and 04.10.2012), and then corn cobs were separated from other parts of the plants. After cobs being treshed manually, mass of corn seeds and straws was determined, to straws stems, rachis and seed covers from cobs.

Spring rape (‘Lariss’ variety in 2011 and ‘Markus’ variety in 2012) was sown in the first year on 20.04., and in the second year on 10.04. For spring rape the following pre-sowing fertilizing was used: 50 kg N, 50 kg P₂O₅, 120 kg K₂O · ha⁻¹, and in further doses of ash applied was 50, 100 and 150 kg of P₂O₅ · ha⁻¹. For topdressing fertilizing (in the green bud stage) applied was 50 kg of N · ha⁻¹. Moreover, during vegetation spring rape was sprayed against rape beetle and weeds were also destroyed. Spring rape harvesting was done by a combine harvester on 26.08.2011 and 19.08.2012.

After harvesting, the mass of crop was determined, samples were taken and initially dried in the temperature of 60 °C. Content of dry mass was marked using oven-drying method and total content of Cd, Ni, and Pb was marked with an ASA method in the dried samples, after prior mineralization of the samples in mixture (3:1, v/v) of nitric acid and chloric(VII) acid. After harvesting of spring rape and corn in 2012, samples of
soil were taken from four repetitions of each fertilizing object, the samples were mixed and average object samples were received. Prior to marking the total content of heavy metals in soil, it was mineralized in the same acids but mixed in 1:1 relation. Variation analysis of corn and spring rape crop results was done in randomized block system according to FR-ANALWAR package. Multiple comparisons of the average were done by using Tukey’s procedure with significance level of $\alpha \leq 0.05$. Standard deviation for content of heavy metals in plants and soil was determined according to STATISTICA 10 program.

**Results and discussion**

In Poland, for over a dozen years, a dynamic increase in agricultural areas cultivation of corn for seeds has been observed, which can be associated with the possibility of using seeds for bioethanol production. In 2012, participation of this plant in grain sown structure constituted 7% (539 thousand ha), and an average seed crop constituted $\approx 6\,\text{Mg} \cdot \text{ha}^{-1}$ [7].

In the discussed researches in both years a richer crop of corn seeds was received – 75 and 95%, respectively (Table 2) when compared to the national average.

| Seeds and straw crop of corn and spring rape [Mg - ha$^{-1}$] |
|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|
| Objects         | Maize           |                 |                 | Spring rape     |                 |                 |
|                 | corn      | straw   | seeds      | straw   | corn      | straw   | seeds      | straw   |
| NK              | 10.13     | 11.99   | 11.06     | 10.06   | 11.21    | 10.64   | 0.51      | 1.67    | 1.09    | 6.25   | 5.84   | 6.05   |
| NPK             | 10.33     | 11.08   | 10.71     | 9.21    | 11.73    | 10.47   | 0.50      | 1.76    | 1.13    | 6.42   | 5.84   | 6.13   |
| NK-P1           | 11.01     | 12.23   | 11.62     | 9.65    | 12.14    | 10.90   | 0.51      | 1.75    | 1.13    | 6.59   | 5.67   | 6.13   |
| NK-P2           | 10.29     | 11.21   | 10.75     | 9.45    | 11.77    | 10.61   | 0.55      | 1.80    | 1.17    | 6.17   | 5.67   | 5.92   |
| NK-P3           | 10.68     | 11.93   | 11.31     | 9.90    | 12.26    | 11.08   | 0.64      | 1.84    | 1.24    | 6.67   | 5.92   | 6.30   |
| Average         | 10.49     | 11.69   | 11.09     | 9.17    | 11.82    | 10.50   | 0.54      | 1.76    | 1.15    | 6.42   | 5.79   | 6.11   |
| LSD$_{0.05}$    | n.s.*     | n.s.    | n.s.      | n.s.    | n.s.     | n.s.    | 0.075     | 0.077   | 0.049   | n.s.   | n.s.   | n.s.   |

* n.s. – non significant.

In respective years, as an average crop from the two years of research was not particularly diversified between objects fertilized with mineral fertilizers (NK and NPK) and objects fertilized with mineral fertilizers with the addition of ash from municipal sewage sludge combustion (NK-P1, NK-P2 and NK-P3). The richest average crop of seeds was harvested from the object where nitrogen and potassium were used as mineral fertilizers and phosphorus in the form of ash (P1) in a dose of 50 kg of P$_2$O$_5$ - ha$^{-1}$ (11.62 Mg - ha$^{-1}$), on other objects crops of seeds was poorer ranging from 0.31 to 0.87 Mg - ha$^{-1}$. Applied fertilizing with mineral fertilizers and ash from municipal sewage sludge combustion did not differentiate crops of corn straw meaningfully (Table 1), and...
the richest average crop of corn was harvested from object NK-P3 (11.08 Mg \cdot ha^{-1}), the poorest from object NPK (10.47 Mg \cdot ha^{-1}). Average crop of corn harvested in the research by Burczyk [8] resulted 9.96 Mg \cdot ha^{-1} in seeds, and 22.10 Mg \cdot ha^{-1} in straw. In a research by Meller and Bilende [9], where ash from biomass combustion was used for fertilizing corn, average crop of fresh corn mass totalled 75.0 Mg \cdot ha^{-1}. The best effects of plants’ growth were achieved with the dose of 60 Mg \cdot ha^{-1} of ash.

In the last years also agricultural area for cultivation of spring rape and agrimony has increased and in 2012 constituted 6.9 %. Still, spring rape represents minor percentage in agricultural area of oil plants, due to its poorer cropping than winter rape.

In 2011, in the following research received was on average ca 3 times poorer crop of spring rape seeds (0.54 Mg \cdot ha^{-1}) than in 2012 (1.76 Mg \cdot ha^{-1}), which was a result of long-lasting drought in the flowering period and seeds forming time of spring rape. The richest crop of seeds in this year was harvested from object NK-P3 (0.64 Mg \cdot ha^{-1}), which was highly different from crops from other objects. In the second year of research, a similar dependence was observed, because from the above mentioned object, the richest crop of seeds was harvested (1.84 Mg \cdot ha^{-1}), but it was not significantly richer than crops from objects NK-P1, NPK and NK. Average crop of seeds from the two years of research from object NK-P3 was the richest and indeed exceeded crops from other objects by 8.7 %. Applied fertilizers did not differentiate crops of spring rape straw significantly, taking into consideration that the richest average crop, similarly to seeds, was received from object NK-P3 (6.30 Mg \cdot ha^{-1}).

Fertilizing with mineral fertilizers and ash from municipal sewage sludge combustion, in comparison to exclusive fertilizing with mineral fertilizers, differentiated content of analysed heavy metals in most cases (cadmium, nickel and lead) in seeds and straw of corn and spring rape. Content of heavy metals in biomass of both plants was presented as weighted average from the two years of research (Table 3).

<table>
<thead>
<tr>
<th>Objects</th>
<th>Maize</th>
<th></th>
<th>Spring rape</th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>corn straw</td>
<td>seeds straw</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Cd</td>
<td>Ni</td>
<td>Pb</td>
<td>Cd</td>
<td>Ni</td>
<td>Pb</td>
</tr>
<tr>
<td>NK</td>
<td>0.325</td>
<td>0.380</td>
<td>2.45</td>
<td>0.571</td>
<td>0.278</td>
</tr>
<tr>
<td>NPK</td>
<td>0.306</td>
<td>0.372</td>
<td>2.49</td>
<td>0.616</td>
<td>0.324</td>
</tr>
<tr>
<td>NK-P1</td>
<td>0.472</td>
<td>0.341</td>
<td>3.13</td>
<td>0.724</td>
<td>0.315</td>
</tr>
<tr>
<td>NK-P2</td>
<td>0.405</td>
<td>0.547</td>
<td>3.01</td>
<td>0.559</td>
<td>0.528</td>
</tr>
<tr>
<td>NK-P3</td>
<td>0.460</td>
<td>0.646</td>
<td>3.16</td>
<td>0.652</td>
<td>0.424</td>
</tr>
<tr>
<td>Average</td>
<td>0.393</td>
<td>0.456</td>
<td>2.85</td>
<td>0.624</td>
<td>0.374</td>
</tr>
<tr>
<td>Standard deviation</td>
<td>0.077</td>
<td>0.133</td>
<td>0.350</td>
<td>0.067</td>
<td>0.102</td>
</tr>
</tbody>
</table>
Content of cadmium in corn seeds from all objects fertilized with mineral fertilizers (NK) and ash from municipal sewage sludge combustion was significantly higher (on average by 45.8 %) than in seeds from objects NPK. An increase in content of nickel in seeds from objects NK + ash, when compared to NPK, was observed after application of two bigger doses of ash (P2 – 100 and P3 – 150 kg of P2O5 · ha⁻¹) by 47.0 and 73.7 %, respectively. All doses of ash with NK, in relation to NPK increased (on average by 24.5 %) the content of lead in corn seeds. The highest content of cadmium was observed in corn straw harvested from object NK-P1 (0.724 mgCd · kg⁻¹ d.m.). In straw from other objects, content of cadmium was lower (by 9.9 to 22.8 %). For more significant increase in content of nickel in corn straw, similarly to seeds, in comparison to NPK, two bigger doses of ash had influence, which caused an increase in content of this metal by 63.0 and 30.9 %. Fertilizing with ash from municipal sewage sludge combustion did not differentiate significantly the content of lead in corn straw. Average content of lead in objects with ash totalled 2.40 mgPb · kg⁻¹ d.m., and in straw from object NPK was higher by 13.0 % when compared to objects fertilized with ash. In the research by Meller and Bilende [9] average content for nickel from objects fertilized with ash from biomass combustion in corn seeds amounted to 3.78, for cadmium 0.37 mg · kg⁻¹, and for straw 9.12 and 0.44 mg · kg⁻¹, respectively.

In seeds of spring rape from objects fertilized with nitrogen and potassium as mineral fertilizers, and phosphorus as ash from municipal sewage sludge combustion an increase in content of cadmium, in relation with NPK, was observed on average by 22.3 %, taking into consideration that the highest advance (by 27.2 %) was caused by the slightest dose of ash (Table 3). Content of nickel in spring rape seeds was slightly differentiated by mineral fertilizers and ash, and average content oscilated from 2.63 to 2.86 mgNi · kg⁻¹ d.m. On the other hand, content of lead, as a result of fertilizing NK and ash in comparison to NPK, increased on average by 9.0 %. In spring rape straw, content of cadmium increased along with the increase of ash dose, and average advance in comparison to NPK amouted to 22.8 % (Table 3). Increase in content of nickel in spring rape straw on the objects NK + ash in relation to objects NPK was observed after second and third dose of ash was applied, but it was slight (on average ca 5.0 %). Content of lead in spring rape straw was slightly differentiated and oscilated from 2.51 to 2.68 mgPb · kg⁻¹ d.m.

Table 4 presents an average total content of heavy metals and their forms dissolved in 1 molHCl · dm⁻³ in soil sampled from 0–20 cm layer from objects fertilized with mineral fertilizers and ash in the second year of corn and spring rape cultivation. In relation to object fertilized with mineral fertilizers (NK and NPK) in soil from objects NK + ash, an average increase in total content of cadmium reached barely 2.81 %. A slight increase in cadmium and nickel content in soil is a result of application of those small amounts of heavy metals to soil with ashes. And average content of cadmium forms dissolved in 1 molHCl · dm⁻³ in objects NK + ash was higher only when compared to content determined in soil fertilized with NK. Average total content of nickel in soil from individual fertilized objects was barely differentiated (from 7.33 to 7.65 mgNi · kg⁻¹ d.m.), similarly to forms of nickel dissolved in 1 molHCl · dm⁻³.
(from 0.611 to 0.681 mgNi \cdot kg^{-1} d.m.). A slight increase of cadmium and nickel in soil is a result of application of small amounts of those heavy metals to the soil with ashes.

A major increase in total content of lead in fertilized soil with NK and ash was observed, which in relation to object NK reached on average 23.7 %, and in relation to NPK 12.3 %. Content of lead forms dissolved in 1 mol HCl \cdot dm^{-3} in soil from objects NK + ash was approximate to content from object NK, and lower than in soil fertilized with NPK. The highest average participation of heavy metals forms dissolved in 1 mol HCl \cdot dm^{-3} in its total content in soil was observed in case of lead (47.9 %), lower for cadmium (28.7 %), and the lowest for nickel (8.52 %).

According to Regulation of Minister of Environment in case of waste catalogue [10] grated slags and ashes from municipal sewage sludge combustion can be classified as combustion slags and ashes, code 19 01 12 (other than mentioned in 19 01 11). Those, however, cannot be recycled R10 according to Regulation of Minister of Environment in case of the process of recycling R10 [11].

Usefullness of ashes from minicipal sewage sludge combustion for agricultural use in literature is estimated on the basis of their psychic-thermal properties and chemical composition. Information provides that ashes are poor in nitrogen and rich in phosphorus, calcium and magnesium, but at the same time they can have higher content of heavy metals [6, 12]. Ash used in own research did not contain particularly high contents of studied heavy metals (Cd, Cu, Ni, Pb and Zn) and did not exceed admissible contents determined in Regulation of Minister of Environment on municipal sewage sludge [13]. Phosphorus, which can be found in ash, can have an important fertilizing meaning, but which has been barely used as fertilizer in recent years. Ottosen et al [14] research gives evidence that ash from municipal sewage sludge combustion may be a significant source of phosphorus, and Wzorek et al [15] maintain the same about ashes from co-combustion of waste from meat processing industry and municipal sewage
sludge. The last mentioned authors, at 1:1 relation of both components, received ash of over 10% of P content.

Conclusions

1. Substitution in the dose of NPK of phosphorus from superphosphate enriched with phosphorus from municipal sewage sludge combustion ash did not cause any significant differences in the crop volume of corn and spring rape seeds and straw. However, average crop of spring rape seeds from object NK with the highest dose of phosphorus from ash significantly exceeded crops from other objects.

2. Mineral fertilizing with nitrogen and potassium as mineral fertilizers and ash from municipal sewage sludge combustion caused an increase in content of cadmium and nickel in seeds and straw, lead content only in corn seeds, content of cadmium and lead in spring rape seeds, and cadmium in straw.

3. After two years of applying nitrogen and potassium as mineral fertilizers and ash from municipal sewage sludge combustion, in comparison to exclusive fertilizing of corn and spring rape with mineral fertilizers, an increase in total content of heavy metals in soil was observed, and total content of cadmium and nickel, as well as their forms dissolved in 1 mol HCl · dm⁻³ was on similar level in soil from all objects.

Acknowledgments

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References


ZAWARTOŚĆ METALI CIĘŻKICH W ROŚLINACH I GŁEBIE NAWOŻONEJ POPIOŁEM ZE SPALANIA OSADÓW ŚCIEKOWYCH

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Abstrakt: W pracy podjęto próbę oceny wartości nawozowej popiołu pochodzącego ze spalania w piecu rusztowym komunalnych osadów ściekowych w Oczyszczalni „Pomorzany” w Szczecinie. Popiół potraktowano jako substytut fosforu z nawozów fosforowych, gdy ogólna zawartość tego składnika przekraczała 20 % P₂O₅. W latach 2011–2012 przeprowadzono doświadczenie polowe na glebie lekkiej. Schemat badań obejmował pięć wariantów nawożenia kukurydzy na ziarno i rzepaku jarego nawozami mineralnymi (NK, NPK) oraz nawozami mineralnymi ze wzrastającymi dodatkami popiołu ze spalania komunalnych osadów ściekowych (NK + P₁, NK + P₂, NK + P₃). Zastąpienie w dawce NPK fosforu z superfosfatu wzbogaconego fosforem z popiołu (P₁, P₂, P₃) ze spalania komunalnych osadów ściekowych nie spowodowało istotnych różnic w wielkości plonu ziarna i słomy kukurydzy oraz słomy rzepaku jarego, a na obiekcie NK + P₃, uzyskano istotnie większy plon nasion rzepaku niż z pozostałych obiektów. Stosowanie popiołu skutkowało zwiększoną zawartością kadmu i niklu w ziarnie i słomie, a ołowiu tylko w ziarnie kukurydzy oraz zawartości kadmu i ołowiu w nasionach rzepaku jarego i nasion słomy. Po dwóch latach stosowania azotu i potasu w postaci nawozów mineralnych i popiołu ze spalania komunalnych osadów ściekowych, w porównaniu z wyłącznym nawożeniem kukurydzy i rzepaku jarego nawozami mineralnymi, odnotowano zwiększenie całkowitej zawartości ołowiu w glebie, natomiast całkowita zawartość kadmu i niklu oraz ich form rozpuszczalnych w 1 mol HCl - dm⁻³ w glebie wszystkich obiektów była podobna.

Słowa kluczowe: komunalne osady ściekowe, popiół z osadów, Cd, Ni, Pb, rośliny, gleba