

Received 15.03.2017
Reviewed 14.05.2017
Accepted 29.06.2017A – study design
B – data collection
C – statistical analysis
D – data interpretation
E – manuscript preparation
F – literature search

Acute toxicity of experimental fertilizers made of blood meal, spent coffee ground and biomass ash

Tomasz CIESIELCZUK¹⁾ ABCDE ✉, Czesława ROSIK-DULEWSKA²⁾ ADEF,
Joanna POLUSZYŃSKA³⁾ ABDF, Irena SŁAWIŃSKA³⁾ BF

¹⁾ Opole University, Department of Land Protection, ul. Oleska 22, 45-052 Opole, Poland; e-mail: tciesielczuk@uni.opole.pl

²⁾ Polish Academy of Sciences, Institute of Environmental Engineering, ul. M. Skłodowskiej-Curie 34, 41-819 Zabrze, Poland; e-mail: czeslawa.rosik-dulewska@ipis.zabrze.pl

³⁾ Institute of Ceramics and Building Materials, ul. Oświęcimska 21, 45-641 Opole, Poland; e-mail: j.poluszynska@icimb.pl, i.slawinska@icimb.pl

For citation: Ciesielczuk T., Rosik-Dulewska Cz., Poluszyńska J., Sławińska I. 2017. Acute toxicity of experimental fertilizers made of blood meal, spent coffee ground and biomass ash. *Journal of Water and Land Development*. No. 34 p. 95–102. DOI: 10.1515/jwld-2017-0042.

Abstract

The study presents the results of research on the acute toxicity of a fertilizer formulas made of spent coffee ground (SCG) with addition of ash from low-temperature combustion of biomass or ash with an admixture of magnesium sulphate and blood meal. The experimental fertilizer formulas included also rape oil used as a plasticizer for controlling the nutrients release from the fertiliser. Mustard (*Sinapis alba* L.), oats (*Avena sativa* sp. L.), cucumber (*Cucumis sativus* L.) and cress (*Lepidium sativum* L.) were used as test plants species in the experiment. The toxicity tests were performed using a standard procedure of 72 h with the use of Phytotoxkit microbio-test and fertilizer application of 2.5; 5 and 10% (v/v). The obtained results indicated an increase of acute toxicity for all tested plant species, proportionally to the applied doses of the fertilizer. During the 72 h period, the strongest inhibition of seedling growth was recorded in samples consisting of 10% of the tested fertilizers, particularly when they showed considerable level of salinity or low pH values. From the tested plant species, cress (*Lepidium sativum* L.) turned out to be the most sensitive to the applied fertilizers, the least was cucumber (*Cucumis sativus* L.) for which only a small inhibition of root system growth was observed. The inhibited growth of roots could be attributed to a reduced oxygen access and excessive salinity of the substratum caused by the applied additives.

Key words: acute toxicity, blood meal, organic fertilizer, spent coffee ground

INTRODUCTION

The amount of waste generated per capita in Poland is relatively low, on the level of 286 kg·yr⁻¹ in 2015 while the corresponding average value for the 28 EU Member States was 476 kg out of which 50% could be biodegradable [Eurostat undated]. This fraction of municipal waste is a potential valuable source of organic matter which could be used for fertilizing purposes, after composting and/or fermentation [KOR-

NER *et al.* 2008]. Selective collection of biodegradable waste at source creates an opportunity of its processing including such options as composting, biogas production or quality fertilizer production. It refers also to waste from the production of beverages made of coffee grains. Due to an increasing consumption of coffee the amount of this waste is significant [MUS-SATTO *et al.* 2011].

Yearly coffee consumption in Poland constantly increases and is currently on the level of 3 kg per cap-

ita. This amount is however several times lower compared to Scandinavian countries. Spent coffee ground (SCG) contains a broad range of valuable organic compounds such as tannin, cellulose, hemicellulose, polyphenols and organic acids which make it suitable for the production of biocarbon and biodiesel as well as a sorbing agent or for composting purposes [CAETANO *et al.* 2014; PUJOL *et al.* 2013]. Attempts have also been made to produce alcohol from SCG, however this process requires high energy consumption [SAMPAIO *et al.* 2013]. SCG processing to fertilizer without an expensive and time consuming composting is a promising alternative, however due to the phytotoxic character of spent coffee ground it is necessary to determine a maximum dose which will not cause a negative effect on plants [MUSSATTO *et al.* 2011]. SCG based fertilizers could be used for organic crop production for which a maximum dose of nitrogen on the level of 170 kg·ha⁻¹ is recommended [CIR(EU) No 354/2014].

The goal of the study was to determine acute toxicity of experimental fertilizers made of spent coffee ground modified by an addition of ash from low-temperature thermal biomass processing and other additives such as expired rape oil. It was used as both plasticizer and an agent slowing down the release of ions into soil solution. Granulated fertilizers produced without cellulose and collagen addition were used for the tests. They are previously discussed in more details as slow-release tableted fertilizers [CIESIELCZUK *et al.* 2015]. It should be noted, that the investigated experimental fertilizers were tested for the first time, therefore no data had been published before in the world literature concerning the proposed crops promoting formulas. Similar research on the application of organic and mineral waste as fertilizers was carried out for vermicomposted manure, sewage sludge and ash from coal combustion [GOSWAMI *et al.* 2013; ROSIK-DULEWSKA *et al.* 2008; 2016]. Granulated fertilizers were also made of a mixture of fly ash from wood and peat combusted in a local power plant and stabilized sewage sludge [PESONEN *et al.* 2016].

The key components of the organic matter include: polysaccharides, proteins and fats which ensure a controlled release of biogens from the decomposing organic matter [BALLESTEROS *et al.* 2014; MUSSATTO *et al.* 2011]. The content of fats in SCG is high and amounts to 15% by average [MUSSATTO *et al.* 2011]. However, research shows that the content of fats is determined by the way in which the coffee is brewed.

MATERIALS AND METHODS

MATERIALS

Spent coffee ground (SCG) from the preparation of coffee beverages (under the pressure of 15 atmospheres) and ash from the thermal processing of oak

biomass (wood with bark) were used for the tests. Chemically pure magnesium sulphate (MS) (POCH) and/or blood meal (BM) were added to the experimental formulas [ROY *et al.* 2013]. Rape oil (O) was used as plasticizing agent. To produce ash, oak wood was combusted in an open furnace at the temperature of 600°C for 3 h. The produced material was additionally combusted at the same temperature by 1 hour in a muffle furnace [CIESIELCZUK *et al.* 2015]. The obtained ash (A) was sieved then through a 2 mm diameter mesh sieve and dried to solid mass. Addition of proteins in a form of blood meal and plant fat provides considerable amounts of chelated iron which is indispensable for a proper synthesis of chlorophyll. The admixture of rape oil stabilizes the fertilizer granules while serving as a source of organic carbon.

Upon initial tests, 2 batches of granulated fertilizer were selected for further testing: K11 and K14, each of a different formula and purpose (Tab. 1).

Table 1. Mass proportions of components in fertilizer tablets

Fertilizer	Spent coffee grounds	Ash	Magnesium sulphate	Blood meal	Gelatin	Rape oil
K11	9	1	0	0	1	1
K14	8	0.3	0.7	1	1	1

Source: own elaboration.

GRANULATED FERTILIZER PRODUCTION METHOD

Spent coffee ground (SCG) dried to solid mass was mixed with ash A. The mixture was treated with the expired rape oil and thoroughly mixed. Magnesium sulphate (MS) and blood meal were added to K14 fertilizer followed by a hot gelatine solution to bind the components. The components were thoroughly mixed and pressed through a matrix with a mesh size of 2 mm in diameter. The produced granules were cooled down and dried in room temperature.

Organic matter content in the produced fertilizer was determined by gravimetric method as loss of ignition after burning in 600°C for 3 h. pH value was determined in H₂O and 1M KCl using the solutions of 1:10 m·v⁻¹. The contents of organic carbon, nitrogen, hydrogen and sulphur were determined by Elementar CHNS Vario Macro Cube analyzer. Phosphorus was determined by a titration method with chinoline phospho-molybdate complex after dry mineralization with sulphur acid. Sodium, potassium and calcium contents were determined in digest solutions after digestion with *aqua-regia* by FES method using BWB XP apparatus [CIESIELCZUK *et al.* 2015]. Magnesium content was determined by atomic absorption spectroscopy using iCE Thermo 3500 apparatus after microwave digestion with *aqua-regia*. The contents of available phosphorus and potassium were determined according to Polish norm PN-R-04024:1997.

ACUTE TOXICITY TESTS

The acute toxicity of the investigated experimental fertilizers was determined using germination test. The following plant species were used in the test: mustard (*Sinapis alba* L.), oats (*Avena sativa* sp. L.), cucumber (*Cucumis sativus* L. 'Krak' cultivar) and cress (*Lepidium sativum* L.). Control test plates (Phytotoxkit microbiotest) were filled with a substratum made of de-acidified peat and outwash sand mixed in proportion 1/1 (v/v). For the experimental test plates the substratum was made of the peat-sand mixture with addition of K11 and K14 fertilizers milled and sieved on a sieve with a mesh diameter of 1mm. For comparison, similar tests were additionally made for a peat-sand substratum with the addition of milled SCG. The substrata were moisturized with water of the following parameters: pH 7.06, EC 0.239 mS·cm⁻¹, mineralization 180 mg·dm⁻³, and ions content: HCO₃⁻ 131,1; F⁻ 0,07; Mg²⁺ 5,62; Ca²⁺ 41,7; Na⁺ 9,65 mg·dm⁻³. The toxicity tests were made in three replicates for each plant species and fertilizer content in substratum.

A total of 10 seeds were planted on each test plate. The plates were incubated without light access in the temperature of 25±0.5°C for 72 h [MOLNAROVA *et al.* 2014]. The test plates were then photographed. The length of roots was determined using ImageTool® software. The germination index for the experimental and control series were calculated using the equation: $GI = G_e/G_c \cdot 100$, where G_e and G_c mean the total number of germinated seeds respectively in the experimental and control series. The root elongation index was calculated using the equation $EI = L_e/L_c \cdot 100$, where L_e and L_c stand for the roots length of the test plants in millimetres. Lack of roots growth difference in the range of 90–110% was considered as no fertilization effect. The value below 80% compared to control was considered as growth inhibition, whereas above 120% as stimulation. Roots growth reaching the level of 80–90% of the control was considered as slight inhibition whereas a growth of 110–120% compared to control as slight stimulation. In order to demonstrate the statistically relevant differences between the roots length of the test species, a one-way variance analysis (ANOVA) was performed using PQstat software. The variance analysis was made separately for each test species. Statistically important differences ($\alpha = 0.05$) were signed with normal letters, very important differences ($\alpha = 0.01$) – with capital letters.

RESULTS AND DISCUSSION

CHARACTERISTICS OF THE GRANULATED FERTILIZERS

Granulated fertilizers without the cellulose-collagen membrane (mentioned by CIESIELCZUK *et al.* [2015]), used in the presented study were character-

ized by high stability in time and were easy to apply. Characteristics of the experimental fertilizers is presented in Table 2 in comparison to pure SCG and requirements of Rozporządzenie MRiRW... [2009], with characteristics of organic fertilizers. High content of organic matter and pH close to neutral are the main advantages of both tested fertilizers. The K11 fertilizer was projected as an universal formula with a significant share of ash from biomass whereas K14 contains an addition of blood meal was made as a donor of chelated iron and magnesium sulphate and is dedicated to serve as an agent preventing chlorosis of plants [YUNTA *et al.* 2013]. Both fertilizers can be applied without the risk of decreasing the content of organic carbon and nitrogen in soil which may be the case when ash from biomass is used only [DEMEYER *et al.* 2001]. Moreover, when applied in combination, mineral and organic fertilization has a beneficial effect allowing to achieve high yield of crops. The investigated granulated fertilizers demonstrate such feature [LUDWIG *et al.* 2011]. In addition, the tested fertilizers can be produced in relatively short time as no composting process is required, which is an unquestionable advantage of the proposed solution, and the

Table 2. Basic characteristics of spent coffee ground (SCG) and investigated fertilizers K11 and K14 ($n = 3$)

Parameter	SCG	K11	K14	Rozporządzenie MRiRW [2009]
Reaction (pH _{KCl})	5.94–5.99	7.4–7.45	6.54–6.62	
Reaction (pH _{H2O})	5.64(0.05)	7.65–7.7	6.10–6.12	
EC, mS·cm ⁻¹	0.95(0.11)	2.75(0.01)	5.37(0.40)	
Organic substances, %	98.5(0.03)	90.7(0.05)	92.43(0.25)	30
TOC, %	53.3(3.57)	49.9(1.3)	48.9(1.3)	
N _{Kjed.} , %	2.45(0.38)	2.96(0.30)	4.01(0.41)	0.3
C:N	21.75	16.86	12.19	
CaO g·kg ⁻¹ DM	0.52(0.12)	12.54	5.10	
MgO g·kg ⁻¹ DM	7.14	7.79	15.08	
MgO (a) g·kg ⁻¹ DM	6.94	7.24	14.44	
S, %	0.132(0.022)	0.152(0.015)	1.19(0.12)	
H, %	7.78(1.43)	6.95(0.7)	7.07(0.71)	
Na ₂ O g·kg ⁻¹ DM	1.21	0.59	0.76	
K ₂ O g·kg ⁻¹ DM	3.05(0.18)	9.32	4.15	2.0
K ₂ O (a) g·kg ⁻¹ DM	2.57	5.52	3.44	
P ₂ O ₅ g·kg ⁻¹ DM	5.27(0.01)	7.4(0.2)	7.5(0.4)	2.0
P ₂ O ₅ (a) g·kg ⁻¹ DM	2.12	2.62	3.52	
Fe g·kg ⁻¹ DM	0.198	0.312	0.272	

Explanations: parameters marked with „a” apply to quantity of bioavailable component. Values in brackets = standard deviation. Source: own elaboration.

use of wet SCG enables the production of fertilizers, which does not depend on the season. Methods with use of dry SCG need a lot of sun heat, so it is complicated during autumn or winter.

The experimental K11 fertilizers were characterized by reaction close to neutral whereas K14 – by a slightly acidic pH (due to magnesium sulphate application). Similar pH values for SCG were obtained by other authors [KIM *et al.* 2014]. However, high values of electrolytic conductivity of the tested K11 and K14 fertilizers, amounting respectively 2.75 and 5.37 mS·cm⁻¹, were quite alarming. Although the decomposition of a fertilizer applied to soil in a form of an oil containing granulate was slow, however a significant salination (especially in the case of K14) was detrimental to plants. High value of electrolytic conductivity was also determined for K12 proposed as tablet fertilizer by CIESIELCZUK *et al.* [2015]. It should be stressed however, that the conductivity tests were performed for the fertilizer in a pulverized not granulated or tablet form thus the ions, mainly sulphate ions, were released in a controlled way [CIESIELCZUK *et al.* 2015]. Problems with high EC value were also reported in case of fertilizers made of coal ash and municipal sewage sludge [ROSIK-DULEWSKA *et al.* 2006].

Due to high contribution of SCG, the experimental fertilizers were featured by a very high organic matter content (>90%) which cannot be found in other types of natural organic fertilizers such as manure or slurry. The content of organic matter negatively correlated with the content of ash from biomass used for the fertilizer production [CIESIELCZUK *et al.* 2015].

The content of organic carbon reaching up to 50% in both experimental fertilizers was particularly beneficial. It was only slightly lower than the content observed for SCG. The amount of TOC determined in SCG by other authors was higher. The differences may however result from the different coffees used or SCG sieving method prior to analysis [PUJOL *et al.* 2013]. The content of organic carbon in the investigated fertilizers was comparable to high quality manure or cattle slurry. From the viewpoint of plant requirements, a significant water retention potential on the level of 5.73 g H₂O·g⁻¹ determined for the tested fertilizers is particularly beneficial [BALLESTEROS *et al.* 2014]. According to the guidelines specified in the Act of Ministry of Agriculture and Rural Development from 18th June 2008 included in the Polish Journal of Laws 2009 No. 224 item1804, all investigated series of fertilizers can be qualified as organic fertilizers.

The content of nitrogen in the tested series of fertilizers was high, reaching ca. 3% and 4%, respectively. Only pure SCG was an exception with nitrogen content of 2.45% and the C:N ratio of 21.7, which has been also confirmed by other authors [BALLESTEROS *et al.* 2014; MUSSATTO *et al.* 2011]. The determined content of nitrogen was comparable to its content in composts and manure [POULSEN *et al.* 2013] and in

SCG samples mentioned by other authors [PUJOL *et al.* 2013]. The amount of nitrogen comparable to data obtained for K14 series (above 4%) was also observed in granulates made of mixtures containing sewage sludge and fly ash from hard and brown coal combustion [ROSIK-DULEWSKA *et al.* 2016]. A considerably lower content of nitrogen (0.26 %) was determined in fertilizers with a high contribution of biomass derived ash [PESONEN *et al.* 2016]. The tested experimental fertilizers met the legal requirements concerning minimum nitrogen content in solid organic fertilizers [Rozporządzenie MRiRW... 2008] on the level of 0.3%. It should be highlighted that the risk of overfertilization is eliminated as the nitrogen present in the tested fertilizers occurs in an organic form, i.e. its availability and mobilization in ionic form to soil solution will be slow [POWLSON *et al.* 2011; ROSIK-DULEWSKA *et al.* 2016]. It is especially important due to fact of high rate of riverine N load from agricultural sources [ILNICKI 2014].

The content of phosphorus in the tested fertilizers was high – above 7%. However only for the rate ranging from 35 and 47% of its content (for K11 and K14 respectively) are bioavailable. Phosphorus is an important element in crop production thus ensuring its availability to plants is essential for an effective fertilization. Much higher contents of phosphorus (above 29 g P₂O₅) were determined in fertilizers made of fly ash and sewage sludge, mainly due to high content of ash (60%) [PESONEN *et al.* 2016].

The contents of calcium and potassium depended on the contribution of ash from biomass in the fertilizer. It should be highlighted that both of these elements occur in a form easily bioavailable to plants. In the case of K14 even 83% of the total phosphorus was bioavailable. This phenomenon of plant active forms of calcium and potassium in biomass ash were published also by other authors [DEMEYER *et al.* 2001]. High content of phosphorus, including bioavailable fraction, was – similar to reported in previous study Phosphorus content, including absorbable phosphorus, is high and similar to the one obtained in earlier studies [CIESIELCZUK *et al.* 2015].

A particularly high content of magnesium was observed for K14 fertilizer series (twice as high as in K11 fertilizer) with the addition of magnesium sulphate and blood meal (Tab. 2). The content of sulphur was low in K11 fertilizer and high in K14 fertilizer which is the result of magnesium sulphate addition. The role of this compound is to supply of plants with magnesium ions which are later leached downwards the soil profile due to, among others, some natural processes. The highest content of sodium in SCG and slightly lower in the investigated K11 and K14 fertilizers indicated its significant content in an organic form which is relevant from the viewpoint of soil salinity reduction.

In case of using organic fertilizers (e.g. manure) to fertilize the crop, acute toxicity can sometimes be caused by an increased heavy metal content, but in

SCG and ash-based fertilizers the content of these micro-contaminants was at a safe level [CIESIELCZUK *et al.* 2015; TELLA *et al.* 2013].

ACUTE TOXICITY

Spent coffee ground (SCG) as waste of plant origin are characterized by a strong phytotoxic effect due to the content of, among others, caffeine, tannin and polyphenols. [COLARIETI *et al.* 2006; KIM *et al.* 2014; MUSSATTO *et al.* 2011]. Other authors [SAADI *et al.* 2007] have not proved a direct relation between polyphenols content and a toxic effect on plants suggesting interactions with other compounds e.g. fatty acids. For that reason tests were made on substrata with K11 and K14 applied in a pulverized form. The applied doses of fertilizers corresponded to manure doses of 6.25; 12.5 and 25 dm³·m⁻² respectively, at ploughing to the depth of 25 cm. Additionally, application of the fertilizer in a pulverized form ensured its maximum effect in soil and thus on plants germination. Only slight differences were observed for germination indices of the individual tested plant species and fertilizer series (Tab. 3). The lowest index was observed for cress (*Lepidium*) and substratum with a 10% non-modified SCG addition.

Table 3. Germination index (*GI*) for tested plants species at 2.5; 5% and 10% fertilizers-rate (%)

Plant	Fertilizer rate (v/v)	Spent coffee grounds	Fertilizer	
			K11	K14
<i>Lepidium</i>	2.5%	100	96.7	93.3
	5%	96.7	93.3	96.7
	10%	80.0	93.3	93.3
<i>Avena</i>	2.5%	100	96.7	96.7
	5%	100	96.7	93.3
	10%	90.0	93.3	96.7
<i>Cucumis</i>	2.5%	100	96.7	93.3
	5%	96.7	86.7	96.7
	10%	90.0	93.3	90.0
<i>Sinapis</i>	2.5%	96.7	93.3	96.7
	5%	100	96.7	93.3
	10%	86.7	90.0	96.7

Source: own elaboration.

All tested species germinated properly and none of the applied fertilizer doses showed a germination inhibiting effect. In the case of *Lepidium*, lower indices were observed for K10 and K12 fertilizers applied in a 10 % dose [CIESIELCZUK *et al.* 2017]. For other tested species i.e. *Lepidium*, *Cucumis*, *Avena*, no direct correlation between the germination index and the applied fertilization level was observed. A significant germination inhibition was expected in the case of samples with SCG addition. During acute toxicity pre-tests with the use of this material as substratum, *Lepidium* seeds had not germinated. Also a lower number of germinated seeds were expected in the case of substratum with the addition of fertilizer from K14 series addition, mainly due to its high electrical conductivity. However, practice showed that the decrease

of germinated seeds could not be directly attributed to the increase of the fertilizer does in the substratum.

Due to high conductivity of K14 fertilizer, it was predicted that smaller amounts of seeds would sprout in soil with the addition of that particular series. In practice, however, the decrease in the amount of sprouted seed was not unequivocal (was not correlated with the growth of fertilizer content in the soil).

In experimental conditions, the sensitivity of plants to salinity can vary significantly and is not only plant species specific but even cultivar specific and is distinctive only to extremely high *EC* values i.e. on the level of 4–8 mS·cm⁻¹ [MAZUR *et al.* 2013]. This is particularly important in case of modifications of fertilizers produced using mineral salts [ROSIK-DULEWSKA *et al.* 2008].

ROOT ELONGATION INDEX (*EI*)

A significant inhibition of roots growth was observed for *Lepidium* and *Sinapis*, especially in samples with a 10% addition of K14 fertilizer. Measurement made at 72 h of the experiment showed only a ca. 20% length of *Lepidium* roots compared to the control (Tab. 4). A particularly high inhibition of roots growth identified for K11 and K14 fertilizers could result from the addition of the rape oil which inhibited oxygen and water access to the developing seedlings. The final form of described fertilizers are granules, so release of oil will be slower than powder used only in experimental plots. The results are comparable to the data obtained for *Lepidium* in the case of a 10% addition of the highly salinating fertilizers from K12 and K14 series. No decrease of germination index was observed for *Lepidium* up to the *EC* value of 2.4 at pH 4.2, however a strong root elongation inhibition was noted at 72 h of the test [SAADI *et al.* 2007].

A small addition of non-modified SCG to the substratum resulted in a slightly stronger growth of roots of the *Cucumis* seedlings (116%), however an increase of the SCG dose caused a gradual decrease of the growth intensity.

The calculated root elongation indices for the tested plant species (Tab. 4) imply their diversified sensitivity to the applied fertilizer additives. The highest root growth inhibition was observed for *Lepidium* and *Sinapis* even at the lowest dose of the tested fertilizers. It should be noted however that the use of the tested fertilizers in a pulverized form enhanced the toxicity effect due to an increased contact surface of the fertilizer with the soil.

The one-way variance analysis (ANOVA) showed statistically relevant differences between the obtained averages from the test data. Although a number of statistically significant and highly significant differences were identified between the experimental groups in which the substrata were modified by the addition of the experimental series of fertilizers, this study presents only these statistical differences which

Table 4. Elongation factor (*EI*) of roots and statistical differences (according to control samples) for plots with 2.5; 5 and 10 fertilizers rate (%) (*n* = 10)

Plant	Fertilizer rate (v/v)	Control	Spent coffee grounds	Fertilizer	
				K11	K14
<i>Lepidium</i>	2.5%	100ABc	85.5c	71.7A	62.0B
	5%	100ABc	78.5c	40.0A	34.8B
	10%	100ABC	49.1C	38.3A	19.9B
<i>Avena</i>	2.5%	100	101.6	108.6	91.3
	5%	100Ab	94.5	72.6A	65.2b
	10%	100ABc	83.8c	57.3A	24.4B
<i>Cucumis</i>	2.5%	100aB	116.6	92.4a	80.2B
	5%	100AB	108.4	72.2A	60.7B
	10%	100ABc	85.4c	64.2A	55.7B
<i>Sinapis</i>	2.5%	100Ab	85.9b	77.8A	82.5
	5%	100AbC	75.0C	42.6A	53.2b
	10%	100ABC	70.9C	39.4A	27.6B

Explanations: A, B significant at $\alpha = 0.01$; a, b significant at $\alpha = 0.05$.

Source: own elaboration.

occurred between the control and the experimental groups of individual species and the given applied dose of the tested fertilizer. Contrary to other tested plant species, *Avena* was the only one for which no statistical differences were observed for the lowest fertilization level. Differences were noted for all levels of fertilization with rape oil addition in the case of *Lepidium* which confirmed its extreme sensitivity to the tested fertilizers. The strongest root growth inhibition was observed for *Lepidium* for substrata with a 10% addition of K11 and K14 fertilizers – 38.3 and 19.9% respectively. A slightly lower inhibition of roots growth was noted for *Sinapis* – 39.4 and 27.6%, respectively. A stronger inhibition observed for K14 fertilizer could result from a strong salinity of the soil solution ($5.37 \text{ mS}\cdot\text{cm}^{-1}$) to which young seedlings are particularly sensitive. A similarly high inhibition of *Lepidium* seedlings growth was observed for substrata with a 6% addition of sewage sludge and sewage sludge made composts [OLESZCZUK 2008].

Cucumis was identified as a species least sensitive to oily and salinated substratum. Although in the case of that species addition of the tested fertilizers (except for non-modified SCG) caused inhibition of roots growth, however this inhibition was on the lowest level among the plant species used in the experiment. No growth stimulation was observed for any of the samples including K11 and K14 fertilizers which has however been observed in similar test with the use of fertilizers of a comparable formula but without rape oil addition [CIESIELCZUK *et al.* 2017]. Thus it was mainly the content of rape oil that was responsible for a strong inhibition of the seedlings growth rather than salinity, which was observed also in the case of K12 fertilizer causing a slowed development of the roots, achieving the length of 49.8 and 67.7% respectively for *Lepidium* and *Sinapis* [CIESIELCZUK *et al.* 2017].

CONCLUSIONS

Organic matter and nutrient analysis of materials was in the last century a base of knowledge about fertilizing properties, but now investigations of potential fertilizers should be much more detailed. Study of the germination and roots growth of different test plant species in the first three days since sieving is gaining an increasing attention as a phytotoxicity index of a material which allows assessing its negative influence on crops. The presented test data showed a diversified acute toxicity level of the fertilizers for the tested plant species. The lowest phytotoxicity was observed for non-modified SCG and the highest for K14 fertilizer which, beside rape oil addition which inhibited water uptake and oxygen access, caused strong salinity of the substratum.

The obtained research results indicate the potential of SCG, blood meal, magnesium sulphate and ash from biomass as key components of low cost fertilizers which may find application in organic farming. Addition of rape oil as an agent controlling the elution of ions from the fertilizer to the soil solution should be eliminated as it probably causes strong inhibition of roots growth. A big advantage of the tablet fertilizers made of SCG and biomass-derived ash is the fact that they can be produced in-house even by unskilled persons. Moreover the reaction of the produced fertilizer can be controlled individually according to the needs of plant species for which it is to be applied.

This publication was supported within the funds of the Chair of Land Protection of the University of Opole and the Institute of Ceramics and Building Materials, Division of Materials Engineering in Opole.

REFERENCES

- BALLESTEROS L.F., TEIXEIRA J.A., MUSSATTO S.I. 2014. Chemical, functional, and structural properties of spent coffee grounds and coffee silverskin. *Food and Bioprocess Technology*. Vol. 7. Iss. 12 p. 3493–3503. DOI 10.1007/s11947-014-1349-z.
- CAETANO N.S., SILVA V.M.F., MELO A.C., MARTINS A.A., MATA T.M. 2014. Spent coffee grounds for biodiesel production and other applications. *Clean Technologies and Environmental Policy*. Vol. 16 p. 1423–1430. DOI 10.1007/s10098-014-0773-0.
- CIESIELCZUK T., ROSIK-DULEWSKA Cz., POLUSZYŃSKA J., MILEK D., SZEWCZYK A., SŁAWIŃSKA I. 2017. Acute toxicity of experimental fertilizers made of spent coffee grounds. *Waste and Biomass Valorization*. DOI 10.1007/s12649-017-9980-3.
- CIESIELCZUK T., ROSIK-DULEWSKA Cz., WIŚNIEWSKA E. 2015. Possibilities of coffee spent ground use as a slow action organo-mineral fertilizer. *Annual Set the Environment Protection*. Vol. 17 p. 422–437.
- CIR(EU) No 354/2014. Commission Implementing Regulation (EU) No 354/2014 of 8 April 2014 amending and correcting Regulation (EC) No 889/2008 laying down

- detailed rules for the implementation of Council Regulation (EC) No 834/2007 on organic production and labelling of organic products with regard to organic production, labelling and control.
- COLARIETI M.L., TOSCANO G., GRECO G. JR. 2006. Toxicity attenuation of olive mill wastewater in soil slurries. *Environmental Chemistry Letters*. Vol. 4. Iss. 2 p. 115–118. DOI 10.1007/s10311-006-0050-5.
- DEMEYER A., VOUNDI-NKANA J.C., VERLOO M.G. 2001. Characteristics of wood ash and influence on soil properties and nutrient uptake: An overview. *Bioresource Technology*. Vol. 77 p. 287–295.
- Eurostat undated. Municipal waste generation and treatment, by type of treatment method [online]. [Access 14.04.2016]. Available at: <http://ec.europa.eu/eurostat/tgm/table.do?tab=table&init=1&plugin=1&language=en&pcode=tsdpc240>
- GOSWAMI L., PATEL A.K., DUTTA G., BHATTACHARYYA P., GOGOI N., BHATTACHARYYA S.S. 2013. Hazard remediation and recycling of tea industry and paper mill bottom ash through vermicomposting. *Chemosphere*. Vol. 92 p. 708–713. DOI 10.1016/j.chemosphere.2013.04.066.
- ILNICKI P. 2014. Emissions of nitrogen and phosphorus in to rivers from agricultural land – selected controversial issues. *Journal of Water and Land Development*. No. 23 p. 31–39. DOI 10.1515/jwld-2014-0027.
- KIM M.K., MIN H.G., KOO N., PARK J., LEE S.H., BAK G.I., KIM J.G. 2014. The effectiveness of spent coffee grounds and its biochar on the amelioration of heavy metals-contaminated water and soil using chemical and biological assessments. *Journal of Environmental Management*. Vol. 146 p. 124–130. DOI 10.1016/j.jenvman.2014.07.001.
- KORNER I., SABORIT-SANCHEZ I., AGUILERA-CORRALES Y. 2008. Proposal for the integration of decentralised composting of the organic fraction of municipal solid waste into the waste management system of Cuba. *Waste Management*. Vol. 28 p. 64–72.
- LUDWIG B., GEISSELER D., MICHEL K., JOERGENSEN R.G., SCHULZ E., MERBACH I., RAUPP J., RAUBER R., HU K., NIU L., LIU X. 2011. Effects of fertilization and soil management on crop yields and carbon stabilization in soils. A review. *Agronomy for Sustainable Development*. Vol. 31. Iss. 2 p. 361–372. DOI 10.1051/agro/2010030.
- MAZUR Z., RADZIEMSKA M., TOMASZEWSKA Z., ŚWIĄTKOWSKI Ł. 2013. Effect of sodium chloride salinization on the seed germination of selected vegetable plants. *Scientific Review – Engineering and Environmental Sciences*. No. 62 p. 444–453.
- MOLNAROVA M., SMELKOVA M., FARGASOVA A. 2014. Assessment of the suitability of Phytotoxkit plastic vertical containers compared with petri dishes for standard seedling growth tests. *Bulletin of Environmental Contamination and Toxicology*. Vol. 92. Iss. 4 p. 497–501. DOI 10.1007/s00128-013-1186-1.
- MUSSATTO S.I., MACHADO E.M.S., MARTINS S., TEIXEIRA J.A. 2011. Production, composition, and application of coffee and its industrial residues. *Food and Bioprocess Technology*. Vol. 4 p. 661–672. DOI 10.1007/s11947-011-0565-z.
- OLESZCZUK P. 2008. The toxicity of composts from sewage sludges evaluated by the direct contact tests Phytotoxkit and Ostracodtoxkit. *Waste Management*. Vol. 28. Iss. 9 p. 1645–1653. DOI 10.1016/j.wasman.2007.06.016.
- PESONEN J., KUOKKANEN V., KUOKKANEN T., ILLIKAINEN M. 2016. Co-granulation of bio-ash with sewage sludge and lime for fertilizer use. *Journal of Environmental Chemical Engineering*. 4. Iss. 4 p. 4817–4821.
- PN-R-04024:1997. Analiza chemiczno-rolnicza gleby – Oznaczenie zawartości przyswajalnego fosforu, potasu, magnezu i manganu w glebach organicznych [Agrochemical soil analyse – Determination of available phosphorus, potassium, magnesium and manganese contents in organic soils].
- POULSEN P.H.B., MAGID J., LUXHØI J., DE NEERGAARD A. 2013. Effects of fertilization with urban and agricultural organic wastes in a field trial – Waste imprint on soil microbial activity. *Soil Biology and Biochemistry*. Vol. 57 p. 794–802. DOI 10.1016/j.soilbio.2012.02.031.
- POWLSON D.S., GREGORY P.J., WHALLEY W.R., QUINTON J.N., HOPKINS D.W., WHITMORE A.P., HIRSCH P.R., GOULDING K.W.T. 2011. Soil management in relation to sustainable agriculture and ecosystem services. *Food Policy*. Vol. 36 p. 72–87. DOI 10.1016/j.foodpol.2010.11.025.
- PUJOL D., LIU C., GOMINHO J., OLIVELLA M.À., FIOL N., VILLAESCUSA I., PEREIRA H. 2013. The chemical composition of exhausted coffee waste. *Industrial Crops and Products*. Vol. 50 p. 423–429. DOI 10.1016/j.indcrop.2013.07.056.
- ROSİK-DULEWSKA CZ., GŁOWALA K., KARWACZYŃSKA U., SZYDŁO E. 2006. The mobility of chosen pollutants from ash-sludge mixtures. *Polish Journal of Environmental Studies*. Vol. 15. Iss. 6 p. 895–904.
- ROSİK-DULEWSKA CZ., KARWACZYŃSKA U., GŁOWALA K., ROBAK J. 2008. Elution of heavy metals from granulates produced from municipal sewage deposits and fly-ash of hard and brown coal in the aspect of recycling for fertilization purposes. *Archives of Environmental Protection*. Vol. 34. Iss. 2 p. 63–71.
- ROSİK-DULEWSKA CZ., NOCOŃ K., KARWACZYŃSKA U. 2016. Wytwarzanie granulatu z komunalnych osadów ściekowych i popiołów lotnych w celu ich przyrodniczego (nawozowego) odzysku [Production of granules from municipal sewage sludge and Ely ashes due to its natural (fertilizer) recycling]. *Works and Studies / Prace i Studia*. Vol. 87. Zabrze. IPiŚ PAN. Z. 87. ISBN 978-83-60877-25-8 pp. 187.
- ROY M., KARMAKAR S., DEBSARCAR A., SEN P.K., MUKHERJEE J. 2013. Application of rural slaughterhouse waste as an organic fertilizer for pot cultivation of solanaceous vegetables in India. *International Journal of Recycling of Organic Waste in Agriculture*. Vol. 2. Iss. 6 p. 1–11. DOI 10.1186/2251-7715-2-6.
- Rozporządzenie Ministra Rolnictwa i Rozwoju Wsi z dnia 18 czerwca 2008 r. w sprawie wykonywania niektórych przepisów ustawy o nawozach i nawożeniu [Regulation of the Minister of Agriculture from 18 June 2008 due to implementation of certain provisions of the Law on fertilizers and fertilization]. *Dz.U.* 2008. Nr 119 poz. 765.
- Rozporządzenie Ministra Rolnictwa i Rozwoju Wsi z dnia 21 grudnia 2009 r. zmieniające rozporządzenie w sprawie wykonywania niektórych przepisów ustawy o nawozach i nawożeniu [Regulation of the Minister of Agriculture from 21 December 2009 due to change of Regulation due to implementation of certain provisions of the Law on fertilizers and fertilization]. *Dz.U.* 2009. Nr 224 poz. 1804.
- SAADI I., LAOR Y., RAVIV M., MEDINA S. 2007. Land spreading of olive mill wastewater: Effects on soil microbial activity and potential phytotoxicity. *Chemosphere*. Vol. 66 p. 75–83. DOI 10.1016/j.chemosphere.2006.05.019.

- SAMPAIO A., DRAGONE G., VILANOVA M., OLIVEIRA J.M., TEIXEIRA J.A., MUSSATTO S.I. 2013. Production, chemical characterization, and sensory profile of a novel spirit elaborated from spent coffee Grodnu. LWT – Food Science and Technology. Vol. 54 p. 557–563. DOI 10.1016/j.lwt.2013.05.042.
- TELLA M., DOELSCH E., LETOURMY P., CHATAING S., CUOQ F., BRAVIN M.N., SAINT MACARY H. 2013. Investigation of potentially toxic heavy metals in different organic wastes used to fertilize market garden crops. Waste Management. Vol. 33 p. 184–192. DOI 10.1016/j.wasman.2012.07.021.
- YUNTA F., DI FOGGIA M., BELLIDO-DIAZ V., MORALES-CALDERON M., TESSARIN P., LOPEZ-RAYO S., TINTI A., KOVACS K., KLENCAR Z., FODOR F., DOMENICO-ROMBOLA A. 2013. Blood meal-based compound. Good choice as iron fertilizer for organic farming. Journal of Agricultural and Food Chemistry. Vol. 61. Iss. 17 p. 3995–4003. DOI 10.1021/jf305563b.

Tomasz CIESIELCZUK, Czesława ROSIK-DULEWSKA, Joanna POLUSZYŃSKA, Irena SŁAWIŃSKA

Toksyczność ostra eksperymentalnych nawozów wytworzonych z mączki z krwi, odpadów poekstrakcyjnych kawy oraz popiołu z biomasy

STRESZCZENIE

Przepisy prawa wymuszają stopniowe ograniczanie deponowania odpadów ulegających biodegradacji na składowiskach. Po wydzieleniu frakcji biodegradowalnej ze zmieszanych odpadów komunalnych lub dzięki selektywnej zbiórce tej frakcji można ją zastosować w nawożeniu organicznym. W szczególności materia organiczna zawarta w odpadach przemysłu spożywczego lub powstająca w gospodarstwach domowych, pod warunkiem braku zanieczyszczeń innymi rodzajami odpadów może być wykorzystywana jako nawóz organiczny. W pracy przedstawiono wyniki badań nad toksycznością ostrą mieszaniny wykonanej z odpadów poekstrakcyjnych kawy (SCG) modyfikowanych popiołem powstającym z niskotemperaturowego spalania biomasy lub popiołem z dodatkiem siarczanu magnezu i mączki z krwi. Dodatkiem uplastyczniającym oraz spowalniającym uwalnianie biogenów z nawozu do roztworu glebowego był olej rzepakowy. Jako rośliny testowe wykorzystano: gorczycę (*Sinapis alba* L.), owies zwyczajny (*Avena sativa* sp. L.), ogórek (*Cucumis sativus* L.) oraz rzeżuchę ogrodową (*Lepidium sativum* L.). Testy toksyczności przeprowadzono z użyciem standardowej procedury 72 h z zastosowaniem płytek Phytotoxkit microbiotest z udziałem nawozu 2,5; 5 i 10% (obj.). Uzyskane wyniki wskazują na zwiększenie toksyczności ostrej w odniesieniu do wszystkich testowanych gatunków. Proporcjonalnie do zastosowanych dawek nawożenia najsilniejszą inhibicję wzrostu siewek w okresie 72 h zanotowano w próbkach z 10-procentowym udziałem badanych nawozów, w szczególności gdy wykazywały one znaczne zasolenie lub niskie pH. Najbardziej wrażliwym gatunkiem na zastosowane dawki nawozów była rzeżucha, a najmniej – ogórek, w przypadku którego inhibicja wzrostu korzeni była niewielka. Prawdopodobną przyczyną spowolnienia wzrostu korzeni było ograniczenie dopływu tlenu oraz nadmierne zasolenie podłoża spowodowane zastosowanymi dodatkami.

Słowa kluczowe: mączka z krwi, nawóz organiczny, odpady poekstrakcyjne kawy, olej rzepakowy, toksyczność ostra