THE INFLUENCE OF BIOMASS ASH ON THE MIGRATION OF HEAVY METALS IN THE FLOODED SOIL PROFILE – MODEL EXPERIMENT

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Abstract: Soils that have been exposed to flood waters can be heavily polluted by inorganic and organic compounds. They are mainly compounds which appear in dissolved or suspended form flowing together with heavily laden floodwater, as well as compounds created as a result of reactions in the soil profile, mostly due to anaerobic transformation of organic matter. Heavy metals brought with flood waters are absorbed by the soil and also washed out from flood sediments by precipitation when the flood recedes. This paper presents the results of research on the effects of fertilization with ash from incineration or pyrolysis of biomass on the migration process of heavy metals (Zn, Cu, Cr, Ni, Pb, Cd, Mn) in the arable layer of soil. It has been shown that the metals in the flood sediment migrate actively in the soil profile what leads to the enrichment of the soils, also in the case of the soil fertilization with biomass ash.

INTRODUCTION

Prolonged torrential rains can cause rising water levels in mountainous and lowland rivers. A common practice of sealing the ground surface with asphalt or concrete causes gathering large amounts of rainwater in a short period of time, while in natural conditions it is stored as a result of soil retention. Water spate results in flooding or permeation and has negative long-term economic, social and environmental effects [2]. The increasingly appearing phenomenon of floods forces us to build new polders and reservoirs and calls for forestation on additional areas of land [28], which significantly changes the usage of terrain. Flooding of soils and the anaerobic processes associated with it, can significantly affect the changes in soil chemistry, even when the flood recedes. Formed in these processes ammonia, and for example drifting with floodwater polyphenolic compounds may delay the efficiency of germination and growth of grass *Elymus virginicus* L. by

ten percent even when the flood recedes [34]. Flood waters contain significant loads of biogenic substances that increase the salinity as well as slurries which contain inorganic and organic contaminants (including heavy metals) [17, 37]. During a rainfall and surface runoff contaminants are washed from roads and transport yards, warehouses and petrol stations [16]. Floods often cover large areas, such as landfills, sewage treatment plants and septic tanks, in which farm and household wastewater is collected. It enriches the flood sediment with significant amounts of organic matter containing absorbed metal ions and leaches large amounts of mineral contaminants in dissolved form, therefore having toxic effects on organisms through contact and ingestion [9]. The increasing prevalence of metal-containing sources means that they are noted in all components of the environment [4, 7]. From the point of view of risks to human health, it is important to identify properly the pollution sources, and it should be a key factor while making the decision on the selection of remediation methods [40]. Flood water leaves sediment on the soil surface which may include a significant amount of metals [3, 20]. Also very toxic metals as cadmium are present in bottom sediments in concentrations up to 285 g/kg d.m. [Ciesielczuk – unpublished data]. In addition, soil fertilization with soild manure, liquid manure, compost or sewage sludge can begin a process of enriching the soil in, among others, metals that frequently occur in easily soluble forms [5, 24, 30, 35, 41]. Allochtonic metal ions permeating into the soil are mainly absorbed in the humus layer [15]. Organic matter present in amounts greater than 0.1% is largely responsible for the sorption of pollutants [39]. Flood sediments brought onto the soil surface may have increased ecotoxicity, but research carried out after the flood, which occurred on the Łaba in 2002, showed no toxic effects of the tested organisms. This is an effect of significant dilution and decrease of the bioavailability of xenobiotics, which is explained by the increase in the amount of organic matter in sediments, compared to the period before the flood [24, 38]. Organic xenobiotics, used in plant protection treatment (cypermethrin, chlorpyrifos, endosulfan and glyphosate) are washed away with rain water and can have severe toxicity and even lethal properties to certain species such as amphibians [23]. In a study on the impact of floods on small invertebrates there was no severe toxicity during the contact of tested organisms with an alkaline soil containing significant content of organic matter and heavy metals brought by floodwater. This is explained by, among others, low bioavailability of the analyzed metals: Cd, Cu and Zn [13]. In a study on the effects of sediment dredged on the yield of maize, which is grown on light acid soils, an increase was recorded directly proportional to the amount of sediment in the soil. In addition, there was a reduction of heavy metals in plant biomass [36]. However, heavy metals that are brought by flood sediment, by migrating into the soil profile, can be dangerous not only for the quality of agricultural soils, but also for the groundwater. There was observed a negative influence of metals (Cr, Ni, Cd, Hg) on plants biological processes. Especially metals lead to the increase of reactive oxygen species (ROS) and free radicals production. Moreover, metals can block activity of important enzymes (e.g. α-amylase, β-amylase and α-glucosidase) or lead to malondialdehyde production as an effect of lipid peroxidation processes. It could be a cause of cell membranes malfunction or damage [31]. To reduce the bioavailability and slow down the migration of metal ions into the soil solution, the liming method is used to increase the soil pH. This process, even if it is carried out only once, effectively reduces the toxicity of metals in the soil [32]. Currently, due to the more widespread use of biomass for energy purposes, there is

a problem arising from the disposal of ashes. In the case of some types of biomass, the ash can be a valuable replacement for calcium fertilizers used on arable soils [1, 8, 26]. This paper presents the results of studies on the effects of soil fertilization by light ash from the incineration of biomass, and its effect on the speed of migration of heavy metals in the experiment simulating short period flood conditions.

MATERIALS AND METHODS

The experiment used soil samples (type: mud river) from arable layer, with grain size characteristic for loamy soils. Before the experiment, the soil was dried at room temperature and sieved through a 2 mm diameter mesh.

The ash was obtained from the incineration of oak biomass in an open small furnace (waste code 10 01 03 according to Polish Waste Catalogue 2001 nr 112 pos. 1206), and was then incinerated at 600°C for 2 hours for small of incompletely burned particles removal and then sieved through a sieve with a mesh diameter of 2 mm. For experimental purposes, the amount of fertilization used is 5.52 Mg to ash/ha (GP), what was calculated on the base standard amount of mineral fertilizing 2 Mg CaO/ha. The same soil without the ash (series G) was used as a reference sample. The test soil was placed in a glass cylinder with dimensions of 3×15 cm (106 cm³). Then on the surface of the soil in the cylinder, some "flood sediment" was placed containing heavy metals in the amount of 2.00 g/cylinder. The "flood sediment" was prepared by mixing sapropel sediment derived from the dam reservoir Turawskie Large Lake, LRM1 certified reference material (Gdansk University of Technology, Poland) and certified reference material LRM-CC013a (BAM, Berlin, Germany) in a 1:1:1 ratio (m/m). For the simulation of fine-grain flood sediment, the sediment, before it was used, was thoroughly ground in an agate mill and sieved through a sieve with a mesh diameter of 0.5 mm. The cylinder's edges were secured with quartz wool. Cylinders were filled completely using the rainwater with capillary ascension method and incubated in the dark at a constant temperature of 22 ± 0.2°C for a period of 7 days. On the 7th day after filling, the water was removed. Then, simulating the precipitation, cylinders with soil were washed with rain water in the amount of 30 mm/week , and the analysis of metals in the soil cores were made on days 7, 14, 28 and 42 after filling the cylinders. After removing the soil from the core glass cylinder, it was separated for analysis into sections *A*, *B*, *C* and *D* corresponding to the depths 0–3, 3–6, 6–9 and 9–12 cm profile, in order to capture the metal penetration into the deeper layers of soil.

Heavy metals (Zn, Cu, Cr, Ni, Pb, Cd, Mn) were determined in averaged samples for each segment, by taking 4 independent samples from the tested core layer. In addition, the following parameters in the soils were determined: pH, electrolytic conductivity (EC), the content of organic matter and organic carbon (methodology according to Polish Standards). The content of calcium, sodium, potassium and lithium was ascertained in wet mineralizates by FES method using *BWB-XP* apparatus. The content of other analysed metals (Zn, Cu, Cr, Ni, Pb, Cd, Mn) was determined by using a spectrophotometer Thermo AAS iCE 3500 after wet microwave assisted mineralization in aqua regia using MARS-X device. Analytical procedure quality was checked with LRM1 certified reference material (Gdansk University of Technology, Poland). Relative standard deviation (RSD) values calculated for metals determined in experimental layers (A–D) were acceptable and did

not exceed 12.5% (higher values were noted for zinc and manganese). The results of metals concentration were processed using the t test ($\alpha = 0.05$), and correlation coefficients were calculated.

RESULTS AND DISCUSSION

The tested soils, before they were flooded, differed in terms of physico-chemical parameters (Table 1). Low organic matter content was characteristic, and was about 2.5 times lower than in the applied flood sediment. The content of ascertained metals was low, typical for uncontaminated soils [14, 29, 33]. Using highly alkaline ash from biomass containing more than 34% of CaO in an amount of 5.52 Mg/ha, soil pH resulted in an increase of reaction by the value of GP 0.74 and conductivity of more than 80 µS/cm compared to the sample without ash fertilization (G).

Table 1. Characteristics of the substrates used in the experiment: mean (SD) (n = 3)

	Ash	G	GP	Flood sediment	Journal of Law No 165 item 1359
Reaction (pH$_{H2O}$)	11.47(0.01)	6.62(0.01)	7.36(0.01)	7.05(0.01)	–
Electrolytic conductivity (µS/cm)	12130 (40)	93.4(5.6)	176.8(4.0)	1378(7.8)	–
Organic matter [g/kg d.m.]	6.3(0.9)	31.1(2.2)	32.8(0.2)	80.2(2.2)	–
Organic carbon [g/kg d.m.]	0.2(0.1)	10.1(0.6)	10.2(0.7)	32.7(1.3)	–
Kjeldahl nitrogen [g N/kg d.m.]	0.5(0.2)	2.0(0.3)	2.3(0.3)	4.4(0.7)	–
Ca [g/kg d.m.]	246(12)	0.28(0.1)	0.35(0.1)	4.77(0.5)	–
P [g/kg d.m.]	7.34(1.2)	1.34(0.6)	1.59(0.6)	4.14(0.8)	–
Na [g/kg d.m.]	1.47(0.5)	0.03(0.01)	0.04(0.01)	0.84(0.3)	–
K [g/kg d.m.]	6.82(1.4)	0.78(0.2)	1.36(0.3)	2.75(0.8)	–
Mg [g/kg d.m.]	28.2(6.7)	0.63(0.2)	0.67(0.2)	3.24(0.9)	
Li2O [mg/kg]	161.9(21)	14.57(4.2)	15.02(4.3)	55.6(11)	–
Zn [mg/kg d.m.]	506.8(28)	59.51(16)	55.59(14)	919.0(34)	300
Cu [mg/kg d.m.]	119.6(6.2)	4.40(0.8)	4.39(0.7)	81.4(4.7)	150
Pb [mg/kg d.m.]	21.2(1.5)	14.0(0.9)	15.1(1.0)	70.8(5.2)	100
Cr [mg/kg d.m.]	21.8(1.1)	4.40(1.7)	5.13(1.8)	32.1(2.6)	150
Cd [mg/kg d.m.]	2.27(0.6)	0.29(0.04)	0.34(0.04)	17.1(0.8)	4
Ni [mg/kg d.m.]	22.2(1.3)	2.46(0.6)	2.51(0.5)	22.7(1.9)	100
Mn [mg/kg d.m.]	4543(51)	261.4(18)	332.0(20)	455.9(29)	–

– not regulated

The addition of ash to the soil (series GP) resulted in an increase of not only the value of pH and conductivity, but also of calcium, sodium, potassium and lithium. The usage of ash also caused a slight increase in the content of heavy metals, except for manganese. The increase in the content of this element by more than 27% was due to its previous significant content in the ash. The prepared flood sediment contained several times more heavy metals than the mineral and mineral-organic deposits which are commonly seen after flooding or Sapropel sediments from unpolluted waters. However, the only exceeded limit values, referring to Journal of Law No 165 item 1359, were noted in the case of zinc [11, 17].

Rainwater (Table 2) used both for filling cylinders with soil and washing cylinders with sediment (simulated rainfall) was contaminated with material from the roof and gutter pipe. It is characterized by alkaline reaction, a small electrolytic conductivity and relatively high levels of ammonium, sulphate and chloride.

Table 2. Rainwater characteristics used to fill the cylinders with the soil and the effluent from the cylinder G and GP after 7 days of the experiment

	Before filling	Effluent G	Effluent GP
pH	8.97	7.25	7.97
EC [μS/cm]	100.4	594	689
NH_4^+ [mg/dm³]	1.40	3.32	6.04
NO_2^- [mg/dm³]	0.029	0.03	0.03
NO_3^- [mg/dm³]	0.785	–	–
PO_4^{-3} [mg/dm³]	0.135	–	–
SO_4^{-2} [mg/dm³]	45.27	–	–
$ChZT_{Mn}$ [mg O_2/dm³]	5.8	56.2	76.2
Cl^- [mg/dm³]	47.04	–	–

– not analyzed

The content of heavy metals in rainwater was low – the highest concentration reported for zinc and copper was respectively – 0.66 and 0.08 mg/dm³. The effluent from the cylinders obtained after 7 days of soil incubation had a reaction close to neutral (G) and slightly alkaline (GP). This was due to the high content of ammonium ions, indicating the process of reducing nitrate ions, more intensive than in the soil G fermentation of organic matter and ash additive used [2]. A higher content of soluble organic matter (DOM) expressed by COD_{Mn} value and twice the ammonium ion content, provides a more intensive process of decomposition in the cylinders with the addition of ash (GP). This can be explained by filled pores of the soil with the ash, and therefore accelerated decomposition of the solution enriched in macro- and trace elements necessary for the proper development of anaerobic bacteria. On the 7th day of the experiment, in samples of soil a slight decrease in the content of the studied metals was observed. This may be caused by the removal of soluble organic matter (DOM) with floodwater of the studied profiles. Flood sediment used in the experiment contained high amounts of the analysed

elements, which is typical for this type of mud. A study on flood deposits conducted in the Czech Republic [27] reported similar levels; with the exception of zinc whose amount did not exceed 369 mg/kg. In the studied model experiment a strong correlation was found between the G and GP in the case of calcium, in which the maximum concentration was noted on the 14th day of the experiment. This indicates the elution of the calcium present in the sediment when its amount exceeds 5.8 g/kg dry matter. A similar situation was observed for sodium (concentration peaks also occurred on the day 14), where highly correlated concentrations of sodium appeared in layers b and c (0.93–0.98) of both series (G and GP) of the experiment.

During the experiment, in G series, zinc contained in the sediment migrated to a small extent, and its amount was not high and reached a maximum of 103.7 mg/kg dry matter. A small migration of this element in soil with pH close to neutral is also confirmed by other authors [12]. Only on the 42nd day of the experiment an increase by over 51% was noted in the content of this element in the layer *A* compared to the initial value, and it was found that the increase in the zinc content of layer *B* in the experiment is highly correlated. In the series GP the correlation was weaker – increasing amount of Zn in layer *A* (which had direct contact with sediment) was lower (80.1 mg/kg d.m.), and finally the maximum content of the element was noticed on the 42nd day and did not exceed 90 mg/kg dm. In this case, on the last day of the experiment, the highest amount observed was in the layer *D*. Copper mobility was high as evidenced by other authors [39]. On the 7th day of the experiment a slight increase in the content of this element was observed in both series of experiments (10% and 7.4% respectively for G and GP) and was recorded only in layer *A* of the profile. On the last day of the experiment in the G series, the highest amount of Cu was observed in layer *D*, in which the content had reached almost 8 mg/kg dm. In addition, there was a strong positive correlation between the concentrations of Cu in layers *B*, *C* and *D*, and also this element correlates with sodium (0.52–0.82) and strongly with potassium (0.83–0.93). In the GP series, the highest concentration of Cu was observed in layer *C* (more than 8.9 mg/kg d.m.), and slightly lower in layers *A* and *B*. In this series highly positive correlations were noted for concentrations listed in layers *A* and *B*, as well as *B* and *C*. However, the correlation with sodium and potassium was lower than that of the control sample and amounted to 0.45–0.68 and -0.21–0.67. The maximum increase in Cu concentration in comparison with the values recorded prior to the experiment, was 65% and 75%, respectively for a series G and GP. The average copper content on the days 28 and 42 was higher for GP, indicating extensive migration of this element, also in the presence of an alkalising agent which is confirmed by other authors [19]. In GP series, copper content increased in layer *A* and was directly connected with the "sediment" and was linear ($y = 0.0649 x + 5.0815$), which was not observed for the other layers and analysed metals in both tested series.

The migration of nickel from sediment to layer *A* was already observed on the 7th day of the experiment, when its increase in series G was 17.8% and in the GP – 11.6%. The mobility of this metal was kept in a series of G until the last day, on which the concentration varied in direct proportion to the order of the layers in the profile – the highest level was recorded in layer *D* (more than 4.6 mg/kg d.m.), and the lowest in layer *D* (3.63 mg/kg d.m.) which had direct contact with the sediment. As in the case of copper, so in the case of nickel, there was a strong positive correlation between the concentration of this element in the layers *B*, *C* and *D*. With the exception of layer *A*,
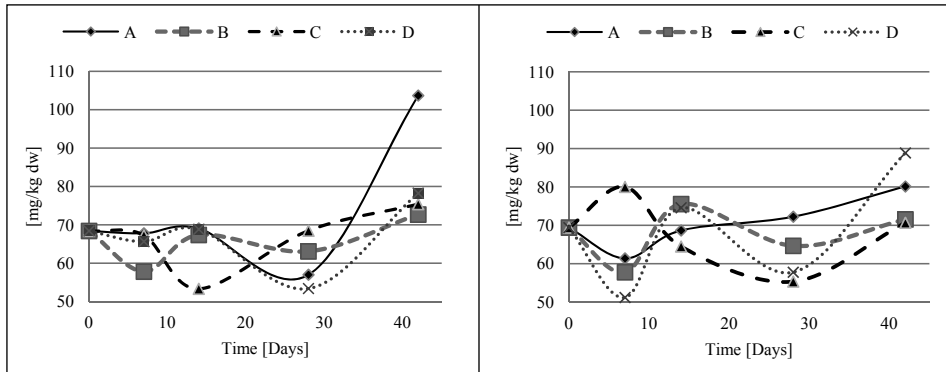
Fig. 1. Zinc content [mg/kg d.w.] changes in control G (left) and experimental GP (right) series, during 42 days model experiment. Experimental layers marked as explained in Materials and methods
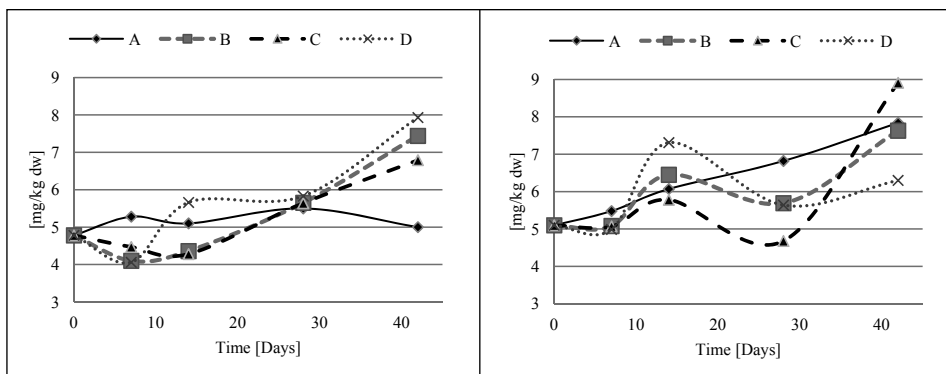

Fig. 2. Copper content [mg/kg d.w.] changes in control G (left) and experimental GP (right) series, during 42 days model experiment. Experimental layers marked as explained in Materials and methods

as in the case of copper, a strong correlation with sodium (0.74–0.94) and potassium (0.88–0.93) was noted. In the GP series the nickel migration rate was significantly slower, and the concentrations recorded on the last day of the experiment were inversely proportional to the depth of the layers, and were slightly higher (on average 4.4 mg/kg d.m.) than those recorded for the series G (4.1 mg/kg d.m.). The correlation of sodium was significant, except for layer C (0.28), and ranged from 0.77 to 0.83. The correlation of potassium (as in the control series) occurred with the exception of layer A, however, it was not as strong (0.49–0.82). In both series, the maximum increase in concentrations was over 50% of the original value.

Migration of cadmium was slow and concentrations increase was observed only on the 42nd day of the experiment. A twofold increase was observed in the layer A while the increase in layers C and D was respectively 62.7 and 76.3%. High correlations were noted between the layers A and B (0.81) and A and D (0.89). In the GP series the recorded concentrations were lower, and their maximum level was 41.2% in layer B on the last day of the experiment.
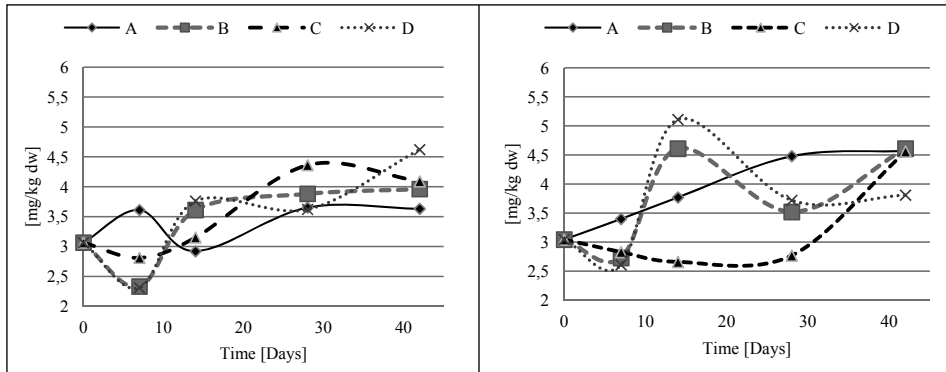
Fig. 3. Nickel content [mg/kg d.w.] changes in control G (left) and experimental GP (right) series, during 42 days model experiment. Experimental layers marked as explained in Materials and methods
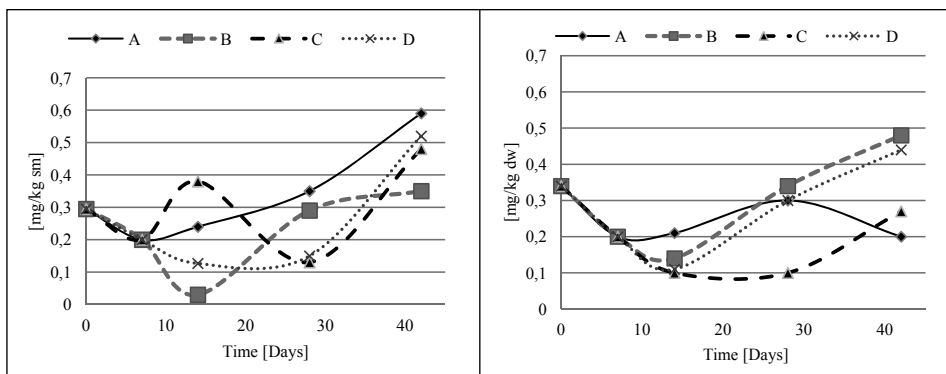

Fig. 4. Cadmium content [mg/kg d.w.] changes in control G (left) and experimental GP (right) series, during 42 days model experiment. Experimental layers marked as explained in Materials and methods

Despite significant lead content in the sediment, the increase of its value was respectively low, however, unexpectedly high in layer *D* for series G (20% increase) and layer *C* for GP (16% increase). On the last day of the experiment there was a significant (p = 0.05) statistic difference between the concentrations of lead in the series G and GP, indicating a significant reduction of the mobility of this element in the soil after the application of ash fertilization [18, 19]. In agricultural Polish soils the average lead content is 14 mg Pb/kg dry matter, which is similar to the concentrations observed in both tested cores at the beginning and the end of the experiment [21].

The migration of chromium to layer *C* (in G series) was observed as early as the 7th and 14th day, but the highest concentration (8.8 mg/kg d.m. representing a 82% increase) was noted on the last day in layer *D*. In GP series chromium migration was slower and elevated concentrations were recorded intermittently in different layers. The maximum increase of the last day was observed in layer *C* (41%). A significant mobility of this element was observed in studies of leaching from the ashes in real--time conditions [30]. During the experiment, the positive correlation between the

concentrations of calcium and copper, nickel and chromium were noted only in GP series for the layer *D*.

Manganese in the series G migrated in small amounts, and the maximum concentration coincided with the last day of the experiment. In layers *A* and *D* the concentration levels rose to 56.7% and 59.8%. The increases in concentration of the element in the two outer layers (*A* and *B*) were highly correlated (0.94) which indicates a slow migration in the profile. The increase in concentrations of manganese in the GP series was lower than in the G-series, and was up to 16.2% in layer *D* on the last day of the experiment, but there was no accumulation of manganese in layer *A*.

Under the conditions of increased pH, mobility of many metals is lower, but similar to the soluble organic matter (DOM), copper, nickel, zinc, and chromium may still be mobile, and their content in the deeper levels of the soil may increase. Moreover, they can be actively taken up by plants [6, 19, 22]. Even alkalinization of soils with $Ca(OH)_2$ does not have to effectively restrict the migration of certain metals (e.g, copper) moving along the profile of the migrating groundwater [40]. The concentrations recorded in the
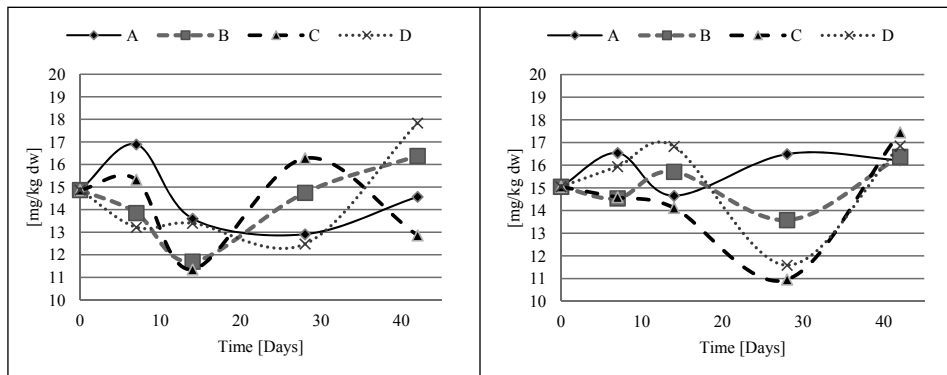

Fig. 5. Lead content [mg/kg d.w.] changes in control G (left) and experimental GP (right) series, during 42 days model experiment. Experimental layers marked as explained in Materials and methods
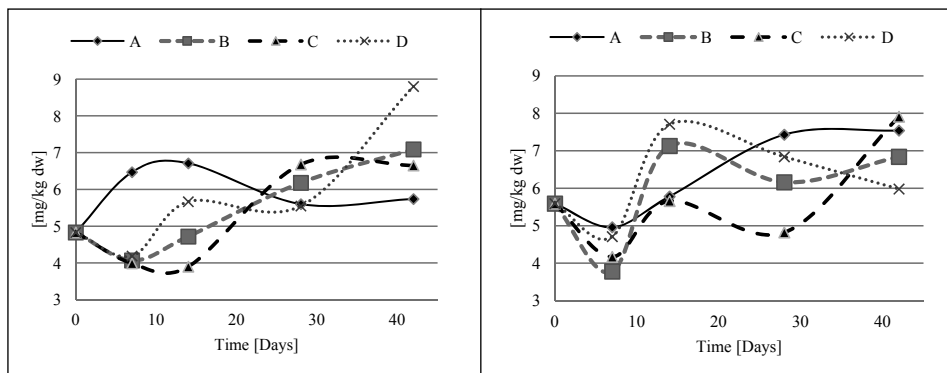

Fig. 6. Chromium content [mg/kg d.w.] changes in control G (left) and experimental GP (right) series, during 42 days model experiment. Experimental layers marked as explained in Materials and methods
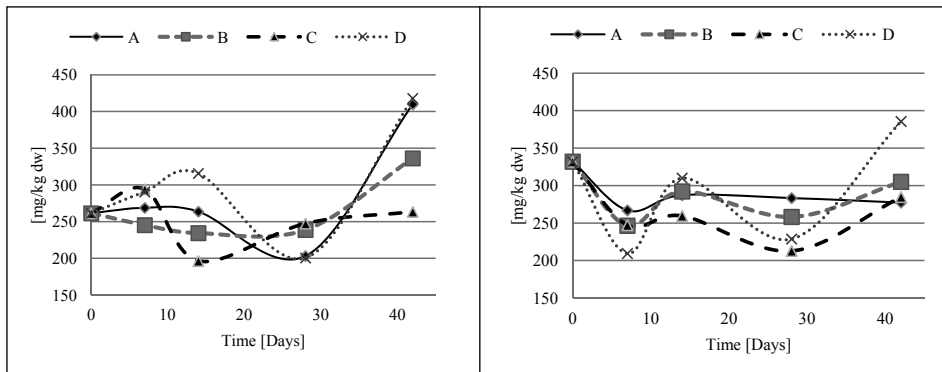
Fig. 7. Manganese content [mg/kg d.w.] changes in control G (left) and experimental GP (right) series, during 42 days model experiment. Experimental layers marked as explained in Materials and methods

experiment were several times lower than those found in the soil on the banks of the river Lahn in Germany, indicating little effect of single impact of flood events simulated in the experiment on the arable soil layer.

CONCLUSIONS

On the ground of the research model, the migration of heavy metals was found from flood sediment to the soil profile. In most cases, the highest concentrations of heavy metals were recorded in layer *A* that had a direct contact with the sediment. The increase in the content of Cu, Ni, Cr and Mn was observed as early as on the 14th day of the experiment. This shows the considerable mobility of the metals in the soil, however, this only applies to the layers *B* and *D* of GP series. In the series G (no added ash) an increased content in these layers was observed only for nickel. A significant increase in the content of the analysed elements was recorded in both series until 42nd day of the experiment, which in the case of the receding flood gives some time to remove the flood sediment. Comparing both series in the layer *D* of the research, greater increases in the concentrations were observed in samples without the addition of ash. It applied to all analysed heavy metals except zinc and indicated reduction in the rate of migration when the soil reaction was increased by fertilization of ashes from biomass. However, taking into account all of the analysed layers of profiles on the 42nd day of the experiment, it appears that in comparison with the control group G, a higher mobility in the experimental group GP was observed for ions Cu, Ni and Pb.

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WPŁYW POPIOŁU Z BIOMASY NA MIGRACJĘ METALI CIĘŻKICH W PROFILU GLEB ZALANYCH WODAMI POWODZIOWYMI – DOŚWIADCZENIE MODELOWE

Gleby, które były narażone na oddziaływanie wód powodziowych w znacznym stopniu mogą być zanieczyszczane związkami nieorganicznymi i organicznymi. Są to przede wszystkim te związki, które są nanoszone w formach rozpuszczonych lub zawieszonych wraz z napływającą silnie obciążoną wodą powodziową, jak i związki powstające w obrębie profilu glebowego, na drodze beztlenowych przemian materii organicznej. Me-

tale ciężkie nanoszone wraz z wodami powodziowymi są sorbowane przez glebę, a także wymywane z nanosów powodziowych przez opady atmosferyczne po ustąpieniu zjawiska powodzi. W niniejszej pracy przedstawiono wyniki badań nad wpływem nawożenia popiołem z termicznego przekształcania biomasy na migrację metali ciężkich (Zn, Cu, Cr, Ni, Pb, Cd, Mn) w warstwie ornej gleb. Wykazano, iż metale pierwotnie zawarte w „nanosie" powodziowym migrują aktywnie w profilu gleby przyczyniając się do wzbogacenia badanych gleb, także w przypadku zastosowania nawożenia popiołem.