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CONTENT OF SELECTED HEAVY METALS IN SOIL AND IN VIRGINIA MALLOW (*SIDA HERMAPHRODITA*) FERTILISED WITH SEWAGE SLUDGE

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Abstract

The aim of this study has been to determine the influence of sewage sludge on the content of selected trace elements in Virginia mallow and in the soil after harvesting these plants. Sewage sludge was the source of nitrogen and phosphorus for the plants, while potassium was supplied in the form of mineral fertilisers. Virginia mallow plants grown without fertilisation served as the contnrol. The form and dose of sewage sludge did not have any larger effect on the concentrations of copper and zinc in the plants. The content of manganese and chromium increased as the the dose of either form of sewage sludge increased. In turn, the content of nickel in Virginia mallow tended to decrease as the doses of sludge rose. The biomass of Virginia mallow contained significantly more cadmium when fertilised with sewage sludge. Significantly more Mn and Pb accumulated in the soil which had been enriched with wet sewage sludge, while the soil treated with pelleted sewage sludge contained more Cd. The soil content of Zn, Mn, Pb and Cr tended to increase as the dose of sewage sludge increased. With respect to nickel and cadmium, their soil content was significantly differentiated by the applied doses of sewage sludge but the direction of these modifications was inconsistent. The forms or doses of sewage sludge applied left the content of copper in soil unaffected. Concerning the accumulation in the aerial parts of Virginia mallow plants, the heavy metals can be ordered as follows: Cd < Cu < Cr < Ni < Zn < Mn. Generally, sewage sludge applied in doses that covered the demand of Virginia mallow for nitrogen and phosphorus did not cause excessive increase in the content of heavy metals in the crop's aerial biomass. However, an increase in the dose of sewage sludge tended to raise the content of mobile forms of heavy metals in soil after harvest of Virginia mallow.

Keywords: wet sewage sludge, pelleted sewage sludge, Virgnia mallow, soil, heavy metals.

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INTRODUCTION

Diminishing resources of fossil fuels as well as the risk of climate change caused by excessive emission of carbon dioxide to the atmosphere force people to search for sources of renewable energy. A possible solution is to use plant biomass. Solid biomass is obtained mostly from waste generated by forestry, agriculture, wood processing and municipal green areas. Some small amounts originate from recycled organic waste collected from households. These resources can be supplemented with biomass grown on field plantations of perennial energy crops (SZCZUKOWSKI et al. 2005, KACPRZAK et al. 2012). According to KABAŁA et al. (2010). Cultivation of energy crops, especially on marginal land and on farmland fertilised with sewage sludge, complies with the principles of permanent and sustainable economic growth because production of biomass for the generation of renewable energy creates an opportunity to reduce the use of non-renewable energy resources. It is also an alternative to using timber from forests for energy generation.

The total amount of municipal sewage sludge produced in Poland in 2013 was 540.3 mln t d.m., of which 105.4 mln t d.m. (19.5%) was used in agriculture and a further 11.7% was dedicated to soil reclamation through the cultivation of plants for composts (*Environmen* 2014). The domestic waste management plan of 2014 envisaged a decrease in the amounts of sewage sludge used in agriculture and land reclamation, but it also highlighted the need of maximising the use of biogenic substances contained in sewage sludge and simultaneously adhering to all sanitary and chemical safety regulations. Numerous studies on the chemical composition of sewage sludge demonstrate that this substance is a rich source of organic matter and nutrients for plants (SMERNIK et al. 2004, WANG et al. 2005, WEI, LIU 2005, FLAVEL, MURPHY 2006, FERNANDEZ et al. 2007, SĄDEJ et al. 2007, BOWSZYS et al. 2009, MTSHALI et al. 2014). When sewage sludge is procesed into fertiliser, the consumption of mineral fertilisers is reduced and biogenic components re-enter the cyclying of elements in terrestrial ecosystems; in addition, the costs of maintaining plant plantations are lower (PETERSEN 2003, STOLARSKI et al. 2014).

The purpose of this study has been to assess the effect of sewage sludge on the content of selected trace elements in Virginia mallow and in the soil after harvesting the plants.

MATERIAL AND METHODS

The study relied on two one-year, two-factor pot experiments performed in four replications (Table 1). The source of nitrogen and phosphprus for plants was wet or pelleted sewage sludge from the Łyna Wastewater Treat-

Table 1

Design of the experiment

Dose of sludge	Pelleted sewage sludge			Wet sewage sludge		
	N	P	K	N	P	K
	(g pot^{-1})					
Control	0.0	0.00	0.0	0.0	0.00	0.0
N1	0.5	0.28	0.5	0.5	0.25	0.5
N2	1.0	0.56	1.0	1.0	0.50	1.0
N3	1.5	0.84	1.5	1.5	0.75	1.5

ment Plant in Olsztyn, while potassium was supplied in the form of mineral fertiliser (potassium chloride). Virginia mallow plants grown without fertilisation served as the control.

Pots were filled with 10 kg of substrate, whose grain-size composition corresponded to light sand. The content of available macroelements, heavy metals soluble in 1 mol dm^{-3} HCl and the pH of soil prior to the experiment are presented in Table 2.

Table 2

Selected chemical properties of the soil before the experiment

pH_{KCl}	Macronutrients (mg kg^{-1})					
	P		K		Mg	
5.6	63.2		125.5		62.0	
Heavy metals soluble in 1 mol dm^{-3} HC (mg kg^{-1})						
Cu	Zn	Mn	Ni	Pb	Cr	Cd
1.479±0.14	9.48±0.95	70.19±19.06	1.765±0.49	3.378±0.57	0.552±0.13	0.049±0.02

The experiment was conducted on Virginia mallow grown from rooted cuttings. After harvest, the plants were weighed, dried and ground. Crushed plant material was digested in a mixture of nitrogen(V) and chloric(VII) acid (in a 4:1 ratio) with added hydrochloric acid; afterwards, the content of trace elements was determined by atomic absorption spectrometry on an AA-6800 Shimadzu apparatus. Determinations of mobile forms of heavy metals in soil prior to the experiment and after harvest were also made by the AAS method, after extraction in 1 mol dm^{-3} HCl.

The results were processed statistically according to analysis of variance (Statistica 10 PL), and the differences between means were compared with the Tukey's test at the significance level of $p = 0.01$.

RESULTS AND DISCUSSION

Two forms of sewage sludge were used in the experiment: wet (dewatered only on a press) and pelleted. The main difference between them was the content of dry matter and organic carbon (Table 3). Except for dry matter, organic carbon, phosphorus, sodium and nickel, wet sludge contained higher amounts of the other components. The content of trace elements in both forms of sewage sludge was below the permitted amounts of heavy metals set in the Regulation of the Minister for the Environment of 13 July 2010, which specifies the legal conditions for using municipal sewage sludge in agriculture.

Table 3

Selected chemical composition of sewage sludge

Specification	Units	Sewage sludge	
		pelleted	wet
Dry matter content	(%)	89.45	20.10
C org.	(g kg^{-1}d.m.)	31.42	25.82
N		61.69	63.88
P		34.70	31.84
Na		0.644	0.291
Ca		57.56	60.66
Mg		22.56	26.65
Pb	(mg kg^{-1}d.m.)	36.60	34.35
Cd		1.26	1.25
Hg		1.37	2.04
Ni		45.14	37.40
Zn		1114.00	1491.17
Cu		223.04	331.33
Cr		89.10	99.37

During wastewater and sewage treatment, heavy metals are removed from the treated substances and accumulated in sludge. Determination of their total content does not fully identify the potential threat they pose to the natural environment. Total content does not enable us to estimate the pool of potentially bioavailable elements. Metals bound to aluminosilicate minerals or sulphates as well as the ones forming permanent organometallic bonds are non-mobile forms and therefore are not perceived as toxicologically dangerous. Metals bound with Fe, Mn and Al oxides or with organic matter can be taken up by plants only under specific conditions including soil reaction, electric conductivity, organic matter content and oxidation and reduction properties of the substrate, as well as the properties of heavy metals in question. Ion-exchangeable oxides and carbonates can permeate into the soil and water environment, where they are a source of nutrients for plants but in excessive amounts create a threat to the plants, soil and water (WILK, GWOREK 2009, GAWDZIK 2012). Whereas LATOSIŃSKA and GAWDZIK (2011) showed that in municipal sewage sludge heavy metals are mainly connected with aluminosilicate. Such sewage sludge is characterised by low mobility Cr, Cd, Cu and Pb. New legal regulations strongly limit the possibility of depositing sewage sludge at municipal waste landfills. A solution to this problem, in particular at small and medium-sized treatment plants, is to dispose of sludge by composting. Composting results in the redistribution of metal forms and the highest decrease of metal mobility in the final product, compared to the feedstock, is achieved for Zn and Cu. The metal binding capacity, however, was observed to have increased for all metals (GUSIATIN, KULIKOWSKA 2012).

Copper, zinc and manganese are the heavy metals that are essential elements for plants. The content of Cu in the aerial biomass of Virginia mallow ranged from 1.90 to 2.67 mg kg^{-1} d.m., while the Zn concentration varied from 15.23 to 24.15 mg kg^{-1} d.m. (Table 4). The form and dose of sewage sludge did not have significant influence on the concentrations of these metals in the plant. By contrast, the content of manganese and chromium increased under the influence of higher doses of either form of sewage sludge. A reverse situation was observed in respect of nickel, whose concentration in Virginia mallow plants tended to decrease as the doses of sewage sludge went up. Significantly less cadmium was detected in the biomass of Virginia mallow fertilised with wet sludge. Most of this metal was determined in the control plants, whilst the lowest cadmium content was found in plants from treatment N1.

According to RYBAK (2006), trace elements contained in biomass play an important role in the process of incineration. Condensation of volatile metals contributes to the formation of submicronic particles of fly ash, which is difficult to remove in filters. Fly ash is dangerous to nature and human health. Allowable concentrations of cadmium and zinc in biofuels, which ensure their safe use, are: Cd < 0.5 and Zn < 80 mg kg^{-1}. The German standard DIN 51731 specifies permissible concentrations of other metals as Cr <8, Cu <5 and Pb <10 mg kg^{-1}.

In our experiment, analogously to the report by SZYSZLAK-BARGŁOWICZ and PIEKARSKI (2009), the biomass of Virginia mallow contained less Cd, Pb and Zn than the threshold levels of allowable amounts. The concentrations of Cd, Cr and Pb did not exceed the limits set out in the aforementioned regulation.

KALEMBASA (2006) reports that the biomass of Virginia mallow contains 16 g kg^{-1} of proper ash, and the total content of heavy metals (Pb, Cd, Cr, Cu, Zn and Ni) corresponds to 0.267% of its amount. Among the heavy metals mentioned, zinc is the most abundant (0.1%), followed by cadmium (0.087%), lead (0.05%), copper (0.018%) and chromium as well as nickel

Table 4

Content of heavy metals in the Virginia mallow (average of two experiments)

Rate of sludge	Cu	Zn	Mn	Ni	Cr	Cd
	(mg kg^{-1} d.m.)					
Pelleted sewage sludge						
Control	2.23	20.35	51.70	18.87	1.67	0.242
N1	2.67	18.71	58.26	19.25	1.46	0.125
N2	2.04	16.35	60.14	15.50	7.84	0.127
N3	1.90	15.23	84.98	12.16	9.60	0.176
Mean	2.21	17.66	63.77	16.44	5.14	0.167
Wet sewage sludge						
Control	2.22	20.26	53.37	18.87	0.67	0.232
N1	2.43	24.15	64.23	11.99	2.32	0.186
N2	2.28	21.17	68.49	11.32	3.53	0.227
N3	2.35	19.27	99.59	12.16	5.35	0.237
Mean	2.32	21.21	71.42	13.58	2.97	0.221
Means for dose						
Control	2.22	20.30	52.53	18.87	1.17	0.237
N1	2.55	21.43	61.25	15.62	1.89	0.156
N2	2.16	18.76	64.31	13.41	5.69	0.177
N3	2.12	17.25	92.29	12.16	7.48	0.207
F emp.						
Form of sludge	0.63	5.39	0.89	4.21	45.05	288.93
Dose	2.00	1.42	4.48	4.45	46.04	127.66
Interaction	1.17	0.66	0.11	1.61	56.78	53.35
Level of significance (p)						
Form of sludge	0.43	0.02	0.35	0.04	0.00	0.00
Dose	0.12	0.25	0.01	0.01	0.00	0.00
Interaction	0.33	0.58	0.95	0.20	0.00	0.00
HSD for $p \leq 0.01$						
Form of sludge					0.65	0.006
Dose			27.64	4.74	1.10	0.011
Interaction					1.55	0.015

(0.006% each). In her study, KRZYWY-GAWROŃSKA (2012) detected more manganese, lead and zinc in Virginia mallow biomass in the first year after an application of sewage sludge, while finding elevated concentrations of cadmium, copper and nickel in the third year. An average content of Cd, Cu, Mn, Ni, Pb and Zn was higher (by 14.3%, 10.0%, 7.3%, 19.3%, 29.1% and 6,9%, respectively) in the biomass of plants grown on plots treated with municipal sewage sludge, with or without brown coal ash possessing a high content of calcium, compared to the treatments with an exclusive application of calcium carbonate or brown coal ash. Helios et al. (2014) concluded that increasing doses of sewage sludge (0-4.2 t ha^{-1} d.m.) did not have any significant effect on the content of heavy metals in the dry matter of prairie cordgrass. In turn, the experiment conducted by Arvas et al. (2013) showed that sewage sludge tested in two doses (25 and 50 t ha^{-1}) did not raise excessively the content of heavy metals in meadow sward. Accordng to Keskin et al. (2010), sewage sludge increased the Cu, Zn, Pb, Cr, Cd concentrations in smooth bromegrass (*Bromus inermis* Leyss.), but its application did not affect the Fe and Mn content of plants. Sewage sludge applied in a fertilising dose stimulated the microbial activity in soil under Virginia mallow and improved the capacity of the plant's photosynthetic apparatus at the end of the growing season (Augustynowicz et al. 2010).

After harvest, the soil in most of the treatments contained more 1 mol dm^{-3} HCl soluble heavy metals than prior to the experiment (Tables 2 and 5). Nickel was an exception in that the soil from the control plots had more of this element than before the onset of the trials. The soil content of the other heavy metals increased, for example there was 2.2-fold more cadmium and 57% more copper. Significantly higher amounts of Mn and Pb were determined in soil which had been enriched with wet sludge, whereas the soil treated with pelleted sewage sludge contained more Cd. The soil content of zinc, manganese, lead and chromium generally increased as the dose of sewage sludge rose. Regarding nickel and cadmium, doses of sewage sludge differentiated the content of these metals in soil, but the direction of these changes was inconsistent. The highest Cu content was determined in the soil from the control trials but neither the form nor the dose of sewage sludge affected significantly the content of this metal in soil. The concentrations of the forms of copper and manganese soluble in 1 mol dm^{-3} HCl implicate moderate soil abundance, while the content of chromium suggests its high richness.

In respect of the content of organic matter and macroelements, sewage sludge from a municipal wastewater treatment plant can be used for agricultural purposes and soil reclamation provided that each batch of sludge is analysed in terms of its content of heavy metals, possibly derived from industrial wastewater, which is often polluted with these elements (Dusza et al. 2009). Numerous research results suggest an increase in the content of heavy metals in soils fertilised with sewage sludge, but it is a relatively small increment and the use of sewage sludge that complies with the currently binding law does not create a risk of soil contamination with heavy metals (Greinert et al. 2009, Mtshali et al. 2014, Bowszys et al. 2015). According to Sienkiewicz and Czarnecka (2012), the rise in the content of available forms of Cu, Zn and Mn detected in alkaline soil fertilised with high doses of sewage sludge (up to 280 t ha^{-1}) did not threaten the natural environment while improving the nutrition of plants with these microelements. Gondek (2010)

Table 5

Content of heavy metals soluble in 1 mol dm^{-3} HCl in soil after plant harvest (average of two experiments)

Rate of sludge	Cu	Zn	Mn	Ni	Pb	Cr	Cd
	(mg kg^{-1})						
Pelleted sewage sludge							
Control	2.36	11.50	80.10	0.81	4.49	0.644	0.113
N1	2.22	11.87	83.86	2.15	5.41	0.802	0.103
N2	1.88	12.69	82.28	1.44	4.88	0.669	0.078
N3	1.93	16.18	87.00	1.92	5.15	0.789	0.113
Mean	2.10	13.06	83.31	1.58	4.98	0.726	0.102
Wet sewage sludge							
Control	2.30	11.85	80.32	0.97	4.49	0.605	0.101
N1	1.80	11.48	84.01	2.08	5.31	0.662	0.097
N2	2.23	15.06	94.31	1.36	5.96	1.010	0.071
N3	2.19	13.18	90.88	2.03	6.30	1.015	0.099
Mean	2.13	12.89	87.38	1.61	5.51	0.823	0.092
Means for dose							
Control	2.33	11.68	80.21	0.89	4.49	0.625	0.107
N1	2.01	11.68	83.94	2.11	5.36	0.732	0.100
N2	2.05	13.87	88.30	1.40	5.42	0.839	0.075
N3	2.06	14.68	88.94	2.00	5.73	0.902	0.106
F emp.							
Form of sludge	0.13	0.25	7.11	2.40	13.51	3.61	7.97
Dose	2.73	20.50	7.16	78.17	5.90	5.76	17.39
Interaction	3.96	10.71	3.35	5.26	4.66	4.86	1.90
Level of significance (p)							
Form of sludge	0.72	0.62	0.01	0.13	0.00	0.06	0.01
Dose	0.05	0.00	0.00	0.00	0.00	0.00	0.00
Interaction	0.01	0.00	0.03	0.00	0.01	0.00	0.14
HSD for $p \leq 0.01$							
Form of sludge			3.05		0.45		0.010
Dose		1.15	5.19	0.24	0.77	0.173	0.024
Interaction	0.42	1.63		0.34	1.09	0.245	

found that fertilisation with manure and sewage sludge in the first year did not cause significant mobilisation of mobile zinc forms in soil. However, due to the mineralisation of organic matter and progressing soil acidification, the content of mobile forms of zinc increased in the second and third year, although the increase was lower than that induced by mineral fertilisation. The research by Możdżer and Krzywy (2013) showed that the total content of manganese, nickel and lead increased (by 17.0, 12.6 and 10.4%, respectively) three years after an application of sewage sludge compared to the soil prior to the experiment. Likewise, the content of 1 mol dm^{-3} HCl soluble forms of Cu, Mn, Ni and Pb as well as the share of soluble forms in the total content of these metals increased. Meanwhile, the share of 1 mol dm^{-3} HCl soluble forms of zinc decreased. Arvas et al. (2013) demonstrated that the content of Cu, Zn, Pb and Cd in the topsoil (0-20 cm) under a meadow tended to increase as higher doses of sewage sludge and the highest amounts of these metals were determined in the second year following an application of sewage sludge. Sewage sludge application had no effect in 20-40 and 40-60 cm soil depth for Fe, Mn, Zn, Cu, Pb and Cd. Applications had very effect in only 0-20 cm soil depth. Compared to control plots, sewage sludge increased DTPA-extractable Fe, Mn, Zn, Cu, Pb and Cd contents of soil (Keskin et al. 2010). Dobrowolska and Janicka (2014) reported that compost substrates made of municipal sewage sludge and used for growing horned violet were rich in nutrients and ensured their adequate supply for the plants. Although such substrates contained large quantities of heavy metals, the concentrations of these elements did not exceed permissible levels established for earth and mineral soils dedicated to plant growing. Milinovic et al. (2014) claimed that by drying sewage sludge before its soil application it was possible to reduce the leaching of zinc while increasing the leaching of copper from this substance.

Compared with the control, sewage sludge in a dose corresponding to 100 kg N ha^{-1} (i.e. 2.8 t ha^{-1} d.m.) significantly increased the height of plants, mass of 1 plant shoot, number of shoots per 1 m^2 and dry matter yield (by 4, 11, 14 and 22%, respectively), but had no influence on the yield structure formation nor did it affect the content of macroelements and heavy metals in the aerial biomass of prairie cordgrass (Helios et al. 2014).

Regarding the level of accumulation in the aerial mass of Virginia mallow plants, heavy metals can be arranged in the following order: $Cd<Cu<Cr<Ni<Zn<Mn$ (Figure 1). Virginia mallow absorbed more Cd, Cu, Zn and Mn from wet sewage sludge, but took up more Ni and Cr from pelleted sludge. Irrespective of the form of sludge, the uptake of cadmium increased as higher doses of sludge, and the highest accumulation of zinc and chromium was detected in Virginia mallow plants fertilised at N2 level. The highest quantities of copper, nickel and manganese were absorbed by Virginia mallow fertilised with the highest dose of wet sludge (N3), whereas the highest uptake of these metals in the series with wet sewage sludge was generally observed on the plots fertilised with the medium dose (N2). According to Ociepa (2013), the uptake of Zn and Cd by plants depended strongly on the concentration of forms of these metals in the soil determined with 0.01 M $CaCl_2$ and 1 mol dm^{-3} HCl. Fertilisation with sewage sludge, despite causing the highest concentration of heavy metals in the soil, did not induce a higher uptake of these elements by plants. The reason was the improved sorption conditions and increased soil pH..

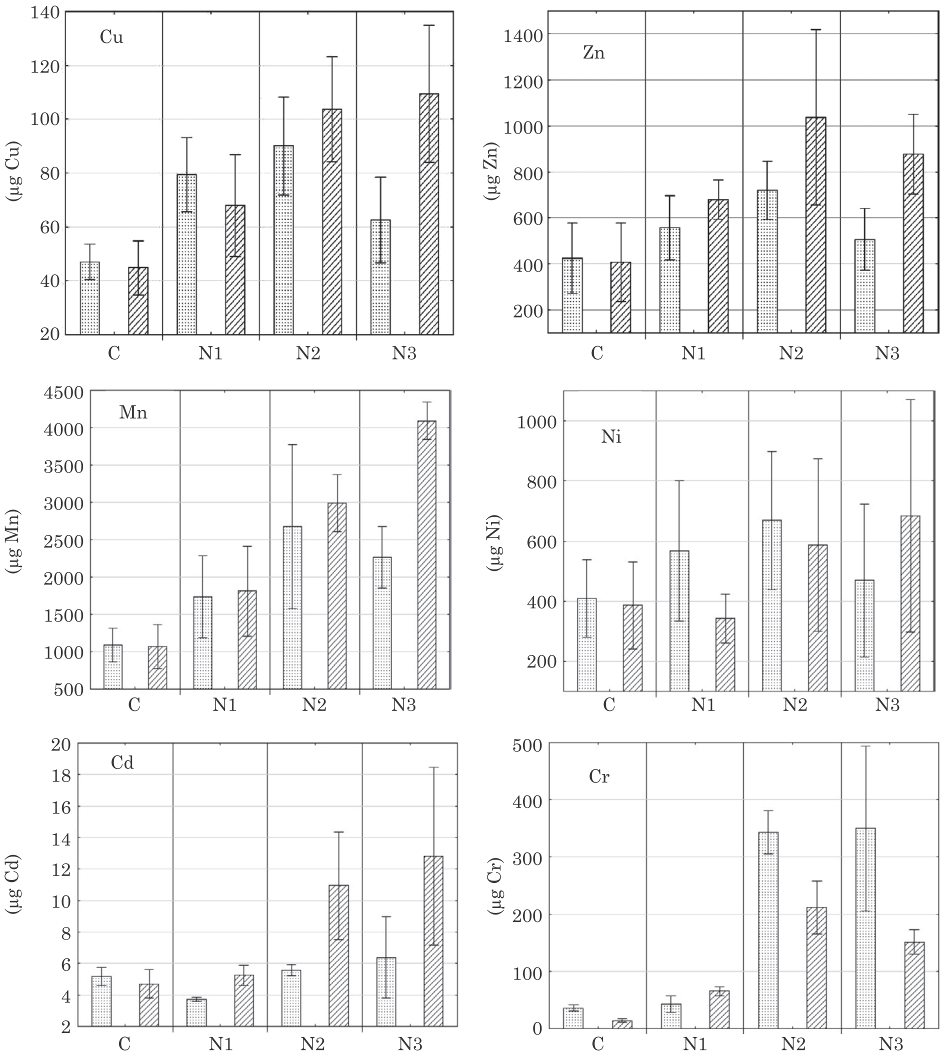

Fig. 1. The uptake of heavy metals with the Virginia mallow harvest depending on the form and dose of sewage sludge (means ± confidence interval, at a level of $p \leq 0.01$)

CONCLUSIONS

1. Sewage sludge did not lead to an excessive increase in the content of heavy metals in the aerial biomass of this plant.

2. Higher doses of sewage sludge tended to result in higher amounts of 1 mol dm^{-3} HCl soluble forms of all the metals except copper. More Mn and Pb were detected in soil treated with wet sewage sludge, while more cadmium accumulated in soil to which pelleted sludge had been applied.

3. Higher amounts of Cd, Cu, Zn and Mn were absorbed by Virginia mallow from wet sewage sludge, while the test plants took up more Ni and Cr from the pelleted form of this substance.

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