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DYNAMICS OF HEAVY METALS LEACHING FROM SOILS WITH SEWAGE SLUDGE ADDITION

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DYNAMIKA ŁUGOWANIA METALI CIĘŻKICH Z GLEB NAWOŻONYCH OSADAMI ŚCIEKOWYMI

Celem prowadzonych badań było określenie zagrożenia dla gleb, wód podziemnych i roślin, jakie może powodować nawożenie osadami z mechaniczno-biologicznej oczyszczalni ścieków o wydajności nominalnej 46 000 m^3/d. Próby gleb pobrano z gospodarstwa rolno-warzywnego. Doświadczenie związane z ługowaniem zanieczyszczeń prowadzono w lizymetrach z PCV (z odpływem wód przesiąkowych).

Analizowane osady spełniają zarówno w zakresie chemizmu, jak i biologii wymogi stawiane osadom przeznaczonym do rolniczego wykorzystania. Odcieki z lizymetrów po 8, 16 i 24 tygodniach symulowanego ługowania opadami atmosferycznymi charakteryzują się w czasie badań zmiennym pH (tendencja wzrostowa), przewodnością elektrolityczną (tendencja spadkowa) oraz obniżającą się nieznacznie zawartością metali ciężkich. Przeprowadzone frakcjonowanie metali ciężkich w osadach ściekowych metodą sekwencyjnej ekstrakcji chemicznej wykazało, że występują one głównie w postaci związków trudno uwalniających się do roztworu glebowego (frakcje III, IV, V). Analiza sekwencyjnej ekstrakcji chemicznej przeprowadzona w glebie z zaaplikowanym osadem ściekowym po 24 tygodniach stosowania symulowanych dawek opadów wykazała podobną tendencję występowania metali ciężkich jak w samych osadach, przy czym sumaryczna zawartość ich we frakcjach I–III wynosiła od 18,6% dla Cr do 44,8% dla Zn. Pozostała ilość metali związana była głównie z frakcją V, całkowicie niedostępną dla roślin.

Summary

The goal of the study was to determine the risk posed to soil, groundwater and plants by the application of sewage sludge from a mechanical-biological wastewater treatment plant of nominal capacity of 46 000 m^3/d. as fertilizer. Soil samples were collected from an agricultural and vegetable production farm. The leaching experiment was carried out in PCV lizymeters (with percolation water outlet).

With respect to the chemistry and biology, the analyzed sludge meets the standards set up for sludge used for agricultural purposes. After 8, 16 and 24 weeks of simulated leaching with atmospheric precipitation, the lecheate from lizymeters showed changes in pH (increasing tendency), electrolytic conductivity (decreasing tendency) as well as slightly lowering content of heavy metals. Heavy metal speciation in sewage sludge showed that they occur in forms of compounds sparingly releasable to the soil solution (fractions III, IV, V). The analysis of sequential chemical extraction carried out in soil with applied sewage sludge, after 24 weeks treatment with simulated atmospheric precipitation doses showed similar heavy metal occurrence tendency as in the case of pure sludge. The total heavy metal content in fractions I–III amounted from 18,6% for Cr to 44,8% for Zn. The remaining content of heavy metals was basically bound with fraction V, which is completely unavailable for plants.

INTRODUCTION

Sewage sludge is a product of the mechanical, biological and physico-chemical treatment of domestic sewage, industrial effluent, agricultural wastewater and storm-water.

In Poland, approximately 17 million tons of sewage sludge is generated yearly. At the hydration level of 98%, the mentioned volume is an equivalent of approx. 340 thousand tons of dry mass per year.

Every year about 300 newly constructed wastewater treatment plants are put to operation, while about one thousand is still under construction. Operation of a wastewater treatment plant is closely related to the necessity of handling the generated sludge in an appropriate manner. Recently, a significant advancement in the development of sewage sludge treatment technologies has been observed. Nevertheless, the problem of its safe utilization is still an open question. Effective solutions to address this problem are even more difficult to find as the volume of the generated sewage sludge systematically increases while its quality does not always meet the requirements of the environmental protection [1, 2, 4, 9].

Nearly 20% of the municipal and industrial wastewater treatment plants dispose their sludge on discharging or landfill sites and only few facilities follow the formally regulated sewage sludge management procedures [10].

Rational utilization of sewage sludge should constitute the basic principle of sludge management in a wastewater treatment facility as the number of barriers in the disposal and storage of sludge continuously increases.

The content of organic matter and nutrients in sludge justifies its suitability for agrotechnical purposes. However, sewage sludge may also contain hazardous substances (e.g. heavy metals), significant amounts of pathogenic bacteria, viruses, protozoans, parasites etc., and toxic organic compounds (e.g. pesticides, polychlorinated biphenyls). All of the mentioned hazardous factors are impediments to utilize the sewage sludge in a natural way, especially for agricultural purposes. Due to that reason, while developing effective methods for sludge stabilization and dehydration, efforts should be made to reduce the volume of generated sludge and the content of hazardous substances in it.

In Poland the investigations of sewage sludge properties were only initiated in mid 60's. Complex studies of the chemical and physical properties of sludge from the viewpoint of its natural utilization have been carried out and are still ongoing in a number of scientific centers across the country

Sewage sludge and sludge compost mixed with other organic waste (wood chips, bark, green waste) can be applied for the following purposes:

– reclamation of waste land and brownfields;

– organic fertilization of soils in agricultural areas, forests (tree nurseries, wood production), in towns and cities (e.g. green areas) industrial sites, communication areas and in a number of other biologically active lands;

– land improvement.

The type of sludge, its sort and chemical composition decide upon its utilization option. Beside the basic criterion i.e. the content of heavy metals, sludge composition analysis must consider also such issues as the content of agriculturally utilizable components, sanitary and microbiological parameters, type of arable land on which the sludge is to be added as well as type of crops. Appropriately prepared, safe sewage sludge applied as compost or component of fertilizing mixtures could become a competitive product for mineral and organic fertilizers, which in some cases may negatively affect the environment and in particular water environment.

Both natural utilization and disposal of sludge are forms of its discharge to soil or on its surface. The impact, which the sludge may cause to the environment, depends to a large degree on the site characteristics and method of sludge discharge to the soil.

The following factors should be considered when making a decision on the application of sewage sludge to a specific site:

– depth of aquifer;

– distance from surface waters;

– land slope;

– soil permeability;

– soil pH;

– capacity of the soil sorption complex;

– depth of the bed rock under the land surface and its type.

The soil forming and fertilizing properties of sewage sludge depend first of all on the content of organic matter, biogens and microelements. Higher content of fertilizing components occurs in non-dehydrated sewage sludge. In general, to the mineral fertilizing components the following are categorized: nitrogen, phosphorus, potassium, calcium, magnesium, manganese, iron, sulfur, boron, zinc, copper, molybdenum and cobalt. The group of toxic mineral components comprises: cadmium, mercury, chromium, nickel, arsenic, as well as zinc, copper, cobalt, boron and molybdenum when occur in excessive concentrations [11, 12].

In the countries where the natural resources economy has been taken under appropriate management, sewage sludge contains less heavy metals due to the fact that wastewater with excessive concentration of heavy metals cannot be discharged to the sewage system i.e. to the treatment plant. It can be expected that similar policy will be enforced in Poland [6].

GOAL OF THE STUDY

The goal of the study was to determine the risk posed to soils, groundwater and plants by the sewage sludge from the Opole treatment plant applied as soil fertilizer.

METHODOLOGY OF THE STUDY

Samples of sewage sludge were collected from the lagoons of the Municipal Wastewater Treatment Plant in Opole. This facility uses a combined mechanical and biological treatment process. Its target capacity is 55 200 m^3/d., the nominal efficiency is 46 000 m^3/d. while the actual throughput of the hydraulic system is 36 000 – 40 000 m^3/d. The facility site is prepared for the expansion of the treatment plant to the capacity of 72 000 m^3/d. The treatment process in the Opole plant comprises wastewater pretreatment i.e. mechanical treatment, first level treatment and biological treatment [1, 5].

Soil was sampled from the agricultural and vegetable production farm in 1998. Soil sampling was carried out in compliance with the branch standard BN-78/9180-02. Sludge samples were collected according to the requirements of the 86/278/EC Directive of 12 June 1986 and the Regulation [8].

The pH of sludge, soil and lecheate was determined by ELMETRON CP 315 pH meter [3]. Specific electrolytic conductivity of the lecheate was determined according to the Polish standard PN-77/C-04542.

Total heavy metal content was determined using the methodology applied by IUNG (Institute of Soil Science and Plant Cultivation) in Puławy, Poland. Analysis of water extracts and lecheate was made using atomic absorption spectrometry (AAS) by PHILIPS PU 9100X spectrometer.

Speciation of the sludge contained metals was carried out using sequential extraction procedure according to [14]. In the course of the extraction the following fractions were separated:

I – exchangeable metals;
II – carbonates bound metals;
III – iron and manganese oxides bound metals;
IV – organic matter bound metals;
V – silicates bounded metal residual.

Analysis of water extracts from individual fractions was carried out using atomic absorption spectrometry (AAS) by PHILIPS PU 9100X spectrometer.

METHODOLOGY OF THE LYSIMETER TEST

The experiment was performed using containers – PCV lysimeters (with percolated water outlet) in three replications.

Sewage sludge (118 g soily sludge), recalculated from the dose of 3.0 Mg dry mass/ha × year, was put to lysimeters filled with soil (43 kg of soil), and poured with a dose simulating atmospheric precipitation typical for the Opole area. The daily dose of 2515 cm^3 was an equivalent of a 15-day precipitation (average for Opole). The experiment was continued for 12 days simulating a 6-month precipitation.

Eluats were collected after each pouring dose and analyzed. Upon the completion of the experiment an analysis of the soil with applied sewage sludge was carried out.

CHARACTERISTICS OF THE INVESTIGATED SLUDGE

A comparative analysis of the microbiological and parasitological data presented in Table 1 with the permissible standards shows that the *Coli* test index (g of dry mass) of the sludge from the lagoons meets the standard effective to August 1999 as well as the standard set up in the Regulation currently in power. Meeting appropriate standards means that the sludge is suitable to be utilized for agricultural purposes. In addition, its suitability is proved also by the values determined in the tests of fecal *Coli*, *Clostridium perfringens* and *Salmonella* rods. The investigated sludge is also parasitologically safe.

Table 1. Microbiological and parasitological characteristics of the sewage sludge in the years 1991–1996

Specification	Sludge from lagoon	Regulation of Polish Ministry of Environment of 1.10.1986*	Recommendations of the Institute of Farming Medicine*	Regulation [8] of 11.08.1999
Coli test (g of dry mass)	0.0026–0.7	not less than 0.01	not less than 0.01	–
Fecal *Coli* test (g of dry mass)	0.13–0.7	0.001	0.001	–
Clostridium perfringens test (g of dry mass)	0.0053–0.02	–	not less than 0.01	–
Salmonella rods in 20 g of sludge	not detected	undetectable	undetectable	undetectable
Ascaris lumbricoides and *Trichocephalus trichuria* eggs in 100 g of sludge	not identified	max 1 in 100 g	max 1 in 100 g	ATT to 10 in 1 kg of dry mass (for agricultural purposes)

* Effective to the date of 11.08.1999.

Source: Data from the research carried out by the Provincional Sanitary and Epidemiological Station in Opole [13].

Total heavy metal contents (Table 2) do not exceed maximum permissible standards set up in the Regulation of the Ministry of Environment (MOŚZNiL) [8].

Table 2. Total heavy metal content in the sewage sludge

	Cd	Cr	Cu	Ni	Pb	Zn
Total metal content in sewage sludge [mg/kg of dry mass]	3.67	117.28	108.12	26.,3	114	1006.12
Permissible standards set up in Regulation [8] applicable in the case of fertilizing medium soils with sewage sludge	2	75	50	35	60	120

Sequential extraction was carried out to determine the species of heavy metals that occur in the analyzed sewage sludge.

Fraction I – exchangeable, contains the most soluble in water metals. Metal content in this fraction is very small. Only nickel (1.9 – 7.2%), lead (1.9 – 2.9%) and zinc in smaller amounts (0.1 – 2.5% – total metal content in fractions I – V) were extracted.

Fraction II – metals bound with carbonate fraction, which can be easily released in acid environment. Attention should be paid to the percentage of nickel (1.3 – 4.4%) and lead (1.3 – 3.3% – total metal content in fractions I – V).

Fraction III – metals bound with iron and manganese oxides, occur in the form of biding agents or coatings covering mineral particles; sensitive to redox potential changes. The contents of individual elements in this fraction are differentiated. In the applied conditions, zinc (31.4 – 62.1%) was leached in the highest amount; then nickel (23.9 – 36.8%) and cadmium (20.0 – 42.9%), chromium (9.3 – 17.0%), lead (1.4 – 6.6%) and copper (0.5 – 3.2% – total metal content in fractions I – V).

Fraction IV – metals from the organic fraction are released in the process of natural mineralization of the organic substance, however they are only partially soluble in soil water. In this fraction the highest determined value was for copper (26.0 – 71.9%), slightly less for nickel (26.1 – 51.6%), chromium (4.0 – 46.5%) and zinc (12.2 – 35.6% – total metal content in fractions I – V value).

Fraction V – contains metals bound with siliceous minerals and other persistent compounds. Metals from this fraction are chemically stable and biologically inactive. This fraction contains significant amounts of all analyzed heavy metals, especially lead (87.5 – 95.2%), cadmium (47.4 – 80.0%), chromium (43.8 – 79.0%), copper (27.6 – 72.9%), zinc (21.7 – 32.1%) and nickel (20.1 – 29.6% – total metal content in fractions I – V).

Generally, it can be stated that only small amounts of nickel, lead and cadmium (the most mobile metals) are bound with fraction II and I. The content of other metals in fraction I and II is insignificant.

CHEMICAL AND PHYSICAL PROPERTIES OF THE ANALYZED SOIL

Based on the above presented data (Table 3) it can be stated that the content of copper, zinc, cadmium, chromium, nickel and lead in the analyzed soil falls under the low values of contents permissible in the case of sludge application for agricultural purposes.

Table 3. Total heavy metal content in soil

	Cd	Cr	Cu	Ni	Pb	Zn
Total heavy metal content in the analyzed soil [mg/kg]	0.22	7.6	4.6	1.94	13.2	33

Table 4. Soil pH and electrical conductivity

Determination		Content
Soil pH before analysis	H_2O	7.31
	1N KCl	6.83
Electrical conductivity [μS/cm]		81.8

The analyzed soil is characterized by very low electrical conductivity 81.8 μS/ cm and the pH of 6.83 in 1N KCl. These parameters allow categorizing the soil as slightly alkaline (Table 4).

LECHEATE ANALYSIS

The pH of the lecheate increased from 6.6 to 8.7 between the 6th and 18th week of the experiment (Fig. 1). Permissible pH values for wastewater discharged to water and soil are 6.5–9.0 pH [7].

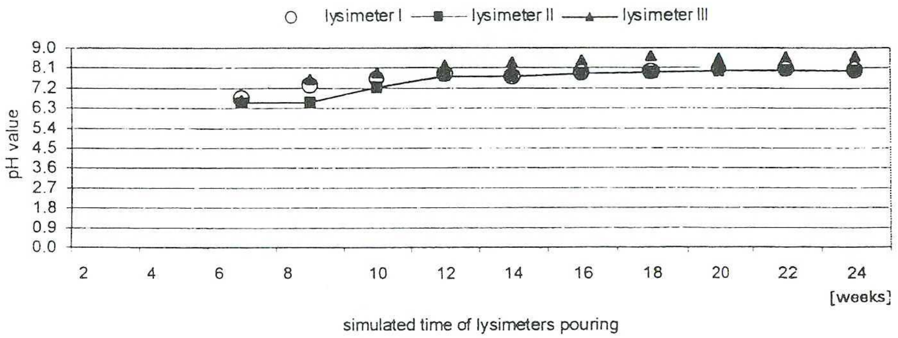

Fig. 1. Changes of lecheate pH during the experiment

Specific electrolytic conductivity in the analyzed lecheate (Fig. 2) showed decreasing tendency. The examined lecheate was characterized by specific electrolytic conductivity in the range of 2.450 (6th week) to 0.637 (24th week) mS/cm (lysimeter I); 1.473 to 0.616 mS/cm (lysimeter II); and 2.080 (6th week) to 0.712 (24th week) mS/cm in lysimeter III. This parameter, however, is not standardized by the Regulation [7] on the permissible contents of substances in wastewater discharged to water and soil.

The total heavy metal content in lecheate (Fig. 3) decreased in time, however it did not exceed the permissible standards for wastewater discharged to water and soil [7].

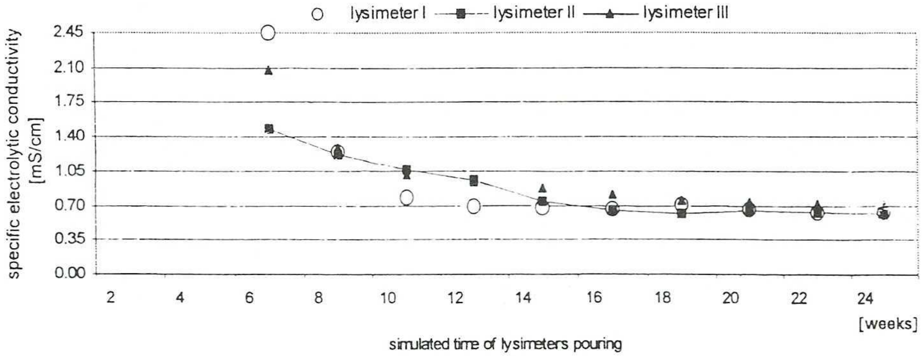

Fig. 2. Changes of conductivity in the lecheate during the experiment [mS/cm]

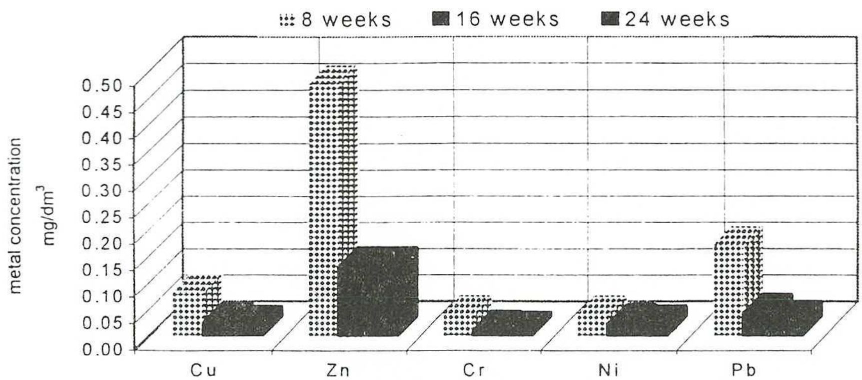

Fig. 3. Total (quantitative) content of heavy metals in lecheate

The highest concentrations of heavy metals in the analyzed lecheate amounted: for copper – 0.08 mg/dm^3, zinc – 0.457 mg/dm^3, chromium – 0.04 mg/dm^3, nickel – 0.04 mg/dm^3 and lead – 0.17 mg/dm^3.

High reduction in heavy metal concentration in the filtrate measured after 4, 8 and 12 days of leaching with water (which is correspondent to respectively 2, 4 and 6-month dose of precipitation for Opole area) was observed in the case of total chromium content (respectively: 0.040; 0.009 and 0.007mg/dm^3); and lead (0.17; 0.042 and 0.028 mg/dm^3). The sequentially descending concentrations in the analyzed lecheate were also observed for zinc (respectively: 0.475; 0.13 and 0.129 mg/dm^3), copper (0.08; 0.024 and 0.019 mg/dm^3) and nickel (0.04; 0.022 and 0.02 mg/dm^3).

During the whole experiment period only trace amounts (<0.002 mg/dm³) of cadmium were determined.

The above presented data shows that in the case of soils with sewage sludge application, heavy metals weakly bound with soil solid fraction are leached as first by atmospheric precipitation. These are (in the descending order): Zn, Cr and Pb, Cu and Ni. Data from chemical sequential extraction of soil with sludge application after 24 weeks of leaching is shown in Figure 4.

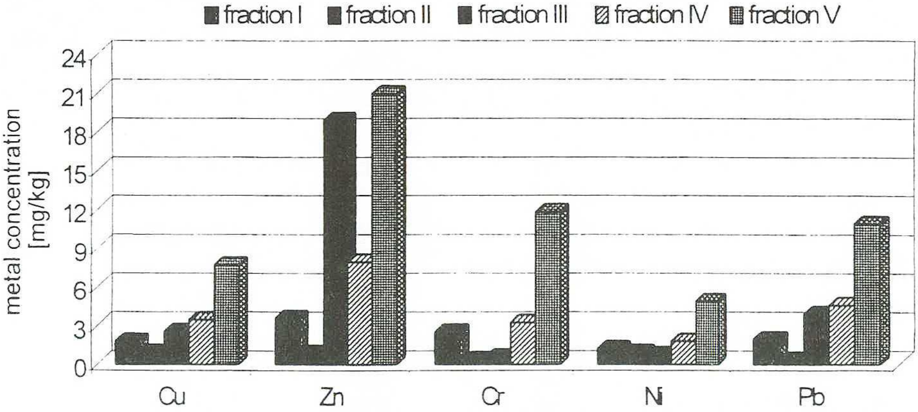

Fig. 4. Heavy metal fractions in soil with sludge application after 24 weeks of simulated washing with atmospheric precipitation

The loads of contaminants [mg/kg] washed out from the soil with sludge application (Table 5, 6) were calculated according to the following formula: $m_w = c*v/m_p$, where: m_w – mass of the substance washed out from the soil [mg/kg]; c – concentration of the washed out substance in the eluat [mg/dm³]; v – total eluat volume [dm³]; m_p – soil mass [kg].

Table 5. Total heavy metal contents in lecheate [mg/dm³]; load of the washed out contaminants [mg/kg]

	Cu		Zn		Cd		Cr		Ni		Pb	
	mg/dm³	m_w [mg/kg]	mg/dm³	m_w [mg/kg]	mg/dm³	m_w [mg/kg]	mg/dm³	m_w [mg/kg]	mg/dm³	m_w [mg/kg]	mg/dm³	m_w [mg/kg]
8 week	0.08	0.005	0.475	0.031	<0.002	Nd.	0.04	0.003	0.04	0.003	0.17	0.011
16 week	0.024	0.005	0.130	0.028	<0.002	Nd.	0.009	0.002	0.022	0.005	0.042	0.009
24 week	0.019	0.004	0.129	0.027	<0.002	Nd.	0.007	0.001	0.02	0.004	0.028	0.006
Total		0.014		0.086		Nd.		0.006		0.012		0.026
Per. Stand. [8]	0.5		2		0.1		0.5		2		0.5	

Table 6. Volume of lecheate during the experiment [dm^3]

Duration of leaching	8 week	16 week	24 week	Total
Average volume of lecheate [dm^3]	2.820	9.182	9.227	21.229

It can be noted that during the period of 24 weeks the following loads of metals were removed from the analyzed soil: zinc – 0.09 mg/kg, lead – 0.026 mg/kg, copper – 0.014 mg/kg, nickel – 0.012 mg/kg and chromium – 0.006 mg/kg.

In the case of copper it was stated that the amounts of load leached after 8 weeks and 16 weeks of washing were the same. The washed out loads of zinc, chromium, and lead showed a decreasing tendency during the leaching period. The lowest washed out load of nickel was recorded after 8-week period while the highest – in the 16th week of the experiment. After the following 8 weeks of leaching the amount of the washed out nickel decreased again.

SOIL ANALYSIS AFTER SLUDGE APPLICATION

The content of copper in the analyzed sample of soil with sludge application was on average 4.6 mg/kg, zinc content was approx. 43.3 mg/kg, chromium content – 7.6 mg/kg, nickel – 6.0 mg/kg and lead – 15.6 mg/kg.

The total heavy metal contents (Cd, Cr, Cu, Ni, Pb i Zn) determined in soil with sewage sludge application (Table 7) proved that the addition of sludge has not resulted in any distinct elevation of metal concentrations in the soil.

Table 7. Total heavy metal contents in soil with sewage sludge application before and after leaching process

	Cd	Cr	Cu	Ni	Pb	Zn
Total heavy metals contents in the soil with sludge addition [mg/kg]	0.22	7.6	4.6	6.0	15.6	43.3
Total heavy metals contents in the soil with sludge addition after leaching [mg/kg]	0.22	6.60	4.20	3.70	13.92	40

ANALYSIS OF SOIL AFTER SLUDGE APPLICATION AND LEACHING

The pH of soil with sewage sludge application was 6.59 in 1N KCl and 7.36 in H_2O, while the electrical conductivity was 74.1 μS/cm (Table 8). The obtained data does not show any meaningful differences in comparison to the corresponding values determined in pure soil (Table 4).

Total content of metals (Cd, Cr, Cu, Ni, Pb and Zn) (Table 7), determined in the soil after sludge application and leaching, proved the fact that the 24-week

Table 8. pH and electrical conductivity of soil with sewage sludge application after the completion of the experiment

Determination		Content
pH	H_2O 1N KCl	7.36 6.59
Electrical conductivity [μS/cm]		74.1

leaching (due to the washed out metal load) resulted in the reduction of Cr, Cu, Pb and Zn by approx. 10%, Ni by approx. 40%, while Cd did not wash out at all.

Qualitative heavy metal analysis allowed identifying metal bindings and forecasting the changes occurring in the soil environment after sludge application and leaching with rainwater.

Based on the performed investigations (Fig. 4), the following was stated:

Fraction I – chromium – 13.5% and nickel – 12.9%, were bound in the highest amounts, then copper – 10.9%, lead – 8.9% and Zn – 6.9% of the total metal content in fractions I–V.

Fraction II – the determined amounts of nickel and copper were, respectively: 9.8 and 5.75% of the total element content, while the content of chromium, lead and zinc did not exceed 2% of their total content.

Fraction III – with this fraction mainly zinc – 36.2%, lead – 18.1% and copper – 15.45% were bound, and nickel and chromium in smaller amount, respectively: 8.2 and 3.2% of the total element value for fractions I–V.

Fraction IV – lead and copper were bound each in: 21.1%, and nickel, chromium and zinc: 18,6–17,6–15,1% of the total contents of these metals.

Fraction V – chromium was bound in 63.8%, nickel – 50.5%, lead – 50.0%, copper – 46.7% and zinc – 40.1% of their total contents.

Only trace amounts ($<0{,}22$ mg/dm^3) of cadmium were determined throughout the experiment period.

Data from the sequential extraction of heavy metals showed that in the first rate the following metals are released from the soil with sludge application: chromium, nickel and copper bound mainly in the exchangeable fraction (I), nickel and copper in the carbonate fraction (II) and zinc, lead and copper with iron and manganese oxides (III). The analysis showed also, that the highest content values of the above mentioned metals are bound in fraction V (40–63.8% of the total element content). This means that their availability for plants is rather limited.

CONCLUSIONS

1. The analyzed sewage sludge meets chemical and biological standards [8] set up for sludge applied for agricultural purposes.

2. Metal speciation by sequential extraction of the analyzed sewage sludge is an additional criterion to decide upon its application for agricultural use. In

the analyzed sludge, heavy metals occur mainly in the forms sparingly releasable to soil solution (fractions III, IV, V).

3. The parameters of the lecheate from the lysimeters after 8, 16 and 24 weeks of simulated leaching with atmospheric precipitation were changing as follows:

– pH 6.6–8.7 (an increasing tendency);

– specific electrolytic conductivity: from 2.45 to 0.616 mS/cm (a decreasing tendency);

– slightly decreasing heavy metal content which does not exceed the permissible standards for wastewater discharged to water and soil.

4. After 24 weeks of simulated precipitation fall, the chemical sequential extraction of soil with sludge application showed that mainly Cr, Ni and Cu are bound in the exchangeable fraction (I), Ni and Cu of iron and manganese oxides in carbonate fraction (II) and Zn, Pb and Cu in fraction III. However, the total content in fractions I–III for Cr is only 18.6%, Ni – 30.9%, Cu – 32%, Zn – 44.8%, Pb – 28.8%, the rest of the metal content is basically bound with fraction V (40–63.8%) – completely unavailable for plants.

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