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Anna JASIŃSKA
Czestochowa University of Technology, Faculty of Infrastructure and Environment
ul. Brzeźnicka 60A, 42-201 Częstochowa
e-mail: aniiaa.jasinska@gmail.com

The Importance of Heavy Metal Speciation from the Standpoint of the Use of Sewage Sludge in Nature

Znaczenie specjacji metali ciężkich w aspekcie przyrodniczego zagospodarowania osadów ściekowych

The development of highly efficient methods of wastewater treatment and the emergence of new wastewater treatment plants is accompanied by the gradual increase in the amount of sewage sludge generated in Poland. However, no waste-free technologies or effective solutions for the complete elimination of the treatment products from the environment have been developed to date, which represents a challenge to the rational management of this type of waste. One of the strategies proposed in this area is the natural use of sewage sludge, mainly to fertilize soils and plants and for reclamation of degraded soils and soilless areas. The prerequisite for finding such a solution is an appropriate quality of sludge intended to be transformed into fertilizers, which has to meet specific hygienic, sanitary and chemical requirements. In the case of the criterion of chemical composition, current standards are mainly applicable for the total heavy metal concentrations in the sludge. In recent years, however, much attention has been paid to the capability of microelements to migrate in the environment, which allows for better evaluation of the quality of biosphere components and the potential risk of the spread of pollution. One of the methods to identify the forms of elements in the medium studied is speciation analysis, based on sequential extraction. The aim of this study was to indicate the importance of speciation in the evaluation of the quality of sewage sludge and the opportunities of its use in nature. Furthermore, one of the most popular methods of determination of the mobility of heavy metals was also discussed. Knowledge of heavy metal forms occurring in the sewage sludge tested allows for preparation of effective waste management strategies.

Keywords: waste management, sewage sludge, speciation, heavy metals

Introduction

Recent years have seen progressive modernization and expansion of the infrastructure in the field of sewage collection and treatment. This phenomenon is primarily connected with the need to adjust environmental protection standards in Poland to the EU requirements [1]. However, this contributes to an increase in the volume of sewage sludge generated as a product of processes used in wastewater treatment plants. No waste-free method or effective solution has been developed so far that would allow for the complete elimination of sewage sludge from the environment [1, 2]. In light of current legal standards concerning waste management, the rational sewage sludge managements represents a major challenge.

The strategies in sewage sludge management are adopted based on legal acts and regulations specific to this type of waste, the way it is generated and processed, and the level of threat to the environment. Therefore, the choice of the method of disposal or use of sewage sludge is determined by strictly defined legal criteria concerning its quality [1, 3]. Figure 1 shows the directions in sewage sludge management in Poland.

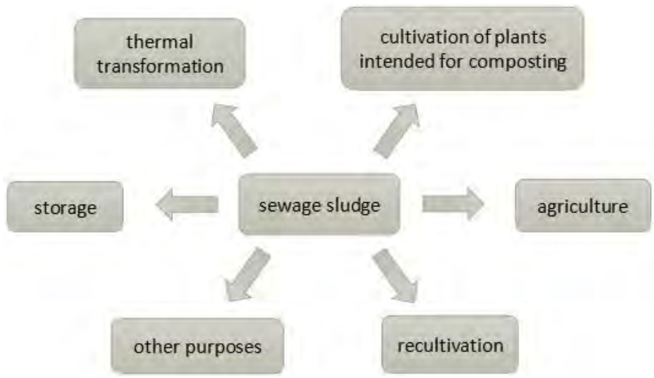

Fig. 1. Directions in sewage sludge management in Poland [3]

According to the assumptions of the 2022 National Waste Management Plan, more and more attention in sewage sludge management should be paid to its use in nature [4]. This approach is justified from both the economic and environmental points of view. However, as with any method, there are some opportunities and limitations [5]. The risk involved in the use of sewage sludge in nature is primarily related to the discharge of additional pollutants that may be present in this waste. Therefore, the decision on their natural use must be based on a thorough analysis of the sludge and the area into which it is planned to be discharged [6-8].

One of the criteria that determine the choice of the use of sewage sludge in nature is its chemical composition, with particular focus on the content of heavy metals [1, 5]. However, determination of the concentration of trace elements in the medium does not allow for a precise analysis of the potential risks involved in sewage sludge discharge into the natural environment. Heavy metals may migrate between soil layers, permeating into groundwater and surface water, and consequently into plants, which, through consumption, represent the sources of such compounds for humans [9]. The mobility of heavy metals is determined by the chemical forms in which they occur in the environment [10].

There are a number of techniques to determine the migration capacity of metals. Most of them are based on extraction by means of various types of reagents, allowing for the isolation of specific fractions of elements from the sample tested. Methods of chemical leaching of metals can be divided into single-stage or multistage. The latter are more popular due to their accuracy. However, they are more time-consuming and require greater volumes of extraction solvents [10, 11].

The most commonly used method of speciation of heavy metals in sewage sludge today is the four-step BCR (Community Bureau of Reference) procedure. This method was developed as a result of activities within the Measurements and Testing Programme, with its aim being unification of extraction-based testing procedures [2, 10].

The assessment of the mobility of heavy metals in sewage sludge allows the researcher to estimate the risk of discharge of toxic elements to the biosphere, which undoubtedly is involved in the natural directions of sewage sludge management [2, 10].

1. Use of sewage sludge in nature

With its application to stabilisation processes limiting the migration of heavy metals in soils, recycling of organic waste such as sewage sludge and compost has been the focus of scientific research for many years. Sewage sludge can be effectively used for fertilization of soil intended for agricultural or non-agricultural use in nature, as well as for reclamation of degraded soils and soilless areas [1, 5]. A study by Ociepa et al. [12], concerning unconventional soil fertilisation demonstrated that the use of a mixture of sewage sludge, brown coal and ash reduces bioavailability of zinc, cadmium and lead. Both fertilization with sewage sludge alone and in the form of a mixture with coal and ash elevates sorption capacity of soil, which is one of the factors determining the presence of mobile forms of metals in the soil environment. However, in all cases of such procedures, sewage sludge has to meet specific chemical and hygienic requirements [13]. First and foremost, it should be subjected to stabilization and hygienization processes [1]. Stabilization of sewage sludge can be achieved through biological, chemical and thermal processes, which leads to changes in the chemical and biological composition of the sludge [3]. The conditions to be met by sewage sludge intended for use in nature were stipulated in the Regulation of the Minister of the Environment of 6 February 2015 on municipal sewage sludge (Item 257) [13].

In the case of the criterion of chemical composition, analysis concerns mainly the total heavy metal concentrations in the sludge tested. Depending on the planned method of sludge management, the permissible levels of these compounds are presented in Table 1 [13].

In hygienic and sanitary terms, sludge is allowed to be used in agriculture if there are no *Salmonella* bacteria and eggs of intestinal parasites such as *Ascaris* sp., *Trichuris* sp., *Toxocara* sp. In the case of reclamation of degraded areas, the permissible number of live eggs of these parasites in 1 kg of dry matter of sludge is 300 [13].

Microorganic pollutants are also an important concern in the field of the use of sewage sludge in nature. These mainly include phenols, chlorophenols, hexachlorobenzene, polychlorinated biphenyls, nitrosamines and pesticide residues. The presence of these compounds in sewage sludge used as a fertilizer is associated with hazards to human health and life in the case of consumption of plants grown in the areas fertilized with such sludge [1].

Table 1. **Permissible concentrations of heavy metals in municipal sewage sludge intended for the use in nature [13]**

Metal	Heavy metal concentration in mg/kg dry matter of sludge not greater than that of municipal sewage sludge		
	In agriculture and for land reclamation for agricultural purposes	For land reclamation for non-agricultural purposes	When adapting land to specific needs specified in waste management plans, land management plans or decisions on land development conditions, to cultivate plants for compost production, to cultivate plants not intended for consumption by humans, and to produce fodder
Cadmium (Cd)	20	25	50
Copper (Cu)	1000	1200	2000
Nickel (Ni)	300	400	500
Lead (Pb)	750	1000	1500
Zinc (Zn)	2500	3500	5000
Mercury (Hg)	16	20	25
Chromium (Cr)	500	1000	2500

The Regulation on Municipal Sewage Sludge also stipulates the permissible levels of sludge that can be applied per year per unit of ground surface area, provided that the permissible heavy metal concentrations are not exceeded. In the case of the use of sewage sludge in agriculture and in land reclamation for agricultural purposes, the permissible level is 3 Mg d.m./ha/year, while for the land reclamation for non-agricultural purposes and for the cultivation of plants not intended for consumption and production of fodder, the permissible dose is 15 Mg d.m./ha/year [13].

In the case of a waste management strategy of the use of sewage sludge in nature, its benefits and risks should be individually analysed. Even if the sludge quality meets the respective legal standards, there is a risk of hazardous compounds being discharged into the environment. In the case of many substances, their long-term effects on living organisms are unknown. Furthermore, legal regulations for some of the well explored toxic compounds have not been specified yet to ensure the high level and broadly understood environmental safety. One example is the levels of mobile fractions of heavy metals, which, due to their properties, may pose a risk of the spread of pollution, but they failed to be stipulated in the waste management law.

2. Mobility of heavy metals

Trace elements may occur in the environment in various chemical forms, as organic compounds, minerals or ions. Some forms have higher migration capacity while others are less mobile. The concept of mobility of heavy metals should be understood as the capability of an element or chemical form to move in the environment. Determination of such properties of microelements is critical to the evaluation of the risks involved in the discharge of sewage sludge into the soil environment [14]. Mobile heavy metal fractions in sewage sludge may migrate deeper into the soil profile, and consequently, become more available to plants [11].

From the legal point of view, analysis of sewage sludge quality can be based only on comparison of total permissible metal concentrations specified in the regulation [13]. In Poland, no regulations have been issued to date to relate directly to the forms of mobile elements. Determination of the concentrations of these compounds does not provide comprehensive information about the risks associated with the use of sewage sludge in nature. The potential risk of pollution in the area subjected to sewage sludge fertilization can be predicted only based on evaluation of the mobility of metals and the possibility of their accumulation in living organisms [11, 15].

The highest risk to soil dwellers is caused by soluble metals, which are highly mobile and readily available. Micronutrients released into groundwater and surface water go up to higher links in the trophic chain. In addition to the evaluation of sewage sludge quality in terms of chemical composition, physicochemical analysis of the soil to be remediated is also useful in determination of the risk of the spread of trace elements in the environment [16, 17].

3. Soil physicochemical parameters affecting the mobility of heavy metals

In the case of solubility of elements, the parameters which are considered decisive include [16, 18]:

- soil pH,
- oxidation-reduction conditions,
- sorption capacity of the soil.

In addition to the above factors, mobility of micronutrients can also be influenced by the activity of various soil dwellers. For example, the presence of some fungi in the soil leads to generation of calcium oxalate in the ecosystem, which in turn is conducive to the formation of heavy metal oxalates which are hardly soluble [19].

3.1. pH value

The pH value of the medium determines the processes of sorption and desorption of hydrogen cations and heavy metal ions. Acid soils are very common in Poland, containing more mobile forms compared to the environment with a lower content of hydrogen ions, where metal oxides are gradually dissolved and toxic elements are released. Cadmium belongs to the fastest soluble metals and is activated at pH of 6.5 [16]. Other metals are released in the following order: Ni > Zn > Mn > Cu > Pb. The content of copper and lead is less correlated with the reaction [19].

Soil acidification is caused both by natural phenomena that occur in the environment and by human activity. The anthropogenic sources responsible for the reduction in pH include forestry, agriculture and products of different sectors of the industry. Excessive acidity can be observed in the areas fertilized with mineral fertilizers for a longer period of time. Furthermore, in industrialised areas, the reduction of concentration of hydrogen ions is caused by the release of nitrogen and sulphur oxides to the atmosphere due to fuel combustion processes. The basic method to reduce soil acidification that allows for the immobilisation of the soluble metal fraction is liming [16, 18]. It is remarkable that in the environment of alkaline reaction, mobile complexes of trace elements may also be formed. However, a noticeable tendency for formation of mobile forms is observed with declining pH values [19].

3.2. Redox potential

The oxide reduction potential of the soil depends on pH and determines the presence of heavy metals in forms susceptible to reduction and oxidation. Oxygen deficiency leads to the predominance of anaerobic organisms in the environment, for which oxidized forms of various elements can represent electron acceptors. Reduction processes determine the release of Mn^{2+} and Fe^{2+} ions from hydrated oxides into soil solution. These phenomena are conducive to an increase in mobility of metals due to their reduction, and, consequently, a change in value.

A change in aerobic conditions can have the opposite effect on the elements when sulphates are reduced. Chemisorption leads to formation of hardly soluble metal sulphides, such as CdS, ZnS, MnS, or CuS. With this pattern, manipulation of the redox potential parameter to obtain metal forms with specific mobility is difficult [20, 21].

3.3. Sorption capacity

Sorption capacity means the total amount of cations which can be bound in the soil environment. This parameter is connected to the content of mineral substance and humus and the presence of hydrated aluminium and iron oxides [20-22]. Soils with high sorption capacity and grain size are able to significantly reduce the migration of heavy metals. One of the methods to immobilize toxic ions is the addition of various types of sorbents (both mineral and organic) to the environment. Placek, Kacprzak and Napora demonstrated that the addition of even up to 1÷2% of organic sorbent prevents from infiltration of xenobiotics into the deeper levels of soil [17]. Sorption capacity of soil also determines the uptake of microelements that are needed by plants to grow. The potential of complexation of elements by soil material can be evaluated by various physical and chemical tests, e.g. determination of organic matter content by means of drying and weighing method or granulometric analysis. It should be remembered, however, that in the case of an exceptional ability to bind elements by the medium, a competitive phenomenon consisting in the displacement of one of the cations from the solution may be observed [3].

4. Speciation of heavy metals in sewage sludge

Chemical forms of metals present in sewage sludge can be identified by means of sequential extraction, i.e. speciation, based on fractionating of compounds. The application of this analytical procedure ensures separation of the tested material into fractions characterized by different mobility levels [23, 24].

Numerous varieties of sequential extraction have been used, but all of them are based on the repetitive process of leaching of different forms of metals using several reagents with increasing aggressiveness. The examined precipitate, after dissolving in the eluent used, is centrifuged in order to separate the solid phase of the sample from the solution with absorbed metals and then leached by the next extractor until all fractions have been determined. The liquid phase with individual forms of microelements is subjected to the subsequent quantitative analysis performed by means of e.g. atomic absorption spectrometry (AAS). The methods of sequential analysis differ essentially in the number of separated element fractions, the reagents used and time. The ability of individual eluents to leach metals depends on their form and reactivity of a given extraction solution [15, 25]. Table 2 shows the reactants which are most commonly used in the procedure.

Table 2. **Reagents used in sequential analysis [26, 27]**

Fraction	Reagents
Soluble in water	H_2O
Adsorbed interchangeably	Salts: Ca^{2+}, K^+ Connections (Ba,Pb,Sr) - Cl
Adsorbed specifically	CH_3COOH, KF
Bound with Mn oxides	Reducing compounds: NH_2OH, $NH_2OH*HCl$ $Na_2S_2O_4$ Strong oxidants: H_2O_2
Bound to crystalline oxides Fe	$(NH_4)_2C_2O_4$ + UV $(NH_4)_2C_2O_4$ + ascorbic acid $Na_2S_2O_4$
Bound to amorphous oxides Fe	$Na_2S_2O_4$, $NH_2OH*HCl$
Bound to organic substance	Dispersive compounds: $K_4P_2O_7$ Chelating compounds: EDTA, DTPA Strong oxidants: H_2O_2, NaOCl
Carbonate	HCl, $CH_3COONa + CH_3COOH$
Sulphide	HNO_3, KCN
Residual	HF, aqua regia, HNO_3, $HClO_4$

Sequential analysis has been in common use, but due to the lack of uniform testing conditions, the problems with comparison of the results obtained by different research teams have been often addressed. One of the most recognized and often used methods of speciation is a technique developed by Tessier et al. [26] used to identify five fractions of trace elements. Initially, the method was used to determine heavy metals in river sediments, but over time, this extraction proved to be effective in the analysis of samples of sewage sludge or those coming from other elements of the environment, such as soil [26].

Despite its popularity and advantages, the analysis proposed by Tessier has not been fully accepted as a universal methodology. The procedure has been modified many times, mainly due to the disproportion of certain fractions compared to other studies, and searching for more efficient solutions.

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4.1. BCR sequential analysis

There has been a tendency for simplifying analyses observed in recent years. Consequently, the four-step BCR procedure has currently become the most popular method. This method was developed as a result of the activities of the Measurements and Testing Programme (formerly the Community Bureau of Reference) responsible for harmonisation of extraction-based studies.

Unlike the Tessier procedure, two fractions were combined in the BCR method: exchangeable and bound to carbonates, obtained by extraction of 0.11 M CH_3COOH and shaking for 12 hours at 20°C. Eventually, four heavy metal fractions can be isolated during the analysis: ion-exchange and carbonate, bound to iron and manganese oxides (reducible), related to organic matter (oxidisable), and residual matter [23, 26, 28]. The diagram of the BCR analysis is shown in Figure 2.

The BCR method has been used in many studies on the quality of sewage sludge and areas contaminated with heavy metals. This technique allows for evaluation of the mobility of elements present in the examined medium and estimation of the risk related to the spread of pollution to the environment [29].

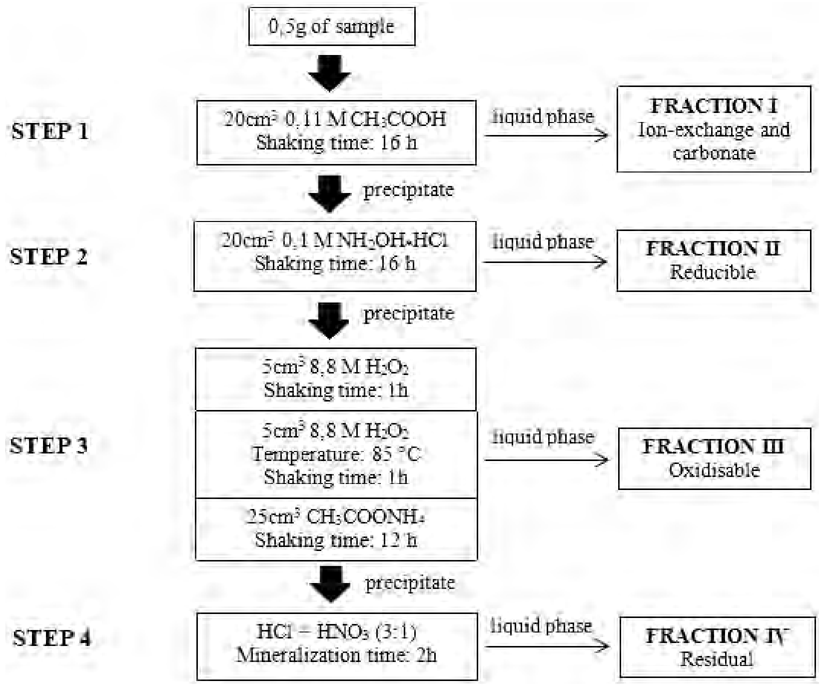

Fig. 2. Diagram of BCR sequential extraction procedure [10, 26]

Despite the many advantages of the BCR method, its disadvantages have also been emphasized, e.g. the lack of selectivity of certain reagents (especially the third fraction), and difficulties related to the appropriate pre-treatment of the sample to be analysed. Therefore, modifications of various stages of this method have been observed in some studies to improve the reliability of the results and their reproducibility [30-33].

Conclusions

With the development of modern technology and the infrastructure in the field of sewage treatment and disposal, the amount of sewage sludge generated is gradually increasing. This unfavourable effect of technological advances has led to an intensive search for the best solution for waste management. One of the preferred methods of sewage sludge management is to use it in nature, consisting mainly in reclamation of degraded soil and soilless areas and fertilisation of soil intended for agricultural use.

Sludge management methods depend strictly on sludge composition. In the case of the use in nature, the requirements to be met by sewage sludge are more stringent compared to other methods of disposal or use of this waste. One of the basic criteria determining the possibility of using sludge for reclamation or fertilization purposes is chemical composition, mainly concerning the heavy metal concentrations in the material tested. The permissible concentrations of these compounds in sewage sludge intended for the use in nature were specified in the Regulation of the Minister of the Environment of 6 February 2015 on municipal sewage sludge.

However, the analysis of heavy metal concentrations in the sewage sludge does not provide comprehensive information about the potential threats to the environment in which the sludge is to be discharged. This is because microelements show migration capabilities depending on the chemical form in which they occur. Metals that are highly mobile can move to different areas of the biosphere and accumulate in living organisms.

Extraction methods are among most popular methods used to determine mobile elements. Sequential extraction is a method that allows for isolation of individual fractions of metals and determination of their migration capacity. Nowadays, the most popular technique of speciation of heavy metals is the four-step BCR procedure, based on chemical leaching of the sample by means of reagents with gradually increasing aggressiveness. The advantages of this method include accuracy, possibility of separation of specific fractions and their quantitative analysis. The disadvantages are time-consumption and lack of selectivity of certain reagents.

Toxic properties of heavy metals are obvious, but neglecting such an important aspect as the ability of microelements to migrate may lead to serious hazards to the environment. The effects of hasty decisions to use sewage sludge in nature without prior analysis of the concentrations of heavy metal forms present in the sludge may therefore be counterproductive.

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Streszczenie

Na skutek rozwoju wysokoefektywnych metod oczyszczania ścieków oraz powstawania nowych oczyszczalni obserwuje się systematyczny wzrost ilości generowanych osadów ściekowych w Polsce. Dotychczas nie opracowano jednak technologii bezosadowych ani skutecznego rozwiązania umożliwiającego całkowitą eliminację produktu oczyszczania ze środowiska, co stanowi problem związany z racjonalną gospodarką tego rodzaju odpadami. Jedną ze strategii proponowanych w tym zakresie jest przyrodnicze wykorzystanie osadów ściekowych, które opiera się głównie na nawożeniu gleb i roślin oraz rekultywacji gleb zdegradowanych i obszarów bezglebowych. Warunkiem takiego rozwiązania jest odpowiednia jakość osadów mających stanowić potencjalny nawóz, a mianowicie powinny one spełniać określone wymagania higieniczno-sanitarne oraz chemiczne. W przypadku kryterium, jakim jest skład chemiczny, obowiązują normy dotyczące przede wszystkim całkowitej zawartości metali ciężkich w osadzie. W ostatnich latach coraz większą uwagę zwraca się jednak na zdolność mikroelementów do przemieszczania się w środowisku, co umożliwia lepszą ocenę jakości elementów biosfery i przewidywanie potencjalnego ryzyka rozprzestrzeniania się zanieczyszczenia. Jedną z metod określania form pierwiastków w badanym medium jest analiza specjacyjna, oparta na ekstrakcji sekwencyjnej. Celem niniejszej pracy było wskazanie znaczenia specjacji w ocenie jakości osadów ściekowych i możliwości ich przyrodniczego wykorzystania. Ponadto, omówiono jedną z najpopularniejszych obecnie metod oznaczania mobilności metali ciężkich. Dysponowanie wiedzą na temat form metali ciężkich występujących w badanych osadach pozwala na precyzyjne ukierunkowanie gospodarki tym odpadem.

Słowa kluczowe: gospodarka odpadami, osady ściekowe, specjacja, metale ciężkie