

# Wastewater sewage sludge leaching and alkaline stabilization

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The constantly growing amount of unutilised wastewater sewage sludge (SS) remains an unsolved environmental problem in Lithuania. SS can serve efficiently as a renewable feedstock in agriculture for the production of composts, for recultivation of exhausted lands and for woodland soil improvement in forestry. Unfortunately, SS application in agriculture is restricted by its contamination with pathogenic microorganisms, organic micro-pollutants and heavy metals (HM). Therefore, investigation of SS leaching behaviour and stabilization processes is of great importance. The aims of the present study were to investigate SS leaching in the environmental conditions and its alkaline stabilization by using an inorganic waste material – cement kiln dust.

SS samples collected in Kaunas (industrial city with a population nearly half a million) and in Raseiniai (small residential town with approximately 12500 inhabitants) wastewater treatment plants (WTP) were investigated. Samples were taken weekly for the period of 7 months from both WTPs. Samples were also collected at the special Ežerėlis sludge landfill in order to evaluate the natural SS leaching behaviour. Samples were taken from sludge piles of an average age of 16 and 40 weeks. Control samples were also taken from recently piled SS (average age 2 weeks) The sampling depths were 0–10 cm, 10–20 cm and 40–50 cm. All samples were air-dried, homogenized and sieved through a 2 mm sieve. SS stabilization was performed using cement kiln dust (CD), which is a waste material of cement industries (JSC “Akmenės cementas”). Parallel experiments with lime and quicklime were carried out for a comparison with CD results. The total HM amounts were determined by flame or electrothermal atomic absorption spectrometry in *aqua regia* digestion solutions. Mobile HM amounts were determined in 0.05M (NH<sub>4</sub>)<sub>2</sub>EDTA (pH 7) extract solutions. The results of the experiment were evaluated by statistical methods.

The data of chemical analysis showed that the total HM amounts in SS varied depending on its origin. HM content in the sludge of Kaunas was 2–3 times greater than in the SS of Raseiniai. The greater standard deviations of HM amounts in the Kaunas SS have shown that HM concentration in the SS of an industrial city is less predictable as it presumably depends on the intensity of industry. Investigation results on SS leaching showed that the storage of sludge in special sites had a positive effect not only on the destruction of pathogens and micro-organic contaminants, but also on the reduction of heavy metal contamination; however, a safe utilization of sludge landfill leachate should be foreseen. Furthermore, such alkaline waste material as cement kiln dust can be successfully applied for SS stabilization, as the lower moisture content after mixing, increase of pH and temperature would enhance microbiological pasteurisation, whereas mixing results in a considerable HM immobilization.

**Key words:** organic wastes, sewage sludge, heavy metals, cement kiln dust, leaching, stabilization, immobilization

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## INTRODUCTION

Processing and reuse of biodegradable organic waste (OW) is the main direction of environmental protection policy in Lithuania (the Law on Waste Managements of the RL, 1998). The importance of OW utilisation is also highlighted in EU directives (Directive 98/31E/EC, Directive 86/278/EEC). This approach to material and energy recycling allows not only saving natural global resources, but also avoiding the negative environmental impact of waste incineration and landfilling. About 6 mln. tons of innocuous wastes accumulate in Lithuania every year, including 2 mln. t (~35 %) of wastes of organic origin. About 3 mln. t are discharged in landfills, though half of this quantity could be utilized. The permanently growing amount of unutilised wastewater sewage sludge (SS), which increases at a rate of more than 500 000 t per year, remains an unsolved environmental problem in Lithuania (Aplinka'99, 2000). Utilisation of other kinds of unstable biodegradable OW of animal and vegetable origin, such as manure, slaughter waste, wastes of food processing industry, forestry, etc., is also a problem of great importance.

OW can serve efficiently as a renewable resource in agriculture for the production of composts and biofertilizers, as soil improvers and growing media, and also for recultivation of exhausted lands such as mining sites or urban brownfields, re-capping of landfills, and for woodland soil improvement in forestry. Unfortunately, such OW application in agriculture, especially SS usage for land reclamation purposes, is restricted by contamination with pathogenic microorganisms, organic micropollutants and heavy metals (HM) (Directive 86/278/EEC). Active and microbiologically contaminated OW (including SS) could be stabilized by long-term (1–3 years) storage at special sites. Alternatively, anaerobic or aerobic OW-treatment technologies include organics, for example, as waste processing in a biogas reactor or conventional composting with peat, dry litter, straw, sawdust, and also alkaline inorganic materials, such as lime, quicklime and waste cement kiln dust (Little et al., 1991; Paulauskas et al., 2004). Most organic micropollutants are unstable and biodegrade during OW storage and processing, whereas HM and their compounds are especially stable, biologically non-degradable, long remaining in OW as well as in the natural environment (Chang et al., 1984; Parveen et al., 1994; Martin et al., 1997). HM have a negative influence on plants, animals and humans, as they tend to accumulate in living organisms disturbing their functioning, so it is important to gain knowledge about HM mobility and behaviour in waste materials in order to understand the mechanisms of leaching processes as well as immobilization effects of the stabilizing agents.

In recent years, discussion of test methods to assess HM mobility and the leaching behaviour of waste materials has entered a new phase, because the urge to recycle and reuse waste materials increases. This policy requires better control over the undesirable release of

contaminants into the environment, which is influenced by a large number of physical (e.g., pH, redox, sorption properties, complexing agents, reaction kinetics) variables (Fytianos, Charantoni, 1998). With large quantities of SS deposited for long periods, hazardous substances may be released in a landfill, percolate through the soil layers and reach groundwater. In order to minimize health risks, it is thus important to know both the total content of hazardous substances and the chemical forms in which they appear in the sludge (Rudd et al., 1988). The distribution ratio of the total HM content between the SS and the water phase depends upon the chemical properties of a metal and the physicochemical properties of sludge. The latter, in turn, depends on the conditions employed in the SS treatment process, such as pH, temperature, redox potential, the presence and concentration of complexing and precipitating agents (Fytianos, Charantoni, 1998).

HM can be leached from OW before utilization in soil or alternatively they can be immobilized by various agents. To reduce the leachability of HM in waste materials, chemical stabilization can be effective. The main aim of stabilization is to form new, less soluble mineral phases more geochemically stable in leaching environments. Also, immobilization is seen as a promising technology for soil HM remediation. In a laboratory test, when the soil was stabilized with reagent grade stabilizers ( $\text{CaHPO}_4$  and  $\text{CaCO}_3$ ), the toxicity of extractable concentrations of Cd, Cu, Pb and Zn was reduced by more than 87%, while under field conditions stabilization with  $\text{Ca}(\text{H}_2\text{PO}_4)_2$  for 30 days gave the ratios of 98% for Cd, 97% Cu, 99% Pb, 96% Zn, and 65% Ni (Wang et al., 2001). Among other agents, general stabilizers such as cement or bentonite can be used (Methods..., 1998). Chemical stabilization of SS with alkaline waste materials such as cement kiln dust (CD) can diminish HM mobility in large amounts of such kind of wastes. However, CD itself may contain high concentrations of HM, and their behaviour in a mixture with SS has not yet been investigated.

In view of recent knowledge of how metals are transported in and bind to the soil, there is no longer a belief that any one single method could successfully remove or stabilise all the different metals in all the different types of soil as well as in SS of different origin (Methods..., 1998). Knowledge of HM migration processes in OW and in the soil allows management or modification of HM by various methods in order to diminish the toxic effects of HM on the environment.

The aims of the present research were to investigate SS leaching in the environmental conditions and its alkaline stabilization by using a waste material – cement kiln dust.

## METHODOLOGY

### Object

Sewage sludge samples were taken after centrifugation from methane tanks at the Kaunas wastewater treatment

plant (WTP) during the period of 7 months. At the Kaunas WTP, all effluents of the Kaunas city and its surrounding areas are being treated. Kaunas is a rather big industrial city with a population reaching nearly half a million inhabitants. Also, SS samples at the Raseiniai WTP were taken after the dewatering with a belt-press and from the SS storing site during the period of 7 months. The main part of the effluents of the Raseiniai WTP comprises household wastewater and drainage water.

Seeking to evaluate leaching processes taking part during SS storage, samples were also taken from a special SS landfill at Ežerėlis. Samples were taken from SS piles of an average age of 16 and 40 weeks. Control samples were also taken from recently piled SS (average age 2 weeks) from different depths: 0–10 cm, 10–20 cm and 40–50 cm. Joint SS samples (weighing about 1–2 kg) were composed from 5 separate subsamples taken randomly from an area of 1 m<sup>2</sup>. All samples were air-dried, homogenized and sieved through a 2 mm sieve.

### Methods

Sewage sludge stabilization was performed using cement kiln dust (CD) which is an inorganic waste material of cement industries (JSC “Akmenes cementas”). It consists of fine mineral particles captured by air cleaning installations (electrostatic filters, cyclones, etc.) during dehydration of cement ingredients in a rotary kiln at a high temperature (above 1400–1500 °C). For the Kaunas SS treatment, more alkaline (pH 13.6) cement kiln dust CD-1 (sampled from the second field of electrostatic filters) was chosen, while the Raseiniai SS stabilization was carried out with less alkaline (pH 12.7) cement kiln dust CD-2 (from the fourth field of electrostatic filters). Parallel experiments with lime (L) and quicklime (QL) were carried out for a comparison with the CD results. The indexes of some characteristics of the alkaline materials are presented in Table 1.

The percentage of air-dried alkaline materials used in the mixtures with dewatered SS was as follows: 10%, 20%, 30%, 40% and 50%. In the course of the stabilization experiment, changes of temperature, moisture and pH (after 2 hours) were recorded, and the loss on ignition was determined. Finally, the amounts of the total

and mobile HM species in the sludge stabilized with CD-1 (volume percentage in the mixture 30%) were determined by atomic absorption spectrometry (AAS).

### Analytical procedures

For HM determination, analytical samples (1–10 g) were taken from homogenized laboratory samples (200–300g) air-dried and sieved with a 2 mm sieve following the standard sampling procedure. Total HM amounts were determined by the flame or electrothermal AAS method with an AAnalyst-100 spectrometer (Perkin–Elmer) in *aqua regia* digestion solutions (ISO 11466:1995, ISO 11047:1998 E). The amounts of mobile HM were determined in 0.05M (NH<sub>4</sub>)<sub>2</sub>EDTA (pH 7) extract solutions (Methods of soil analysis, 1996). Samples with the extractant were agitated for 1 hour at room temperature (sludge mass to extractant volume ratio 10:50), centrifuged and filtered. All the extractions were carried out in triplicates and all the data were corrected to the oven-dry (105 °C) SS moisture content.

### Statistical calculations

Results of the experiments were evaluated by the statistical methods for calculating means, standard deviations and correlations using MS' EXCEL program.

## RESULTS AND DISCUSSION

### Total HM amounts in sewage sludge of different origin

Results of chemical analysis showed, that total HM amounts in SS varied and depended on its origin. HM amounts in the SS of the industrial city (Kaunas) were 2–3 times greater than in SS of the small town Raseiniai. A notably high contamination level by Cd was found in the Kaunas SS. Quite high levels of Zn were determined in the SS of both Kaunas and Raseiniai. Furthermore, the greater standard deviations of HM amounts in the Kaunas SS showed that HM content in the sludge of an industrial city are less predictable as presumably they depend on the intensity of industry. An especially great deviation was observed in the case of Cd (21 ± 25.89 mg/kg d.m.).

Analysing the relation between different HM changes in the Kaunas SS during the period of 7 months,

Table 1. Characteristics of cement kiln dust, lime and quicklime

Indexes	CD-1	CD-2	L	QL
Na <sub>2</sub> O (%)	0.21	0.40	n	n
K <sub>2</sub> O (%)	3.50	10.09	n	n
CaO (%)	44.63	38.81	n	n
pH <sub>KCl</sub>	13.6	10.6	12.5	12.5
Cd (mg/kg d.m.)	10.6	8.4	n	n
Pb (mg/kg d.m.)	154.0	214.2	n	n
Ni (mg/kg d.m.)	27.6	58.3	n	n
Cu (mg/kg d.m.)	20.0	28.6	n	n
Zn (mg/kg d.m.)	61.0	48.7	n	n

\* n – no results, d.m. – dry matter.

a high correlation was determined between Cd–Pb and Cd–Cu ( $r = 0.7$  and  $0.95$ , respectively). The variation of Zn concentration correlated well with changes of Ni content in SS ( $r = 0.86$ ). This is presumably related with the same original source of these anthropogenic contaminants. Meanwhile, alteration margins of HM amounts in the Raseiniai SS were much narrower than in the case of Kaunas SS, and no correlation among HM was observed. Sewage sludge of a small town, such as Raseiniai, with a rather low level of HM and comparably stable composition, can be successfully used for the production of biofertilizers, composts and soil improvers or, alternatively, applied on-land directly (Table 2).

Application of the sludge of an industrial city like Kaunas on agricultural land is more complicated and limited by the EU and Lithuanian legislative documents regarding HM content, especially Cd (Directive 86/278/EEC; LAND 20-2005). According to Cd content, Kaunas SS should be attributed to the lowest sludge quality category 3, on-land application of which is forbidden (Table 2). Moreover, as the concentration of HM in SS collected at different time varies greatly, and individual analysis of chemical composition before its on-land application is necessary.

#### Leaching processes during sewage sludge storage

A possibility of HM leaching from sewage sludge was investigated under natural conditions in sludge piles at the Ežerėlis SS landfill after 16 and 40 weeks of storage. HM migration down the profile of the piles was observed in most of the cases (Fig. 1).

The main causes of these changes are supposed to be rainfall exudation (percolation), pH changes due to ion

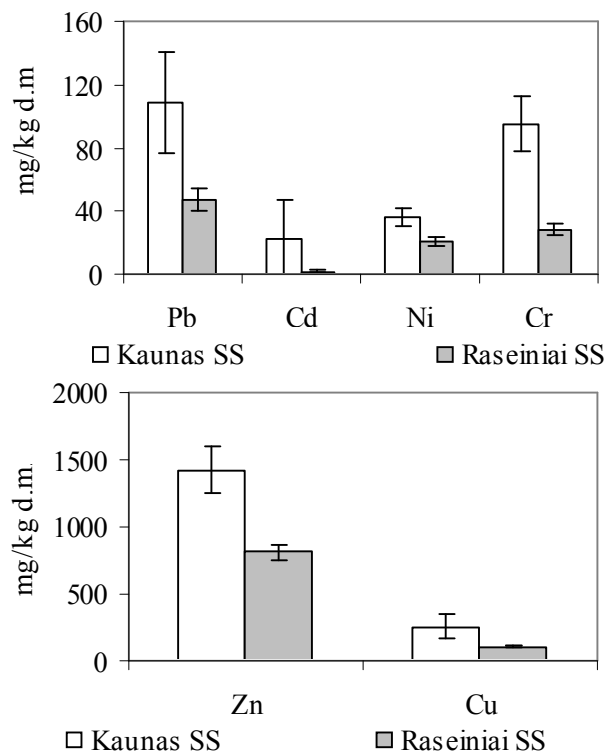


Fig. 1. Total HM amounts (mean values and standard deviations) in the sewage sludge of Kaunas and Raseiniai WTPs

leaching as well as mineralization and evaporation from the surface layers (Table 3).

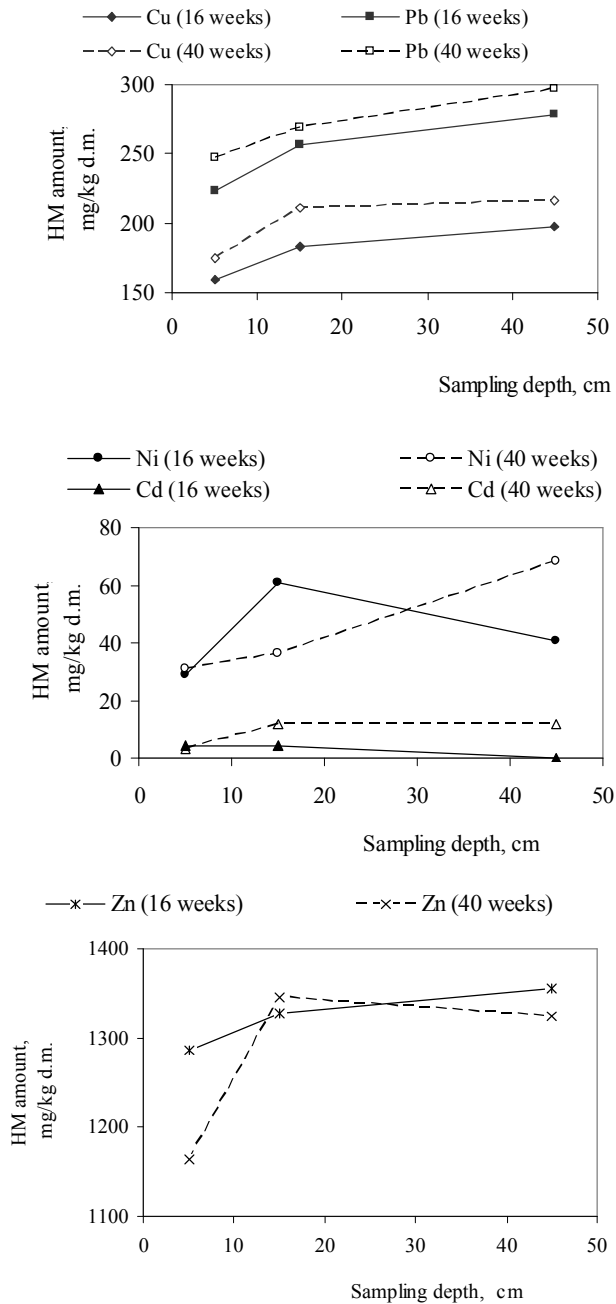
The most intensive downward migration was observed in the case of Cu and Pb, while Cd and Zn accumulated mainly in the upper layers (20 cm) of the SS piles. Leaching of Ni to the lower layers of sludge was slower than that of Cu or Pb (Fig. 2).

Table 2. Categories of sewage sludge depending on heavy metal concentrations (LAND 20-2005)

Category of sewage sludge	Concentration of heavy metals, mg/kg						
	Pb	Cd	Cr	Cu	Ni	Zn	Hg
I	<140	<1.5	<140	<75	<50	<300	<1.0
II	140–750	1.5–20	140–400	75–1000	50–300	300–2500	1.0–8.0
III	>750	>20	>400	>1000	>300	>2500	>8.0

Table 3. Variation of SS mineralization degree (calculated as C/N ratio), moisture content and pH in the SS pile profile during SS storage

No.	Period of piling (average age)	Depth of sampling	Moisture content, % d. m.	Total N, % d. m.	pH	C/N ratio
1	40 weeks	0–10	62.15	2.47	6.96	12.5
2		10–20	71.50	2.52	7.42	12.7
3		40–50	73.42	2.73	7.87	11.0
4	16 weeks	0–10	61.50	2.75	7.25	11.3
5		10–20	69.79	2.73	7.46	11.1
6		40–50	73.02	2.90	7.72	10.7
7	2 weeks	0–10	75.52	3.5	7.92	9.1

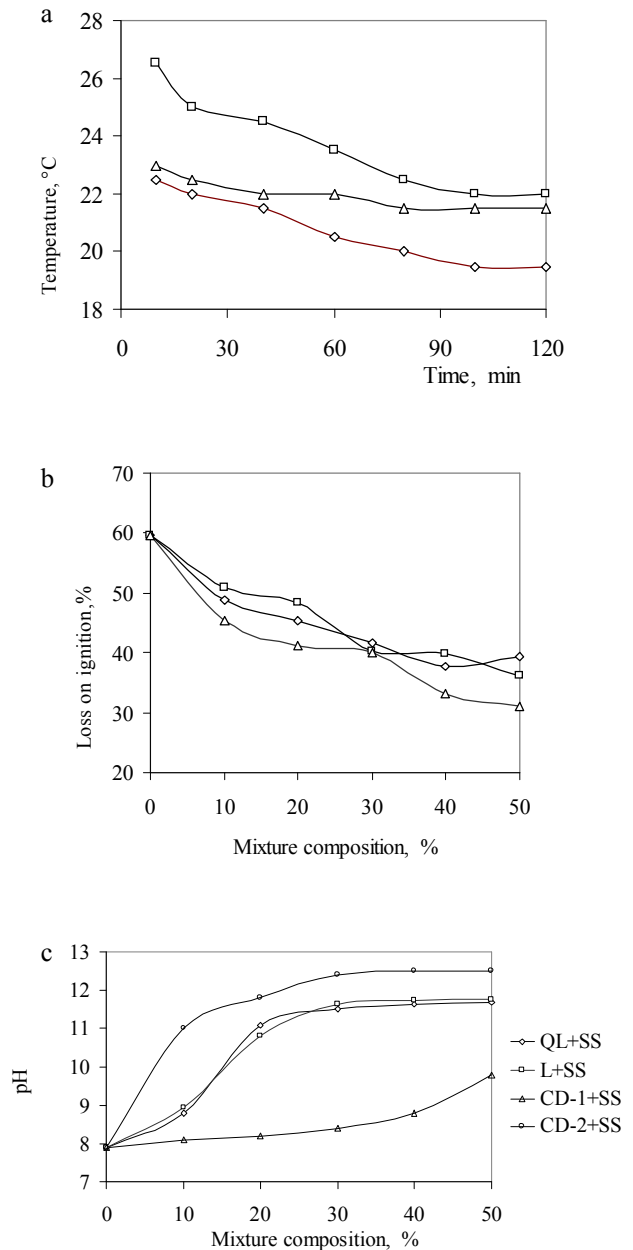


**Fig. 2.** HM amounts at different depths of the SS pile profile after 16 and 40 weeks of storage

### Sewage sludge alkaline stabilization

Results of SS stabilization using different alkaline materials are presented in Fig. 3.

The initial increase of temperature right after SS mixing with alkaline materials reached 4–9 °C above room temperature (18 °C), but afterwards the temperature decreased (Fig. 3, a). During the period of 2 hours, the rise of temperature was most stable only in the case of CD+SS mixture. Nevertheless, temperature uprise was not as high as reported in the literature (up to 40–60 °C), presumably due to the small volume of the mixture components (Little et al., 1991; Logan, Harrison, 1995), whereas the value of loss on ignition is



**Fig. 3.** Changes of temperature, loss on ignition and pH in SS mixtures with alkaline materials (QL – quicklime, L – lime, CD – cement kiln dust, SS – sewage sludge)

important when organic wastes are recycled as soil conditioners. The obtained results showed that addition of CD reduced the content of organic matter in the mixtures significantly, but even in 30–50% of mixtures it remained still high (Fig. 3, b). pH alteration of the CD+SS mixture depended greatly on the initial pH of cement kiln dust. The CD-1 influence on the mixture pH increase was less than that of CD-2 (Fig. 3, c), due to the lower amount of free alkaline materials in this waste material (Table 1). However, CD-2 addition raised the mixture pH even more effectively than did L or QL (up to 11–12.5 times) depending on the SS/CD ratio in the mixture. Therefore, such kind of mixtures can be successfully applied for soil liming.

### Heavy metal immobilization

In order to investigate the influence of cement kiln dust on HM mobility, the amounts of total and mobile HM were determined in 30% CD+SS mixtures. The data presented in Fig. 4 show, that the investigated alkaline waste dust changed the total HM concentration in the mixtures depending on the initial HM amounts in the mixture components. The total Cd amount in the CD mixture with the Raseiniai SS increased from 2.9 up to 5.2 mg/kg. The total Pb amount increased almost twice (from 68.4 to 123.3 mg/kg), while Ni from 15.4 to 39.1 mg/kg. But the concentrations of the total Cu and Zn in the mixtures decreased markedly from 115.8 to 62.5 mg/kg and from 1158.0 to 547.2 mg/kg, respectively.

Consequently, SS stabilization with CD could be a good measure to diminish the total Cu and Zn amounts when recycling SS in agriculture, but it should be taken into account that the total amounts of all the other HM should not exceed the permissible limits (Table 2, LAND 20-2005).

Results on HM mobility in a CD-SS mixture and in the initial mixture components using EDTA extraction are presented in Fig. 4. It is obvious that alkaline dust immobilized most of the HM due to pH increase and formation of less soluble metal compounds in the mixture. The EDTA extractable Cd amount in the SS+CD mixture decreased from 41 to 12%, Pb from 10 to 4%, Ni from 63 to 17%, and Zn from 17 to 7%, while only Cu amount slightly increased (from 42 to 53%).

Another cause of HM immobilization in the investigated mixtures is the mineral origin of alkaline waste predetermining the initial low HM mobility. The obtained results show that CD as inorganic waste material

could be successfully used for both SS stabilization and HM immobilization, but investigations of the long-term behaviour in acid soils of such kind of mixtures should be carried out, considering a possible HM release to the natural environment.

### CONCLUSIONS

It could be maintained that storage of the sewage sludge in special sites has a positive impact not only on the destruction of pathogens and micro-organic contaminants, but also on the reduction of heavy metal contamination; however, safe utilization of sludge landfilling leachate should be foreseen. Furthermore, such alkaline waste material as cement kiln dust can be successfully applied for wastewater sewage sludge stabilization, because the lower moisture content after mixing, increased pH and temperature would enhance microbiological pasteurisation, and the mixing facilitates a considerable immobilization of heavy metals.

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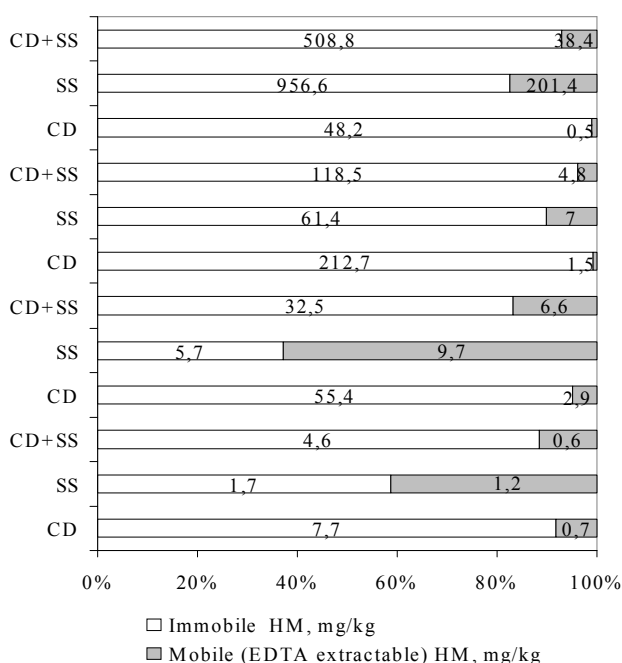


Fig. 4. Ratio of immobile and mobile HM species in cement kiln dust (CD), sewage sludge (SS) and 30% CD+SS mixture

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#### **NUOTEKŲ DUMBLO IŠPLOVIMAS IR ŠARMINIS STABILIZAVIMAS**

##### **S a n t r a u k a**

Nuolat didėjantis nepanaudoto nuotekų dumblo kiekis yra aktuali aplinkos apsaugos problema Lietuvoje. Nuotekų dumblas yra pedirbimui tinkama medžiaga. Jis gali būti efektyviai nau-

dojamas žemės ūkyje kompostų ir biotrašų gamybai, pramonėje karjerų rekultivavimui, miškininkystėje miško dirvožemių praturtinimui. Deja, nuotekų dumblo panaudojimą žemės ūkyje riboja jo užterštumas patogeniniais mikroorganizmais, organiniais mikroteršalais, sunkiaisiais metalais. Dėl to nuotekų dumblo išsiplovimo bei stabilizavimo procesų tyrimai yra labai svarbūs. Šio tyrimo tikslas buvo iširti nuotekų dumblo išsiplovimo procesą natūraliomis aplinkos sąlygomis bei šarminį stabilizavimą su cemento gamybos atliekomis – cemento dulkėmis.

Nuotekų dumblo ėminiai buvo imami iš Kauno (pramoninio miesto su 0,5 mln. gyventojų) ir Raseinių (mažo nepramoninio miestelio su 12 500 gyventojų) nuotekų valymo įrenginių kas savaitę 7 mėnesius. Taip pat nuotekų dumblo ėminiai buvo imami iš Ežerėlio nuotekų dumblo saugyklos plovimo procesams natūraliomis aplinkos sąlygomis įvertinti. Ėminiai buvo imami iš 0–10 cm, 10–20 cm ir 40–50 cm gylio po 16 ir 40 savaičių laikymo. Be to, buvo paimti atitinkami kontroliniai naujai supulto dumblo (laikyto iki 2 savaičių) ėminiai. Nuotekų dumblo mėginiai buvo išdžiovinti, homogenizuoti ir persijoti per 2 mm sietą. Nuotekų dumblo stabilizavimas atliktas panaudojant AB „Akmenės cementas“ gamybos atliekas – cemento dulkes, taip pat, palyginimui, gesintas bei negesintas kalkes. Sunkiųjų metalų bendrosios koncentracijos nustatytos liepsnos arba elektroterminiu atominės absorbcinės spektrometrijos metodu *aqua regia* mineralizavimo tirpale, o judriųjų jonų koncentracijos – 0,05 M (NH<sub>4</sub>)<sub>2</sub>EDTA (pH 7) tirpalo eksakte. Rezultatai įvertinti statistiniais metodais.

Cheminės analizės rezultatai parodė, kad bendrosios sunkiųjų metalų koncentracijos nuotekų dumble priklauso nuo jo prigimties. Pramoninio miesto Kauno nuotekų dumble sunkiųjų metalų koncentracijos buvo 2–3 kartus didesnės nei Raseinių nuotekų dumble. Be to, didesni sunkiųjų metalų koncentracijų standartiniai nuokrypiai Kauno nuotekų dumble rodo, kad jos labiau priklauso nuo pramonės intensyvumo ir jas sunkiau numatyti. Nuotekų dumblo išsiplovimo tyrimų rezultatai parodė, kad nuotekų dumblo laikymas specialiose atvirose saugyklose turi teigiamą įtaką ne tik patogeninių mikroorganizmų bei organinių mikroteršalų irimui, bet ir užteršimo sunkiaisiais metalais sumažinimui. Tačiau yra svarbu numatyti saugius filtrato, į kurį išsiplauna pavojingos medžiagos, utilizavimo būdus. Stabilizavimo cemento dulkėmis eksperimento rezultatai parodė, kad jis gali būti naudojamas nuotekų dumblui stabilizuoti, nes efektyviai sumažina mišinio drėgmės kiekį, padidina pH ir temperatūrą taip sustiprindamas pasterizavimo procesus. Be to, cemento dulkių priedas nuotekų dumble sumažino sunkiųjų metalų judriųjų jonų masės dalis.

**Raktažodžiai:** organinės atliekos, nuotekų dumblas, sunkieji metalai, cemento dulkės, išplovimas, stabilizavimas, imobilizavimas