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CHALLENGES OF THE REPUBLIC OF SERBIA IN THE TREATMENT OF WASTE SLUDGE CONTAMINATED WITH HEAVY METALS

The interactions of heavy metals and cement phases in the solidification/stabilization (S/S) process have been examined. The S/S method was applied to the waste sludge contaminated with heavy metals to determine the conditions under which the treated materials can be safely disposed of at landfills for inert, non-hazardous, or hazardous waste. Sludge samples were mixed with soil and sand to simulate improper sludge disposal directly into the soil. S/S technology was selected using different cement fractions to treat these contaminated samples. The use of Portland cement or mixtures of different types of cement for the treatment of sludge containing heavy metals is widespread. Samples of leached content were analyzed for the presence of the following metals: arsenic (As), cadmium (Cd), chromium (Cr), copper (Cu), nickel (Ni), and zinc (Zn). The study results lead to the conclusion that the sludge contaminated with heavy metals after the application of S/S with the addition of different cement fractions is transformed into non-hazardous, monolithic material.

1. INTRODUCTION

The term sludge refers to mixtures of fine particles, which differ in their origin, composition, content, the concentration of certain substances, dry matter content, different properties, processing and disposal methods, and the final purpose [1]. Other

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types of waste sludge, especially those containing heavy metals, arise in metal processing, galvanic protection, oil refining, and municipal and industrial wastewater production and treatment.

Different categories of waste sludge are defined in the *Republic of Serbia Waste Catalog*, which is harmonized with the *European Waste Catalog* [2]. Thus, for example, the sludge from the municipal wastewater treatment plant is classified into the waste group 19 08 05. In Serbia, the sludge created after the wastewater treatment is deposited in landfills. Currently, it is about 4000 tons/year. The mentioned quantity will be significantly increased by constructing a plant for municipal wastewater treatment, as at present, only 5.3% is adequately treated in the Republic of Serbia. Treatment of wastewater, that is, the construction of a wastewater plant is one of the biggest challenges for the Republic of Serbia when it comes to meeting the requirements of the EU. The membership in the European Union represents the national interest and strategic commitment of the Republic of Serbia. In this regard, it is necessary to integrate European values and policies in the territory of the Republic of Serbia. The biggest challenges regarding EU membership are related to environmental protection and climate change, as defined in the negotiating Chapter 27: *Environment and climate change*. According to estimates, the most significant financial investments in the coming period will be needed for infrastructure projects in waste management and wastewater treatment. According to the European Commission's Report on the Republic of Serbia's progress towards European Integration from 2021 [3], a moderate level of harmonization with the acquis communautaire in water quality has been set. Nevertheless, untreated sewage sludge and wastewater remain the primary source of water pollution. Therefore, improving local governance, especially concerning the operation and maintenance of water and wastewater facilities, remains a priority.

Waste sludge requires adequate treatment before use or final disposal. To achieve the standards of Chapter 27, the prohibition of waste disposal without prior treatment is necessary, particularly waste of hazardous characteristics. In Serbia, despite the need for a previous treatment, waste sludge is, in most cases, deposited on land near the plants from which they originate or in landfills of municipal waste. They often contain heavy metals in high concentrations and contaminate soil and groundwater.

The main goal of the research is to examine the conditions under which the waste sludge contaminated with heavy metals treated with the stabilization/solidification method can be safely disposed of in landfills for inert, non-hazardous or hazardous waste. Conditions for sludge disposal (sanitary landfill) are regulated by the Technical Directives in each country [4]. For example, in Serbia, the disposal of waste in landfills is regulated by the *Regulation on Waste Disposal* [5], but unfortunately, its applicability is unsatisfactory. La Grega et al. [6] suggest that today S/S technology is widely used as a method of remediation to treat contaminated soil. It is also successfully used to treat hazardous waste, waste streams before their disposal in landfills, and industrial waste. The mentioned technology includes mixing cement or other binder materials with contaminated media and immobilizing harmful constituents, preventing their penetration into the environment. According to Malone and Lundquist [7], stabilization involves chemical reactions between stabilizing agents and pollutants to reduce their mobility. In contrast to stabilization, solidification is the physical immobilization of pollutants into a stabilized mass of material. Generally speaking, stabilization is a process in which additives and binders are mixed with waste materials or contaminated soil to reduce the toxicity of the treated material. Solidification is a process that uses binders and additives to change its physical nature during the process. Stabilization and solidification processes often take place in parallel.

Marjanović et al. [8] state that S/S is a remediation technology based on the reaction between binder and contaminated soil to stop, prevent or reduce the mobility of pollutants. The difference between the stabilization and solidification process is shown as follows:

- stabilization involves the addition of reagents to contaminated material (soil or sludge) to produce chemically stable constituents,
- solidification includes the addition of reagents to contaminated material to obtain physical/dimensional stability of contaminants in the form of a solid product, resistant to external factors (such as rain and landslides).

Conventional S/S is an established method of remediation for contaminated soils and treatment of waste sludge in many countries worldwide. However, the understanding of technology is relatively modest, and there are numerous barriers to its practical application. One of the most significant limitations is the lack of accurate data on the duration and level of pollutants released from the treated material.

2. EXPERIMENTAL

To determine the content of the selected heavy metal group in different types of waste sludge, samples of waste sludge were taken from the following sites:

sample 1 – wastewater treatment plant Vodokanal Bečej,
sample 2 – water filter factory of Vodovod i Kanalizacija Subotica,
sample 3 – the process of enameling Metalac Gornji Milanovac,
sample 4 – the process of tefloning Metalac Gornji Milanovac.

The samples were representative samples for different sludge categories most commonly generated in Serbia and require treatment before final disposal.

Analysis of heavy metal content in sludge samples. Samples of waste sludge were subjected to laboratory analysis on the content of the following heavy metals: As, Cd, Cr, Cu, Ni, and Zn. Also, the contents of dry matter and pH were determined in the samples. Standard analytical methods were used for the analysis of the collected sludge samples. The content of heavy metals was determined by the EPA 6010c method *Inductively coupled plasma-atomic emission spectrometry* on the ICP-OES system Thermo iCAP 6500 Duo (Thermo Fisher Scientific, Waltham, MA, USA) The dry matter content in the samples was determined by the EPA 3051 method *Microwave-assisted acid digestion of sediments, sludges, soils, and oils.* Further, the pH was determined by the EPA 9045D method *Solid and waste pH*. All values are expressed on a dry matter basis.

Application of S/S to contaminated waste sludge and soil samples. Initially, the contaminated sludge samples were mixed with the soil and sand in a particular proportion. Then, the mixture was made to simulate improper disposal of sludge directly into the soil and its consequences in the environment in terms of heavy metals content. The soil used to prepare S/S samples was taken from two sites. The first site was the perimeter of the non-sanitary landfill of municipal waste in the town of Sombor (sample 1a). The second is a waste dump located on the stretch between rural settlements Svetozar Miletic and Čonoplja (samples 1b and 1c). Samples of soil were taken from the surface layer of soil and at a depth of 30 cm. Sekulić et al. [9] reported that a large number of metals such as Cd, Hg, Pb, Ag, As, Sb, and Zn accumulate in the upper layer of the soil profile. This phenomenon can be attributed to the adsorption process because heavy metals are adsorbed by soil organic matter. Also, they are deposited from the atmosphere on the soil surface and absorbed by many plants. The collected soil samples were analyzed using standard analytical methods. The heavy metals content was determined by the EPA 6010C method *Inductively coupled plasma-atomic emission spectrometry* on the camera ICP-OES system Thermo ICAP 6500 Duo (Thermo Fisher Scientific, Waltham, MA, USA).

The S/S technology with different cement components manufactured by the Lafarge was selected to treat contaminated samples. An innovative additive based on natural and synthetic zeolites (ImmoCem, PowerCem Technologies, Moerdijk, Netherlands) was selected and added to individual samples. The use of Portland cement or mixtures of different types of cement for the treatment of liquids, sludges, or heavy metal-containing heavy metals is widespread [10]. According to Santanu et al. [11], alkaline materials, primarily cement, are often used for S/S treatment for several reasons: cheaper than other binding materials; easily incorporate waste, and alkalinity reduces the solubility of inorganic pollutants, primarily toxic metals.

S/S samples were formed by mixing contaminated samples with cement and adding a hand mixer for a few minutes or until a homogeneous mixture was formed. Then, a certain amount of water was added to accelerate the hydration process during the mixing process. After adding water, the mixing process is continued for a few minutes to achieve a predefined water and cement ratio. After the mixing process, the formed content is placed in specially prepared modules. In the modules, the content is manually compacted. The prepared samples were left in the modules for seven days at room temperature between 15 and 20 °C. Their wetting was carried out daily using deionized water to prevent the bursting of S/S material. After seven days, S/S samples were taken out of the mold and left to dry for another 27 days at room temperature.

Application of leaching test on S/S samples. On all S/S samples, a modified semi-dynamic ANS 16.1 leaching test was applied. The test was carried out at room temperature with deionized water as a leaching solution. The samples were placed in a suspended position from the top of the glass beaker filled with deionized water, wherein the ratio of liquid/solid (L/S) was 5:1. The test was performed under static conditions. First, the monoliths were immersed in deionized water. Then, after 1, 8, 24, and 72 h, as well as 7, 14, 21, 28, and 56 days the samples (25 cm^3) were taken from deionized water. Samples of leached content after 7, 14, 21, 28, and 56 days were filtrated on a membrane filter (0.45 µm), and analyzed for the content of the following metals: As, Cd, Cr, Cu, Ni, Zn. The ICP-OES system Thermo iCAP 6500 Duo (Thermo Fisher Scientific, Waltham, MA, USA) was used for the analysis.

3. RESULTS

The results of the heavy metal content analysis in the waste sludge samples are shown in Table 1.

Table 1

Heavy metal contents in waste sludge samples [mg/kg]

Sample	As	Cd	Cr	Cu	Ni	Zn
1	**37.55**	**3.644**	16.23	**213.5**	22.26	**1032**
2	**1057**	**3.593**	0.1	<0.03	6.976	15.71
3	1.951	**1048**	55.78	**575.0**	**1222**	**1463**
4	0.2093	**2.631**	**318.0**	3.266	9.499	42.76
ELV*	29	0,8	100	36	35	140

*ELV – environmental limit value. Regulation on a program of systematic monitoring of soil quality, indicators for assessing the risk of soil degradation, and method for the development of remediation programs (Official Gazette of the Republic of Serbia, No. 88/2010). The heavy metal concentrations exceeding the ELV are bold.

In samples of sludge from the plant for conditioning of raw water, increased content of arsenic and cadmium was detected. In addition, significant quantities of chromium, nickel, copper, zinc, and cadmium were observed in sludge from industrial enameling processes. For example, the sludge from the tefloning process contains chromium at three times higher concentrations than the limit value. The dry matter content and the pH value in the waste sludge samples are shown in Table 2.

Table 2

Content of dry matter and pH in samples of waste sludge

Sample	Moisture content [%]	Dry matter content [%]	pH
1	7.84	92.16	7.08
2	95.62	4.38	7.47
3	0.39	99.61	8.66
4	51.96	48.04	6.84

The results of the content of heavy metals in soil samples are presented in Table 3.

Table 3

Contents of heavy metals in soil samples [mg/kg]

Sample	As	Cd	Cr	Cu	Ni	Zn
1a	4.095	**3.559**	22.61	10.66	12.67	28.42
1b	8.695	**3.653**	55.68	**36.72**	**63.81**	136.5
1c	4.284	**3.152**	10.62	10.14	12.28	31.40
ELV	29	0.8	100	36	35	140

The heavy metal concentrations exceeding the EVL) are bold.

The measured concentrations of heavy metals in soil samples are compared with the limit values for the content of hazardous and harmful substances in soil, given in the *Regulation on a program of systematic monitoring of soil quality, indicators for assessing the risk of soil degradation, and methodology for the development of remediation programs* (2010). Border values were exceeded for cadmium in all three soil samples and copper and nickel in sample 1b. In sample 1c, the heavy metal content is below the ELV for all analyzed heavy metals. The dry matter content (gravimetric) is 98.91% in sample 1a, 97.99% in sample 1b and 97.61% in sample 1c. The measured pH value was 10.36 in sample 1a, 7.37 in sample 1b, and 7.47 in sample 1c.

Table 4

Composition of the samples prepared by mixing the sewage sludge with soil and sand samples

<table>
<tr><td>Sample</td><td>1a</td><td>2a</td><td>3a</td><td>3b</td></tr>
<tr><td rowspan="3">Composition</td><td>sample 3 (60%)
+ sample 1a (40%)</td><td>sample 1 (45%)
+ sample 1b (55%)</td><td colspan="2">sample 2 (33%)
+ sample 1a (33%)
+ sand (33%)</td></tr>
<tr><td>4A</td><td>5A</td><td colspan="2">5B</td></tr>
<tr><td>sample 4 (50%)
+ sample 1a (25%)
+ sand (25%)</td><td colspan="3">sample 2 (15%) + sample 1a (27%)
+ sample 1c (28%) + sand (30%)</td></tr>
</table>

After mixing sludge samples with soil and pure sand (purchased from Thermo Scientific), new samples of contaminated material were obtained. The following table (Table 4) presents the percentage share of soil, sludge, and sand in newly formed samples (the composition of samples).

Composition and characteristics of S/S samples. Table 5 presents data on the content of each of the sample components separately.

Table 5

Composition of S/S samples [kg/m^3]

Sample	Material (waste sludge, soil and sand)	Portland cement	Additive ImmoCem	Water
1a	1934	618.88	9.28	748.07
2a	1196.58	95.28	–	150.97
3a	1404	831.75	–	–
3b	1404	831.75	8.32	1
4a	1626	251	–	–
5a	1720	270.44	16.23	–
5b	1720	270.44	–	–

After removing the samples from the mold, the labeling was done. The dimensions of all samples were then determined, and their surface area and volume were calculated. The mass of S/S samples was measured on the analytical balance. The characteristic of S/S samples is given in Table 6.

Table 6

Characteristics of S/S samples

Sample	Mass [g]	Side a [cm]	Side b [cm]	Side c [cm]	Surface area [cm^2]	Volume [cm^3]
1a	172.55	4.1	5.6	5.1	144.86	117.096
2a	161.78	4.1	5.7	5.2	148.66	121.524
3a	197.15	4.1	5.7	5.2	148.66	121.524
3b	196.24	4.1	5.7	5.2	148.66	121.524
4a	163.83	4.1	5.6	5.3	148.74	121.688
5a	175.63	4.1	5.7	5.2	148.66	121.524
5b	178.06	4.1	5.7	5.1	146.70	119.187

Leaching test results. The results from the leaching test S/S samples for As, Cd, Cr, Cu, Ni, and Zn are presented in Figs. 1–7.

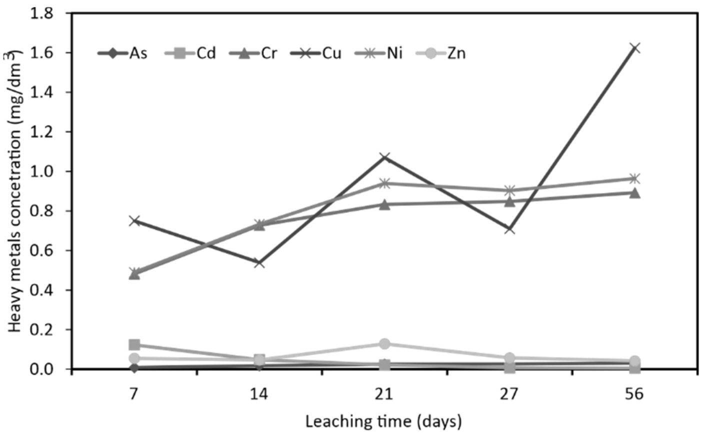

Fig. 1. The cumulative fractions of heavy metals leached from sample 1a

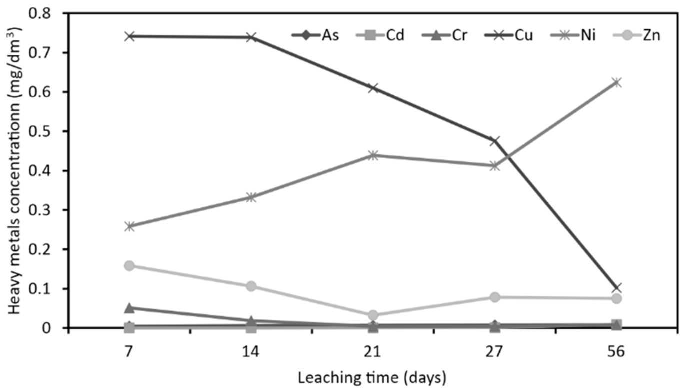

Fig. 2. The cumulative fractions of heavy metals leached from the sample 2a

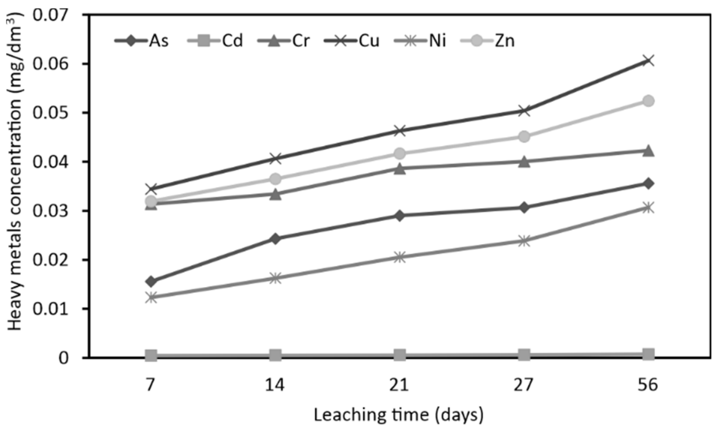

Fig. 3. The cumulative fractions of heavy metals leached from the sample 3a

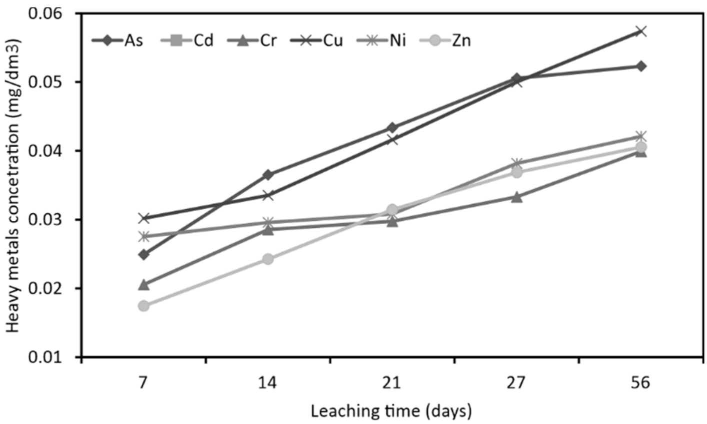

Fig. 4. The cumulative fractions of heavy metals leached from the sample 3b

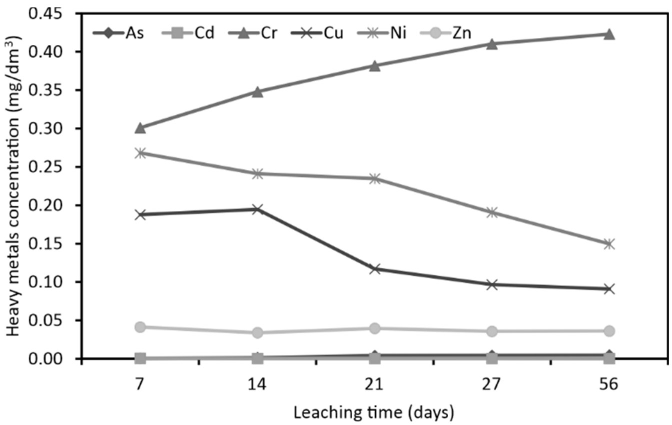

Fig. 5. The cumulative fractions of heavy metals leached from the sample 4b

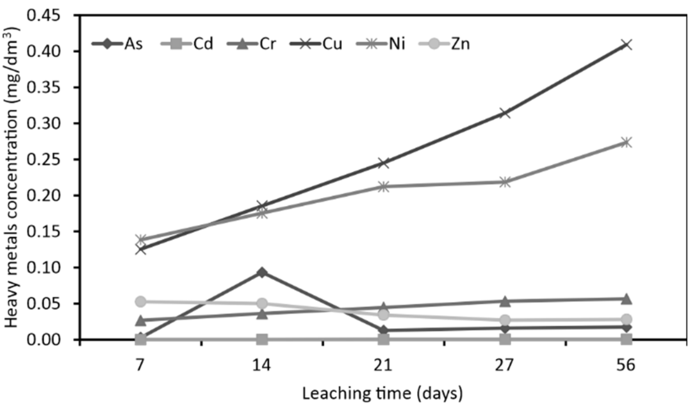

Fig. 6. The cumulative fractions of heavy metals leached from the sample 5a

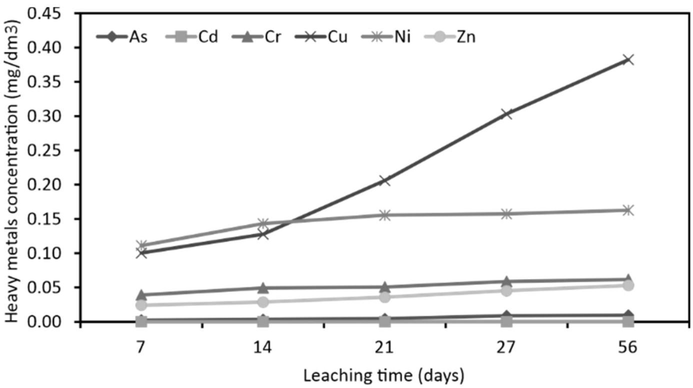

Fig. 7. The cumulative fractions of heavy metals leached from the sample 5b

4. DISCUSSION

Leach testing is arguably the best indicator of the effectiveness of pre-landfill treatments, including solidification/stabilization, in reducing the mobility of waste constituents [12]. However, further recommendations suggest that leaching tests should be related to the conditions governing the leaching mechanisms in actual disposal conditions [13]. Therefore, several methods for leaching analysis have been developed in Europe today to link leaching tests with specific disposal scenarios [14]. The leaching of metals from S/S mixtures is highly complex, as many factors can affect the release of specific constituents over time. As previously mentioned, leaching is influenced by many characteristics such as chemical characteristics of the elements, pH value, redox potential, complexing, the ratio of solid-liquid, and contact time [15]. The EU countries have reported several requirements regarding the quality of waste that can be safely accepted and disposed of in landfills [16]. To meet the requirements of Directive 99/31/EC, Member States have developed several procedures to assess the level of pollutants leaching from waste.

In 2002, the Council of Europe adopted Decision 2003/33/EC to unify waste characterization in all EU Member States before its acceptance into landfills. The Decision points out that one of the main parameters for the characterization of waste before its disposal in landfills is leaching monitoring of pollutants. Decision 2003/33/EC has prescribed the appropriate leaching tests: granular waste compliance leaching test (EN 12457/1-4) and percolation test (prEN 14405). These tests were not used in the paper because they are not adjusted to analyze leached pollutants from monolithic samples. In both prescribed tests, the shredding of monolithic material should be done. However, that is not a realistic solution for this study because it does not correspond to the expected conditions in the field. In future research, it is possible to carry out these test types on formed S/S samples to examine their further application in the construction industry.

Regulation on categories, testing, and classification of waste [6] prescribes a list of parameters for testing waste for disposal using the TCLP test (*Toxicity characteristic leaching procedure*). Also, the Regulation defines the list of parameters for testing waste and process waters from inert, non-hazardous, or hazardous waste landfills using the EN 12457/1-4 test. These tests are not suitable for application to the S/S samples because the goal of S/S is the production of monolithic samples, which these leaching tests do not take into account. The Rulebook also prescribes a list of parameters for determining hazardous waste physical and chemical characteristics intended for physical and chemical treatment, containing minimum criteria for disposing of the monolithic waste in hazardous waste landfills. The Rulebook prescribes the leaching tests for monolithic waste according to NEN 7345, which is based on the same principle as applied ANS 16.1 test. Namely, both tests belong to the group of semi-dynamic tests that evaluate the leaching of metals in diffusion-controlled conditions. By using NEN 7345, as well as ANS 16.1, it is possible to determine the cumulative amount of metal leaching out from the solidified mixture over a specific time. Since these tests are based on the same principle, it is possible to compare them.

Models of leaching presented on analyzed samples follow the expected behavior for monolithic samples. The penetration of liquids along the depth of the sample is limited to a relatively thin surface layer. The main goal of S/S is to immobilize pollutants by preventing their contact with the liquid. Analysis of the content of arsenic, cadmium, chromium, copper, nickel, and zinc in contaminated sludge before the application of S/S methods showed a very high concentration in some samples (Table 2). The highest concentration of As was identified in a sludge sample from the water filter factory (Subotica) and was 1057 mg/kg. The highest concentrations of Cd (1048 mg/kg), Cu (575 mg/kg), Ni (1222 mg/kg), and Zn (1463 mg/kg) were identified in the sample of sludge from the enameling process (Gornji Milanovac).

After applying S/S methods to contaminated material samples, some heavy metals were liberated from the surface layer by diffusion. At the same time, the solid crystal structure was formed inside the S/S samples due to chemical reactions between soil and cement. The level of As, Cd, Cr, Cu, Ni, and Zn leaching has increased over time. However, the leaching trend increases with a much lower intensity after a certain period. Halim et al. [17] explained the dependence of Cd leaching and pH value, stating that the lower amount of Cd would leach at a pH value greater than 5. According to the literature data [18], the monitoring of As, Cd, Cr, Cu, Ni, and Zn levels from S/S samples over a more extended period (100 days) shows that the concentration of leached heavy metals in most cases increases with low intensity up to a certain point. After that point, it indicates a tendency to stagnation.

Cumulative concentrations of As, Cd, Cr, Cu, Ni, and Zn leached from S/S samples were compared with the concentrations prescribed by the EU Directive (2003/33/EC).

As a result, all samples can be considered inert or non-hazardous waste because the leachable concentration of analyzed heavy metals in the first 56 days of leaching monitoring is below the concentration prescribed by Decision 2003/33/EC.

According to the Regulations of the Republic of Serbia [5] and the criteria for the maximum permissible concentration of As, Cd, Cr, Cu, Ni, Zn, and pH in leached liquid, the treated S/S samples do not fall into the category of hazardous waste. Therefore, S/S waste samples can be disposed of at the landfill for inert or non-hazardous waste without negatively impacting human health and the environment. The efficiency in As immobilization is already noticeable with the application of 6.6% of Portland cement, i.e., in sample 2a (Table 5). After 56 days of leaching, 0.0082 mg/dm^3 of As was released from the sample. The sample was made without any additives and with the addition of 10.5% of water.

By comparing all S/S samples, the lowest concentrations of Cd and Ni leached from the sample were obtained using 13.5% cement, without additives and water. In addition, comparing Cd and Ni leaching levels from S/S samples was performed between samples with and without additives. 0.8% cement additive for Cd and 0.4% cement additive for Ni were used in the samples with the additive. In both cases, it was concluded that the addition of cement additives did not significantly increase the efficiency of heavy metals immobilization.

The lowest concentration of Cr, Cu, and Zn was leached from a sample obtained using 37% cement, 0.4% additives, and 0.04% water, comparing all S/S samples. The leaching level of Cr, Cu, and Zn was compared between samples with and without additives. It can be concluded that the addition of additives in samples influenced the enhancement of the efficiency of heavy metals immobilization to a lesser extent. Montañés et al. [19] conclude that chromium retention decreases as the relative amount of cement and water increases, probably due to cement's additional chromium and increased porosity in the mixtures. Gracia et al. [20] reported that Cu ranges from 0.02 to 0.04 mg/dm^3 in the leachate, and it depends on the share of lime in the sample.

5. CONCLUSIONS

EU membership imposes on the Republic of Serbia several obligations and requirements for implementing measures in waste management and wastewater treatment. For example, it is forbidden to dispose of untreated waste sludge directly on the land, and its adequate treatment is required before the final disposal.

Serbia currently produces about 4000 tons/year of waste sludge from wastewater treatment plants. It is expected that this amount will increase significantly due to the construction of a municipal wastewater treatment plant. Therefore, large amounts of waste sludge are expected, which should be disposed of properly. However, the current practice of treating waste sludge is unacceptable due to the migration of pollutants and potential negative impacts on the environment. Hence, it is necessary to find possible methods of waste sludge treatment to be adequately handled and disposed of properly without being a threat to human health and the environment.

Generally, it can be concluded that the sludge contaminated with heavy metals after applying S/S with the addition of different portions of the cement is converted into a non-hazardous, monolithic material. Therefore, waste sludge contaminated with heavy metals (As, Cd, Cr, Cu, Ni, Zn) after treatment with S/S technology can be safely disposed of in landfills for non-hazardous waste without negative consequences for human health and the environment. Arsenic immobilization's highest efficiency is formed by adding 13.4% cement to the contaminated material without additives and water. The highest efficiency in cadmium immobilization is achieved by using 13.5% cement without impurities. In contrast, the highest efficiency in chromium, copper, and nickel immobilization is observed when using 37% cement, 0.4% additives, and 0.04% water. The highest efficiency in nickel immobilization is achieved when using 37% cement without the addition of additives and water.

Based on the comparison of total cumulative concentrations of metals with the concentrations prescribed by the EU Directive (2003/33/EC), only a mixture of soil and 37% cement in terms of concentration of As, Cd, Cr, Cu, Ni, and Zn can be considered as inert waste. At the same time, other S/S materials can be classified as non-hazardous waste. According to the Republic of Serbia Regulations on categories, testing, and classification of waste, none of the considered S/S samples does fall into the category of hazardous waste. Considering the limits on the concentration of certain heavy metals in the procedure, analyzed S/S samples can be disposed of in an inert or non-hazardous waste landfill with aspects of the concentration of As, Cd, Cr, Cu, Ni, and Zn.

The primary conclusion drawn from this study is that the S/S method should be used to transform waste sludge, i.e., hazardous waste, into material that can be safely disposed of in landfills for non-hazardous or inert waste. The S/S method is fundamental in the Republic of Serbia, as it is common practice to dispose of waste sludge improperly.

Future research should focus on examining the characteristics of S/S samples of waste sludge to confirm the possibility of their commercial use.

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