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Utilization of Galvanic Sewage Sludge to Produce Alkali-Activated Materials

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ABSTRACT'

The development of civilization is causing an increase in the amount of industrial wastes, especially hazardous wastes. Among the methods of utilisation of this type of waste, the methods of stabilisation and solidification deserve attention, which enable the transformation of hazardous waste and the heavy metals it contains into an environmentally safe form, that is, immobilised. This paper proposes a method to stabilize and solidify galvanic sludge using geopolymerization reactions. The surface morphology was studied, and the chemical composition was analyzed using the SEM-EDS method. The presence of characteristic functional groups on the surface of the galvanic sludge and the geopolymer obtained on its basis was determined by FTIR spectroscopic analysis. Moreover, we evaluated of the recovery of selected heavy metals were performed on the basis of leaching tests. It was found that, as a consequence of geopolymerization of galvanic sludge, this hazardous waste is transformed in such a way that the heavy metals it contains, which occur in the form of soluble compounds are immobilised. In relation to the metals analyzed, reduction in solubility were obtained at a practical level of 100% for Zn and Mn, for the remaining metals, respectively, 94% for Cu and in the range of 40 to 90% for Pb. Analysis of the FTIR spectra showed that the ions of the metals studied were permanently immobilised in the aluminosilicate structure of the geopolymers obtained. This shows, that galvanic sewage sludge, as hazardous waste, is chemically transformed into inert waste that may be deposited in landfills.

Keywords: galvanic sludge, heavy metal, leaching test, geopolymers.

INTRODUCTION

Industrial waste is a threat to human health, animal health, and each of the components of the environment (atmosphere, hydrosphere, biosphere, as well as the earth's surface). These hazards arise at all stages of waste management, including generation and collection, transport, treatment, and disposal, as well as during waste disposal. Numerous substances belonging to the heavy metal group, as well as aromatic and aliphatic hydrocarbons, pesticides, phthalates, are present in the composition of industrial waste, which pose a threat to humans and the environment due to their toxic and physicochemical properties (Jeyasundar et al., 2020). Due to the scale of exposure to toxic substances contained in waste and the wide range of possible effects, hazardous wastes associated with the production and manufacture of metals deserve special attention. An example of such waste is electroplating sludge, which comes from electroplating and electronics plants. It has a different chemical composition, which depends on the production process and the type of reactants used in the neutralization of this type of waste (Letcher et al., 2019).

According to environmental requirements, waste management should focus on waste prevention, preparation for reuse, recycling, resource recovery, and disposal (Li et al., 2018). The hazardous waste disposal process can be carried out by biological (Ayilara et al., 2020; Luo et al., 2017) thermal (Irisawa 2021), chemical (Mwembeshi et al., 2004; Westlake et al., 2013), and by landfilling (Jarnerud et al., 2021) methods. The choice of a particular method is possible after a thorough analysis of the composition and properties of the waste. It should follow the principle of sustainable development and meet the so-called triad of conditions that determines the success of an investment, i.e., economic efficiency, ecological effectiveness, and social acceptance. This is due to the fact that in the disposal process, in addition to hazardous waste, water, air, and appropriately selected reactants are supplied depending on the disposal process being carried out, and the course of the process results in the emission of gases, the emission of water-soluble pollutants, and the generation of secondary waste that does not contain hazardous components (Toledo et al., 2018).

One method of disposing of hazardous waste is solidification and stabilization. As a result of this process, the waste is transformed in such a way that the harmful substances contained, which are in the form of water-soluble compounds, are not washed out but immobilized. The waste thus transformed, with improved mechanical strength, can be a raw material practically used in the economy, e.g. as construction materials. Wastes whose physical properties cannot be improved by solidification and stabilization can be treated as inert waste, i.e. less harmful than hazardous waste, and can then be deposited in landfills (Basegio et al., 2009; Bednarik et al., 2005; Luz et al., 2006).

Binders play a key role in the immobilization of heavy metals, the most commonly used in solidification and stabilization systems being Portland cement. An alternative to cements is geopolymers, which exhibit excellent properties, including favorable pore structure, low permeability, very high alkalinity, good chemical stability, and a three-dimensional microstructure (Jia et al., 2020; Nergis et al., 2018). Recently, many studies on geopolymers have been conducted on the degree of immobilization of toxic wastes (Nohajerani et al., 2019; Vu and Gowripalan 2018). The process to obtain geopolymers is not only a method for synthesis of compounds widely used in the construction industry as a replacement for Portland cement but also a way to dispose of and recycle hazardous and toxic waste. In the geopolymerization process, hazardous waste is disposed of simultaneously through a physical process (encapsulation of elements in the material) and by embedding them into the resulting network through chemical reactions. The geopolymer matrix is a good material for heavy metal immobilization because of its low permeability and resistance to acid and chloride ions. Waste-based geopolymers show a higher heavy metal-ion immobilization ability than Portland cement or waste alone (Mikuła et al., 2017 and Zhang et al., 2016). Studies conducted by Zhang et al. (2008) show that Pb(II) ions can be effectively immobilized with a fly ash-based geopolymer by binding Pb(II) in the form of insoluble silicate (Pb_3SiO_5). El-Eswed et al. (2017) also showed that the heavy metals Pb(II), Cu(II), Cd(II), and Cr(III) can be immobilized using a geopolymer based on kaolin. There are three possible mechanisms for the immobilization of heavy metals in the geopolymer matrix:

- ion exchange of Na^+, K^+ cations by heavy metal ions, which balances the negative charge of fragments of geopolymer chains containing aluminum (electrostatic interaction);
- formation of covalent bonds between heavy metals and the aluminosilicate chain;
- formation of hydroxides, carbonates, silicates and heavy metal aluminates (El-Eswed et al., 2017).

The present work focuses on this issue, concerning utilization of hazardous waste, namely galvanic sewage sludge, to produce alkali activated materials (geopolymers).

MATERIALS AND METHODS

Galvanic sewage sludge from the Sewage Treatment Plant of the Screw Factory in Łańcut (Poland) and a geopolymer obtained based on this sewage sludge were used as objects of the study. Galvanic sewage sludge is produced during production processes, such as etching and electroplating of metal components. During the etching process, the metal components are treated with sulfuric acid(VI) and washed with water after etching. During the electroplating process, metal components are electroplated with a thin layer of zinc to increase their resistance and protect them from corrosion. The byproducts of the above-mentioned processes are solid and semi-liquid impurities that constitute galvanic sewage sludge. It was used as a solid mixture composed of equal parts by weight of quartz sand and sewage. A mixture of sodium silicate (oxide content $SiO_2 + Na_2O$ – not less than 39% by weight; molar modulus SiO_2/Na_2O = 2.4–2.6) and solid sodium hydroxide (purity 97% by weight) was used as an alkaline activator. To obtain the geopolymer paste, the mixture of solid components was mixed with an alkaline activator in a ratio of the aqueous phase [cm^3] to the solid phase [g] of 50:50. After obtaining a homogeneous mixture, the geopolymer mortars were transferred to cylindrical molds (14×25 mm). The samples were removed from the molds after 24 h and then cured at room temperature (~20 °C) and humidity (~68%) for 28 days. A detailed characterization of the starting raw materials and geopolymers obtained, as well as their synthesis conditions, is included in the work (Sitarz-Palczak and Kalembkiewicz 2021).

Characterization of the physicochemical properties of galvanic sewage sludge and geopolymers obtained on the basis of this sludge included examination of the morphology by scanning electron microscopy and determination of the chemical composition by the Scanning Electron Microscopy/Energy Dyspersive X-ray Spectroscopy (SEM/EDS) technique; Fourier Transform Infrared (FTIR) spectroscopic analysis; standard leachability tests American Society for Testing and Materials (ASTM), Toxicity Characteristic Leaching Procedure (TCLP), United States Environmental Agency (USEPA) under static and dynamic conditions; sequential extraction according to the Tessier procedure. The content of the metals tested in the solutions obtained was determined using the Flame Atomic Absorption Spectrometry (FAAS) method.

Scanning electron microscopy (SEM) was used to characterize the surface of the geopolymers, using a scanning electron microscope equipped with a microarea chemical analysis (EDS) attachment, model S-3400 N (HITACHI, Japan). Preparations were made by placing the ash on carbon tape with gold sputtering as a necessary condition to avoid artifacts resulting from electrical charging of the surface of nonconductive materials during scanning by the electron beam.

Infrared absorption spectra were recorded in the fundamental range of 4000–400 cm^{-1} with a resolution of 2 cm^{-1} using an FTIR spectrometer (Alpha model, BRUKER, Germany). Powder preparations for analysis were prepared by mixing the investigated samples with spectrally pure KBr. The mixture prepared in this way was pressed under vacuum at a pressure of 10 MPa, obtaining a preparation in the form of a lozenge.

The conditions for the leaching tests are described in the article (Sitarz-Palczak et al., 2019), while sequential extraction by the Tessier method is described in the article (Galas et al., 2016). All eluates from the leaching tests and the sequential extraction were filtered through membrane filters (0.45 μm). Then they were fixed by 65% HNO_3 and analysed. The concentrations of Zn, Cu, Pb, and Zn in solutions were determined by Flame Atomic Absorption Spectrometry (FAAS) using PERKIN ELMER model 3100 spectrometer. Each measurement was carried out with three repetitions holding relative standard deviation (RSD) < 5%.

RESULTS AND DISCUSSION

The SEM image of the galvanic sewage sludge presented in Figure 1 shows the complex morphology of the sludge studied. This is typical for this group of wastes. A large number of irregularly shaped particles are observed. The sizes of individual grains range from less than 10 μm to more than 300 μm. The smallest grains are found in the form of aggregates that bind the larger particles, this is due to the hygroscopic properties exhibited by the sediment samples. The highest concentrations of the elements determined were observed for zinc (36.8%), iron (27.97%), and oxygen (21.3%). Such a high content of these elements is related to the production processes that produce the sludge under study (etching, plating). The content of Cr, Mg, and Cu in the sludge is about 1%, the concentration of Si is about 4% and the concentration of Ca is about 11% (Fig. 2–4).

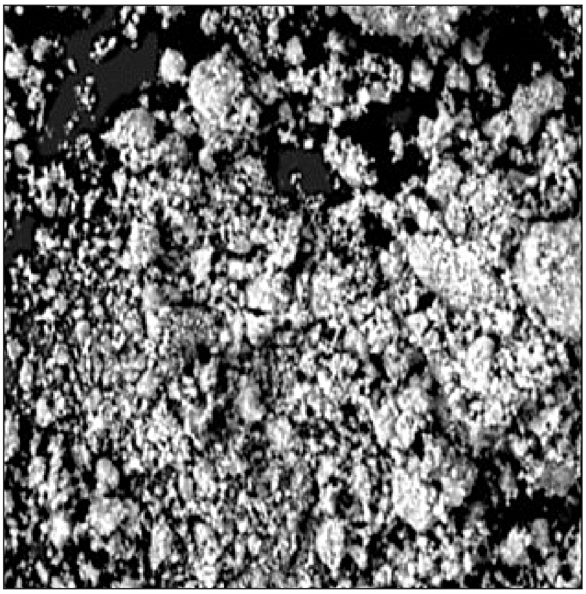

Figure 1. SEM image of galvanic sewage sludge

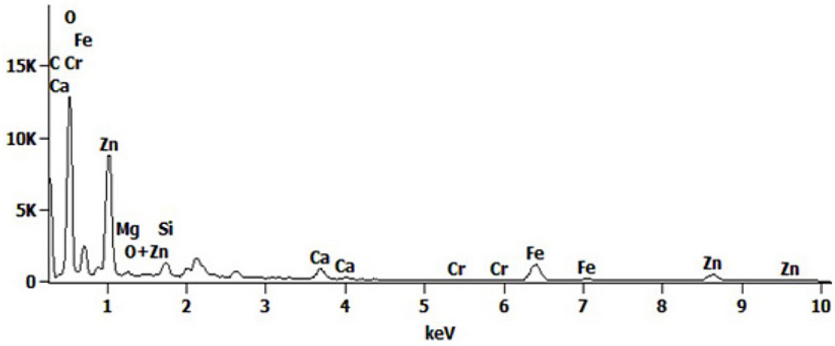

Figure 2. EDS analysis (research area – 1) of galvanic sewage sludge

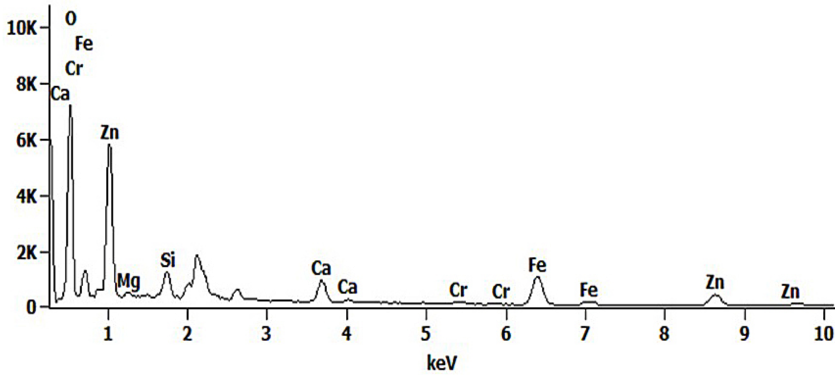

Figure 3. EDS analysis (research area – 2) of galvanic sewage sludge

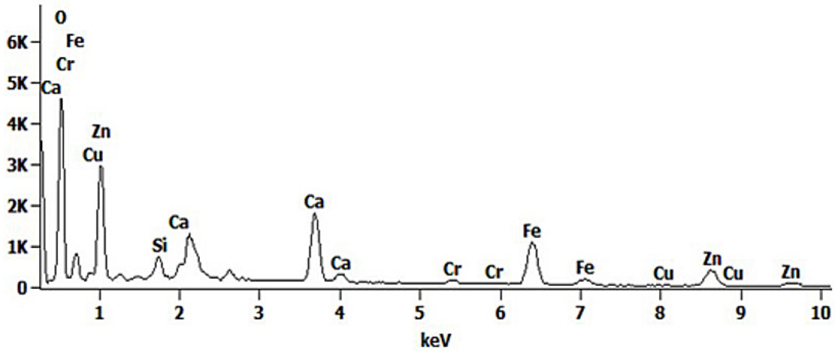

Figure 4. EDS analysis (research area – 3) of galvanic sewage sludge

Figure 5 shows an SEM image of the morphology of the geopolymer obtained from the galvanic sewage sludge.

A predominance of irregular particles with sharp edges is observed. The sizes of individual grains range from 1 μm to more than 1 mm. Large numbers of irregularly shaped grains shape the specific surface of this material. The highest concentrations were recorded for oxygen (36.45%) and sodium (32.93%) and silicon (18.13%). Such high values for these elements are due to the fact that the resulting geopolymer is composed of long-chain copolymers of aluminium and silicon oxides, admixtures of metal cations, such as sodium, which constitute the stabilizing material here, and bound water. The low calcium content corresponds to the presence of gypsum, which significantly reduces the porosity of the resulting material (Fig. 6-8).

The results of the FTIR spectroscopic analysis are shown in Figure 9. In the recorded spectra, changes were found, mainly in the bands associated with Si-O-Si(Al) vibrations occurring in the range 1200–450 cm^{-1} and changes in the bands attributed to OH group vibrations occurring in the range 2800–3500 cm^{-1}. The difference is a slight decrease in the intensity of the band at 3400 cm^{-1}, which corresponds to OH stretching vibrations.

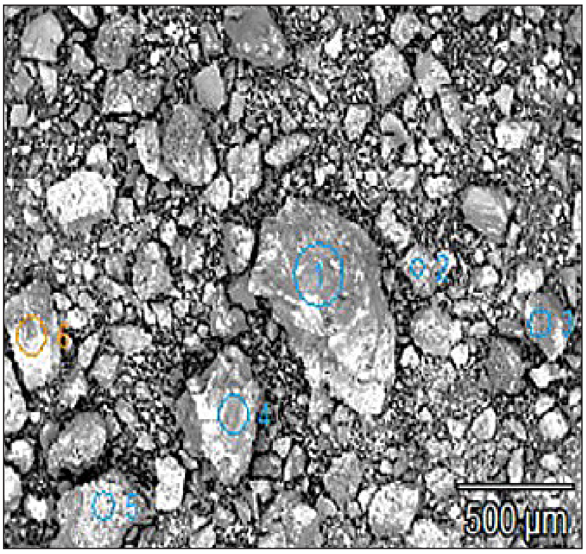

Figure 5. SEM image of geopolymer obtained from galvanic sewage sludge

Also observed is a shift in the stretching Si-O-Si bond, from 1035 cm^{-1} to a position of 990 cm^{-1} which indicates its elongation and a decrease in the angle of this bond. This shift can be attributed to an increase in the silicon fraction bonded to non-bridging oxygen atoms. A 562 cm^{-1} band appeared in the geopolymer spectrum, which is responsible for the vibrations that occur in AlO_6.

The term geopolymers refers to alkali-activated materials. Geopolymers exhibit a lack of long-range ordering in their structure, and there can be 3 basic structural units in the short-range ordering composed of sialane, siloxo-sialane, and disiloxo-sialane units. The sialate network contains tetrahedral SiO_4 and AlO_4, which are linked by a common oxygen atom. In all polymerized structures, aluminium has a coordination number of 4 and thus forms a negative charge in the molecule,

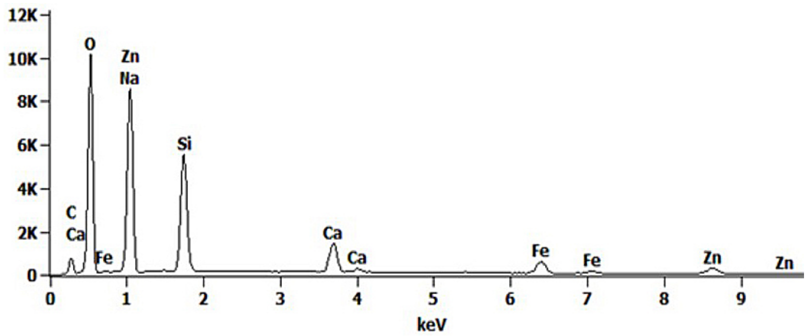

Figure 6. EDS analysis (research area – 1) of geopolymer obtained from galvanic sewage sludge

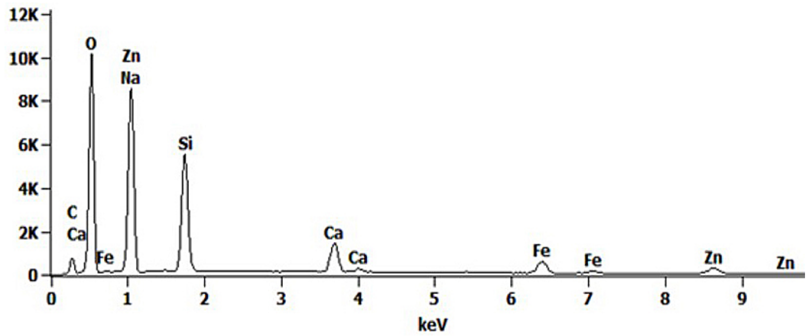

Figure 7. EDS analysis (research area – 2) of geopolymer obtained from galvanic sewage sludge

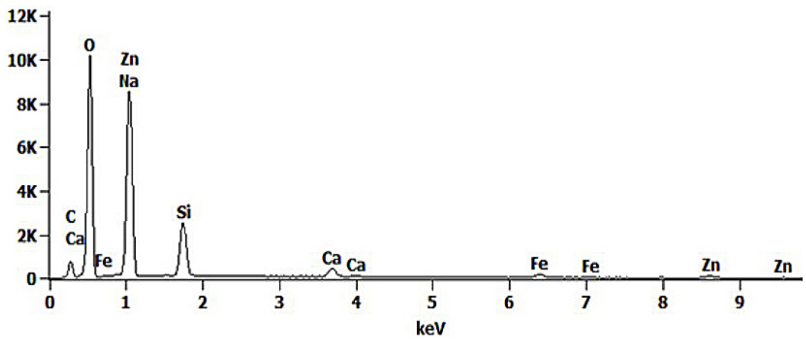

Figure 8. EDS analysis (research area – 3) of geopolymer obtained from galvanic sewage sludge

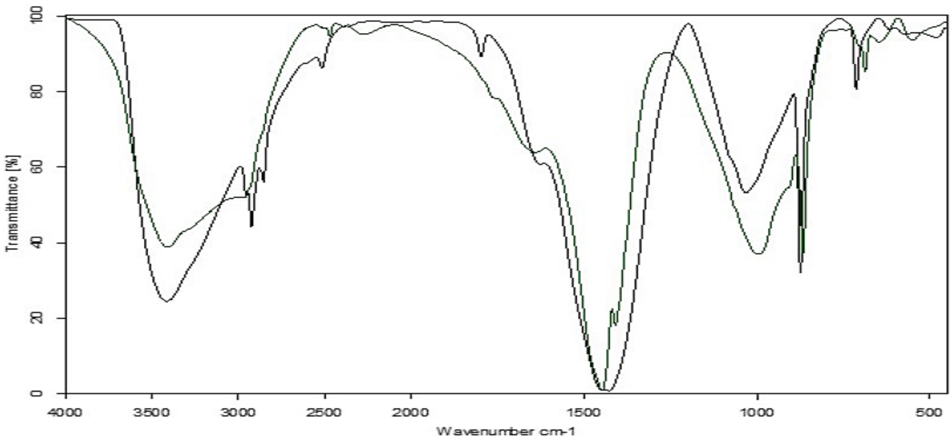

Figure 9. FTIR spectra of galvanic sewage sludge (black) and geopolymer obtained from galvanic sewage sludge (green)

which is compensated for by the presence of a monovalent cation such as sodium and potassium. Both the galvanization precipitate and the geopolymer exhibit a heterogeneous morphology, so a scattering of the results obtained is observed, which is characteristic of EDS microanalysis (Alvee et al., 2022). Chromium and magnesium were not detected in the geopolymer material (unlike in the precipitate). The geopolymer materials contained an excess of Na^+ ions in the form of salts, indicating an incomplete geopolymerization reaction. The reason for this may have been that the aluminosilicate gel formed shields the surface of the sediment particles preventing the release of Al and Si from the surface, therefore stopping further geopolymerization (Kosanović et al., 2008).

Solid samples, such as galvanic sewage sludge, represent characteristic matrices for potentially toxic metal species. The availability of these metals is greatly dependent on the characteristics of the surface of the particle, the kind and strength of the bond, and the properties of the solution in contact with the solid sample. It is the first step in the evaluation of the environmental and biological risk associated with this material; on the basis of this information, it can be decided if more information on the metals' availability should be gained. The determination of the toxic element in solid samples is of great importance for health and for the environment.

Leaching tests were carried out on the basis of the principles laid in standard procedures. Three standard leaching tests: The American Society for Testing and Materials (ASTM), the toxicity characteristic leaching procedure (TCLP), and the United States Environmental Agency (USEPA) were selected for this study to determine the stability of trace elements in galvanic sewage sludge. The degree of leaching of heavy metals is influenced by the pH value of the environment. In alkaline environments, Cu, Zn, and Mn form insoluble compounds, while in acidic environments their solubility increases. Lead belongs to amphoteric metals and shows the highest solubility at pH 8–10. On the basis of the results of the leaching tests (Table 1), the degree of leaching of the metals tested was determined in relation to their total content.

The tests carried out showed 100% immobilization for Cu using HOAc and H_2O under dynamic conditions and 94% under the other conditions of the tests carried out. Zinc and manganese were present in virtually insoluble form, resulting in virtually 100% immobilization of these elements. In general, the leaching rate of heavy metals from the plating sludge was low, with the exception of Pb. Despite the lowest total lead content compared to the other heavy metals, the highest leaching rate was observed for Pb under dynamic conditions, a value of 40%, while under static conditions it was 90%. The leachability of heavy metals from the surface of geopolymers obtained from galvanic sewage sludge is determined by the type of sample, i.e. monolithic form (static tests) or fragmented form (dynamic tests). In the case of monolithic forms, the degree of leaching of heavy metals is determined by surface leaching and diffusion processes. On the other hand, in the case of compressed forms, heavy metals leaching is related to the percolation process.

The mobility and biological availability of trace metals depend not only on their total

Table 1. Results of the standard leaching tests for the samples studied

Leaching tests	Leaching solutions (value of pH)	Recovey of metal [%]			
		Cu	Mn	Pb	Zn
Galvanic sevage sludge					
USEPA	HNO_3 (pH = 0.90)	2.31	9.12	6.93	14.17
TCLP	HOAc (pH = 3.00)	0.57	0.29	1.84	0.02
ASTM	H_2O (pH = 6.00)	-	0.17	-	0.01
Geopolymer					
USEPA	HNO_3 (pH = 0.90)	5.95	0.34	10.21	0.06
TCLP	HOAc (pH = 3.00)	-	1.03	30.00	0.05
ASTM	H_2O (pH = 6.00)	-	1.03	30.00	0.05

concentration but also on the physicochemical forms in which they occur. To evaluate the availability of metals from galvanic sewage sludge, the sequential extraction procedure by the Tessier method has recently been applied. The composition and concentration of the extraction solutions are given in Table 2.

Sequential extraction makes it possible to predict the potential for metal contamination of natural ecosystems and to assess the risks arising from the short-and long-term storage of the plating sludge and its geopolymerization product. Sequential extraction of samples by the Tessier method allowed the partitioning of metals between the individual chemical fractions defined as: water-soluble, exchangeable, carbonate, oxide, organic, and residual. To assess the risk arising from the storage of galvanized sewage sludge and geopolymer, the mobility index of the analyzed metals was calculated according to the following formula (Puga et al., 2016):

$$M = \frac{F0 + F1 + F2}{M_{total}} \cdot 100\% \tag{1}$$

where: M – mobility index of metal; F0 – metal content in the water-soluble fraction; F1 – metal content in the exchangeable fraction; F2 – metal content in the carbonate fraction [$mg \cdot kg^{-1}$]; M_{total} – total metal content [$mg \cdot kg^{-1}$].

The results for the mobility index of the studied metals are summarized in Table 3. The lowest mobility in galvanic sewage sludge, as well as in geopolymer, is shown by zinc, respectively: 0.06% and 0.005% and manganese: 0.68% and 0.55%. In general, higher values of the mobility coefficient were obtained for the sludge than for the geopolymer (with the exception of Cu and Pb); therefore, it can be concluded that

Table 2. Sequential extraction procedure using the Tessier method

Step	Chemical fraction	Extraction solutions
0	F0 - water soluble	H_2O (pH = 7)
1	F1 - exchangeable	1 M $MgCl_2$ (pH = 7)
2	F2 - carbonate	HOAc/NaOAc (pH = 5)
3	F3 - oxide	0.04 M $NH_2OH \cdot HCl$ in 25% (v/v) HOAc
4	F4 - organic	30% H_2O_2 (pH = 2) 3.2 M NH_4OAc in 20% (v/v) HNO_3
5	F5 - residual	$HClO_4$(conc.):HNO_3 (conc.) (1:2)

Table 3 Results of the sequential extraction procedure for the samples studied

Metal	Sum of fraction F0 – F2 [mg/kg]	Mobility index [%]
Galvanic sewage sludge		
Cu	-	-
Mn	8.28	0.68
Pb	8.12	9.90
Zn	155.07	0.06
Geopolymer		
Cu	2.81	1.34
Mn	6.64	0.55
Pb	9.69	11.82
Zn	12.05	0.01

geopolymerization of the galvanic sewage sludge is an effective method for reducing the mobility of selected metals (Mn and Zn). In the case of lead, there was an increase in mobility of about 10%, which may have been caused by the effect of pH. In an alkaline environment, copper, manganese, and zinc form sparingly soluble compounds, e.g. hydroxides, carbonates, silicates, and sulfides, and in an acidic environment their solubility increases. Lead is an amphoteric metal and has the highest solubility in the pH range of 8-10. The formation of hydroxysodalite in the geopolymer matrix affects the efficiency of immobilization of heavy metals. Copper occurs in galvanic sewage sludge mainly in a stable organic form, which is confirmed by the increase in the Cu^{2+} content in the organic fraction of the galvanic sludge. The Pb^{2+} cations are coordinated octahedrally by oxygen atoms in the 6-membered ring of the sodalite cage and by three water molecules. The higher leachability of Pb than other heavy metals may also result from the presence of crystalline $PbO·xH_2O$ inclusions formed in an alkaline environment and characterized by significant solubility in water (El-Eswed, 2020).

The permanent immobilization of Cu, Zn, Mn, and Pb ions in the aluminosilicate structure of the obtained geopolymers is also evidenced by changes recorded on FTIR spectra within the 1200–450 cm^{-1} bands, which relate to internal vibrations in the Si-O(Al) and Si-O-(Si) tetrahedral oxygen bridges (Zehua et al., 2020).

CONCLUSIONS

The consequence of geopolymerization of galvanic sewage sludge is that this hazardous waste is transformed in such a way that the heavy metals it contains, which occur in the form of soluble compounds, are not leached but immobilized. The applied method of solidification of galvanic sludge by geopolymerization allows the physical and chemical properties of the tested sludge to be changed, and, above all, results in reduced leachability of Cu, Mn, and Zn. The proposed waste disposal process is safe for the environment and is consistent with the idea of sustainable development. Geopolymers obtained on the basis of hazardous waste can be treated as inert waste, which can be landfilled.

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