Phosphating sludge: properties and immobilization in ceramics

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Insertion of industrial waste into the ceramic matrix is one of the heavy metal binding technologies, allowing to obtain products fit for use or to reduce the volume of waste before final disposal at the same time. The properties of phosphating process waste and opportunities for its stabilization in the ceramic matrix are investigated in this study.

Before investigation of stabilization a thermogravimetric analysis of phosphating sludge has been performed. The sludge was heated at 400, 600 and 1000 °C, and SEM images of sludge were made. The SEM images show that re-crystallization processes occur during the heating of the sludge.

Ceramic samples for leaching tests were formed by adding 5 and 10% of phosphating sludge to the clay mass and by burning them at 900–1050 °C for 1–4 hours. The results of the leaching tests showed that the concentration of heavy metals from the burned ceramic samples containing 5 and 10% of phosphating sludge is less than the detection limit of the instrument, and at the same time it is below the allowable limits set out in the Wastewater Management Regulation. In this way, phosphating sludge stabilization in the ceramic proved as effective heavy metals immobilization and hazardous waste decontamination.

Keywords: waste stabilization, phosphating waste, heavy metal leaching, ceramics

INTRODUCTION

Secondary products are inherent to any industrial process and normally cannot be avoided. In many cases, these types of materials cannot be re-used by other means and may become not marketable. These materials are typically given to third parties for further treatment. According to the content of the Reference Document on Best Available Techniques for the Waste Treatments Industries, reducing of the hazardous nature and transforming into useful materials belong to the basic reasons for treating waste. The main goal in the physico-chemical treatment of waste solids and waste sludge is to minimize the long-term release by leaching out primarily heavy metals and low biodegradable compounds. In principle, all treatment options can be applied to waste solids and waste sludge. However, the characteristics of the treated material and the effectiveness of a treatment technology can vary significantly depending on the specific properties of the original waste IN and on the type of a cleaning system applied. One of the treatment options may be immobilization [7].

The aim of immobilization is to minimize the rate of contaminants migration to the environment and/or to reduce the level of toxicity, in order to alter or improve the characteristics of the waste so that it can be disposed of. Two types of processes have been developed; these are commonly referred as stabilization and solidification [7].

Stabilization is a process, by which contaminants (e. g. heavy metals) are fully or partially bound by the addition of supporting media, binders, or other modifiers. Stabilization is accomplished by mixing the waste with a reagent (depending on the type of waste and reaction planned, this can be, for example, clay particles) to minimize the rate of contamination migration from the waste, thereby reducing the toxicity of the waste and improving the handling properties of the waste at the landfill. To achieve this, a process should include a physico-chemical interaction between the reagent and waste, rather than just a dilution. The physical mechanisms used in stabilization are as follows: macro-encapsulation, micro-encapsulation, absorption, adsorption, precipitation and detoxification.

Solidification uses additives to change the physical properties of the waste (as measured by its engineering properties such as strength, compressibility, and/or permeability). The term 'solidification' (and encapsulation or fixation) relates to the mixing of wastes with a reagent to produce a solid waste form (with low porosity and low permeability matrix) for landfill disposal. The solidified product is landfilled in either

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surface level or underground deposits. In some countries, it may be utilized as backfilling material in old salt mines.

Researches carried out on stabilization of galvanic and phosphating sludge in organic [2, 3, 11] and non-organic matrices, i. e. cement [10, 15, 19] and ceramics [1, 6, 8, 9], have been described.

However, a different approach to the stabilization of waste is noteworthy, namely, the cases where stabilized material is used in other sectors of economy. This is particularly the case for the heavy metal containing waste. This waste can be added to the concrete, and as a result, it can become an integral part of paving blocks, pavement slabs or kerbstones. Trials to reuse the sludge of metal surface treatment and electrochemical coating industry in production of cement clinker have been described [18]. Stabilization of electrochemical processes waste (plating process solutions and wash/flush waters neutralization sludge) in ceramic matrices can be attributed to a similar kind of stabilization methods. The results of the research [16, 20, 23] shows that a metal hydroxide additive affects the physico-mechanical properties of ceramics [20, 21]. On the other hand, the degree of immobilization of environmentally hazardous components largely depends on the properties of the additive, which, in turn, may affect the usage conditions of their leaching with a waste additive by-product.

The phosphating process can be defined as the treatment of a metal surface so as to give a reasonably hard, electrically non-conducting surface coating of insoluble phosphate, which is contiguous and highly adherent to the underlying metal and is considerably more absorptive than the metal. The coating is formed as a result of a topochemical reaction, which causes the surface of the base metal to integrate itself as a part of the corrosion resistant film [17].

The environmental impact of metal finishing operations is a matter of serious concern and the phosphate pretreatment operation is no exception to this. The phosphating sludge is generally considered as hazardous waste materials and is, therefore, subject to strict regulations as to disposal.

EXPERIMENTAL

Types and properties of sludge
This paper was written based on research of waste from three companies operating the phosphating process:
  • crystalline phosphating precipitation (U) obtained by filtering working phosphating solution in the company Elameta, UAB;
  • amorphous phosphating solution neutralization sludge (L) obtained from the neutralization of wastewater in the company Lanksti Linija, UAB;
  • amorphous phosphating neutralization sludge (S) after the neutralization of sludge in the company Snaigė, AB.

The investigated sludge is of a variable composition that depends on the metal being treated, the composition of the commercial product used for phosphating and the reagents used for neutralization. The sludge consists of phosphates and

hydroxides of metals given in Table 3 containing small amounts of sulphate, carbonate and fluoride impurities.

All the three types of sludge were dried at 105 °C and disrupted to less than a 0.5 mm particle size. The properties of the sludge are given in Table 1 [24].

Table 1. **Physical and chemical properties of the sludge**

Sludge	Properties				
	Color	Moisture (fresh sludge), %	Bulk density (dried sludge), kg/m³	Hexane soluble substances, %	Temperature of sintering, °C
Sludge L	Yellowish	57	0.441	5.60	960
Sludge S	Light brown	62	0.507	7.87	>1100
Sludge U	Gray	30	0.447	0.00	900

The clay from the Girininkai (Lithuania) pit was used, it was dried at 105–110 °C and was grained by a dismembrator into grains smaller than 1 mm. The average chemical composition of clay is given in Table 2 [25].

Table 2. **Chemical composition of used clay, in wt.%**

Clay	SiO_2	TiO_2	Al_2O_3	Fe_2O_3	CaO	MnO	MgO	Na_2O	K_2O	LOI
	47.69	0.76	13.35	6.19	9.05	0.06	3.59	0.96	3.32	11.9

LOI is loss on ignition.

Determination of metals in the dried sludge
Sludge identified metal ions were chosen based on the literature sources [4, 5], and most phosphating compositions contained those metals. Concentrations of the metals in the dried sludge were determined by a PerkinElmer Optima 7000 DV ICP-OES spectrometer.

Simultaneous thermal analysis (STA)
Differential scanning calorimetry (DSC) and thermogravimetry (TG) were employed for measuring the thermal stability and phase transformation of all three dried sludges at a heating rate of 10 °C/min in the temperature range from 30 °C up to 800 °C under air atmosphere. The tests were carried out on a Netzsch instrument STA 409 PC Luxx. The ceramic sample handlers and crucibles of Pt-Rh were used.

Heating of dried sludge
Each type of pure sludge was heated for 1 hour at 400, 600 and 1000 °C. The purpose of this investigation is to assess the changing of the sludge morphological structure at the high temperature conditions. Later these changes are fixed by the SEM method.

Scanning electron microscopy of the sludge
Scanning electron microscopy (SEM) images of the unheated dried and heated sludge were taken using a FEI Quanta 200 FEG scanning electron microscope.

Preparing and burning of ceramic samples

When preparing the samples, the amounts of dry powder of clay and sludge additive (5 and 10%) were mixed for one hour. Then, the water was poured into mixtures for preparation of a plastic forming mixture with humidity of 20–24%. Slabs of the size 60 × 30 × 10 mm were formed from these mixtures, dried at 105–110 °C and burned in a laboratory furnace SNOL 30/1300, a temperature bias no larger than ±3 °C. The temperature was increased up to 120 °C at 200 °C/h rate, afterwards, at 500 °C/h rate up to the desirable temperature. The burning temperature of the samples was 900, 1000, 1025, 1050 °C. The duration of isothermal exposure in the desirable temperature was 1–4 hours.

Leaching test for ceramic samples

Leaching tests of the dried sludge and prepared ceramic samples were done by immersion of sludge in different extraction solutions: pH 2.1, pH 4, pH 6.2–6.3, pH 8.1 and pH 10. The leaching tests were performed using the solids/liquids ratio 1/10. Concentrations of heavy metals (Cr, Cu, Mn, Ni and Zn) in the extracted solutions were determined after 24-hour exposition by an atomic absorption spectrophotometer Shimadzu AA-6800.

The detectable concentration limit for all determined metals was 0.01 mg/l.

RESULTS AND DISCUSSION

Properties of Sludge L

The obtained TG and DSC curves show that the first big decrease in mass may be due to the evaporation of water of crystallization. This is confirmed by the endothermic peak at 152 °C. Another exothermic peak at 212 °C may be due to burning of the absorbed polymer – flocculent from the waste water cleaning step. Exothermic peaks at 346, 523 and 566 °C may be related to the crystallization processes.

The application of X-ray diffraction analysis shows that Sludge L is of an amorphous structure; the X-ray image shows only a peak characteristic of magnesium ferrite $MgFe_2O_4$ (Fig. 2). The sludge after heating at 400 °C has crystallized, and the crystals became enlarged and slightly melted (Fig. 3). Sludge crystals after heating at 600 °C became enlarged and slightly melted (Fig. 4). The sludge melted fully at approximately 960 °C, and it was impossible to make an image of the melt.
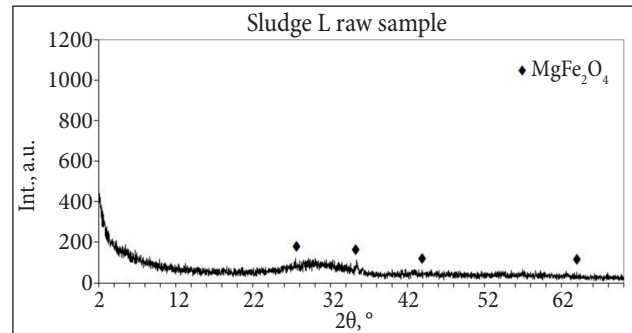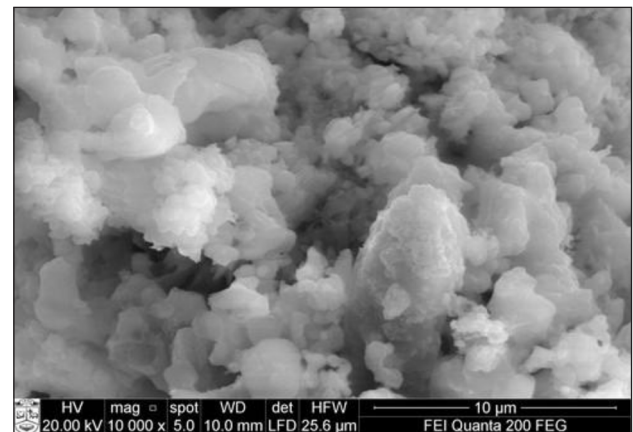

Fig. 2. Sludge L raw sample XRD analysis

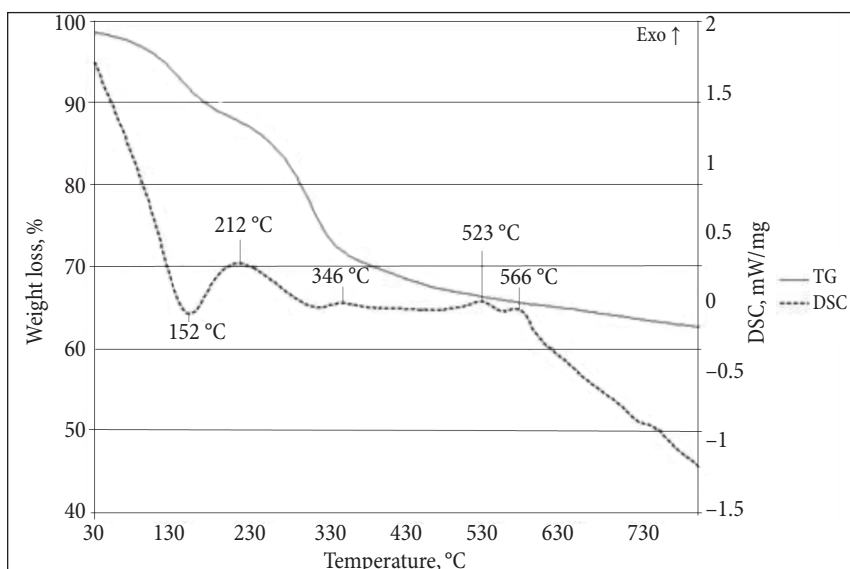**Fig. 3.** Sludge L heated at 400 °C

Fig. 1. TG and DSC curves for Sludge L

Properties of Sludge S

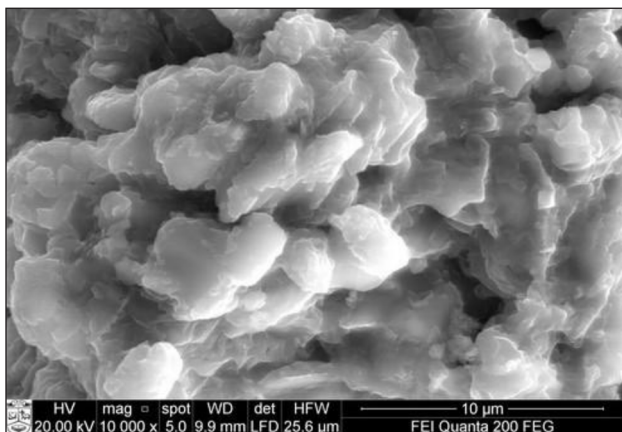The thermogravimetric curve shows a steeper and more abrupt decrease in mass from ~680–700 °C, when the mass

Fig. 6. Sludge S raw sample XRD analysis

Fig. 4. Sludge L heated at 600 °C

begins decreasing possibly due to carbonate decomposition that corresponds to the endothermic peak at 710 °C. The DSC curve shows a slight endothermic peak at 148 °C that can be attributed to the coming off of crystallization water.

The application of X-ray diffraction analysis shows that Sludge S is of an amorphous structure, but the X-ray image shows bright peaks of calcite $CaCO_3$ (Fig. 6). There were no significant visual changes in the structure after heating the sludge at 400 and 600 °C (Figs. 7, 8). However, the exothermic peak of crystallization is observed already at 494 °C (Fig. 5). The sludge becomes a clear crystalline structure at 1000 °C temperature, it is obviously visual from the SEM image (Fig. 9). Unfortunately, we did not have a possibility to record TG and DSC curves before reaching this temperature.
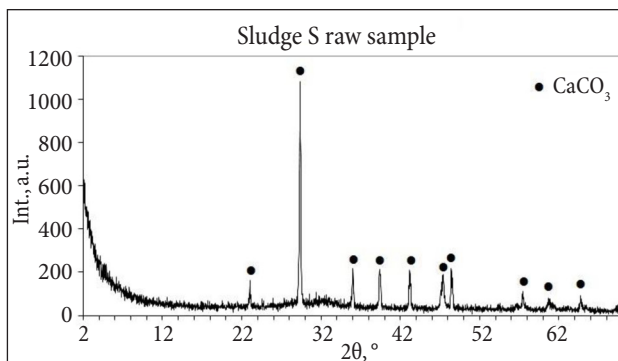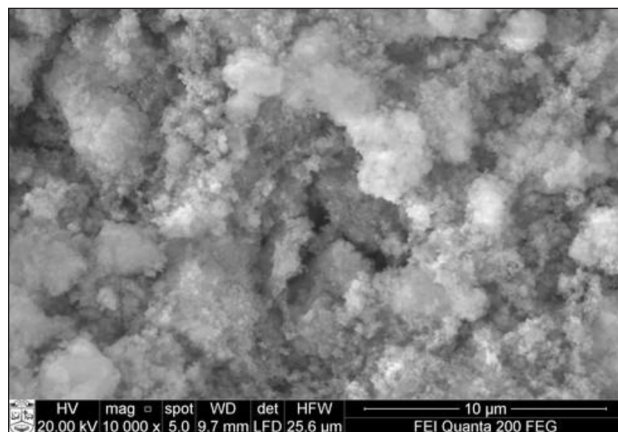

Fig. 7. Sludge S heated at 400 °C

Properties of Sludge U

The TG curve of Sludge U (Fig. 10) shows a steeper decrease in mass that corresponds to an endothermic peak at 141 °C of the DSC curve, showing the coming off of the crystallization water. A slight exothermic peak at 251 °C is most probably attributed to the combustion of organic impurities.
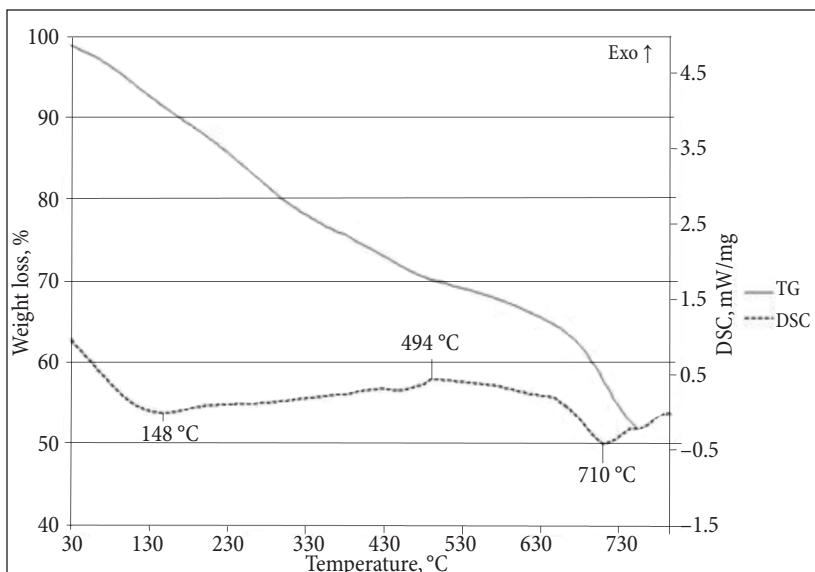

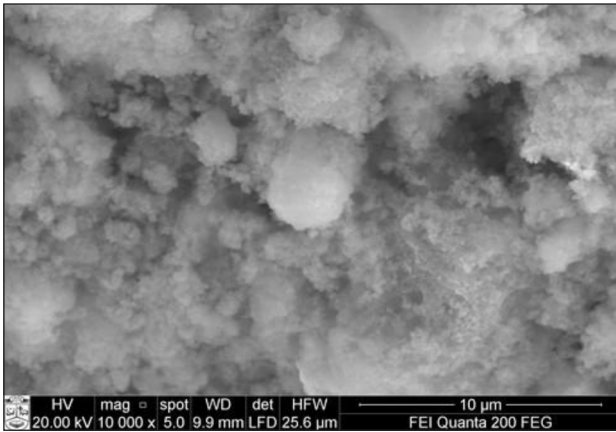**Fig. 5.** TG and DSC curves for sludge S
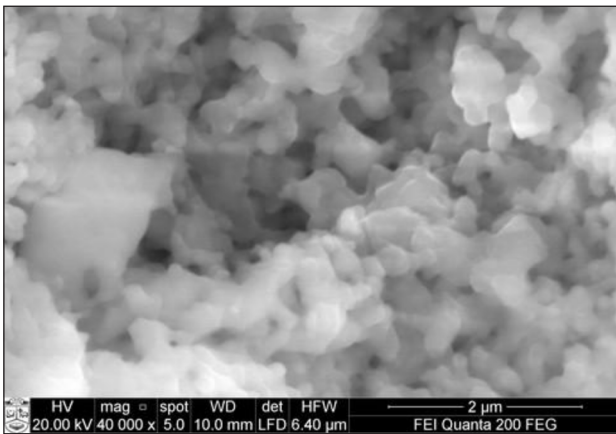
Fig. 8. Sludge S heated at 600 °C

Fig. 9. Sludge S heated at 1000 °C

The exothermic peak at 562 °C may be attributed to the formation of α-Fe_2O_3 as Mutlu Can et al. mentioned in [12].

Nariai et al. in the study [13] showed that the exothermic peak at 589 °C reflected the crystallization of $Ca(PO_3)_2$.
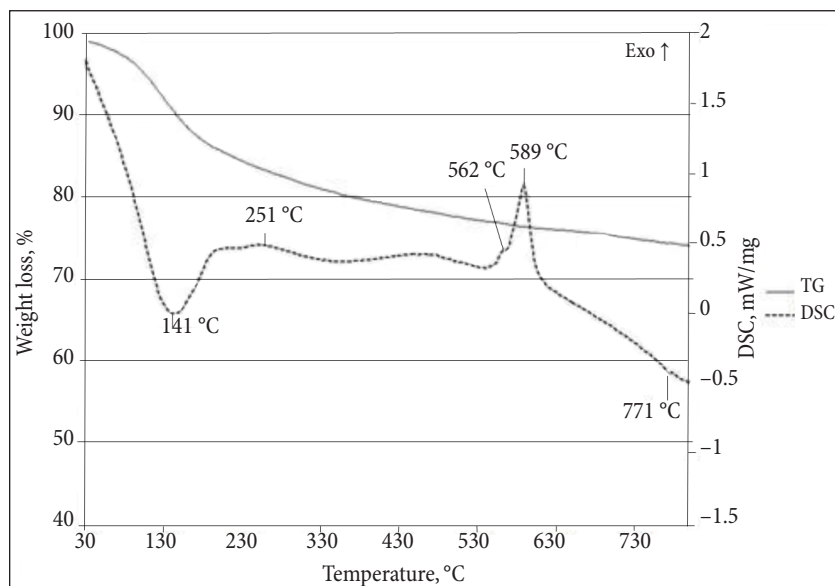
The endothermic peak at 710 °C may be attributed to formations of a liquid phase.

Such exothermic peaks are not visible in other types of sludge probably due to the fact that iron and calcium are in the form of other compounds.

The application of X-ray diffraction analysis shows that Sludge U is of an amorphous structure, but peaks characteristic of iron sulfate hydrate $Fe_2(SO_4)_3 \cdot 8H_2O$ and loseyite $(Mn, Zn)_7(OH)_{10}(CO_3)_2$ can be identified in its X-ray image (Fig. 11). At 400 °C temperature the sludge slightly melts without changing its structure (Fig. 12). The image of the sludge heated at 600 °C shows that the sludge mass is slightly melted (Fig. 13). The sludge melted fully at approximately 900 °C, and we had no possibility to make SEM images of the sludge.
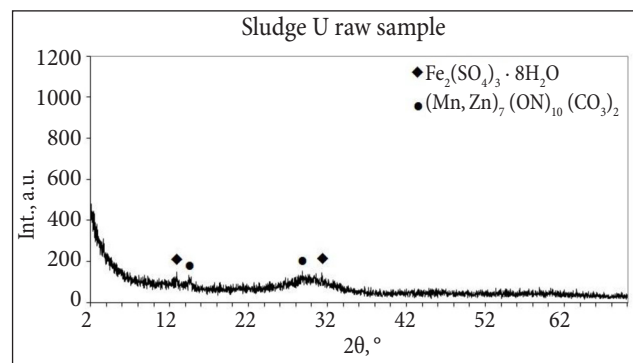
Fig. 11. Sludge U raw sample XRD analysis

Metal content in the sludge

The studies showed that, as was to be expected, sludge may be very different. A large amount of calcium was determined in Sludge L and S (12.2 and 20.0%, respectively), that got into the sludge during the neutralization of waste water with lime. Large amounts of manganese (0.364%), nickel (0.111%) and zinc (0.609%) were determined in Sludge U; meanwhile,

Fig. 10. TG and DSC curves for Sludge U
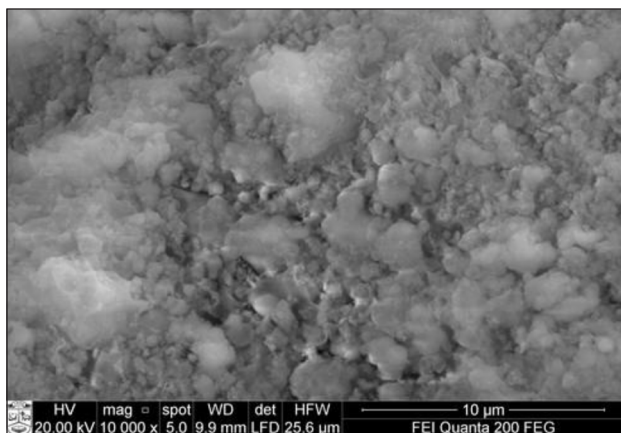
Fig. 12. Sludge U heated at 400 °C
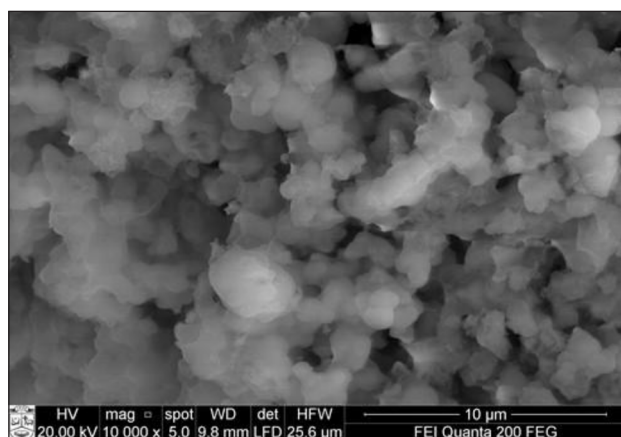
Fig. 13. Sludge U heated at 600 °C

the amounts of the metals specified hereinabove were under 0.1% in other two types of sludge. All types of sludge contained a similar amount of chromium: 0.127% in Sludge S, 0.146 in Sludge L and 0.161% in Sludge U. These data are presented in Table 3. Detectable concentrations of heavy metals did not leach from the blank ceramic samples, so the table does not contain any clay leaching data.

Table 3. **Amounts of metals contained in the sludge, %**

Sludge	L		S		U	
Metals	mg/kg	%	mg/kg	%	mg/kg	%
Al	460	0.046	2240	0.224	1520	0.152
Ca	122000	12.2	200000	20.0	40000	4.0
Cr	**1460**	**0.146**	**1270**	**0.127**	**1610**	**0.161**
Cu	74	0.0074	61	0.0061	15	0.0015
Fe	1850	0.185	132000	13.2	19300	1.93
K	15700	1.57	810	0.081	1630	0.163
Mn	**350**	**0.035**	**420**	**0.042**	**3640**	**0.364**
Na	5150	0.515	1240	0.124	5270	0.527
Ni	**17**	**0.0017**	**16**	**0.0016**	**1110**	**0.111**
Zn	**90**	**0.009**	**140**	**0.014**	**6090**	**0.609**
Total:	147151	14.7151	338197	33.8197	80185	8.0185

Studies of heavy metal leaching from sludge

Only leaching of Cr, Cu, Mn, Ni and Zn was evaluated. The leaching results are given in Tables 4 and 5.

Table 4. **Leaching amounts of heavy metals from the sludge, mg/kg**

	Sludge U				
	Cr	Cu	Mn	Ni	Zn
pH = 2.1	2.51	0.89	137.54	265.97	47.96
pH = 4	0.44	0.78	136.83	259.66	47.46
pH = 6.2–6.3	0.41	0.58	136.22	259.83	47.32
pH = 8.1	0.50	0.58	136.16	258.43	47.39
pH = 10	0.48	0.53	135.96	257.66	47.40
	Sludge L				
	Cr	Cu	Mn	Ni	Zn
pH = 2.1	b.d.l.	0.67	0.78	0.17	0.22
pH = 4	b.d.l.	0.67	0.68	0.20	0.02
pH = 6.2–6.3	b.d.l.	0.73	0.75	0.22	0.22
pH = 8.1	b.d.l.	0.64	0.77	0.27	0.18
pH = 10	b.d.l.	0.71	0.77	0.25	0.22
	Sludge S				
	Cr	Cu	Mn	Ni	Zn
pH = 2.1	b.d.l.	0.96	1.27	b.d.l.	0.33
pH = 4	b.d.l.	0.97	1.03	b.d.l.	0.20
pH = 6.2–6.3	b.d.l.	0.91	1.22	b.d.l.	0.15
pH = 8.1	b.d.l.	0.87	1.23	b.d.l.	0.15
pH = 10	b.d.l.	0.87	1.25	b.d.l.	0.11

b.d.l. = below detectable limit.

Table 5. **Leaching of heavy metals from the sludge expressed in percent (%) from the initial metal content in the sludge**

	Sludge U				
	Cr	Cu	Mn	Ni	Zn
pH = 2.1	0.16	5.93	3.78	23.96	0.79
pH = 4	0.03	5.20	3.76	23.39	0.78
pH = 6.2–6.3	0.03	3.87	3.74	23.41	0.78
pH = 8.1	0.03	3.87	3.74	23.28	0.78
pH = 10	0.03	3.53	3.74	23.21	0.78
	Sludge L				
	Cr	Cu	Mn	Ni	Zn
pH = 2.1	b.d.l.	0.15	0.22	1.00	0.24
pH = 4	b.d.l.	0.15	0.19	1.18	0.02
pH = 6.2–6.3	b.d.l.	0.16	0.21	1.29	0.24
pH = 8.1	b.d.l.	0.14	0.22	1.59	0.20
pH = 10	b.d.l.	0.15	0.22	1.47	0.24
	Sludge S				
	Cr	Cu	Mn	Ni	Zn
pH = 2.1	b.d.l.	0.04	0.30	b.d.l.	0.24
pH = 4	b.d.l.	0.04	0.25	b.d.l.	0.14
pH = 6.2–6.3	b.d.l.	0.04	0.29	b.d.l.	0.11
pH = 8.1	b.d.l.	0.04	0.29	b.d.l.	0.11
pH = 10	b.d.l.	0.04	0.29	b.d.l.	0.08

b.d.l. = below detectable limit.

The largest amounts of nickel (265.97–257.66 mg/kg or 23.96–23.21% from the initial amount) and manganese (137.54–135.96 mg/kg or 3.78–3.74% from the initial amount of metals) leached from Sludge U. The amounts of leached zinc were 47.96–47.40 mg/kg, which makes only 0.79–0.78% from the initial amount of metals. Meanwhile, the leaching of copper 0.89–0.53 mg/kg seems to be small, but it makes 5.93 up to 3.53% from the initial amount. High concentrations of manganese and nickel leached from Sludge U can be explained by the fact that these elements were not fully transferred to insoluble compounds during the neutralization of solutions.

The largest amounts of manganese Mn (0.78–0.68 mg/kg or 0.22–0.19% from the initial amount of metal) and copper Cu (0.73 0.64 mg/kg or 0.16–1.14% from the initial amount of metals) leached from Sludge L. The amounts of leached zinc Zn and nickel Ni are not large (0.22–0.02 and 0.27–0.17 mg/kg, respectively, which makes 0.24–0.02 and 1.59–1.000%, respectively, from the initial amount of metals. No detectable amount of chromium Cr leached from Sludge L.

No detectable amounts of chromium and nickel leached from Sludge S, and only small amounts of the following metals leached out: zinc 0.33–0.11 mg/kg or 0.236–0.079% from the initial amount of metals, and copper 0.96–0.87 mg/kg or 0.043–0.039% from the initial amount of metals. The largest amount of manganese leached from Sludge S is 1.27–1.03 mg/kg; however, this makes only 0.302–0.245% from the initial amount of metal.

The leaching of metals from sludge is very different; therefore, it can be assumed that metals are bound to different compounds in the sludge.

The metal leaching results from ceramic samples burned in all test modes (at 900, 1000, 1025 and 1050 °C, respectively) were below the detectable limit of the measuring device, which is 0.01 mg/l for all the tested metals.

The Wastewater Management Regulation [14] lays down the permissible concentrations of heavy metals in wastewater discharged to sewerage systems and natural environment. The maximum permissible concentrations of metals under our study and the obtained ceramic sample leaching results are

given in Table 6. It may be concluded that the tested metals were stabilized in ceramic sufficiently in our test conditions.

CONCLUSIONS

1. The structure of the phosphating sludge changes during the heating not only because of evaporation of some substances, but, as the inserted SEM images and DTG curves show, also because of crystallization processes in the sludge that need to be given a more detailed study.

2. The difference of heavy metals, such as Mn, Cu, Zn, Ni and Cr, leaching from different phosphating sludge is determined by the sources and processes of sludge formation; meanwhile, media pH is negligible in this case.

3. In all cases heavy metal leaching from the formed and heated ceramic samples (containing 5 and 10% sludge additives) were below 0.01 mg/l, and at the same time below the maximum permissible concentrations laid down in the Wastewater Management Regulation. This testifies good efficiency of immobilization of heavy metals contained in phosphating sludge into ceramic matrices.

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Table 6. **The maximum permissible concentration of hazardous substances from ceramic samples**

Metal	Maximum permissible concentration discharged into sewerage system, mg/l	Maximum permissible concentration discharged into natural environment, mg/l	Sludge U	Sludge L	Sludge S
Ni	0.5	0.2			
Cr (total)	2	0.5	The concentrations of leached metals are below detectable limits for each burned ceramic sample		
Cu	2	0.5			
Zn	3	0.4			
Mn	No limit is established	No limit is established			

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FOSFATAVIMO DUMBLAS: SAVYBĖS IR IMOBILIZACIJA KERAMIKOJE

S a n t r a u k a

Pramonės atliekų įvedimas į keraminę matricą yra viena iš sunkiųjų metalų stabilizavimo technologijų, leidžiančių gauti naudojimui tinkamus produktus ir / arba sumažinti sąvartyne šalinamus atliekų kiekius. Šiame darbe ištirtos fosfatavimo procese susidarančių atliekų – fosfatavimo dumblo – savybės ir jų stabilizavimo keraminėje matricoje galimybės. Atlikta fosfatavimo dumblo termogravimetrinė analizė, taip pat dumblo bandiniai atskirai iškaitinti 400, 600 ir 1 000 °C temperatūrose, ir kaitinimo produktams atlikta skenuojanti elektroninė mikroskopija. Rezultatai rodo, kad dumblo kaitinimo metu vyksta rekristalizacijos procesai.

Sunkiųjų metalų išplovimo testui atlikti molio pagrindu buvo suformuoti keraminiai bandiniai (dumblo kiekis juose sudarė atitinkamai 5 ir 10 %, išdegant juos 900–1 050 °C temperatūroje 1–4 val.). Išplovimo testo iš šių išdegtų bandinių rezultatai parodė, kad sunkiųjų metalų Mn, Cu, Zn, Ni ir Cr koncentracija eliuate yra mažesnė už aptikimo ribą, taip pat neviršija nuotekoms keliamų reikalavimų. Fosfatavimo dumblo stabilizavimas keramikoje pasiteisina kaip efektyvus sunkiųjų metalų imobilizacijos ir pavojingų atliekų nukenksminimo metodas.