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Microencapsulation of zinc plating waste using silicone polymers

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ABSTRACT

This paper deals with the treatment of hazardous zinc-bearing waste using hydraulic binders and silicone polymers, with the aim to allow its safe disposal into landfill. The waste was solidified using hydraulic binders in the first step and then encapsulated using silicone polymers. Samples were characterised using x-ray fluorescence, x-ray diffraction, and scanning electron microscopy. The effectiveness of the process was evaluated by leaching tests in distilled water and in an acidic environment according to Toxicity Characteristic Leaching Procedure. The effect of porosity and pH on the release of pollutants was also studied. Zinc and chloride were identified as the most significant pollutants in the waste. Portland cement did not stabilize them efficiently. The two-step treatment with Portland cement and silicone binders decreased, in the best case, the concentration of zinc and chloride in acidic extracts from 12,400 mg/L and 38,300 mg/L to 21.9 mg/L and 74 mg/L, respectively, and the treated waste complied with regulatory requirements for hazardous waste disposal into landfills. The two-step treatment was also found as a more effective method than microencapsulation using a silicone binder alone. The factor that most affects leachability appears to be the porosity of the encapsulated waste.

Keywords: Metal plating wastes; Zinc; Silicone polymers; Stabilisation/solidification; Leaching tests

1. INTRODUCTION

Industrial processes produce many types of waste, which could pose an elevated risk to the environment or human health. They are usually classified as hazardous due to the content of toxic components. The amount of industrial hazardous waste produced by 28 European Union members was 14,360,000 tonnes in 2018 (Eurostat, 2020; Camenzuli and Gore, 2013; EUROSTAT, 2010). One of the industrial processes generating hazardous waste is the metal surface plating industry producing waste contaminated with heavy metals such as zinc, chromium, or nickel. Zinc has been identified as one of the fifty-four material that is important to the EU's economy, and its consumption rate increased by 7% in the last five years (Ng et al., 2016). The galvanising process, in which zinc plays a vital role, produces many types of waste collected in different steel coating steps. Flux waste and zinc ash are waste, containing mainly zinc and zinc ammonium chloride, from the galvanising process, and they are treated to the recovery of metal zinc. Other types, which contain a smaller amount of zinc, are disposed on landfills. One of the most used waste treatments prior to landfill disposal is the stabilisation and solidification (S/S) method (Ng et al., 2016; Cobournestu, 2010).

S/S method is a process which converts a toxic waste into more physically and chemically stable form. It is achieved by the additions of various additives or binders to solidify the mixture. Moreover, it could produce materials which can be valuable and suitable for many other applications. The most used binders are ordinary Portland cement (OPC), activated fly ash and coal fluidised-bed combustion ash. It is also the technique used for dealing with contaminants such as metals, soluble salts and organic compounds (Rozumova et al., 2015; Silva et al., 2011). Furthermore, it has been reported that the use of cement binders reduces the leachability of metals such as Fe, Zn, Pb, Cu, Ni and Mn (Song et al., 2013; Malviya and Chaudhary, 2006).

In some cases, the treatment using common hydraulic binders may not be effective as predicted because the interactions between waste constituents and the binder particles impede the cement hydration process (Kalb, 2004). Zinc is known to slow this process because it interacts with the cement clinker grains during hydration. It is probably caused by the thin layer formation of zinc hydroxide or crystalline $Ca[Zn(OH)_3 \cdot H_2O]_2$ around anhydrous grains (Trezza, 2007; Stumm et al., 2005). Other pollutants that slow cement hydration process are chlorides because they exceed the cement system's binding capacity in high concentrations (Lago et al., 2017). They are chemically bound in compounds such as Friedel's salt; however, they can be released in the presence of sulphates (Galan and Glasser, 2015; Justnes, 1997). Thus, in many studies, the waste was pre-treated by leaching in distilled water before stabilisation/solidification technique (Colangelo et al., 2012; Nzihou and Sharrock, 2002). More specific techniques were cationic lignin and ettringite for stabilising waste containing chlorides (Gougar et al., 1996; Fink, 2017). It has also been reported that retarding effect on cement hydration can be caused by organic contaminants which form a protective layer around the cement grains (Pollard et al., 1991; Ge et al., 2020; Paria and Yuet, 2006; Zhan et al., 2020).

Some recent studies on S/S of zinc-containing waste (Wang et al., 2020; Zhao et al., 2021; Souza Barreto et al., 2020) introduced the usage of red mud, rock solid waste or geopolymers to reduce zinc leachability. Various toxic metal immobilisation mechanisms are applied, such as adsorption, physical encapsulation, or chemical fixation. As with the use of Portland cement, zinc retention is relatively high; however, the residual leachability of zinc is still in the order of tens or hundreds of mg/L, which in some countries would not meet the regulatory limits for landfill disposal. The solubility of many toxic elements depends on pH, which is one of the mechanisms, which S/S technique uses. Thus, metal ions are transformed from soluble form to less or practically insoluble form by changing the pH of solution (Hamilton and Sammes, 1999). From the view of the leachability zinc ions are very leachable at $pH < 8$ because they are presented as Zn^{2+} and at

pH > 11 are presented as zincate in a solution (Hamilton and Sammes, 1999). Zinc ions form low soluble hydroxides between pH values from 8 to 11 (Hamilton and Sammes, 1999; Damir et al., 2012). Conversely, chlorides leachability was found independent on the pH of the solution in a static extraction test by the Quina et al. (2009). Moreover, the presence of salts in a solution could increase the leachability of metal ions (van der Sloot and Dijkstra, 2004; Ding et al., 2013).

Another possibility of reducing the toxicity of hazardous wastes is to encapsulate them using hydrophobic thermoplastic binders (e.g. bitumen or elementary sulphur), which is a well-known technique (Chang, 2001). The advantages of silicone polymers used in this study as the binder for encapsulation of waste are their better thermal resistance long term-resistance to weather conditions, chemical inertness, and resistance to most chemicals (Labouriau et al., 2015; Miller et al., 2000; Yilgor and Yilgor, 2014; Colas, 2005; Sastri, 2014). On the other hand, some silicone polymers are known to degrade rapidly in dry soil and undergo UV degradation and are susceptible to hydrolysis (Lehmann et al., 2000; Kumagai and Yoshimura, 1999). Therefore, silicone polymers with high mechanical and weather resistance, designed for outdoor use should be chosen. The silicone polymers are prepared by the polycondensation or polyaddition reaction and usually are produced as liquids or in a pasty consistency. A final product (elastomer or resin) is then obtained after chemical crosslinking of polymeric chains, which can be achieved either using a catalyst, higher temperature or mixing a two-component blend. The crosslinking reaction using a catalyst is generally described by the scheme (1).

$$\sim Si{-}H + \sim Si{-}OH + \text{catalyst} \rightarrow \sim Si{-}O{-}Si\sim + H_2\ (\text{gas}) \qquad (1)$$

When these components are mixed together, the formation of Si–O– Si linkage, as the essence of silicone binder curing process, take place and hydrogen gas is released (Miller et al., 2000; Colas, 2005). The possible use of polysiloxanes was studied by Miller et al. (2000) who has reported the capability of two commercial silicone products (RTV 664 and ELECTROGUARD 2100) to encapsulate the mixed waste mainly consist of salts and chromium. Their results showed that the chromium concentration in leachate exceeded the Universal Treatment Standards (UTS) when RTV 664 was used, whereas the second silicone binder showed promising results because complied with the required limits for Toxicity Characteristic Leaching Procedure (TCLP) (Miller et al., 2000). Another study successfully tested ceramic silicone foam (CSF) on chromium-contaminated waste containing nitrate salts. The chromium concentration was at about 1 g/L, and the samples were successfully stabilised with waste loadings as high as 50% by mass (DOE, 1999).

The aim of this study is to evaluate the effectiveness of the S/S treatment of residual waste using different, commercially available silicone polymers. These commercial products are used for toxic waste microencapsulation. The effectiveness of the process is evaluated by leaching test in distilled water according to EN 12457-4:2002 and by US EPA TCLP 1311 (EPA, 1992). The studied parameters are zinc concentration, the concentration of chlorides and concentration of total dissolved solids (TDS). These values are then compared with the limits for landfill disposal set by EU council decision 2003/33/EC and by UTS set by Federal Code of Regulations (Title 36 Code of Federal Regulations, 2020).

2. EXPERIMENTAL

2.1. Waste

The sample of the waste was obtained from a factory near Valencia in Spain. It was collected at the end of the galvanising process in which steel pieces gain a thin layer of metal (e.g. zinc) for protection against corrosion. The location of the waste collection was under the bag filters, which are automatically cleaned by a periodic blast of compressed air (Cobournestu, 2010). The annual production of this type of waste is 6678 kg, and it mainly consists of zinc ammonium chloride (Valenciana and Mvlat, 2010). According to the European Waste Catalogue (EWC) (EPA, 2002), this waste should be classified as hazardous with the code 11 05 03, solid waste from gas treatment.

2.2. Silicone polymers

Silicone polymers used in this study were obtained as samples of commercial products with different composition and characteristics as can be seen in Table 1.

N1522 – a two-component silicone rubber produced in the factory Lucebni zavody a.s. Kolin in the Czech Republic. It is cured by condensation reaction and needs to be mixed with a catalyst to be cured thorough entire mass within a few hours at ambient temperature.

RTV 20 – a condensation silicone rubber produced by Lianhuan Group Limited, Shenzhen, China.

ESSIL 291/292 – a transparent two-component addition-type silicone resin from Sika AG, Switzerland.

GMS 2628 – a two-component addition silicone resin from Dawex chemical, Czech Republic.

MM730FG – food-grade silicone moulding rubber from ACC Silicones Ltd., United Kingdom.

Table 1
Basic characterization of silicone polymers according to the product sheets.

Characteristics	N 1552	RTV 20	ESSIL 291/ 292	GMS 2628	MM730FG
Viscosity (Pa.s)	10–15	15	42	5	15
Density (kg/m^3)	1250	1090	1080	1150	1250
Hardness (°ShA)	53–55	20–22	38	26–28	30
Tensile strength (MPa)	3.4–3.6	> 2.8	5	4–5	4.4

2.3. Hydraulic binders

Ordinary Portland Cement (OPC) II/B-S was obtained from the company CEMMAC Inc. (Horn´e Srnie, Slovakia). Ash from fluidised-bed combustion of coal (FBCA) was obtained from the heating plant of the city of Zlín (Czech Republic), and its properties were described earlier Table 1 (Vinter et al., 2016).

2.4. Waste encapsulation using silicone polymers

Liquid silicone rubber was stirred shortly, or in the case of twocomponent binders (ESSIL 291/292 and GMS 2628), the two components were mixed in the recommended ratio. Then, the waste in an amount ranging from 10% to 70% of the total weight was mixed into the prepared silicone binder and homogenised for 5 min by a glass rod in a plastic vessel. In the case of silicones N 1552 and RTV 20, the catalyst at the recommended dosage (1–1.5 wt% according to product sheets) was added, and the mixture was mixed for an additional 5 min. The mixtures were then degassed using a water vacuum pump for ten minutes at the pressure 1 kPa and left in closed cylindric plastic moulds (diameter 28 mm, height 69 mm) for 72 h to solidify before testing.

2.5. Two-step treatment of zinc waste

As a first step, the waste was mixed with hydraulic binder and water according to the procedure optimised in our previous study (Vinter et al., 2016), and left in closed moulds to solidify and harden for 28 days. The content of waste was 62 or 50% of total mixture weight for mixtures with OPC (marked as samples A and B), and 33% for the mixture with FBCA (sample C). As a second step, solidified waste specimens were crushed down to the particle size < 4 mm and encapsulated using a silicone polymer, similarly as described in Section 2.3. The mixing ratio of silicones and OPC/FBCA pre-treated waste was 1:1 by weight, so the contents of silicone polymers in the final specimens were 50% of the total mass. All samples were prepared and tested in triplicates. For a shorter reference, the samples were marked as P1-P10 in the chapter Results and discussion. Different approaches used in this study for treating the waste are shown in Fig. 1. The red line indicates stabilisation/solidification using hydraulic binders, the blue line indicates microencapsulation using silicone polymers, and the grey line indicates two-step treatment of zinc waste using both types of binders together. Furthermore, a series of blank samples without zinc waste was prepared. The procedures of blank samples preparation were identical as preparation of solidified waste samples, except the pure silica sand was used instead of waste.

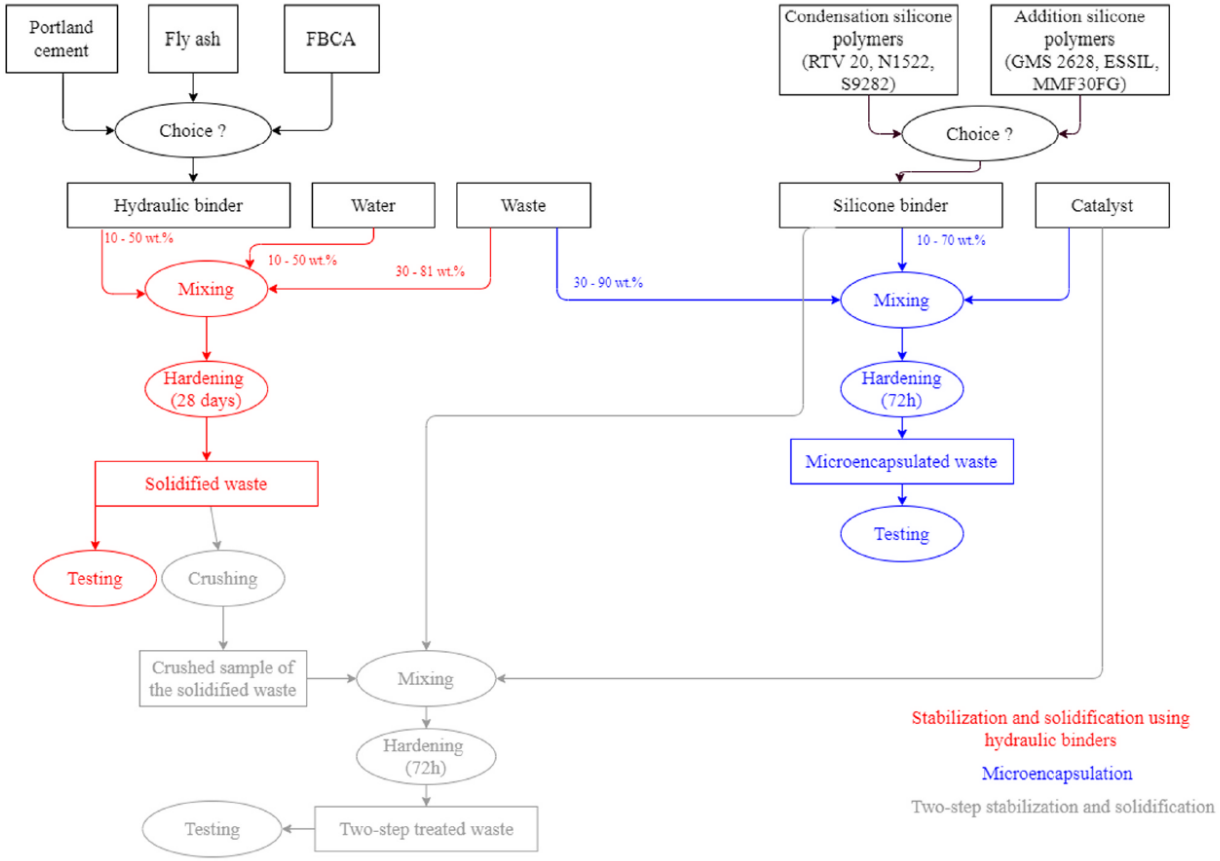

Fig. 1. A block scheme of S/S approaches used.

2.6. Acid digestion

To determine the total content of zinc, plating waste was dissolved using the acid digestion in sulphuric acid. The process was performed in the following way: 5 g of sample was weighed and put into a beaker with 60 ml of 0.5 M H_2SO_4. The mixture was stirred for 15 min. The liquid phase was then filtered through a 0.45-micron filter and subjected to the chemical analysis.

2.7. Leaching tests

Leaching tests were based on European standard EN 12457-4:2002 (Kumagai and Yoshimura, 1999). Samples were extracted using distilled water (or an extraction liquid with the pH set to selected value) at the liquid/solid weight ratio 10:1 on a vibrating shaker at the shaking frequency 150 rpm for 24 h. The liquid phase was then filtered through a 0.45-micron filter and subjected to chemical analysis. The limit concentrations of pollutant defined by European regulations (European Commision, 2003) were used as the reference values for the assessment.

2.8. Toxicity characteristic leaching procedure

The untreated waste and selected solidified samples were also evaluated using Toxicity Characteristic Leaching Procedure (US EPA Method 1311 (EPA, 1992)). Extraction fluid # 2 (acetic acid) with pH 2.88 ± 0.05, liquid/solid ratio 20:1 and extraction time 18 h were used. Universal Treatment Standards (UTS) (US EPA, 2016) were applied as the reference values for pollutant concentrations.

2.9. Analytical methods

Total dissolved solids (TDS) were determined according to the standard method ASTM D5907-13 (ASTM, 2018). A leachate sample of volume of 10 ml was dried-out at 105 °C to the constant weight, and the concentration of TDS was calculated. The concentrations of heavy metals were determined using GBC 933A atomic absorption spectrometer with the air-acetylene flame (GBC 933 AA, GBC Scientific Equipment Pty. Ltd., Australia). Chloride was determined by the argentometric titration with potassium chromate as the indicator. The ammonium was determined using the alkalimetric formaldehyde titration method. The pH value was measured using the InoLab 730 pH meter equipped with the Sentix 81 glass-electrode (WTW, Germany). X-ray diffraction (XRD) analysis was carried out on powdered samples using PANalytical Model X' Pert PRO MPD with CuKα radiation source, generated at 40 kV and 40 mA, 2θ range from 5° to 90°. Crystalline phases were identified according to the International Centre of Diffraction Data PDF-2. The elementary composition of solids was analysed using an energy-dispersive X-ray fluorescence (XRF) spectrometer ElvaX (Elvatech Ltd., Ukraine) equipped with an Rh X-ray tube. The instrument settings were as follows: voltage on X-ray tube 10 kV, current 64 μA, and the spectrum acquiring time 100 s. Scanning electron microscope (SEM) analysis was carried out using a Phenom Pro microscope (Phenom World, United Kingdom) equipped with mini sputter coater SC7620 (Quorum, United Kingdom). The samples were coated with a mixture of gold and palladium, and then the specimens were analysed at 10 kV.

2.10. The compressive strength measurement

The compressive strength (CS) of tested solids was measured after 28 days of curing according to standard EN 196-1:2005 (EN 196-1, 2016) using a universal laboratory press BSML 21 (Brio Hranice, Czech Republic). The tested solids were of the cylinder shape with a diameter of 27 mm and height from 57 to 63 mm. The diameter values of each test specimen were computed as the averages of 10 measures with precision ± 0.1 mm.

2.11. Determination of open porosity

The percentage of open pores by volume in the solidified samples were determined on the basis of mass water saturation measurements according to standard method EN 1936:2007 (EN 1936, 2007) using Eq. (2).

$$\rho_0 = \frac{m_s - m_d}{\rho_L \times V_S} \times 100(\%vol.) \quad (2)$$

Where ρ_0 – open porosity as a ratio of pore volume and apparent volume of specimen m_s – weight of saturated specimen (g) m_d – weight of dry specimen (g) V_s – a bulk volume of the cylindrical specimen (cm^3) ρ_L – density of the liquid used for pore saturation (g/cm^3).

3. RESULTS AND DISCUSSION

3.1. Waste characterisation

The waste was a fine light grey powder with apparent density 0.80 ± 0.03 g cm^{-3}, particle density of 2.10 ± 0.36 g cm^{-3} and dry mass of 98.4 ± 0.3%. The dry sieve analysis (see Fig. 2) showed the most considerable fraction (35.6 ± 0.5%) of particles retained on mesh sieve size 0.2 mm. The content of coarse-grained particles with size > 1 mm was 21.7 ± 0.4%. The SEM image of the waste sample (Fig. 3) revealed small particles ranging in size from units to tens of microns, apparently crystalline with sharp edges, aggregated into larger clusters. The XRD analysis (Fig. 4) showed that the most dominant crystalline compounds were zinc-ammonium chlorides and zinc oxide. A quantitative analysis of the digested waste sample resulted in composition as follows: chlorides 38.3 ± 0.5 wt%, zinc 27.5 ± 0.7 wt% and ammonium ions 14.9 ± 0.5 wt%. Those values correspond to an empiric formula $(NH_4)_{1.95}ZnCl_{2.55}$, which is in a fair agreement with the results of XRD analysis. According to the spectrum obtained by the XRF measurement (Fig. 5), the untreated waste sample contained, besides Zn and Cl as the major compounds, also Ca, Fe and Al in amounts estimated on the basis of fundamental parameters calculation to 1.8 ± 0.2, 0.59 ± 0.03 and < 1.25 wt%, respectively. Trace elements detected by the XRF methods were P, S, Mn and Pb, estimated to < 0.25, 0.17 ± 0.09, 0.08 ± 0.02 and 0.03 ± 0.01 wt%, respectively.

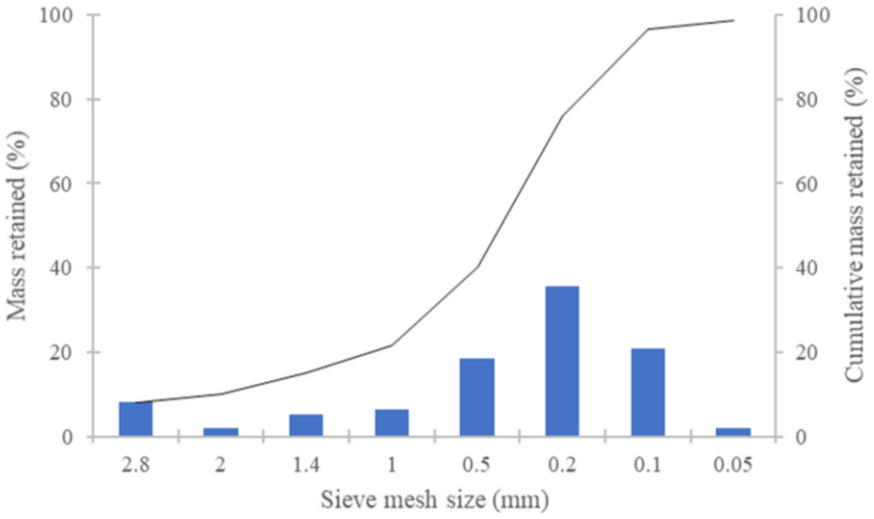

Fig. 2. Dry sieve analysis of the untreated waste sample.

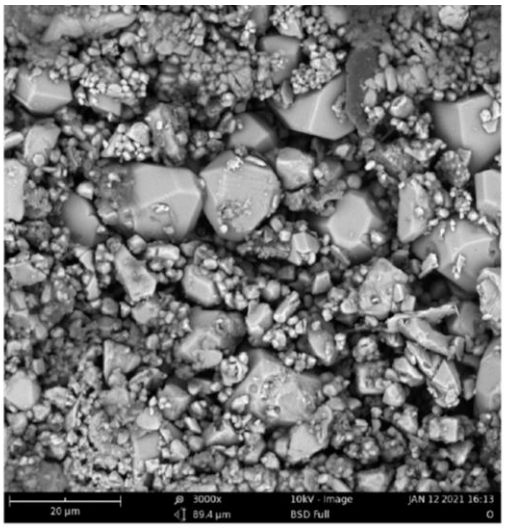

Fig. 3. SEM image of the untreated waste sample.

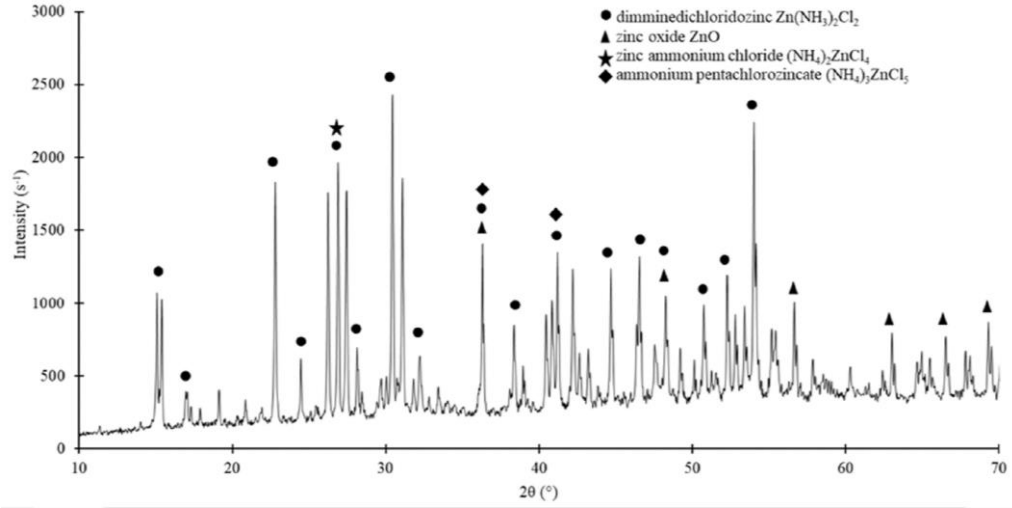

Fig. 4. XRD pattern of the untreated waste sample.

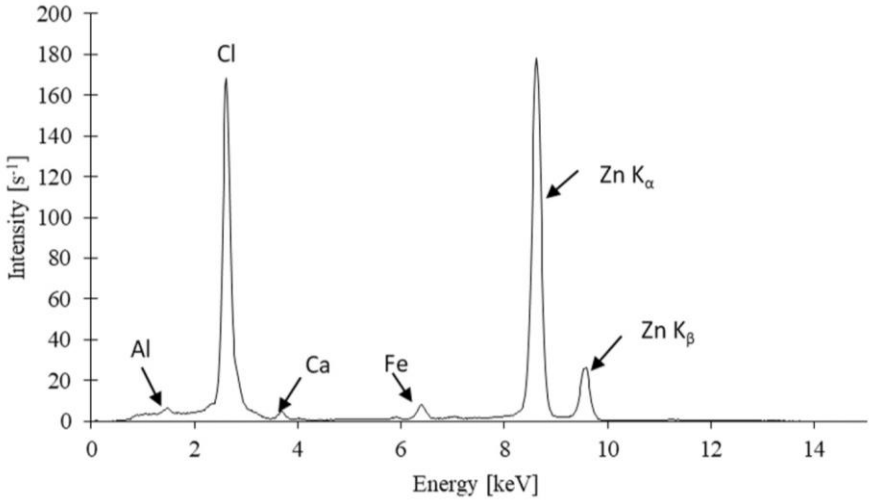

Fig. 5. XRF spectrum of the untreated waste sample.

3.2. Leaching tests of untreated waste

The results of leaching tests of the untreated waste sample both in distilled water and acetic acid solution are shown in Table 2, where the highlighted values did not meet the regulatory levels. Even if the waste was extracted in distilled water, the final pH value was below 7, which is obviously caused by the slightly acidic nature of ammonium chloride salts. The total zinc content determined using acid digestion of the waste sample was 2.75×10^5 mg/kg; however, the amounts of zinc extracted in both leaching tests were lower. The leaching test in distilled water released only 16.7% (4.6×10^4 mg/kg) of total zinc, in the case of TCLP, it was 45.1% (1.24×10^5 mg/kg). Chloride ions were leached in higher efficiency - 62.1% and 99.7% in distilled water and TCLP tests, respectively. The concentration of total dissolved solids was another parameter, of which value did not comply with the limits for the waste acceptance to a hazardous waste landfill according to the council decision 2003/33/EC (European Commision, 2003). In addition, the TCLP test found an unsatisfactory concentration of lead. All other parameters met the regulatory criteria, so the concentrations of Zn, Cl, TDS and Pb were selected as the critical parameters for an efficient assessment of the waste treatment process.

Table 2
Determined values in the water and acetic acid leachate of the waste.

Parameter	Unit	EN 12457-4	TCLP 1311
pH	–	6.5 ± 0.1	6.3 ± 0.1
TDS	mg/L	$\mathbf{(39.7 \pm 1.3) \times 10^3}$	NM*
Cl^-	mg/L	$\mathbf{(23.8 \pm 0.9) \times 10^3}$	$\mathbf{(38.3 \pm 0.7) \times 10^3}$
SO_4^{2-}	mg/L	284 ± 3	NM*
F^-	mg/L	< 0.01	< 0.01
Cr	mg/L	< 0.01	0.71 ± 0.05
Ba	mg/L	< 0.01	< 0.01
Zn	mg/L	$\mathbf{(4.6 \pm 0.2) \times 10^3}$	$\mathbf{(12.4 \pm 0.3) \times 10^3}$
Ni	mg/L	0.21 ± 0.03	0.16 ± 0.03
Cu	mg/L	0.13 ± 0.01	0.62 ± 0.01
Hg	μg/L	1.60 ± 0.07	2.31 ± 0.09
As	mg/L	< 0.01	< 0.01
Se	mg/L	0.33 ± 0.14	0.29 ± 0.09
Sb	mg/L	< 0.01	< 0.01
Pb	mg/L	2.50 ± 0.03	$\mathbf{7.35 \pm 0.09}$
Cd	mg/L	0.22 ± 0.01	0.20 ± 0.01
Mo	mg/L	< 0.01	< 0.01

NM* – determination of those parameters is not mandatory in TCLP.
The highlighted values did not meet the regulatory levels.

3.3. Characterisation of waste pre-treated with hydraulic binders

In our previous study (Vinter et al., 2016), a procedure for zinc-bearing waste treatment using cementation was proposed; however, that procedure did not comply with the regulatory levels for land disposal. Therefore, the best three recipes from that study were selected as a basis for a combined cement/silicone treatment. An overview of prepared pre-treated waste mixtures together with the results of leaching tests and physical-mechanical properties measurement after 28 days of curing can be seen in Table 3. Compared with the untreated waste, the zinc and lead concentrations dropped significantly, whereas the chlorides and TDS remain at high levels. However, in the TCLP of these mixtures, the concentrations of zinc were an order of magnitude higher than in aqueous extracts, and therefore the treatment of waste with cementitious binders can be assessed as successful only in terms of lead immobilization. The highest value of compressive strength (CS) and the lowest value of porosity were measured for sample B, which correlate with the lowest lead leachability in TCLP.

Table 3
Basic characterization of the mixtures used for the two-step treatment of zinc waste.

Sample	A	B	C	A	B	C
Extraction method	EN 12457-4			TCLP 1311		
W-B-W ratio	2:1:1	5:3:2	1:1:1	2:1:1	5:3:2	1:1:1
pH	8.7	7.9	8.0	6.5	6.6	6.6
c_{Zn} (mg/L)	379 ± 21	366 ± 34	204 ± 59	2188 ± 90	1812 ± 90	3012 ± 120
c_{Pb} (mg/L)	< 0.01	< 0.01	< 0.01	0.03 ± 0.01	< 0.01	0.22 ± 0.03
c_{Cl} (mg/L)	12,600 ± 1130	14,100 ± 2613	9200 ± 475	9924 ± 500	9037 ± 375	8067 ± 563
c_{TDS} (mg/L)	29,400 ± 1633	34,400 ± 1853	28,200 ± 1048	×	×	×
	Physical-mechanical properties					
CS (MPa)	0.16 ± 0.05	1.71 ± 0.20	< 0.1			
ρ_0 (vol%)	16.6 ± 2.8	6.0 ± 1.8	45.3 ± 7.8			

W-B-W = waste-binder-water ratio.

The SEM images of pre-treated samples are shown in Figs. 6–8. Samples treated with OPC show an almost continuous but clearly porous structure encapsulating the waste particles. In sample B, where a higher dosage of OPC was used, plate-shaped crystals of Portlandite were also observed. On the other hand, the sample treated with FBCA shows predominantly separated grains, which corresponds to a lower measured value of compressive strength and higher porosity.

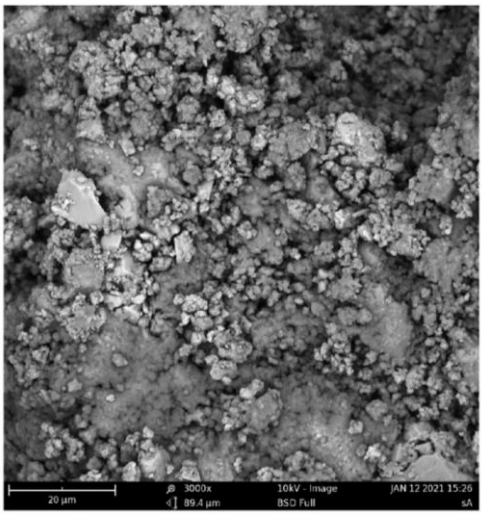

Fig. 6. SEM image of the waste pre-treated with OPC (sample A).

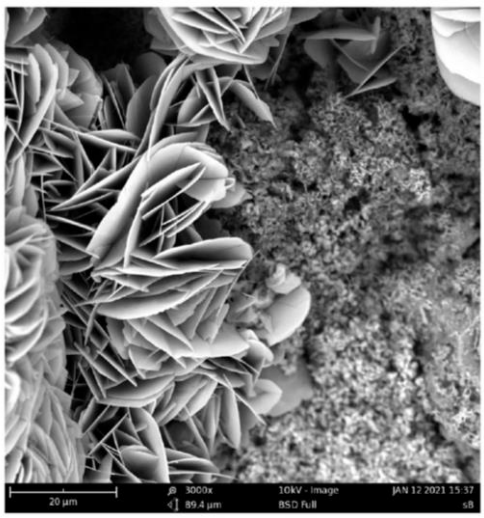

Fig. 7. SEM image of the waste pre-treated with OPC (sample B).

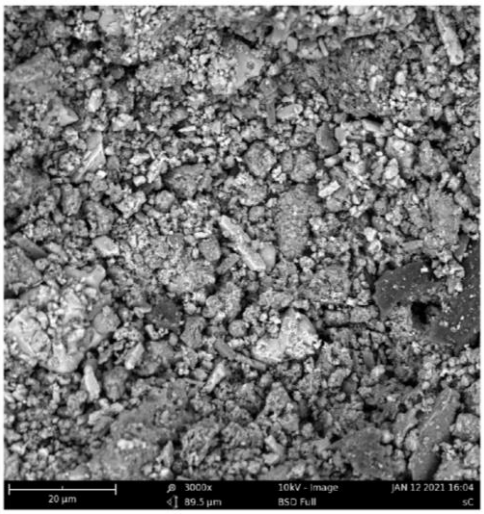

Fig. 8. SEM image of the waste pre-treated with FBCA (sample C).

The pre-treated samples A, B and C were crushed and then analysed using XRD technique. As shown in Fig. 9, the XRD analysis of the pretreated waste sample showed that the dominant crystalline phases were the same as in the untreated waste. In the case of FBCA binder (sample C), an additional band of calcium sulphate ($CaSO_4$) was detected, that agree with expected FBCA composition, as it should contain flue-gas desulphurisation products (Vondruska et al., 2001). On the other hand, there are missing bands at $2\theta = 37°$ in the treated waste sample XRD patterns compared to the untreated waste. It could be caused by chemical reactions of calcium oxide contained in binders with zinc-ammonium chloride in the pre-treatment procedure. A release of ammonia was also observed by smell during the mixing waste with hydraulic binders. This unwanted phenomenon should be easily solved by means of air cleaning, or even generated ammonia could be isolated and utilised; however, it was not the subject of this study.

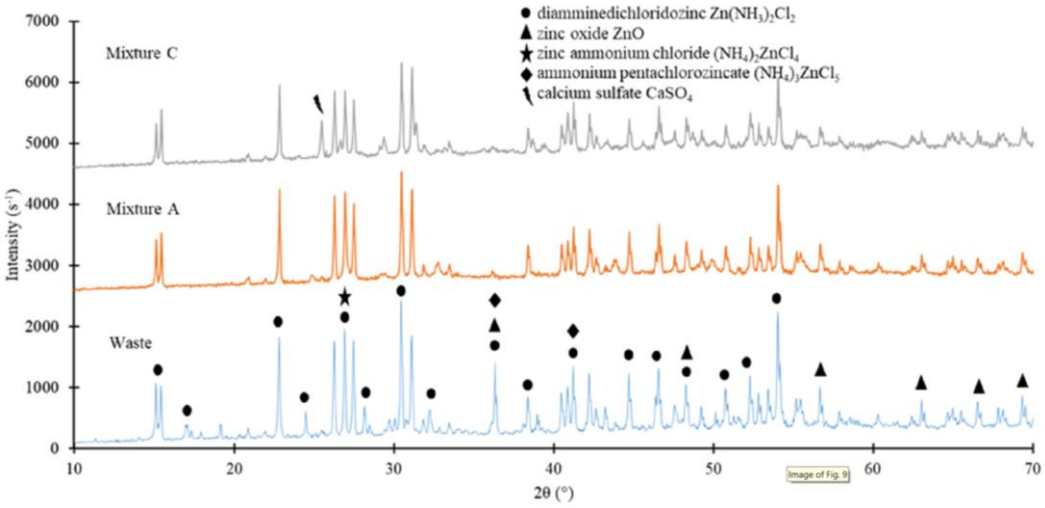

Fig. 9. XRD patterns of the pre-treated samples A and C in comparison with the untreated waste.

3.4. Evaluation of blanks samples

The results of blank samples are summarised in Table 4. As can be seen from the table, zinc was released from some silicone binders up to concentration 15 mg/L in the case of GMS2628. It is probably due to zinc oxide pigments used in those silicone binders. However, all observed values in leaching test of blank samples met the regulatory limits for landfills. The values of compressive strength for silicone blanks could not be measured due to the elastic properties of the silicone rubber. The highest value of compressive strength was measured for the blank sample with Portland cement corresponding to pre-treated sample B, which also correlate with the best S/S efficiency of that sample. The results emerged that the most porous blank samples were those which were prepared using OPC as a binder. Nevertheless, the lowest porosity in the set of hydraulic binder blanks was found in sample OPC B, which is in a good agreement with the best value of compressive strength. The blank silicone samples showed low values of porosity around 2%.

Table 4
Measured parameters of blank samples.

Binder	pH (1)	c_{Zn} (mg/L)	c_{Cl}^- (mg/L)	c_{TDS} (mg/L)	CS (MPa)	ρ_0 (vol%)
OPC A	12.2	0.329 ± 0.019	44.7 ± 3.1	3.53 ± 0.22	6.2 ± 0.5	39.1 ± 2.0
OPC B	12.3	0.305 ± 0.021	41.2 ± 2.9	2.98 ± 0.18	15.7 ± 0.8	28.6 ± 2.3
FBC-A	12.1	0.064 ± 0.010	89.5 ± 6.3	5.18 ± 0.32	1.6 ± 0.8	38.3 ± 6.3
N1522	7.6	1.89 ± 0.08	< 0.01	0.02 ± 0.01	×	1.87 ± 0.69
RTV20	7.1	2.94 ± 0.12	< 0.01	0.01 ± 0.005	×	1.53 ± 0.41
GMS2628	8.2	15 ± 0.6	< 0.01	0.2 ± 0.02	×	1.44 ± 0.47
ESSIL 291/292	7.1	< 0.01	< 0.01	0.05 ± 0.01	×	1.88 ± 0.25
MM730FG	4.3	< 0.01	< 0.01	0.02 ± 0.01	×	2.13 ± 0.29

c – concentrations, CS – compressive strength, ρ_0 – open porosity, × – not measured.

3.5. Encapsulation using silicone polymers

In total, 35 solidified samples were prepared with different waste loadings and different silicone binders. It was determined that the zinc concentration, shown in Fig. 10, decreased sharply at the binder content around 30 wt%, that was probably caused by the formation of the monolithic specimen in which waste particles were encapsulated, and the most of open pores were filled with the silicone binder. The zinc concentration range in the leachates of solidified waste varied from 40 to approximately 5000 mg/L. The best results were obtained for samples containing the silicone binder RTV20 or GMS 2628, where the lowest measured values of zinc concentration were 40 mg/L and 110 mg/L, respectively, that were nearly an order of magnitude less in comparison with the results of waste treatment using hydraulic binders (see Table 3). Despite the fact the zinc concentrations decreased with an increasing amount of the silicone binder at all samples, no sample has met the class III limit value for land disposal, which is set to 20 mg/L (European Commision, 2003). There were observed no significant differences between the condensation (RTV20, N1522) and addition (GMS2628, ESSIL, MM730FG) type silicone binders at low binder contents. At the binder content at least 40%, the zinc immobilisation was clearly higher in the case of RTV20, compared to all other binders.

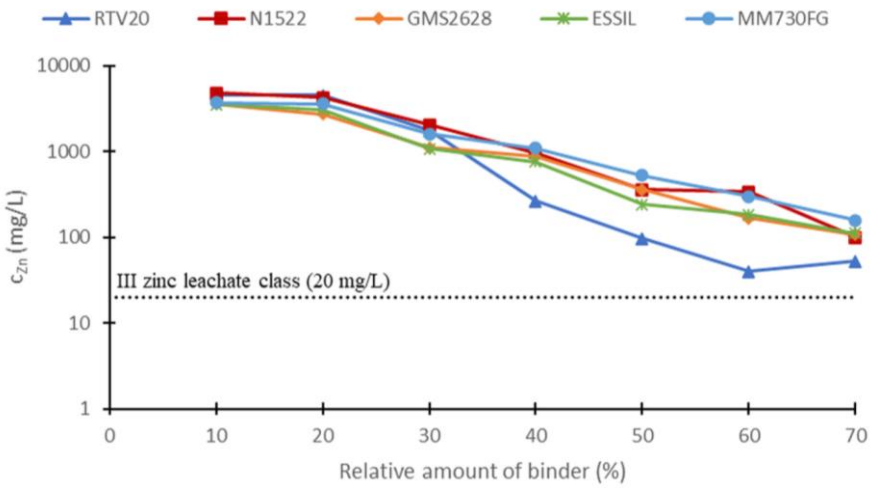

Fig. 10. The dependency of zinc concentration in the leachate of encapsulated waste on the silicone binder content (logarithmic scale).

The concentration of chloride ions, as can be seen in Fig. 11, decreased approximately from 29 g/L to 280 mg/L, depending on the silicone binder content. All silicone binders showed similar trends, except the case of RTV20, that seemed to be more effective than other silicones at the binder contents over 30%, as was already observed for zinc concentration. Samples of waste encapsulated using N1522, compared with other silicone binders, showed a significantly lower value of the chloride concentration at the initial binder loading; however, the class III limit value for the chloride concentration (European Commision, 2003) was met at the binder contents 35–50%, depending on the binder used, with the best value for RTV20. The lowest concentration of chlorides, of 283 mg/L, was found for the sample containing 70 wt% of the RTV20 binder. As a result, all samples with more than 50 wt% of a silicone binder loading could be considered sufficiently productive from the viewpoint of immobilisation of chlorides. A similar observation was also reported by Miller et al., which treated waste containing a high amount of salts using vinyl-methyl-polysiloxane (Duirk and Miller, 2002). Thus, the results indicated that silicone polymers were capable of chloride immobilisation with higher efficiency than the hydraulic binders.

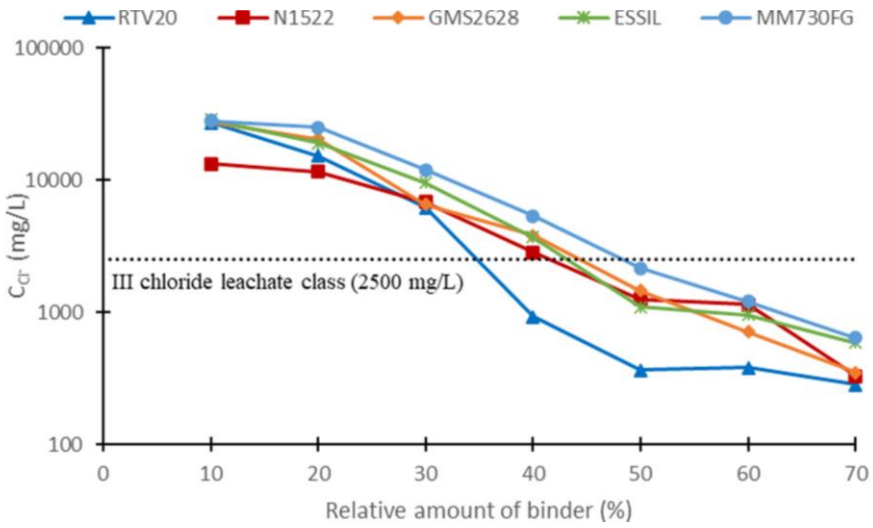

Fig. 11. The dependency of chloride concentration in the leachate of encapsulated waste on the silicone binder content (logarithmic scale).

As shown in Fig. 12, the concentration of total dissolved solids (TDS) decreased from 37 g/L to 280 mg/L. A marginal decrease was observed up to 20 wt% of the binder dosage for all silicone binders. The lowest TDS concentration was determined at 70 wt% silicone loadings, and all samples with 30 wt% or more of the binder met the class III limit value for TDS (European Commision, 2003). Observed trends in TDS concentrations were very similar to zinc and chloride concentrations plots, including a slightly different behaviour of RTV20 binder.

Fig. 12. The dependency of TDS concentration on the binder addition in the leachate using different silicone polymers.

To conclude, the silicone binder RTV20 showed the best efficiency for waste encapsulation using a one-step treatment procedure. The observed values of pollutant immobilisation were significantly higher than the waste treatment using hydraulic binders, reaching over 97%. Nevertheless, the treated waste did not meet the zinc concentration regulatory level for landfill disposal. Therefore, in the next stage, a two-step treatment of the waste was studied.

3.6. Solidification using two-step treatment

The purpose of the two-step treatment was to improve the process of the zinc-containing waste stabilisation/solidification, which was effective in case of a single binder usage, but not sufficient in terms of regulatory limits. Table 5 presents the evaluation results of 10 samples of waste treated by the two-step procedure with different binders. Parameters that did not meet a class III regulatory limit are underlined, and the highlighted lines represent samples that complied with all limit values. The sample P9 was found as the best one with the zinc concentration in the leachate 0.5 mg/L, whereas the waste treated only by OPC showed value 379 mg/L. Other experimental results of test solids determined that most silicone test solids prepared using the addition of silicone polymers performed well under the given set of experimental conditions. All studied parameters were below the III leachate class limit criteria for land disposal. However, the test solids prepared from the condensation type of silicone polymers were sufficiently treated in only two cases. Five samples met all tested limit values, which means that such a treated waste could be disposed of in a landfill. However, the samples that showed the concentration of chloride ions above the limit could also be considered an effective treatment because they met the limit criteria for the concentration of dissolved solids. It was also found that the addition of silicone polymers (ESSIL and GMS) was for the purpose of two-step solidification a more suitable than those of condensation type (RTV20 and N1522).

Table 5
The nomenclature of samples used in a two-step treatment and the results of studied parameters.

Sample	Pretreatment / first binder	Second binder	pH	c_{Zn} (mg/L)	c_{Cl^-} (mg/L)	c_{TDS} (mg/L)	ρ_0 (%)
P1	C / FBCA	RTV20	8.6	12.0 ± 1.6	3660 ± 180	6140 ± 492	×
P2	C / FBCA	N1522	7.8	36.0 ± 3.2	1820 ± 165	2210 ± 154	×
P3	**C / FBCA**	**ESSIL**	**8.2**	**2.00 ± 0.28**	**1490 ± 142**	**1910 ± 92**	**1.91 ± 0.35**
P4	C / FBCA	GMS	8.1	4.84 ± 0.39	4030 ± 208	4550 ± 215	×
P5	A / OPC	RTV20	8.6	76.0 ± 7.5	630 ± 49	800 ± 50	×
P6	A / OPC	N1522	8.3	78.0 ± 6.7	4200 ± 401	7100 ± 542	×
P7	**A / OPC**	**ESSIL**	**8.1**	**3.87 ± 0.52**	**2080 ± 203**	**2020 ± 173**	**1.83 ± 0.26**
P8	**B / OPC**	**ESSIL**	**8.0**	**4.67 ± 0.72**	**840 ± 42**	**1410 ± 90**	**1.63 ± 0.43**
P9	**A / OPC**	**GMS**	**8.7**	**0.50 ± 0.05**	**350 ± 27**	**370 ± 24**	**2.11 ± 0.62**
P10	**B / OPC**	**GMS**	**8.6**	**0.57 ± 0.04**	**490 ± 38**	**620 ± 30**	**1.59 ± 0.75**

c – concentrations, ρ_0 – open porosity, × – not measured.
The highlighted lines represent samples that complied with all limit values.

For comparison, the SEM image of the cut of the sample P8 (OPC + ESSIL) is shown in Fig. 13. Grains coated with a continuous phase of silicone polymer are clearly visible. The pores are insulated, preventing easy penetration of water into the matrix, and ensuring effective encapsulation. As a result, the silicone polymers showed the ability to form a secondary barrier surrounding the solidified particles of the zinc waste pre-treated with common hydraulic binders. The immobilisation of observed pollutants in the two-step treatment procedure could be rated as effective in the case of waste pre-treatment with OPC and encapsulation with GMS silicone binder. The encapsulation of waste pretreated with FBCA was rated as less effective, despite the low zinc concentrations, because the chloride ions were leached out. The incorporation of waste into a silicone polymer matrix was also evaluated by porosity measurement. The results demonstrated that open porosity values are very low and close to the values of blanks, which confirmed the quality of the encapsulation process. The low porosity and continuous layer of encapsulation silicone binder were found as the main mechanisms for decreasing the leachability of pollutants contained. Above mentioned results demonstrated that silicone binders can encapsulate zinc-containing waste effectively.

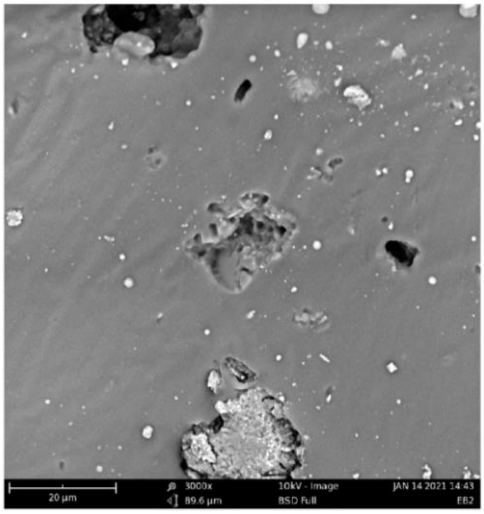

Fig. 13. SEM image of a sample cut of waste treated with OPC and silicone binder (sample P8).

3.7. Evaluation using the TCLP procedure

The results of the TCLP leaching test of selected samples are shown in Table 6. As can be seen, the change of pH value of the extraction liquid significantly increased the zinc concentration in the leachate compared to leaching test in distilled water. A correlation between pH value at the end of the leaching test with the concentration of both zinc and chlorides indicated that the crucial influence on the leachability is the quality of waste particles encapsulation. A contact of extraction acid with an imperfectly encapsulated waste particle on the test specimen surface or in an open pore is manifested by the neutralisation of the acid (an increase of the pH value) and simultaneously by the pollutants leaching (an increase of Zn and Cl concentrations). The changes in leachability between samples are probably not pH-controlled because pH values closer to the neutral should lead to lower zinc solubility, and the chloride concentration should be nearly independent on pH. The best encapsulation efficiency in the TCLP test was obtained for the sample P8 (OPC pre-treatment + ESSIL silicone binder), which was the third-best in the distilled water leaching test. The action of acetic acid on silicone polymers did not show observable deteriorations on the surface. The TLCP procedure confirmed the previous observation that waste particles are encapsulated by silicone polymers; however, these particles can be more easily washed off from material due to a more acidic environment. A comparison of the TCLP

results with UTS (US EPA, 2016) showed that the zinc concentration in the leachate of none sample tested met the limit value (4.6 mg/L). Other measured elements concentration was mostly negligible except lead concentration at sample P3 and P4, but these concentrations were still below the UTS limits.

Table 6
The results obtained using TCLP.

Sample	P7	P9	P8	P10	P3	P4
			Concentration,mg/L			
Cd	< 0.01	0.01 ± 0.005	< 0.01	< 0.01	< 0.01	< 0.01
Cu	0.01 ± 0.005	0.01 ± 0.005	< 0.01	< 0.01	0.02 ± 0.01	0.03 ± 0.01
Ni	0.01 ± 0.002	0.01 ± 0.001	0.003 ± 0.001	0.007 ± 0.001	0.03 ± 0.004	0.05 ± 0.006
Pb	0.02 ± 0.003	0.11 ± 0.01	0.001 ± 0.0005	0.01 ± 0.001	0.17 ± 0.02	0.23 ± 0.03
Se	< 0.01	0.01 ± 0.005	< 0.01	< 0.01	< 0.01	< 0.01
Cr	< 0.01	< 0.01	< 0.01	< 0.01	0.01 ± 0.005	0.02 ± 0.01
As	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
Zn	45.1 ± 8.3	115.6 ± 3.5	21.9 ± 11.5	45.4 ± 6.0	241.2 ± 21.8	349.7 ± 22.3
Cl^-	163.8 ± 9.3	Image of Fig. 13	74.0 ± 8.8	148.1 ± 11.3	374.7 ± 15.5	630.5 ± 24.8
Final pH value	3.5	3.7	3.3	3.3	3.8	4.3

3.8. Influence of pH value and porosity on zinc leaching

To analyse the influence of pH on the leaching of pollutants, a series of leaching tests was carried out using extraction solutions with the initial pH value of 1, 4, 7, 10 or 12.5. The solid-liquid ratio was maintained at L/S = 10. The results for the selected samples are shown in Fig. 14. As can be seen, the highest zinc concentrations were measured at pH 1 and 12.5 with a local minimum in a slightly alkaline environment. However, as can also be seen in Fig. 14, those concentration maxima in a strongly acidic/basic environment increase rapidly with increasing sample porosity. The zinc leachability is governed by the solubility of the metal hydroxides, which is a well-known effect. The solubility in the high alkaline environment is caused by a formation of soluble zincates (Dutra et al., 2006). Nevertheless, in the case of waste encapsulation with silicone binders, the porosity of the sample has a decisive influence on the leachability of zinc. The leachability of chlorides and TDS was not so dependent on pH values, which agreed with the observation by other authors, for example, Quina et al. (2009). In the leaching test according to EN 12457-4, the final pH values of extracts were between 7.8 and 8.7, that means the zinc was probably presented in the form of zinc cations in the solution. The lowest zinc concentrations were measured for the sample with the highest pH value. On the other hand, the dependence of zinc concentration on the pH value could be only a correlation, as a better encapsulation and lower porosity of a sample means a lower leachability of both the zinc ions from the waste and hydroxide ions from cement. Thus, the zinc leachability is a more likely controlled by the sample porosity, as indicated the TCLP results where the best two samples were the samples with the lowest measured values of open porosity.

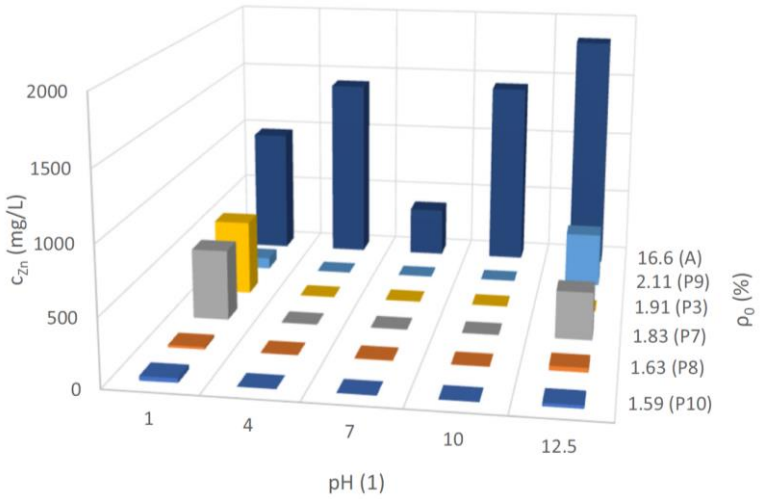

Fig. 14. Influence of pH value and sample porosity on the zinc leachability.

4. CONCLUSIONS

• The encapsulation of the waste with a high zinc content using silicone binders provided a better immobilisation efficiency for zinc, chlorides, and dissolved solids than the solidification using hydraulic binders, however, did not comply with the regulatory limit value for zinc.

• The combined two-step treatment of the waste using hydraulic binders and silicone polymers improved the pollutant immobilisation efficiency and met the regulatory limit values in the aqueous leaching test for all pollutants observed.

• Although the leachability of zinc is highly pH dependent, the releasing of zinc is controlled rather by the porosity of the encapsulated waste than its solubility in acids or bases.

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