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Scientific Paper

INERTISATION OF GALVANIC SLUDGE WITH CALCIUM OXIDE, ACTIVATED CARBON, AND PHOSPHORIC ACID

Višnja OREŠČANIN[1], Ivanka LOVRENČIĆ MIKELIĆ[2], Robert KOLLAR[1], Nenad MIKULIĆ[3], and Gordana MEDUNIĆ[4]

Advanced Energy Ltd.[1]*, Laboratory of Radioecology, R. Boskovic Institute*[2]*, EKO INVEST, Ltd., Zagreb, Croatia*[3]*, Institute of Mineralogy and Petrography, Faculty of Science*[4]*, Zagreb, Croatia*

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In this study we compared three methods for the treatment of electroplating sludge highly loaded with zinc and iron: (1) calcium oxide-based solidification/stabilisation; (2) conversion into inert material by adsorption of organic and inorganic pollutants onto activated carbon; and (3) conversion of mobile waste components into insoluble phosphates. All three methods proved highly efficient in the conversion of hazardous waste into inert material. Under optimum treatment conditions zinc concentration in the leachate of solidified waste was reduced by 99.7 % compared to untreated sludge. Zinc retention efficiency in the waste treated with activated carbon and phosphoric acid was 99.9 % and 98.7 %, respectively. The advantages of electroplating sludge treatment with activated carbon over the other two methods are high sorption capacity, insignificant pH and volume changes of the sludge, and simple use.

KEY WORDS: *heavy metals, immobilization, leaching, solidification*

Because of the high content of toxic heavy metals electroplating sludge is considered hazardous waste (1). On the other hand, this toxic waste could be reused as raw material for the extraction of zinc, chromium, and other valuable components to save natural resources. Various alkaline or acid based hydrometallurgical and electrochemical processes have been used for this purpose (1-6), but their major disadvantage is that they yield large amounts of sludge that needs to be neutralised (rendered inert) before landfilling. If metal extraction is not economically justified, such material has to be converted to non-hazardous waste by solidification/stabilisation and can be reused in cement industry (7-11). Although there are more than a hundred electroplating facilities in Croatia, most of them zinc plating, there either recovery or inertisation of electroplating sludge have been regulated (12). As this sludge contains high concentrations of zinc in the leachate, and the pH value is usually below 3, it poses a considerable threat to the environment and people, which has been confirmed by toxicological tests on various bio-systems for the concentration of zinc that are even lower than 2 mg L^{-1} (13-19).

Oreščanin et al. (20) developed the first method to treat electroplating sludge in Croatia that is based on solidification/stabilisation of sludge with CaO in order to convert hazardous waste into inert material. Our preliminary results have confirmed that the method is highly efficient for the specific purpose. In this study we wanted to analyse two new methods for sludge inertisation based on the application of powdered activated carbon and phosphoric acid and to compare them with the CaO method in terms of efficiency in binding heavy metals, sludge production, simplicity of implementation, and cost.

MATERIALS AND METHODS

Sampling and sample handling

For inertisation experiments we took samples of electroplating sludge from Tvik electroplating facility (Knin, Croatia). Sampling and sample handling has already been described in detail in our previous article (20).

Solidification/stabilization procedure

The electroplating sludge was solidified/stabilised with calcium oxide (Lika lime factory, Ličko Lešće, Croatia) as described in our previous work (20) and after air-drying subjected to the leaching test according to DIN38414-S4 procedure.

Sludge inertisation using activated carbon

A composite sample of sludge was mixed with 1 %, 2 %, 3 %, 4 %, 5 %, and 6 % of powdered activated carbon (*p.a.* Kemika, Zagreb) in relation to the dry weight of the sludge. The mixture (5.9 g) was homogenised for 10 min, air dried at room temperature (22 °C), and subjected to the DIN38414-S4 leaching test.

Sludge inertisation using phosphoric acid

A composite sample of sludge was mixed with 1 %, 2 %, 3 %, 4 %, and 5 % of phosphoric acid (Kemika, Zagreb) in relation to the dry weight of the sludge. The mixture (5.9 g) was homogenised for 10 min, air dried at room temperature (22 °C), and subjected to the DIN38414-S4 leaching test.

Analysis of parameters in solid and liquid samples

Dry matter and organic matter content were measured overnight by loss on ignition at 105 °C and 375 °C, respectively. Elemental concentrations in solid samples were determined using the energy dispersive X-ray fluorescence method (EDXRF) (21). DIN 38414-S4 leachates of original and inertised samples were prepared as described in our earlier studies (20, 22). Instrumental settings as well as quality control have been described in detail in our earlier research (21, 22).

RESULTS AND DISCUSSION

Basic physico-chemical characteristic of the electroplating sludge

Table 1 shows that the prevailing elements of toxicological significance in untreated electroplating sludge were iron, zinc, chromium, nickel, and lead. Sludge also contained a significant amount of organic matter (9.4 % to 29.4 %). High chelating potential of these organic constituents was the most probable reason for low leaching of chromium, lead, and copper from the sludge. Lead concentration in the DIN38414-S4 leachate was below the detection limit (0.001 mg L^{-1}) in all tested samples while mean chromium and copper values were 0.096 mg L^{-1} and 0.053 mg L^{-1} of leachate, respectively (Table 2). These findings have

Table 1 *Element content in bulk samples of untreated electroplating sludge*

Measured parameter	Statistical parameter					
	Mean	SD	RSD	Median	Minimum	Maximum
Ca / mg kg^{-1}	73000	39900	0.55	65000	13000	152000
Ti / mg kg^{-1}	451.7	53.61	0.12	463	365	557
Cr / mg kg^{-1}	207.2	197.42	0.95	131	21	759
Mn / mg kg^{-1}	439.4	110.23	0.25	440.5	207	598
Fe / mg kg^{-1}	136000	47900	0.35	151600	38400	191400
Ni / mg kg^{-1}	278.3	67.99	0.24	273	185	418
Cu / mg kg^{-1}	133.3	72	0.54	122.5	16	317
Zn / mg kg^{-1}	33400	12600	0.38	31800	10900	58200
Pb / mg kg^{-1}	109.9	62.25	0.57	114.5	11	235
Dry Matter / mg kg^{-1}	253000	83200	0.33	243000	125000	455000
LOI 375 ˚C / mg kg^{-1}	197000	52900	0.27	183000	94000	294000

SD-standard deviation; RSD-relative standard deviation confirmed our previous research (22-24) showing high susceptibility of these three elements to organic lygands. The DIN38414-S4 leachate composition shows that this sludge was not suitable for landfilling with inert waste since mean zinc and nickel were 10 and 1.5 times higher than the upper permissible limit, and the maximum zinc and nickel values were 27 and 2.5 times higher than the upper permissible limit, respectively (25).

Zinc concentration in the leachate of untreated sludge ranged from 0.3 mg L^{-1} to up to 107 mg L^{-1} with the mean value of 40.3 mg L^{-1}. In our earlier study (13), we found that zinc concentrations higher than 25 mg L^{-1} were cytotoxic to TA98 and TA100 *Salmonella typhimurium* strains while 100 mg L^{-1} of zinc significantly increased the frequency of micronucleated cells and reduced mitotic activity of human peripheral blood lymphocytes. In another study (14) the survival rates for HeLa and HEp2 human cells were only 2.3 % and 0.3 %, respectively, after treatment with electroplating wastewater having zinc concentration of 562 mg L^{-1}, while 99.5 % diluted sample significantly increased all comet assay parameters compared to negative control.

Durgo et al. reported (15) less than 15 % survival of the TA98 and TA100 *Salmonella typhimurium* strains after the treatment with electroplating wastewater having zinc concentration of 505 mg L^{-1} (15) while Horvat et al. (17) found that 51 mg L^{-1} and 126 mg L^{-1} of zinc have a significant toxic effect on *Lemna minor*.

In another study (19), zinc concentration of 86 mg L^{-1} significantly increased all comet assay parameters after exposure of peripheral blood lymphocytes to the leachate of electric arc furnace dust. All these findings suggest that untreated electroplating sludge could endanger human health and plant bio-systems if released directly into the environment (leaching by precipitation).

Sludge inertisation with CaO

Figure 1a shows to which extent calcium oxide treatment lowered heavy metal concentrations in the

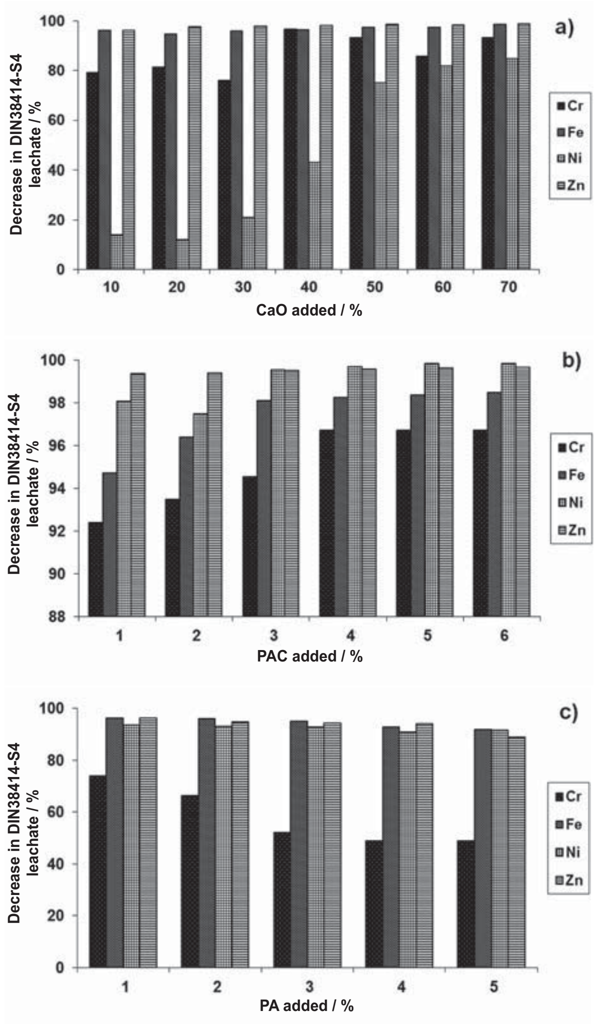

Figure 1 *Decrease in heavy metal concentrations in the DIN38414-S4 leachate of treated waste compared to untreated electroplating sludge for different dosages of: a) calcium oxide; b) powdered activated carbon (PAC); c) phosphoric acid (PA)*

Table 2 *Heavy metal concentrations in the leachate DIN38414-S4 of untreated electroplating sludge and maximum allowed concentrations*

Statistical parameter	Measured parameter					
	Cr / mg L^{-1}	Fe / mg L^{-1}	Ni / mg L^{-1}	Cu / mg L^{-1}	Zn /mg L^{-1}	pH
Mean	0.096	15.725	0.634	0.053	40.320	4.34
SD	0.047	9.970	0.306	0.194	27.997	0.80
RSD	0.486	0.634	0.483	3.691	0.694	0.19
Median	0.090	14.307	0.634	0.003	34.746	4.06
Minimum	0.006	0.345	0.082	0.001	0.349	3.42
Maximum	0.183	34.085	1.052	0.829	107.475	5.90
MAV (25)	0.5	-	0.4	2	4	-

SD-standard deviation; RSD-relative standard deviation

DIN38414-S4 leachate of electroplating sludge compared to untreated sludge. Treatment with the lowest dosage of CaO (10 %) already lowered all elemental concentrations below the lowest limit for the landfilling (25) and these concentrations dropped linearly with increasing CaO concentration. The exception is nickel, which dropped below the acceptable limit only at 40 % CaO.

Although, lime-based solidification is a method of choice for solidification of municipal solid waste (26-28), the use of this method for solidification of waste oil, oily wastes, and other waste by-products with high organic content has scarcely been investigated and only a few studies investigated its use in the solidification of inorganic sludges. Silva et al. (3) showed high efficiency of a two-phase sludge solidification method that included clay and lime in the first step and Portland cement, sand, and water in the second. Other authors (29) found that hydrated lime and black rice husk ash in the presence of either Na_2SiO_3 or Na_2CO_3 activators significantly reduced heavy metals leaching, especially of zinc, which was immobilised in the form of calcium zincate and calcium zinc silicate.

Extremely low leaching of arsenic was observed after solidification of inorganic sludge with cement, fly ash, and $Ca(OH)_2$ in the weight ratio 3:1:0.5:0.5 (30) confirming excellent binding capacity of this mixture.

Hydrated lime was also used for the immobilisation of heavy metals in highly contaminated soil having a similar chemical composition as electroplating sludge. At $Ca(OH)_2$ and soil ratio of 0.0375 the concentration of heavy metals Cd, Co, Ni, Pb and Zn in the leachate were decreased below minimum detection limit (31).

The advantage of CaO-based solidification is that solidification and chemical stabilisation of waste sludge occur simultaneously due to exothermic hydration of calcium oxide. The porosity, hydrophobicity, fire resistance, and good thermal and acoustic properties of the obtained solid matter make it suitable for use in sectors such as civil engineering. Moreover, CaO is readily available and less expensive than the other two additives. Solidification is less expensive in terms of equipment and trained operators and can be used easily in large companies and small family businesses alike.

The major disadvantage is that for CaO to be effective with heavy metals retention it has to be added in large quantities (at least 40 %), which generates more waste than the other two methods.

Sludge inertisation with powdered activated carbon

The regulatory requirements for landfilling of inert waste (25) were met for all elements with only 1 % of PAC (Table 4). Zinc concentration in the leachate dropped 159 times compared to untreated sludge. Cr, Fe, Ni and Zn concentration dropped 92.4 %, 94.7 %, 98.1 %, and 99.4 %, respectively (Figure 1b). Heavy metal concentrations dropped linearly with further addition of PAC.

Although, activated carbon, either in granular or powdered form, is commonly used for the adsorption of organic (32-34) and inorganic matter (35-37) in wastewaters, we have not found a single article on immobilization of heavy metals with this adsorbent in industrial sludges.

PAC turned out to be more effective in reducing heavy metal leaching than the other two methods (Figure 2) due to high adsorption capacity. PAC only

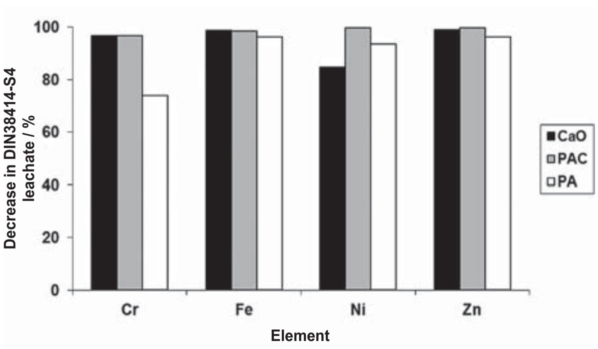

Figure 2 *Comparison of the best performance results for three treatment method of electroplating sludge. PAC-powdered activated carbon; PA-phosphoric acid*

slightly changes pH and the volume of the treated sludge in relation to untreated sludge. Treatment process is simple, does not require expensive equipment and trained personnel, and is easily applied by large companies and small family businesses alike. The adsorbent does not require special precautions for handling and storage. The major disadvantages over CaO are that PAC is less available and more expensive and that treated sludge needs additional drying.

Sludge inertisation with phosphoric acid

The addition of 1 % of phosphoric acid showed the best results in the retention of all elements in waste sludge. Cr, Fe, Ni, and Zn concentration dropped in the leachate by 73.9 %, 96.2 %, 93.7 %, and 96.3 %, respectively (Figure 1c) and all elements met regulatory requirements for landfilling of inert waste

Table 3 *Elemental concentration in DIN38414-S4 leachate of galvanic sludge before/after treatment with different dosages of CaO and upper permissible limit for inert waste (MAV)*

Element	Concentration before treatment / mg L^{-1}	MAV (25)	Concentration after CaO added / mg L^{-1}						
			10 %	20 %	30 %	40 %	50 %	60 %	70 %
Cr	0.092	0.5	0.019	0.017	0.022	0.003	0.006	0.013	0.006
Fe	13.113		0.477	0.673	0.504	0.439	0.333	0.317	0.175
Ni	0.681	0.4	0.586	0.599	0.537	0.389	0.169	0.122	0.103
Zn	37.900	4	1.380	0.787	0.781	0.679	0.488	0.532	0.348

Table 4 *Elemental concentration in DIN38414-S4 leachate of galvanic sludge before/after treatment with different dosages of powdered activated carbon (PAC) and upper permissible limit for inert waste (MAV)*

Element	Concentration before treatment / mg L^{-1}	MAV (25)	Concentrationa after PAC added / mg L^{-1}					
			1 %	2 %	3 %	4 %	5 %	6 %
Cr	0.092	0.5	0.007	0.006	0.005	0.003	0.003	0.003
Fe	13.113		0.691	0.473	0.247	0.229	0.213	0.198
Ni	0.681	0.4	0.013	0.017	0.003	0.002	0.001	0.001
Zn	37.900	4	0.239	0.222	0.174	0.153	0.139	0.127

Table 5 *Elemental concentration in DIN38414-S4 leachate of galvanic sludge before/after treatment with different dosages of phosphoric acid (PA) and upper permissible limit for inert waste (MAV)*

Element	Concentration before treatment / mg $L^{-}1$	MAV (25)	Concentration after PA added / mg L^{-1}				
			1 %	2 %	3 %	4 %	5 %
Cr	0.092	0.5	0.024	0.031	0.044	0.047	0.047
Fe	13.113		0.495	0.528	0.653	0.941	1.059
Ni	0.681	0.4	0.043	0.047	0.049	0.061	0.057
Zn	37.900	4	1.393	1.964	2.101	2.201	4.201

(Table 5). Further addition of phosphoric acid caused linear increase in leachate heavy metals concentrations probably trough the formation of soluble complexes.

Over the last few years a new effective method has been introduced for waste inertisation with phosphoric acid. This method has been developed by Solvay for inertisation of sediments in ports and shipyards with high heavy metal and organic matter loads and has been patented under the name of Novosol®. It can transform highly mobile heavy metals into hardly soluble metal phosphates and destroy organic matter with heat (38, 39). Yang and Mosby (40) used phosphoric acid for successful *in situ* immobilisation of lead in contaminated soil while Zupančič et al. (41) used the same method to immobilise nickel and zinc in sewage sludge and reduced significantly leaching of either metal.

High retention rate of heavy metals (Table 5) and other components in treated sludge, slight changes in pH, and a small amount needed are the major advantages of the phosphoric acid-based method. Its major disadvantage is that it involves special handling and storage, higher operational costs, lower efficiency in heavy metal binding, treated sludge needs additional drying.

CONCLUSION

All three treatment methods can efficiently convert hazardous waste into inert material and reduce its adverse effects on the environment and human health (13-19). Inertization with powdered activated carbon showed best performance (Figure 2). Its other advantages are insignificant changes in pH and volume of treated sludge, small amount needed, and simplicity of the application. On the other hand, CaO-based solidification is the least expensive method and the obtained solidificate is suitable for reuse.

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Sažetak

INERTIZACIJA GALVANSKOG MULJA S POMOĆU KALCIJEVA OKSIDA, AKTIVNOG UGLJENA I FOSFORNE KISELINE

U radu su prikazani i uspoređeni rezultati triju metoda obrade galvanskog mulja visoko opterećenog cinkom i željezom: (1) solidifikacija/stabilizacija otpada primjenom kalcijeva oksida; (2) inertizacija otpada adsorpcijom organskog i anorganskog opterećenja na aktivni ugljen; (3) prevođenje mobilnih komponenata otpada u teško topljive fosfate. Sve tri metode pokazale su se efikasnima u prevođenju opasnog otpada u inertno stanje. Kod optimalnih uvjeta koncentracija cinka u eluatu solidificiranog otpada snizila se za 99,7 % u odnosu prema neobrađenom otpadu. Efikasnost retencije cinka u otpadu inertiziranom aktivnim ugljenom iznosila je 99,9 %, a fosfornom kiselinom 98,7 %. Prednost inertizacije aktivnim ugljenom u odnosu prema ostalim dvjema metodama očituje se visokim sorpcijskim kapacitetom, neznatnim promjenama pH-vrijednosti i volumena tretiranog otpada, kao i jednostavnošću primjene.

KLJUČNE RIJEČI: *CaO, izluživanje, praškasti aktivni ugljen, teški metali*

CORRESPONDING AUTHOR:

Višnja Oreščanin
Advanced Energy Ltd.,
V. Prekrata 43, Zagreb, Croatia
E-mail: *vorescan@gmail.com*