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STABILIZATION AND CHARACTERIZATION OF THE SOLID WASTE GENERATED IN NEUTRALIZATION SLUDGE LEACHING PROCESS

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ABSTRACT – Acid mine drainage from Robule Lake is treated by neutralization process. Generated sludge is reach in copper and because of that is treated by leaching process, during which generate new type of waste. Characterization of this type of waste shows that is non-hazardous waste with high mobility of sulphate ions in leachability test and high mobility of lead and cadmium ions in toxicity test. Developed stabilization process shows low influence on immobilization of sulfuric ions, but high influence on immobilization of cadmium and lead ions presents in solid waste.

Keywords: Characterization, Waste, Sludge, Stabilization, Leaching.

INTRODUCTION

The sulfidic minerals present in active or abounded tailing ponds during short or long disposal period, under oxidation condition, generates free acid. The leachate from this mine facilities are known as acid mine drainage (AMD). Simultaneous, under the acidic conditions trace metals became liberated from the tailings and reaches into solution causing acidification/heavy metals pollution of the surrounding soil, groundwater as well as rivers and streams that receiving such effluent. Further this phenomenon leads to the serious pollution of drinking water as well as loss of the agricultural land [1,2,3].

Low-cost treatment method for AMD is neutralization process. Neutralization sludge that is generated during the AMD neutralization treatment can be reach in some value elements. Aim to valorized present elements, leaching process is applying. During leaching process another type of sludge is generated.

The aim of this work is characterization and stabilization this type of waste.

EXPERIMENTAL

Experimental work consists of the following activities:

- Characterization of solid waste generated in neutralization sludge leaching process;

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- Stabilization of solid waste generated in neutralization sludge leaching process;
- Characterization of stabilized solid waste.

Characterization of solid waste generated in neutralization sludge leaching process

Sludge generated in neutralization process of selected AMD – W1 is leached with sulfuric acid in controlled conditions (temperature and stirring).

Characterization of the solid waste generated in neutralization sludge leaching process was carried out in two phases:

- First - investigation of the waste leachability (LP procedure) by providing leaching test according to standard methods EN 12457-2 (Characterization of waste - Leaching - Compliance test for leaching of granular waste materials and sludges - Part 2: One stage batch test at a liquid-solid ratio of 10 l/kg for materials with a high content solids and particle size of less than 4 mm (with decreasing particle size, or without decreasing)) and
- Second - investigation of the waste toxicity (TCLP procedure) by providing Toxicity Characteristic Leaching Procedure according to standard method EPA 1311.

Stabilization of solid waste generated in neutralization sludge leaching process

Aim to ensure non-hazardous character of generated solid wastes after leaching for disposal on landfill for non-hazardous wastes, stabilization process is carried out.

Stabilization of the waste includes the following steps: determine the homogeneity of the waste; determine the moisture of the waste; determination the optimal ration lime and waste; determination of optimal contact time needed for stabilization process; confirmation defined parameters of stabilization by minimum five times repeated tests of leachability and toxicity of waste.

Characterization of the stabilized solid waste

Characterization of stabilized solid waste was carried out in two phases, same as for the solid waste generated in neutralization sludge leaching process.

RESULTS AND DISCUSSION

In the Table 1 are given the results of physicochemical testing waste (solid waste remaining after leaching) investigation of waste leachability according with SRPS EN 12457-2:2008. Moisture content in the sample is 45.44% and solid content is 54.56%.

Method SRPS EN ISO 11885 is used for As, Cu, Mo, Cd, Se, Ni, Hg, Pb, Cr, Zn, SO_4^{2-}, F^- and Cl^- determination, method SRPS EN 12506 for pH determination and EPA 120.1 for determination the conductivity.

In the Table 2 are given the results of physicochemical testing of toxic waste characteristics for disposal -TCLP test (EPA 1311).

Table 1 Results of physicochemical testing waste (solid waste remaining after leaching) leachability according with SRPS EN 12457-2:2008

Parameter	Unit	Measured value Label of the sample 01.20.11 OR-10 L/kg	The reference value for non-hazardous waste[2)]	The reference value for non-hazardous waste[3)]	The reference value for inert waste[4)]
Content in LP extract (neutral test, L/S = 10/1)					
Conductivity	µS/cm	2007	-	-	-
pH (25 °C)[1)]	-	8.96	-	6-13	-
Arsenic, As	mg/kg dm	<0.020	25	2	0.5
Antimony, Sb	mg/kg dm	<0.5	5	0.7	0.06
Copper, Cu	mg/kg dm	0.096	100	50	2
Molybdenum, Mo	mg/kg dm	<0.007	30	10	0.5
Cadmium, Cd	mg/kg dm	<0.008	5	1	0.04
Selenium, Se	mg/kg dm	<0.033	7	0.5	0.1
Nickel, Ni	mg/kg dm	<0.007	40	10	0.4
Mercury, Hg	mg/kg dm	<0.005	2	0.2	0.01
Lead, Pb	mg/kg dm	0.44	50	10	0.5
Chromium, Cr	mg/kg dm	<0.020	70	10	0.5
Zinc, Zn	mg/kg dm	0.097	200	50	4
Sulphate, SO_4^{2-}	mg/kg dm	13 000	50000	20 000	1 000
Fluoride, F^-	mg/kg dm	10.4	500	150	10
Chloride, Cl^-	mg/kg dm	10	25000	15 000	800

[1)] Annex 7 of the Regulation on waste categories, testing, and classification (Official gazette RS 56/2010. and 93/2019), H15 waste characteristics.

[2), 3), 4)] Annex 10 of the Rules on categories, testing and classification of waste (Official Gazette RS 56/2010 and 93/2019), Article 2, Parameters for testing waste and process water from hazardous waste landfill[2)], non-hazardous waste landfill[3)] and inert waste[4)].

Based on the limit values given in Annex 10 of the Regulation on categories, testing and classification of waste (Office Gazete RS 56/2010.) for LP and TCLP procedure:

- concentration of sulfuric ions in LP test is near the limit value for disposal on landfill for non-hazardous waste and
- concentration of lead and cadmium in TCLP test is near the limit value for waste toxicity criteria.

Optimal parameters for solid waste stabilization process are defined based on the realized investigations. Homogeneity of the waste has to be high, moisture content has to be in range from 42 to 46%, optimal ratio of lime: waste has to be 1:10 (m/m) and optimal contact time is 12 h.

Efficiency of the stabilization process can be seen by the results of stabilized waste testing according to LP and TCLP procedure. Results of this testing are shown in table 3, and the results of the waste testing according to TCLP procedure are shown in table 4.

In the samples (residue after leaching) for LP and TCLP characterization, moisture content is 42.30% and solid content is 57.7%.

Table 2 Results of physicochemical testing of toxic waste characteristics for disposal - TCLP test (EPA 1311)

Parameter	Unit	Measured value Label of the sample 02.20.11 OR-TCLP	The reference value for non-hazardous waste[5)]	Method
Content in Eluate (TCLP test)				
Antimony, Sb	mg/L	<0.011	15	SRPS EN ISO 11885
Chromium, Cr	mg/L	0.018	5	SRPS EN ISO 11885
Molybdenum, Mo	mg/L	<0.007	350	SRPS EN ISO 11885
Nickel, Ni	mg/L	0.048	20	SRPS EN ISO 11885
Selenium, Se	mg/L	<0.033	1	SRPS EN ISO 11885
Zinc, Zn	mg/L	22.6	250	SRPS EN ISO 11885
Copper, Cu	mg/L	1.6	25	SRPS EN ISO 11885
Arsenic, As	mg/L	<0.020	5	SRPS EN ISO 11885
Cadmium, Cd	mg/L	0.77	1	SRPS EN ISO 11885
Lead, Pb	mg/L	4.6	5	SRPS EN ISO 11885
Mercury, Hg	mg/L	<0.0005	0.2	SRPS EN ISO 11885
Vanadium, V	mg/L	0.032	24	SRPS EN ISO 11885
Silver, Ag	mg/L	0.016	5	SRPS EN ISO 11885

[5)] Annex 10 of the Role Book on the categories, testing and classification of waste (Official Gazette RS 56/2010), Article 1, Parameters for testing the toxic characteristics of waste intended for disposal.

Table 3 Results of physicochemical testing waste (residue after leaching) leachability according with SRPS EN 12457-2:2008

Parameter	Unit	Measured value Label of the sample 01.20.11 ST-10 L/kg	The reference value for non-hazardous waste[2)]	The reference value for non-hazardous waste[3)]	The reference value for inert waste[4)]
Content in LP extract (neutral test, L/S = 10/1)					
Conductivity	µS/cm	6658	-	-	-
pH (25 °C)[1)]	-	12.34	-	6-13	-
Arsenic, As	mg/kg dm	<0.020	25	2	0.5
Antimony, Sb	mg/kg dm	<0.5	5	0.7	0.06
Copper, Cu	mg/kg dm	<0.005	100	50	2
Molybdenum, Mo	mg/kg dm	<0.007	30	10	0.5
Cadmium, Cd	mg/kg dm	<0.008	5	1	0.04
Selenium, Se	mg/kg dm	<0.033	7	0.5	0.1
Nickel, Ni	mg/kg dm	<0.007	40	10	0.4
Mercury, Hg	mg/kg dm	<0.005	2	0.2	0.01
Lead, Pb	mg/kg dm	0.012	50	10	0.5
Chromium, Cr	mg/kg dm	<0.020	70	10	0.5
Zinc, Zn	mg/kg dm	0.015	200	50	4
Sulphate, SO_4^{2-}	mg/kg dm	11400	50000	20 000	1 000
Fluoride, F^-	mg/kg dm	12.3	500	150	10
Chloride, Cl^-	mg/kg dm	10	25000	15 000	800

[1] Annex 7 of the Regulation on waste categories, testing, and classification (Official gazette RS 56/2010. and 93/2019), H15 waste characteristics.
[2], [3], [4] Annex 10 of the Rules on categories, testing and classification of waste (Official Gazette RS 56/2010 and 93/2019), Article 2, Parameters for testing waste and process water from hazardous waste landfill[2], non-hazardous waste landfill[3] and inert waste[4].

Method SRPS EN ISO 11885 is used for As, Cu, Mo, Cd, Se, Ni, Hg, Pb, Cr, Zn, SO_4^{2-}, F^- and Cl^- determination, method SRPS EN 12506 for pH determination and EPA 120.1 for determination the conductivity.

Table 4 Results of physicochemical testing of toxic waste characteristics for disposal - TCLP test (EPA 1311)

Parameter	Unit	Measured value Label of the sample 02.20.11 ST-TCLP	The reference value for non-hazardous waste[5]	Method
Content in Eluate (TCLP test)				
Antimony, Sb	mg/L	<0.011	15	SRPS EN ISO 11885
Chromium, Cr	mg/L	0.052	5	SRPS EN ISO 11885
Molybdenum, Mo	mg/L	<0.007	350	SRPS EN ISO 11885
Nickel, Ni	mg/L	0.035	20	SRPS EN ISO 11885
Selenium, Se	mg/L	<0.033	1	SRPS EN ISO 11885
Zinc, Zn	mg/L	0.60	250	SRPS EN ISO 11885
Copper, Cu	mg/L	0.35	25	SRPS EN ISO 11885
Arsenic, As	mg/L	<0.020	5	SRPS EN ISO 11885
Cadmium, Cd	mg/L	0.017	1	SRPS EN ISO 11885
Lead, Pb	mg/L	0.16	5	SRPS EN ISO 11885
Mercury, Hg	mg/L	<0.0005	0.2	SRPS EN ISO 11885
Vanadium, V	mg/L	0.045	24	SRPS EN ISO 11885
Silver, Ag	mg/L	0.010	5	SRPS EN ISO 11885

[5] Annex 10 of the Role Book on the categories, testing and classification of waste (Official Gazette RS 56/2010), Article 1, Parameters for testing the toxic characteristics of waste intended for disposal.

Based on comparison with the limits given in Annex 10 of the Regulation on categories, testing and classification of waste (Office Gazete RS 56/2010.) for LP and TCLP procedure comment are as follows:

- concentration of sulfuric ions in LP test of stabilized waste is lower than in unstabilized waste, but influence of stabilization process on immobilization of sulfuric ion is not high;
- concentration of lead and cadmium ions in TCLP test is lower than in unstabilized waste, and influence of stabilization process on immobilization of lead and cadmium ions is very high.

Proposed stabilization process is applicable for stabilization of solid waste generated in neutralization sludge leaching process.

CONCLUSION

The experiment results show that new type of waste generated after treatment process of AMD consists of neutralization process and leaching process is non-

hazardous waste, but concentration of sulfuric ions in LP test is near the limit value for disposal on landfill for non-hazardous waste and concentration of lead and cadmium in TCLP test is near the limit value for waste toxicity criteria.

The efficiency of stabilization process is investigated. Results are shown that stabilization process doesn't have the significant influence on immobilization of sulfuric ions, but influence on immobilization of lead and cadmium ions is very high. Concentration of lead and cadmium ions in TCLP test is the lower than in unstabilized waste, and influence of stabilization process on immobilization of lead and cadmium ions is very high.

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