THE STUDY OF LEACHABILITY AND TOXICITY OF SLUDGE AFTER NEUTRALIZATION OF SARAKA AND ROBULE AMD WASTEWATERS

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Abstract

Acid mine drainage (AMD) waters are one of the most important ecological risks at the global level because of its high heavy metals content and strong acidity. Treatment of AMD water is a complex and expensive. One of the most widely used treatment process is the neutralization process of AMD. The result of neutralization is the production of sludge which may contain various other (heavy) metals, depending on the chemical characteristics of the mine water treated.

In this paper, leachability and toxicity of the sludges obtained during the neutralization process of wastewater from Saraka and Robule acid mine drainage and the sludges after the stabilization process at different temperatures is tested. Sludge produced in the neutralization process of Robule AMD R4 (40) and stabilized on 105 ºC and 200 ºC shows a H8-corrosiveness characteristic. Stabilized sludge show tendency to lower leachability of zinc and copper, but without influence on sulphate leachability. Sludges that show a H8-corrosiveness needs additional stabilization/neutralization pretreatment prior temperature treatment.

Key words: *sludge; neutralization; heavy metals; leachability; TCLP.*

1. Introduction

Mining and processing of metal ores cause degradation and pollution of land due to deposition of large mining wastes (overburden and flotation tailings) as well as AMD formation.

Hundreds of hectares of land and thousands of kilometers of rivers and springs all over the world are endangered by circulation of AMD [1]. It is characterized by low pH value and high concentrations of potentially toxic dissolved metals, metalloids and sulphate [2-7].

The formation of AMD water is caused by uncontrolled oxidation of sulphide minerals, especially pyrite (FeS_2), resulting from the exposure of these minerals to oxygen, water and microorganisms [8-14], as a consequence of the mining and processing of metal ores and coals [1]. The mechanism of acid mine drainage (AMD) water formation was the subject of intensive research during last twenty years. Forming of AMD water from pyrite ore (FeS_2) is usually described by the following reactions [4, 10, 15-21]:

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$$FeS_2 + 7/2O_2 + H_2O \rightarrow Fe^{2+} + 2SO_4^{2-} + 2H^+ \tag{1}$$
$$Fe^{2+} + 1/4O_2 + H^+ \rightarrow Fe^{3+} + 1/2H_2O \tag{2}$$
$$Fe^{3+} + 3H_2O \rightarrow Fe(OH)_3 + 3H^+ \tag{3}$$
$$FeS_2 + 15/2O_2 + 7/2\,H_2O \rightarrow Fe(OH)_3 + 2SO_4^{2-} + 4H^+ \tag{4}$$
$$FeS_2 + 14\,Fe^{3+} + 8H_2O \rightarrow 15Fe^{2+} + 2SO_4^{2-} + 16H^+ \tag{5}$$
$$FeS_2 + 15/4O_2 + 1/2H_2O \rightarrow Fe^{3+} + 2SO_4^{2-} + H^+ \tag{6}$$
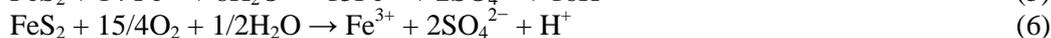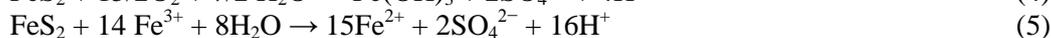
The oxidation of the pyrite (or sulphide) into dissolved iron, sulphate and hydrogen is the first and most important reaction (Eq. (1)) [15]. Also, the presence of sulphate is typically the first indicator of sulphide mineral oxidation [17].

The overall reaction of pyrite oxidation (eq. (1)-(3)) is represented by equation 4. Equation 5 shows complete oxidation of pyrite where ferric acts as the oxidizing agent, and is also deemed a faster reaction than reaction [12]. As can be seen from reaction (5), the oxidation of pyrite by ferric iron results in a further decrease in pH [20]. However, the rate of pyrite oxidation and the resulting acid production depend on various variables such as: composite solid phase composition and microbial activity, as well as the availability of oxygen and water [17].

The complete oxidation of pyrite in the presence of low water content is represented by reaction (6) [21]. It must be noted that reactions (2) and (4) can be significantly accelerated by the presence of acidophilus bacteria, such as Thiobacillus ferroxidans [17, 20]. It has been shown that these bacteria increase the iron conversion rate factor from hundreds to a million times [20, 22].

Acid mine drainage, as one of the major environmental pollution problems, is associated with historic and current mining industries [1, 23]. It causes problems to current and future generations because of its high heavy metal content and strong acidity [24]. The type and metals concentration present at mine drainage depends on nature of ore deposits.

Copper mines are mostly responsible for acid water forming. Uncontrolled drainage or pumping out of such water into nearest water streams pollutes living environment in large scale. At some copper mines, AMD is detected after 2 – 5 years from the beginning of mining operations. Abandoned copper mines and tailing dumps can generate acid water for tens, hundreds and even thousand of years [16]. One of examples of such pollution is abandoned pyrite mine Richmond in California (USA) for which one it is estimated that it can generate extremely acid solutions for the next 3000 years. For a small zinc and copper mine at north-west Ontario (Canada) it is estimated that it will create AMD waters for 10 000 – 35 000 years [16]. Although these two examples are extreme, it is not unusual that abandoned mines and tailing dumps have a capacity of generating acid solutions for hundreds of years. It is supposed that ten thousand of active or abandoned mines exist in Europe from where $5\text{-}10 \times 10^9$ m^3 of polluted mine water is discharged [16]. The data scale of the world problem of AMD is argumented by the fact that more than half of mines within European Union has been closed up to now and almost all of them are metals and coal mines.

Acid mine drainage contains heavy metals such as: Cu, Cd, Co, Cr, Mn, Ni, Pb and Zn, which have the potential to become major contamination sources, particularly in soil, and water resources [25-26]. AMD with these characteristics has not been discharged into public streams. Once AMD is generated, it is difficult to control the process and the treatment also requires high cost [1].

Many traditional processes for the treatment of acid mine water was briefly discussed by Kefeni et al. (2017). There may be divided into active and passive treatment options.

Among the conventional active chemical treatment options, the neutralization of AMD using various neutralizing reagents such as calcium hydroxide (CA (OH)$_2$) or limestone (CaCO$_3$) is a common method that has been widely applied for the removal of metals as metal hydroxide precipitates and sulphate as gypsum (CaSO$_4$·2H$_2$O) sludge [27, 28].The result is the production of an iron-rich sludge that may also contain various other metals, depending on the chemistry of the mine water treated. The sludge produced by this process is voluminous, with low solid content [29], low chemical stability [30] and has no economical value and that require further treatment or disposal [17, 31].

The disposed sludge produced in the neutralization process of AMD presents the potential risks to human health and the environment, particularly in the area there is the possibility of occurrence acid rain. Toxic element present in neutralization sludge, Zn, Cd, Pb, Cu, As, Sb and etc., can become mobile and polluted surrounding environment [32]. The sludge should be characterized in term of leachability test (mobility test of heavy metals contents in sludge). But, sludge disposal in line ponds is costly due to the following: large masses of sludge are produced. An estimated amount of 20 t/day of sludge is produced from 1 M l/day of discard leachate when neutralized with lime or limestone; plastic lining of sludge pond. Sludge produced from acid mine water is classified as a class 3 waste due to its metal content and must be discharged into a lined pond [33].

In the different European countries, tests have developed to characterize the constituents which can be leached from waste materials. The release of soluble constituents upon contact with water is regarded as a main mechanism of release in potential risk to the environment, especially during the reuse or disposal of waste materials. The intent of these tests is to identify and determine the leaching properties of waste materials. Due to the complexity of the leaching process in environmental conditions, it is necessary to simplify for laboratory research [34].

Toxicity Characteristic Leaching Procedure (TCLP) developed in USA in term of determining if a waste is hazardous or non-hazardous for disposal purposes. Also, the TCLP is used for legal waste classification. The TCLP test was designed to test toxicity characteristics by simulating leaching in an acidic landfill environment [35]. The TCLP is the current protocol required by Serbian low to characterize a waste for disposal purposes [36].

2. Experimental

2.1. Materials and methods

2.1.1. Study site

The Bor mining area is one of the important copper-producing areas in Serbia. The ore deposits in the mining area are porphyry copper deposits. The copper mine Bor, within the operation scope of Mining & Smelting Basin (RTB) Bor, started mining operations in the year 1903. For more than one hundred years of mining, about 673.5 million tons of overburden and flotation tailings were deposited in and near the municiplity of Bor [37-40]. Also, over 29,000 hectares of land it has been by quality strongly degraded. The area of such agricultural degraded land from the total percentage of agricultural land of the Bor District amounts over 60 % [40].

The area of Copper mine Bor represents a classical example of land degradation due to mining and processing of copper ore and disposing of mining wastes and flotation tailings. During the decades of exploitation of ore from the open pit Bor at different locations ("Visoki Planir" – also called "Oštreljski planir", "Severni planir" dump of ore body "H" (RTH)) mine overburden and tailings were delayed [40].

Active mine and flotation tailing dumps and industrial operations of RTB Bor create large amounts of acid mine water polluting natural rivers and springs with approximately 1285 t of Fe, 500 t of Cu, 52 t of Zn, 62 t of Mn, 1.8 t of Pb, 1.3 t of Ni, 400 kg of As and about 250 kg of Cd [41]. This water flows into the Robule lake and the Bor River and Krivelj River, which further flow into the Timok River and Danube River.

The Robule lake (figure 1) located close to the Bor city is characterized by low pH value and high concentration of metals [40]. It was built during 1970s as a consequence of the depositing of the large amount of overburden, about 150 million tons, that formed Veliki Planir. As a result of weathering, the products of oxidation are dissolved and leached in the form of AMD wastewaters, whose influence on the environment is extremely harmful due to a high content of heavy metal ions and sulphuric acid [37].

Figure 1. Robule lake

The ore exploitation at open pit mine Veliki Krivelj started in 1983. Since then, south of the open pit Veliki Krivelj, in the valley of the Saraka stream (figure 2), is situated a landfill for disposing of overburden. Since 1998, when the transportation system was built, overburden from the mine Veliki Krivelj is transported to the old open pit of Bor.

Figure 2. Saraka stream

Several thousands of cubic meters of AMD, as a consequence of the weathering from mine dumps, flows into the Borska and Kriveljska river every day [42]. Accordingly, both the Borska and the Kriveljska river are open collectors of acid mine drainage. Besides polluting the living environment, free discharge of acid waste water containing remarkable amount of various metals is a rather important economic loss [4].

The aims of this study are to determine the characteristics of the AMD from the Robule lake and Saraka stream and testing the leachability of sludges obtained during the neutralization process of these AMD and sludges after the stabilization process on different stabilization temperature.

2.1.2. Materials

The study was carried out in the region of the Bor mining area. Water samples were taken periodically from the Robule lake and Saraka stream.

Water sampling locations are shown in Figure 3 and are given by the following labels:

P-1 Robule lake (Figure 1);
P-2 Saraka stream (Figure 2).
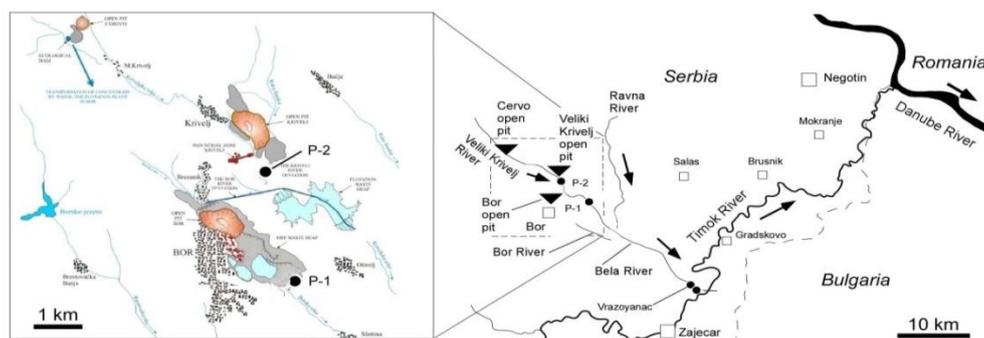
Figure 3. Map of the sampling locations in the area of the cities of Bor and Zaječar

As shown in Fig. 3, both samples were collected around the Bor mine and were then filtered through the 0.45 μm membrane to remove any debris or suspended solids. The samples were stored in closed high-density polyethylene bottles and kept at 4 °C [43].

All of the chemical reagents used were analytical reagent (AR) grade chemicals.

The characteristics of the AMD from the Robule lake and Saraka stream are presented in Table 1, which shows the mean values for all the water samples.

Table 1. Characteristic of AMD water samples

Parameter	Measure unit	Mark of water samples	
		P-1	P-2
pH	-	2.7	4.3
Al	mg/l	330.8	71.005
Cu	mg/l	64.4	75.112
Fe	mg/l	554.5	0.263
Mn	mg/l	122.6	16.769
S	mg/l	3843.2	936.707
Zn	mg/l	26.5	3.5
Ca	mg/l	590.4	529.715
Mg	mg/l	1478.7	226.173
Cr	μg/L	<1.7	<0.50
Co	μg/L	1354.4	1121.1
Ni	μg/L	643.4	280.1
As	μg/L	6.9	3.3
Se	μg/L	17.8	27
Sr	μg/L	1074.6	1143.7
Cd	μg/L	73.1	21.6
Cs	μg/L	<0.5	<0.10
Pb	μg/L	<2.1	0.2

Mass absorption method with coupled plasma (ICP-MS) on apparatus AGILEND 7700 was used for chemical analysis of: As, Cd, Co, Cr, Cs, Ni, Pb, Sr, Se

Atomic absorption method with inductive coupled plasma (ICP-AES) on apparatus SPECTRO BLUE was used for chemical analysis of: Al, Ca, Cu, Fe, Mg, Mn, S and Zn.

From table 1 it can be seen that the major metal contaminant was Fe, and others, such as Cu, Zn, and Mn were also present.

2.1.3. Methods

Sludge obtained during neutralization process of Saraka AMD on pH 7 and pH 11 and sludge obtained during neutralization process of Robule AMD on pH 4 and pH 8 are testing by leachability test and toxicity characteristic test in first phase of waste characterization.

Sludges after neutralization were treated on different temperatures of stabilization and also are testing by leachability test and toxicity characteristic test in second phase of waste characterization. Mark of tested sludges are given in Table 2.

Content of Zn and Cu were determined by atomic emission spectrometry method with Induction Coupled Plasma (ICP-AES) on apparatus Ciros Vision and content of Cr, Co, Ni, As, Se, Cd, Cs and Pb was determined by mass spectrometry method with Induction Coupled Plasma (ICP-MS) on apparatus AGILEND 7700.

Table 2. Mark of sludge samples

Sludge description	Mark of sample
Sludge after neutralization process of Robule AMD wastewaters up to pH 4 (dry on 40 ^{o}C)	R4 (40)
Sludge after neutralization process of Robule AMD wastewaters from pH 4 up to pH 8 (dry on 40 ^{o}C)	R8 (40)
Sludge after neutralization process of Saraka AMD wastewaters up to pH 7 (dry on 40 ^{o}C)	S7 (40)
Sludge after neutralization process of Saraka AMD wastewaters from pH 7 up to pH 11 (dry on 40 ^{o}C)	S11 (40)
Sludge after neutralization process of Robule AMD wastewaters up to pH 4 and stabilization on 105 ^{o}C	Rs4 (100)
Sludge after neutralization process of Robule AMD wastewaters from pH 4 up to pH 8 and stabilization on 105 ^{o}C	Rs8 (100)
Sludge after neutralization process of Robule AMD wastewaters up to pH 4 and stabilization on 200 ^{o}C	Rs4 (200)
Sludge after neutralization process of Robule AMD wastewaters from pH 4 up to pH 8 and stabilization on 200 ^{o}C	Rs8 (200)

The impact of sludges obtained in neutralization process of AMD and sludges after stabilization process on the environment were carried out by leaching test according to standard methods EN 12457-2 (Characterization of waste - Leaching - Compliance test for leaching of granular waste materials and sludge - 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.

The investigated materials which originally have at least 95 % mass ratio of particles below 4 mm, brought into contact with

leachant (water) under defined conditions. The Standard is based on the assumption that equilibrium or near-equilibrium is achieved between the liquid and solid phases during the test duration. The solid residue of eluate is separated by filtering. After the test, the leaching conditions in term of pH, conductivity (optionally redox potential) dictated by the waste are measured) [34].

Toxicity characteristic of sludges obtained in neutralization process of AMD and sludges after stabilization process were carried out by TCLP test according to standard methods EPA 1311 (Toxicity Characteristic Leaching Procedure).

The solid phase is extracted with an amount of extraction fluid equal to 20 times the weight of the solid phase. The extraction fluid employed is a function of the alkalinity of the solid phase of the waste.

Following extraction, the liquid extract is separated from the solid phase by filtration through a 0.45 µm glass fiber filter [35].

The results of waste testing are compared with limits given in Annex 10 of the Regulation on categories, testing and classification of waste (Office Gazette RS 56/2010. [36].

3. Results and discussion

To predicted amount/concentration of pollutant that can be released in environmental, leachability test (mobility test of heavy metals contents in sludge) is performed in Mining and Metallurgy Institute laboratories. Results of these tests are shown in Table 3 (sludges after neutralization process) and Table 4 (stabilized sludges).

Table 3. The results of the sludges characterization by leaching standard test method EN 12457-2 [4]

Parameter	Measure unit	Content				Limit value [36] **	Analytical testing standard method
		R4 (40)	R8 (40)	S7 (40)	S11 (40)		
pH of eluate	-	4.31	8.14	8.11	9.27	6-13	SRPS EN 12506:2007
Conductivity of eluate	µS/cm	3427	3293	1925	3530	-	SRPS EN 13370:2003
Lead, Pb	mg/kg dm*	0.23	<0.20	<0.20	<0.20	10	EN ISO11885
Cadmium, Cd	mg/kg dm*	0.24	<0.08	<0.08	<0.08	1	EN ISO11885
Zinc, Zn	mg/kg dm*	146	0.07	0.49	<0.05	50	EN ISO11885
Copper, Cu	mg/kg dm*	797	0.10	12	<0.05	50	EN ISO11885
Nickel, Ni	mg/kg dm*	3.6	<0.07	<0.07	<0.07	10	EN ISO11885
Chromium total, Cr_{tot}	mg/kg dm*	<0.05	<0.05	<0.05	<0.05	10	EN ISO11885
Mercury, Hg	mg/kg dm*	<0.05	<0.05	<0.05	<0.05	0.2	VMK C.h.1
Arsenic, As	mg/kg dm*	<0.20	<0.20	<0.20	<0.20	2	EN ISO11885
Antimony, Sb	mg/kg dm*	<0.5	<0.5	<0.5	<0.5	0.7	VMK C.G.1
Barium, Ba	mg/kg dm*	0.46	<0.09	0.11	<0.09	100	EN ISO11885
Selenium, Se	mg/kg dm*	<0.33	<0.33	<0.33	<0.33	0.5	EN ISO11885
Molibdenium, Mo	mg/kg dm*	<0.07	<0.07	<0.07	<0.07	10	EN ISO11885
Chloride (Cl^-)	mg/kg dm*	6	7	18	16	15000	SRPS EN 12506:2007
Sulphate (SO_4^{2-})	mg/kg dm*	28000	23500	21000	24500	20000	SRPS EN 12506:2007

Table 4. The results of stabilized sludges on 105 ^{o}C and 200 ^{o}C characterization by leaching standard test method EN 12457-2 [4]

Parameter	Measure unit	Content				Limit value [36] **	Analytical testing standard method
		R4 (105)	R8 (105)	R4 (200)	R8 (200)		
pH of eluate	-	4.44	7.91	4.50	8.37	6-13	SRPS EN 12506:2007
Conductivity of eluate	µS/cm	3313	2803	2623	3128	-	SRPS EN 13370:2003
Lead, Pb	mg/kg dm*	0.68	<0.20	<0.20	<0.20	10	EN ISO11885
Cadmium, Cd	mg/kg dm*	0.09	<0.08	0.11	<0.08	1	EN ISO11885
Zinc, Zn	mg/kg dm*	127	<0.05	39	0.68	50	EN ISO11885
Copper, Cu	mg/kg dm*	828	<0.05	287	1.3	50	EN ISO11885
Nickel, Ni	mg/kg dm*	2.2	<0.07	0.7	<0.07	10	EN ISO11885
Chromium total, Cr_{tot}	mg/kg dm*	<0.05	<0.05	<0.05	<0.05	10	EN ISO11885
Mercury, Hg	mg/kg dm*	<0.05	<0.05	<0.05	<0.05	0.2	VMK C.h.1
Arsenic, As	mg/kg dm*	<0.20	<0.20	<0.20	<0.20	2	EN ISO11885
Antimony, Sb	mg/kg dm*	<0.5	<0.5	<0.5	<0.5	0.7	VMK C.G.1
Barium, Ba	mg/kg dm*	0.22	0.10	0.31	<0.09	100	EN ISO11885
Selenium, Se	mg/kg dm*	<0.33	<0.33	<0.33	<0.33	0.5	EN ISO11885
Molibdenium, Mo	mg/kg dm*	<0.07	<0.07	<0.07	<0.07	10	EN ISO11885
Chloride (Cl^-)	mg/kg dm*	5	7	6	7	15000	SRPS EN 12506:2007
Sulphate (SO_4^{2-})	mg/kg dm*	28500	21500	21000	26000	20000	SRPS EN 12506:2007

*dm – dry mass

Sludge obtained in the neutralization process of Robule AMD R4 (40) and sludges stabilized on 105 ^{o}C and 200 ^{o}C shows a H8-corrosiveness characteristic, because of eluate pH values significantly lower (4.31, 4.44 and 4.50, respectly) than the limit value (6.0) and indicates the acidity eluate generated during the leaching test. Also, content of sulphate is higher than limit for unhazardous waste for all tested sludge samples.

Sludge produced in the neutralization process of Robule AMD R4 (40) and stabilized on 105 ^{o}C shows higher leachability of zinc and copper than limit values. Sludge produced in the neutralization process of Robule AMD and stabilized on 200 ^{o}C shows higher leachability only for copper.

Sludge produced in the neutralization process of Saraka AMD S7 and S11 are in accordance with applicable laws and regulations of the Republic of Serbia, except of sulphate content.

To predicted amount/concentration of pollutant that can be released in environmental, leaching in an acidic landfill environment (toxicity characteristic of sludges) is performed in Mining and Metallurgy Institute laboratories especially on samples that not shows hazardous characteristic by leachability test.

The result of these tests is shown in Table 5 (sludge after neutralization process) and Table 6 (stabilized sludge).

Table 5. The results of toxicity characteristic of sludge by TCLP test method

Parameter	Measure unit	Content			Limit value[36]	Analytical testing standard method
		R8 (40)	S7 (40)	S11 (40)		
Lead, Pb	μg/L	<2.1	<2.1	<2.1	5 000	EN ISO11885
Cadmium, Cd	μg/L	284.8	53.9	0.19	1 000	EN ISO11885
Zinc, Zn	mg/L	24.9	1.8	0.009	250	EN ISO11885
Copper, Cu	mg/L	5.8	2.9	0.080	25	EN ISO11885
Nickel, Ni	mg/L	2.0	0.20	<0.007	20	EN ISO11885
Chromium total, Cr_{tot}	μg/L	1.9	<1.7	20.0	5 000	EN ISO11885
Mercury, Hg	mg/L	<0.0005	<0.0005	<0.0005	0.2	VMK C.h.1
Arsenic, As	μg/L	<2.1	<2.1	4.1	5 000	EN ISO11885
Antimony, Sb	μg/L	<0.86	<0.86	10.4	15000	VMK C.G.1
Barium, Ba	μg/L	32.5	38.4	6.8	100 000	EN ISO11885
Selenium, Se	μg/L	<4.5	9.7	21.0	1 000	EN ISO11885
Molibdenium, Mo	mg/L	<0.0023	0.0239	0.0101	350	EN ISO11885
Vanadium, V	mg/L	<0.008	0.048	<0.008	24	EN ISO11885
Silver, Ag	mg/L	<0.4	<0.4	<0.4	5	EN ISO11885

Table 6. The results of ^{o}C toxicity characteristic stabilized sludge on 105 ^{o}C and 200 by TCLP test method

Parameter	Measure unit	Content				Limit value [36]	Analytical testing standard method
		R4 (105)	R8 (105)	R4 (200)	R8 (200)		
Lead, Pb	μg/L	3.8	<2.1	7.2	<2.1	5 000	EN ISO11885
Cadmium, Cd	μg/L	9.1	203.9	3.2	186.5	1 000	EN ISO11885
Zinc, Zn	mg/L	4.7	9.6	1.2	20.1	250	EN ISO11885
Copper, Cu	mg/L	26.7	3.0	9.4	2.4	25	EN ISO11885
Nickel, Ni	mg/L	0.12	1.3	0.23	1.9	20	EN ISO11885
Chromium total, Cr_{tot}	μg/L	26.3	5.6	5.0	2.7	5 000	EN ISO11885
Mercury, Hg	mg/L	<0.0005	<0.0005	<0.0005	<0.0005	0.2	VMK C.h.1
Arsenic, As	μg/L	21.6	10.2	<2.1	<2.1	5 000	EN ISO11885
Antimony, Sb	μg/L	<0.86	<0.86	<0.86	<0.86	15000	VMK C.G.1
Barium, Ba	μg/L	37.4	29.6	30.9	21.2	100 000	EN ISO11885
Selenium, Se	μg/L	10.0	<4.5	7.6	<4.5	1 000	EN ISO11885
Molibdenium, Mo	mg/L	<2.3	<2.3	<2.3	2.7	350	EN ISO11885
Vanadium, V	mg/L	<0.008	<0.008	0.10	<0.008	24	EN ISO11885
Silver, Ag	mg/L	<0.4	<0.4	<0.4	<0.4	5	EN ISO11885

Based on the results presented in Table 5 and 6 it is seen that Cu concentration in sludge obtained by neutralization process from Robule Lake on pH 4, marked as R4 (105), and stabilized on 105 ^{o}C, has the higher value than the limited value. Result. For all other samples the obtained values for concentration of Pb, Cd, Zn, Cu, Ni, Cr tot., Hg, As, Sb, Ba, Se, Mo, V and Ag are highly below the limit values. In accordance with the Regulation on categories, testing and classification of waste in Republic of Serbia, this sludge has not be disposal before the prior treatment.

The value for copper concentration in TCLP extractit obtained from sludge in neutralization process of Robule AMD on pH 4 and stabilized on on 105 ^{o}C is higher than limit value for toxicity characteristic of waste. Taking into account the method uncertainty (uncertainty of preratation and determination), monitoring of R4 (105) sludge toxicity characteristic should be carried out in future period.

4. Conclusions

Acid mine drainage (AMD) is a consequence of uncontrolled oxidation of sulphide minerals, especially pyrite, which are initially exposed to the environment by intensive mining activities in Bor area.

The results of leachability test are not within the permitted limits for the disposal of nonhazardous waste to the landfill. Stabilized sludges show tendency to lower leachability of zinc and copper, but without influence on sulphate leachability.

Sludge that shows a H8-corrosiveness characteristic needs additional pretreatment stabilization/neutralization method prior temperature treatment stabilization.

The value for copper concentration in TCLP extractit obtained from sludge in neutralization process of Robule AMD on pH

4 and stabilized on on 105 ^{o}C is higher than limit value for toxicity characteristic of waste, but this results is in area of method unceratainty. Monitoring of R4 (105) sludge toxicity characteristic should be carried out in future period.

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