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A combined CaO/electrochemical treatment of the acid mine drainage from the "Robule" Lake

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The purpose of this work was development and application of the purification system suitable for the treatment of the acid mine drainage (AMD) accumulated in the "Robule" Lake, which represents the part of the Bor copper mining and smelting complex, Serbia. The study was undertaken in order to minimize adverse effect on the environment caused by the discharge of untreated AMD, which was characterized with low pH value (2.63) and high concentration of heavy metals (up to 610 mg/L) and sulfates (up to 12,000 mg/L). The treatment of the effluent included pretreatment/pH adjustment with CaO followed by electrocoagulation using iron and aluminum electrode sets. Following the final treatment, the decrease in the concentration of heavy metals ranged from 40 up to 61000 times depending on the metal and its initial concentration. The parameters, color and turbidity were removed completely in the pretreatment step, while the removal efficiencies for other considered parameters were as follows: EC = 55.48%, SO_4^{2-} = 70.83%, Hg = 98.36%, Pb = 97.50%, V = 98.43%, Cr = 99.86%, Mn = 97.96%, Fe = 100.00%, Co = 99.96%, Ni = 99.78%, Cu = 99.99% and Zn = 99.94%. Because the concentrations of heavy metals in the electrochemically treated AMD (ranging from 0.001 to 0.336 mg/L) are very low, the negative impact of this effluent on the aquatic life and humans is not expected. The sludge generated during the treatment of AMD is suitable for reuse for at least two purposes (pretreatment of AMD and covering of the flotation waste heap). From the presented results, it could be concluded that electrochemical treatment is a suitable approach for the treatment of AMD.

Keywords: Acid mine drainage, "Robule" Lake, CaO, electrocoagulation, iron anode, aluminum anode.

Introduction

Acid Mine Drainage (AMD) originates in the reaction of sulfide minerals from an open pit or the mine tailings with water and oxygen creating sulfuric acid. This process is largely accelerated by the activity of *Thiobacillus ferroxidans* bacteria. Under the acidic conditions trace metals are liberated from the ore/tailings into solution causing acidification/heavy metals pollution of the surrounding soil, groundwater as well as rivers and streams that receiving such effluent. Consequently, this activity leads to the serious pollution of drinking water sources in the area of the influence as well as loss of the agricultural land which is not suitable for agricultural production due to the pollution with metals and high acidity.

The acid mine drainage from the Bor mining complex (Bor-Krivelj-Cerovo) and smelting /refining of copper was characterized by low pH value (usually below 4) and high concentrations of heavy metals whose concentration varies depending on the source. If entire complex was considered 7 different sources[1] of the wastewaters could be defined.

(1) The AMD from "Veliki Krivelj" open pit representing the precipitation and groundwater collected on the bottom of the open pit from which it is pumped into the Kriveljska River in the amount of 2333 m^3/day. The average pH value is 4.4 and from the analyzed heavy metals, copper (127.5 mg/L), zinc (3.1 mg/L) and iron (0.38 mg/L) predominate, while the sulfates (360 mg/L) and chlorides (151 mg/L) are the dominant anions.
(2) Wastewaters of the Saraka River occur by the leaching of heavy metals and other components by the flow of the Saraka River through the open pit "Veliki Krivelj" with an average daily flow of 3802 m^3/day and flowing into the Kriveljska River. The pH ranges from 3.6 to 7.74, copper from 1.86 to 218 mg/L, iron from 0.21 to 85 mg/L, zinc from 0.12 to 2.31 mg/L, manganese 0.28 to 28 mg/L, sulfates from 41 to 319 mg/L and chlorides from 8 to 61 mg/L.
(3) Wastewater from the open pit "Bor" originate from the groundwater collected on the bottom of the open as well as the precipitation. Dailey input into the Kriveljska River is 2599 m^3/day. pH ranges from 2.49 to 8.27,

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copper from 0.028 to 389 mg/L, iron from 0.15 to 1834 mg/L, zinc from 0.02 to 287 mg/L, manganese from 0.01 to 20 mg/L, nickel from 0 to 0.74 mg/L, arsenic from 0 to1.22 mg/L, sulfates from 2.99 to 829 mg/L chloride and 6-720 mg/L.

(4) Wastewaters of the 1A flotation waste heap dam “Veliki Krivelj” represent the surface drainage of wastewaters that are discharged into Kriveljska River in the average amount of 1944 m^3/day. pH ranges from 6.08 to 8.12, copper from 0.043 to 0.43 mg/L, iron from 0.014 to 4.49 mg/L, zinc from 0.001 to 2.61 mg/L, manganese from 0.18 to 1.7 mg/L, nickel from 0 to 0.014 mg/L, arsenic from 0 to 0.016 mg/L, sulfates from 59 to 730 mg/L and chlorides from 20 to 29 mg/L.

(5) Wastewaters of the 3A flotation waste heap dam “Veliki Krivelj” represent the surface drainage of wastewaters that are discharged into Kriveljska River in the average amount of 3024 m^3/day. pH ranges from 4.6 to 8.24, copper from 0 to 0.276 mg/L, iron from 0.009 to 11 mg/L, zinc from 0.001 to 1.95 mg/L, manganese from 0.348 to 1.922 mg/L, nickel from 0 to 0.015 mg/L, arsenic from 0 to 0.201 mg/L, sulfates from 12 to 700 mg/L and chlorides from 12 to 26 mg/L.

(6) Wastewaters from the “Robule” Lake originate by the accumulation of wastewaters that leaching the waste heap of the “Bor” open pit. The average input from that source in the Bor River is 484 m^3/day. pH is 2.97; copper 55.16 mg/L, iron 895 mg/L, zinc 26.5 mg/L, manganese 125 mg/L, nickel 0.322 mg/L, arsenic 0.001 mg/L, sulfates 4145 mg/L and chlorides 42 mg/L.

(7) Mixed wastewaters originate from the “Bor” open pit and copper smelter and various metallurgical processes and drainage water. The average flow of the water in the Bor River is 2323 m^3/day. pH is 2.35; copper 54 mg/L, iron 323 mg/L, zinc 1.92 mg/L, manganese 9.63 mg/L, nickel 1.05 mg/L, arsenic 0.017 mg/L, sulfates 1670 mg/L and chlorides 22 mg/L.

From the preceding research, it could be concluded that there is high variability in the composition of the AMD among respective sources as well as within each source. The highest values of heavy metals in all these sources were found for copper, iron, manganese and zinc.

Due to the complex composition of the effluent, Pavlovic et al.[2] used cascade line reactor for the precipitation of heavy metals from AMD originated in Bor mining complex. Using 1M NaOH as a precipitant after 4 hours of the treatment in three parallel reactors the removal efficiencies of the elements copper, iron, manganese, zinc and nickel were 99.30%, 99.99%, 99.24%, 72.30%, 76.30%, respectively.

More than 92% of the copper was removed from the acid mine drainage from the open pit “Cerovo” using high degree of electrowinning. The initial concentration was reduced from 1300 mg/L to less than 0.1 mg/L.[3]

Johnson and Hallberg[4] summarized the treatment methods of the acid mine drainage. Among them aeration and lime addition, anoxic limestone drains, off-line sulfidogenic bioreactors, aerobic wetlands, compost reactors/wetlands, permeable reactive barriers and packed bed iron-oxidation bioreactors were described in details.

In our previous research, we demonstrated that electrocoagulation (EC) could be used successfully for the treatment of the effluents[5–7] laden with heavy metals. The theory and application of EC was summarized in the work of Mollah et al.[8] and Emamjomeh and Sivakumar.[9] This process involves the application of the electric current to the sacrificial electrodes inside the reactor tank where the current generates a coagulating agent and gas bubbles. These ions coagulate with pollutants in the water, similar to the addition of coagulating chemicals such as alum and ferric salts, allowing easier removal of the pollutants by sedimentation and flotation.[8] Iron and aluminum are the most commonly used as the sacrificial anodes in the electrocoagulation unit.[5–12]

Materials and methods

Sampling and purification

The acid mine drainage was collected from the “Robule” Lake (Bor mining complex, Serbia). The volume of 10 L of water was pumped into two acid cleaned polyethylene containers. The samples were kept at +4°C until the analysis and the purification experiments.

Prior to the purification experiments, the water samples were homogenized for 10 minutes. For each experiment 2.0 L of the effluent was taken and subjected to the pH adjustment by calcium oxide (Lika lime factroy, Licko Lesce, Croatia).[13]

Calcium oxide was of a technical grade with less than 5% of impurities and particle size less than 90 μm. The addition of CaO stopped when pH of the solution increased over 7. The suspension was mixed with air bubbles for 10 minutes and subjected to electrocoagulation process using iron electrode plates for 15 minutes, followed by electrocoagulation using aluminum electrode set for additional 10 minutes. [5–7] Each electrode set contained of six quadratic, 1 millimeter thick plates (75 × 250 mm) in the parallel arrangement, separated by an electro-insulator. The distance between plates = 10 mm; I = 6 A; U = 15 V. Each even plate was connected to + pole of DC power supply and represented sacrificial anode, while odd plates connected to the – pole of DC served as the cathodes. Either Fe or Al electrode set was placed at the bottom of the reaction vessel perpendicular to the vessel bottom.

The electrocoagulation by Fe electrode set was applied first. The mixing of the suspension was carried out with air bubbles supplied trough the diffuser placed at the bottom of the reaction vessel as well as hydrogen gas bubbles produced by the water reduction on the cathode plates. In the end of the reaction, Fe electrodes were replaced by Al electrode set and continued electrocoagulation for additional 10 minutes. In the end of the reaction Al electrode set was removed from the vessel while the suspension of the electrochemically generated flocks and water was mixed for additional 10 minutes in order to oxidize the excess of ferrous iron and to complete removal of pollutants by flocculation. The settlement time lasted 30 minutes. The whole procedure was repeated three times and the removal efficiency for each parameter was expressed as a mean value obtained from these three experiments.

After the settlement, the clear water was decanted and analyzed while the waste sludge was dried at 105°C to the constant weight and analyzed for bulk and leachate composition. In the second treatment experiment, the sludge collected from the treatment of AMD was used instead of CaO for pH adjustment and pretreatment of the initial AMD. The addition of sludge stopped when pH of the solution increased over 7. Pretreated effluent was subjected to electrochemical treatment as described above. After the settlement, the clear water was decanted and analyzed.

Preparation of liquid samples for EDXRF analysis

The volume of 0.5 mL of the original acid mine drainage and 100 mL of each purified sample or DIN38414-S4 leachate was adjusted to pH 3 by the addition of hydrochloric acid and ammonium hydroxide, preconcentrated by ammonium pyrrolidine-dithiocarbamate (APDC), then filtered through a Millipore HAWP filter (pore size 0.45 μm; diameter 25 mm) using a Millipore micro filtration system)[5–7] and analyzed by energy dispersive X-ray spectrometry (EDXRF).

Preparation of solid samples for EDXRF analysis

The samples of dried waste sludge were prepared for the analysis as follows: 4 grams of each powdered sample (particle size <0.071 mm) was pressed into pellets of 20 mm in diameter (15 tons pressure, 30 s dwelling time). No binder material was applied. The samples were placed in the standard sample holders and loaded into X-ray spectrometer. Five replicate samples of the sludge were taken.[5]

Leaching test

DIN 38414-S4 leachates[5] of the waste sludge were prepared by mixing the sample with deionized distilled water for 24 hours on a rotary shaker (3 rpm). Solid/liquid ratio was 1:10. The test was carried out at a room temperature. The suspended solid matter was removed from the liquid phase by filtration through a glass fiber filter. The obtained leachate was prepared and analyzed as described for other liquid samples.

Elemental analysis of the targets

All samples were irradiated by X-rays generated from X-ray tube ("Oxford instruments") with Mo anode (high voltage: 40 kV; current: 900 μA; cooling medium: Air).[5,7,8] The detection of the characteristic X-ray radiation from the sample was conducted with the Si drift detector - model SXD15C-150-500 (Canberra, Meriden, USA) (surface: 15 mm^2; FWHM for 5.9 keV ^{55}Fe: 145 eV; Window: 13 μm Be; cooling: thermo-electrical (peltier)). The incident and emerging angles were 45°.

For signal processing DSA 2000 (Canberra) was used. Spectral data were collected by Genie-2000 software (Canberra) while for spectrum deconvolution and quantitative analysis WinAxil software package (Canberra) was used. In order to obtain a good counting statistic, collecting time for thick targets was 5000 seconds, while thin targets were measured 10,000 seconds. The calibration model for the qualitative and quantitative analysis of the thick targets was created on the basis of the measurements of the following standard reference materials from the National Institute of Standards and Technology (Gaithersburg, MD, USA): SRM 2782-Industrial sludge, SRM 2781-Domestic sludge, SRM 2702-Inorganic in marine sediment. Calibration model for the qualitative and quantitative analysis of thin targets was created on the basis of the measurements of the standard solutions (Merck) having the concentration range from 10 to 200 μg/L, prepared and measured in the same way as unknown samples.

Determination of other parameters

Color was determined according to the 8025 APHA platinum-cobalt standard method (adopted from Standard Methods for the Examination of Water and Wastewater) using HACH DR890 colorimeter (Hach Company, Loveland, Colorado, USA). For accuracy and performance check, a 500 platinum-cobalt unit's color standard solution (Cole-Parmer, USA) was used.

Turbidity was determined by 8237 absorptometric method using HACH DR890 colorimeter. For accuracy and performance check 4000 NTU Formazin stock solution (Cole-Parmer, USA) was used. Suspended solids were determined by the 8006 photometric method using a HACH DR890 colorimeter.

Sulfates were determined by SulfaVer 4 method which is equivalent to USEPA method 3754 for wastewater. Measurements were done by HACH DR890 colorimeter. Estimated detection limit is 4.9 mg/L SO_4^{2-} and standard deviation ±0.5 mg/L sulfate.

Manganese was determined by 8149 PAN method using HACH DR890 colorimeter. For accuracy and performance check 1000 mg/L Merck standard solution was used. Estimated detection limit is 0.002 mg/L Mn and standard deviation ± 0.013 mg/L Mn.

Table 1. Physical and chemical parameters determined in untreated acid mine drainage from "Robule" Lake and after each treatment step.

		Treated effluent	
Parameter	*Initial effluent*	*Pretreatment*	*Final treatment*
Color (PtCo)	476	0	0
Turbidity (NTU)	7	0	0
SS (mg/L)	0	0	0
pH	2.63	7.41	6.68
EC (mS/cm)	9.95	5.52	4.43
SO_4^{2-} (mg/L)	12000	5000	3500
Hg (mg/L)	0.061	0.005	0.001
Pb (mg/L)	0.080	0.006	0.002
V (mg/L)	0.127	0.007	0.002
Cr (mg/L)	2.210	0.010	0.003
Mn (mg/L)	59.003	1.011	0.336
Fe (mg/L)	610.000	0.060	0.010
Co (mg/L)	0.098	0.006	0.002
Ni (mg/L)	1.840	0.012	0.004
Cu (mg/L)	82.500	0.007	0.006
Zn (mg/L)	41.600	0.036	0.024

pH value, electrical conductivity (EC) and total dissolved solids (TDS) were determined by PHT-027 – water quality multi-parameter monitor (Kelilong Electron, Fuan Fujian, China). For calibration purpose Cole-Parmer standard solutions were used.

Results and discussion

The values of physicochemical parameters in the AMD before and after the treatment which combine the pretreatment/pH adjustment with CaO and final treatment by electrochemical methods are shown in Table 1, and decrease of the initial values following the treatment in Table 2. The highest values of heavy metals in the initial AMD have been found for iron (610 mg/L), copper (82.5 mg/L), manganese (59 mg/L) and zinc (41.6 mg/L). Extremely high concentrations of sulfates were also determined (12,000 mg/L).

Table 2. Decrease of the initial concentration of the parameters in the acid mine drainage from "Robule" Lake after pretreatment with CaO and final electrochemical treatment.

	Decrease of the initial concentration	
Parameter	*Pretreatment*	*Final treatment*
EC (mS/cm)	1.8	2.2
SO_4^{2-} (mg/L)	2.4	3.4
Hg (mg/L)	12.2	61.0
Pb (mg/L)	13.3	40.0
V (mg/L)	18.1	63.5
Cr (mg/L)	221.0	736.7
Mn (mg/L)	58.4	175.6
Fe (mg/L)	10166.7	61000.0
Co (mg/L)	16.3	49.0
Ni (mg/L)	153.3	460.0
Cu (mg/L)	11785.7	13750.0
Zn (mg/L)	1155.6	1733.3

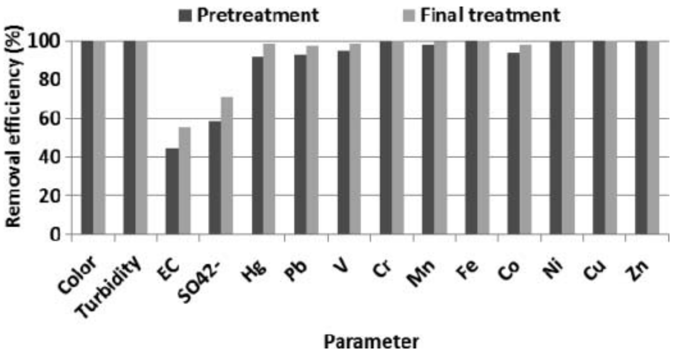

Fig. 1. The removal efficiencies of the parameters from the acid mine drainage of the "Robule" Lake after pretreatment with CaO and final electrochemical treatment.

After the pretreatment/pH adjustment with CaO pH of the effluent increased from 2.63 to 7.41 resulting in a 100% removal of the color and turbidity and 58.33% removal of the sulfates (Fig. 1), and the electrical conductivity was reduced for 44.52%. In the case of the selected heavy metals, the removal efficiency was 91.80% for Hg, 92.50% for Pb, 94.49% for V, 99.95% for Cr, 98.29% for Mn, 99.99% for iron and copper, 93.88% for Co, 99.35% for Ni and 99.91% for Zn. Decrease of the initial concentration of heavy metals ranged from 13.3 times for lead to 11785.7 times in the case of copper (Table 2). The formation of the poorly soluble hydroxides and gypsum was the most probable mechanism of the removal of heavy metals and sulfates at this stage of the treatment.

After the main electrochemical treatment including the electrocoagulation by electrochemically generated iron and aluminum flocs the decrease of the parameters in the solution compared to the previous treatment step was 1.2 times for EC and Cu, 1.4 times for sulfates, 1.5 times for Zn, 3 times for Pb, Mn, Co and Ni, 3.3 and 3.5 times for Cr and V respectively and 6 times for iron (Table 2). Because the color and turbidity were removed completely in the first step, the removal efficiencies of other considered parameters: EC, SO_4^{2-}, Hg, Pb, V, Cr, Mn, Fe, Co, Ni, Cu and Zn were 55.48%, 70.83%, 98.36%, 97.50%, 98.43%, 99.86%, 99.43%, 100%, 97.96%, 99.78%, 99.99% i 99.94%, respectively (Fig. 1).

The waste sludge collected during the first treatment experiment was collected, dried and used for the pH adjustment and pretreatment of the acid mine drainage in the second treatment experiment of AMD. The elemental concentrations in the effluent before/after the treatment and removal efficiencies were presented in Table 3. There was no significant difference in the quality of the final effluent pretreated with the waste sludge compared to this pretreated with CaO for any of the measured parameters.

Table 3. Physical and chemical parameters determined in the acid mine drainage from "Robule" Lake before and after combined treatment with waste sludge from the previous treatment of acid mine drainage followed by electrochemical methods.

Parameter	*Initial effluent*	*Final effluent*	*Removal efficiency (%)*
Color (PtCo)	476	0	100
Turbidity (NTU)	7	0	100
SS (mg/L)	0	0	–
pH	2.63	7.23	–
EC (mS/cm)	9.95	4.58	53.97
SO_4^{2-} (mg/L)	12000	3690	69.25
Hg (mg/L)	0.061	0.001	98.36
Pb (mg/L)	0.080	0.001	98.75
V (mg/L)	0.127	0.003	97.64
Cr (mg/L)	2.210	0.004	99.82
Mn (mg/L)	59.003	0.287	99.51
Fe (mg/L)	610.000	0.013	100.00
Co (mg/L)	0.098	0.001	98.98
Ni (mg/L)	1.840	0.003	99.84
Cu (mg/L)	82.500	0.006	99.99
Zn (mg/L)	41.600	0.027	99.94

The values of the parameters in the solid sample of the sludge, by-product of the purification of the wastewater from the "Robule" Lake are shown in Table 4 and the composition of the DIN38414-S4 leachate of the same sludge in Table 5. Due to the extremely low concentrations of the elements in the leachate and neutral pH value it could be concluded that such waste sludge represents the inert mixture of the hardly soluble phosphate, gypsum as well as hydroxide and oxy-hydroxides of iron and aluminum that bind heavy metals, and consequently, significantly reduce their possible release into the environment. Moreover, based on the preliminary results it is found that this sludge can be used for another cycle of the AMD treatment (pH adjustment, pre-treatment) instead of CaO and subsequently, due to its inertness it is also applicable for overlaying layer of the flotation waste heap during its recovery work.

Table 4. Elemental concentrations (C) and uncertainties (U) measured in the waste sludge by-product of the purification of the acid mine drainage with CaO/electrochemical methods.

Element	$C \pm U$
Mg (%)	0.26 ± 0.03
Al (%)	0.79 ± 0.07
P (%)	25.91 ± 0.24
S (%)	34.24 ± 0.31
Ca (%)	12.73 ± 0.04
Ti (ppm)	33 ± 4
V (ppm)	58 ± 5
Cr (ppm)	50 ± 1
Mn (%)	0.676 ± 0.005
Fe (%)	3.208 ± 0.001
Co (ppm)	13.22 ± 0.10
Ni (ppm)	184 ± 9
Cu (%)	0.3446 ± 0.0006
Zn (%)	0.1321 ± 0.0008
Hg (ppm)	0.40 ± 0.05
Pb (ppm)	8 ± 1

Table 5. The mass concentrations of the parameters determined in DIN38414-S4 leachate of the waste sludge by-product of the purification of the acid mine drainage with CaO/electrochemical methods.

Parameter	*Concentration*
pH	7.03
EC (mS/cm)	2.51
TDS (mg/L)	1750
SO_4^{2-} (mg/L)	870
Hg (mg/L)	< 0.001
Pb (mg/L)	< 0.001
V (mg/L)	< 0.001
Cr (mg/L)	< 0.001
Mn (mg/L)	0.011
Fe (mg/L)	0.028
Co (mg/L)	< 0.001
Ni (mg/L)	0.003
Cu (mg/L)	0.012
Zn (mg/L)	0.021

Conclusion

Preliminary results showed that the application of the combination of the electrochemical methods for the treatment of the acid mine drainage pretreated/pH adjusted with CaO resulted in the extremely high removal efficiencies of heavy metals from the waste effluent usually above 99%. The removal efficiency increased with increasing initial metal concentration. High degree of the removal of sulfates (over 70%) was also achieved. The concentration of iron in the treated effluent was reduced from 610 to 0.010 mg/L, copper from 82.5 to 0.006 mg/L, manganese from 59 to 0.336 mg/L and zinc from 41.6 to 0.024 mg/L. The concentrations of other heavy metals in the final effluent were below 0.005 mg/L.

The waste sludge from the combined treatment process could be reused for the pH adjustment/pretreatment of the acid mine drainage instead of CaO, and afterwards, due to its inertness, it could be used for overlaying layer of the flotation waste heap during its recovery work.

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