University of Belgrade
Technical Faculty in Bor and
Mining and Metallurgy Institue Bor

51st International October Conference on Mining and Metallurgy

PROCEEDINGS

Editors:

Prof. dr Srba Mladenović
Prof. dr Čedomir Maluckov

Bor Lake, Serbia,
October 16-19, 2019

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PREFACE

On behalf of the Organizing Committee, it is a great honor and pleasure to wish all the participants a warm welcome to the 51st International October Conference on Mining and Metallurgy (IOC 2019) held at Bor Lake, Serbia, 16 – 19 October 2019.

The IOC 2019 has been organized by the University of Belgrade, Technical Faculty in Bor, in cooperation with Mining and Metallurgy Institute Bor. It is devoted to presenting recent research results and advances in the fields of geology, mining, metallurgy, materials science, technology, environmental protection, and related engineering topics. The primary goal of IOC is to bring together academics, researchers, and industry engineers to exchange their experiences, expertise and ideas, and also to consider possibilities for collaborative research.

These proceedings include 81 papers from authors coming from universities, research institutes and industries in 15 countries: Bosnia and Herzegovina, Croatia, Japan, Kazakhstan, México, Montenegro, Poland, Romania, Russia, Slovenia, Turkey, Ukraine, Switzerland, Brasil and Serbia.

Financial assistance provided by the Ministry of Education, Science and Technological Development of the Republic of Serbia is gratefully acknowledged. The support of the sponsors and their willingness and ability to cooperate has been of great importance for the success of IOC 2019. The Organizing Committee would like to extend their appreciation and gratitude to all the donors and friends of the Conference for their donations and support.

We would like to thank all the authors who have contributed to these proceedings, and also to the members of the scientific and organizing committees, reviewers, speakers, chairpersons and all the Conference participants for their support to IOC 2019. Sincere thanks to all the people who have contributed to the successful organization of IOC 2019.

We look forward to welcoming you to the 52nd International October Conference on Mining and Metallurgy (IOC 2020), which will be held in October 2020.

On behalf of the 51st IOC Organizing Committee,
Prof. dr Srba Mladenović

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Abstract

The present work explored the effectiveness of Parsovići pyrophyllite ore as sorbent material for heavy metals such as Pb, Cu, Zn, Cd and Ni from aqueous solution. The analysis of chemical composition of PIR samples revealed the presence of silica and alumina besides Ca bearing phase; along with FTIR analysis which confirmed presence of hydroxyl groups attached to Si or Al, the composition of the ore sample indicates that this sample might be a prospective source for the effective metal sorbent. The preliminary batch sorption studies supported this hypothesis, indicating that the PIR sample poses highest affinity (mmol/g) in single ion solution towards Pb, followed by Zn, Cu, Ni and Cd. In multi metal solution the sorption of Zn was lowest. The sorption capacity under described operational conditions for Pb (single metal solution) was 47.25 mg/g.

Keywords*: pyrophilite, contaminated water, heavy metals, sorption*

1. INTRODUCTION

Among the many pollutants nowadays, heavy metals occupy a special place in the environment pollution, entering the food chain and transferring easily from one medium to another. Heavy metals belong to the group of very dangerous pollutants, greatly affecting all living organisms. Unlike organic pollutants, the metal ions do not degrade into harmless end products. The particular concern caused by these heavy metals is not only because of their potential toxicity but also of their accumulative character [1]. Because of these facts, special attention is directed to finding the best method to eliminate them from the environment. There are many conventional methods for removal of heavy metal ions from water and other solutions. These methods are ion exchange, solvent extraction, participation, phyto extraction, ultrafiltration, reverse osmosis, electro dialysis and adsorption [2]. One of the most used techniques, nowadays, for removing heavy metals is sorption, which was used in our experiment. Sorption is a physical and chemical process by which one substance becomes attached to another. This connection is enabled by either physical or chemical forms, depending on sorbent nature and sorbate characteristics. Numerous approaches have been studied for the development of cheaper metal sorbents, applying fly ash, peat, clays and related minerals, phosphate minerals, etc. Many of minerals like hydroxyapatite, rock phosphates, pyrophyllite, and zeolite are used as adsorbents for the removal of heavy metals [3, 4]. Based on the foregoing, in our experiment is tested a possibility of using raw material pyrophyllite like adsorbate for heavy metals. The research is based on ore body pyrophyllite from the deposit "Parsovići" from Bosnia and Hercegovina. The objective of the present studies described here was to investigate sorption characteristics of Pb^{2+}, Cu^{2+}, Ni^{2+}, Cd^{2+} and Zn^{2+} onto mention sorbent in sole and multi metal solution.

2. EXPERIMENTAL

The material used in this experiment is pyrophyllite from Bosnian deposit "Parsovići". The deposit of pyrophyllite ore in Parsovići, Konjic, is located around 25 km northwest of Konjic [5]. After receiving, the starting raw ore was grinded in vibratory disc mill "Siebtechnik - TS250" (Siebtechnik GmbH, Germany) that operates discontinuously in batch conditions, applying the pressure, impact and friction action. During the milling time, both grinded and coarse materials were continuously exposed to vibratory mill action. After milling, the grinded material was sieved. For the purpose of experiments presented in this paper, the granulation below 0.1 mm was used. The samples were dried at 105 ^{o}C until constant mass, marked as PIR and kept in desiccator prior to further use.

Sorption experiments were conducted in batch conditions at room temperature. Stock solutions were prepared by dissolving separately nitrate salts of Pb^{2+}, Ni^{2+}, Cd^{2+}, Cu^{2+} and Zn^{2+} (Merck) using distilled water. Five individual 1 mM solutions of zinc, copper, lead, nickel and cadmium were brought into contact with the solid substance (powder) of PIR at a ratio of 2 g/l. Therefore, the experiment was performed in two replicates, where 0.1 g of sample was added to 50 ml of volume of individual metal solutions. The samples were placed on the Heidolph Unimax 1010 shaker (200rpm) at contact time of 2 h. Multi metal solutions were made up of mention nitrate salts solution, and the sorption experiments were performed under the same operational conditions described above. The pHs of the solutions were measured during the whole contact time on pH meter SensION3 (Hach, SAD). The determination of heavy metals was performed by atomic absorption spectroscopy (AAS) on a Perkin Elmer AAS Analyst 300; this technique is used to determine the concentration of an individual element (metal) in the sample analyzed. The amount of metal ions adsorbed per mass unit of adsorbent was obtained by the following equation:

$$q = \frac{(C_i - C_e) \times V}{M} \quad (1)$$

where C_i and C_e (mg L^{-1}) are initial and equilibrium of metal ions concentration, respectively, M (g) is the mass of sorbent and V (L) is the volume of the of metal ions solution. Along with sorption capacity, removal efficiency was calculated as following:

$$R(\%) = (Ci - Ce) / Ci * 100 \quad (2)$$

3. RESULTS AND DISCUSSION

3.1 Pyrophyllite ore characterization

Pyrophyllite is a phyllosilicate mineral composed of aluminum silicate hydroxide: $Al_2Si_4O_{10}(OH)_2$. Pyrophyllite is abundantly found Al-Si clay mineral that has sorption properties thanks to the large surface area and negative layer charge in contact with water [6]. Generally, sorption onto clay materials can be two-dimensional, physical and/or chemical removal adsorption process; these processes can occur via two different mechanisms: (i) cation exchange in the interlayers resulting from the interactions between ions and negative permanent charge and (ii) formation of inner-sphere metal complexes through Si–O and Al–O groups at the both edge sites of the clay particles [7]. According to the results described in literature [5], the composition of pyrophyllite sample was (mass %): SiO_2 64.14%, Al_2O_3 15.92%, CaO 6.65%, Fe_2O_3 1.57%, MgO 1.06%, Na_2O 0.1%, K_2O 0.64%, FeO 0.37%, P_2O_5 0.18%, kr. H_2O 5.18%, LOI 9.46% and accompanying phases present in lower percentages titanium dioxide, sulfur trioxide and barium oxide (lower than 0.02%). Compared with other authors who investigated pyrophillite ore, e.g. pyrophillite from India, presented results show a little different composition of pyrophillite ore [8]. For example, in Indian deposits the contents of SiO_2 and CaO are lower for 11.4% and 5.02% respectively, while the Al_2O_3 and K_2O amounts are bigger for 12.22% and 8.57% respectively; the other components have very similar values. This induces lower cation exchange capacity of PIR compared to Indian ore, 59.69 and 123.8 meq/100g. Also, X-ray diffraction analyses have confirmed that the main mineral components present in the ore sample were: pyrophyllite (app. 50%), quartz (app. 30%), and carbonates (calcite app. 10% and dolomite less than 5 %), while the FTIR analysis revealed the presence of spectral lines characteristic of pyrophyllite and quartz, along with peaks those originating from vibrations in line –OH (1620 cm^{-1}) and outside the plane OH group (837 and 880 cm^{-1}) [5]. According to Panda et al [9], the most reactive surface functional groups on the geopolymer made up from pyrophillite mine waste are the surface hydroxyl groups attached to either Al or Si, and the sample PIR pose them.

3.2 Sorption results

The results of Gucek et al [6] show that pyrophyllite in water exhibits a negative surface charge within the range pH 2–12. Also, Prasad have showed that pH value have no significant impact on metal sorption by sericitic pyrophyllite from Jhansi [8], in the pH range from 5 to 7. Concerning this, the initial values of solution pH were at the range 5-5.5, while the final measured ranged from 5.7 (for Pb solution) to 7.3 (for Ni solution). This increase in pH value might be explained by different metal behavior. Thus, the chosen pH values enhance the removal efficiency of the metals avoiding metal precipitation at designated concentrations.

Regarding the contact time, many authors have concluded that equilibrium of metals sorption onto clay mineral surface is achieved in very short time period. For example, Prasad concluded that the time of 30 minutes was long enough to attain equilibrium in removal of Pb^{2+}, Cu^{2+} and Zn^{2+} from water solutions [8], while the geo polymer made up from pyrophillite ore attained equilibrium in less than 30 minutes [9]. Knowing that the sorption is a surface phenomenon, with rapid filling of active sites at the beginning of the process, and slower diffusion of the metal ions through the PIR pores, the authors have decided to fix the operational time to 2 h in order to ensure complete sorption.

Having in mind all previously described, the sorption experiments are conducted under chosen operational parameters, described in experimental part. The results of single metal and multi metal sorption experiments are presented on Figure 2.

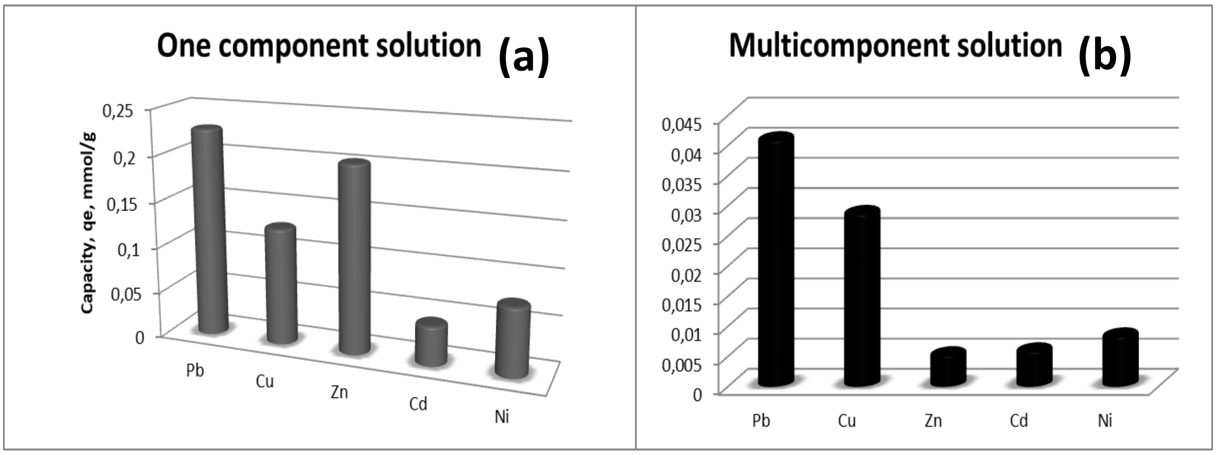

Figure 2. Preliminary results of PIR sorption (a) single metal solution and (b) multi metal solution

As it can be seen from Figure 2 (a) the sorption affinity under described operational conditions is biggest for lead ions in single metal solution. The sorption capacity of PIR for this metal was 47.25 mg/g which is equivalent to 0.23 mmol/g. Related to initial lead concentration which was app 1 mmol/L, this represents 53,08% of lead removal. The following metal ions are zinc and copper, with 42.04% and 27.9% of initial concentration removal. On the other hand, nickel and cadmium showed a small percentage of removal. In the case of multi metal solution (Figure 2. (b)), the lead and copper stood out with 53.1 mass.% and 35.1 mass.%, while for other metals the removal values were much lower.

The PIR sorption capacities for investigated metals in single water solution were: 47.25 mg/g (Pb), 13.32 mg/g (Zn), 8.10 mg/g (Cu), 4.81 mg/g (Cd) and 4.38 mg/g (Ni), which are much higher amounts than many other similar sorbents found in the literature. This is especially important to notify because the optimization of operational parameters as well as maximum sorption capacity should be investigated in the future.

4. CONCLUSION

Contamination of water by heavy metals in industries is an acute environmental problem worldwide. The preliminary investigation results presented in this paper are focused on sorption characteristics of PIR sample towards heavy metals such as Pb(II), Ni(II), Cu(II), Zn(II) and Cd(II). This material was found to be a potent sorbent of metal ions from single and multimetal water solutions through batch sorption studies. The obtained results in single metal solution have indicated that sorption efficiency in lead removal was the highest, with 47.25 mg/g, while the lowest was for Cd, 4.50 mg/g. In multicomponent metal solution, lead removal was highest again, while the lowest removal efficiency was with Zn, only 5.4% of initial concentration which was 13 mg/L. The presented results indicate that the Parsovići pyrophillite ore could be used as an inexpensive and promising adsorbent for removal of heavy metal ions. Further investigation on its application should be conducted.

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