THE INFLUENCE OF ADSORBENT MASS AND INITIAL ION CONCENTRATION ON THE CAPACITY AND EFFICIENCY OF LEAD (II) IONS ADSORPTION FROM AQUEOUS SOLUTIONS USING NATURAL BENTONITE AS ADSORBENT

ORIGINAL SCIENTIFIC ARTICLE

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ABSTRACT:
In this research, the results of bentonite characterization by XRF and SEM/EDS before and after adsorption are presented. The influence of adsorbent mass and initial ion concentration on the capacity and efficiency of adsorption of lead (II) ions using bentonite is determined by adsorption experiments for initial lead (II) concentrations of 200, 300 and 400 mg/L, adsorbent dosage of 0,5, 1 and 2 g, at pH 5, temperature 25 °C, stirring rate of 200 rpm, contact time between adsorbent and lead (II) ions of 2,5 minutes. The obtained results showed that with an increase in the adsorbent mass, the adsorption capacity decreases for all three tested initial concentrations, while the adsorption capacity increases with an increase in the initial concentration of Pb(II) ions for all three investigated masses of adsorbent. On the other hand, the adsorption efficiency increases with increasing adsorbent mass, while the adsorption efficiency decreases with increasing initial ion concentration.

KEYWORDS: adsorption; lead; bentonite; mass; concentration

INTRODUCTION

To ensure quality of life, protect human health, and safeguard the environment, the removal of lead from wastewater before it is discharged into natural water recipient is of vital importance [1, 2]. Lead is toxic to all organisms. Lead salts and organic lead compounds have the highest ecotoxicity. Lead affects chlorophyll synthesis in plants and reduces plant growth and development [3]. Lead is easily absorbed into the tissues of plants and animals, accumulates, and thus directly enters the food chain of humans [4]. In addition to living organisms, lead also accumulates in sediments and sludge [3].

Methods that can be used for the removal of Pb(II) ions from drinking water and wastewater include ion exchange, solvent extraction, reverse osmosis, precipitation, and adsorption. Among all these methods, adsorption has proven to be cost-effective (compared to ion exchange) and technically simple (compared to precipitation or reverse osmosis) [5].

The selection of an adsorbent is the first and fundamental step in the application of adsorption for the removal of heavy metal ions [6]. Adsorption using natural materials is a technique that offers several advantages over other methods used for heavy metal ion removal from wastewater, including cost-effectiveness, availability, environmental sustainability, low cost, high efficiency, and operational simplicity [7].

The term bentonite refers to a broad group of hydrated silicates with layered structures of colloidal dimensions in the range of 1 nm to to 2 μm [8]. The primary component of bentonite is the clay mineral montmorillonite. The name montmorillonite originates from the location of its discovery, Montmorillon, France. It consists of plate-like particles and is chemically represented as $(Na, Ca)_{0.33}(Al, Mg)_2Si_4O_{10}(OH)_2 \cdot nH_2O$, forming through the alteration of volcanic ash [7]. Montmorillonite has an amphoteric surface, whose charge depends on the pH value. Due to the presence of montmorillonite, bentonite exhibits a large specific surface area, high cation exchange capacity, and a significant number of acid-base centers on its surface. These properties, along with its widespread availability and low cost, make bentonite an effective adsorbent for heavy metals (Pb^{2+}, Fe^{2+}, Zn^{2+} i Cr^{3+}) from waste waters, significantly contributing to its application in environmental protection [9, 10]. The cations present in bentonite, Ca^{2+}, Mg^{2+}, Na^{+}, and K^{+}, can be exchanged with heavy metal ions in the order of their reactivity: $K^{+} > Na^{+} > Ca^{2+} > Mg^{2+}$, where higher reactivity corresponds to a greater tendency for ion exchange. The nature, composition, and characteristics of bentonite clays can vary from one location to another. As a result, the efficiency of heavy metal ion removal can also vary significantly [11].

EXPERIMENTAL

The characterization of the natural bentonite from the "Šipovo" deposit in Bosnia and Herzegovina was performed using X-ray fluorescence (XRF) and SEM/EDS analysis, before and after adsorption.

Experimental investigations on the removal of Pb(II) ions from aqueous solutions through adsorption using bentonite as an adsorbent were conducted in a batch process. Samples of 50 mL solutions were prepared with a standard Pb(II) solution with a concentration of 1000 mg/L ($Pb(NO_3)_2$ in 0.5 mol/L HNO_3). The samples were transferred into Erlenmeyer flasks, and the pH value 5 was adjusted using NaOH and HCl solutions of varying concentrations (0.01 mol/L to 0.1 mol/L).

After pH adjustment, certain amount of bentonite was added to each sample. The samples were stirred using a magnetic stirrer with a thermostat at a mixing speed of 200 rpm while maintaining the temperature at 25 °C for a contact time of 2.5 minutes. After the adsorption process, the samples were filtered using a vacuum pump and two filter papers (blue stripe). The Pb(II) ion concentration in the filtrate was determined using atomic absorption spectrophotometry.

The influence of mass and initial ion concentration on the capacity and efficiency of adsorption of lead (II) ions using bentonite was examined for three initial Pb(II) ion concentrations (200, 300, and 400 mg/L), and three adsorbent dosages (0,5, 1 and 2 g). Each experiment was conducted in triplicate, and the average Pb(II) ion concentration after adsorption was used for calculations.

The adsorption capacity of Pb(II) ions using bentonite was calculated by applying the equation [12, 13]:

$$q_t = \frac{(C_i - C_f) \times V}{m} \qquad (1)$$

where:
q_t – adsorption capacity of Pb(II) ions at time t, (mg/g),
C_i – initial concentration of Pb(II) ions in solution before adsorption, (mg/L),
C_f – final concentration of Pb(II) ions in solution after adsorption, (mg/L),
V – volume of the Pb(II) ion solution, (L),
m – mass of bentonite, (g).

To determine the adsorption efficiency of Pb(II) ions using bentonite, the following equation was applied [12, 13]:

$$E_a = \frac{C_i - C_f}{C_i} \times 100 \qquad (2)$$

where:
E_a – adsorption efficiency of Pb(II) ions (%),
C_i and C_f have the same meaning as in equation (1) for adsorption capacity.

The selected bentonite samples, after adsorption and filtration, were dried and ground, and then characterized using XRF analysis.

RESULTS AND DISCUSSION

The chemical composition (in the form of oxides) of bentonite before and after adsorption, determined by using XRF analysis, is presented in Table 1. As previously stated in their work A. Karic et al. [14], the natural bentonite from the "Šipovo" deposit primarily consists of SiO_2 (55.6%) and Al_2O_3 (24.1%), indicating that it is an aluminosilicate material. The high mass fraction of Al_2O_3 suggests the presence of aluminum oxides or substitution of Si with Al [25].Oxides such as Fe_2O_3 (8.71%), Na_2O (4.34%), MgO (2.40%), CaO (2.05%), and Cr_2O_3 (1.91%) are present in smaller quantities, while other oxides are present at less than 1% or in the ppm range.

The presence of MgO, Na_2O, and CaO indicates that these are the major exchangeable cations in bentonite [15], which is confirmed by the analysis results of the bentonite after adsorption, as the mass fraction of these oxides decreased, while the PbO oxide was present in an amount of 2.03 mass % after adsorption. The fraction of MnO, ZrO_2, and ZnO oxides also slightly decreased.

The results presented in Table 1 clearly indicate high percentages (mass %) of silicon and aluminum, which is consistent with expectations, as the chemical composition of the natural clay is largely determined by its phase composition, i.e., the presence of clay and non-clay minerals. The presence of calcium, magnesium, sodium, and potassium oxides can be explained by the fact that bentonite is a clay predominantly composed of smectite clay minerals, which contain exchangeable ions such as Ca^{2+}, Mg^{2+}, Na^{+}, and K^{+} in their interlayer spaces.

Table 1. Chemical composition of bentonite before and after adsorption determined by XRF analysis

Component	Bentonite composition	Bentonite composition after adsorption
SiO_2 (mass %)	55.6	54.2
Al_2O_3 (mass %)	24.1	24.3
Fe_2O_3 (mass %)	8.71	8.98
Na_2O (mass %)	**4.34**	**3.29**
MgO (mass %)	**2.40**	**2.34**
PbO (mass %)	/	2.03
CaO (mass %)	**2.05**	**1.67**
Cr_2O_3 (mass %)	1.91	2.01
TiO_2 (mass %)	0.665	0.666
K_2O (mass %)	0.299	0.332
Br (mass %)	0.281	0.471
SO_3 (mass %)	0.164	0.159
I (mass %)	/	0.810
Cl (ppm)	846	/
MnO (ppm)	836	792
ZrO_2 (ppm)	409	370
Rb_2O (ppm)	/	324
SrO (ppm)	244	257
CuO (ppm)	210	223
ZnO (ppm)	204	193
NiO (ppm)	/	202

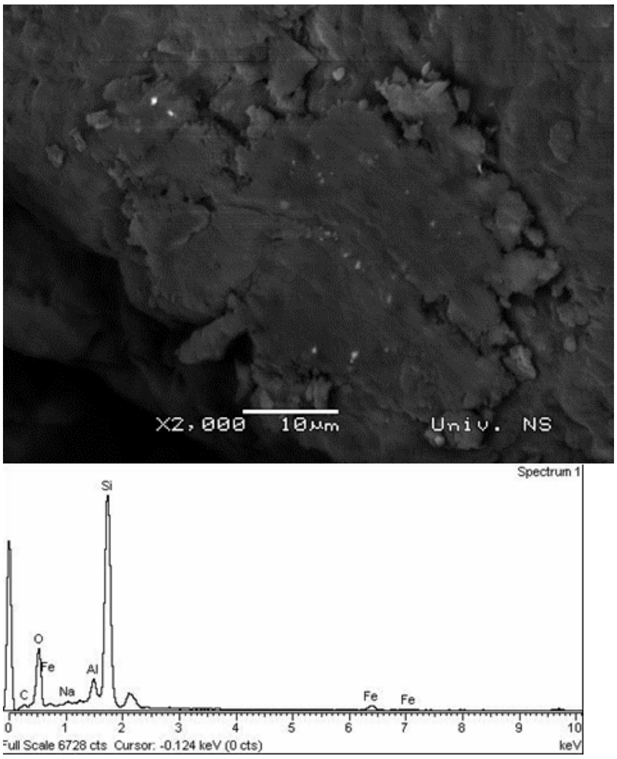

Figure 1. SEM image and EDS spectrogram of a pure bentonite sample

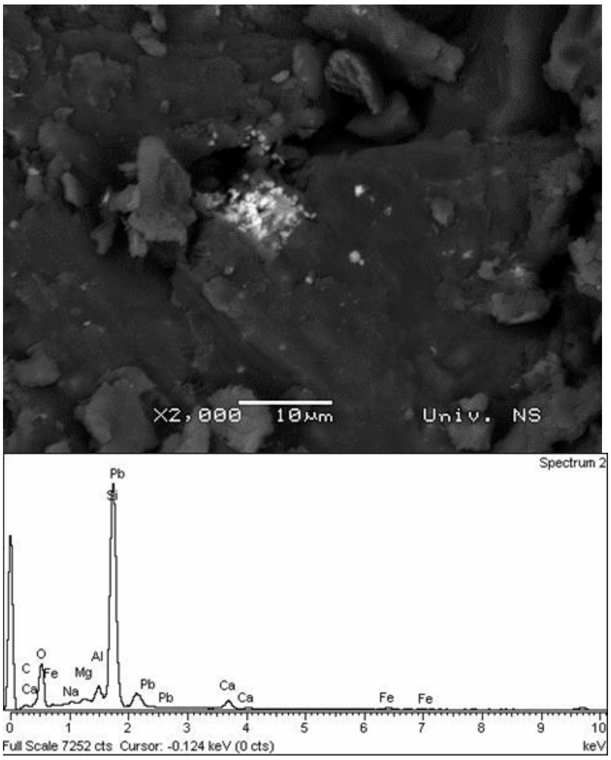

Figure 2. SEM image and EDS spectrogram of a bentonite after adsorption, for initial Pb(II) ion concentration of 400 mg/L. in selected points

SEM/EDS characterization of pure bentonite sample before and after adsorption, for initial Pb(II) ion concentration of 400 mg/L, is shown in Figures 1 and 2. As previously stated in their work A. Odobašić et al. [16], on SEM image of pure bentonite "fine powder" can be seen on the surface, which indicates that there is a developed porous structure in the powder structure. Presence of gaps in the material indicates that the structure of the shown particles is chaotic, irregular and that there is a lot of empty space in the structure. SEM image of the bentonite sample after adsorption (Figure 2) shows the places where the composition and structure of the bentonite has changed, which is also confirmed by the EDS spectrogram.

The mass of the adsorbent is an important experimental parameter in the adsorption process, as it influences both the adsorption capacity and efficiency. Furthermore, the adsorbent mass is also a key parameter for the economic optimization of the adsorption process. The effect of adsorbent mass and initial concentration of Pb(II) ions was investigated at pH 5, a temperature of 25°C, stirring speed of 200 rpm, and a contact time of 2.5 minutes. The results of the investigation of the influence of adsorbent mass and initial Pb(II) ion concentration on adsorption capacity and efficiency, using bentonite as the adsorbent are shown in Figures 3 and 4.

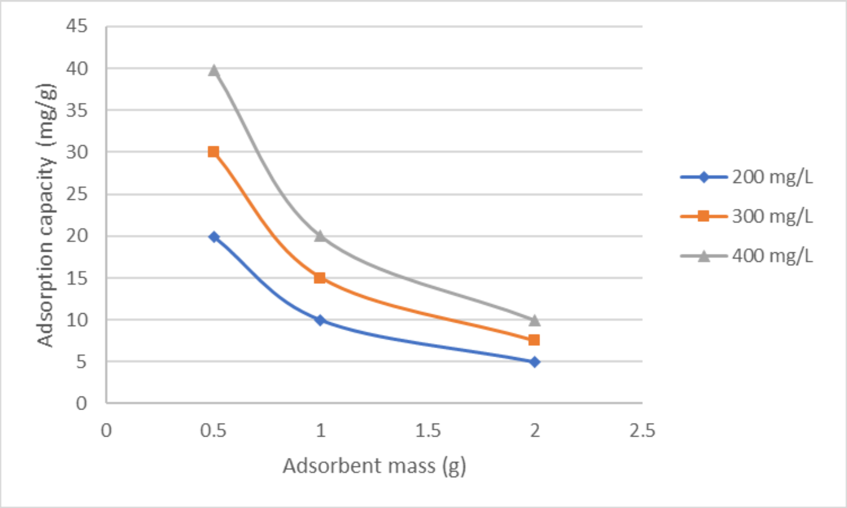

Figure 3. The influence of adsorbent mass and initial Pb(II) ion concentration on adsorption capacity

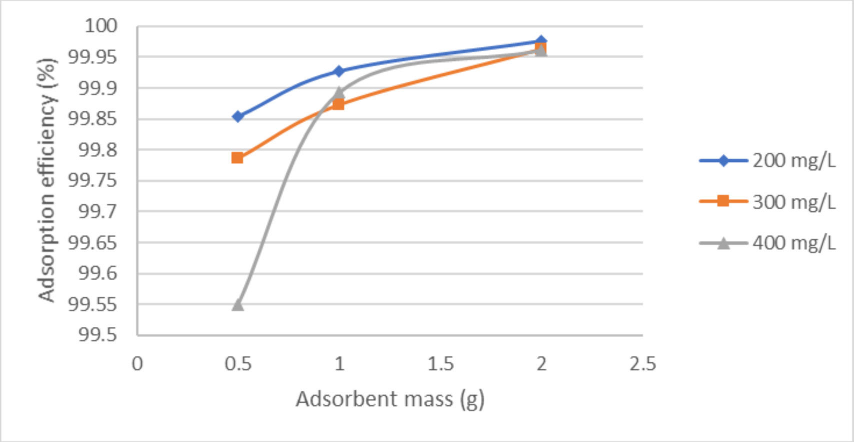

Figure 4. The influence of adsorbent mass and initial Pb(II) ion concentration on adsorption efficiency

Although it is expected that the adsorption capacity would increase with the increase in adsorbent mass due to a larger number of active sites in the solution, the results shown in Figure 3 indicate an opposite trend. The decrease in Pb(II) ion adsorption capacity with increasing bentonite mass can be explained in two ways.

First, during the adsorption process, binding sites on the surface may remain unsaturated, i.e., the adsorption capacity of the adsorbent is not fully utilized. Second, a larger amount of adsorbent may lead to particle aggregation, resulting in a reduced effective surface area, decreased availability of active sites, and an increased diffusion path length, all of which contribute to a reduction in the amount of adsorbed ions per unit mass [12, 17, 18]. Additionally, increasing the sorbent mass may not only reduce mixing efficiency but also hinder ion diffusion through the solution, which slows down mass transfer [19].

The decline in adsorption capacity with increasing adsorbent mass for the adsorption of Pb(II) ions was also confirmed in the studies of S. P. Singh et al. [11], H. Chen and A. Wang [20] for various tested adsorbents, as well as by M. R. Khan et al. [21], J. A. Hefne et al. [22], and Z. Melichova and L. Hromada [23] for bentonite.

On the other hand, the adsorption efficiency increases with increasing adsorbent mass, since a higher adsorbent dose provides more active sites for Pb(II) ion adsorption, resulting in a greater number of ions being adsorbed on the adsorbent surface. This has also been confirmed in previous studies by J. H. Potgieter [24], I. Chaari et al. [9] for other tested adsorbents, as well as by S. Al-Sharani [25], R. Naseem and S. S. Tahir [26], D. Xu et al. [27], J. A. Hefne et al. [22], M. Hamidpour et al. [17], D. John Babu et al. [28], and Z. Melichova and L. Hromada [23] for bentonite.

When observing the influence of the initial Pb(II) ion concentration on adsorption capacity, the diagram in Figure 3 indicates that the adsorption capacity increases with increasing initial Pb(II) ion concentration for all three tested adsorbent masses. This can be explained by the fact that more adsorption sites are occupied as the concentration of Pb(II) ions increases [15]. These findings are consistent with the results reported by N. Bektas et al. [29], J. H. Potgieter et al. [24], Y. El Mouzdahir et al. [8], and S. S. Gupta and K. G. Bhattacharyya [12] for various adsorbents, as well as by Z. Melichova and L. Hromada [23] for bentonite.

On the other hand, for a bentonite mass of 0.5 g, the adsorption efficiency is the lowest at an initial Pb(II) ion concentration of 400 mg/L, with amount of 99.5501%, and the highest at 200 mg/L, with amaount of 99.8536%. It can thus be concluded that under these conditions, the efficiency decreases with increasing initial Pb(II) ion concentration. Similar results were reported in studies by I. Chaari et al. [9] for clay, and by S. Al-Sharani [25], R. Naseem and S. S. Tahir [26], and Z. Melichova and L. Hromada [23] for bentonite. This decrease can be explained by the fact that increasing the Pb(II) ion concentration leads to a higher ratio of Pb(II) ions in solution relative to the number of available adsorption sites.

For a bentonite mass of 2 g, the adsorption efficiency remains almost the same across all three initial Pb(II) ion concentrations, with a variation of only 0.01%, which is consistent with earlier findings reported by J. H. Potgieter et al. [24] for clay. At higher adsorbent dosages, the number of available adsorption sites exceeds the number of Pb(II) ions present in the solution, making the adsorption efficiency independent of the Pb(II) ion concentration, i.e., the efficiency values remain approximately the same across different concentrations [20].

Considering that a bentonite mass of 1 g achieved an adsorption efficiency greater than 99.87% for all three initial Pb(II) ion concentrations, and that the increase in efficiency with a doubled adsorbent mass (2 g) was negligible (only 0.05%–0.07%), and taking into account the economic aspect of the process, it can be concluded that the optimal mass of the adsorbent, for this volume of solution and the given range of initial Pb(II) ion concentrations, is 1 g.

CONCLUSION

According to XRF analysis of bentonite before and after adsorption, MgO, Na_2O, and CaO were identified as the main exchangeable cations in the bentonite, as the mass fraction of these oxides was found to decrease. Furthermore, PbO was detected after adsorption at a content of 2.03 wt.%. SEM image of bentonite before the adsorption process showed a "fine powder" on the particle surface, suggesting the presence of a well-developed porous structure within the material. SEM image of bentonite sample after adsorption showed regions where changes in composition and structure had occurred. EDS analysis of bentonite samples before and after adsorption confirmed the XRF results, further verifying the adsorption of Pb(II) ions onto the bentonite.

The results of the study of the effect of adsorbent mass on adsorption capacity and efficiency showed that increasing the adsorbent mass leads to a decrease in adsorption capacity, while adsorption efficiency increases with adsorbent mass. Adsorption capacity increases with increasing initial Pb(II) ion concentration for all three tested adsorbent masses. For a bentonite mass of 0.5 g, adsorption efficiency decreases with increasing initial Pb(II) ion concentration, whereas at higher adsorbent masses, adsorption efficiency becomes independent of the Pb(II) ion concentration, remaining approximately the same across different concentrations. The optimal mass of bentonite, for a solution volume of 50 mL and an initial Pb(II) ion concentration range from 200 mg/L to 400 mg/L, is 1 g.

REFERENCES

[1] Mina B. Jovanović (2016). Uklanjanje jona metala iz vodenih rastvora korišćenjem zeolita: mehanizam, kinetika i primena u fluidizovanom sloju. *Doktorska disertacija, Univerzitet u Beogradu, Tehnološko-metalurški fakultet.*

[2] Asli Baysal, Nil Ozbek and Suleyman Akman (2013). Determination of Trace Metals in Waste Water and Their Removal Processes. *Waste Water – Treatment Technologies and Recent Analytical Developments,* Chapter 7, (145-171).

[3] Xiaoxing Zhang, Hui Liu, Jin Yang, Li Zhang, Binxia Cao, Libo Liu, and Weimin Gong (2021). Removal of cadmium and lead from aqueous solutions using iron phosphate-modified pollen microspheres as adsorbents. *Reviews on Advanced Materials Science.* 60: 365–376.

[4] Qiaoqiao Zhou, Nan Yang, Youzhi Li, Bo Ren, Xiaohui Ding, Hualin Bian, Xin Yao (2020). Total concentrations and sources of heavy metal pollution in global river and lake water bodies from 1972 to 2017. *Global Ecology and Conservation,* 22.

[5] Marin Ugrina (2014). Kemijska modifikacija prirodnog zeolita klinoptilolita-analiza ravnoteže i kinetike vezivanja kadmija i cinka iz vodenih otopina. *Doktorski rad, Sveučilište u Splitu, Kemijsko-tehnološki fakultet.*

[6] Geoffrey K. Kinuthia, Veronica Ngure, Dunstone Beti, Reuben Lugalia, Agnes Wangila, Luna Kamau (2020). Levels of heavy metals in wastewater and soil samples from open drainage channels in Nairobi, Kenya: comunity health implication. *Scientific reports*, 10:8434.

[7] N. K. Lazaridis, M. Jekel, A. I. Zouboulis (2003). Removal of Cr(VI), Mo(VI), and V(V) Ions from Single Metal

Aqueous Solutions by Sorption of Nanofiltration. *Separation Science and Technology,* 38:10, 2201-2219.

[8] Y. El Mouzdahir, A. Elmchaouri, R. Mahboub, A. ElAnssari, A. Gil, S. A. Korili, M. A. Vicente (2007). Interaction of stevensite with Cd^{2+} and Pb^{2+} in aqueous dispersion. *Applied Clay Science*, 35, 47-58.

[9] Islem Chaari, Emna Fakhfakh, Salima Chakroun, Jalel Bouzid, Nesrine Boujelben, Mongi Feki, Fernando Rocha, Fakher Jamoussi (2008). Lead removal from aqueous solutions by a Tunisian smectitic clay. *Journal of Hazardous Materials,* 156, 545-551.

[10] Murari Prasad, Sona Saxena (2008). Attenuation of divalent toxic metal ions using natural sericitic pyrophyllite. *Journal of Environmental Management,* 88, 1273-1279.

[11] S. P. Singh, L. Q. Ma, M. J. Hendry (2006). Characterization of aqueous lead removal by phosphatic clay: Equilibrium and kinetic studies. *Journal of Hazardous Materials,* B136, 654-662.

[12] Susmita Sen Gupta, Krishna G. Bhattacharyya (2008). Immobilization of Pb(II), Cd(II) and Ni(II) ions on kaolinite and montmorillonite surfaces from aqueous medium. *Journal of Environmental Management,* 87, 46-58.

[13] Mabrouk Eloussaief, Mourad Benzina (2010). Efficiency of natural and acid-activated clays in the removal of Pb(II) from aqueous solutions. *Journal of Hazardous Materials,* 178, 753-757.

[14] Amna Karić, Amra Odobašić, Edisa Papraćanin, Azra Halilović (2025.) Influence of temperature and pH value on the capacity and efficiency of lead (II) ions adsorption from aqueous solutions using natural bentonite as adsorbent. 15th Scientific/Research Symposium with International Participation „METALLIC AND NONMETALLIC MATERIALS“ B&H, 24th and 25th April 2025.- Proceedings, 2566-4344

[15] Joseph Ketcha Mbadcam, Solomon Gabche Anagho, Julius Ndi Nsami, Adelaide Maguie Kammegne (2011). Kinetic and equilibrium studies of the adsorption of lead (II) ions from aqueous solution onto two Cameroon clays: Kaolinite and smectite. *Journal of Environmental Chemistry and Ecotoxicology,* 3(11), 290-297.

[16] Amra Odobašić, Indira Šestan, Amna Karić, Melisa Ahmetović, Husejin Keran (2025). Characterization and possibility of bentonite regeneration after adsorption of Pb and Cu ions. International Research Journal of Engineering and Technology (IRJET), Volume 12, Issue 8, 2395-0056.

[17] Mohasen Hamidpour, Mahmoud Kalbasi, Majid Afyuni, Hossein Shariatmadari, Gerhard Furrer (2011). Sorption of lead on Iranian bentonite and zeolite: Kinetics and isotherms. *Environmental Earth Sciences.*

[18] Runping Han, Weihua Zou, Zongpei Zhang, Jie Shi, Jiujun Yang (2006). Removal of copper(II) and lead(II) from aqueous solution by manganese oxide coated sand I. Characterization and kinetic study. *Journal of Hazardous Materials* B137, 384–395.

[19] Abida Kausar, Haq Nawaz Bhatti, Gillian MacKinnon (2013). Equilibrium, kinetic and thermodynamic studies on the removal of U(VI) by low cost agricultural waste. *Colloids and Surfaces B: Biointerfaces* 111, 124–133.

[20] Hao Chen, Aiqin Wang (2007). Kinetic and isothermal studies of lead ion adsorption onto palygorskite clay. *Journal of Colloid and Interface Science,* 307, 309-316.

[21] Khan M. R., Hegde R. A., Shabiimam M. A. (2017). Adsorption of Lead by Bentonite Clay. *International Journal of Scientific Research and Management (IJSRM),* 5 (07), 5800-5804.

[22] J. A. Hefne, W. K. Mekhemer, N. M. Alandis, O. A. Aldayel, T. Alajyan (2008). Kinetic and thermodynamic study of the adsorption of Pb(II) from aqueous solution to the natural and treated bentonite. *International Journal of Physical Sciences,* 3(11), 281-288.

[23] Zuzana Melichova, Ladislav Hromada (2013). Adsorption of Pb^{2+} and Cu^{2+} Ions from Aqueous Solutions on Natural Bentonite. *Polish Journal of Environmental Studies,* 22(2), 457-464.

[24] J. H. Potgieter, S. S. Potgieter-Vermaak, P. D. Kalibantonga (2006). Heavy metals removal from solution by palygorskite clay. *Minerals Engineering,* 19, 463-470.

[25] S. Al-Shahrani (2010). Removal of lead from aqueous solutions using Saudi activated bentonite. *WIT Transactions on Ecology and the Environment*, 135.

[26] R. Naseem, S. S. Tahir (2001). Removal of Pb(II) from aqueous/acidic solutions by using bentonite as an adsorbent. *Wat. Res.,* 35(16), 3982-3986.

[27] D. Xu, X. L. Tan, C. L. Chen, X. K. Wang (2008). Adsorption of Pb(II) from aqueous solution to MX-80 bentonite: Effect of pH, ionic strength, foreign ions and temperature. *Applied Clay Science,* 41, 37-46.

[28] D.John Babu, G.Kalyani, H.Joga Rao, Y.Prasanna Kumar, P.King (2012). Biosorption characteristics of Lead onto Bentonite Clay, as Low-cost natural biosorbent. *International Journal of Scientific & Engineering Research,* 3(8).

[29] Nihal Bektas, Burcu Akman Agim, Serdar Kara (2004). Kinetic and equilibrium studies in removing lead ions from aqueous solutions by natural sepiolite. *Journal of Hazardous Materials,* B112, 115-122.