15th Scientific/Research Symposium with International Participation
„METALLIC AND NONMETALLIC MATERIALS“ B&H, 24th and 25th April 2025

INFLUENCE OF TEMPERATURE AND PH ON THE CAPACITY AND EFFICIENCY OF LEAD (II) IONS ADSORPTION FROM AQUEOUS SOLUTIONS USING NATURAL BENTONITE AS ADSORBENT

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Keywords: adsorption, lead, bentonite, temperature, pH

ABSTRACT

In this research, the resulsts of bentonite characterization by XRF before and after adsorption are presented. The influence of temperature and pH on the capacity and efficiency of adsoption 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 1 gram, stirring rate of 200 rpm, contact time between adsorbent and lead (II) ions of 2,5 minutes, for temperature of 25, 30 and 40 °C, and pH of 5 and 8.5. The obtained results showed that at pH 5 adsorption capacity and efficiency decrease with increasing temperature, while for pH 8.5 adsorption capacity and efficiency increase with increasing temperature.

1. INTRODUCTION

1.1. Lead

Lead enters the environment as a result of natural processes and anthropogenic activities. Natural processes include pedogenic breakdown of minerals and the translocation of the resulting products, as well as sedimentation due to dust storms, volcanic eruptions, and forest fires [1]. Lead has become one of the main environmental pollutants and is identified as a global issue due to its presence in batteries, ammunition, automotive fuel, and exhaust gases after fuel combustion. Rainwater collected near highways can contain significant concentrations of lead (over 50 mg/dm^3). Lead pollution is contributed by mining and smelting, lead exploitation, alloy production and metal surface finishing, battery recycling, lead glass production, cables, plastics, and pulp, the use of lead-based paints and pigments, and lead-containing pipes in water supply systems, continuous use of fertilizers and pesticides, as well as the and urban and industrial waste. Lead also enters the environment from photographic materials, ceramic glazes, and cosmetics. Global lead production has exceeded 3 million tons per year [1, 2, 3, 4, 5, 6].

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 [5, 7]. 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 [2]. Lead is easily absorbed into the tissues of plants and animals, accumulates, and thus directly enters the food chain of humans [8]. In addition to living organisms, lead also accumulates in sediments and sludge [2].

1.2. Adsorption

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) [3].

Adsorption is a process in which certain chemical species (atoms, ions, or molecules) from a liquid or gaseous phase accumulate on the surface of a solid phase upon contact [2]. Adsorption is a highly suitable technique for treating wastewater with low concentrations of heavy metals [9].

The efficiency of the adsorption process depends on various parameters, including the properties of the adsorbent (surface area, pore structure, surface chemistry, particle size, etc.) and the properties of the adsorbate (such as molecular weight, molecular structure, oxidation state, particle size, dissociation ability, polarity, etc.). Additionally, several physicochemical parameters, such as solution pH, temperature, contact time, adsorbent mass, adsorbate concentration, and the presence of other ions, also influence the adsorbate/adsorbent interaction during the adsorption process. Studying these parameters can provide valuable insights into the adsorption mechanism, which is crucial for selecting the appropriate adsorbent and operating conditions for efficient adsorption of a specific adsorbate [2,10].

The most influential parameter is the pH of the solution from which the metal is adsorbed [11], as it directly affects the adsorption capacity, the adsorption mechanism, the ionic species of the metal in the solution, and the functional surface groups present on the adsorbent. With changes in the solution's pH, the functional groups on the adsorbent surface can be protonated or deprotonated, resulting in different surface charges on the adsorbent. This leads to electrostatic attraction or repulsion between the charged adsorbate and adsorbent particles [10].

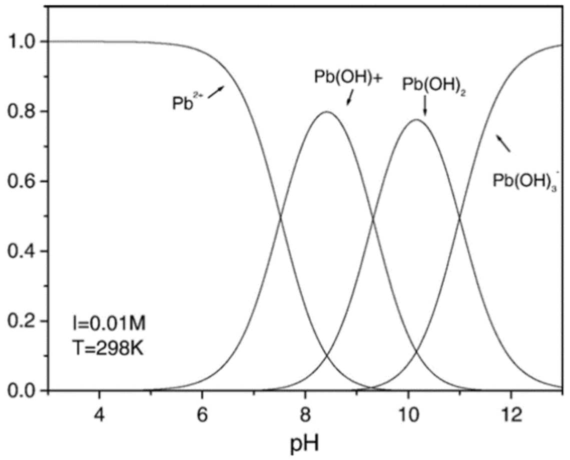


Figure 1. The distribution of lead ionic species in an aqueous medium as a function of solution pH [12]

As shown in Figure 1, at pH lower than 7, the free Pb^{2+} ion is the dominant ionic species. At a pH 7.5, the ionic species Pb^{2+} and $Pb(OH)^+$ are present in approximately equal concentrations, while in the pH range between 7.5 and 9, $Pb(OH)^+$ becomes the dominant species. In the pH range between 9 and 11, $Pb(OH)_2$ dominates, whereas at pH greater than 11, the predominant ionic species is $Pb(OH)_3^-$. Due to this ionic distribution, the best results for Pb(II) ion adsorption in most studies have been achieved at pH between 4 and 7.

The temperature of the solution plays a significant role in the adsorption process. An increase in temperature typically enhances the mobility of heavy metal ions due to the rise in their kinetic energy and also increases the diffusion rate of metal ions within the adsorbent. Furthermore, temperature affects the equilibrium and kinetics of the adsorption process [10], as well as the interaction between the adsorbent and metal ions, primarily by influencing the stability of metal ions in the solution and the stability of metal-adsorbent complexes [11].

The thermal effect of adsorption can be either endothermic or exothermic [2]. In the case of an exothermic process, increasing the temperature would reduce the efficiency of metal ion removal, whereas for an endothermic process, the opposite trend is observed. Therefore, optimizing the adsorption temperature is essential. However, it is preferable for adsorption to occur at ambient temperature to avoid additional heating or cooling of the solution. Additionally, conducting the adsorption process at higher temperatures increases operational costs [10].

1.3. Adsorbents

The selection of an adsorbent is the first and fundamental step in the application of adsorption for the removal of heavy metal ions [13]. 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 [14].

A wide range of materials in nature possess ion-exchange and adsorption properties and can be used for the removal of heavy metal ions from wastewater [4]. These include tannin-, lignin-, and chitosan-rich materials, marine algae, peat moss, mold, moss, dead biomass, sawdust, and agricultural waste (e.g., corn husks, peanut shells, walnut shells, rice husks, olive pits, grape stalks, lemon and orange peels, and coffee and tea waste), as well as widely available natural materials such as zeolite, apatite, and bentonite [6, 9, 15, 16, 17, 18, 19].

1.3.1. Bentonite

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 [20]. 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 [14]. Montmorillonite has an amphoteric surface, whose charge depends on the pH. 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 [21, 22]. 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 [18].

2. EXPERIMENTAL PART

The characterization of the natural bentonite from the "Šipovo" deposit in Bosnia and Herzegovina was performed using X-ray fluorescence (XRF) analysis.

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 three different initial Pb(II) ion concentrations (200, 300, and 400 mg/L). A standard Pb(II) solution with a concentration of 1000 mg/L ($Pb(NO_3)_2$ in 0.5 mol/L HNO_3) was used for solution preparation. The samples were transferred into Erlenmeyer flasks, and the pH (5 and 8.5) were adjusted using NaOH and HCl solutions of varying concentrations (0.01 mol/L to 0.1 mol/L).

After pH adjustment, 1 g 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 desired temperature (25, 30, and 40°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 pH and temperature was examined for all three initial Pb(II) ion concentrations. 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 [23, 24]:

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

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_k – 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 [23, 24]:

$$E_a = \frac{C_i - C_f}{C_i} \times 100 \tag{2}$$

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.

3. RESULTS AND DISCUSSION

The chemical composition (in the form of oxides) of bentonite before and after adsorption was determined using XRF analysis, and the results are presented in Table 1. As can be seen, 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.

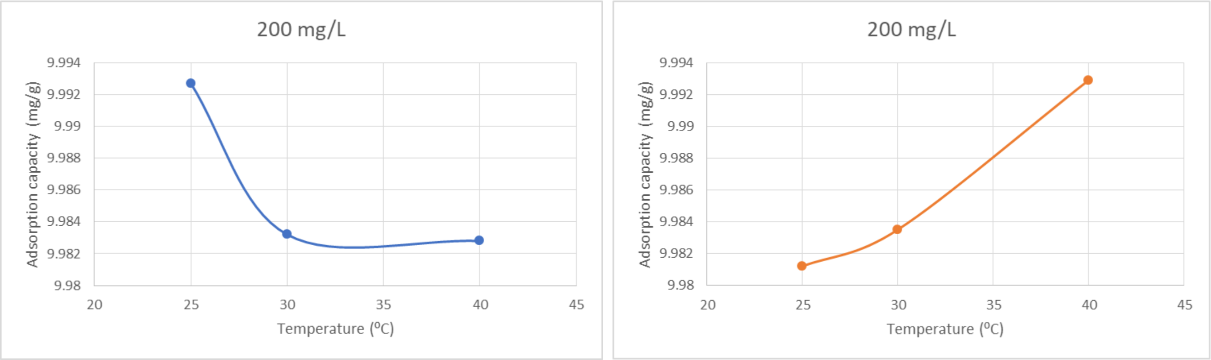
The presence of MgO, Na_2O, and CaO indicates that these are the major exchangeable cations in bentonite [25], which is confirmed by the analysis results of the bentonite after adsorption, as the mass fraction of these oxides decreased, while the PbO oxide was present in an amount of 2.03 mass % after adsorption. The fraction of MnO, ZrO_2, and ZnO oxides also slightly decreased.

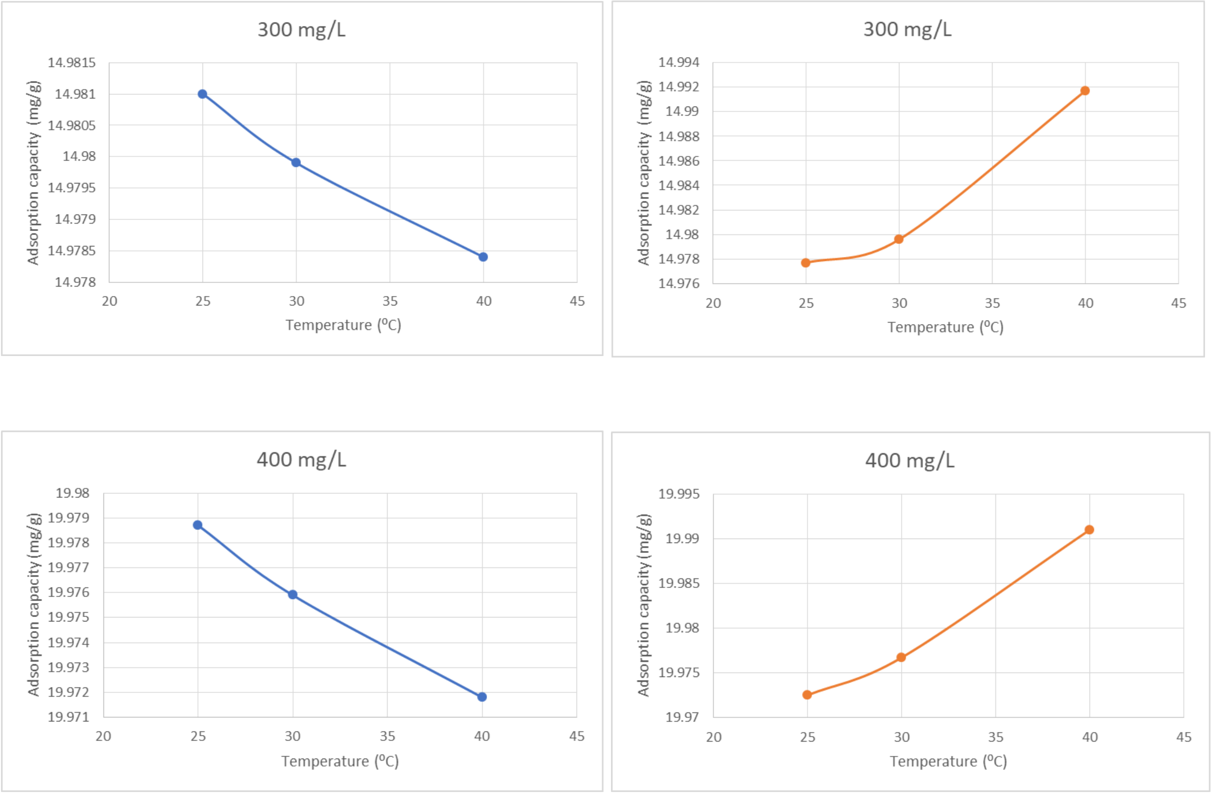
Table 1. Chemical composition of bentonite before and after adsorption determined by XRF analysis

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

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.

The results of the examination of the effects of temperature and pH on adsorption capacity and efficiency, for three initial concentrations of Pb(II) ions, are shown in Figures 2 and 3.





a) pH 5 *b) pH 8.5*

Figure 2. The effect of temperature and pH on the adsorption capacity for three different initial concentrations of Pb(II) ions.

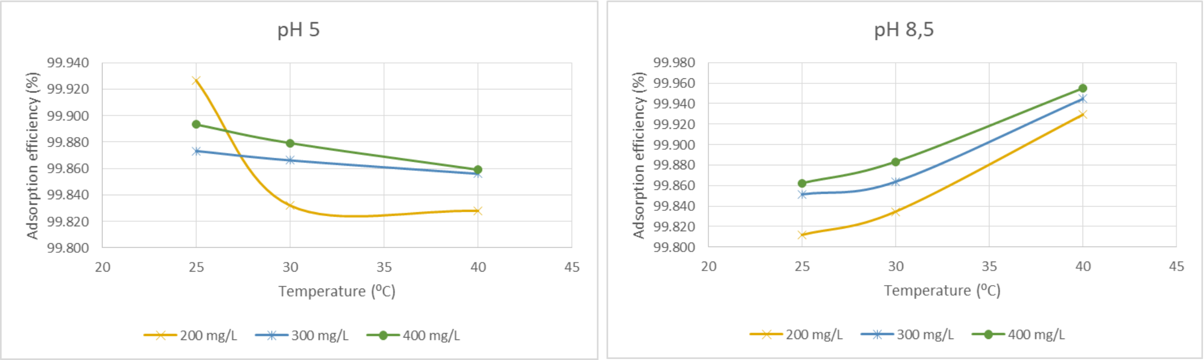


Figure 3. The effect of temperature and pH on adsorption efficiency for three different initial concentrations of Pb(II) ions.

The results showed that adsorption is efficient at both pH tested at all temperatures (>99%), which was expected due to the distribution of lead ionic species in aqueous solutions. At pH below 7, the free Pb^{2+} ion is the dominant ionic species, while at pH between 7.5 and 9, the $Pb(OH)^+$ ionic species dominates. At pH lower than 5, H^+ ions are present in high concentrations and compete with Pb(II) ions for the available adsorption sites on the bentonite surface, thus reducing the adsorption efficiency of Pb(II) ions [3, 8, 26]. At pH between 9 and 11, $Pb(OH)_2$ dominates, while at pH above 11, the $Pb(OH)_3^-$ ionic species dominates, which results in low adsorption efficiency at these pH [12].

As seen in the diagrams presented in Figures 2 and 3, the adsorption capacity and efficiency at a pH of 5 decrease with increasing temperature for all three initial concentrations of Pb(II) ions, while at a pH 8.5, the adsorption capacity and efficiency increase with temperature. However, considering the initial concentration of 200 mg/L and a temperature of 25°C, the adsorption capacity at pH 5 is 9.9927 mg/g, while at pH 8.5 it is 9.9812. If the same concentration is observed at a temperature of 40°C, the adsorption capacity at pH 5 is 9.9828, while at pH 8.5 it is 9.9929. Based on these results, it can be concluded that at pH 8.5 and a temperature of 40°C, the capacity reached the same as at pH 5 and a temperature of 25°C (the difference is 0.0002 mg/g). Therefore, it can be concluded that a pH 5 is more optimal compared to a pH 8.5, as no additional heating is required to achieve the same capacity, resulting in lower process costs. Results from other researchers also show that the highest adsorption efficiency of Pb(II) ions using bentonite is achieved at pH ranging from 3 to 6 (P.C. Mishra and R.K. Patel [4]), from 4.5 to 7 (M. Hamidpour et al. [26]), at pH 6 (J.A. Hefne et al. [27]), and at pH 5 (S. Al-Sharani [8] and D. John Babu et al. [28]).

Based on the obtained results, it can be concluded that the adsorption process at a pH 5 is exothermic, while at a pH 8.5 it is endothermic. Results published in other studies show that the adsorption process of Pb(II) ions onto bentonite can be either exothermic (D. Xu et al. [12]) or endothermic (S. Wang et al. [29], R. Naseem and S.S. Tahir [30], V.J. Inglezakis et al. [31]), depending on the other process parameters.

4. CONCLUSION

XRF analysis of natural bentonite from the "Šipovo" deposit in Bosnia and Herzegovina showed that it mainly consists of SiO_2 (55.6%) and Al_2O_3 (24.1%), indicating that it is an aluminosilicate material. 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. The presence of MgO, Na_2O, and CaO suggests that these are the main exchangeable cations in bentonite, which is further confirmed by the post-adsorption analysis results, as the mass percentage of these oxides decreased and the PbO oxide was present at 2.03 mass % after adsorption. The results of the examination of the effects of temperature and pH showed that adsorption is effective at both tested pH, 5 and 8.5, at all temperatures (>99%). It was determined that the adsorption capacity and efficiency at pH 5 decrease with increasing temperature for all three initial concentrations of Pb(II) ions, while at pH 8.5, the adsorption capacity and efficiency increase with incrising temperature. At pH 8.5 and 40°C, the capacity achieved wich was similar to the that was obtained at pH 5 and 25°C, with a difference of 0.0002 mg/g. Therefore, pH 5 is more optimal compared to pH 8.5, as no additional heating is required to achieve the same capacity, resulting in lower process costs.

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