

The Influence of Structural Parameters of Georgian Natural and Modified Mordenite over its Adsorption Properties Towards Lead Ions

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ABSTRACT

The potential of Georgian natural Mordenite has been studied as a low-cost, eco-friendly adsorbent for the removal of lead (II) ions from aqueous solutions in various experimental environments. The natural and modified Mordenite structural characterization was carried out using XRD, SEM-EDS, XRF, methods. The following parameters were studied for adsorption: dependence of solution pH, adsorbent dose, and initial concentration of lead (II) ions. The results of the optimization study showed that a high dose of adsorbent and a low concentration of Pb (II) ion increase the sorption. The optimum pH was found to be 6-8. Sorption capacity increased along with pH. Langmuir and Freundlich isotherms were used to model the adsorption data. Correlation coefficient values (R^2) were 0.975 and 0.922, respectively. Isotherm studies showed that the uptake equilibrium is best described by the Langmuir adsorption isotherm. The maximum sorption capacity was found to be 11.439 mg/g. The research results showed that natural Georgian Mordenite can be used to remove Pb (II) ions from aqueous solutions.

Keywords: mordenite, structural parameters, adsorption, lead, solution.

INTRODUCTION

An important concern of the modern world is the accumulation of a large amount of harmful waste in the environment. Among them is the pollution of wastewater. Water is essential for life on Earth; The level of freshwater pollution is increasing every day, which is mainly caused by the increase in production, high level of agriculture and energy consumption, as well as weak management. Globally, millions of tons of waste and toxic substances are released into the environment, such as industrial, domestic, food, mining, and agricultural waste. They are regularly generated and enter the environment without any purification. Consequently, the accumulation of inorganic and organic substances dangerous to health increases in water systems.

Contamination of water with heavy metals from various toxins is a serious problem (Matouq et al., 2015a). When their concentration reaches a certain limit, it poses a significant threat to the

health of the environment, animals, and society. Lead is one of the most toxic heavy metals (Ayodhya, 2023). Even its small concentration, it can endanger the nervous system, gastrointestinal tract, reproductive system, liver, and brain.

There are various methods of wastewater treatment, which can be divided into three main classes: physical, chemical, and biological methods (Sahu et al., 2023). Chemical water purification technologies are very expensive in terms of energy; they require a lot of energy and material costs. The waste left after the use of chemical technology cannot be reused, so physical methods are actively used, which mainly include reverse osmosis, membrane filtration, and adsorption. The main disadvantage of membrane filtration is its high cost (Amy et al., 2017). The biological process of water purification involves the removal of dissolved and suspended organic compounds using natural microorganisms. Due to certain technical limitations, their use is restricted.

The process is time-consuming and requires a large working area (Fawcett-Hirst et al., 2021). Literature data confirm (Guo & Wang, 2019) that adsorption and ion exchange constitute some of the most popular and attractive alternative methods of water purification. By developing the appropriate innovative technology, it is possible to clean wastewater with high quality. For this, it is necessary to select cheap raw materials. For the removal of heavy metals, adsorption is considered to be one of the most popular techniques compared to other methods due to its low cost, abundant availability, high absorbency, simple operation mode, and biodegradability (Hu & Xu, 2020).

Today, the purification of wastewater from heavy metals using adsorption processes is considered a simple, cheap, and effective method. The efficiency of this method depends significantly on the nature of the sorbent. In this regard, natural zeolites are recognized and among them the most common and used – Clinoptilolite and Mordenite, which are found in several regions of Georgia. At the same time, improved methods of modification (activation) of zeolites are being developed, which make the sorbent even more effective. Different types of zeolites have different adsorption capacities. Mordenite's unique adsorption and cation-exchange properties, high content in tuff, thermal stability, and acid resistance allow raising the issue of its widespread use. Mordenite has a fairly low Si/Al ratio (5:1), which leads to good adsorption of some heavy metals, including lead (Zhang et al., 2021). The study of the sorption of heavy metal ions using mordenite is very rare.

This study showed that the sorption of lead ions is significantly dependent on the initial lead concentration and adsorption conditions. Comparative characterization of the original and modified Mordenite is very important in evaluating the adsorption effect. The main goal of this study was to optimize the conditions of adsorption of lead ions by Mordenite (initial and modified). To increase the level of adsorption, Mordenite (MOR) was modified with acid (DE-MOR) and alkali (MOR-OH). Effects of variables including initial lead concentration, contact time, and temperature on adsorption yield. Langmuir and Freundlich isotherm models were used to determine the kinetic parameters. Moreover, the thermodynamic parameters of adsorption were calculated.

MATERIALS AND METHODS

From different types of zeolites, Mordenite was selected as a sorbent with high ion exchange capacity, high surface area, high acid- and thermal stability, and low Si/Al ratio. Natural Georgian Mordenite from Ratevani deposit was used.

Various analytical methods were used in the study of natural and modified Mordenite: scanning electron microscopy (SEM), energy-dispersive spectroscopy (EDS), X-ray diffractometry (XRD), and X-ray fluorescence (XRF). Adsorption parameters were measured using Atomic Absorption Spectrometry method (AAS).

The initial model solution of lead (Pb^{2+}), used in experiments, was prepared by dissolving 1.5999 g of $Pb(NO_3)_2$ in deionized water, filled to the brim in a 1 dm³ volumetric flask. Adsorbates (0–200 ppm) were prepared from this solution by serial dilution. The concentration of heavy metals was measured on an atomic absorption instrument (Perkin Elmer AAnalyst200). To ensure the quality, the non-cooling graph was built on 3 points; as a result, a linear calibration graph ($R^2 \geq 0.997$) was obtained. 0.1 N solutions of NaOH and HNO_3 were used to adjust the pH (Milwaukee 150 pH meter).

The Mordenite from the Ratevani deposit was washed with distilled water and dried at 105 °C, a part of the sample was treated three times with increasing concentrations of hydrochloric acid (1N, 2N, 3N) in a water bath. The duration of each treatment is one hour. The ratio of zeolite: acid was 1:10. The treated Mordenite was washed with distilled water until a negative reaction to chlorine ion. The obtained sample was dried at 105 °C.

To obtain the MOR-OH form, the initial, washed sample was treated three times with increasing concentrations (1N, 2N, 3N) of a mixture of sodium and potassium alkalis (1:1 ratio). The duration of each treatment was one hour. The ratio of zeolite: alkali 1:10. The treated Mordenite was washed with distilled water until the pH was neutral.

The X-ray phase analysis of the initial and modified Mordenite samples was performed with X-ray diffractometer (XRD) (X-ray diffractometer by PANalytical, MPD X, PERT PRO PW3040 / 60). For typical powder samples, data were collected over a 2θ angle data range from $\approx 5^\circ$ to 90° , which are pre-scanned in an X-ray scanner. The morphology and elemental analysis of the sorbents were carried out using SEM-EDS under the

following analytical conditions: VEGA 3, LMU cathode of LaB 6 was used as the source of electrons. The accelerating voltage was 0–30 kV, and the resolution was 2.0 nanometers at 30 kV accelerating voltage. The maximum magnification reaches 1,000,000. The chemical composition of the sorbents was analyzed by the energy-dispersive X-ray fluorescence method (XRF).

Firstly, 50 cm³ of the initial solution of Pb²⁺ (5, 10, 20, 40, 60, 80, 100 ppm) was added to 1 gram of sorbent and mixing was done for 1 hour. The self-absorbed mixture was then filtered and the lead content in the filtrate was determined by using the Atomic Absorption method.

The adsorbent of different weights (0.1 g, 0.5 g, 1.0 g, 1.5 g, 2.0 g, 5.0 g) was added to the initial solution of 50 cm³ of Pb²⁺ where the optimal sorption (10 ppm) was obtained and stirred for 1 hour.

50 cm³ of an initial 10 ppm Pb²⁺ solution was added to 1 gram of sorbent, with adsorbate pH ranging from 2 to 10. Contact time was 1 hour. The suspension was filtered immediately and analyzed with an Atomic Absorption Spectrometer.

The adsorption capacity was calculated by the formula (Matouq et al., 2015b):

$$E = \frac{(C_0 - C)V}{m} \quad (1)$$

where: E (mg/g) – the equilibrium adsorption capacity;

C_0 and C (mg/dm³) – initial and equilibrium concentrations of a lead ion in the solution;
 V (dm³) – adsorbate volume and m (g) amount of adsorbent.

Study of the adsorption isotherm: the adsorption isotherm gives an idea about the sorption process and the degree of surface coverage by the adsorbate. This helps to select a suitable sorbent and determine the required sorbent dose for the process.

Langmuir isotherm: The Langmuir isotherm model is one of the most commonly used adsorption models, where sorption occurs only by monolayer formation. This isotherm predicts that intermolecular forces decrease rapidly with increasing distance. The Langmuir isotherm equation has the following form (Guo & Wang, 2019b):

$$q_e = \frac{q_{\max} K_L C_e}{1 + K_L C_e} \quad (2)$$

The linear form of the Langmuir isotherm is expressed by the equation (Manyangadze et al., 2020):

$$\frac{C_e}{q_e} = \frac{1}{q_{\max} K_L} + \frac{C_e}{q_{\max}} \quad (3)$$

where: C_e – the equilibrium concentration of adsorption (mg/dm³);

q_e – the amount of adsorbed metal cations per mass unit (mg/g);

q_{\max} (mg/g) and K_L (dm³/mg) – Langmuir constants, which are related to the maximum monolayer adsorption capacity and adsorption rate, respectively;

q_{\max} and K_L – are determined from the graph of $1/C_e$ versus $1/q_e$.

Freundlich isotherm: The Freundlich adsorption isotherm is an empirical equation used to describe a heterogeneous system (Li et al., 2018), It is expressed by the equation (Georgescu et al., 2018):

$$q_e = K_F C_e^{1/n} \quad (4)$$

The well-known logarithmic form of the Freundlich isotherm is given by the following equation (Huang et al., 2017):

$$\ln C_e = \ln K_F + (1/n) \quad (5)$$

where: C_e (mg/dm³) – the equilibrium concentration of the adsorbate;

q_e (mg/g) – the amount of adsorbed metal cation per unit mass;

K_F and n – Freundlich constants. n indicates how favorable the adsorption process is; K_F – defined as the adsorption or distribution coefficient. It is the amount of adsorbate that is adsorbed per unit mass of adsorbent at equilibrium (DeMessie et al., 2022). Adsorption in the range $0.1 < 1/n < 1$ is favorable.

RESULTS AND DISCUSSION

XRF characterization

The chemical composition of the obtained samples by EDXRF is represented in Table 1. Mordenite acid treatment resulted in an increase in the Si/Al ratio from 5.8 to 6.2. H⁺ ions of hydrochloric acid break the Al-O bond (Huntley et al., 2021).

Zeolite crystals are partially dissolved when the Mordenite from Georgian deposit is treated with NaOH and KOH solutions. Prolonged contact with alkali causes the amorphization of the outer surface and pores too, and high concentration of

Table 1. The results measurement of the initial and modified mordenite mineral composition using XRF

Name	Oxide content, %									
	SiO ₂	Al ₂ O ₃	CaO	MgO	Na ₂ O	K ₂ O	Fe ₂ O ₃	TiO ₂	P ₂ O ₅	Si/Al
MOR	70.6	10.79	3.55	0.29	2.00	4.4	1.77	0.12	0.19	5.8
De-MOR	71.29	10.03	1.96	0.15	1.40	2.2	1.06	0.09	0.18	6.2
MOR-OH	67.98	9.09	3.45	0.18	3.15	5.04	1.72	0.11	0.19	6.6

alkali and long contact with Mordenite can cause the amorphization of structure. It is known that alkali modification is used to increase the porosity of zeolites (Zhu et al., 2021).

XRD analysis

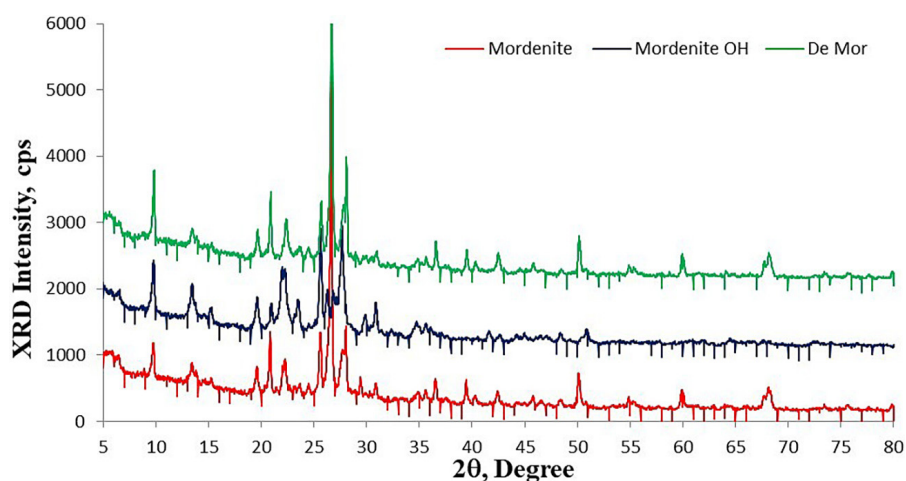
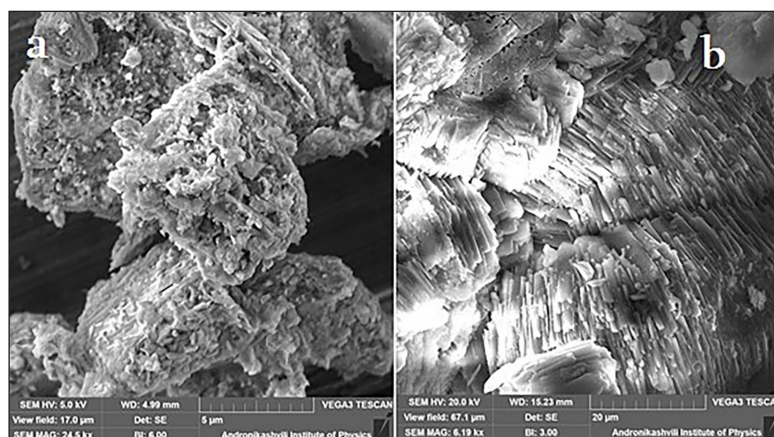
XRD patterns for initial and De-MOR and MOR-OH forms are shown in Figure 1. As it can be seen from the diffractograms for the studied three sorbents, all the diffraction peaks are actually at the same positions (with insignificant angular shifts), which indicates that no structural changes occurred during acid and alkali modification.

The results showed that Georgian natural Mordenite mainly consists of the mordenite phase. In addition to the mordenite phase, the mineral also contains quartz and clay impurities.

Therefore, rather mild conditions of acid and alkali treatment caused only insignificant dealumination and de-cationization of the samples but led to the removal of impurity phases and cations from Mordenite channels without breaking the structure.

SEM-EDS

Microscopic observations showed that the microstructures of the initial and modified forms

**Figure 1.** XRD patterns for MOR, MOR-OH, and H-MOR**Figure 2.** SEM images natural (a) and modified mordenite (b)

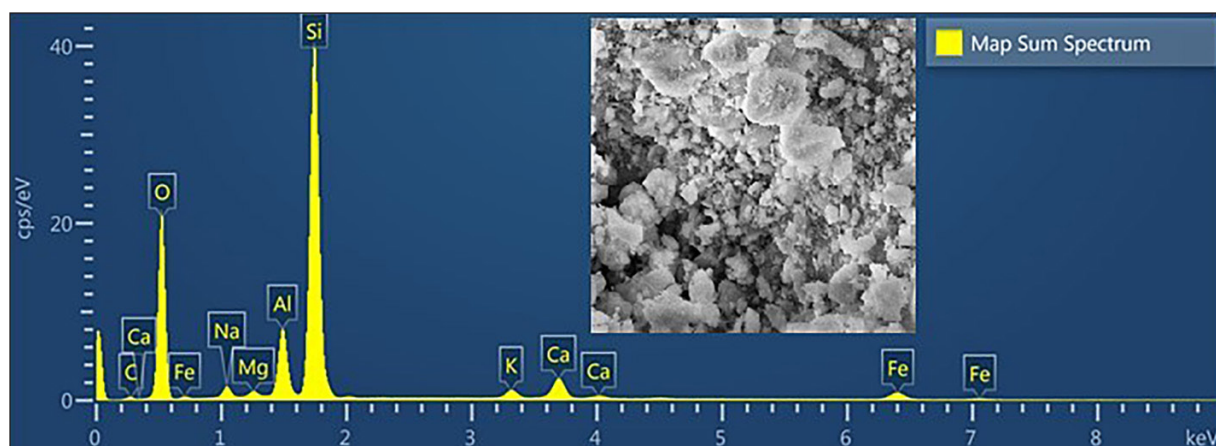


Figure 3. EDS analysis showing the elemental composition of natural mordenite and a scanned image

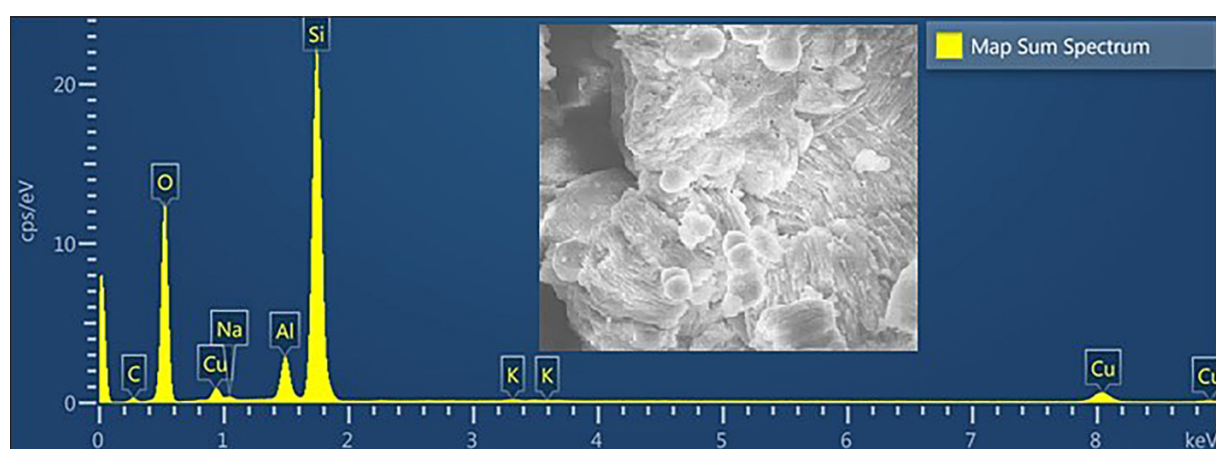


Figure 4. EDS analysis showing an elemental composition of DE-MOR and scanned image

of zeolites give quite a similar picture. The natural zeolite was treated with 1 M hydrochloric acid to remove the interfering substances from the surface and to obtain a clean surface as shown in Figure 2.

The chemical composition of the natural and modified Mordenite was analyzed by EDS, as shown in Figures 3 and 4. EDS is an analytical method used to determine the elemental composition and perform chemical characterization of zeolite samples. The morphology of the initial zeolite is represented by dense formations with strong agglomerates and irregularly shaped particles of various sizes. As a result of physical and mechanical influences on the zeolite (grinding) and modification, the surface structure of the material changes.

Effect of contact time and concentration

Absorption of Pb^{2+} ions occurs mainly in the first 10 minutes, which emphasizes the efficiency

of the adsorbent (Figure 5). The initial concentration of the adsorbate solution has a great influence on the sorption degree because a fixed amount of adsorbent can absorb a fixed amount of Pb^{2+} . Also, as can be seen from the graph in Figure 6, when the initial concentration of lead ions increases, the adsorption capacity increases. In case of high concentration, the degree of adsorption decreases. The maximum sorption of Pb^{2+} ions occurred in 40 minutes, since then there have been no significant changes. Sorption can be divided into two stages. Initially, a significant part of the lead ions were adsorbed very quickly, on average in 5 minutes. In the next stage, the adsorption was reduced and the equilibrium is reached in 40 minutes, after which the dynamic equilibrium was established (from 40 to 60 minutes). At equilibrium, the adsorption capacity increases from 0.25 to 5 mg/g, (in intervals of the initial concentrations from 5 to 100 mg/l) so the initial concentrations increase from 5 to 100 mg/l. At the initial stage, rapid adsorption may

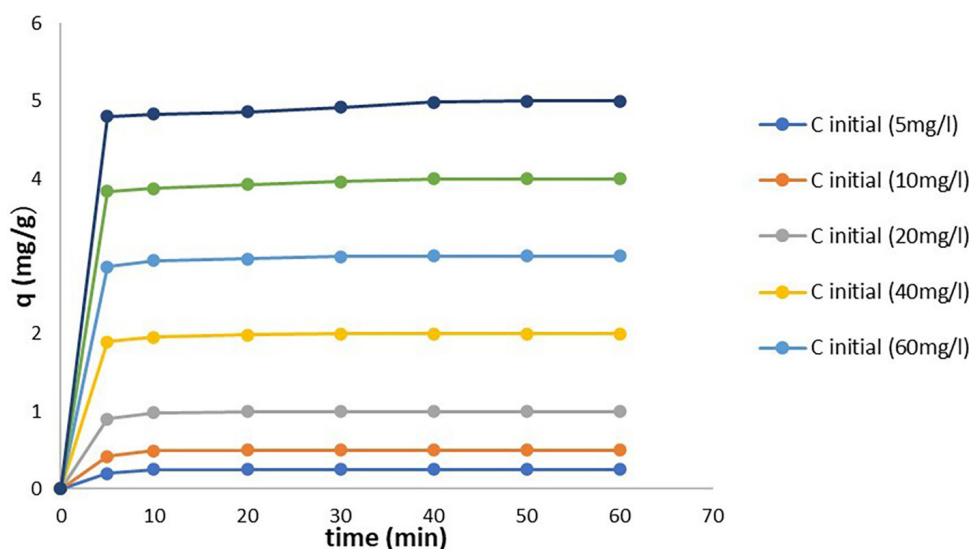


Figure 5. Effect of agitation time and initial concentrations of adsorbates' (T: 25 °C, pH: 6, agitation rate: 200 rpm)

occur at the expense of filling the large channels on the surface. However, over time, the active sites become saturated, leading to equilibrium after 40 min (Ofudje et al., 2020).

Effect of adsorbent mass

Adsorption of lead ions on the initial and modified Mordenite was studied for different amounts of adsorbent (0.1; 0.5; 1.0; 2.0 g). The initial concentration of lead ions is 30 mg/l (Fig. 6). As it can be seen from the graph, the degree of adsorption of Pb (II) ions increases along with the increase in the amount of sorbent. Lead ions were adsorbed to a high degree even in the case

of 0.1 grams, 100% removal occurs from 2 g. Therefore, 2 g of Mordenite is considered to be the optimal dose of sorbent in this study.

Effect of pH

The removal of metal ions from an aqueous solution depends on the pH of the solution, since pH affects the surface charge of the adsorbent, the degree of ionization of functional groups, and the type of metal ion (Ogunlalu et al., 2021). The dependence of the adsorption of lead ions on different pH is shown in Figure 7. Alkali and acid modification did not change the influence of pH on adsorption value.

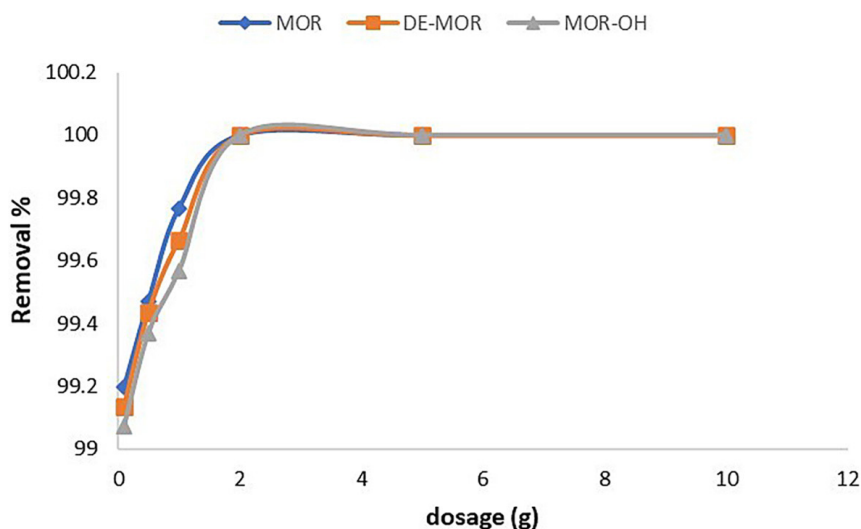


Figure 6. The effect of adsorbent dose on the adsorption of lead ions using natural and modified Mordenite (C_0 : 30 mg/l, T: 25 °C, pH: 6, agitation rate: 200 rpm)

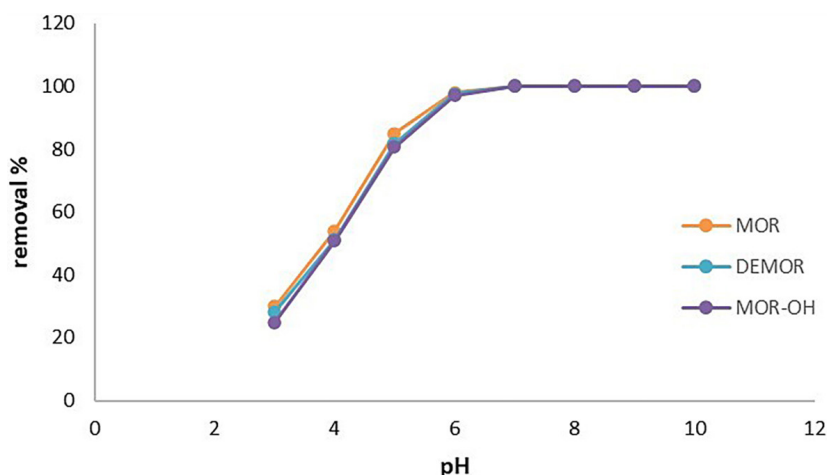


Figure 7. Effect of solution pH on uptake of lead (II) ions on natural and modified Mordenite ($C_0 = 30$ mg/l, $T = 25$ °C, agitation rate – 200 rpm)

Adsorption occurred even at low pH values. This may be due to the formation of stable complexes with the functional groups on the surface of the adsorbent. Also, at low pH values, the surface of Mordenite is covered by H^+ ions, which reduces the interaction of lead ions with Mordenite adsorption centers (Khan et al., 2021). For the pH range above 7, the lead ions present are hydrolyzed and the precipitate may be adsorbed as hydroxide.

Adsorption isotherms

Analysis of equilibrium data is important in developing equations that can be used to characterize different materials. The equilibrium adsorption isotherm is one of the most important forms of information, using which it is possible to determine the mechanism of adsorption process systems, to develop and optimize various operating procedures (Arshadi et al., 2014).

The experimental results of lead adsorption on zeolite were analyzed according to well-known Freundlich and Langmuir isotherm models.

The main criterion for selecting the most appropriate isothermal model is the average percentage error and deviation from experimental values (Ahmadi et al., 2022).

The Langmuir and Freundlich isotherms used for lead ion adsorption analysis are shown in Figures 8 and 9. The data obtained from the adsorption experiments (Table 2) fit well with both models, indicating that the adsorption sites are non-uniform and non-specific. The maximum adsorption capacity (q_{max}) from the Langmuir isotherm is 11.439 mg/g. From the Freundlich isotherm, the value of n is greater than 1, indicating favorable adsorption conditions. By comparing the correlation coefficients, the isothermal fit can be arranged as Langmuir > Freundlich. Therefore, the experimental results of adsorption fit best with the Langmuir isotherm model.

Table 2. Langmuir and Freundlich isotherm parameters for adsorption systems

Isotherm Type	Parameters					Note
Langmuir $\frac{C_e}{q_e} = \frac{1}{q_{max} K_L} + \frac{C_e}{q_{max}}$ (3)	$\frac{1}{C_e}$	$\frac{1}{q_e}$	q_{max} (mg/g)	K_L (L/mg)	R^2	$q_{max} = \frac{1}{intercept}$ $K_L = \frac{1}{q_{max} * slope}$ $R^2 > 0$, monolayer adsorption
	28.571	0.200	11.439	21.751	0.975	
	20.000	0.168				
	6.250	0.125				
	4.348	0.100				
	0.765	0.084				
Freundlich $q_e = K_F C_e^{1/n}$ (4)	$\ln C_e$	$\ln q_e$	$\ln K_f$	n	R^2	$\ln K_f = intercept$ $\frac{1}{n} = slope$ $R^2 > 0$, multilayer adsorption
	-3.352	1.609	2.197	4.196	0.922	
	-2.996	1.791				
	-1.832	2.077				
	-1.469	2.300				
	0.267	2.474				

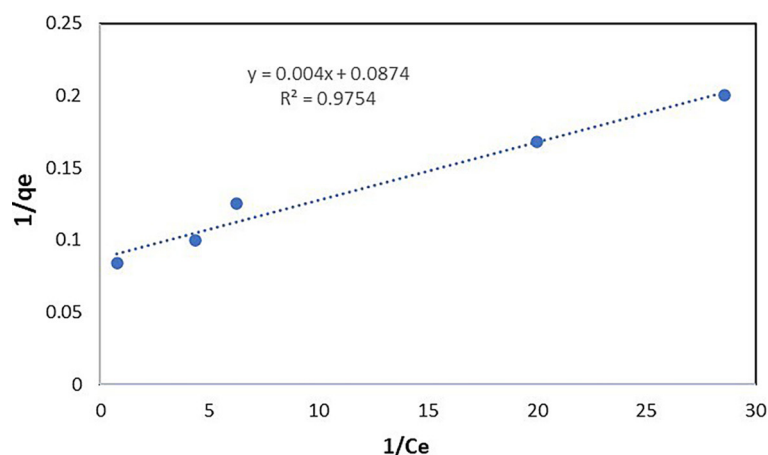


Figure 8. Langmuir isotherm model for adsorption system using natural Mordenite

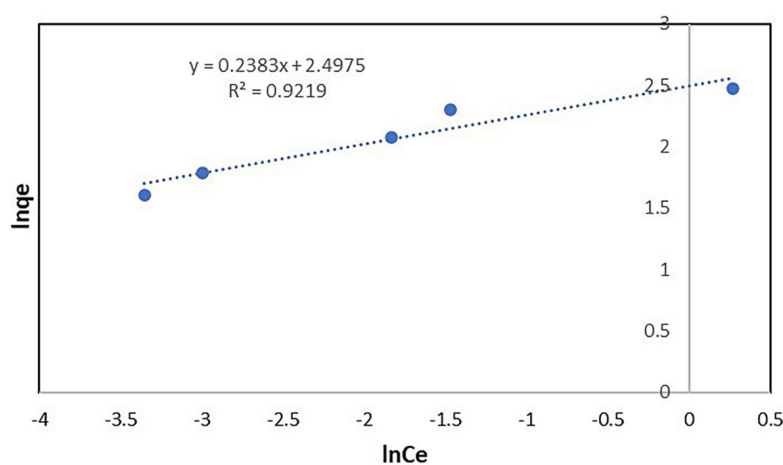


Figure 9. Freundlich isotherm model for adsorption system using natural Mordenite

CONCLUSIONS

The adsorption properties of Mordenite towards lead (II) ions have been studied. The study of its structural characteristics was carried out by XRD, SEM-EDS, and EDXRF. Surface modification of Mordenite has a negligible effect on the sorption ability, therefore, for the adsorption of lead (II) ions, it is possible to use initial Mordenite without pretreatment. It should be noted that the sorption takes place to a higher degree when the sorbent dose increases in progress. Sorption also depends on contact time, solution pH, and initial lead concentration, but equally for all three samples. As it turned out, the adsorption of lead (II) is spontaneous which is an indicator of good economic value. The adsorption capacity does not depend on the insignificant changes in structure and chemical composition of natural Mordenite.

Two isotherms were used to model the adsorption results, which fit the experimental data

well, although the Langmuir isotherm offers a better correlation to the kinetic data.

On the basis of this study, it is clear that Georgian natural Mordenite can be a useful adsorbent for absorbing lead (II) ions from polluted environments without any pre-treatment.

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