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Kinetic and Equilibrium Studies of Cd (II) Adsorption from Aqueous Solutions Using Natural Clay

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ABSTRACT

This study investigates the kinetics and equilibrium of cadmium Cd (II) ion removal from aqueous solutions using natural clay collected from Al-Khums, Libya, as a low-cost adsorbent. Batch adsorption experiments were conducted to evaluate the effects of contact time, solution pH, adsorbent dosage, initial Cd(II) concentration, and temperature on adsorption performance. E. The optimum adsorption conditions were pH 6.0, an adsorbent dosage of 3 g, and an equilibrium time of 60 min. Under these conditions, the maximum Cd (II) removal efficiency reached 94.3%, and the adsorption efficiency increased with increasing temperature, indicating a favorable effect of temperature on the adsorption process. The Langmuir isotherm provided the best fit to the equilibrium data, suggesting predominantly monolayer adsorption, while the pseudo-first-order kinetic model adequately described the adsorption behavior. These findings demonstrate that the natural clay from Al-Khums is an effective, inexpensive, and environmentally friendly adsorbent for the removal of Cd (II) ions from contaminated aqueous solutions.

الحركية والتوازن في إزالة أيونات الكادميوم باستخدام الطين من المحاليل المائية

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الكلمات المفتاحية:
الكادميوم (II)
الامتزاز
الطين الطبيعي
الحركية
الاتزان

الملخص

تتناول هذه الدراسة حركية وتوازن إزالة أيونات الكادميوم Cd (II) من المحاليل المائية باستخدام الطين الطبيعي المستخرج من مدينة الخمس، ليبيا، كمادة مازة منخفضة التكلفة. أُجريت تجارب امتزاز متقطعة لتقييم تأثير زمن التلامس، ودرجة حموضة المحلول، وجرعة المادة الماصة، والتركيز الابتدائي لأيونات الكادميوم، ودرجة الحرارة على كفاءة الامتزاز. وقد تحققت ظروف الامتزاز المثلى عند درجة حموضة 6.0، بجرعة مادة ماصة 3 غرامات، وزمن توازن 60 دقيقة. في ظل هذه الظروف، بلغت كفاءة إزالة الكادميوم القصوى 94.3%، وازدادت كفاءة الامتزاز مع ارتفاع درجة الحرارة، مما يشير إلى تأثير إيجابي لدرجة الحرارة على عملية الامتزاز. أظهر نموذج لانغمويير أفضل تطابق مع بيانات التوازن، مما يشير إلى امتزاز أحادي الطبقة، بينما وصف نموذج الحركية من الرتبة الأولى الزائفة سلوك الامتزاز بدقة. تُبين هذه النتائج أن الطين الطبيعي مادة مازة فعالة وغير مكلفة، وصديقة للبيئة لإزالة أيونات الكادميوم من المحاليل المائية الملوثة.

1. Introduction

Heavy metals are extensively utilized in several industrial processes and manufacturing facilities, including mining and metal-processing activities, paper industry, paint plants, paper mills, electroplating, fertilizer and chemical industry, pharmaceutical industry and detergent industry. These industries are potential sources of heavy metal ions like zinc (II), copper (II) and cadmium (II) in liquids and industrial waste [1]. The contamination of drinking water by heavy metals is a significant environmental and public health concern due to their toxicity and potential adverse effects even at relatively low concentrations. The World Health Organization (WHO) has established specific guideline values for individual heavy metals in drinking water based on their toxicity and potential health risks. [2].

The effective removal of heavy metal ions from wastewater and aqueous solutions has become extremely important to prevent their harmful effects on humans, ecosystems and health. Cadmium is considered a heavy metal that is highly toxic to human health and

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various biological systems. This metal is considered to be of particular importance because it is non-degradable and enters water bodies through various industries such as the production of metal, pesticides, phosphate fertilizers, textile mills, electroplating, refining processes, and battery and dye production [3.4].

Cadmium can cause a range of acute and chronic problems and damage, including kidney damage, high blood pressure, lung swelling, kidney, lung and liver damage, and cancer [5]. Therefore, there is great scientific interest in removing low concentrations of cadmium from industrial wastewater. However, environmental protection laws and civil society concerns about the environment have prompted the development of innovative technologies for removing heavy metals from industrial wastewater. Several conventional techniques have been employed for the removal of heavy metals from contaminated water, including reverse osmosis, ultrafiltration, electrodialysis, chemical precipitation, ion exchange, and phytoremediation [6]. However, low efficiency, time required, treatment process, and high operating and removal costs often make these methods impractical and lead to further environmental damage. Therefore, there is a need to explore methods for treating industrial liquid wastewater using relatively inexpensive, environmentally friendly natural absorbent materials [7].

The most cost-effective and efficient technologies are based on the combination of metals with biomaterials suitable for the removal of heavy metals from wastewater [8]. Therefore, there is a high demand for suitable and relatively inexpensive natural absorbent materials that can remove large amounts of heavy metals from wastewater [9,8]. Adsorption is regarded as one of the most effective methods., economical and advantageous methods for the removal of toxic heavy metals from aqueous solutions by selecting different adsorbents under optimal operating conditions [10]. The removal of cadmium ions from aqueous systems is extremely important. Adsorption on clay metal oxide surfaces is often used as a very effective method for the removal of unwanted metals from aqueous phases [11,12].

Clay materials are generally characterized by a high specific surface area, a layered crystalline structure, permanent and pH-variable surface charges, and high cation exchange capacity, providing numerous active sites for the adsorption of metal ions [13, 14]. Furthermore, the abundance, low cost, natural availability, chemical stability, and environmental compatibility of clay materials make them potential alternatives to conventional commercial adsorbents such as activated carbon [14, 15]. Common clay compounds, such as kaolinite, Montmorillonite, Illite, and bentonite, have demonstrated excellent performance in the adsorption of various toxic metal ions through ion exchange, surface complex formation, and electrostatic interactions [15, 16]. These properties justify the selection of natural clay as a sustainable and economically viable adsorbent for the efficient removal of cadmium ions from wastewater [13-16].

Numerous studies have demonstrated the effectiveness of clay-based and other low-cost sorbents in removing cadmium Cd (II) from aqueous solutions. Gupta and Bhattacharya reported that kaolinite and montmorillonite exhibited high cadmium adsorption capacities through ion exchange mechanisms and the formation of surface complexes [15]. Murray observed increased cadmium (II) adsorption on activated palygorskite due to its increased surface area and the availability of active sites [16]. More recently, Odin concluded that natural clay materials remain among the most promising low-cost sorbents due to their high adsorption efficiency, environmental friendliness, and wide availability [14]. In addition to clay minerals, various agricultural residues, biochar, fly ash, and industrial byproducts have also been investigated as economical cadmium removal sorbents. However, adsorption performance varies considerably depending on the mineral composition, surface properties, and local availability of the sorbent. Therefore, evaluating the locally available natural clay in Al-Khums, Libya is crucial to determining its absorption properties and assessing its potential as an effective and sustainable absorbent for removing cadmium Cd (II) ions.

In this study, clay from the region was used as a natural adsorption material. Different shades contribute to the removal process. The most important of these are kaolinite, illite and bentonite, which are used to remove almost all types of pollutants from water. The clay was obtained from a quarry in the Souq Al-Khamis area of the city of Homs. It was investigated whether the clay could be used to adsorb cadmium (II) ions under different conditions. The adsorption performance was systematically investigated under different conditions, including the influence of key operating parameters, such as solution pH, adsorbent dosage, contact time, and initial Cd (II) concentration. The equilibrium adsorption data were analyzed using the Langmuir and Freundlich isotherm models to characterize the adsorption behavior and equilibrium relationship. Furthermore, the experimental kinetic data were analyzed using the pseudo-first-order kinetic model to evaluate the adsorption kinetics.

2. Materials and Methods

2.1. Materials

In this study, green clay was selected as the adsorbent and collected from a specific site in the Souq Al-Khamis area located within the city of Al-Khums. The clay was washed to remove dirt using distilled water, dried in an oven at a temperature of 120 degrees Celsius for 3 hours, ground to obtain fine particles using an electric grinder, and the powder was sieved using sieves of different sizes ranging from (0.075-0.15-0.3-0.4-0.5) mm. Finally, the sample powder was stored in the laboratory for use in batch experiments.



Fig 1: Natural Clay

2.2. Preparation of the solution

Cadmium solutions of different concentrations were prepared by diluting 100 mg L^{-1} of stock solution from Cadmium sulphate heptahydrate ($CdSO_4 \cdot 7H_2O$) analytical grade) with. The optimal wavelength was chosen to be 340 nanometres (nm). The adsorption experiments were performed in batch mode using 100 mL conical flasks in which a constant amount of adsorbent was mixed with a fixed volume of aqueous cadmium salt solution.

2.3. Experimental adsorption studies

Batch adsorption experiments were conducted to investigate the adsorption of cadmium from aqueous solutions. Different concentrations of cadmium (1000 to 5000 mg L^{-1}) and different amounts of clay (adsorbent material) were used at a temperature of 25 degrees Celsius. One gram of clay was added to 100 mL of solution containing cadmium concentrations (1000 to 5000 mg L^{-1}), the solution was separated via filtration using filter paper and then the cadmium (II) concentration was measured by atomic absorption spectrophotometry (AAS). For all batch experiments, the pH of the solution was adjusted to the required level using 1 M HCl or NaOH solutions, in order to study the effect of contact time on the absorption of cadmium (II) ions as well as the absorbance and clay.

After completion of the adsorption process, the adsorbent was

separated from the aqueous solution by filtration, and the residual Cd (II) concentration was determined by atomic absorption spectrophotometry [17,18]. The cadmium (II) content was determined by the following equation:

$$Q_e = \frac{(C_i - C_f)\,V}{m} \qquad (1)$$

Where: Q_e is the capacity of Cd (II) adsorbed at equilibrium (mg/g), C_i is the initial concentration of cadmium (mg L^{-1}), C_f is the concentration of cadmium in equilibrium (mg L^{-1}), m is adsorbents dose (g), and V is volume of solution (L).

The percentage removal of Cd (II) from the aqueous solution was calculated using the following equation (2) [18.19].

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

where: Ci (mg L^{-1}) represents the initial ion concentration, Cf (mg L^{-1}) denotes the final ion concentration, and R signifies the removal efficiency (%).

3. Results and discussions

3.1 Properties of Clay

The chemical composition of the natural clay used in this work was analysed using X-ray fluorescence spectroscopy (XRF), and the results showed that the clay primarily consists of Al_2O_3 and SiO_2 in varying proportions, as well as an amount of CaO and an amount of Fe_2O_3, as shown in Table 1.

Table 1: Chemical analysis of Clay

Constituent	*AL_2O_3*	*CaO*	*MgO*	*Fe_2O_3*
wt. (%)	*19.12*	*3.48*	*0.26*	*6.89*
Constituent	*SiO_2*	*CO_2*	*H_2O*	*others*
wt. (%)	*53.01*	*13.3*	*2.42*	*1.52*

3.2 Calibration curve

Calibration curves are performed before operating the Atomic Absorption Spectrometer to measure Cadmium ions in solution to obtain accurate results. The following figure shows an example:

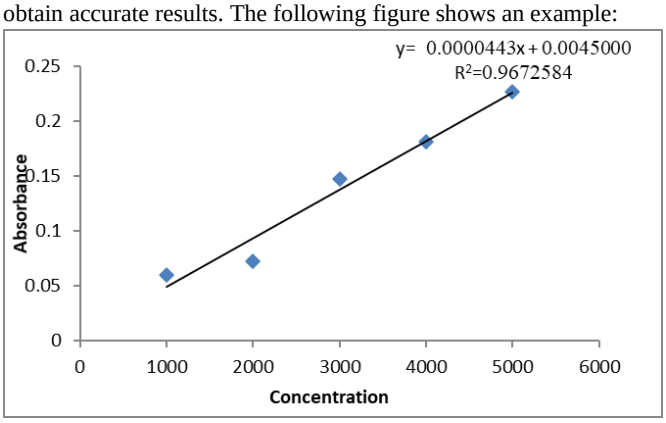


Fig 2: Calibration curve

3.2. Effect of pH

The pH of the aqueous solution is one of the most important variables controlling mineral adsorption at soil-water contact surfaces. The effect of pH on cadmium (II) adsorption on clay was studied in a range of 2.0 to 9.0, as shown in Figure 3. It was observed that at lower pH levels, cadmium (II) ions predominate in the solution, resulting in relatively low adsorption. However, as pH increases, cadmium (II) ions acquire free binding sites on the clay, thus increasing their adsorption. [21, 20, 19] The results indicate that the optimal adsorption of cadmium (II) occurs at pH 6.0.

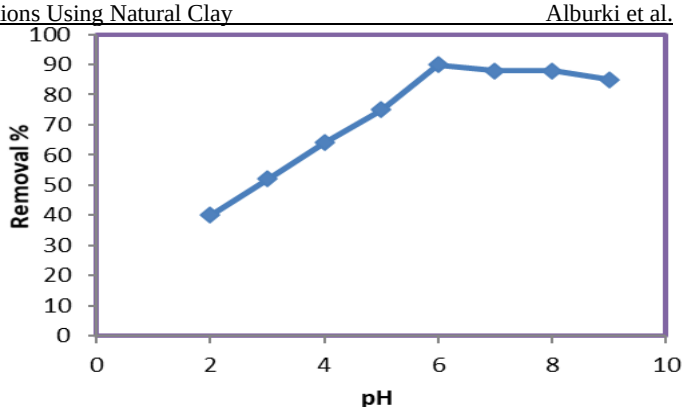


Fig. 3: Effect of pH on the adsorption of Cd(II) onto Natural clay

3.3. Effect of adsorbent dose

The quantity of adsorbent significantly influences the removal efficiency of cadmium (II), as it determines the adsorbent's capacity and reflects the initial concentration of adsorbent materials. Findings indicate that the adsorption efficiency improves with an increased amount and dosage of adsorbent, primarily due to the expanded surface area and the availability of more adsorption sites within the clay. This expansion facilitates easier penetration of metal ions into these sites. Additionally, the particle size in higher doses contributes to enhancing the binding sites for cadmium (II) ions, further improving adsorption.

The removal rate of cadmium (II) increases proportionally with the adsorbent dose, ranging from 1 to 5 grams. At a concentration of 1000 parts per million, the peak removal rate is achieved using an adsorbent dose of 3 grams with a contact time of 60 minutes. However, when the dose is increased to 4 grams, the removal efficiency remains nearly unchanged, as 3 grams is sufficient to maintain equilibrium conditions for effective cadmium ion removal. Similar findings have been reported by Wang et al. (2007) and Gupta & Bhattacharya (2006) in studies examining cadmium (II) adsorption on various clay materials [24.26].

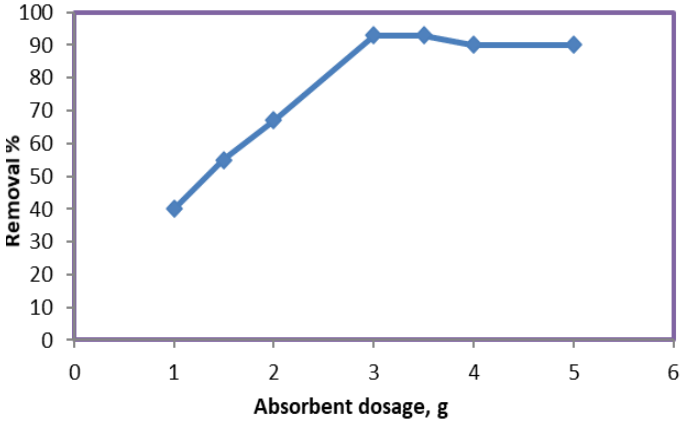


Fig. 4: Effect of adsorbent dosage on Cd (II) adsorption

3.4. Effect of Ion concentration

The effect of Cd (II) concentration on the adsorption process was studied under optimal conditions. According to the results, the adsorption of Cd2+ ions was almost constant up to 3000 parts per million of Cd (II), and then it decreased. This can be explained by the relatively smaller number of active sites available for higher concentrations of Cd (II) ions.

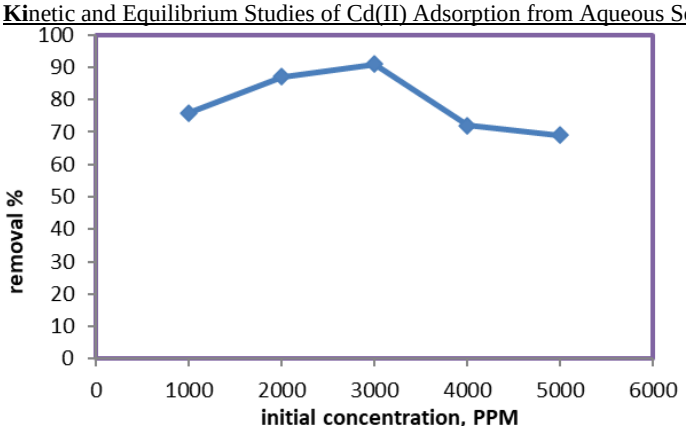


Fig. 5: Effect of Initial Cd (II) Concentration on Removal Efficiency

3.5. Effect of Contact Time on Cd (II) Adsorption

The effect of contact time on cadmium adsorption was evaluated by varying the equilibrium time between the adsorbent and adsorbate from 30 to 120 minutes, while keeping the cadmium ion (II) concentration constant at 3000 mg L^{-1} relative to the clay dose [25].. The findings are illustrated in Figure 6 and demonstrate that cadmium ion (II) adsorption by clay increased progressively with longer contact times, reaching its peak value at 60 minutes

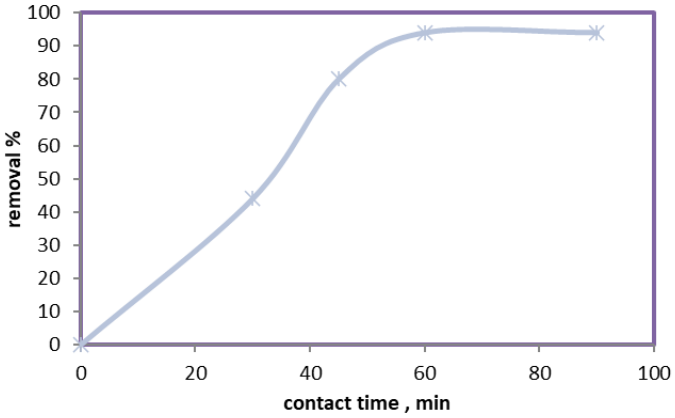


Fig. 6: Effect of contact time on Cadmium adsorption

3.6. Adsorption kinetics

The adsorption kinetics of 100 mL containing a known concentration of Cd (II) were studied. Concentration of 3000 mg L^{-1} at a contact time of 20-100 minutes. A pseudo-first-order kinetic model, expressed in Equation 3, was applied to fit the experimental data. [22,23.27].

$$\ln(qe - qt) = -K_1 t + \ln(qe) \qquad (3a)$$

Where: qe represents the capacity of equilibrium adsorption (mg/g), qt denotes the adsorption capacity (mg/g) at time t, K_1 is the pseudo-first-order rate constant (min^{-1}), and t is the contact time (min).

$$\frac{t}{qt} = \frac{1}{k_2\, qe^2} + \frac{t}{qe} \qquad (3b)$$

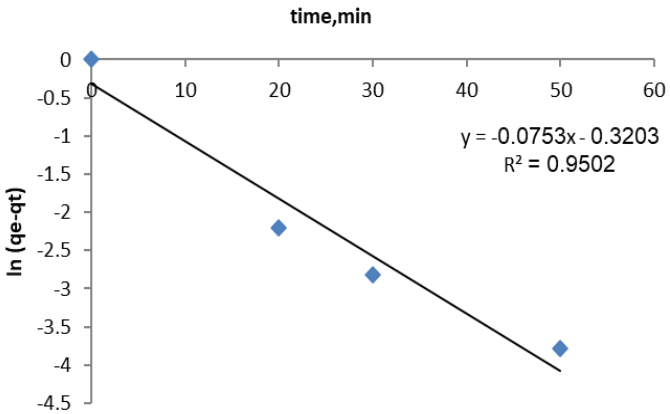
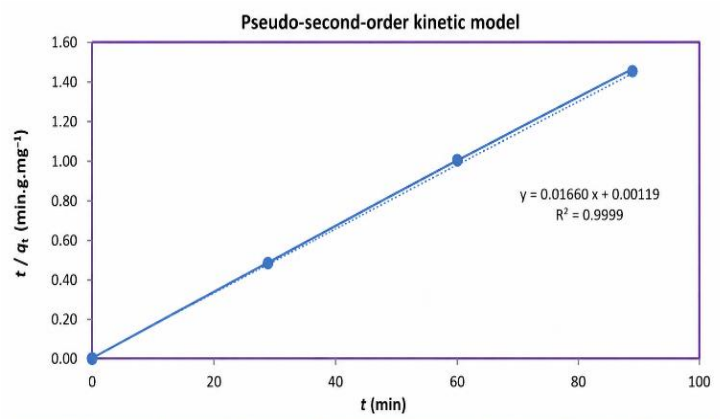


Fig. 7a: Pseudo-first-order kinetic model for Cadmium



Kinetic model	$q_{e,exp}$ (mg/g)	$q_{e,cal}$ (mg/g)	k (min^{-1} or $g \cdot mg^{-1} \cdot min^{-1}$)	R^2	RMSE (mg/g)	χ^2 (mg^2/g^2)
Pseudo-second-order	94.30	60.24	0.00455 ($g \cdot mg^{-1} \cdot min^{-1}$)	0.9999	1.12	1.38

Fig. 7b: Pseudo second order kinetic model for Cadmium

3. 7 .Adsorption Isotherm

The adsorption isotherm study was carried out to evaluate the equilibrium behaviour of Cd (II) adsorption onto the studied adsorbent and to assess the feasibility of the adsorption process for Cd (II) removal from aqueous solutions. Adsorption isotherms describe the relationship between the equilibrium concentration of the adsorbate remaining in the liquid phase and the corresponding amount adsorbed onto the adsorbent surface at equilibrium. Understanding this relationship provides valuable information regarding the adsorption capacity and surface characteristics of the adsorbent.

The experimental equilibrium data obtained at different initial Cd (II) concentrations under identical operating conditions were analysed using two widely applied adsorption isotherm models, namely the Langmuir and Freundlich models. These models were selected to evaluate the nature of the adsorption process and to determine the corresponding equilibrium parameters. The experimental data were fitted to the respective mathematical equations given by Eqs. (4) and (5). The resulting Langmuir and Freundlich plots are presented in Figures 8 and 9, respectively. [28.29].

$$\frac{Ce}{qe} = \frac{1}{qm * k_l} + \frac{C_e}{qm} \qquad (4)$$

Where: Ce represents the equilibrium Cd (II) concentration in solution (mg/L), qe denotes the equilibrium adsorption capacity (mg/g), KL is the Langmuir equilibrium constant, and qm signifies the maximum sorption capacity (mg/g). The value of qe was determined using Eq. (1).

$$\ln(qe) = \ln kF + \frac{1}{n \ln ce} \qquad (5)$$

Where: qe represents the equilibrium adsorption capacity (mg/g), Ce denotes the equilibrium concentration of Cd (II) ions (mg L^{-1}), and KF and n are Freundlich constants indicative of adsorption capacity and intensity, respectively. A linear relationship is established by plotting ln qe against ln Ce, enabling the determination of KF and n from the intercept and slope, respectively.

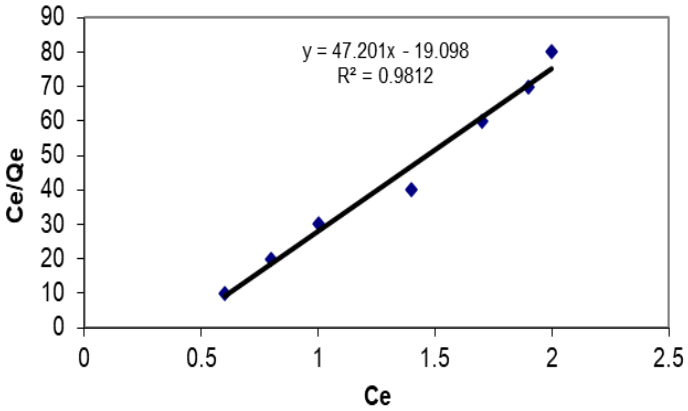


Fig. 8: Langmuir adsorption isotherm for Cd (II) adsorption

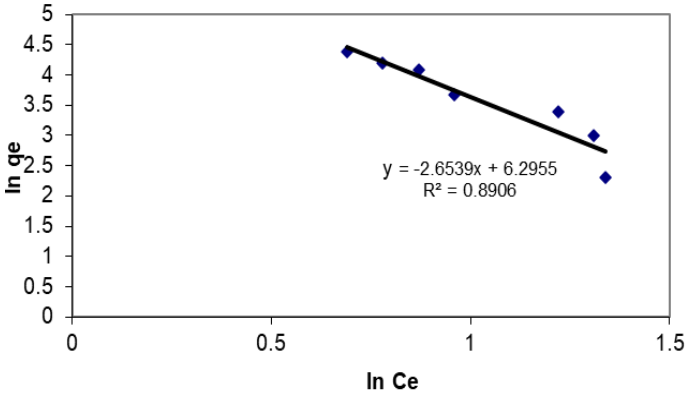


Fig. 9: Freundlich adsorption isotherm for Cd (II) adsorption

The results obtained were compared with previous studies on clay-based adsorbents. Gupta and Bhattacharya (2006) and Wang et al. (2007) reported that clay minerals exhibit a high capacity for adsorbing cadmium Cd (II) ions through ion exchange mechanisms and the formation of surface complexes [24, 26]. Odin (2017) highlighted natural clay as a promising and low-cost adsorbent due to its high cation exchange capacity and abundance of active sites [14]. In this study, the natural clay from the Al-Khums area achieved a removal efficiency of 94.3% at pH 6.0, using an adsorbent dose of 3 grams and a contact time of 60 minutes. These results are consistent with those reported in the scientific literature, confirming the effectiveness of the studied clay as an efficient, low-cost, and locally available adsorbent. Furthermore, the Langmuir model provided the best match with the equilibrium data, indicating monolayer adsorption, consistent with previous studies

4. Conclusion

This study investigated natural clay collected from the Al-Khums region, Libya, as a low-cost adsorbent for the removal of Cd (II) ions from aqueous solutions. The adsorption performance was evaluated under different operating conditions, including pH, initial Cd (II) concentration, adsorbent dosage, temperature, and contact time. The results demonstrated that these parameters significantly affected the adsorption efficiency.

The optimum adsorption conditions were achieved at pH 6.0, using an adsorbent dosage of 3 g and an equilibrium contact time of 60 min, resulting in a maximum Cd (II) removal efficiency of 94.3%. The adsorption efficiency increased with increasing temperature, indicating the favorable effect of temperature on the adsorption process. Equilibrium data were analyzed using Freundlich and Langmuir isotherm models. The Langmuir model provided the best fit, suggesting monolayer adsorption of Cd (II) ions onto the available active sites of the natural clay surface. The results of the kinetic study showed that adsorption process followed the pseudo-first-order kinetic model describing the interaction between Cd (II) ions and the clay surface.

The findings confirm that the locally available natural clay from Al-Khums exhibits effective adsorption performance for Cadmium removal, with a high removal efficiency of 94.3% under optimized conditions. Therefore, this material represents a promising, inexpensive, and environmentally friendly alternative adsorbent for treating cadmium-contaminated aqueous solutions.

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