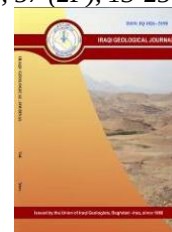




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Assessing the Efficiency of Injana Clay for Cadmium Sorption: Implications for Environmental Sustainability

Khalid S. Mohammed^{1,*}, Saadi A. Ouda², Ahmed F. Al-lahaibi¹, Mohammed I. Mohammed³, and Omar H. Jasim⁴

¹Department of Geography, College of Education for Humanities, University of Anbar, Iraq

²Department of Geography, College of Education for Girls, University of Anbar, Iraq

³Department of Geology, College of Science, University of Baghdad, Baghdad, Iraq

⁴Department of Civil Engineering, College of Engineering University of Al-Maarif, Anbar, Iraq

Correspondence: ed.khalid.sabar@uoanbar.edu.iq

Abstract

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The study aims to assess the effectiveness of Injana Formation clay in removing cadmium from solutions. The clay samples were collected from the Shekhasuod area east of Ramadi City in the Governorate of Al-Anbar, west of Iraq, from the Injana Formation (Late Miocene). The effect of temperature and time on adsorption was identified through several experiments conducted at temperatures of 15, 25, 35, and 45 °C for solutions at eight durations 1, 10, 20, 30, 40, 50, 60, and 70 minutes. The procedure included determining the adsorption efficiency by adding three grams of clay (solid) to 40 ml of cadmium solution (liquid) under the effect of changing, the pH and TDS. The equilibrium state (equilibrium time) at the different temperature degrees is determined as 20 min in most experiments. The equations of Langmuir and Freundlich showed that the capacity of adsorption (Q_0) is equal to 0.6224 mg/g, and the constants of Langmuir empirical (KL) is 22.6. The results show that the intensity (n) is equal to 30.03, and the Freundlich isotherm constant (Kf) is equal to 1.5282. The equations of cadmium adsorption are identical with the Langmuir isotherm and Freundlich isotherm models. The efficiency of utilizing clay for removing cadmium from solutions was discussed practical solution to enhance the aquatic environment.

Keywords: Injana; Clay; Cadmium; Pollution; Sorption; Solutions

1. Introduction

Heavy metals are among the most complex and persistent pollutants in nature (Ozyigit and Genc, 2020). These metals increased in the surrounding environment as a result of human activities such as mine influences, metallurgical industries, metal smelting, fossil fuel combustion, and waste disposal (Kitula, 2006; Awadh, 2018). Water is greatly affected by increasing environmental pollution. It is very necessary to enhance treatment processes for the aquatic environment, whether from chemical or biological pollutants (Ghayeb and Mohammed, 2024). Therefore, it was necessary to experiment with new adsorbents that reduce the spread of heavy metals. The adsorbent material used in this research is clay, which is found in huge reserves in the Injana Formation, and appears in several areas in Al-Anbar Governorate. Cadmium in the environment is one of the most dangerous contaminant metals that is easily transmitted to plants and other living organisms. Cadmium affects human health even in low concentrations and could cause serious diseases (Ozyigit and Genc, 2020). Because of the environmental and health risks associated with cadmium, researchers are actively seeking to eliminate or minimize its

presence in various environments through adsorption techniques employing diverse adsorbent materials (Filho and Carmo, 2006; Al-Hamdani, 2016).

The process of adsorption of heavy metals from solutions depends on the adsorbent materials, whether natural or synthetic (Awadh, 2018). The adsorbent material used in this research is Injana Formation clay to adsorb cadmium ions from solutions. There are many locations of outcrops of the Injana Formation clays inside and outside of Al-Anbar Governorate such as Hasaiba Alsharqiea area, Al-Hujaira area, at the east of Habbaniyah Lake, and Al-Chiffa area (Mansor and Al-Nuaimy, 2023). The selected exposed clay bed is located in the Sheikhamasud area east of Ramadi. The clay fraction having particle size in the range of 2-4 μm often contains non-phyllsilicate minerals, such as feldspar, carbonates, quartz and the hydroxides of aluminum and iron. These minerals don't impact plasticity of clay; therefore, they have been referred to as accessory minerals or non-clay constituents (Hall, 1987). The clay is used in several industries, including the ceramic industry (Farhan et al., 2022). Also, there are wide uses of clay in several applications, including environmental applications. It is used in backfilling, building dams and containing waste, sewer pipes, and stoneware (Grim, 1962).

The use of clay to improve the environment and combat pollution represents an effective and powerful tool because of its wide availability in the local environment in addition to its cheap price and high effectiveness. Clay was used in lead adsorption experiments from solutions. Previous studies have provided several evidence of the possibility of inexpensive adsorbents to remove heavy metals from wastewater and solutions. Mohammed and Awadh (2023) used ceramic waste, to determine the efficiency in adsorbing nickel from solutions and protecting the environment, with the ultimate goal of reducing the concentration of nickel in aqueous solutions. They used the glass waste to remove cadmium from solutions. Also, they used the glass sand to remove chromium from aqueous solutions. Awadh and Tawfik (2013) used clay minerals such as palygorskite and montmorillonite in adsorption processes. Awadh and Abdulla (2017) used natural bentonite to purification of aqueous solutions from lead ions (Pb^{2+}). Hussain et al. (2008) used the clay to adsorption of cobalt ions (Co^{2+}) from aqueous solution. This research presents a widely available material (clay) to be used as an adsorbent to reduce or remove the cadmium ion from solutions, thus protecting the environment.

This research aims to determine the absorption efficiency of Injana Formation clay for removing cadmium ions from solutions.

1.1. Geological Setting

The type locality of the Injana Formation (Late Miocene) is located at the extent of the northeastern limb of Himreen anticline in the Injana area (Sissakian and Mohammed, 2007). In the typical section, the thickness of the formation is about 620 m. The thickness of the formation in the study area at the Shekhamasud area in eastern Ramadi City is between 12 and 20 m. The Longitudes 43°47'30" E Latitudes 33° 4'44" N, which consist of red clay beds interblended with silt. The lower contact of this formation ends with the Fatha formation, and the upper contact ends with the Muqdadiya Formation (Jassim and Goff, 2006). Injana Formation consists of claystone, sand, and siltstone. Sedimentary sources are considered the most dominant as the proposed source for the Injana Formation, then igneous sources, with the least dominant being the metamorphic source (Al-Salmani et al., 2024). The environment of the Injana Formation is lagoon and transformed into a marine environment, continental environment in many cases be mountainous environment (Al-Sayab et al., 1982).

2. Materials and Methods

The adsorbent material (clay) was collected from the Shekhamasud area near Ramadi City (Fig. 1). Clay reserves are present within the Injana Formation (Late Miocene) (Fig. 2).

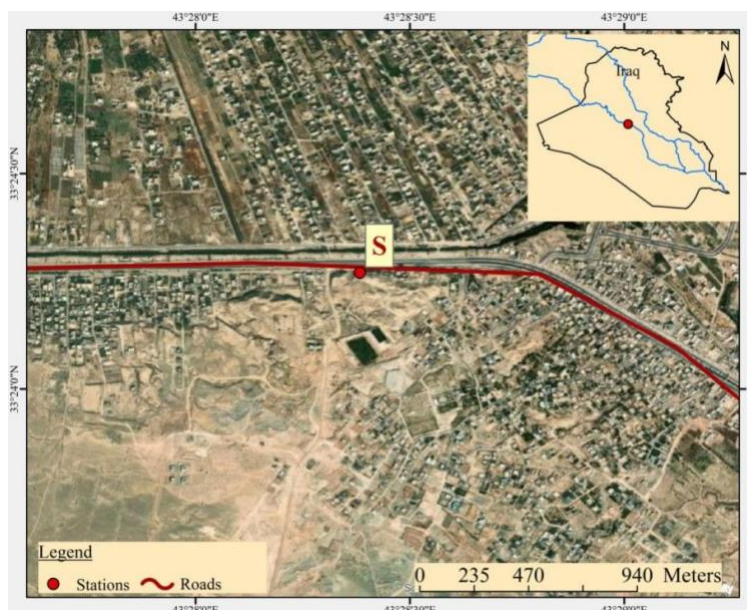


Fig.1. The Location map for the sample in Shekhamasoud area at the east of Ramadi City



Fig.2. The clay layers interbedded with silt at the Shekhamasoud area

Samples were prepared, and laboratory tests were conducted in the Soil and Water Lab and Chemistry Department in Al-Anbar University. The grain size of the clay sample is $<2\ \mu\text{m}$ in diameter, for experiment the particle size of clay for the cadmium adsorption tests, clay as a powder is shown in Fig. 3. The clay samples were tested for chemical analysis (XRF) in the University of Baghdad at the Department of Geology in the Iraqi Germany Laboratory, the device of (XRF) receive the sample in the form of powder, and to obtain a high accuracy for analysis, the powder is compressed and converted into pellet using a pressure of five tons (Fig. 4). The mineralogical analysis (XRD) for the clay sample tested in XRD Laboratory at the Department of Material Sciences in Authority of Science and Technology for Regions and Governorates at Ministry of higher education in a method of the bulk sample. The weight of cadmium nitrate diluted in distilled water is 2.7443 g at a concentration of 1000 ppm, this was diluted to 50 ppm. 3g of adsorbent (clay) and 40 ml of diluted cadmium solution were used to conduct the experiments. The pH, TDS, and EC of the diluted solution (50 ppm) were tested before sorption experiments using different measuring devices (Fig. 6). 32 laboratory experiments were tested at four different temperatures (15, 25, 35, and 45 °C), using a vibrating (shaker) (Fig. 5). At each

temperature degree, eight experiments were conducted at 8 different times 1, 10, 20, 30, 40, 50, 60, and 70 min. That is, every 10 minutes, we take a sample from the 8 samples in the shaker, filter it with filter paper, and examine the remaining concentration using an atomic absorption device (Fig. 6). They are measured pH, TDS, and EC again after the experiments.



Fig.3. Sample of clay as a powder

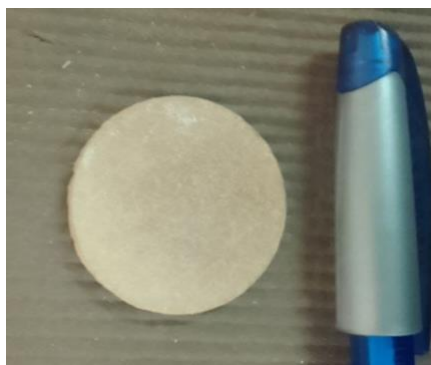


Fig.4. Clay sample compressed into pellets for XRF analysis

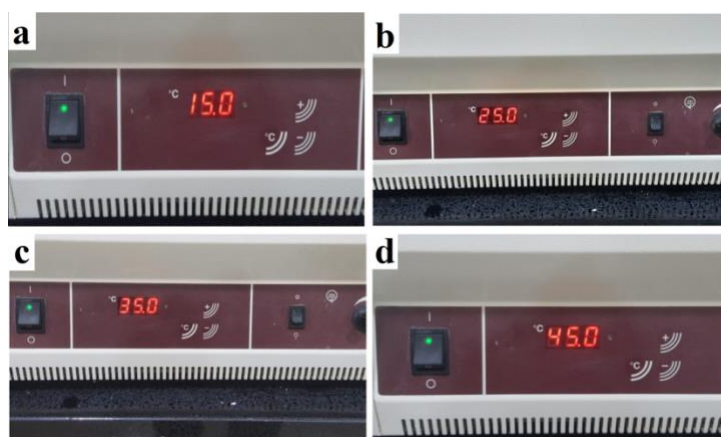


Fig.5. The device used for experiments (vibrator), at four temperature degrees, 15 °C (a); 25 °C (b); 35 °C (c); and 45 °C (d)



Fig.6. Atomic absorption spectrophotometer (a); pH meter (b); EC, TDS meter (c)

3. Results and Discussion

3.1 Mineralogy and Geochemistry for the Clay Sample

Mineralogically, the clay sample is consisted of quartz, calcite, albite, dolomite, illite, chlorite, gypsum, palygorskite, and halite. The mineralogy of the clay is shown by the diffractogram of XRD (Fig. 7).

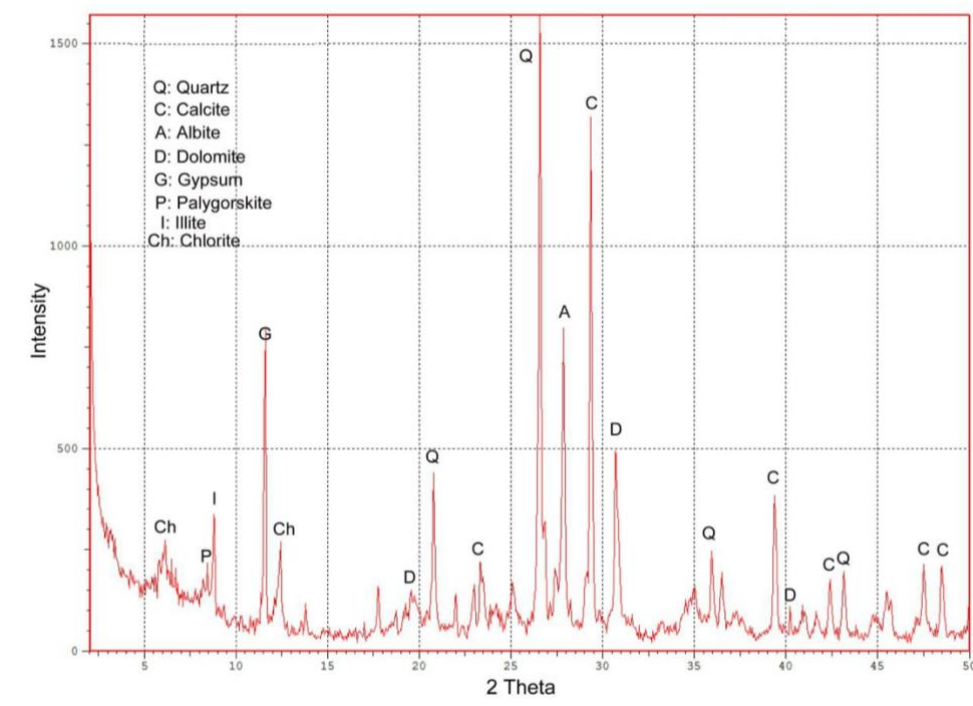


Fig.7. The diffractogram of clay sample

The geochemistry of the clay sample is summarized as the composed of clay includes several components, SiO₂ (38.33), CaO (17.96), Al₂O₃ (8.66), MgO (6.57), Fe₂O₃ (6.15), Na₂O (1.51), K₂O (1.49), SO₃ (1.31), TiO₂ (0.91), P₂O₅ (0.12) and MnO (0.1). The chemical composition of the clay sample and its percentage are given in Table 1.

Table 1. The composition of clay sample using XRF.

Oxides	SiO ₂	CaO	Al ₂ O ₃	MgO	Fe ₂ O ₃	Na ₂ O	K ₂ O	SO ₃	TiO ₂	P ₂ O ₅	MnO	LOI	Total
%	38.33	17.96	8.66	6.57	6.15	1.51	1.49	1.31	0.91	0.12	0.10	15.45	98.56

3.2. Sorption Efficiency

3.2.1 Effects of temperature, time, and concentration

To calculating the efficiency of clay absorption requires several factors are manipulated including temperatures, times, cadmium concentration, volume of solution, and weight of adsorbent (clay). The ends time of the reactions was determined by adsorption efficiency when the equilibrium time was known from the absorption results, 32 laboratory experiments on cadmium adsorption with clay were conducted by specifying 8 time periods (1, 10, 20, 30, 40, 50, 60, and 70 min) for four temperatures 15, 25, 35, and 45 °C, the volume of the solution to the weight of adsorbent material (clay), solid: liquid (3g: 40ml). At the temperature of 15 °C, the concentration of cadmium changed (decreased) from 50 to 2.23 ppm (95.54%) as shown in Table 2 and Fig. 8. At the temperature of 25 °C, the concentration of cadmium changed from 50 to 2.28 ppm (95.44%) (Table 3; Fig. 9). At the temperature of 35 °C, the concentration of cadmium changed from 50 to 1.97 ppm (96.06%) (Table 4; Fig. 10). While at the temperature of 45 °C, the concentration of cadmium changed from 50 to 1.09 (97.82%) ppm (Table 5; Fig. 11).

Table 2. Results of the laboratory tests for the cadmium adsorption using clay for 8 times experiments with temperature of 15 °C and cadmium concentration of 50 ppm.

Sample	Before the experiments				After the experiments					
	Time (min)	pH	EC (μs/cm)	(mg/l) TDS	pH	EC (μs/cm)	TDS (mg/l)	Final conc. (ppm)	Sorption (ppm)	Sorption (%)
1 Cd	1	5.1	129	91	7.78	1377	869	2.93	47.07	94.14
2 Cd	10	5.1	129	91	8.00	1325	823	2.83	47.17	94.34
3 Cd	20	5.1	129	91	7.75	1304	802	2.45	47.55	95.1
4 Cd	30	5.1	129	91	7.76	1289	795	2.30	47.70	95.40
5 Cd	40	5.1	129	91	7.82	1264	780	2.31	47.69	95.38
6 Cd	50	5.1	129	91	7.90	1247	773	2.25	47.75	95.5
7 Cd	60	5.1	129	91	8.00	1254	778	2.24	47.76	95.52
8 Cd	70	5.1	129	91	8.00	1250	776	2.24	47.76	95.52

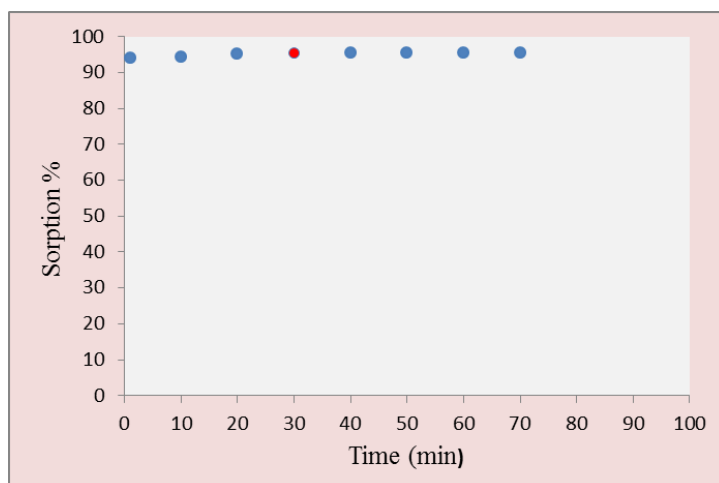


Fig. 8. The efficiency of adsorption and equilibrium state of the clay for the cadmium sorption from solutions (10 min) at 15 °C

Table 3. Results for the laboratory tests to sorption of cadmium using clay for 8 times experiments at the temperature of 25 °C and 50 ppm.

Samples	Before experiment				After experiment					
	Time (min)	pH	EC (µs/cm)	TDS (mg/l)	pH	EC (µs/cm)	TDS (mg/l)	Final conc. (ppm)	Sorption (ppm)	Sorption (%)
1 Cd	1	5.1	129	91	7.59	1413	887	4.18	45.82	91.64
2 Cd	10	5.1	129	91	7.48	1357	855	3.31	46.69	93.38
3 Cd	20	5.1	129	91	7.78	1319	820	2.70	47.3	94.6
4 Cd	30	5.1	129	91	7.91	1302	796	2.28	47.72	95.44
5 Cd	40	5.1	129	91	7.65	1280	784	2.19	47.81	95.62
6 Cd	50	5.1	129	91	7.82	1271	779	2.16	47.84	95.68
7 Cd	60	5.1	129	91	8.05	1263	775	2.20	47.8	95.6
8 Cd	70	5.1	129	91	8.17	1263	775	2.21	47.79	95.58

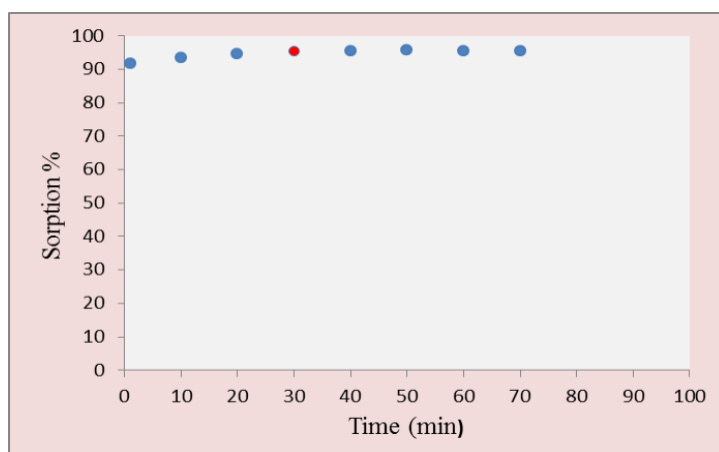
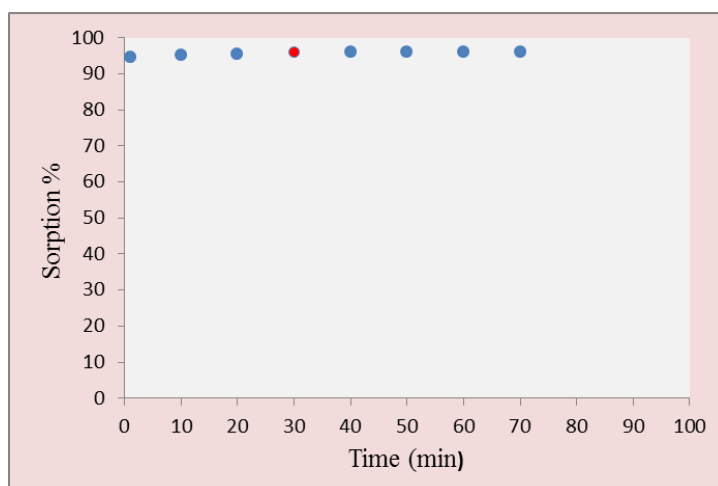


Fig. 9. The efficiency of adsorption and equilibrium state of the clay for the cadmium sorption from solutions (10 min) at 25 °C.

Table 4. Results for the laboratory tests to sorption of cadmium using clay for 8 times experiments at the temperature of 35 °C and 50 ppm.

Samples	Before experiment				After experiment					
	Time (min)	pH	EC ($\mu\text{s}/\text{cm}$)	TDS (mg/l)	pH	EC ($\mu\text{s}/\text{cm}$)	TDS (mg/l)	Final conc. (ppm)	Sorption (ppm)	Sorption (%)
1 Cd	1	5.1	129	91	7.61	1450	893	2.74	47.26	94.52
2 Cd	10	5.1	129	91	7.76	1393	867	2.44	47.56	95.12
3 Cd	20	5.1	129	91	7.68	1345	837	2.23	47.77	95.54
4 Cd	30	5.1	129	91	7.75	1329	801	1.97	48.03	96.06
5 Cd	40	5.1	129	91	7.76	1307	793	1.93	48.07	96.14
6 Cd	50	5.1	129	91	7.81	1287	788	2.02	47.98	95.96
7 Cd	60	5.1	129	91	7.78	1281	791	1.99	48.01	96.02
8 Cd	70	5.1	129	91	7.82	1285	789	1.95	48.05	96.1

**Fig. 10.** The efficiency of adsorption and equilibrium state of the clay for the cadmium sorption from solutions (10 min) at 35 °C.**Table 5.** Results for the laboratory tests to sorption of cadmium using clay for 8 times experiments at the temperature of 45 °C and 50 ppm.

Samples	Before experiment				After experiment					
	Time (min)	pH	EC ($\mu\text{s}/\text{cm}$)	TDS (mg/l)	pH	EC ($\mu\text{s}/\text{cm}$)	TDS (mg/l)	Final conc. (ppm)	Sorption (ppm)	Sorption (%)
1Cd	1	5.1	129	91	8.1	1478	902	3.13	46.87	93.74
2 Cd	10	5.1	129	91	8.2	1431	883	1.73	48.27	96.54
3 Cd	20	5.1	129	91	8.13	1366	842	1.42	48.58	97.16
4 Cd	30	5.1	129	91	8.15	1337	807	1.09	48.91	97.82
5 Cd	40	5.1	129	91	8.12	1315	798	1.13	48.87	97.74
6 Cd	50	5.1	129	91	8.29	1307	805	1.07	48.93	97.86
7 Cd	60	5.1	129	91	8.35	1299	793	1.02	48.98	97.96
8 Cd	70	5.1	129	91	8.22	1294	796	1.006	48.994	97.98

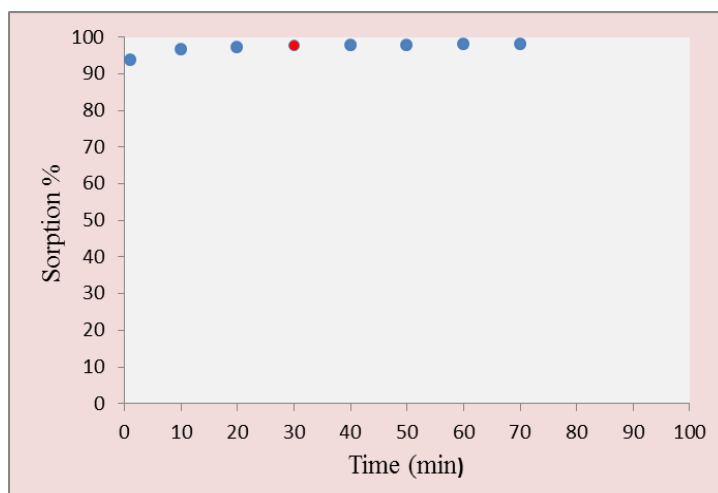


Fig. 11. The efficiency of adsorption and equilibrium state of the clay for the cadmium sorption from solutions (10 min) at 45 °C

There is a direct relationship between high temperature and sorption efficiency (sorption %). The clay absorption of cadmium (%) was studied at four temperatures (15, 25, 35, and 45 °C), when the temperature increases, the absorption efficiency increases slightly (Fig. 12). As a result, the best absorption (97.82%) occurred when the cadmium solution had a 45 °C at the initial concentration of 50 ppm.

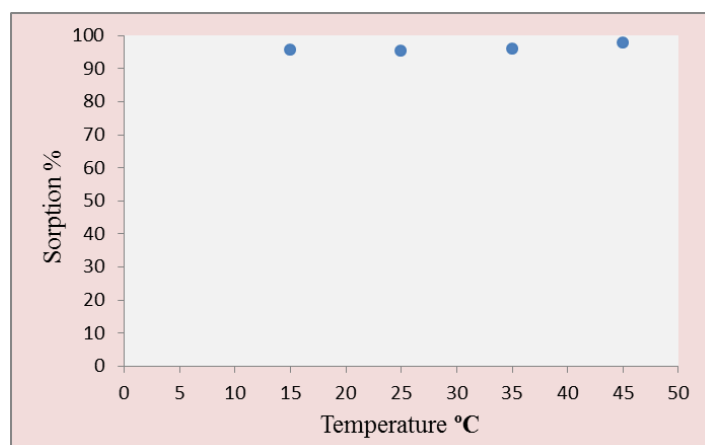


Fig. 12. The difference in the ability of adsorption for cadmium at four temperature degrees at the equilibrium time

4. Adsorption Isotherms

In general, the adsorption isotherms are used to measure the absorption equilibrium in any process between an adsorbent and an adsorbate, the ionic distribution between the adsorbed and liquid state is also elucidated by the same process (equilibrium adsorption isotherms) (Piccin et al., 2017). To know the adsorption systems and the ability of adsorbent materials to remove pollutants, it is necessary to measure the pH of the solution, and know the initial concentration, and time, from here the importance of using the Langmuir and Freundlich equations emerges (Gunasundari, 2017).

4.1. Langmuir Isothermal Model

This model is very effective and common in most adsorption processes, as well as the capture of solute from the solution, as it is considered that adsorption occurs in a single layer and the reaction does not continue after that layer. The reactions also include the maximum adsorption capacity (Q_0) corresponding to the monolayer, where K_L values vary as shown by the initial slope of the adsorption isotherm. The mathematical equations of the Langmuir model are as follows:

$$\frac{C_e}{Q_e} = \frac{1}{K_L Q_0} + \frac{C_e}{Q_0} \quad (1)$$

Where: C_e = equilibrium concentration for adsorbate (mg/L), Q_0 = the maximum monolayer coverage capacity (mg/g), Q_e = amount of adsorbed of material per gram for the adsorbent in equilibrium (mg/g). $K_L Q_0$: the constants of Langmuir empirical.

From the model of C_e/Q_e against C_e , it was showed a slope with a line for $1/Q_0$ and an intersection for the values of $1/K_L Q_0$, where K_L and Q_0 were identified from the slope and intercept for the model (Adamson and Gast., 2001). as shown in Fig. 13.

The Q_0 represents the maximum sorption ability, and K_L is an equilibrium constant representing the bond energy between the adsorbate and adsorbent (Adamson and Gast., 2001). The amounts of adsorbent materials were identified based on the equation:

$$Q_e = \frac{V_{sol} (C_0 - C_e)}{M} \quad (2)$$

V_{sol} = adsorbent solution volume (L). C_0 = initial concentration for the adsorbent solution (mg/l). M = adsorbent mass (grams).

The efficiency of removal was determined by computing the sorption of percentage by the following formula:

$$R\% = \frac{(C_0 - C_e)}{C_0} \times 100 \quad (3)$$

Where: $R\%$ = percentage of adsorption (sorption efficiency). C_0 and C_e are the initial and final concentrations for heavy metal Cd at the equilibrium concentration.

Table 6. Sorption (%) for cadmium by clay at the concentration of 50 ppm and their corresponding values of Q_e and C_e .

Sample	Temperature (°C)	R (%)	C_e (mg/L)	Q_e (mg/g)	C_e / Q_e (g/L)
1	15	95.40	2.30	0.6360	3.6163
2	25	95.44	2.28	0.6362	3.5837
3	35	96.06	1.97	0.6404	3.0762
4	45	97.82	1.09	0.6521	1.6715

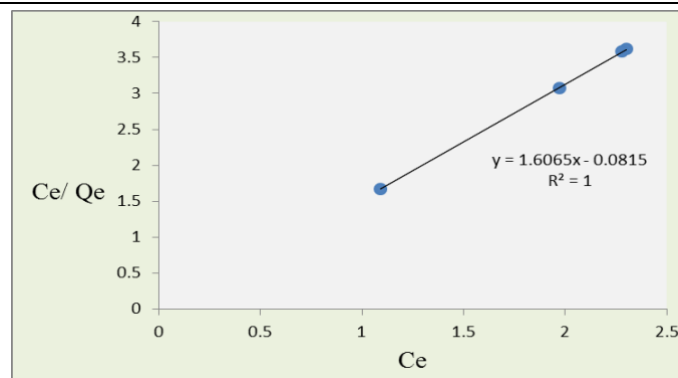


Fig. 13. Langmuir model for adsorption of cadmium with clay

The maximum capacity (Q_o) at the Langmuir isotherm was determined to be 0.6224 mg/g. Langmuir isotherm constant related to the energy of the adsorption (K_L) is 22.6. The adsorption is well correlated to the Langmuir isotherm model, where the correlation coefficient (R^2) is significantly strong and reach to 1 (Fig. 13) m which means that the adsorption processes are fitted with the Langmuir isotherm.

4.2. Freundlich Isothermal Model

The basis of this equation refers to the variation and difference in the adsorption energies of the adsorbent materials (heterogeneity), this model shows the ease or difficulty of adsorption (Lau et al., 2016). The mathematical equations are as follows:

$$Q_e = Kf C_e^{1/n} \quad (4)$$

Where: Q_e = amount of metal adsorbed per grams for the adsorbent at equilibrium time (mg/g). C_e = equilibrium concentration for adsorbate material (mg/L). n = intensity of adsorption. Kf = Freundlich isotherm constant (mg/g). The equation 5 is derived from the previous equation:

$$\text{Log } Q_e = \text{Log } Kf + \frac{1}{n} \text{Log } C_e \quad (5)$$

The Kf constant is an approximate indicator of sorption capacity, the value of n constant represents adsorption intensity, and $1/n$ is the strength of adsorption at the adsorption processes (Voudrias et al., 2002). The value of $1/n$ and constant of $\text{Log } Kf$ were computed from the intercept, slope for the Freundlich isotherm plot (Fig. 14).

Table 7. Sorption percentage (%) for cadmium by clay at the concentration of 50 ppm and their corresponding values of $\text{Log } Q_e$ and $\text{Log } C_e$

Sample	Temperature (°C)	R (%)	C_e (mg/L)	Q_e (mg/g)	$\text{Log } C_e$	$\text{Log } Q_e$
1	15	95.40	2.30	0.6360	0.3617	-0.1965
2	25	95.44	2.28	0.6362	0.3579	-0.1964
3	35	96.06	1.97	0.6404	0.2944	-0.1935
4	45	97.82	1.09	0.6521	0.0374	-0.1856

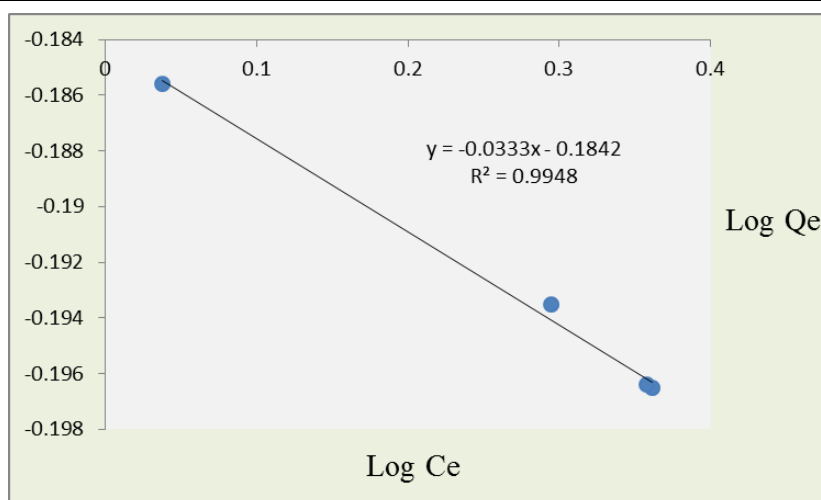


Fig. 14. Freundlich diagram for adsorption of cadmium with clay

From the values in Table 7, and Fig. 14, the intensity of adsorption (n) = 30.03, Kf = 1.5282. The (R^2) value is 0.994, This shows the adsorption of cadmium is consistent with Freundlich isotherm.

5. Conclusions

Most cadmium concentrations can be removed within the first 1-10 minutes of experiments.

The adsorption efficiency increased slightly with increasing temperature from 15 to 45 °C, where the sorption percentage for these two temperatures was 95.54% - 97.82%, respectively, at the equilibrium time.

The equilibrium time for the absorption processes did not change with changing temperatures, as it was recorded as 20 minutes for all temperature experiments (15, 25, 35, and 45 °C).

The increase of pH (towards the alkalinity of the solution) with the adsorption processes is due to the presence of alkaline metals in the clay composition (Na, K, Ca, and Mg).

The slight increase in TDS with increasing temperature is evidence of increased dissolution of salinity from adsorbent materials in the solution. At the same time, the decrease in TDS after the experiment of adsorption is evidence of the adsorption process.

In general, the adsorption efficiency of Injana clay for cadmium is excellent in terms of efficiency and time.

Acknowledgements

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