



REMOVAL OF LEAD FROM POLLUTED WATER BY ADSORPTION ON BENTONITE CLAY PARTICLES

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Abstract: This study investigates the capability of a certain type of natural Bentonite clay to remove the poisonous element; lead, from polluted water by adsorption phenomena on the clay particles at different temperature levels. Many high lead concentration synthetic solutions had been prepared in laboratory (24.3, 56.85, 146.55, 269.99, 284.72, 418.86, 465.61, 658.80 mg/L), and a volume of 250 ml from each solution being mixed with 2.5 g of Bentonite clay for a period of one hour at a certain temperature level (three levels were used, 20, 40, and 60 °C) and pH=7, until an equilibrium adsorption process being obtained, then the samples filtered through a porous paper, and the filtrate tested by atomic absorption technique to determine the remaining lead concentration. Very high removal efficiencies have been obtained (more than 98%), and the results were drawn on graphs to show the relations at test conditions. The above mentioned procedure was repeated using the same prepared lead solutions but mixed with 0.5 g of Bentonite clay and tested at temperature of 20 °C, and very good lead removals also were obtained. The adsorbed lead ions mass in mg, on Bentonite mass in gram (Me) were ranged from 2.387 to 65.848 at temperature levels of 20, 40, 60 °C and Bentonite clay dosage of 2.5 g per 250 ml of polluted waters ranged in initial Lead concentrations from 24 to approximately 700 mg/L, and Me from 24.808 to 202.938 for dosage 0.5 g at 20 °C at same lead concentration range. The Langmuir and Freundlich isotherm models for adsorption were used in analyzing the adsorption data and find their constants at different temperature levels to investigate if they can describe the obtained data well, Langmuir model was more representative of the data than Freundlich model. Bentonite clay would be very efficient in removing lead from industrial wastewater.

Keywords: Lead, Bentonitey, Adsorption, Atomic absorption, Langmuir & Freunlich models.,

ازالة الرصاص من المياه الملوثة بواسطة الادمصاص على دقائق طين البنتونايت

الخلاصة: تهدف الدراسة الى التحري عن قابلية نوع معين من طين البنتونايت لازالة عنصرالرصاص السام من المياه الملوثة بالادمصاص بدرجات حرارة مختلفة. عدة محاليل مركبة بتراكيز رصاص عالية تم تحضيرها مختبريا (24.3، 56.85، 146.55، 269.99، 284.72، 418.86، 465.61، 658.80 مليلتر)، و باستخدام حجم 250 مليلتر من كل محلول تم مزجه مع 2.5 غرام من البنتونايت تماما و وضع على صفيحة تسخين و باستخدام مازجة مغناطيسية لفترة ساعة بدرجة حرارة معينة (20، 40، 60 مئوية) و pH=7 لحين الوصول لحالة التوازن في عملية الادمصاص، بعد ذلك رشحت المحاليل بورقة نفاذة و الراشح تم فحصه بجهاز الامتصاص الذري لتحديد تركيز الرصاص المتبقي. تم الحصول على كفاءات ازالة عالية جدا (اكثر من 98%) و النتائج رسمت بمنحنيات لتبين العلاقات بين العوامل بظروف الفحص. اعيد اجراء نفس الخطوات السابقة باستخدام نفس التراكيز لمحاليل الرصاص المحضرة مع مزج 0.5 غرام من البنتونايت و فحصت بدرجة حرارة 20 مئوية و ايضا تم الحصول على نتائج ازالة عالية للرصاص. ان كتلة ايونات الرصاص المدمصة بوحدات المليغرام على كتلة البنتونايت بالغرام تراوحت من 2.387 الى 65.848 في الحالة الاولى عند استخدام 2.5 غرام بنتونايت و من 24.808 الى 202.938 عند استخدام 0.5 غرام بنتونايت بدرجة حرارة 20 مئوية و لنفس مدى تراكيز الرصاص. النماذج الرياضية ل Langmuir and Freundlich عند حالة التوازن لعملية الادمصاص استخدمت لتحليل النتائج المحصلة و ايجاد ثوابتهما و تحديد مدى تعبيرها عن النتائج و وجد ان نموذج Langmuir اكثر ملائمة من نموذج Freundlich. طين البنتونايت سيكون عالي الكفاءة في ازالة الرصاص من المياه الملوثة الصناعية.

1. Introduction

Lead is a chemical element in the carbon group with symbol Pb (from Latin: *plumbum*) and atomic number 82. Lead is a soft, malleable and heavy post-transition metal. Metallic lead has a bluish-white color after being freshly cut, but it soon tarnishes to a dull grayish color when exposed to air. Lead has a shiny chrome-silver luster when it is melted into a liquid. It is also the heaviest non-radioactive element. Lead is used in building construction, lead-acid batteries, bullets and shot, weights, as part of solders, pewters, fusible alloys, and as a radiation shield.

Toxic metals and metalloid such as lead, cadmium, arsenic, chromium, copper and mercury are released into the environment as a result of rapid industrialization, they cause damage in ecosystems and human health. Unlike pollutants of organic origin, heavy metals are non-biodegradable and can accumulate in living tissues in human bodies and other animals and plants inducing significant physiological disorders such as damage of central nervous system, blood structure, metabolism of energy and vital organs. Lead go into the natural waters from wastewater of various industrial activities such as metal plating, oil refining and battery manufacturing [1]. Human take lead ions through body via inhalation, ingestion or skin adsorption. It can act as a cumulative poison. Lead accumulates mainly in bones, brain, kidney and muscles and may cause many serious disorders like anaemia, kidney disease, nervous disorder and sickness even death [2, 3], lead can replace calcium, which is an essential mineral for strong bones and teeth, while play important role in sympathetic actions of nerve and blood vessel for normal functioning of nervous system. The high level of lead damages cognitive development especially in children. It also acts as an enzyme inhibitor in body, e.g. replaces essential element zinc from heme enzymes [4, 5]. World Health Organization standard give a maximum allowable limit of total Pb of $50 \mu\text{gL}^{-1}$ In drinking water while in USEPA standards is less than $15 \mu\text{gL}^{-1}$ [6].

Presently, heavy metals are be removed from wastewater by several methods include ion exchange, solvent extraction, foam floatation coagulation, electro-deposition, chemical precipitation, and membrane. Among these methods adsorption by clay is considered effective and cheap as compared to more expensive water treatment method such as ion exchange [7], precipitation with chemical and electrochemical methods or sometimes using sulphides [8-10]. A major problem with this type of treatment is the disposal of the precipitated wastes. Ion exchange treatment which is the second most widely used method for metal ion removal does not present a sludge disposal problem and has the advantage of reclamation of Cu (II) [11]. This method can reduce heavy metals to very low levels. However, ion exchange treatment does not appear to be economical. Activated carbon is also efficient for removal of trace elements from the waste water, but its high cost has prevented its wide usage. The adsorption phenomenon has still been found economically appealing for the removal of toxic metals from wastewater by choosing some adsorbents under optimum operation conditions. orange barks from commercial oranges may act as adsorbent for the removal of Pb^{2+} [12].

The adsorption properties of natural American Bentonite and activated carbon, the removal of Pb^{2+} from aqueous solution were studied, the results showed that the amount of adsorption of Pb^{2+} increases with initial metal ion concentration, contact time and solution pH but decreases with amount of adsorbent and temperatures, and it

was found that Bentonite is more effective for removal of Pb^{2+} than activated carbon. [13]. A lead (II) ion adsorption capacity of a natural Zeolite (clinoptilolite activated with HCl) from artificial polluted tap water was investigated in the study conducted by [14], and the highest removal efficiency was found being 89.95% at pH 5, and 30 °C.

This study investigated the adsorption capacity of a certain type of natural Bentonite clay to remove lead ions from polluted waters by adsorption phenomena, several synthetic solutions of certain lead ion concentration were prepared, mixed thoroughly with Bentonite clay at temperatures levels of 20, 30, and 40 °C with pH=7, then the samples filtered through a 0.2 micron porous paper, and tested for the remaining lead concentration by atomic absorption technique. Two types of adsorption isotherm models at equilibrium were studied; Langmuir and Freundlich models, if they can represent the obtained test data well using MatLap software drawing the relations between the variables, finding the model constants, and correlation coefficient. The Bentonite clay composition was determined by X-ray diffraction technique to identify its qualitative and quantitative characteristics.

2. Materials and Method

2.1. Materials

- Absorbent: In this research the absorbent used is a natural Bentonite clay.
- Adsorbate: Purified $Pb(NO_3)_2$ was used to prepare Lead solutions, it was supplied from Thomas Baker-chem. Limited.

2.2. Instruments

- Hot plate with thermostat and magnetic stirrer.
- Atomic absorption device (Model shimadzu, AA-7000, Flame-Flameless-MVU-1a, HVG-1), (Figure 3.).
- X-ray diffraction device (XRD-6000, Shimadzu, device for qualitative analysis & EDX-7000, Shimadzu, device for quantitative analysis) (Figure 4.)

2.3. Procedure

Lead solutions were prepared to having a concentration of 1000 mg/L as a stock using distilled water and Purified lead nitrate, then diluted by distilled water to get samples for equilibrium experiments in the required concentrations range (25 – 700 mg/L). After that, the initial concentration of the metal ion samples and the final remaining lead concentration for these filtered samples (after the equilibrium experiment completed) diluted and analyzed by the atomic absorption spectroscopy instrument.

Lead Concentration measurements of the unknown samples were conducted, by preparing standard solutions having different concentrations from 5 ppm to 40 ppm using distilled water as a solvent, and calibration curve was constructed by reading the absorbance values for this range of known concentrations using atomic absorption

spectroscopy instrument, a linear relation between absorbance and concentration for these sufficiently diluted solutions was gotten. then the absorbance of each sample solution that passing the equilibrium experiment and being filtered was measured.

Equilibrium state isotherms were established by adding a constant Bentonite clay mass of 2.5 g to 250 milliliters of lead solution in a glass beaker placed on hot plate controlled by a thermostat and subjected to constant agitation by magnetic stirrer for being thoroughly mixed (Figure 1.). In each isotherm run, the solute solution concentration was in the range of 25 – 700 ppm, and the study temperature levels were 20, 40, 60 °C. The experiments continued for enough time to ensure equilibrium of the adsorption process, after that the samples were filtered by micron permeable laboratory paper, put in test tubes which were numbered and designated for test conditions (Figure 2.), and their absorption measured by atomic absorption spectroscopy technique (Figure 3.).

Using the calibration curve for lead and the atomic absorption the remaining lead concentrations were specified. The results obtained in these test runs are shown in Table 2. for the test conditions indicated and using Bentonite mass of 2.5 g with the diluted samples of 250 ml volume of different concentration lead solutions, another test runs were conducted for the most favorable temperature concluded from the previous tests results using Bentonite clay mass of 0.5 g added to the same sample volume mentioned above also for different lead concentrations in the range 50-700 mg/L, and the results are shown in Table 3.



Figure 1.



Figure 2.



Figure 3.

The following mass balance equation was used to calculate the amount of lead ions adsorption on the adsorbent in the batch reactor:

$$M_e = V(C_i - C_e)/m \dots\dots\dots(1)$$

Where:

M_e indicates the adsorbed Lead ions mass in mg, to Bentonite mass in gram.

V is the solution volume, in liter.

C_i is the initial concentration of lead before adsorption on Bentonite clay in mg/L.

C_e is the concentration of lead after adsorption in equilibrium for the filtered samples.

The Bentonite clay chemical analysis identification was conducted using the X-ray diffraction instrument mentioned above, which indicated the clay to consist of the components as shown in Table 1.

Table 1. Chemical analysis of bentonite clay by XRF.

Composition	Content (wt %)	Composition	Content (wt %)
SiO ₂	50.459	MnO	0.021
Al ₂ O ₃	33.235	SrO	0.018
CaO	8.043	ZnO	0.010
Fe ₂ O ₃	5.581	ZrO ₂	0.008
K ₂ O	1.535	CuO	0.005
TiO ₂	0.501	NiO	0.003
SO ₃	0.497	MoO ₃	0.003
V ₂ O ₅	0.040	Y ₂ O ₃	0.001
Cr ₂ O ₃	0.030	CO ₂	0.010

3. Results and Discussion

The test runs results (Table 2., and 3.) show that, the type of Bentonite clay used in this study whose composition illustrated in Table 1., was very effective in removing lead ions from polluted water for different lead concentrations at good practical range of temperature.

The percentages of lead removals were more than 98% (98.18% ~ 99.95%) at temperature levels of 20, 40, 60 °C even for the very high lead concentration solutions that approaching 700 mg/L (Table 4.), when using 2.5 gm of the Bentonite clay added to 250 ml of polluted solutions.

Table 2. Lead concentration in filtered samples (mg/L), using 2.5 g of Bentonite adsorbent mass.

Lead initial concentration mg/L	24.309	56.859	146.551	269.996	284.725	418.866	465.613	658.809
At 20 °C	0.441	0.443	0.879	0.7221	0.279	0.468	0.605	0.330
At 40 °C	0.433	0.392	0.875	0.7016	0.247	0.582	0.723	0.527
At 60 °C	0.394	0.572	0.734	0.6914	0.893	1.243	0.818	0.712

Table 3. Lead concentration in filtered samples (mg/L), using 0.5 g of Bentonite adsorbent mass.

Lead initial concentration mg/L	50	100	200	300	400	500	600	700
At 20 °C	0.384	0.584	10.025	85.719	123.592	154.914	251.132	294.123

As well as the percentage of removal when using 0.5 g of Bentonite clay is still very high (Table 5.), taking in consideration that the anticipated lead pollution concentrations from different related industries are much less than these ranges (may be less than 10 mg/L) but the study considers the investigation of full capability of this specified type of Bentonite clay and its behavior under theoretical wide range of lead pollution concentrations, therefore this Bentonite was very efficient in removing Lead ions from polluted waters.

The mass balance equation (1) was used to calculate M_e ; the amount of lead ions adsorbed on the Bentonite clay adsorbent mass in the batch reactor at equilibrium for the different Lead concentration synthetic polluted solutions tested (C_i) which were ranged from 25 to approximately 700 mg/L at temperatures 20, 40, 60 °C, using an adsorbent mass of 2.5 g added to 250 ml of the above mentioned solutions and the equilibrium Lead concentrations (C_e) measured by the atomic adsorption technique, and in other test runs a mass of 0.5 g was used at 20 °C.

Table 4. Lead percent of removal at different Lead initial concentration in waste water when using 2.5 g of Bentonite clay as adsorbent mass.

Lead initial concentration mg/L	24.309	56.859	146.551	269.996	284.725	418.866	465.613	658.809
At 20 °C	98.18	99.22	99.40	99.73	99.90	99.89	99.87	99.95
At 40 °C	98.22	99.31	99.40	99.74	99.91	99.86	99.84	99.92
At 60 °C	98.38	98.99	99.50	99.74	99.68	99.70	99.82	99.89

Table 5. Lead percent of removal at different Lead initial concentration in waste water when using 0.5 g of Bentonite clay as adsorbent mass.

Lead initial concentration mg/L	50	100	200	300	400	500	600	700
At 20 °C	99.23	99.42	94.99	71.43	69.10	69.02	58.14	57.98

The relations given by the results are shown in Figures 5 ~ 9. The removal efficiency is little affected by temperature change for this range of lead concentrations solutions, therefore it would be economical to work at normal room temperature (around 20°C) without heating when applying this technique for treatment in practice. Results show little increase in lead removal efficiency when the temperature increased from 20 to 40, then to 60 °C at Lead concentration less than 250 mg/L, while little decrease with higher concentration at good dose of 2.5 g per 250 ml of polluted water volume.

Table 6. The amount of Lead ions adsorption on the adsorbent mass (M_e) in the batch reactor at equilibrium when using 2.5 g of Bentonite clay as adsorbent in units of mg/g.

Lead initial concentration mg/L	24.309	56.859	146.551	269.996	284.725	418.866	465.613	658.809
At 20 °C	2.387	5.642	14.567	26.927	28.444	41.840	46.500	65.848
At 40 °C	2.387	5.647	14.568	26.929	28.448	41.828	46.489	65.828
At 60 °C	2.392	5.535	14.582	26.930	28.383	41.762	46.479	65.809

Table 7. The amount of Lead ions adsorption on the adsorbent mass (M_e) in the batch reactor at equilibrium when using 0.5 gm of Bentonite clay as adsorbent in units of mg/g.

Lead initial concentration- mg/L	50	100	200	300	400	500	600	700
At 20 °C	24.808	49.708	94.987	107.140	138.204	172.543	174.434	202.938

Langmuir and Freundlich isotherm models of adsorption were used to analyse the obtained results and their constants [K , b , K_F , and n] calculated by nonlinear regression to indicate if they can describe the obtained test results well. The Langmuir isotherm equation is written as follows:

$$M_e = K C_e / (1 + b C_e) \dots\dots (2)$$

, the Langmuir equation supposes that Adsorption of metal ions on Bentonite clay is a monolayer and is applied to evaluate the maximum capacity of the clay.



Figure 4.

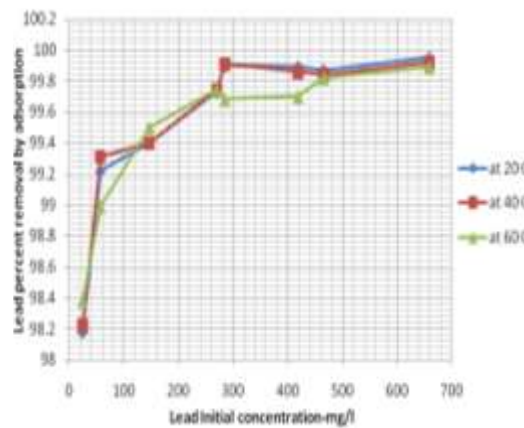


Figure 5. Lead ion removal efficiency versus Initial concentration at different temperatures using 2.5 g of Bentonite clay added to 250ml polluted solution

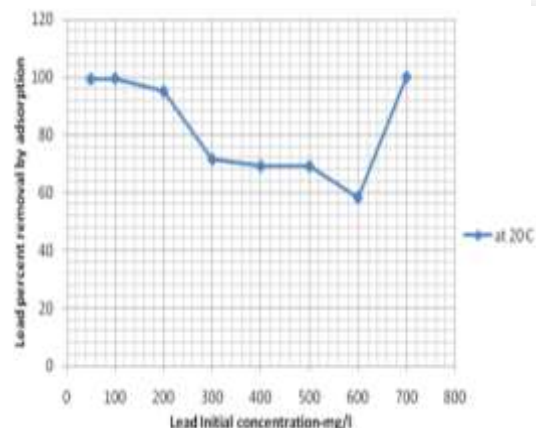


Figure 6. Lead ion removal efficiency versus Initial concentration at 20 °C using 0.5 g of Bentonite clay added to 250ml polluted solution

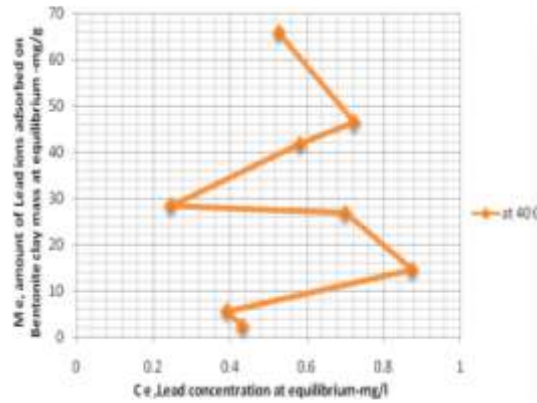


Figure 7. Adsorption equilibrium isotherm of lead in polluted water on Bentonite clay at temperature 20 °C, using 2.5 g of Bentonite clay added to 250ml polluted

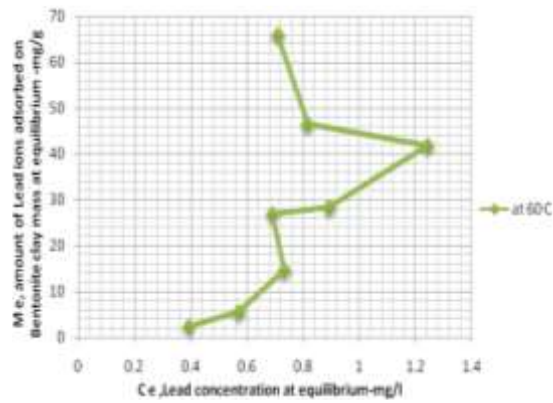


Figure 8. Adsorption equilibrium isotherm of lead in polluted water on Bentonite clay at temperature 40 °C, using 2.5 g of Bentonite clay added to 250ml polluted solution.

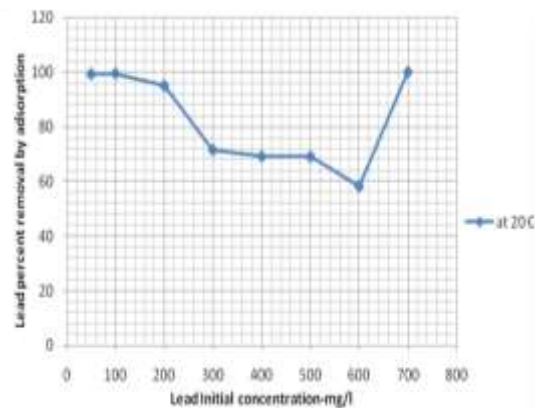


Figure 8. Adsorption equilibrium isotherm of lead in polluted water on Bentonite clay at temperature 60 °C, using 2.5 g of Bentonite clay added to 250ml polluted solution

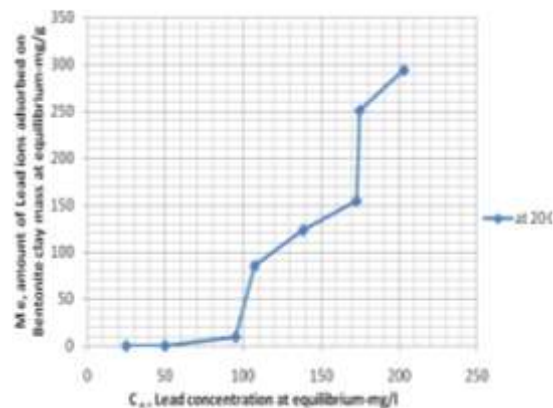


Figure 9. Adsorption equilibrium isotherm of lead in polluted water on Bentonite clay at temperature 20 °C, using 0.5 g of Bentonite clay added to 250ml polluted solution

The Freundlich isotherm model described the experimental data for heterogeneous surface, the model is written as follows:

$$M_e = K_F C_e^{1/n} \dots\dots (3)$$

The results of the non linear regression are shown in Table 8. and 9., they show that, Langmuir model describe the data well more than Freunlich model which is weak when high dosages of Bentonite clay is used and good at lower ones, in practice the range of pollution is much less than that investigated in this study since the object was to find the maximum capacity of removal for the Bentonite clay at different conditions.

Table 8. Langmuir constants for the Lead Ions Adsorption on Bentonite Clay at different temperature levels.

Bentonite clay dosage	Temperature ($^{\circ}\text{C}$)	K	b	R^2
2.5 g per 250 mL	20	-2.737	-11.95	0.835
	40	5.533	-1.753	0.688
	60	0.489	-1.23	0.907
0.5 g per 250 mL	20	74.05	0.444	0.670

Table 9. Freundlich constants for the Lead Ions Adsorption on Bentonite Clay at different temperature levels.

Bentonite clay dosage	Temperature ($^{\circ}\text{C}$)	K_F	n	R^2
2.5 g per 250 mL	20	16.255	-4.219	0.006
	40	29.991	1.459	0.059
	60	46.773	0.365	0.638
0.5 g per 250 mL	20	43.251	3.906	0.921

4. Conclusions and Recommendations

The Bentonite clay used in this research whose composition shown in table 1. determined by X-ray diffraction technique, was very effective in removing lead ions from polluted waters at different temperature levels, it works very good at high lead ions concentrations (24 ~ 700 mg/L), using enough dosage and suitable temperature, the removal percentage reached more than 98% (98.18% ~ 99.95%). The use of this high range of lead ions concentrations although in practice the generated ones from different industries are much less than these levels is to investigate the potential extreme adsorption capacity of this clay. It was seen that a dosage of 2.5 g clay per 250 mL of polluted water achieved very high removals for high concentrations approaching 700 mg/L and it looks possible if higher concentration solutions than 700 mg/L be used to still have very high removals on wide range until the adsorption capacity of the Bentonite is exhausted and its particle surfaces saturated and this can be studied in future.

The use of lower dosage of Bentonite (0.5 g per 250 mL) for the same range of lead ions concentration mentioned above achieved very good results of removals ;more than 95% for Lead concentrations less than 200 mg/L, but decreases gradually at higher concentration because of depletion of clay adsorption capacity and therefore needs higher dosage. The temperature effect on clay adsorption capacity was also studied and the results show minor effect for this type of clay, where little increase in removal efficiency at lead concentrations of less than 250 mg/L when temperature increased from 20 to 40, then to 60 $^{\circ}\text{C}$, while little decrease in removal at concentrations that are higher than 250 mg/L. The adsorbed lead ions mass in mg, on Bentonite mass in gram (M_e) were ranged from 2.387 to 65.848 at temperature levels of 20, 40, 60 $^{\circ}\text{C}$ and Bentonite clay dosage of 2.5 g per 250 ml of polluted waters ranged in initial lead concentrations from 24 to approximately 700 mg/L, and M_e from 24.808 to 202.938 for dosage 0.5 g at 20 $^{\circ}\text{C}$ at same lead concentration range.

Therefore from previous review the use of this type of Bentonite clay for adsorption of lead ions from high concentration polluted waters will be very economical and effective treatment method in practice (same as that found by Yarkandi [13], first removing large quantities of lead by adsorption then using chemical precipitation with or without filtration or other techniques to get very low lead concentration water be used for drinking or industrial sensitive purposes at low costs. The treatment in prototype scale can be done in normal room temperature without heating as long as temperature has minor effect on removal efficiency.

Two adsorption models were tested if they can match the obtained results of this study, Langmuir adsorption isotherm model and Freundlich adsorption model [7], it was found from non linear regression that, Langmuir model is well representing the result data with good correlation coefficients as stated in table above at the test conditions indicated in this research, while Freundlich model was far from that at high Bentonite clay dosage of 2.5 g per 250 ml of polluted waters and temperature levels of 20, 40, and 60 °C, where low value of correlation coefficients were obtained, but when Bentonite dosage of 0.5 g was used at temperature, a very good correlation being obtained by Freundlich model ($R^2=0.921$), which means good representation of the data and we can make good inferences for other results at different test variables. Other more accurate adsorption models by non linear regression can be found.

Future studies are recommended to investigate the adsorption capacity of this Bentonite clay type at different range of pH than used in this study (pH=7), as well as the effect of mixing time and mixing intensity to reach the adsorption equilibrium state in shorter time than used in this study(one hour) and medium mixing which was fixed during test runs as a parameter affecting the results. Further studies to determine the ability of this type of Bentonite clay in removing other contaminants such as Chromium, Zinc, Cadmium, Copper, Zinc, ...etc, or halogenated organic persistent compounds, such as pesticides and THMs will be of great benefit to treat drinking water and industrial waste water from different origins.

5. References

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