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Granules of brick waste (GBW) as low-cost sorbent for removal of Pb^{+2} ions from aqueous solutions

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Abstract— The potential application of granules of Granular brick waste as a low-cost sorbent for removal of Pb^{+2} ions from aqueous solutions has been studied. The properties of Granular brick waste were determined through several tests such as X-Ray diffraction , Energy dispersive X-ray, Scanning electron microscopy , and surface area. In batch tests, the influence of several operating parameters including contact time, initial concentration, agitation speed, and the dose of GBW was investigated. The best values of these parameters that provided maximum removal efficiency of lead (89.5 %) were 2.5 hr, 50 mg/L, 250 rpm, and 1.8 g/100mL, respectively. The sorption data obtained by batch experiments subjected to the three isotherm models called Langmuir, Freundlich and Elovich. The results showed that the Langmuir isotherm model described well the sorption data (R^2= 0.9866) in comparison with other models. The kinetic data were analyzed using two kinetic models called pseudo_first_order and pseudo_second_order. The pseudo-second-order kinetic model was found to agree well with the experimental data.

Keywords— Sorption; Pb^{+2} ions,Granular brick waste, Isotherms, Kinetics.

1. Introduction

Many branches of industry nowadays produce large quantities of wastewater containing danger and carcinogenic organic and inorganic compounds. Heavy metals are one of the most common toxic contaminants that involve cadmium, lead, cobalt, zinc, nickel, and manganese etc. ,it's not decomposed, so metal concentrations should be minimum to reasonable concentrations levels before drain it to the environment, [26] . For example, lead presented in the wastewater of many industrial processes, such as the fabrication of dyes, batteries, glass, and paint coatings etc., [18]. The maximum acceptable concentration of Pb^{+2} ions recommended by the World Health Organization (WHO) for drinking water is 2 μg/L, [1]. The exposure of humans to lead element creates acute toxicity and diseases such as edema , immune system disorders ,and damage to organs (liver and heart etc.), [25]. When permissible concentrations of metals ions exceeded, these metal ions exhibit toxic characteristics and non-biodegradable compounds. For that, creator methods of wastewater handling are continuously being developed to treat polluted water. The conventional methods such as Precipitation, ion exchange, solvent extraction ,and sorption are the commonly utilized methods for treating water polluted with metals, [10]. Many sorbents can be utilized for removal of toxic metals from contaminated water but porous carbons are utilized extensively in contrast to other methods because these sorbents have high sorption capacity, and easily regenerated, but is considered an expensive sorbent material. For that reason, different cheap materials such as metal oxides, [6]; zeolites ,[23]; iron oxide-coated sand , [4]; and clay minerals, [13], had recently been examined with the purpose to prove their capability of heavy metal removal from wastewater. But, the solution of specific water issues encountered in Societies of developing countries had required the elaboration of proven and locally more suitable water treatment procedures at low costs. In the last years, the brick waste utilized as a cheap material to remove soluble toxic metal ions contaminants from wastewaters had been studied. [3] ;[7].The nature of brick material, surface area, and surface charge influence the extent of interaction with sorbates. As brick granular are negatively charged, therefore cations will be strongly attached to brick granules [22]. Within this scope, the present work examines the feasibility of using granules of brick waste as abundant, available, and low-cost sorbent material for removal of Pb^{+2} ions from contaminated water.

2. Experimental work

2.1 *Granular of brick waste (GBW) preparation*

Pieces of brick waste, which are left unused after construction, were used as sorbent in this study. It were crushed and sieved with size ranging from 1.18 to 1.7 mm. The obtained granular were washed several times with distilled water then dried as shows in figure 1.

Figure 1: Granular of brick waste (GBW)

2.2 *Characterization of GBW*

2.2.1 X-Ray diffraction (XRD) analysis

The surface qualitative analysis was carried out to characterize and confirm the existence of the major components; samples were analyzed before uptake of lead. This analysis was accomplished using (BRUKER , D2 PHASER, Germany) ,at the German Laboratory/ Geology Department/ College of Science/ University of Baghdad.

2.2.2 Energy dispersive X-ray (EDX) analysis

Energy Dispersive X-Ray analysis is a chemical microanalysis technique used in conjunction with scanning electron microscopy (SEM). It's used to recognize the elemental composition of materials. This test carried out using (TESCAN, Vega III , Czech Republic), at Production Engineering and Metallurgy Department University of Technology - Iraq .

2.2.3 Scanning electron microscopy (SEM)

Scanning Electron Microscope (TESCAN, Vega III , Czech Republic) was utilized for surface studies of GBW,the test measure at at Production Engineering and Metallurgy Department University of Technology - Iraq . Using the optimized conditions for the sorption of Pb^{+2} ions , the loaded mass was filtered, washes ,and dried at 105 ^{o}C for 30 min. A blank mass (unloaded) was also subject to the same conditions, and both the loaded and unloaded mass was subjected to SEM test to identify the influences and changes on the surface of GBW before and after lead molecules sorption.

2.2.4 Surface area

Surface area is an important factor in determining the active sites that will be occupied with the contaminants. Therefore, increasing the surface area of the material increases its susceptibility to adsorb more quantity of pollutants. The BET surface area was tested using (Quanta chrome , USA), at the Petroleum Research and Development Center-Ministry of Oil/Baghdad-Iraq.

2.3 *Preparation of synthetic wastewater*

The synthetic solution of lead with a concentration of 1000 mg/l was prepared by dissolving a 1.5985g of lead nitrate ($Pb(NO_3)_2$) in 1 L of distilled water .The pH of synthetic solution was set using 0.1 M of HNO_3 or NaOH as needed.

2.4 *Sorption experiments*

The experiments were achieved to identify the better conditions of the contact time , initial concentration of metal ions (Pb^{+2}), agitation speed ,and dosage of the sorbent. A number of flasks (250 ml) are utilized and each one is filled with Pb^{+2} ions solution (100 ml) which has (50 mg/L) initial concentration , initial pH= 4 ;and about 1 g/100ml of GBW was added into each flask . the flasks were preserved stirred in 200 rpm (speed orbital shaker) at ambient temperature. Then the sorbent was separated from the pollutant solution by filtration.

These tests were conducted at different time (10,20,30,50,90,150,180 and 240 min.) **,** initial concentration (50, 100,150,200,250 mg/L) **,** agitation speed (0, 50, 100, 150, 200 ,and 250 rpm), and sorbent dosage (0.2, 0.4, 0.6, 0.8,1 ,1.1 ,1.2 ,1.3,1.5,1.8 and 2 g/100 ml). The metal ion concentration at saturation was measured by atomic absorption spectrometry (AAS). The concentration of Pb^{+2} ions sorbed by GBW was determined from the difference between the initial and final concentration of Pb^{+2} ions solution which obtained before and after the communicate between the GBW and the pb^{+2} ions solution. The sorption capacities were determined using Eq. (1), **[16]:**

$$q_e = \frac{C_o - C_e}{m} V \qquad (1)$$

Where:

q_e : is the amount of sorbed Pb^{+2} ion per unit mass of GBW (mg/g).

C_o and C_e: are the initial and equilibrium concentrations of lead in the solution (mg/L).

V: is the volume of solution (L).

m: is the mass of the sorbent GBW (g).

The removal efficiency (R %) of the Pb^{+2} ions was calculated using Eq. (2), **[16]:**

$$R\% = \frac{C_o - C_e}{C_o} x100 \qquad (2)$$

3. Isotherm Models

In the current study, three isotherm models is used to simulate the performance of GBW in removing lead ions from wastewater. A summary of these models is presented below:

- **Langmuir model:** assumes a surface with homogeneous binding sites, equivalent sorption energies, and no interactions between sorbed species, **[14]**.The linear equation of langmuir model can be written as follows:

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

where :

q_{max} : is the maximum sorption capacity (mg/g).

K_L : is the Langmuir model sorption constant (L/mg).

C_e : is the concentration of Pb^{+2} ions (mg/L) at equilibrium.

The plot of (C_e/q_e) against (C_e) gives a straight line with a slope (1/qmax) and (1/qmax K_L) intercept .

- **Freundlich model**: It's an empirical model not limited to monolayer coverage alone but also describe multilayer adsorption , **[14]**. It is expressed linearly as in Eq.4:

$$\ln q_e = \frac{1}{\mathrm{n}} \ln C_e + \ln \mathrm{K_F} \qquad (4)$$

Where:

K_F: is the Freundlich model adsorption coefficient.

C_e : is the concentration (mg/L) of Pb^{+2} ions at equilibrium .

n : is an empirical coefficient indicative of the intensity of the sorption.

The Plots of (log qe) against (log Ce) gives a linear graph with slope (1/n) and intercept (log K_F) from which (n) and (K_F) can be determined respectively.

- **Elovich model:** is instituted on a kinetic basis, suppose that the sorption effective sites rise exponentially with sorption, which denote a multilayer adsorption, **[2]**. It can be written as follows:

$$\ln \frac{q_e}{C_e} = \ln \mathrm{K_E}\, q_m - \frac{q_e}{q_m} \qquad (5)$$

Where:

K_E : is the Elovich equilibrium constant (L/mg) and qm :is the Elovich maximum adsorption capacity (mg/g).

4. Kinetic models

Kinetic sorption models are helpful to understand the mechanism of the sorption process of lead onto GBW. These models include pseudo first order and pseudo-second order, **[8]**.

- The pseudo - first - order kinetic rate equation is :

$$\ln(q_e - q_t) = \ln(q_e) - \mathrm{K_1 t} \qquad (6)$$

Where:

q_e and q_t: is represent the amounts of Pb^{+2} ions sorbed per unit mass of GBW at equilibrium (mg/g) and time t (min) respectively.

K_1 : is represent the rate constant of pseudo-first-order sorption (1/min).

- The pseudo - second - order model kinetics rate equation is:

$$\frac{\mathrm{t}}{q_t} = \frac{1}{\mathrm{K_2}\, \mathrm{q}_e{}^2} + \frac{\mathrm{t}}{q_e} \qquad (7)$$

Where K_2 (g / (mg.min)) is the rate constant of pseudo-second-order adsorption.

5. Results and discussion

5.1 *Characterization of GBW*

5.1.1 XRD analysis

The XRD measurement was performed to identify the mineralogical composition of GBW. Figure 2 illustrates that GBW was composed mainly of Diopside (29.9%), Quartz (22.4%), wollastonit (22.2%), akermente (20.5%) and Mellite (5%). Diopside Originating from dolomite (CaO, MgO, $2CO_2$),also show that quartz and calcite compound are the popular compounds, with with the addition of other clays and clay minerals. However, due to the application of high temperature handling during the manufacturing process, the decomposition of clay minerals occurred which forming (SiO_2) compound , followed by the loss of their crystal structure.The $CaCO_3$ compound also decompose and resulting (CaO) compound that may react with the clay which resulting calcium-silicate called wollastonite**[12].**

Figure 2: XRD of GBW.

5.1.2 EDX analysis

The EDX analysis was carried out and the spectra is given in figure 3. This figure indicates that the GBW composed of oxygen, calcium ,silicon , aluminium , iron , magnesium, sodium, potassium and sulfur, with percentage of 44.2, 21.7, 18.1, 5.7,5, 2.8, 1.4, 0.6 and 0.6% , respectively. The test for GBW showed the presence of silicon, oxygen , calcium and other small percentages of metals. The existence of these oxides and hydroxides in GBW results in it's having a various sorption capacity, **(24)**

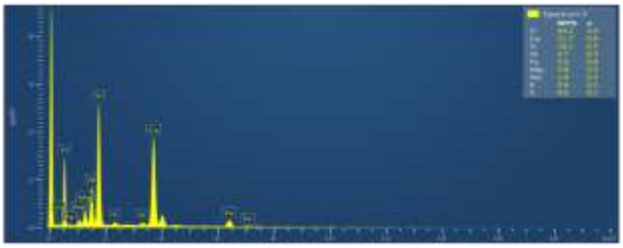

Figure 3: EDX spectra of GBW.

5.1.3 SEM analysis

The SEM analysis images at 50 µm obtained before and after sorption to determine the surface morphology. Figure 4 (a) represents the SEM spectra of the of GBW before lead loading, its shows irregular structure having small pores, which facilitate the process of sorption, while the SEM micrograph after lead sorption figure 4 (b) indicating that these pores become filled with the sorbate (lead), **[11].**

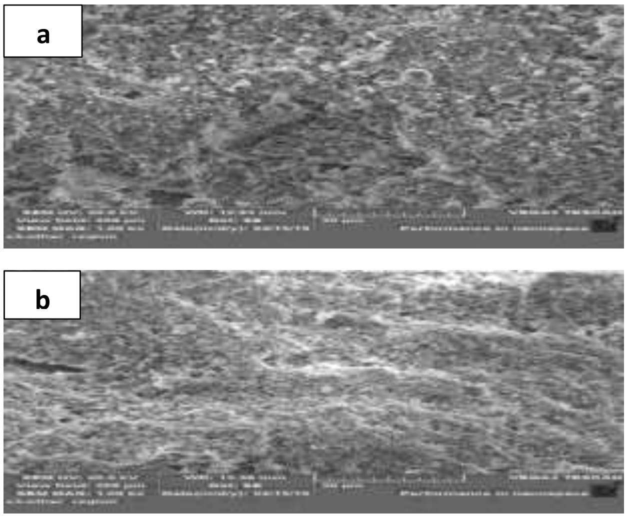

Figure 4: SEM spectra of the of GBW (a) before and (b) after removal of pb^{+2} .

5.1.4 Surface area

The results of this test clarify that the GBW sample show a low surface area of ($1m^2/g$).this results in a good agreement with previous study of **[15]**.

5.2 ***Influence of batch operating parameters***

5.2.1 Effect of contact time

The impact of the contact time on sorption of pb^{+2} ions using GBW was studied by using a contaminated aqueous solution with initial lead concentration 50 (mg/L) at pH=4.The relation between the contact time and removal efficiency of lead ions is shown in Fig.5, the best removal efficiency (77%) was reached within about 150 (min). The sorption of Pb^{+2} ions occurred in two periods, initial rapid sorption followed by subsequent slow sorption. The sorption process appeared to proceed rapidly when the numbers of active sites are higher than the number of metal species to be sorbed, [20]. The increase of solution pH during the contact with GBW due to dissolution some of the sorbent (GBW) components**, [12].**

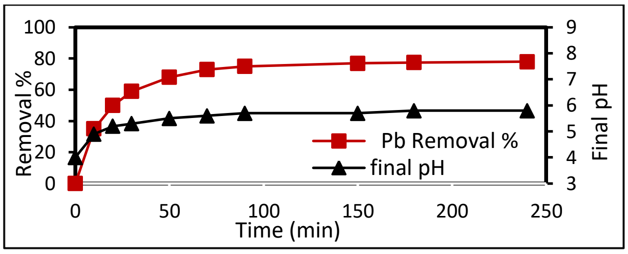

Figure 5: Effect of contact time on Pb^{+2} removal percent (C_o = 50 mg/L, pH= 4, agitation speed = 200 rpm, and mass of GBW = 1g /100ml).

5.2.2 Effect of initial concentration

The initial concentration of metal ions is a very important parameter to be investigated in sorption studies as most polluted wastewaters usually exist at various concentrations of metal ions, so the determination of its influence is important for an elaborate sorption study. The influences of initial Pb^{+2} ions concentration on the percentage removal of Pb^{+2} ions using GBW is shown in figure 6. The removal efficiency of lead decreased with an increase in the initial Pb^{+2} ions concentration from 50 to 250 (mg/L). A decrease in percentage removal from 77 to 48 % of Pb^{+2} ions was obtained. This decrease is due to the fact that the sorbent material (GBW) has a fixed number of active sites and at higher concentrations, the active sites become saturated, **[19].**The simple hydrolysis of generality divalent metal ions can be written as follows:

$$M^{+2} + H_2O \rightleftharpoons M(OH)^{+} + H^{+} \quad (8)$$

where M^{+2} is either Pb^{+2} or any metal ions.The final pH increase when the concentration of the solution decreases. This happens when GBW is being taken up by the GBW . As represented in the reaction above , the reaction shifts to the left, leading to the depletion of protons and hence increase in pH ,**[9]**

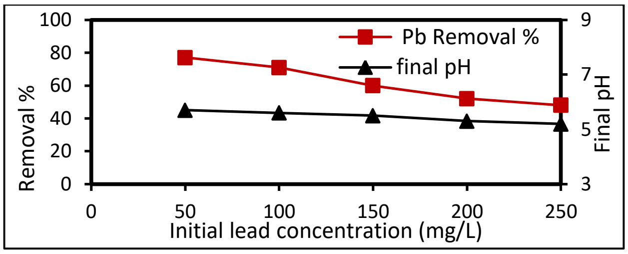

Figure 6: Effect of initial concentration on Pb^{+2} removal percent (Time=150 min, pH=4 , mass of GBW = 1g/100 ml, and agitation speed = 200 rpm).

5.2.3 Effect of agitation speed

The effect of agitation speed of the sorbent/sorbate system was monitored at (0, 50, 100, 150, 200, and 250 rpm) as shown in figure 7.The slight increase in percentage removal primarily cause the fact that agitation facilitates proper contact between the lead ions and the GBW the effective sites and consequently promoting effective transfer of Pb^{+2} ions to the GBW effective sites, **[4]**. In addition, the increase of the pH of the solution during the contact with GBW due to the dissolution of some GBW components as reported by ,**[12]**.

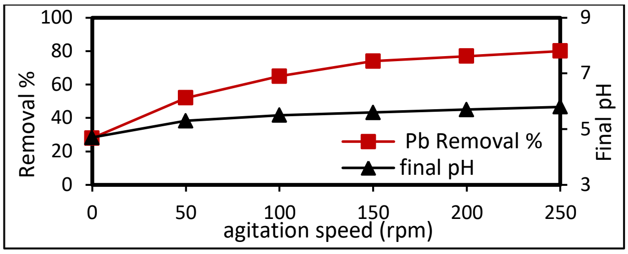

Figure 7: Effect of agitation speed on Pb^{+2} removal percent (C_o = 50 mg/L, Time=150 min, pH=4, and mass of GBW = 1g /100ml)**.**

5.2.4 Effect of the dosage of GBW

The study of the amount of GBW that utilized for the removal of pb^{+2} ions was carried out using the various dosage of GBW range from 0.2 - 2 g.

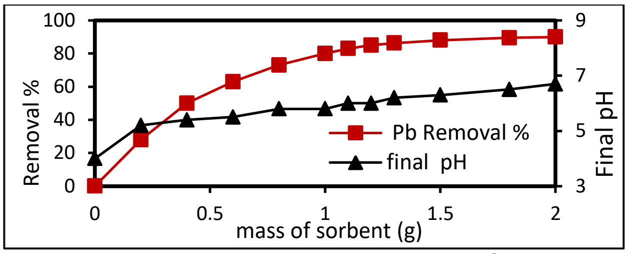

Figure 8: Effect of sorbent dosage on Pb^{+2} removal percent (C_o=50 mg/L, Time=150 min , pH=4 ,agitation speed = 250 rpm).

The influence of sorbent dose on the sorption of lead by GBW was shown in figure 8. As represent in figure 8, the lead sorption percent increased with increase of sorbent (GBW dosage) . The increase in the GBW mass improved the availability of more effective sites for the sorption, thus making easier penetration of Pb^{+2} ions to the sorption effective sites, **[20].** Moreover, the final pH increase due to release the amounts of dissolved Ca^{+2} and other light metal alkalis in solution during the reaction between GBW and Pb^{+2} ions, **[12].**

5.3 *Sorption isotherms*

The sorption data for Pb^{+2} ions are fitted with linearized equations of three models namely Langmuir, Freundlich and Elovich. Accordingly, the empirical coefficients for each model were determined from the slope and intercept of the linear plot using Microsoft Excel 2013 software. The isotherm graphical representations of these models are shown in figure 9. All constants are presented in Table 1. Since the value of R^2 nearer to 1 denotes that the respective equation a good fits the experimental data **[17]**. So, the Langmuir isotherm model was concluded to be preferred models for the experimental data.

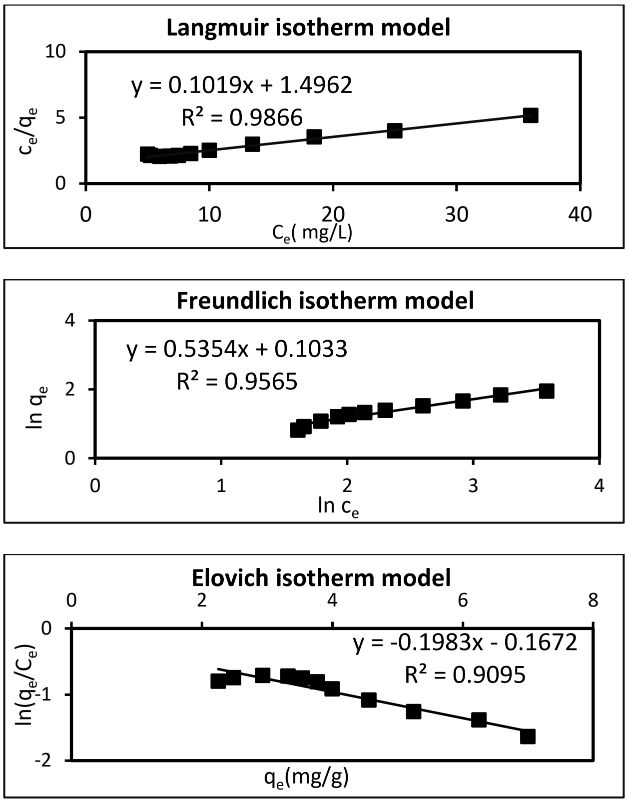

Figure 9: Isotherm models plot.

Table 1: Sorption isotherm constants with coefficients of determination for Pb $^{+2}$ onto GBW.

Isotherm model	Parameters	GBW
Langmuir	q_m (mg/g)	9.81
	b (L/mg)	0.1019
	R^2	0.9866
Freundlich	K_F (L/mg)	1.108
	n	1.8677
	R^2	0.9565
Elovich	q_m (mg/g)	5.04
	K_E (L/mg)	0.967
	R^2	0.9557

5.4 *Kinetic study*

To identify the type of sorption mechanism occurs, the kinetic equations namely pseudo first order and pseudo second order were utilized. It is clear from figure 10 and Table.2 that the reaction for GBW is second order because the value of the experimental qe was the closest to the qe calculated from the pseudo second order in compared with the pseudo-first-order model, irrespective to the amount of the correlation coefficient (R^2) , so that the mechanisms will be chemisorpion.

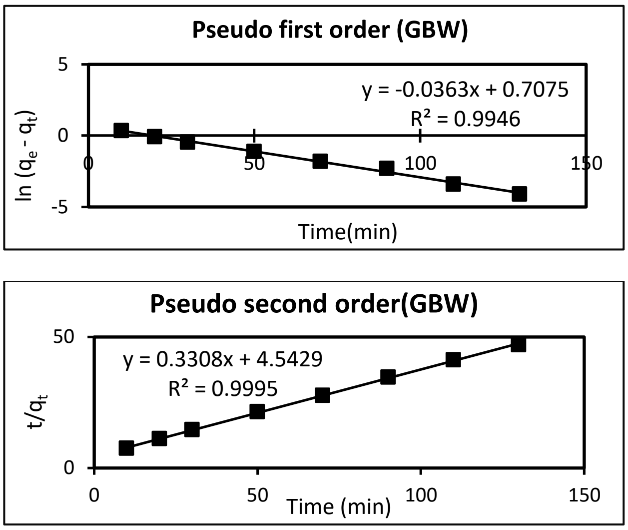

Figure 10: The kinetic models for sorption pb $^{+2}$ onto GBW.

Table 2: The kinetic constants for the sorption of Pb^{+2} onto GBW.

qe (mg/g) Exp.	Pseudo first order	Pseudo second order
	K1(1/min) 0.0363	K2(g/mg.min)0.0241
2.93	qe calc..(mg/g) 2.02	qe (calc.) (mg/g) 3.02
	R2 0.9964	R2 0.9995

6. Conclusion

Based on the results obtained from the experimental work, the following conclusions can be drawn:

- The granular of brick waste (GBW) material proved effectiveness in removing Pb^{+2} ions from aqueous solutions with removal percent of 89.5% according to the experimental conditions. Therefore, it can be utilized as efficient and low- cost sorbent for the removal of pb^{+2} ions from contaminated solutions
- The batch results indicated that several parameters including contact time, initial concentration, agitation speed, and granular brick waste dose affect the sorption process. The optimum values of these factors which provided maximum removal percent (

89.5 %) of Pb^{+2} with initial pH of 4 were 150 min, 50 mg/l, 250rpm, and 1.8/100ml, respectively. The maximum sorption capacity for GBW (2.93mg/g)

- The isotherm study refers that the sorption data correlated well with Langmuir isotherm model which showed the highest value of the correlation coefficient (R^2= 0.9866).
- The kinetic study showed that the pseudo second order kinetic model was conformed better than pseudo first order kinetic model. This result clarifies that chemisorption has been predominant in the sorption of Pb^{+2} ions using GBW.

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حبيبات نفايات الطابوق كمادة منخفضة التكلفة في ازالة أيونات الرصاص pb^{+2} من المحاليل المائية

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الخلاصة – تمت دراسة امكانية تطبيق حبيبات مخلفات الطوب (GBW) كمواد منخفضة التكلفة لإزالة أيونات Pb^{+2} من المحاليل المائية. تم تحديد خصائص GBW من خلال العديد من الفحوصات مثل حيود الأشعة السينية (XRD) ، الأشعة السينية المشتتة للطاقة (EDX) ، المجهر الإلكتروني المسح الضوئي (SEM) ، والمساحة السطحية. في اختبارات الدُفعات ، تم فحص تأثير العديد من العوامل بما في ذلك وقت الاتصال والتركيزالابتدائي وسرعة الرج وجرعة GBW. كانت أفضل قيم المعاملات التي وفرت أقصى كفاءة إزالة للرصاص (89.5%) : 2.5 hr ، 50mg/L ، 250rpm ، و 1.8 g/100ml ، على التوالي. بيانات الامتصاص التي تم الحصول عليها من خلال تجارب الدُفعات التي خضعت لنماذج isotherm الثلاثة المسماة (Langmuir ، Freundlich ،و Elovich). أظهرت النتائج أن نموذج Langmuir وصف بشكل جيد بيانات الامتصاص (R^2 =0.9866) بالمقارنة مع النماذج الأخرى. وقد تم تحليل البيانات الحركية باستخدام نموذجين : pseudo-first-order وpseudo-second-order). تم ايجاد ان pseudo-second-order يمثل جيدا البيانات التجريبية.

الكلمات الرئيسية –" Sorption; Pb^{+2}; brick waste; Isotherms; Kinetics ".