Performance of zero-valent iron barrier through the migration of lead-contaminated groundwater

Prof.Dr. Ayad Abdulhamza Faisal
Rawaa Jaffar Mohammed
Department of Environmental
Engineering-University of Baghdad
E-mail: ayadabedalhamzafaisal@yahoo.com
rawaajaffar@gmail.com

ABSTRACT

The Freundlich and Langmuir isotherm models are frequently used for modeling of sorption data at constant value of pH. The present study modified the Langmuir isotherm model for simulating the sorption effects as a function of initial pH of the aqueous solutions. This model was developed using the batch results for sorption of lead from contaminated solution onto zero-valent iron (ZVI) sorbent under specified parameters. These parameters were; initial pH of (2–7), contact time (≤120 min), agitation speed of (0–250 rpm) and ZVI weight not exceeding 2 g/100 mL) for initial lead concentration ranged from 50 to 250 mg/L. Experimental results proved that the sorption capacity and affinity constants of the developed isotherm model are changed dramatically with initial pH of aqueous solution. These constants have values of 23.62 mg/g and 0.3566 L/mg at best initial pH of 5. The sorption capacity-initial pH was correlated using polynomial regression, while the affinity coefficient–initial pH was represented by linear relationship. In addition, stoichiometric ratios between cumulative iron elution and cumulative lead removal at best operation conditions proved that about 0.34 mg of iron is required to remove 1 mg of lead ions. The kinetic rate constants showed that the lead uptake was increased threefold when pH changed from 2 to 5, while this value decreased to its half when pH changed from 5 to 7.

Keywords: Lead removal, sorption process, ZVI, permeable barrier, groundwater remediation.

1. Introduction

Water found beneath the ground surface and seeped down from the surface by migrating through the soil matrix and spaces in geologic formations is defined as "groundwater" **[3]**. This water can be utilized for domestic, irrigation and industrial uses and according to available literatures more than half of the population in the United States depends on this water as water supply. In addition, groundwater used to cover the requirements of the factories with process water and farms with irrigation water. On the national level, these literatures signified that twenty five percent of used water (included water uses in the power production and irrigation) taken from groundwater. Accordingly, efforts of scientists and researchers are directed toward the protection of this water from contamination and finding the efficient techniques to remediate it if this water is polluted **[7]**.

The ‘pump-and-treat’ technology is conventional system used since the beginning of the past century in the remediation of contaminated groundwater. However, the previous literatures proved that this technique cannot be achieved the clean-up goals in comparison with acceptable environmental regulations. Thus, efforts for finding suitable and innovative sustainable techniques used in remediation of groundwater were begun from the beginning of 1990s. In this direction, permeable reactive barrier (PRB) is developed which represents a novel technology for in situ remediation process **[16]**. A wide spectrum of contaminants (organic and/or inorganic) such as chlorinated solvents, petroleum hydrocarbons, metals and radionuclides can be removed by this technology **[6]**.

The principle of PRB technique is depended on the creation of a vertical wall across the flow of water and this wall must be packed with material of high permeability and reactivity. Through the movement of contaminated groundwater in this wall, the contaminants can be retarded by sorption or transformed (biologically or abiotically) in such away where the treated water in the downstream side of PRB doesn't cause any risk for water resources and receptors. Lower costs of operation, maintenance and monitoring as well as complete capture of contamination plume and treatment of this plume in the subsurface are the most characteristics of this approach in comparison with other remediation technologies **[10]**.

Reactive media used in these barriers should be compatible with the subsurface environment and no cause adverse chemical reactions or byproducts when reacting with constituents in the contaminant plume. Also, this media must be persistent with time to maintain the minimum cost of PRB. The availability of PRB materials in suitable cost and adequate their particle sizes to avoid the blockage are the most conditions required for efficient application of this technique **[11]**.

Theoretical and experimental studies on PRBs using activated carbon, zeolite and zero-valent iron (ZVI) for the removal of heavy metals from groundwater have been performed such as **[2,9]**. For example, a continuous column experiment was conducted under dynamic flow conditions to study the efficiency of low-cost PRBs to remove several inorganic contaminants from acidic solutions. A 50:50 w/w waste iron/sand mixture was used as the candidate reactive medium to activate precipitation and promote sorption and reduction–oxidation mechanisms Also, the treatment of the groundwater contaminated with metals by dumping sites located in the provinces of southern Poland was investigated. The simulated groundwater circulated through the column filled with ZVI in the laboratory tests. Chromium, Copper, Nickel, Cobalt, Lead, Cadmium, and Zinc, occurring in the water as cations and anions, were removed in the iron bed **[15]**. Accordingly, the present study is aimed to derive a sorption isotherm expression for simulating pH-dependent sorption effects on the removal of lead from aqueous solution using ZVI as sorbent

2. Experimental Methodology

2.1 Materials

Lead stock solution of 1000 mg/L was prepared using 1.5985 g of lead nitrate **(Table 1)** in one liter of distilled water. A certain quantity of HNO_3 or NaOH with concentration of 0.1 M was used as required for controlling the pH of the aqueous solution at room temperature.

Zero-valent iron (ZVI, Fe^0) (with oxidation number of 0, 2, 3) was represented the best choice for using it as fixed-bed barrier in the in-situ remediation of contaminated groundwater. It is promising medium due to its availability, low cost, and sustainable development benefits in terms of reuse of solid wastes as well as a means of cutting disposal costs. Safety, stability and adequate hydraulic conductivity are the most favorable properties for using the ZVI reactive material in the remediation process **[13].** Scrap iron was used to prepare the ZVI which has particle size distribution ranged from 0.6 to 1 mm **(Fig.1)** and porosity of 0.51. Acetone was used in the washing of the prepared iron. Then, the iron was dried and stored in the aerated environment until needed in the experiments. Physico-chemical characteristics of the used ZVI are listed in the **Table2.**

Table 1. Characteristics of lead nitrate [8].

Property	Description
Molecular formula	$Pb(NO_3)_2$
Structure	
Appearance	White colorless crystals
Formula weight (g/mole)	331.2
Atomic weight	207.2
Solubility in water (g/100 mL)	52
Charge	+2
Density (g/cm^3)	4.53
Melting point (K)	543
Manufacturing company	BDH, England
Purity (%)	99.5
Wavelength (nm)	283.3

Table 2. Characteristics of ZVI used in the present study

Property	Value
Fe^0 (%)	90
Real density (g/cm^3)	6.542
Average surface area (m^2/g)	0.555
Atomic number	26
Atomic mass	55.85
Melting point	1536
Boiling point	2861

Fig.1: Prepared ZVI from scrap iron.

2.2 Batch Experiments

The effect of different operation conditions for specifying the maximum removal efficiency of lead ions were studied by batch experiments which are conducted in four steps. These conditions are included ZVI dosage, initial metal concentration, agitation speed, initial pH of the aqueous solution and contact time. These steps were:

1-required the variation of ZVI weight (0.05, 0.1, 0.3, 0.5, 0.8, 1 and 2 g) added into 100 mL of contaminated solution; while the other parameters; initial lead concentration, agitation speed, initial pH and contact time are fixed on the values of 50 mg/L, 250 rpm, 3 and 60 min respectively.

2- required the using a constant sorbent dosage chosen from the results of the first step with changing the initial lead concentration from 50 to 250 mg/L. Agitation speed, initial pH, and contact time were maintained at their previous values.

3- Required the changing of the agitation speed from 0 to 250 rpm with an initial concentration chosen from the results of the second step. Other operating conditions were maintained as in the second step.

4- Contact time and initial pH were changed from (10 to 120 min) and (2 to 7) respectively in the last step with agitation speed value taken from the results of the third step; other conditions being fixed at their third step values. Flasks with capacity of 250 mL were used in the aforementioned experiments; where 100 mL of lead solution was placed in each flask and then ZVI dosage was added. The prepared flasks were agitated using an orbital shaker (Edmund Buhler SM25, German) until achieving the equilibrium state. Exactly, 20 mL of treated water was pipetted out from each flask at the end of any experiment and subsequently filter paper was used to separate the ZVI from withdrawal samples. A certain quantity of the clear solution (10 mL) was used for lead concentration determinations by Atomic Absorption Spectrophotometer (Sens AA, Austeria). Steps of experimental procedure adopted in the batch mode experiments are illustrated in **Fig.2.**

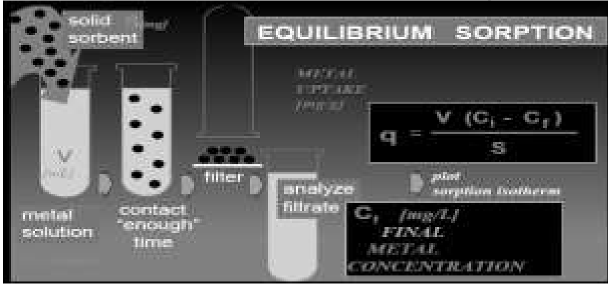

Fig.2 Steps of experimental procedure adopted in the batch mode experiments [17].

3. Results and discussion

3.1 ZVI weight

The amount of ZVI was changed from 0.05 to 2 g using 100 mL of metal solution for investigating the effect of sorbent dosage on the sorption of lead ions at 25ºC. These batch experiments were conducted with contact time of 60 min, initial pH of aqueous solution equal to 3 and agitation speed of 250 rpm. **Fig.3** shows that the removal efficiency of Pb^{+2} was improved with increasing the sorbent dosage from 0.05 g to 0.8g at a fixed initial metal concentration of 50 mg/L. This was expected due to the fact that the higher dose of sorbents in the solution leads to the greater availability of sorption sites. This means that the more ZVI particles are able to provide more iron surface-active sites for collision with metal molecules to accelerate the metal removal efficiencies **[12].** This also suggests that after a certain dose of sorbent, the maximum sorption sets in and hence the amount of Pb^{+2} bound to the sorbent and the amount of this metal in solution remains constant even with further addition of the dose of sorbent. Accordingly, the value of 0.8g/ 100 mL was chosen as a best dosage of ZVI for further batch experiments.

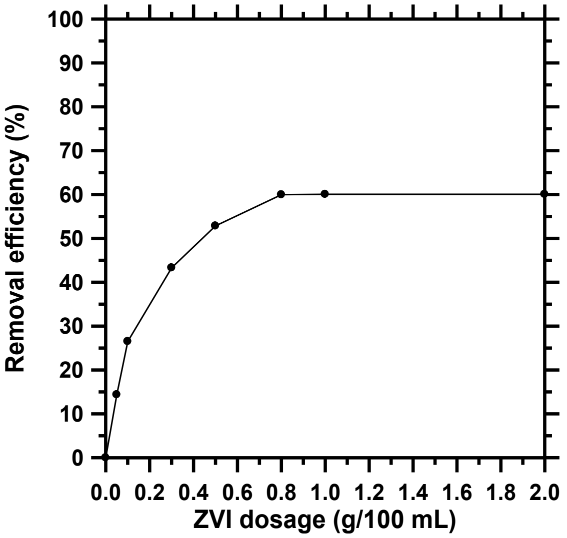

Fig.3 Removal efficiency of lead onto ZVI as a function of dosage (pH=3; C_o=50 mg/L; contact time=60 min; agitation speed=250 rpm; T=25ºC).

3.2 Initial Concentration

The ratio between the metal concentration removed from the contaminated water and the initial concentration is defined the "removal efficiency". The batch experiments in this step were implemented with using the concentration values ranged from 50 to 250 mg/L while other condition used as follows; contact time of 60 min, pH of contaminated water equal to 3, dosage of ZVI equal to 0.8g/ 100 mL and shaking speed of 250 rpm. It is clear from **Fig.4** that the concentration has significant influence on the lead removal efficiency using ZVI as sorbent where this removal was changed from 60% to 18% with changing the initial concentration from 50 to 250 mg/L. The increase of pollutant concentration can produce less favorable active sites and, consequently, reduction in removed pollutant can be recognized **[14]**.

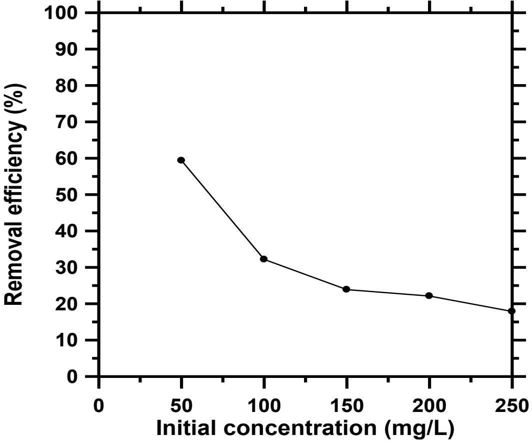

Fig.4: Removal efficiency of lead onto ZVI as a function of initial metal concentration (pH=3; contact time=60 min; ZVI dosage=0.8 g/100mL; agitation speed=250 rpm; T=25ºC).

3.3 Agitation Speed

To find the variation between lead removal percentage and agitation speed, the speed was changed from zero to 250 rpm with using best conditions specified from previous experiments (i.e. C_o=50 mg/L and ZVI dosage=0.8 g/100mL) at time and pH of 60 min and 3 respectively. Approximately ten percent of the lead was eliminated with agitation speed of zero and the removal efficiency was increased gradually until reached the value of 60% at 250 rpm as illustrated in **Fig.5**. It is clear that the speed of 250 rpm is sufficient for ensuring the proper contact between active sites of sorbent and lead ions in the aqueous solution. The gradually increase of the lead removal efficiency may be resulted from improving the diffusion of ions through the sorbent in response to increase of agitation speed where agitation process will enhance the mass transfer of ions towards the sorbent.

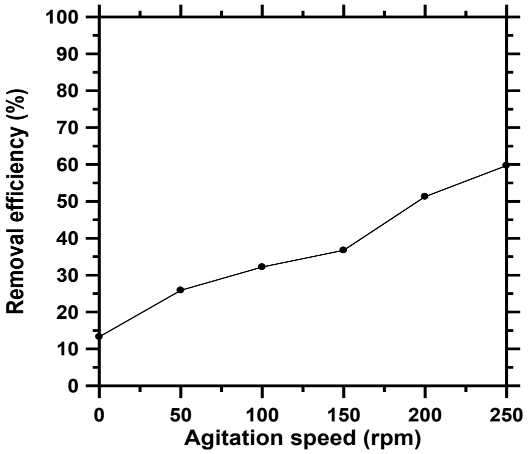

Fig.5: Removal efficiency of lead onto ZVI as a function of agitation speed (pH=3; C_o=50 mg/L; contact time=60 min; ZVI dosage=0.8 g/100mL; T=25ºC).

3.4 Equilibrium Time and Initial pH

The time in the batch experiments must be specified at certain value which is achieved the equilibrium state for partitioning. The influence of elapsed time on the lead removal efficiency at different values of initial pH ranged from 2 to 7 with added 0.8 g of ZVI to 100 mL of contaminated water in the room temperature (25ºC) is plotted in **Fig.6**. It is clear that there is a significant increase in the removal efficiency as a result of increasing of contact time where a rapid increase was recognized in the sorption rate for the initial stages. Then, the rate was gradually stabilized and this may be attributed to occupy all active sites of ZVI sorbent. The results signified that the achieved maximum efficiency was 99% at equilibrium time of 60 min and pH of 5 for the aqueous solution where these values are represented the best conditions for sorption of lead ions onto ZVI reactive material.

The sorption processes between solid and liquid are influenced by the pH of the aqueous solution which is considered a most important parameter for interaction of water with ZVI as mentioned and certified by many studies such as **[13]**. The pH_{pzc} is the pH at point of zero charge which means that the positive charges generated by protonation equal to negative charges caused by deprotonation on the iron surface and this parameter is very important in the specified the affinity of sorption process. Hydrogen ions (protons) at pH less than pH_{pzc} are sorbed on the functional groups of the ZVI surface and this will generate a net positive charge that inhibits the sorption of cations. On the other hand, net negative charge on the iron surface due to deprotonize of oxygen atoms for pH greater than pH_{pzc} will enhance the sorption of cations [13].

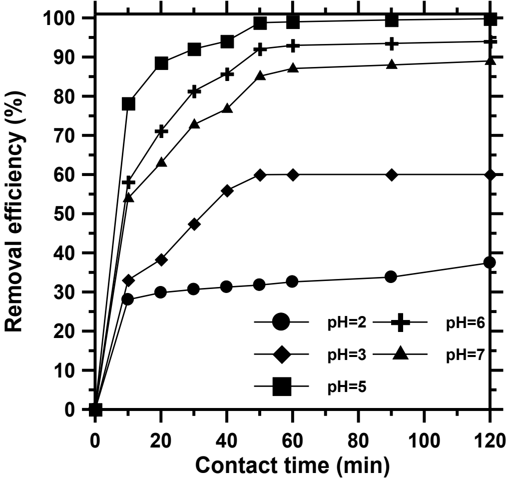

Fig.6: Removal efficiency of lead onto ZVI as a function of contact time and initial pH of aqueous solution(Co=50mg/L;ZVIdosage=0.8g/100mL;agitation speed= 250 rpm; T=25ºC).

3.5 Sorption Equilibrium in Metal Solution

Mathematical models describe the partitioning process of contaminant between the water and solid phases at certain temperature are called "sorption isotherms". These isotherms are depended on a set of assumptions related to the homogeneity /heterogeneity of reactive material, interaction between the contaminants and the type of coverage. These isotherms relate metal uptake per unit mass of sorbent (q_e) to the equilibrium sorbate concentration in the bulk phase (C_e) **[5]**. Sorption results of lead onto ZVI reactive material are fitted with two models namely Langmuir and Freundlich **(Eqs.1 and 2)** respectively for each value of initial pH of aqueous solution. **Figs.7 and 8** showed that there is a good agreement between the experimental data-set (symbols) and predicted values (lines).The constants for isotherm models **(Table 3)** were estimated by linearization using the GRAPHER Version 1.09 – 1993. This table showed that the best correlation was represented by the Langmuir isotherm model in comparison with Freundlich isotherm model for sorption of lead on the ZVI.

$$q_e = \frac{q_m b C_e}{1+bC_e} \qquad (1)$$

$$q_e = K_F C_e^{1/n} \qquad (2)$$

where q_e is the sorbed quantity of contaminant per unit mass of the sorbent (mg/g), q_m is the maximum sorption capacity of the sorbent (mg/g), b is the affinity constant (L/mg), K_F is the Freundlich sorption coefficient ((mg/g)(L/mg)$^{1/n}$), n is a constant indicative of the intensity of the adsorption and C_e is the contaminant concentration remaining in the solution at equilibrium (mg/L).

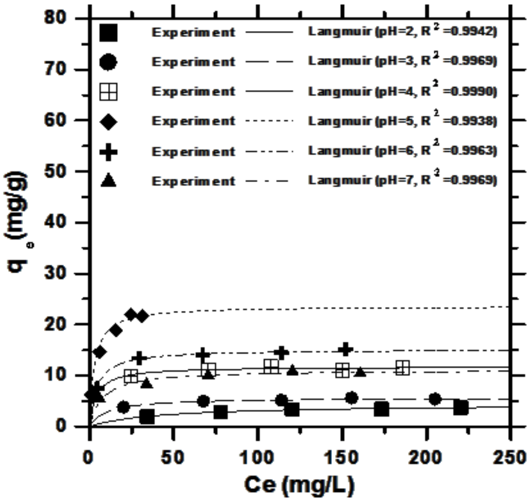

Fig.7. Comparison of the experimental results with the q_e values obtained by Langmuir isotherm model for lead sorption onto ZVI.

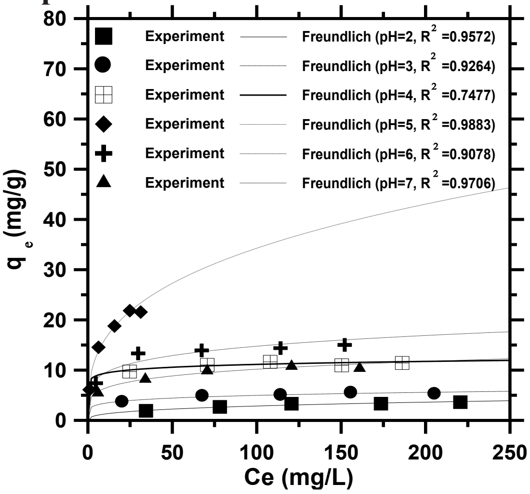

Fig 8.Comparison of the experimental results with the q_e valus obtained by Freundlich isotherm model for lead sorotion onto ZVI.

Table 3. Sorption isotherm constants with coefficients of determination for lead onto ZVI.

Model	Parameter	pH					
		2	3	4	5	6	7
Langmuir	q_m	4.3612	5.6557	11.756	23.6233	15.125	11.1223
	b	0.0241	0.0963	0.251	0.3566	0.235	0.1289
	R^2	0.9942	0.9969	0.9990	0.9938	0.9963	0.9969
Freundlich	K_F	0.6306	2.3148	7.8713	7.5292	6.0097	3.9845
	N	0.3345	0.1657	0.0753	0.3291	0.195	0.2036
	R^2	0.9572	0.9264	0.7477	0.9883	0.9078	0.9706

3.6 Modeling the pH Dependent Sorption Effects Using Modified Langmuir Isotherm

It is clear from previous section that the magnitudes of b and q_m are changed with pH of the influent water. The fitted values for two constants were calculated from linearized form of Langmuir model **(Table 3)** and plotted against the pH as illustrated in **Fig. 9** for understanding the trends of q_m-pH and b-pH relationship. The values of q_m were fitted with the polynomial regression while linear relationship was used for representing the b values. It is clear from these relationships that the increase of the pH was corresponded with the increasing of these constants until pH equal to 5. However, the constants were decreased after this value of pH with similar trends and types of fitted relationships. The variation of initial pH can be affected on the surface structure of the sorbent due to the protonation and deprotonation and, consequently, this may be reflected on the interaction between the sorbent and pollutants and the formation of metal hydroxides **[1]**. In this direction, **Fig.9** signifies that the affinity of the pollutant for surface of sorbent, i.e. b, is decreased in response to increase or decrease of pH from the value of 5. Competition between the cations and protons for the binding sites at low values of pH may be the reason for the reduction of affinity. However, the binding sites may not be activated at higher values of pH and this is explained the reduction of affinity for high pH **[8]**. The relationships related between the values of (q_m or b) and pH of influent water are listed in **Fig.9** which are fitted with **Eqs.3 and 4**. Accordingly, the relationships represented the value of retardation factor (R) can be written by substituting the equations in the **Fig.9** into **Eq.5** and the resulting equations will be showed in the **Eqs.6 and 7**:

$$q_m = \alpha_1 + \alpha_2\, pH + \alpha_3 pH^2 \qquad (3)$$

$$b = \alpha_4\, pH + \alpha_5 \qquad (4)$$

$$R = 1 + \frac{\rho_b}{n}\left(\frac{(\alpha_1+\alpha_2\, pH+\alpha_3 pH^2)(\alpha_4\, pH+\alpha_5)}{(1+(\alpha_4\, pH+\alpha_5)C)^2}\right) \qquad (5)$$

$$R = 1 + \frac{\rho_b}{n}\left(\frac{(18.0639 - 12.1137\, pH+2.6432\, pH^2)(0.115204\, pH- 0.221191)}{(1+(0.115204\, pH- 0.221191)C)^2}\right) \quad \text{pH} \leq 5 \qquad (6)$$

$$R = 1 + \frac{\rho_b}{n}\left(\frac{(133.549 - 33.2241\, pH+2.2478\, pH^2)(-0.113896\, pH+ 0.923512)}{(1+(-0.113896\, pH+ 0.923512)C)^2}\right) \quad \text{pH} > 5 \qquad (7)$$

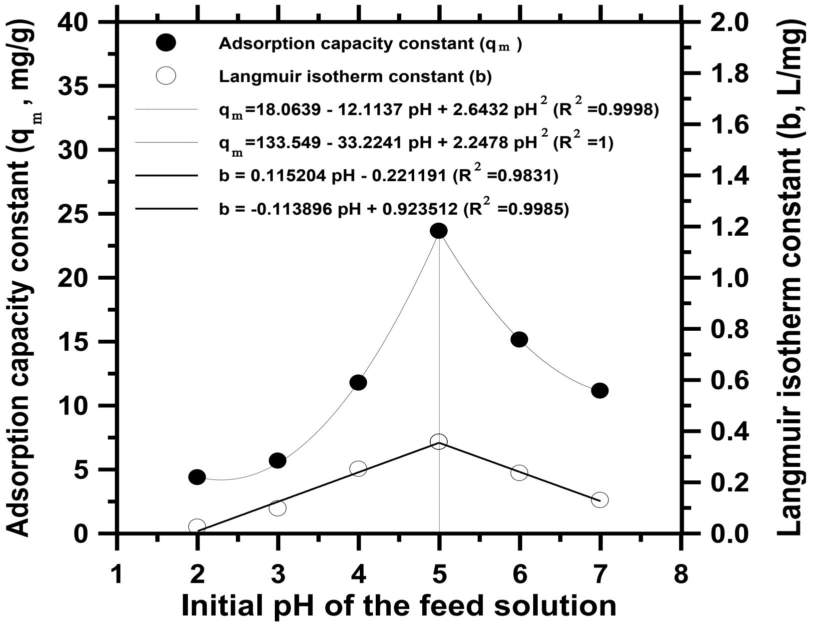

Fig.9: Variation of q_m and b calculated using Langmuir isotherm with initial pH for lead sorption onto ZVI sorbent.

3.7 Kinetics and Stoichiometry of Metal Uptake

Fig.10 is depicted the relationships between lead normalized concentrations and contact time where these curves are measured from kinetic experiments under controlled pH of aqueous solution. The results of these experiments are satisfied with pseudo first order rate law;

$$\frac{dC_t}{dt} = -k_{obs}C_t \qquad (8)$$

Integration of this equation and incorporation of C_o as the initial concentration gives:

$$C_t = C_o e^{-k_{obs}t} \qquad (9)$$

where C_t are metal ion concentration at time t, k_{obs} is the observed kinetic rate constant, and t is time. By fitting the concentration data in the previous figure to the rate equation where the number of correlated data ranged from 3 to 6, observed kinetic rate constants were calculated as explained in **Table 4** and used to interpret the experimental results. Accordingly, these constants show that lead uptake is changed dramatically with the initial pH of the treated water. It is clear that the changed pH from 2 to 5 will increase the lead uptake approximately threefold, while the changed pH from 5 to 7 will decrease the lead uptake approximately to its half value.

Fig.11 shows the relationship between the cumulative iron elution and cumulative lead removal as a function of contact time. Stoichiometric ratio (i.e. the dissolved iron divided by the corresponding removed of lead, $\Delta Fe^{+2}/\ \Delta Pb^{+2}$) as a function of time is illustrated in the **Table 5**. Depended on the measured values of $\Delta Fe^{+2}/\Delta Pb^{+2}$, every 1 mg of lead was removed by consuming 0.34 mg of iron. This value is consistent with one reported by **[13]** for same metal and reactive material equal to 0.3 mg/mg.

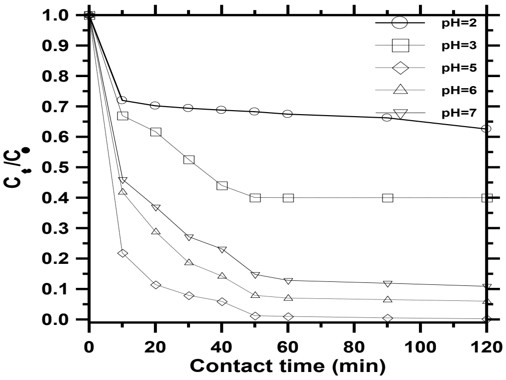

Fig10:Variation of normalized lead concentration with contact time (Co=50mg/l;ZVIdosage=0.8g/100ml agitation speed=250rpm;T=25 ºC)

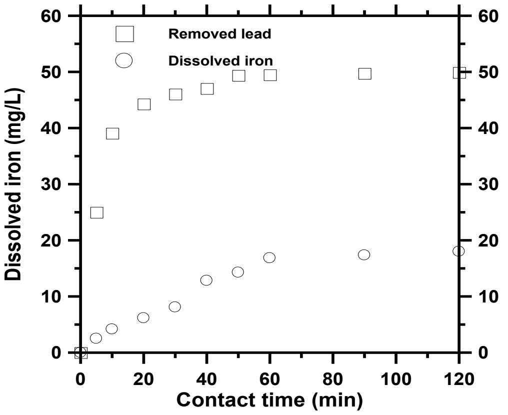

Fig11:Variation of dissolved iron and removed lead with contact time(pH=5;Co=50mg/L ZVI=0.8g/100ml;agitation speed=250rpm;T=25 ºC)

Table 4. Variation of observed kinetic rate constants with initial pH of aqueous solution for removal of lead onto ZVI reactive material.

pH	k_{obs} (per min)	R^2
2	0.262185	0.885621
3	0.0202739	0.975709
5	0.0851021	0.974974
6	0.0524592	0.987101
7	0.0400442	0.975987

Table 5. Stoichiometry ratios as a function of time for removal of lead onto ZVI reactive material.

Time (min)	5	10	20	30	40	50	60	90	120
$\Delta Fe^{+2}/\Delta Pb^{+2}$ (mg/mg)	0.10	0.11	0.14	0.18	0.27	0.29	0.34	0.35	0.36

4. Conclusions

1) Batch sorption experiments for removal of lead ions from aqueous solutions using ZVI sorbent at room temperature signified that the parameters influenced on sorption process are sorbent dosage, initial concentration, agitation speed, contact time and initial pH. These parameters have the values of 0.8g/ 100 mL, 50 mg/L, 250 rpm, 60 min, and 5 respectively to achieve the maximum removal efficiency of 99%.

2) Batch results proved that the sorption capacity and affinity coefficients are influenced by the variation of initial pH of aqueous solution. The coefficients have values of 23.62 mg/g and 0.3566 L/mg at best initial pH of 5. A significant reduction in these coefficients was recognized with an

increase or decrease with respect to the best value of the pH.

3) Langmuir isotherm model was more representative for experimental results in comparison with Freundlich model where coefficient of determination (R^2) not less than 0.9938. This model was modified by finding the relationships between sorption capacity and affinity constants as a function of initial pH.

4) Kinetic rate constants showed that lead uptake is changed dramatically with initial pH of the aqueous solution. This uptake was increased threefold when pH changed from 2 to 5, while this value decreased to its half when pH changed from 5 to 7.

5) Stoichiometric ratios of lead were found less than 1 based on relationship between cumulative iron elution and cumulative lead removal ($\Delta Fe^{+2}/\ \Delta Pb^{+2}$). The values of $\Delta Fe^{+2}/\ \Delta Pb^{+2}$ demonstrated that about 0.34 mg of iron is required to remove 1 mg of Pb^{+2} at best operating conditions specified in point 1.

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كلية الهندسة- جامعة بغداد/ العراق

خلاصة

نماذج الأيزوثيرم لفريندلج ولانكمير هي كثيرا ما تستخدم لنمذجة بيانات الامتصاص عند قيمة ثابتة من درجة الحموضة. تهدف الدراسة الحالية لتعديل نموذج الأيزوثيرم لانكمير ليتناسب مع آثار الامتصاص كدالة لدرجة الحموضة الأولية في المحاليل المائية. وقد تم تطوير هذا النموذج باستخدام نتائج الدفعة لامتصاص الرصاص من المحاليل الملوثة على الحديد صفر التكافؤ (ZVI)المازة تحت المعايير المحددة. وهذه المعايير كانت؛ درجة الحموضة الأولية من (2-7)، زمن التماس (≥120 دقيقة)، سرعة الاهتزاز (0-250 دورة في الدقيقة) ووزن الحديد صفر التكافؤ لا يتجاوز(2 غرام / 100 مل) لكل تراكيز الرصاص الأولي التي تتراوح بين (50-250 ملغم / لتر). أثبتت نتائج التجارب أن قدرة الامتصاص وتقارب الثوابت لنموذج الأيزوثيرم المطور يتغير بشكل كبير مع درجة الحموضة الأولية للمحلول المائي. هذه الثوابت تمتلك قيم من (23.62 ملغ / غرام) و(0.3566 لتر / ملغ) عند افضل درجة حموضة وهي 5. وبناء على ذلك، فأن قدرة الأمتصاص لدرجة الحموضة الأولية قد ارتبطت بأستخدام الأنحدار متعدد الحدود، بينما مثل معامل التقارب- درجة الحموضة الأولي بواسطة علاقة خطية.

المفاتيح :ازالة الرصاص، عمليات الامتزاز، حديد صفر التكافؤ، الحواجز النفاذة، معالجة المياه الجوفية