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Removal of Lead Ions from Waste Water Using Modified Jordanian Zeolite

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Author's contribution

The sole author designed, analysed, interpreted and prepared the manuscript.

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ABSTRACT

In this study a Jordanian Zeolite was modified using anionic surfactant which is sodium dodecyl sulfate (sodium dodecyl sulfate). The sorption of Pb(II) from synthetic wastewater by surfactant modified Zeolite (SMZ) was investigated as a function of temperature. The experimental data was analysed using isotherm models namely Langmuir, Freundlich, Redlich-Peterson and Temkin and kinetic models such as the pseudo- second-order, intraparticle diffusion and the Elovich models in order to understand the mechanism of the interaction between this SMZ and the lead ions. All the isotherm models showed good correlation with the experimental results but Freundlich was the best. The calculated ΔH was obtained using Langmuir constant (a_L), its value of 8.29kJ/mol revealed that the type of sorption is physical oneThe values of R_L at all temperatures reflect the favorability of this interaction. The calculated activation energy was 21.126 kJ/mol using the pseudo-second order constant (k_2), which indicates that the sorption is physisorption. The intraparticle diffusion model showed multilinearity which means multiple stages there occurred to achieve the removal of lead ions, the first linear curve is due to the boundary layer diffusion and the second linear curve isfor the intraparticle diffusion effect. The adsorption kinetics data fitted also Elovich model.

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1. INTRODUCTION

Water pollution represents a real danger to the environment and consequently to man. This pollution is primarily due to industrial wastes. Heavy metal water pollution is an area of major concern today in both developed and developing countries. It is therefore mandatory that their levels in drinking water, wastewater, and water used for agricultural and recreational purposes must be reduced to within the maximum allowable concentrations recommended by national and international health authorities such as the World Health Organization (WHO, 2004), the removal of heavy metal ion from aqueous solution is important in environmental concern because heavy metal can induce serious problems to human health through the water resources.

The concentration and the mobility of heavy metals in soils and sediments have been widely studied in the last decades. Although many heavy metals are necessary in small amounts for the normal development of the biological cycles, most of them become toxic at high concentrations. The presence of high levels of these metals in the environment may cause long-term health risks to humans and ecosystems. They occur naturally in soils, and they are widely used in industry, especially in electronics and the rapidly growing information technology sector. Common anthropogenic sources include agricultural activities, atmospheric deposition, road run off, discharges from industrial plants and sewage works, acidic mine effluents and building of reservoirs. Lead (PbPb) is one of the most toxic and easily encountered metals released into the environment through various industrial activities, consumer products, and waste disposal. Lead is used in the production of batteries, ammunition, sheet lead, solder, pipes, paints, medical equipment, ceramic glazes, and electronic products [1,2]. Therefore, so many projects focus on the removal of heavy metal ion to protect water resources. Numerous treatment techniques have been used for heavy metal ion removal from the aquatic environment such as adsorption, ion exchange, coagulation/flocculation, chemical precipitation, photodegradation, and electrochemical oxidation. Among these techniques, adsorption is one of the most common technologies for removing heavy metal ions. Although activated carbon and activated metal oxides are effective adsorbent for the removal of heavy metal ions [3,4-5].

Zeolites play an important role in the environment by acting as a natural scavenger of pollutants by taking up cations and anions either through ion exchange or adsorption or both. There is an increased interest in using surfactants to increase the efficiency of metal removal by treated Zeolite. Treatment of Zeolite with anionic surfactant to convert it to organo-Zeolite may improve the removal of metal ion from water. sodium dodecyl sulfate (SDS) is one of the anion surfactant that can be use to make complexation with metal ions.

In the present work, a method is proposed for wastewater treatment in Jordan for removing the lead ions using modified Jordanian Zeolite, the modification was done with anionic surfactant (SDS) by batch and column experiments. The well-characterized Zeolite modified with (SDS) was used for the sorption PbPbof $PbPb^{2+}$ ions at different temperatures values. To evaluate and to understand the affinity and the mechanism of interaction between metal ion and the adsorbent, different adsorption isotherm and kinetic models were applied on the experimental results [6].

Four different isotherm models were tested Langmuir, Freundlich, Redlich-Peterson and Temkin, Three types of kinetic models which are pseudo-second order, an intraparticle diffusion and Elovich were used. All of these models were used to study the adsorption equilibrium by fitting the experimental data with these isotherm models.

2. EXPERIMENTAL SECTION

2.1 Sampling of Sorbent

The Zeolite deposits samples were collected from Jebal Aritain in Jordan. These deposits samples were treated by washing them with deionized distilled water many times; the washed samples were air dried and stored for further use.

2.2 Modification of Zeolite

Sodium dodecyl sulfate (SDS) –Zeolite was prepared based on the cation exchange capacity (CEC), which was determined in the previous work [7] the CEC was 40.6 mg/g. SDS (analytical grade) was obtained from Sigma prepared in amount which is equivalent to the CEC. One hundred g of Zeolite was accurately weighed then added to 400 mL of SDS solution. This mixture was stirred using mechanical stirrer for 24 hour at 600 rpm and at 25°C. The surfactant modified Zeolite (SMZ) was obtained by filtration using 45 μm filter paper. The treated Zeolite was dried at 95°C for 24 hours then grinded and sieved to particle size less than 100 mesh [8] and stored in tight glass bottle.

2.3 Preparation of $PbPb^{2+}$ Solution

All the chemical reagents that are used in the preparation of solutions were analytical grade from BHD (Analar). A stock solution of lead ions (1000 mg/L) was prepared by dissolving an appropriate amount of this metal ion in 0.01 M of $NaClO_4$ to keep ionic strength constant. All the diluted lead ion solutions ranging from 5 to 35 mg/L adjusted to pH 6 using 0.1 M of NaOH and/ or HNO_3.

2.4 Equilibrium Batch Experiment

The isotherm batch experiments were carried out as follows: accurate 25 mL volume of metal ion solutions in the range of 5-35 mg/L were transferred to a series of 50 mL stoppered flasks, an accurate weight of 0.125 g of the SMZ was added to each flask. All the flasks containing mixture were shaken using shaker water bath at constant temperature until reaching the equilibrium. All the experiments were done at different temperatures different temperatures (25, 35, 45, and 55°C). The equilibrium attained after 8 hours (the equilibrium time determined experimentally). The SMZ was isolated from the solution by filtration using 45 μm Whatman filter paper. After filtration centrifugation is done with centrifugal force (g) of 2000.

2.5 Column Extraction Kinetics

The evaluation of extraction of $PbPb^{2+}$ ions using SMZ was investigated experimentally using glass column reactor. $PbPb^{2+}$ solutions of 10.0 ppm were prepared under the conditions described in sections 2.3 and 2.4. The glass reactor (20 cm ×1.4 cm id) was plugged with glass wool to support the modified Zeolite bed and was filled with 2.0000 gram modified SMZ, the solid extractant was saturated with 0.01 M of $NaClO_4$, the solution of $PbPb^{2+}$ ions was placed at the top filled reactor, the obtained solution was collected at the end of the desired time interval with flow rate of 1.3 mL/min under gravity force. The collection eluates were done at controlled pH of 5. The eluates were collected at different time intervals of 5, 10, 15, 20, …….1,440 minutes and measured using CV 797 VA Computrance (Metrohm). The column experiments were done at different temperatures (25, 35, 45, and 55°C). All the experimental aquarium concentrations for equilibrium isotherm and kinetic adsorption models were determined using CV 797 VA Computrance (Metrohm).

3. RESULTS AND DISCUSSION

3.1 Adsorption Isotherms Analysis for Removal of $PbPb^{+2}$ by SMZ

To describe the adsorption behavior, such as mechanism, affinity and thermodynamic concepts from the experimental data, the equilibrium isotherm equations which belong to Langmuir, Freundlich, Redlich-Peterson and Temkin are used.

3.1.1 Langmuir adsorption isotherm model

The Langmuir isotherm model was chosen for the estimation of maximum adsorption capacity corresponding to complete monolayer coverage of the Zeolite surface, it is used for homogeneous sites with monolayer adsorption surface, the maximum adsorption capacity can be detected which is related to complete monolayer coverage on the SMZ surface. The linearized Langmuir expression equation is

$$\frac{C_e}{q_e} = \frac{1}{q_{\max} a_L} + \frac{C_e}{q_{\max}} \qquad (1)$$

where q_e is the amount adsorbed on solid at equilibrium time (mg/g), Ce is the equilibrium liquid concentration (mg/L), q_{max} is the monolayer adsorption capacity or adsorption maximum (mg/g), and a_L is constant related to the adsorption intensity or Langmuir coefficient (L/mg). The amount of removal at time t and at equilibrium q_t and q_e respectively is calculated using the following equation

$$q_t = \frac{C_o - C_t}{w} \times v \qquad (2)$$

Where: C_o and C_e are the initial and equilibrium concentration respectively, w is the weight of SMZ and v is the total volume of solution. All the experiments are performed in triplicate and the average is used for calculation.

The Langmuir isotherm results for removal of lead ions by SMZ were shown in Table 1 and illustrated in Fig. 1 at different temperatures. All data were fitted using the linearized equation for Langmuir isotherm, high correlation coefficients were obtained using SMZ.

It was found that the values of q_{max} and a_L increased by increasing temperature. The separation factor (R_L) which can characterized the Langmuir isotherm [9] is calculated using the following equation

$$R_L = \frac{1}{1 + a_L C_O} \tag{3}$$

The value of R_L indicates the shape of the isotherms to be either unfavorable ($R_L > 1$), linear ($R_L = 1$), favorable ($0 < R_L < 1$) or irreversible ($R_L = 0$). It is noticed from Table 1 that the values of R_L at all temperatures are less than 1 which means this isotherm is favorable one.

By calculating the specific surface area (S) [9], it was found that the value of S increased by increasing the temperature as shown in Table 1, this is may be due to the increasing of the capacity of the monolayer of the adsorbent.

3.1.2 Freundlich adsorption isotherm model

Freundlich isotherm based on the removal of adsorbate on heterogeneous surface of adsorbent, the sorption will be non-ideal with assumption of multilayer sorption which means that the distribution of sorption heat is not uniform. The Freundlich linearized equation is expressed as:

$$\ln q_e = \ln K_f + \frac{1}{n} \ln C_e \tag{4}$$

The slope and intercept for this linear equation are K_f (mg/g (L/mg)$^{1/n}$) and 1/n respectively (the Freundlich constants). Where K_f represents the capacity of adsorbent, the favorability of this adsorption model is known from the value of *n* which expresses the adsorption intensity. As we can see from the results in Table 1 and illustrated in Fig. [2] that the plot of ln qe vs ln Ce gives straight lines with high correlation coefficients at all temperature values. This adsorption is favorable because the n values are higher than unity, the adsorption is physical in nature. Also the values of K_f increases by increasing the temperature, indicating that the capacity of sorption increases by increasing the temperature and the sorption is an endothermic process [10].

3.1.3 Redlich–peterson isotherm

In Redlich–Peterson isotherm, the equation includes three adjustable parameters. This isotherm model makes combination of both Langmuir and Freundlich isotherm models. The linearized form of this model is expressed by this equation:

$$\ln((K_R C_e / q_e) - 1) = \beta \ln(Ce) + \ln(\alpha) \tag{5}$$

The three parameters K_R, β and α are obtained from the above pseudo-linearized equation using trial- and -error to get the highest value of the R^2. Where K_R is (L g^{-1}), α is (L mg^{-1}) and values of α lies in this range $0 < \alpha < 1$. A comparison between experimental data and theoretical data was done using solver of Excel program. From Table 1 and Fig. 3 it is found that the K_R increases by increasing the temperature and β has 1.000 value at all temperatures while α between 0 and 1.

When α constant approaches zero then this model will tend to Freundlich model and when α approaches 1then this model will tend to Langmuir model. Rdlich-Petrson showed that the mechanism of removal was not exactly followed nor Langmuir neither Freundlich, it considered to be a hybrid case that is neither following monolayer nor following multilayer adsorption [10,11].

3.1.4 Temkin adsorption isotherm model

This adsorption model deals with the interaction between adsorbent and adsorbate whatever be the concentration value is whether it is high or low. The heat of adsorption is calculated from this model as a function of temperature, it assumes that the heat of adsorption of molecules in the layer will decrease with the coverage. The linearized equation of Temkin adsorption isotherm is expressed as:

$$q_e = \frac{RT}{b_T} \ln A_T + \frac{RT}{b_T} \ln C_e \tag{6}$$

Table 1. Isotherm parameters obtained using the linear method for removal of ions from aqueous solutions using modified zeolite at different temperatures

	Temp (°C)	Langmuir				Freundlich			Redlich-Peterson				Temkin			
		q_{max} (mg g^{-1})	a_L (L mg^{-1})	R_L	R^2	K_F (L g^{-1})	n	R^2	K_R	α	β	R^2	A_T	b_T	B	R^2
Pb^{-2}	25	70.922	0.371	0.212	0.999	18.298	1.281	0.994	31.514	0.621	1.000	0.991	6.179	216.775	11.435	0.963
	35	76.9231	0.396	0.201	0.994	21.115	1.207	0.9964	34.533	0.615	1.000	0.990	10.653	329.322	9.690	0.9105
	45	80.6452	0.316	0.241	0.9780	24.415	1.719	0.9968	38.431	0.409	1.000	0.996	10.110	240.704	10.989	0.948
	55	106.383	0.500	0.167	0.9506	37.319	1.183	0.9998	46.550	0.148	1.000	0.961	13.720	216.510	12.601	0.979

Table 2. Pseudo-second order model for removal of lead ions from aqueous solutions using modified zeolite at different temperatures

$q_{e,exp}$	$q_{e,theo}$	$K_2 \times 10^{-3}$	R^2	%SSE
25°C				
6.775	6.817	2.700	0.9895	0.7095
35°C				
8.620	8.425	3.200	0.9888	0.5797
45°C				
8.643	8.137	4.970	0.991	0.3171
55°C				
9.250	9.294	5.540	0.9996	0.1596

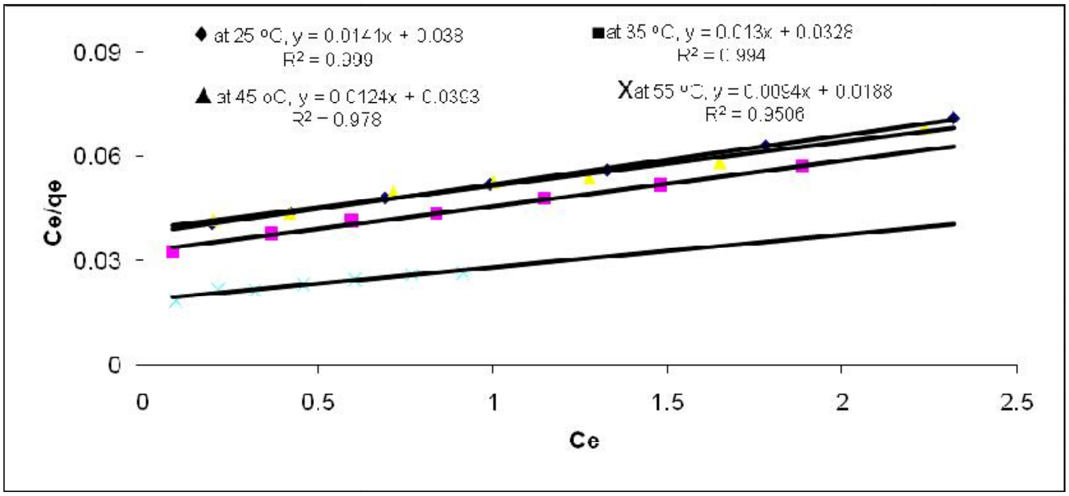

Fig. 1. Langmuir isotherm model for the removal of lead ions from aqueous solutions using modified zeolite at different temperatures (Conditions: pH = 6, particle size < 100 mesh, adsorbent dose 0.125 g)

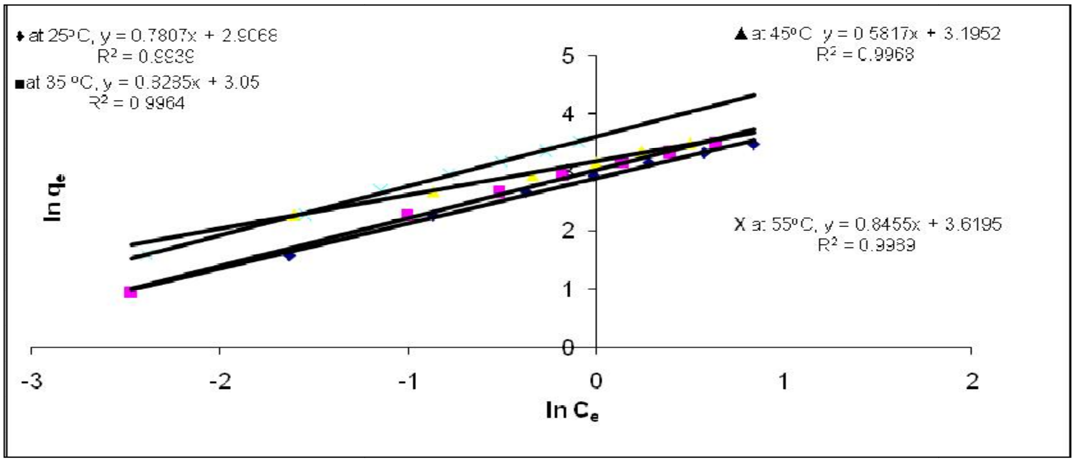

Fig. 2. Frendluich isotherm model for the removal of lead ions from aqueous solutions using modified zeolite at different temperatures (Conditions: pH = 6, particle size < 100 mesh, adsorbent dose 0.125 g)

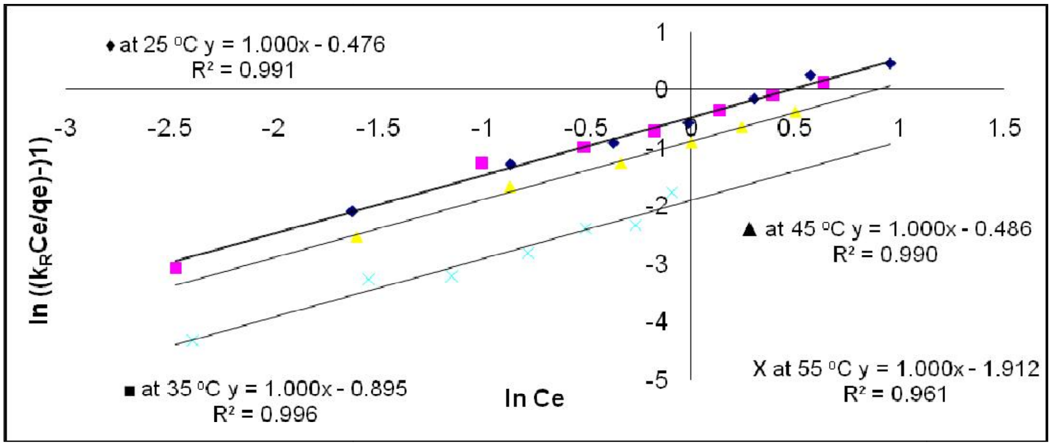

Fig. 3. Redlich-Peterson isotherm model for the removal of lead ions from aqueous solutions using modified zeolite at various temperatures (Conditions: pH = 6, particle size < 100 mesh, adsorbent dose 0.125 g)

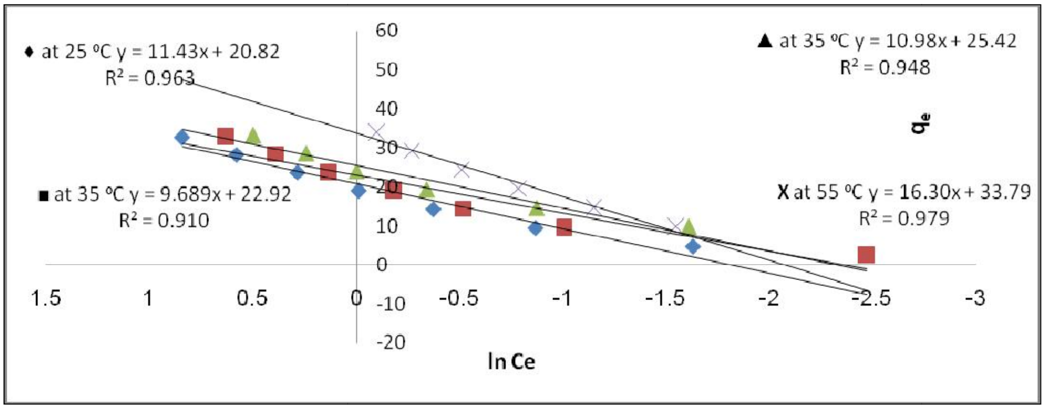

Fig. 4. Temkin isotherm model for the removal of lead ions from aqueous solutions using modified zeolite at different temperatures (Conditions: pH =6, particle size < 100 mesh, adsorbent dose 0.125 g)

Where A_T is the Temkin isotherm equilibrium binding constant (L/g), b_T is the Temkin isotherm constant related to the adsorption potential of the adsorbent (J/mol), B is constant related to heat of sorption and can be calculated from this equation B= $\frac{RT}{b_T}$, R is the universal gas constant (8.314J/mol*K), and T is the Temperature at 298K. These constants are determined from the slope and intercept.

The estimated values of B obtained from Fig. 4, as shown in Table 1 are less than 40 kJmol^{-1} that means the adsorption of lead ions is physical one [12].

3.1.5 Thermodynamic parameters

The enthalpy ΔH, entropy ΔS and the free energy ΔG can be calculated using Langmuir isotherm constant a_L by applying equations (7) and (8)

$$\ln(a_L) = \frac{\Delta S^o}{R} - \frac{\Delta H^O}{RT} \tag{7}$$

$$\Delta G^o = -RT \ln a_L \tag{8}$$

Where R is the universal gas constant (8.314 J/mol K). The thermodynamic parameters were estimated in the range of temperature from 25°C to 55°C. From the experimental results of the Langmuir's constants which are shown in Table 1, ΔH, ΔG and ΔS were calculated, by drawing ln(a_L) verses 1/T using slope and intercept, the values were 8.29 kJmol^{-1}, 2.50 kJ/mol and 19.42 J mol^{-1}respectively. ΔH has positive value which means the process is endothermic [13,14], and the classification of adsorption tend to be physical because the calculated value of ΔH is <84 kJ mol^{-1}, the value of the free energy is also positive but the value is so small, the process is almost spontaneous with positive entropy, the value of the ΔS indicates that when the lead ions interact with the solid surface of the SMZ the randomness increases.

3.1.6 Kinetic models

To analyze the removal kinetics of lead ions by the SMZ pseudo second-order, Intra-particle diffusion and Elovich kinetic models were applied to the experimental data.

3.1.7 Pseudo second-order

The pseudo second -order model was based on the assumption that the rate limiting step might be chemisorption caused by the sharing of the valence electrons between the adsorbent and the adsorbate [11,15].

The experimental data was found to be fitted to the pseudo second-order rather than the pseudo first-order R^2 values for pseudo second-order at all temperature values are high, the K_2 and the q_e were calculated from the slope and intercepts of the linear curve after applying the linearized equation of this model,

$$\frac{t}{q_t} = \frac{1}{k_2 q_e{}^2} + \left(\frac{1}{q_e}\right)t \qquad (9)$$

The values of K_2 and the q_e increased by increasing the temperature as shown from Fig. [5] and Table 2, this process is an endothermic with high correlation coefficient. the straight lines for this model showed a good fit for the experimental data.

3.1.7.1 Validation test

To determine the validity and the applicability of pseudo second- order kinetic model the sum of error squares (SSE %) is calculated using the following equation:-

$$\Delta q\% = S.D = \sqrt{\sum \frac{\left[(q_{e,\exp} - q_{e,cal} / q_{e,\exp})\right]^2}{n-1}} \qquad (10)$$

The calculated $q_{e,cal}$ were found close the experimental $q_{e,exp}$ at all temperature Table 2, these results indicate that this model fits well to describe the adsorption kinetics and the chemisorptions may be the rate-controlling step [16-18].

3.1.7.2 Activation energy

The activation energy was calculated using Arrhenius equation which is

$$\ln k_2 = -(Ea/RT) + \ln A \qquad (11)$$

Where E_a is the activation energy in (kJ/mol), k_2 is the second rate constant for removal (g mg^{-1}s^{-1}), R is the gas constant, T is temperature (K) and *A* is the Arrhenius factor. Determination of the activation energy was achieved by plotting ln (k2) versus 1/T, from the slope of this plot, the E_a was 21.126 kJ mol^{-1}, if the activation energy is in the range of 5 to 40 kJ/mol then the process is physisorption while it will be chemisorption if the activation energy is in the range of (40–800 kJ mol^{-1}) which is higher than the physisorption [17, 19]. Based on the the experimental activation energy our process is physisorption.

3.1.8 Intra-particle diffusion model

The rate of the movement of the lead ions from the bulk solution towards the solid surface of the modified-Zeolite which is the solid phase can be described by Intraparticle diffusion model, this model is applied onto the porous adsorbent. The following equation described this model is:-

$$q_t = k_{id}(t)^{0.5} \qquad (12)$$

Where, k_{id} is the intraparticle diffusion rate constant (mg/g min$^{1/2}$) and can be obtained from the slope of qt versus $t^{0.5}$. If the intraparticle diffusion is the rate determining step then the plot will pass through the origin according the experimental results the plots at temperatures (shown in Table 3) did not pass through the origin which means that this diffusion is not the only controlling step, the boundary layer diffusion somewhat controls the process of removal.

Also multilinearity (two linear curves) was observed for all plots as illustrated in Fig. [6], indicating that more than one steps are involved in the removal of lead ions, the first linear curve is due to the boundary layer diffusion and the second one is because of the intraparticle diffusion effect [18].

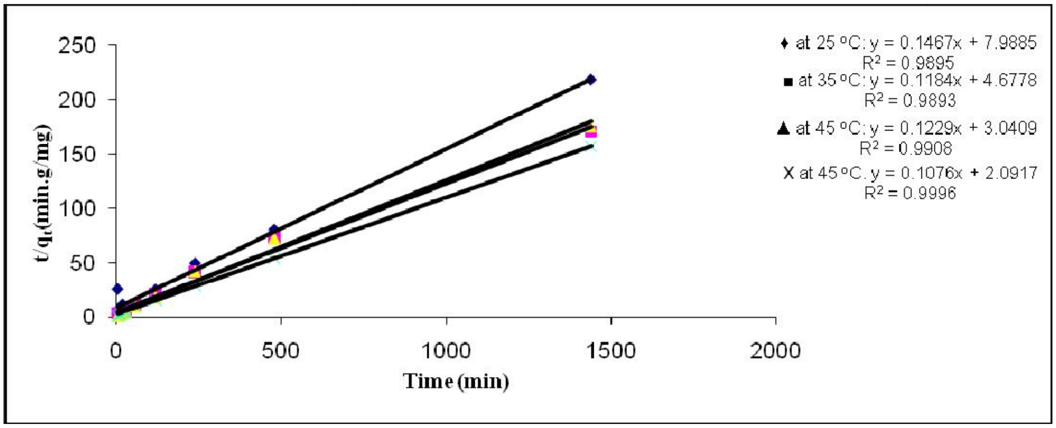

Fig. 5. Pseudo-second order model for the removal of lead ions from aqueous solutions using modified zeolite at different temperatures (Conditions: pH = 6, particle size less than 100 mesh, adsorbent dose 0.125 g)

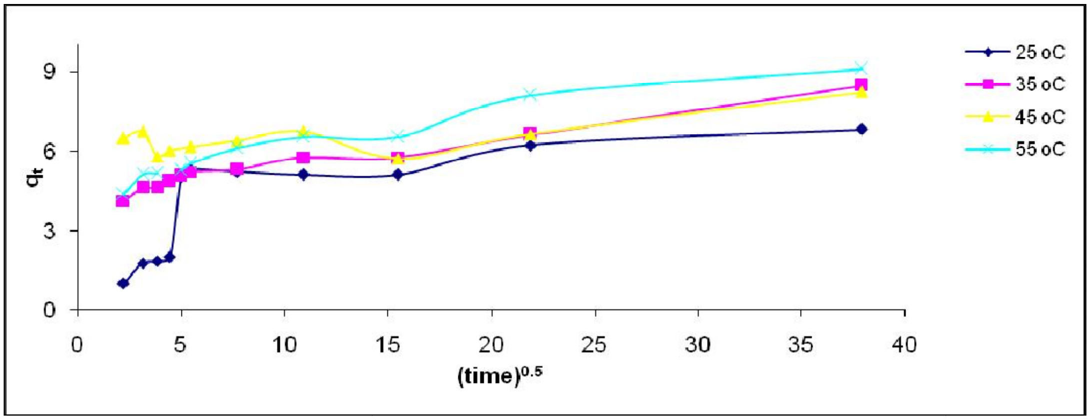

Fig. 6. Intra-particle diffusion model for the removal of lead ions from aqueous solutions using modified zeolite at different temperatures (Conditions: pH = 6, particle size < 100 mesh, adsorbent dose 0.125 g)

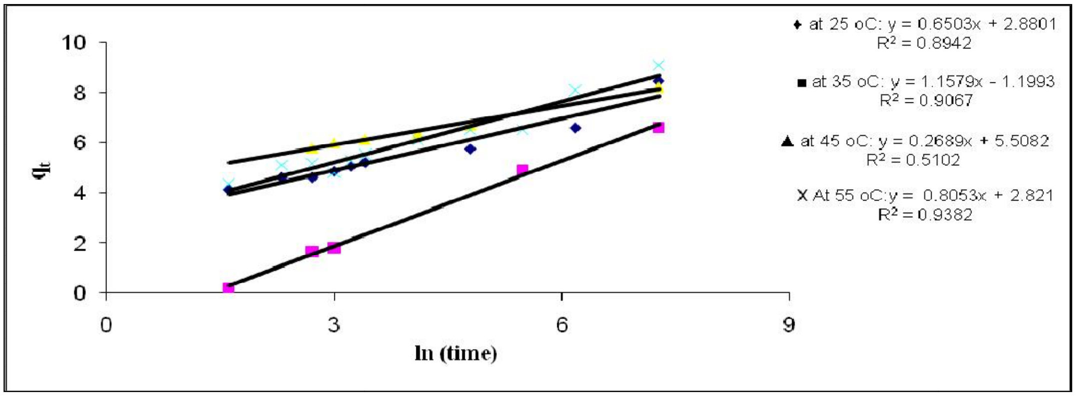

Fig. 7. Elovich kinetic model for the removal of lead ion from aqueous solutions using modified clay with sodium dodecyl sulfate at various temperatures. Conditions: pH = 5, particle size less than 100 mesh, adsorbent dose 0.125 g

Table 3. Intra-particle diffusion model for removal of lead ions from aqueous solutions using modified zeolite at different temperatures

25°C						35°C						45°C						55°C					
k_{id1} mg g - 1min- 0.5	k_{id2}	C_1	C_2	R_1^2	R_2^2	k_{id1}	k_{id2}	C_1	C_2	R_1^2	R^2_2	k_{id1}	k_{id2}	C_1	C_2	R^2_1	R^2_2	K_{id1}	k_{id2}	C_1	C_2	R^2_1	R^2_2
0.318	0.104	3.455	4.420	0.960	0.977	0.135	0.071	4.094	4.554	0.923	0.996	0.244	0.094	3.923	4.570	0.962	0.979	0.308	0.109	0.308	5.170	0.740	5.170

Table 4. Elovich kinetic model for the removal of lead ions from aqueous solutions using modified zeolite at different temperatures Table 4

Temperatures (°C)											
25			35			45			55		
β	δ	R^2	β	δ	R^2	β	δ	R^2	β	δ	R^2
1.538	54.657	0.941	2.114	106.658	0.929	1.634	107.949	0.926	1.242	26.749	0.9382

3.1.9 Elovich kinetic model

The Elovich equation describes predominantly chemical adsorption on highly heterogeneous adsorbents, but the equation does not propose any definite mechanism for adsorbate-adsorbent interaction [14,15].

Elovich model equation is given as:

$$q_t = 1/\beta \ln(\delta \beta) + 1/\beta \ln t \quad (13)$$

Where δ is the initial adsorption rate (mg g^{-1} min^{-1}) and β is desorption constant (g mg^{-1}). The Elovich constants were obtained from the linearized plots (Fig. 7) and shown in Table 4. The initial rate of adsorption which is described by δ increases as the temperature increases in the range of 25-45°C and better adsorption mechanism is observed, but δ decreases at 55°C. β values ranged from 1.538 to 1.242, the increasing of temperature is not really affecting the value of β which represents surface coverage [19].

4 CONCLUSIONS

This work showed that we can use the modified Jordanian Zeolite (SMZ) as low cost adsorbent to remove lead ions from waste water. This study for lead ion removal was conducted at constant pH of 5, the removal was investigated at different temperatures (25-65°C) the analysis of results was done using four isotherm models namely Langmuir, Freundlich, Redlich-Peterson and Temkin. Freundlich isotherm is found to best fit the experimental data as indicated by the highest correlation coefficient. The enthalpy was calculated using Langmuir constant, the process is found to be is an endothermic as ∆H has positive value, and the adsorption nature is physical one. The kinetic models were used to investigate the adsorption mechanism; the removal data followed the pseudo-second-order kinetic model. The activation energy (Ea) was obtained from the plotting of Ln k_2 versus 1/T, the value of Ea showed that the process is physisorption. The intra-particle diffusion showed that the removal occurred in two steps, the first is due to the boundary layer diffusion and the second is for to the intraparticle diffusion effect. The straight line did not pass through the origin which mean the diffusion is not the only controlling step. The lead ion removal has two resistances; the first one is due to the boundary layer diffusion and the second one is due to the intraparticle diffusion. The straight line did not pass through the origin which means that the diffusion is not the only controlling step.

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COMPETING INTERESTS

Author has declared that no competing interests exist.

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