



Removing Thallium (I) Ion from Aqueous Solutions Using Modified ZnO Nanopowder

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ABSTRACT

In this study, the adsorption of thallium (I) ion as a dangerous pollutant from aqueous solution onto modified ZnO nanopowder as a fairly cheap adsorbent has been examined in batch mode. It was known that modification of the adsorbent was necessary to reach a significant adsorption percentage. The adsorbent used here was modified by sodium phosphate solution. The effect of experimental conditions such as initial pH of solution, contact time, adsorbent dosage, initial concentration of thallium and temperature is studied. The results showed the dependence of the adsorption percentage to these conditions specially its pH. The maximum adsorption percentage of Tl (I) ions at $25\pm 1^\circ\text{C}$ was 92.8%. Freundlich isotherm model provided a better fit with the experimental data than Langmuir and Temkin isotherm models by high correlation. Separation factor, R_L , values showed that modified ZnO nanopowder was favorable for the adsorption of Tl (I) ion. The negative value of ΔH^0 showed that Tl (I) sorption is an exothermic process and the negative value of ΔS^0 represented that there is a little decrease of randomness at the solid-solution interface during sorption.

Keywords

Adsorption; Adsorbent; Modified ZnO nanopowder; Thallium (I) ion; Adsorption isotherms.



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1. INTRODUCTION

Among various pollutants in waters and wastewaters, heavy metals are very toxic and dangerous for environment. Thallium is one of these pollutants that its toxicity and harm is more than the other heavy metals such as lead, cadmium, mercury, copper and zinc for mammals [1, 2]. Thallium compounds enter to the human body mainly through inhalation, food [3] and by eating vegetables and fruits grown in polluted soils [4-6].

After entering to the body, thallium compounds accumulate in bones, renal medulla, and central nervous system and throughout all the body [1]. They can harm the bones, heart, lungs, kidneys, liver and nervous system. A trace of these compounds may cause anorexia, diarrhea, vomiting, headache, temporary hair loss and their higher quantities can cause blindness and death [3, 6]. Therefore, removing the thallium compounds from waters and wastewaters is really necessary to prevent their pollution from hurting humans and environment.

Adsorption is an effective method among different methods for removing this pollutant due to low cost, ease of accessibility, ease of operation and low time of operation [7]. Thallium compounds until now, have been removed from aqueous solutions by some adsorbents such as ion-exchange resin [8], iron powder method [9], polyurethane foam [10], titania particle surfaces [11], silica gel [12], modified aspergillus niger biomass [13], dry biofilm biomass collected from a eutrophic lake [14], activated carbon [15], modified ulmus carpinifolia tree leaves [16], modified sugar beet pulp [17], geological materials [18], pistachio hull [19] and eucalyptus leaves powder [20, 21].

In recent years, nano materials have been also used as adsorbents due to their special properties. Their surface has a lot of unsaturated atoms that can easily attach to other atoms, thus they have a high capacity for the adsorption. The examples of nanocompounds for the adsorption of thallium ions are nano- Al_2O_3 [22], nano- TiO_2 [23] and multiwalled carbon nanotubes [24].

In this paper, ZnO nanopowder was used as an adsorbent to remove Tl (I) ions from aqueous solutions, for the first time. In nano materials, nano-ZnO is abundant and a cheap adsorbent. It was known that net charge of ZnO nanopowder in aqueous solution is positive, thus surface modification is essential to obtain negatively charged ZnO nanopowder [25]. We accomplished the modification by sodium phosphate solution to adsorb Tl (I) ions.

2. EXPERIMENTALS

2.1. Apparatus

An Atomic Absorption Spectrometer Model AA220 (VARIAN Co., USA) to determine the concentration of Tl (I) ion and a 420A model pH meter (ORION Co., USA) was used to measure the pH of the solutions. A TDL80-2B model centrifugal machine (Shanghai Anting Scientific Instrument Co., China) was used to separate the adsorbent from the mixture. We also used Ultrasonic Bath (71020-DTH-E, Model 1510 DTH, 220 v, EMS Company) for agitation and to prevent aggregation.

2.2. Materials

Tl (I) stock solution with proper concentration (250 mg L^{-1}) was prepared by dissolving 0.0815 g of its nitrate ($TlNO_3$, Merck, Germany) in distilled water. ZnO nanopowder was purchased from TECNAN, Spain (Purity 99.983%, Average Particle Size (APS) 20-25 nm, Specific Surface Area (SSA) $5\text{-}50 \text{ m}^2/\text{g}$). Sodium phosphate ($Na_3PO_4 \cdot 12 H_2O$) was prepared from Merck, Germany. All chemicals used in this study were of an analytical grade.

2.3. Modification of adsorbent

For adsorbing position thallium ions onto the ZnO nanopowder, it should be modified by a suitable anion to obtain the negative surface charge. Definite amount of ZnO nanopowder (0.05 g) was suspended into 10 ml from the various compound solutions (the suitable compound was tri-sodium phosphate). The mixture was then placed in the ultrasonic bath for 60 minutes for agitation and to prevent aggregation. The modified ZnO nanopowder was then used for the adsorption experiments.

2.4. Adsorption experiments

The adsorption experiments were done in 100 mL flasks containing 50 mL of Tl (I) solution of known concentration (10 mg L^{-1}) obtained from the dilution of 250 mg L^{-1} stock solutions. The initial pH of the solution was adjusted to optimum value (pH 6) with 0.1 M HCl or 0.1 M NaOH solution. The mixture was then added to each flask and it was placed in the ultrasonic bath for 60 min for agitation and to prevent aggregation. After completion, the sample was removed from the flask and was separated by centrifuging at 4000 rpm for 5 min. The obtained solution was analyzed for residual Tl (I) ion. Then, the adsorption percentage (% Adsorption) was obtained as

$$\% \text{ Adsorption} = \frac{C_i - C_f}{C_i} \times 100 \quad (1)$$

where C_i and C_f are the initial and the final concentrations of Tl (I) ions in solution, respectively.

The amount of Tl (I) ions adsorbed per unit mass of adsorbent (q_e) was obtained by following equation:

$$q_e = \frac{V}{m} (C_i - C_e) \quad (2)$$

where C_i and C_e are initial and equilibrium concentrations TI (I) ions (mg L^{-1}), respectively and V is the volume of the solution (L) and m is the mass of the adsorbent (g).

The average absolute value of relative error, AARE, compares the predicted results with experimental data. This is defined as follows:

$$AARE\% = \frac{1}{N} \sum_{i=1}^N \frac{|\text{Predicted value} - \text{Experimental value}|}{\text{Experimental value}} \times 100 \quad (3)$$

in which N is the number of data points.

In this study, various experimental conditions including concentration of modifier agent (sodium phosphate solution), solution initial pH (5-11), contact time (15-75 min), amount of sorbent (0.05-0.18 g), thallium initial concentration (5-50 mg L^{-1}) and temperature (25-60°C) on the adsorption percentage of TI (I) ion were controlled.

3. RESULTS AND DISCUSSION

3.1. Modification of the adsorbent by various compounds

First, we modified ZnO nanopowder (as adsorbent) by various compounds and then, we studied the adsorption percentage of TI (I) ion in them and we selected the adsorbent gained by higher adsorption. These compounds were: 1M NaOH, 2M NaOH, 0.1M HCl, 0.1M NaCl, 1% sodium citrate and 1% tri-sodium phosphate. As seen in Figure 1, the adsorbent modified by sodium phosphate gave the best adsorption. Upon modification, PO_4^{3-} ions attach to the adsorbent sites and subsequently to the adsorbent better than other ions, due to more aggregation of the negative charge. This favored TI (I) ion adsorption onto the surfaces of the adsorbent.

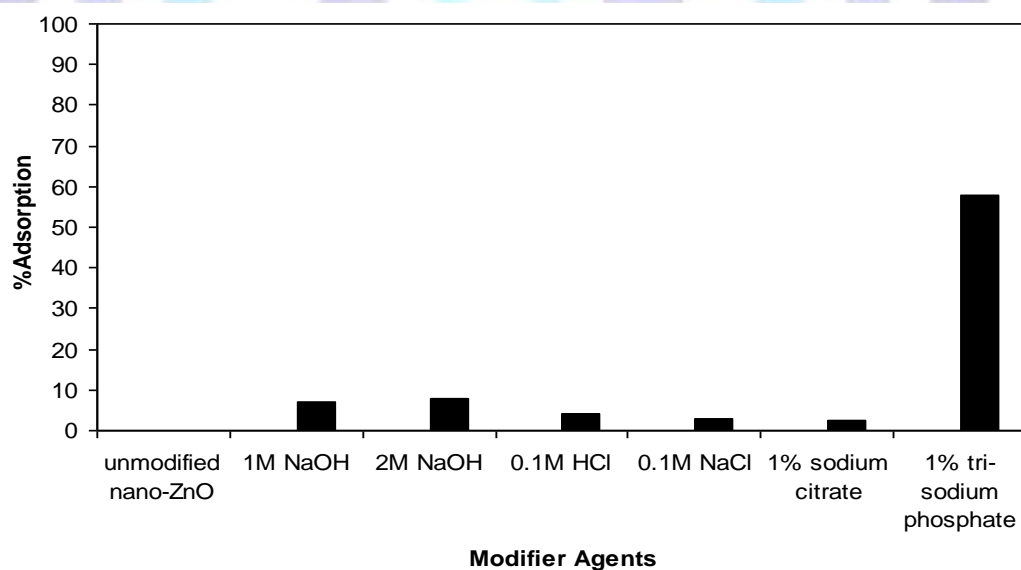


Fig 1: Modification of the adsorbent by the various modifier compounds.

3.2. Modification of the adsorbent by sodium phosphate solution

The adsorbent was modified by 1-15% w/v sodium phosphate solutions and adsorption process was done again. The results given in Figure 2 showed that the modified adsorbent by 5% w/v sodium phosphate solution is a more suitable adsorbent. Extra PO_4^{3-} ions disturb the desirable adsorption.

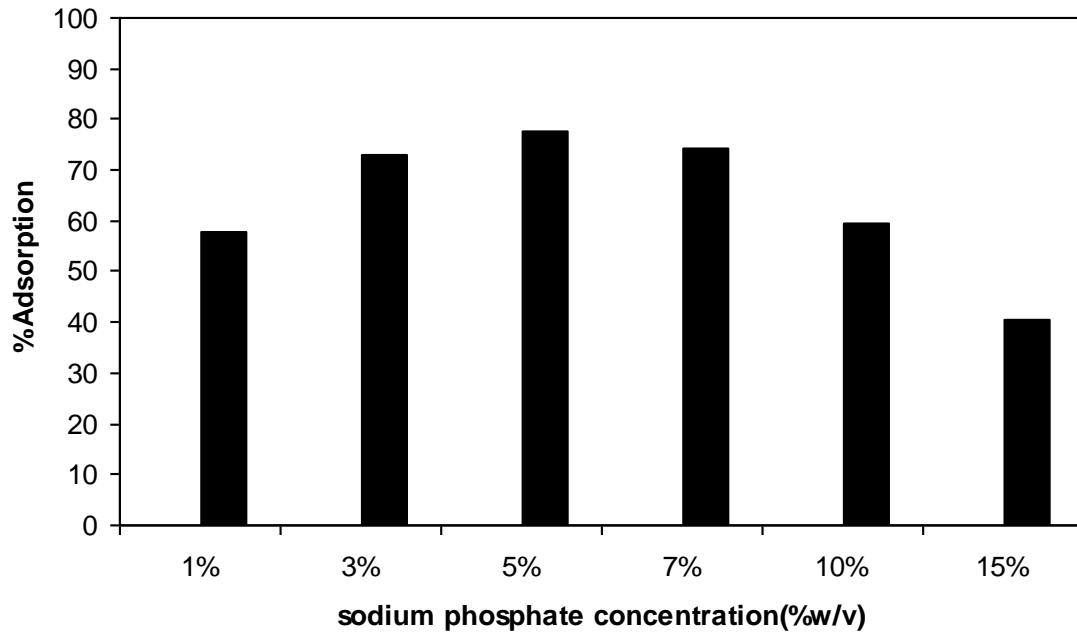


Fig 2: Optimizing concentration of modifier agent (tri-sodium phosphate).

3.3. The effect of solution initial pH on adsorption percentage

The solution initial pH is the major parameter controlling sorption processes. For study this parameter, the solution initial pH was varied in the range of 5-11. At pH<5, ZnO nanopowder is dissolved in the solution. As seen in Figure 3, Tl (I) sorption on ZnO nanopowder is increased from pH=5 to pH=6 and then it is drastically decreased by increasing pH. The satisfactory Tl (I) sorption occurred at pH=6. When the pH increase, with increasing OH⁻ ions, they compete with PO₄³⁻ ions for adsorption of the positive thallium ions and this deteriorate the adsorption process.

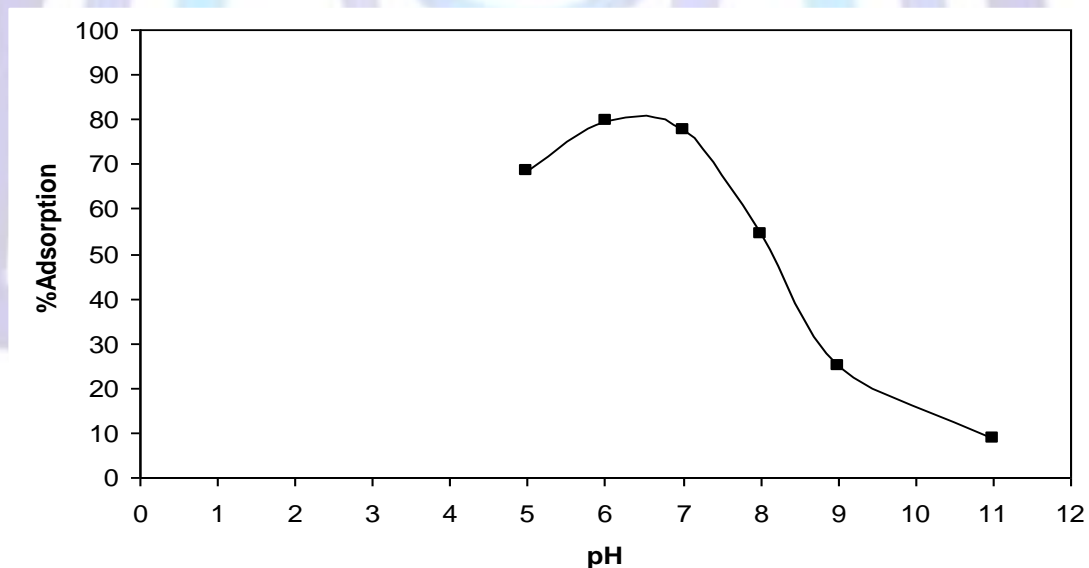


Fig 3: The effect of solution initial pH on the adsorption percentage of Tl (I) ion, initial concentration, 10 mg L⁻¹; adsorbent dosage, 0.05 g; contact time, 60 min and temperature =25±1°C.

3.4. The effect of contact time

Contact time in the test solutions was varied from 15 to 75 min, where the other conditions were constant. The results are gathered in Figure 4. We observed that the satisfactory Tl (I) sorption obtained at 60 min but the adsorption changed by contact time gently.

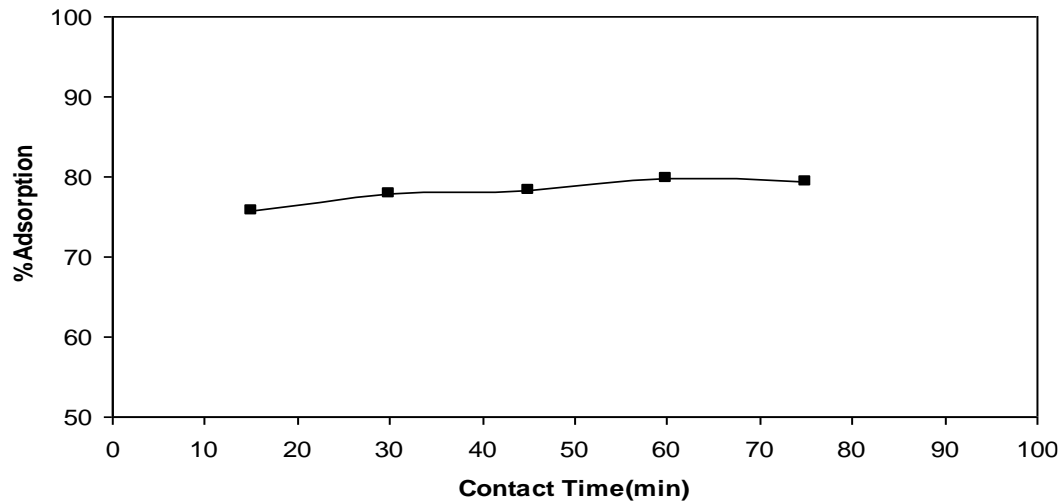


Fig 4: The effect of contact time on the adsorption percentage, initial concentration, 10 mg L^{-1} ; pH 6; adsorbent dosage, 0.05 g and temperature $=25\pm 1^\circ\text{C}$.

3.5. The effect of sorbent dosage

The adsorption percentage of TI (I) ion onto the modified ZnO nanopowder affected the dosage of the sorbent (Figure 5), while the other conditions maintained constant. The adsorption percentage increased with an increase in sorbent dosage from 0.03 g up to 0.1 g and then, it remains constant nearly. This may probably be due to increased adsorbent surface area and availability of more adsorption sites. The optimum sorbent dosage was 0.1 g.

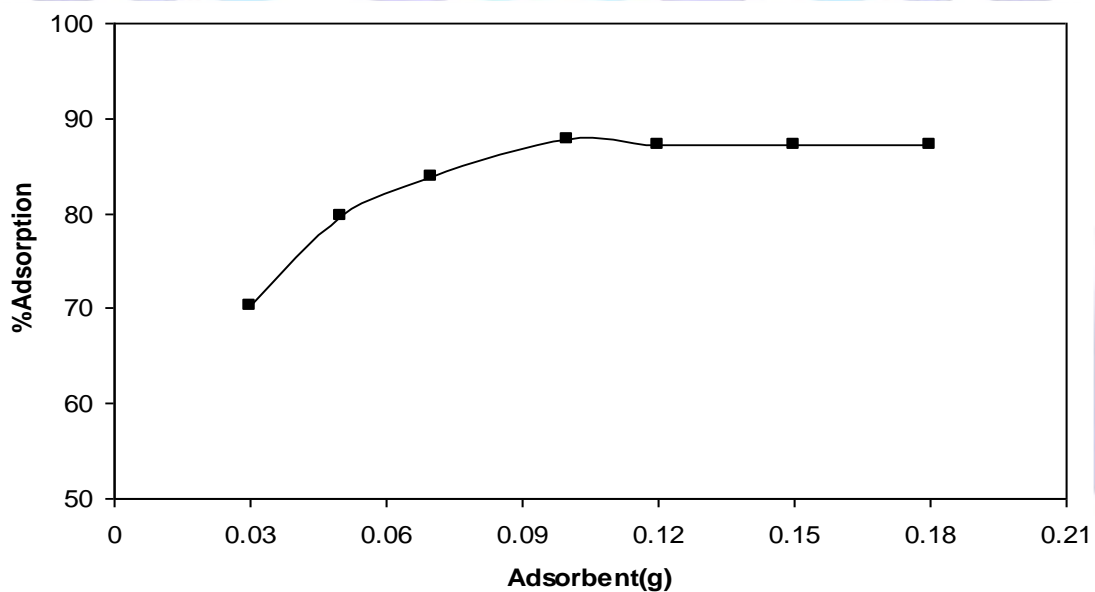


Fig 5: The effect of sorbent dosage on the adsorbent percentage of TI (I) ion, initial concentration, 10 mg L^{-1} ; pH 6; contact time, 60 min and temperature $=25\pm 1^\circ\text{C}$.

3.6. The effect of temperature

Temperature is the other parameter that affect to the adsorption percentage. Thus, temperature was varied within the range of $25\text{-}60^\circ\text{C}$, while the other conditions were constant. As seen in Figure 6, the adsorption percentage decrease with an increase in temperature. It shows that the adsorption is an exothermic process. This agrees with the process enthalpy ($\Delta H^\circ = -6.92 \text{ kJ mol}^{-1}$, Table 3). The suitable temperature was 25°C .

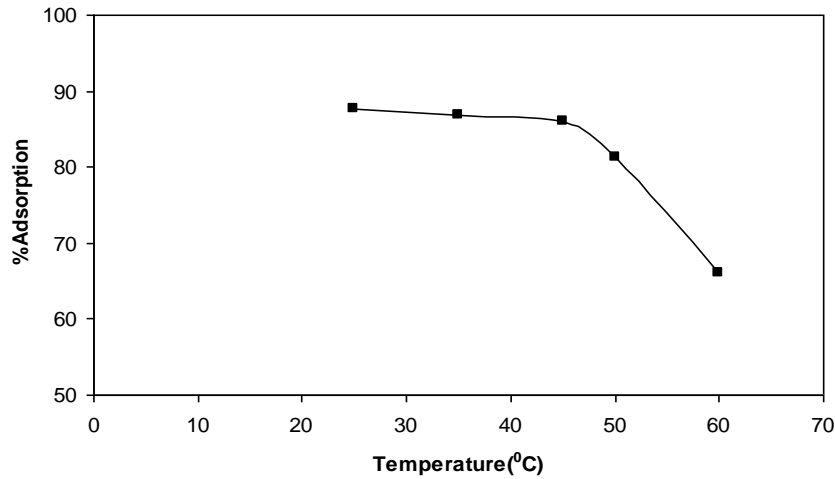


Fig 6: The effect of temperature on the adsorption percentage of Tl (I) ion, initial concentration, 10 mg L⁻¹; pH 6; contact time, 60 min and adsorbent dosage, 0.1 g.

3.7. The effect of initial concentration of thallium

Adsorption percentage also influenced by initial concentration of the thallium. In this stage, initial concentration of Tl (I) ion was changed within 5-50 mg L⁻¹, where the other conditions were constant and then the adsorption percentage of Tl (I) ion was measured. The results are shown in Figure 7. The adsorption percentage increased when the initial concentration of thallium increased. The suitable concentration range of Tl (I) ions was 50 mg L⁻¹. This may be attributed to the increase in the driving force of the concentration gradient.

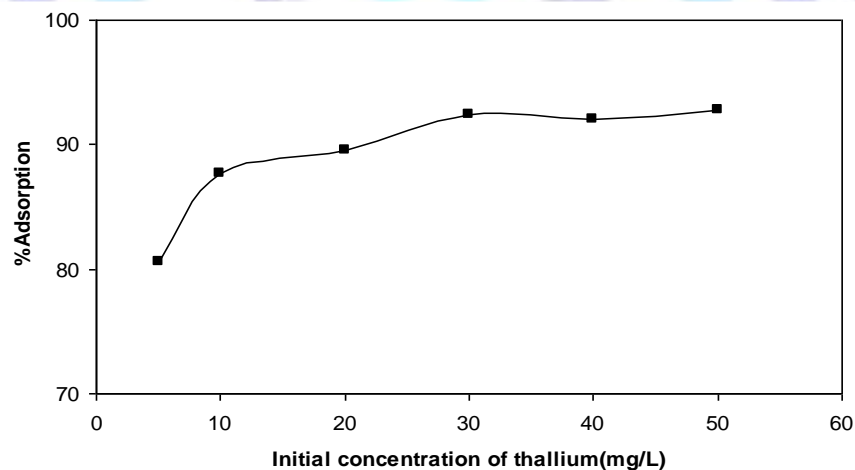


Fig 7: The effect of initial concentration of Tl (I) ion on its adsorption percentage, pH 6; contact time, 60 min; adsorbent dosage, 0.1 g and temperature = 25±1°C.

3.8. Adsorption isotherms

In order to find the suitable model for the adsorption of Tl (I) ion onto modified ZnO nanopowder, the experimental data were correlated by Langmuir, Freundlich and Temkin models. The related linear equations are:

$$\text{Langmuir equation: } \frac{1}{q_e} = \frac{1}{q_m} + \left(\frac{1}{q_m K_L}\right) \frac{1}{C_e} \quad (4)$$

$$\text{Freundlich equation: } \log q_e = \log K_F + \frac{1}{n} \log C_e \quad (5)$$

$$\text{Temkin equation: } q_e = B_1 \ln C_e + B_1 \ln K_T \quad (6)$$

where C_e (mg L⁻¹) is the liquid phase concentration of Tl (I) ions at equilibrium; q_e (mg g⁻¹) is the amount of Tl (I) ions adsorbed per unit mass of adsorbent at equilibrium; K_L (L mg⁻¹) is the Langmuir isotherm constant; q_m (mg g⁻¹) is the maximum sorption capacity of Langmuir model; K_F (mg^{1-(1/n)} L^{1/n} g⁻¹) is the Freundlich constant, and n is the heterogeneity factor; B_1 is a constant that it depend on the adsorption heat and K_T (L g⁻¹) is the Temkin isotherm constant.



We correlated the adsorption data at different initial concentrations of TI (I) ion in terms of the Langmuir isotherm (Eq. (4)). The curve of $1/q_e$ versus $1/C_e$ gave a straight line with a slope of $1/K_L q_m$ and intercept of $1/q_m$ (Figure 8 a). We also examined the data according to the Freundlich isotherm (Eq. (5)). The plot of $\log q_e$ versus $\log C_e$ gives a straight line with slope $1/n$ and intercept of $\log K_F$ (Figure 8 b). We also studied the data based on the Temkin isotherm (Eq. (6)). The plot of q_e against $\ln C_e$ gave a straight line with a slope of B_1 and intercept of $B_1 \ln K_T$ (Figure 8 c). As it was seen, the experimental data have a better agreement with the Freundlich isotherm than those of Temkin and Langmuir because the values of regression coefficient and AARE % for the Freundlich isotherm are higher than Temkin and Langmuir. Table 1(a, b, c) shows the parameters of the Langmuir, Freundlich and Temkin models and their regression coefficients and AARE%.

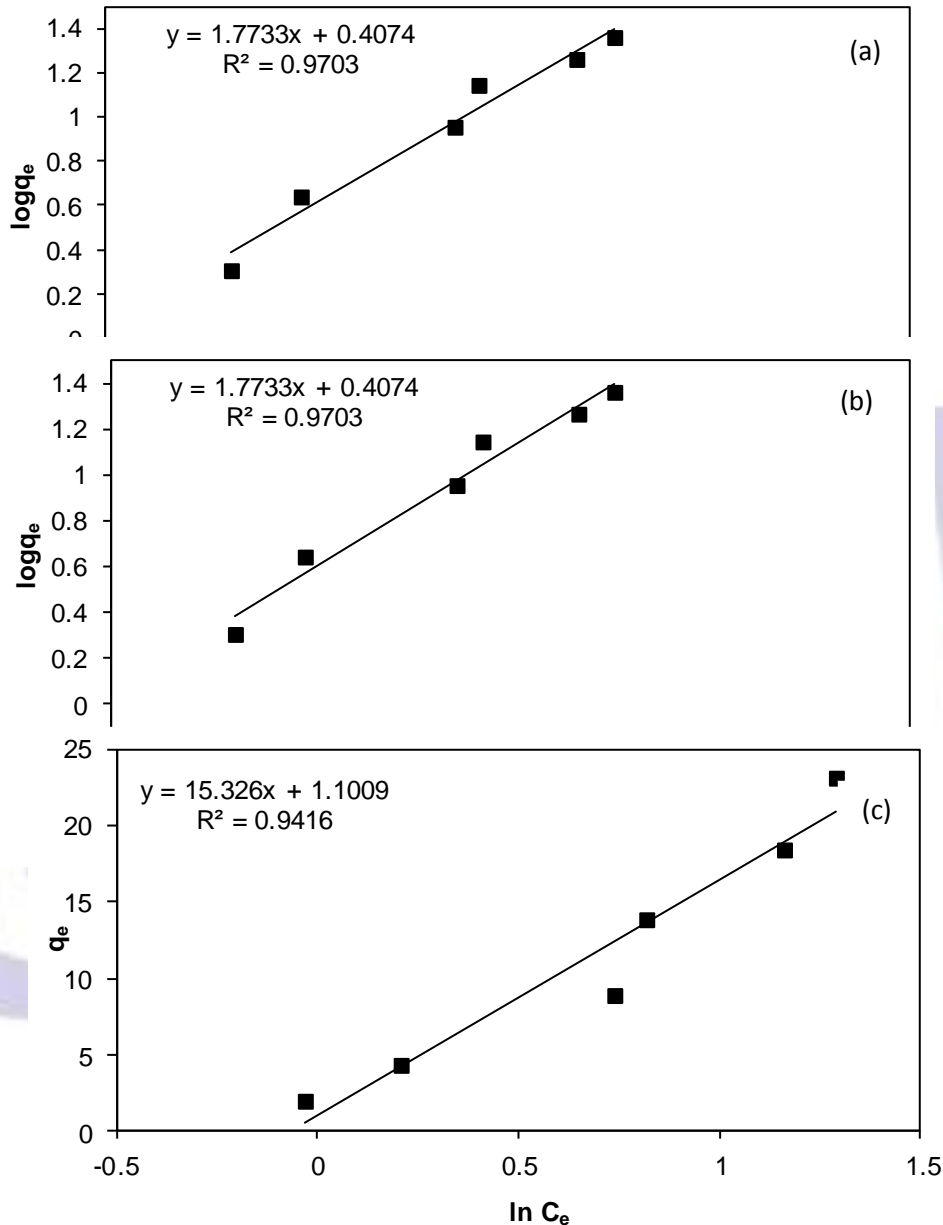


Fig 8: Sorption isotherms for TI (I) ion onto the modified ZnO nanopowder: (a) Langmuir isotherm, (b) Freundlich isotherm and (c) Temkin isotherms (conditions: pH 6; contact time, 60 min; adsorbent dosage, 0.1 g and temperature= $25 \pm 1^\circ\text{C}$).



Table 1. Parameters of the various isotherms for TI (I) ion adsorption onto the modified ZnO nanopowder (constant conditions: pH 6, contact time 60 min, adsorbent dosage 0.1 g and temperature 25±1°C)

a) Parameters of the Langmuir isotherm

K_L (L mg ⁻¹)	q_m (mg g ⁻¹)	R^2	AARE %
0.258	6.95	0.9136	51.82

b) Parameters of the Freundlich isotherm

K_F (mg ^{1-(1/n)} L ^{1/n} g ⁻¹)	n	R^2	AARE %
2.55	0.564	0.9703	17.64

c) Parameters of the Temkin isotherm

K_T (L g ⁻¹)	B_1	R^2	AARE %
1.075	15.33	0.9416	35.76

Constant separation factor, R_L , determines the essential characteristics of the Langmuir isotherm by Eq. (7) [26]:

$$R_L = \frac{1}{1 + K_L C_i} \tag{7}$$

Where K_L is the Langmuir constant and C_i is the initial concentration of TI (I) ion in solution.

This factor illustrates the shape of the isotherm and the nature of the adsorption process as below:

R_L value	Nature of the process
$R_L > 1$	unfavorable
$R_L = 1$	linear
$0 < R_L < 1$	favorable
$R_L = 0$	irreversible

The calculated R_L values against initial TI (I) concentration were shown in Figure 9. The value of R_L in the range of 0-1 at all initial TI (I) concentrations shows the favorable adsorption of TI (I) ion.

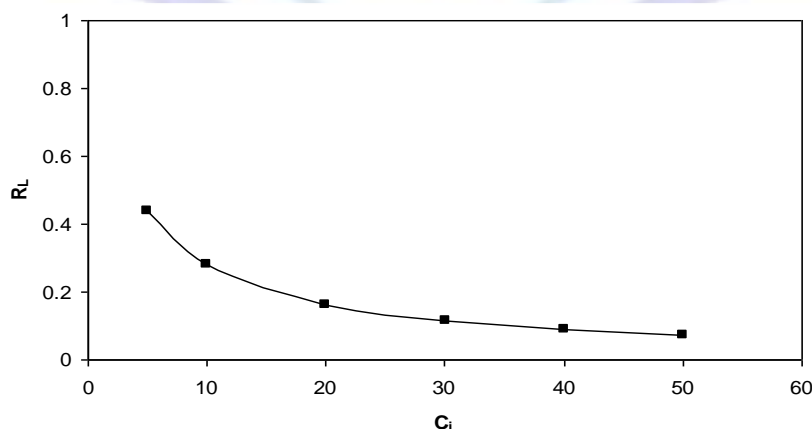


Fig 9: Separation factor for the adsorption of TI (I) onto the modified ZnO nanopowder in terms of initial concentration of TI (I) ion.

3.9. Adsorption Thermodynamics

Thermodynamic analysis helps us obtain useful information on energetic changes during adsorption process. The change in free energy (ΔG^0) can be calculated as:

$$\Delta G^0 = -RT \ln K_0 \quad (8)$$

Where R ($\text{kJ mol}^{-1} \text{K}^{-1}$) is the gas constant, T is the absolute temperature, and K_0 (L mol^{-1}) is the thermodynamics equilibrium constant. The equilibrium constant (K_0) can be defined as [27]:

$$K_0 = \frac{a_s}{a_e} = \frac{\gamma_s q_s}{\gamma_e C_e} \quad (9)$$

Where a_s and a_e are activity of ions adsorbed onto modified ZnO nanopowder as an adsorbent and the ions in the equilibrium solution, respectively. γ_s and γ_e show activity coefficients of ions adsorbed and the ions in the equilibrium solution. q_s (mg g^{-1}) is the concentration of ion adsorbed onto the surface and C_e (mg L^{-1}) is the concentration of ions in the equilibrium condition. When the solution is very dilute, the activity coefficient approaches unity and equation (9) is written as:

$$K_0 = \frac{a_s}{a_e} = \frac{q_s}{C_e} \quad (10)$$

ΔG^0 is a function that it shows the change in standard enthalpy (ΔH^0) and the change in standard entropy (ΔS^0):

$$\Delta G^0 = \Delta H^0 - T\Delta S^0 \quad (11)$$

If Eq. (11) is placed into Eq. (8), it gives

$$\ln K_0 = \frac{\Delta S^0}{R} - \frac{\Delta H^0}{RT} \quad (12)$$

Thus, ΔH^0 and ΔS^0 of the adsorption can be determined by the slope and intercept of the line obtained from plot of $\ln K_0$ versus $1/T$, respectively (Figure 10). Values C_e , q_e and K_0 in different temperatures and thermodynamic parameters are shown in Tables 2 and 3, respectively. The negative values of ΔG^0 indicate that the TI (I) adsorption process onto modified ZnO nanopowder could occur spontaneously. Generally speaking, the heat released during physical adsorption has the same order of magnitude for the heat of condensation, i.e., 2.1-20.9 kJ mol^{-1} [28], while the heat of chemisorption changes in the range of 80-200 kJ mol^{-1} [29]. Therefore, it seems that TI (I) adsorption onto modified ZnO nanopowder could be attributed to a physical sorption process. The negative value of ΔH^0 shows that TI (I) sorption is an exothermic process. The low and negative value of ΔS^0 may state that the entropy have not changed significantly during the adsorption but there is a little decrease of randomness at the solid-solution interface during sorption.

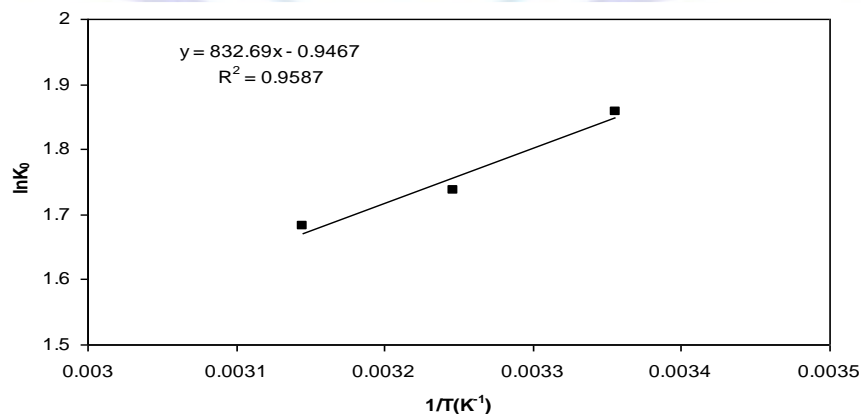


Fig 10: $\ln K_0$ vs. $1/T$ plot for the thermodynamics parameters for the adsorption percentage of TI (I) ions onto modified ZnO nanopowder (constant conditions: pH 6; contact time, 60 min; adsorbent dosage, 0.1 g and temperature= $25 \pm 1^\circ\text{C}$).



Table 2. Values C_e , q_e and K_0 in different temperatures for the adsorption of Tl (I) onto modified ZnO nanopowder (constant conditions: pH 6; contact time, 60 min; adsorbent dosage, 0.1 g and thallium initial concentration, 50 mg L⁻¹).

T (K)	q_e (mg g ⁻¹)	C_e (mg L ⁻¹)	K_0
298	23.19	3.62	6.41
308	22.98	4.05	5.67
318	22.88	4.25	5.38

Table 3. Thermodynamics parameters for the adsorption of Tl (I) onto modified ZnO nanopowder (constant conditions: pH 6; contact time, 60 min; adsorbent dosage, 0.1 g and thallium initial concentration, 50 mg L⁻¹).

T (K)	ΔG^0 (kJ mol ⁻¹)	ΔH^0 (kJ mol ⁻¹)	ΔS^0 (J mol ⁻¹ .K ⁻¹)
298	-4.60	-6.92	-7.87
308	-4.44		
318	-4.45		

3.10. Comparing nano-adsorbents

In this study, several nano-adsorbents have been compared and results were shown in Table 4. It can be seen that the highest values of the maximum adsorption capacity, q_m , are related to the modified ZnO nanopowder. Among the nano-adsorbents, nano-ZnO is an abundant and cheap adsorbent. It seems that, among the nano-adsorbents, the modified ZnO nanopowder is the most suitable nano-adsorbent for removing Tl (I) ions from aqueous solutions.

Table 4. Comparison of the maximum adsorption capacity for Tl (I) adsorption onto several nano-adsorbents.

nano-adsorbent	q_m (mg g ⁻¹)	Reference
nano-Al ₂ O ₃	6.28	[22]
nano-TiO ₂	4.09	[23]
multiwalled carbon nanotubes	0.658	[24]
nano-ZnO	6.95	current work

4. CONCLUSION

In this study, we showed that ZnO nanopowder, after modification with sodium phosphate solution, can be used as an adsorbent to adsorb Tl (I) ion from aqueous solution. We showed that 5% w/v sodium phosphate solution gave the better adsorption percentage. Experimental data indicated that the adsorption percentage was depended on parameters such as initial pH of solution, contact time, dosage of adsorbent, temperature and the initial concentration of thallium. The optimum conditions were: pH 6; contact time, 60 min; adsorbent dosage, 0.1 g; initial concentration of thallium, 50 mg L⁻¹ and temperature, 25°C. Under these conditions, the maximum adsorption percentage of Tl (I) ion on modified ZnO nanopowder obtained was 92.8%. Freundlich isotherm model provided a better fit with the experimental data than Langmuir and Temkin isotherm models by high correlation. The R_L values showed that the modified ZnO nanopowder was preferred for the adsorption of Tl (I) ion. The negative value of ΔH^0 shows that Tl (I) adsorption is an exothermic process. The low and negative value of ΔS^0 may state that the entropy was not changed significantly during the adsorption but there was a little decrease in randomness at the solid-solution interface during sorption. Among several nano-adsorbents, nano-ZnO is a more suitable adsorbent because it is abundant and cheap and its maximum adsorption capacity is higher than others.

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