

Kinetic and thermodynamics study of heavy metal ions adsorption from aqueous solution using new resin 8-hydroxyquinoline-furfural-hydroquinone (HQFH).

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ABSTRACT:

Used in this study to prepare for the new cross linked polymer adsorption to remove heavy metals(Hg^{+2} , Cd^{+2} , Zn^{+2} , Pb^{+2}) in aqueous solution . Using UV-Visible spectrophotometer technique to determine the amount of adsorption and factors affecting the adsorption of PH and the distribution coefficient and the effect of temperature and study the thermodynamic functions process. The results showed that the highest amount of adsorption was ion (Zn^{+2}), which was 98.9% , $Pb^{+2}= 80.2\%$, $Cd^{+2}=75.3\%$ and $Hg^{+2}=67.9\%$. The results the thermodynamic parameter showed that reaction exothermic and value of ΔG is spontaneous. The adsorption kinetic was found to follow pseudo-second order rate model, due to the high correlation coefficient(R^2) and agreement between the experimental and calculated value of q_e (mg/l). The results proved the applicability of isotherm Freundlich equation for adsorption coefficient higher than for Langmuir correlation equation.

Keywords: Adsorption , isotherm , distribution coefficient.

1- Introduction

Heavy metals are generally considered to be those whose density exceeds(5 g/cm^3). Removal of heavy metals from industrial wastewater is of primary importance because they are not only causing contamination of water bodies but they are also toxic to many life forms[1]. In recent years, co polymers have found widespread applications for the separation and removal of pollutant and toxic elements from waters and others media .many study was carried out of removal of heavy metals from aqueous solutions by using chelating resin [2] .removal , separation and enrichment

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of trace metals in aqueous solutions play an important role for the analysis of wastewaters, industrial and geological samples, as well for environmental remediation. The guarantetra ethylene pentamine resin was used to removal of heavy metal ions(Fe^{+2} , Co^{+2} , Ni^{+2} , Cu^{+2} , Zn^{+2} , Cd^{+2} , and Pd^{+2})from aqueous solutions[3]. the synthetic resins have been found selective for certain metal ions over a wide pH range of substituted 8-hydroxyquinoline with 1,2-dichloroethylene[4].the cross linked polymer [5] from the reaction with different amounts of disphenol-a to certain trivalent ions of lanthanides was investigated by static a load balancing method. Copolymer resin derived from 8- hydroxyquinoline –formaldehyde –pyrogallol was synthesized and studied to removal of toxic heavy metals from aqueous media and industrial wastewater containing Zn(II)and Pd(II) [6].

The present paper included preparation and identification of co-polymer properties of poly(8-hydroxyquinoline –furfural-hydroquinone) towards some pollutant ions such as Pb^{+2} , Zn^{+2} , Cd^{+2} , and Hg^{+2} . The rate of metal ion uptake and the pH-binding capacity profiles were investigated with the view to determine the efficiency and selectivity of the chelate-forming resin, and Studies on adsorption kinetics and adsorption isotherms have been carried out, and the best fitting models for the rate kinetics and isotherms have been proposed.

2.MATREIAL AND METHODS

2-1:Materials

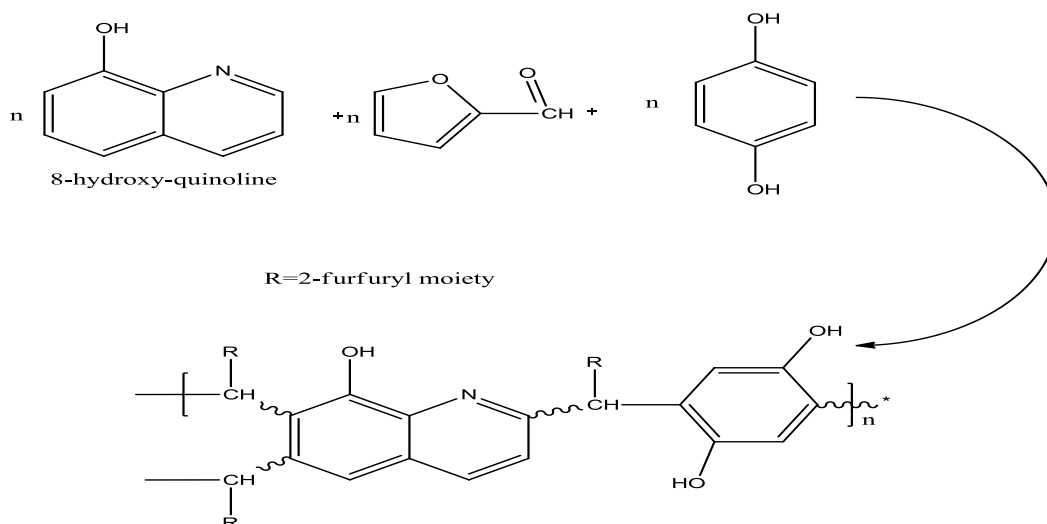
materials	Hydrochloric Acide HCl	Sodium hydroxide	Diphenyl carbazone	Ethanol	8-hydroxy qoinoline	Furfural	Hydro quinonline
company	Fluka.AG	Fluka.AG	B.D.H	B.D.H	Fluka.swit.	Sigma-Aldrich	B.D.H
purity		98%	97%	99%	99.99%	99%	99.9%

2-2: Synthesis of (8-hydroxyquinoline-furfural-hydroquinone) copolymer:

The cross linked polymer were prepared by the following procedure. A mixture of 8-hydroxyquinoline(2.91gm,0.02 mole) and distilled furfural(1.97gm,0.021mole) with small amounts of cross-linking hydroquinone(2gm,0.018 mole) and 100 mL of methanol was adjusted to pH 6.0 with 0.2 M HCl. The reaction mixture was stirred at room temperature for two hours until get homogeneous solution, then refluxed on water bath for 8 hours to afford dark brown viscous mass of terepolymer of HQFH which washed several times with ethanol, methanol to remove un reacted materials. The purification of the HQFH terepolymer was done by fractional precipitation via dissolving the new terepolymer in DMF then its

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precipitation in aqueous ethanol, thus dried in desiccators over calcium chloride pellets, scheme(1).



Scheme (1) synthesis of (8-hydroxy quinoline-furfural-hydroquinone) copolymer.

2:2:1. H NMR and FT.IR characterization of HQFH.

The proton NMR spectra of the copolymer in DMSO-d6 solution, figure (1) displays the following absorptions: 9.75-8.99 ppm(-OH,2H),6.6-7.02 PPM(Ar-H,5-6H of Quinoline-H), 2.8-3.34 ppm(C-H bridge of cross linked to Fur.).

The data of IR(cm-1) spectra(shimadzu 8400 Japan) of copolymer analysis was carried out in order to identify the functional groups in HQFH. The FTIR spectra in the range of 400-4000cm is show in fig.(2).the data function groups are = 3436 cm⁻¹ is due to (O-H), 3054-2931 due to (C-H Ar-H,Fur-H), 1650-1592(C=N-Quinoline ring and C=C-), 1025-1268(C-O), 817-937(γ C-C bending of cross liked bonds). The results of elemental analysis are in good agreement with calculated value of % C, %H, %N as scheme (1). The micro-elemental analyses for the HQFH copolymer was Found (Calc.) %C=71.11(72.06), N%=3.66(4.21), H%=4.03(4.55).

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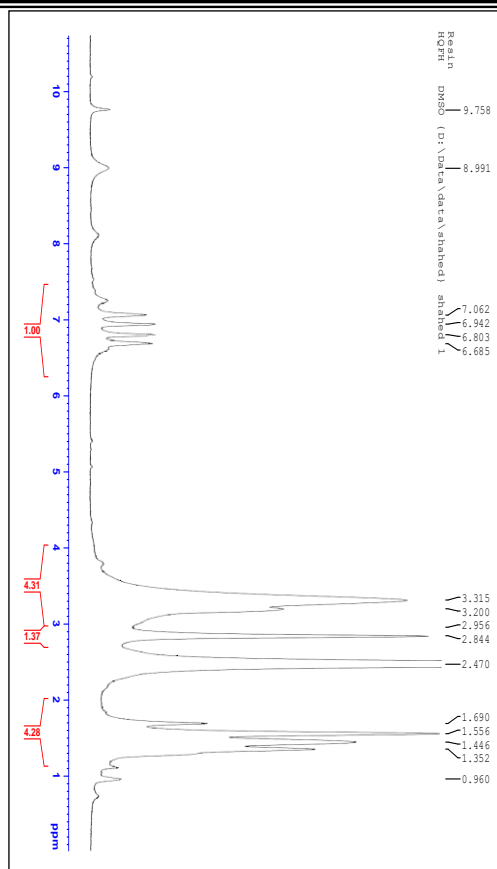


Figure (1) .H NMR spectra of HQFH resin.

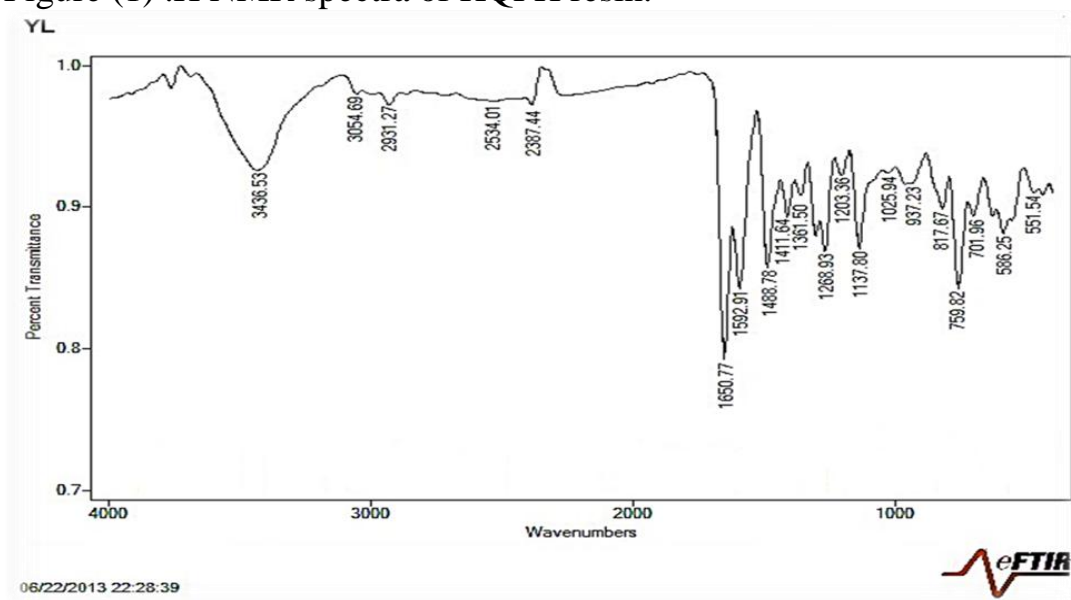


Figure (2). FT-IR spectra of HQFH .

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2.3: preparation of heavy metals Ions:

Stock solution :A stock solution of aqueous solution of Hg^{+2} , Zn^{+2} , Cd^{+2} , and Pb^{+2} was obtained by dissolving (0.6184gm) of mercury nitrate and (0.4786gm) of zinc chloride and (0.4745gm) of cadmium nitrate and (0.4608gm) of lead nitrate in 1000ml of double distilled water to give 1000ppm solution .

2.3: Hg, Cd, Zn,Pb determination :

The change in heavy metals ions concentration due to adsorption was determined by UV-vis spectrophotometer by formation complex soluble with diphenylcarbazone .Absorbance was measured at wavelength 523nm of Hg^{+2} and 525nm of Zn^{+2} , Cd^{+2} , Pb^{+2} .

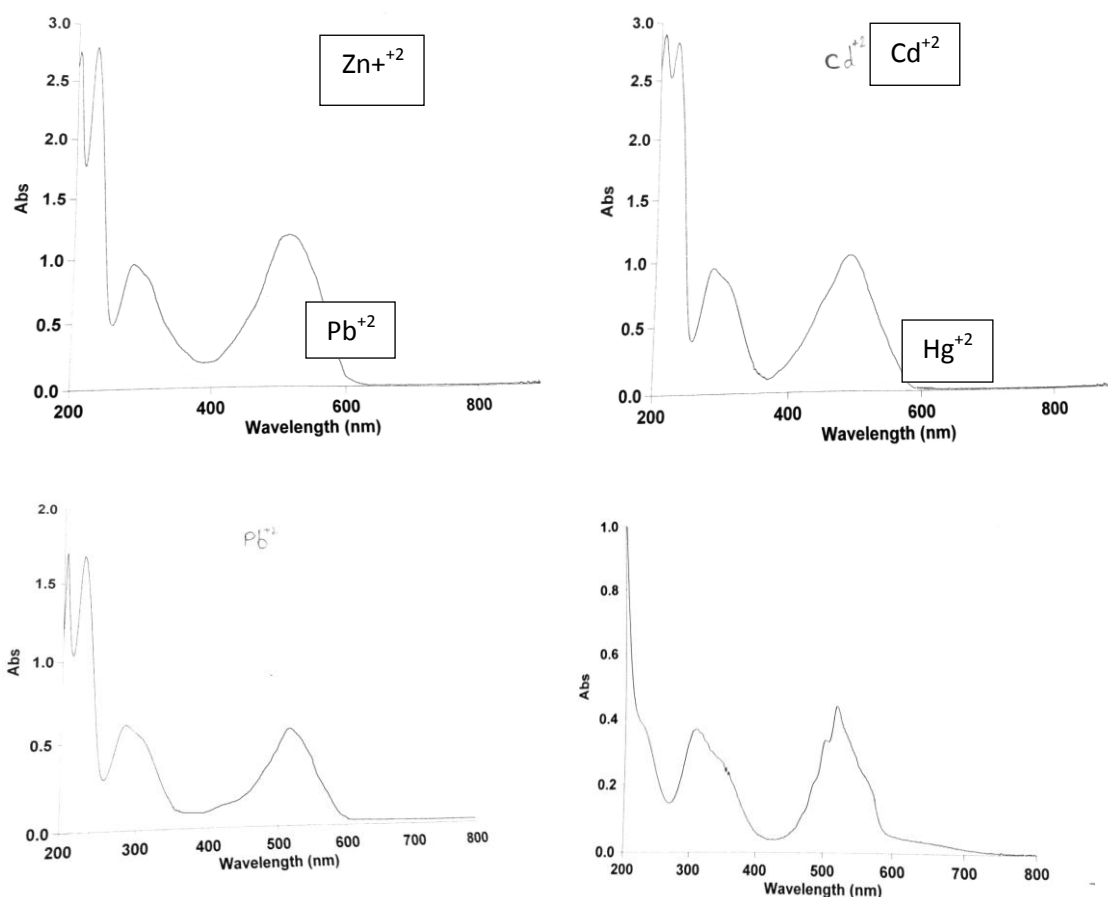


Fig-3: UV-visible spectral of Hg^{+2} , Cd^{+2} , Zn^{+2} , Pb^{+2} with diphenylcarbazone. The percentage of (Hg^{+2} , Cd^{+2} , Zn^{+2} , Pb^{+2}) ion removed by resin was calculated as % ions as removal = $[(C_o - C_e) / C_o] \times 100 \%$, where C_o and C_e are the initial and final concentration of ions solution(mg/L) respectively.

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2.4. Calibration curve of heavy metals:

Different concentrations of(Hg^{+2} , Cd^{+2} , Zn^{+2} , Pb^{+2})complex solutions were prepared by serial dilutions of the stock solution(20-300ppm) . Absorbance values of these solutions were measured at the specified λ_{max} value (523nm and 525nm) and plotted versus the concentration values Figure (4).

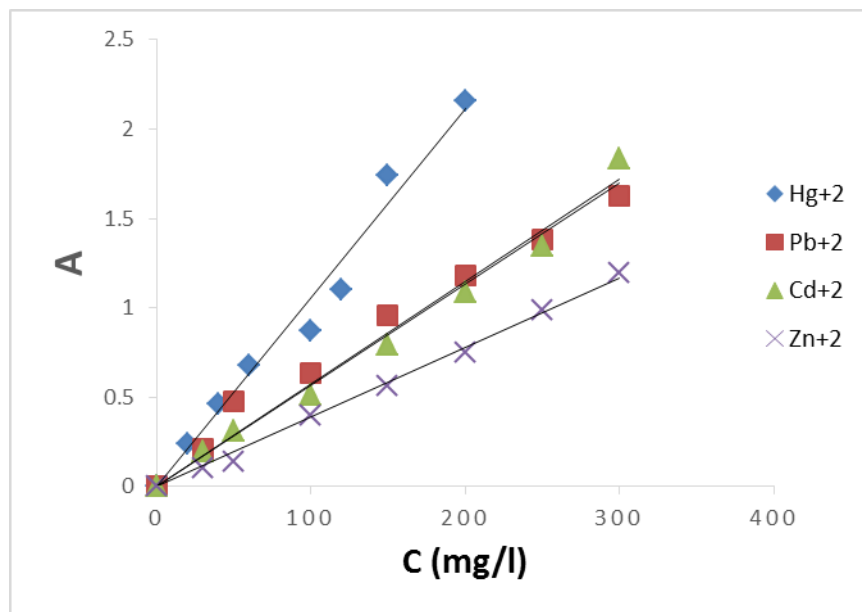


Fig.(4) calibration curve of (Hg^{+2} , Cd^{+2} , Zn^{+2} , Pb^{+2})

Results and discussion

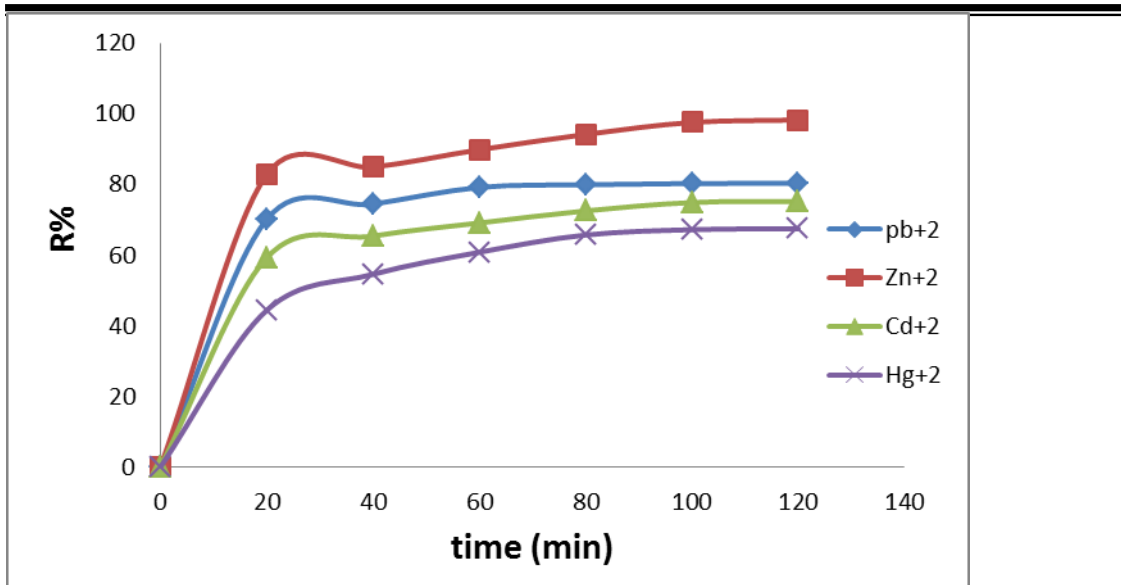
1- Effect of contact time:

The effect of contact time on the removal of metals ions Hg^{+2} , Cd^{+2} , Zn^{+2} , Pb was carried out deferent intervals ranging from (20-120 minutes).in each case 50 ml of metals ions solution of initial concentration (100mg/L) was added to each of the conical flasks and added 0.5 gm Of resin and the mixture agitated at constant oscillation of 120prm. After the state time the samples were filtered (whatman NO. 40)and absorbance (100conc./varian .USA)of each solution was measured by using UV-vis spectrophotometer fug.(5). The removal of metals ions adsorption was determined by the following equation :

$$\% \text{removal} = \frac{C_o - C_e}{C_o} \times 100 \dots\dots\dots (1)$$

Where C_o and C_e are the concentration initial and equilibrium before and after adsorption process respectively [7].

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Fig(5) :Effect of contact time on removal of (Hg⁺²,Cd⁺² ,Zn⁺² ,Pb⁺²) by HQFH resin.

It is observed that in all cases percentage removal is comparatively lower for (20min) contact time ,with increasing removal at higher contact time up to (100min) and then gradually decreased at (120min) .In case of (Zn⁺²)and Pb⁺² ions, the percentage removal reaching nearly (80-98%) only around (100min). In this time the heavy metal are constant because the resin was saturated.

2- Effect of PH and Distribution coefficient

The pH is an important parameter that effects the adsorption behavior of metal ions in aqueous systems , metal removal ions from an aqueous solution by sorption are dependent on pH of the solution which influence the surface charge of the sorbent[8,9].The distribution coefficient of Hg⁺² ,Cd⁺²,Pd⁺², and Zn⁺² ions was carried out a PH range (2-8) , whilst maintaining all other parameter as constant (C_o= 100mg/l and weight of resin (0.5g) and KD was calculated according to equation (1).

$$KD = \frac{C_o - C_e}{C_e} \times \frac{V}{M} \dots\dots\dots(2)$$

- Where: C_o= initial concentration of metal ion (mg/L)
- C_e= equilibrium concentration of metal ion (mg/L)
- V= volume of solution (ml)
- M =adsorbent of mass (gm).

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Table(1): Distribution coefficients of various metal ions at different PH.

KD				PH
Cd ⁺²	Pb ⁺²	Hg ⁺² Zn ⁺²		
235.57	77.26	78.57	88.3	2
293.7	136.96	125.73	125.72	3
468.18	205.81	227.86	198.47	4
646.26	313.22	148.75	229.83	5
1288.88	227.86	405.05	300.92	6
834.57	122.71	108.76	187.66	7
163.15	100.88	53.13	120.37	8

The results show that the distribution coefficient value first increased and then decreased with increasing PH .the order of the KD value for metal ions at PH of their maximum adsorption follows the sequence : Zn⁺² > Pb⁺² > Cd⁺² > Hg⁺² show strong adsorption in low PH range (5-6) because the copolymer (HQFH) was effective in the adsorption of heavy metal ions in this range . The Zn⁺² ion shows maximum adsorption at PH 6 ,with KD=1288.88 whereas Pb⁺² and Hg⁺² show an appreciably high adsorption at PH 6 with value of KD=405.05 and 300.92 respectively. The difference in distribution coefficients at the same PH for different metal ions suggests possible strategy for separation of these ions from their mixtures.

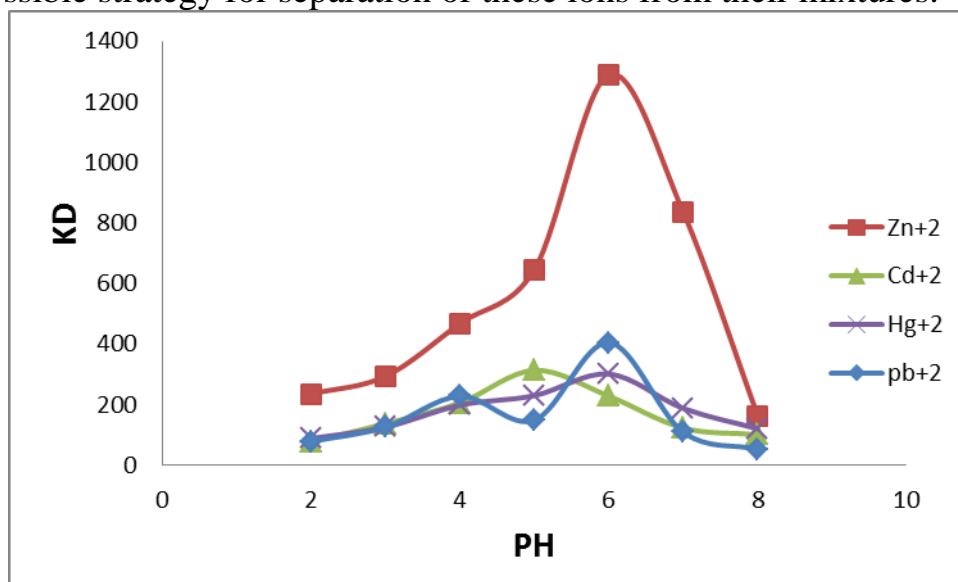


Fig (6) :Effect of PH on removal of (Hg⁺²,Cd⁺² ,Zn⁺² ,Pb⁺²) by HQFH resin.

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3. Effect of temperature

The effect of the solution temperature on the adsorption capacity was investigated for Hg(II) ,Cd(II),Pb(II)and Zn(II) ions solutions at initial metal Concentration 100 mg/L and adsorbent dose 0.5 g. three different temperatures of 25, 35 and 45 °C and for a contact time 100 minutes with PH constant were considered. The following formula used to calculate the concentration of sorbate after sorption experiments [10]:

$$Q_e = V_{sol} (C_o - C_e) / m \dots\dots\dots (3)$$

Where Q_e , V_{sol} , C_o , C_e , and m are quantity of sorbet in mg.g-1, volume of solution in L, initial concentration of sorbet in mg/L, concentration of sorbet at equilibrium in mg/L, and weight of sorbent in(g) respectively. From Figure 7,table (2) it can be observed the removal ions are exothermic in nature [11].

Table(2).Effect of temperature on adsorption heavy metal by HQFH.

Temperature (K)	Hg ⁺²		Cd ⁺²		Pb ⁺²		Zn ⁺²	
	C _e	Q _e	C _e	Q _e	C _e	Q _e	C _e	Q _e
298	28.37	7.163	29.84	7.016	13.62	8.638	2.84	9.716
308	39.49	6.051	34.96	6.504	26.85	7.315	13.17	8.683
318	46.53	5.347	37.28	6.272	41.37	5.863	29.30	7.070

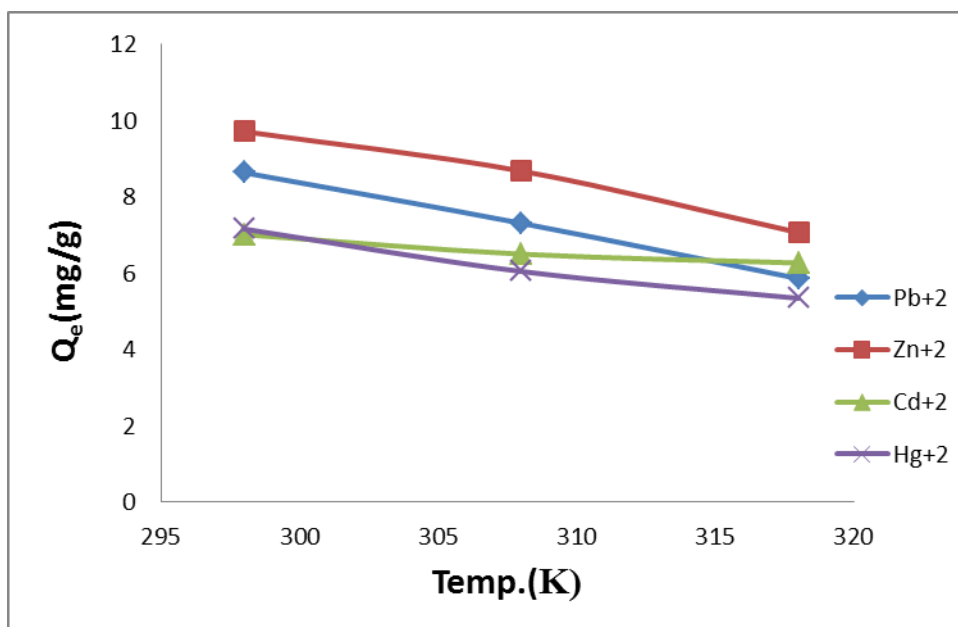


Figure (7). adsorption capacity (Q_e) vs. Temperature (K), C_o=100 mg/l.

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4- Adsorption Isotherms:

The equilibrium relationships between adsorbent and adsorbate are described by adsorption isotherms, usually the ratio between the quantity adsorbed and that remaining in solution at a fixed temperature at equilibrium. The adsorption of a substance from one phase to another leads to a thermodynamically defined distribution of that substance between the phases as the system reaches equilibrium state [12]. This distribution can be expressed in terms of adsorption isotherms. In this investigation, Langmuir and Freundlich isotherm models were used to analyze the equilibrium data.

The Langmuir adsorption isotherm is developed assuming that a fixed number of accessible sites are available on the adsorbent surface, all of which have the same energy; adsorption is reversible; monolayer adsorption occurs and there are no lateral interactions among the adsorbents. The Langmuir adsorption isotherm is defined as

$$C_e/q_e = 1/q_m b + C_e/q_m \dots\dots\dots(4)$$

Where,

q_e : Amount adsorbed per unit weight of adsorbent at equilibrium (mg/g)

C_e : Equilibrium concentration of adsorbate in solution after adsorption (mg/g)

q_m : Empirical Langmuir constant which represents maximum adsorption capacity (mg/g).

b : Empirical Langmuir constant (L/mg)

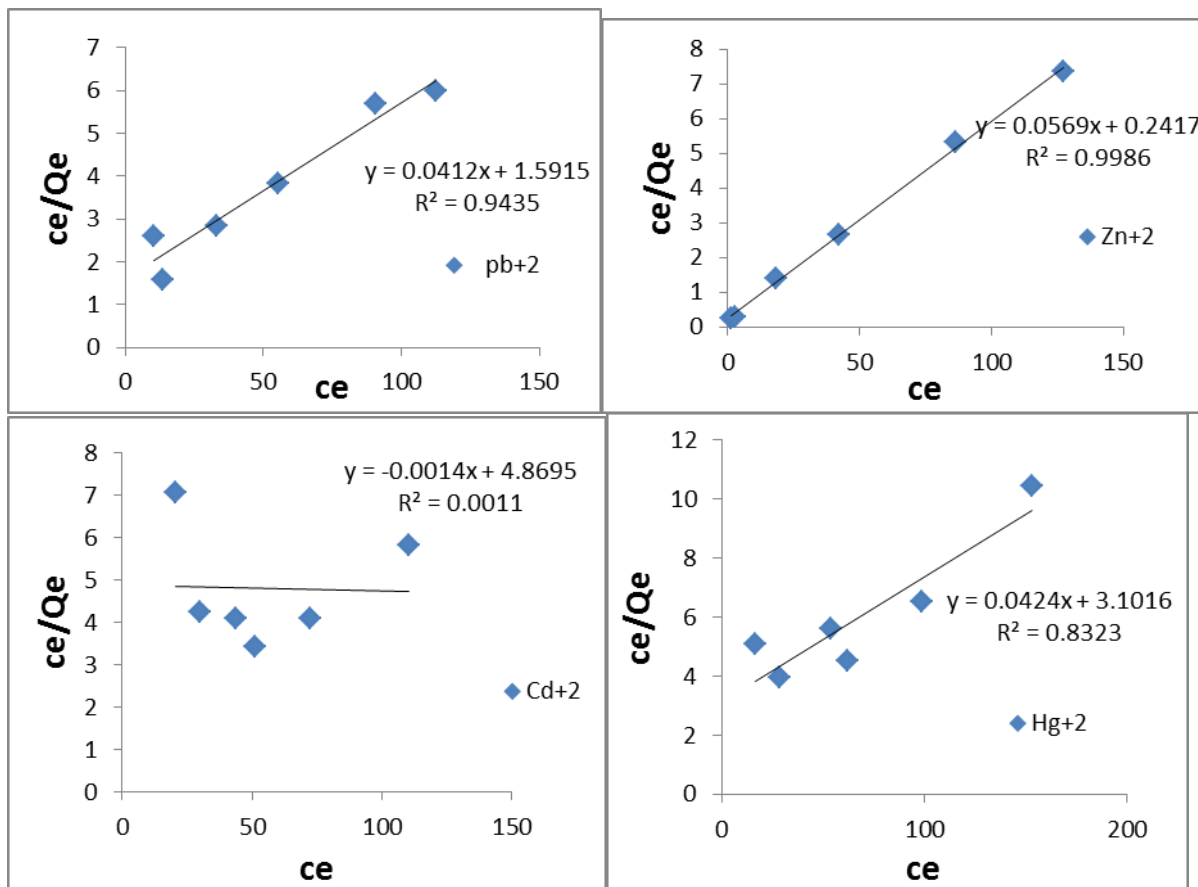
Plot of C_e/q_e vs C_e is linear shown in Figure(8). The values of (q_m) and R_L shown in Table (4).

The essential characteristics of the Langmuir isotherm can be expressed by a dimensionless constant called equilibrium parameter R_L .

$$R_L = 1/(1 + bC_o) \dots\dots\dots (5)$$

R_L values indicate the type of isotherm. The R_L value between 0 and 1 indicates favorable adsorption [13].

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Figure(8).the Langmuir adsorption isotherm for Hg⁺²,Cd⁺²,Pd⁺²,Zn⁺² onto HQFH

Freundlich adsorption isotherm

The Freundlich adsorption isotherm is an empirical equation based on the adsorption on a heterogeneous surface. The linear form of Freundlich adsorption isotherm can be defined by the following equation:

$$\log q_e = \log k_F + 1/n \log C_e \dots\dots\dots(6)$$

The constant n is an empirical parameter that varies with the degree of heterogeneity and (K_F) is a constant related to adsorption capacity. The constants n and (K_F) were calculated from the previous equation by plotting $\ln C_e$ against $\ln q_e$ (the slope = $1/n$ and the intercept = $\ln K_F$) (Figure 5). The values of n (which reflect the intensity of adsorption) are between (1 and 10)

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represent a favorable adsorption. From the results are presented in Table (4). It can be seen that the adsorption of Hg^{+2} , Cd^{+2} , Pd^{+2} , Zn^{+2} on resin adsorption models, where as Langmuir model is more fitted in for Zn^{+2} , Pb^{+2} adsorption (depending of the values of $R^2=0.998$, $R^2=0.943$ respectively).while in Cd^{+2} , Hg^{+2} the freundlich is fitted ($R^2=0.865$, $R^2=0.858$ respectively).

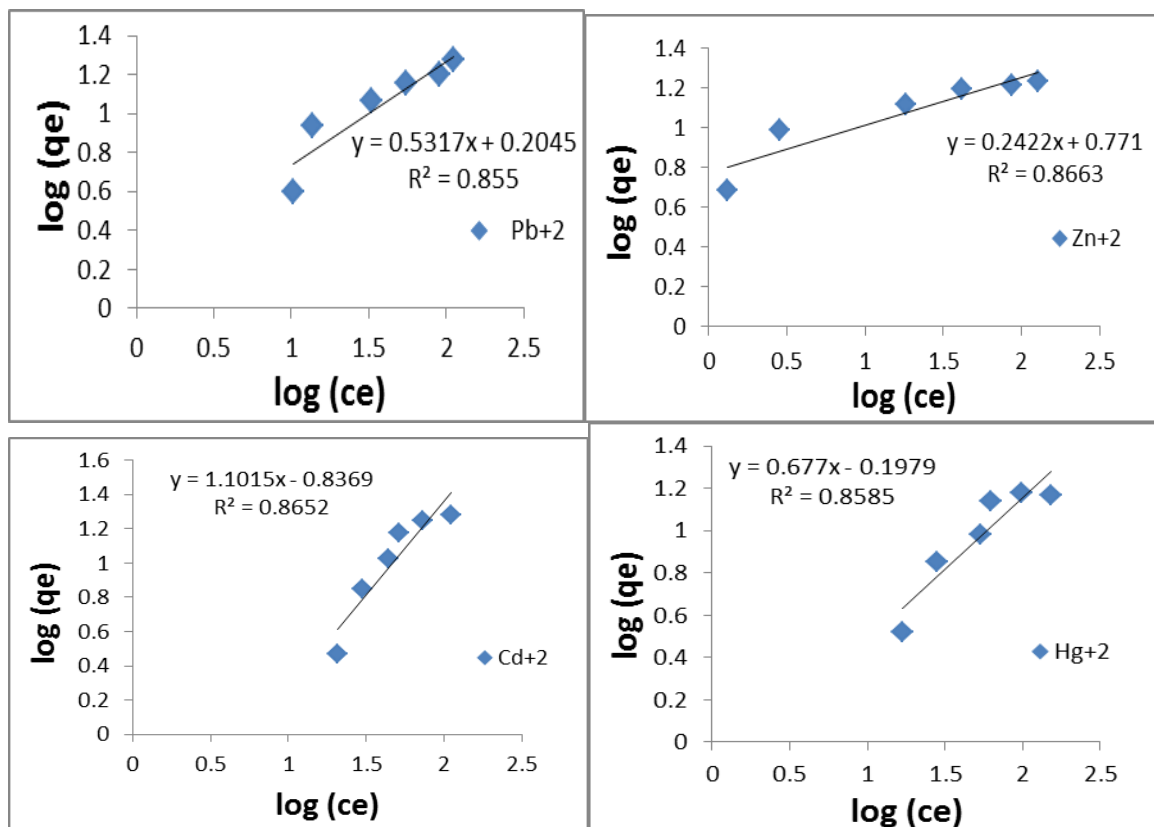


Figure (9).the Freundlich adsorption isotherm for Hg^{+2} , Cd^{+2} , Pd^{+2} , Zn^{+2} onto HQFH

Table(4) fitted parameter Freundlich and Langmuir

Metal ions	Freundlich constants			Langmuir constants		
	Kf	n	R ²	q _m	RL	R ²
Hg	1.967	1.477	0.858	23.584	0.4237	0.832
Cd	2.307	0.907	0.865	0.0011
Pb	1.226	1.880	0.866	24.271	0.2793	0.943
Zn	2.161	4.128	0.855	17.574	0.0407	0.998

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5- Adsorption thermodynamics

Thermodynamic behavior of the adsorption of Hg,Cd,Pb,Zn on HQFH was evaluated by the thermodynamic parameters including the change in free energy (ΔG), enthalpy (ΔH), and entropy (ΔS). These parameters are calculated from the following equations[14]:

$$K_{ad} = \frac{C_s}{C_e} \quad C_s = C_o - C_e \dots\dots\dots(7)$$

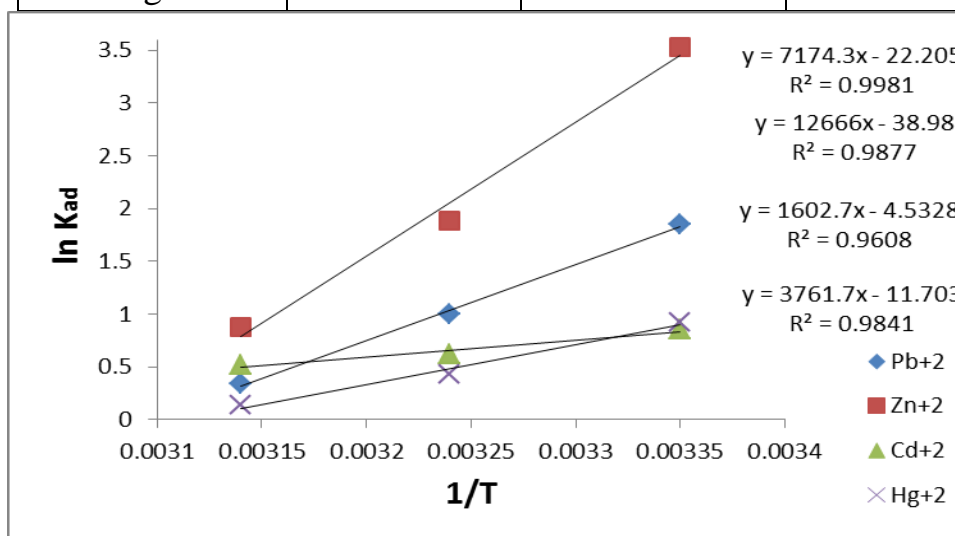
$$\ln K_{ad} = \frac{-\Delta H^0}{RT} + \frac{\Delta S^0}{R} \dots\dots\dots(8)$$

$$\Delta G^0 = \Delta H^0 - T\Delta S^0 \dots\dots\dots(9)$$

Where, K_{ad} is the equilibrium constant, C solid is the concentration of $Hg^{+2}, Cd^{+2}, Pb^{+2}, Zn^{+2}$ at the solid phase (adsorbent) at equilibrium, C liquid is the liquid phase concentration of $Hg^{+2}, Cd^{+2}, Pb^{+2}, Zn^{+2}$ at equilibrium, (T) is the absolute temperature (K) and ($R=8.314$ j/mol.k) is the gas constant. Vant Hoff equation was used to estimate the values of ΔH^0 and ΔS^0 . From the intercept and slope of the plot of $\log K_d$ vs. $1/T$ (Figure 10) These values are presented in Table 5.

Table 5. Thermodynamic parameters of Hg,Cd,Pb,Zn adsorption on HQFH

Metal ions	ΔH^0 (KJ/mol)	ΔS^0 (KJ/mol.K)	ΔG^0 (KJ/mol)
Pb^{+2}	-59.647	-0.1846	-4.6362
Zn^{+2}	-105.305	-0.3240	-8.7530
Cd^{+2}	-13.324	-0.03768	-2.0954
Hg^{+2}	-31.274	-0.09729	-2.2820



Figure(10). Plot of Van't Hoff equation for the adsorption of heavy metal ions onto HQFH.

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The plot of $\ln K$ against $1/T$ (Vant's Hoff plot) of the metal ion solution was carried out as indicated in Figure 7, in which the slope equals to $-\Delta H^\circ/R$ and the intercept equals to $\Delta S^\circ/R$. These thermodynamic parameters are presented in Table 5. The Gibbs free energy ΔG° were determined from equation 9. All heavy metal ions the ΔH s were negative; thereafter the sorption processes were exothermic physical in nature. Hence, the adsorption is highly expected at the entire range of temperatures. The negative values of ΔG° of all metal ions examined indicate the feasibility of the process and the spontaneous nature of the adsorption. Its

Magnitude was consistent with electrostatic type of interactions between the adsorbate and the adsorbent surfaces (physical adsorption) [15]. The negative values of ΔS° suggest a decrease in the randomness at the solid/solution

Interface during the adsorption of $Hg^{+2}, Cd^{+2}, Pb^{+2}, Zn^{+2}$ ions onto HQFH.

6- Kinetic Study of adsorption

The study of adsorption kinetic describes the solute uptake rate and evidently this rate controls the residence time of adsorbate uptake at the solid-solution interface. The kinetics of $Hg^{+2}, Pb^{+2}, Cd^{+2},$ and Zn^{+2} adsorption on (8-hydroxy quinoline-furfural-hydroquinone) copolymer were analyzed using pseudo first order[16], pseudo second order [17], kinetic models. The conformity between experimental data and the model predicted values was expressed by the correlation coefficients (R^2 values close or equal to 1). A relatively high R^2 value indicates that the model successfully describes the kinetics of the adsorption.

Pseudo First Order Equation

The pseudo first order equation (Lagergren model) is generally expressed as follows:

$$dq_t / dt = k_1 (q_e - q_t) \dots\dots\dots(10)$$

Where, q_e is the equilibrium adsorption capacity(mg/l) and q_t is the quantity of heavy metal ions adsorbed at time t (mg/l) , k_1 is the rate constant of pseudo first order adsorption (min^{-1}). After integration and applying boundary conditions, $t = 0$

to $t = t$ and $q_t = 0$ to $q_t = q_t$, the integrated form of Equation (10) becomes:

$$\ln (q_e - q_t) = \ln (q_e) - k_1 t \dots\dots\dots(11)$$

A plot of $\ln (q_e - q_t)$ against(t) should give a linear relationship for the applicability of the first order kinetic.

The pseudo second order adsorption kinetic rate equation is expressed as:

$$dq_t / dt = k_2 (q_e - q_t)^2 \dots\dots\dots(12)$$

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Where k_2 is the rate constant of pseudo-second-order adsorption (g/mg min^{-1}).we have a linear form as: $t/q_t = 1/(K_2 q_e^2) + t/q_e$(13)

The plot of t/q_t versus (t) should give a linear relationship for the applicability of the second order kinetic, and q_e , K_2 can be determined from the slope and intercept of the plot, respectively, show fig.(11,12)and table(6).

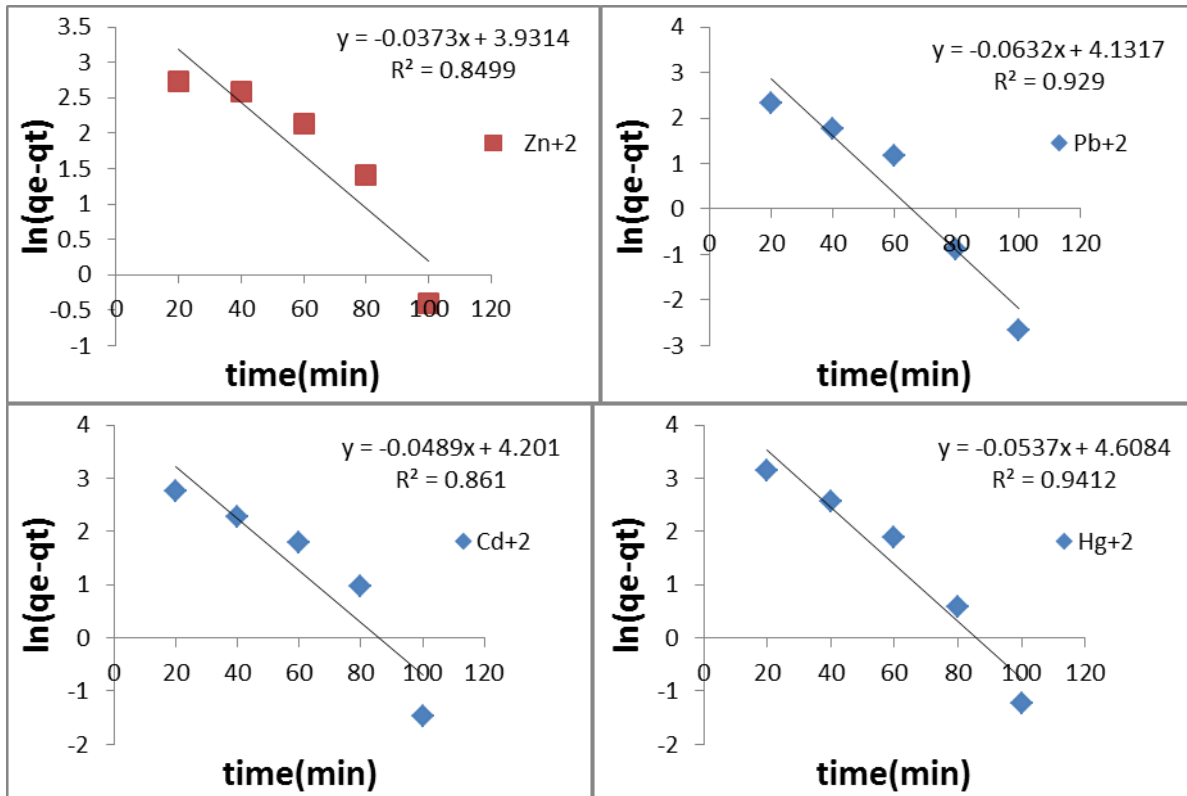


Figure (11). Pseudo First order kinetic plot for the adsorption of Hg^{+2} , Cd^{+2} , Pb^{+2} , Zn^{+2} ions on to HQFH.

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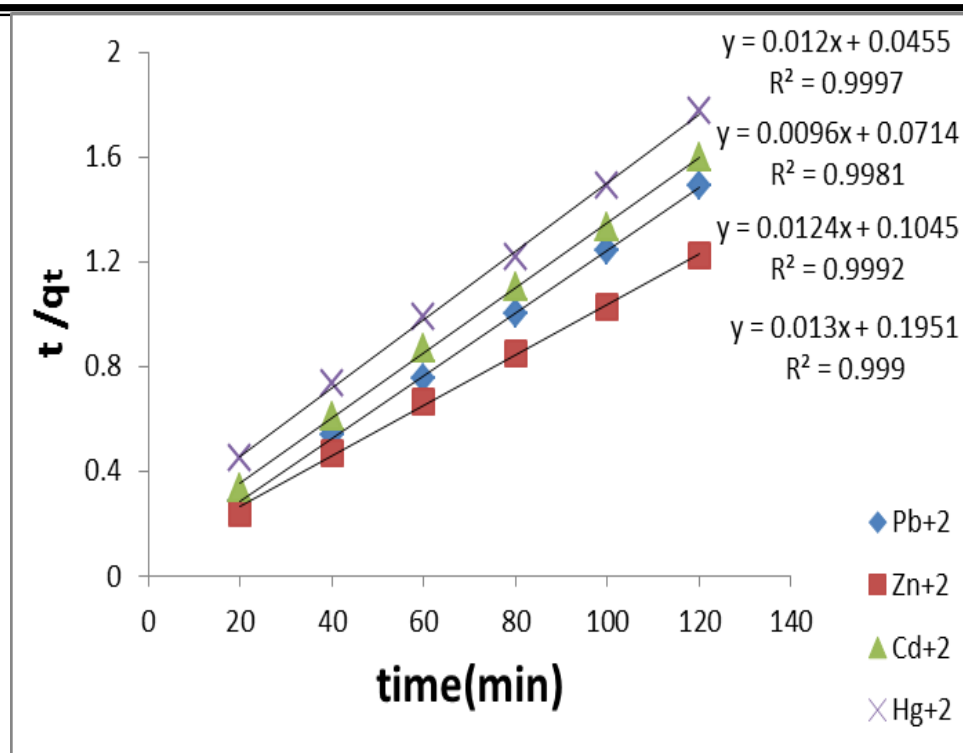


Figure (12). Pseudo second order kinetic plot for the adsorption of Hg^{+2} , Cd^{+2} , Pb^{+2} , Zn^{+2} ions on to HQFH.

Table 6. comparison of the first-order ,Second –order ,calculation and experimental (q_e) values for adsorption of heavy metal

Metal ions	First- order kinetic model				Second –order kinetic model			
	$q_{e.exp.}$ (mg/l)	K (min^{-1})	$q_{e.calc.}$ (mg/l)	R^2	$q_{e.exp.}$ (mg/l)	K (g/mg.min)	R^2	$q_{e.calc.}$ (mg/l)
Pb^{+2}	80.34	0.0632	62.28	0.929	80.34	0.00316	0.9997	83.33
Zn^{+2}	98.25	0.0373	50.97	0.849	98.25	0.00129	0.9981	104.16
Cd^{+2}	75.14	0.0489	66.75	0.861	75.14	0.00147	0.9992	80.64
Hg^{+2}	67.5	0.0537	100.32	0.941	67.5	0.00086	0.9990	76.92

The linear regression values obtained from the first-order kinetic model were lower than the second –order model and the q_e values acquired from the Pseudo First order sorption kinetics were contrasted with the experimental values. But in the case of Pseudo Second order model, the linear regression values were considerably higher(correlation coefficient $R^2 > 0.99$), and also the calculated q_e values agreed better with the experimental data.

Conclusion

This study suggest that the copolymer of 8-hydroxyquinoline-furfural-hydroquinone is an effective adsorbent for removal of Hg^{+2} , Cd^{+2} , Zn^{+2} , Pb^{+2} from its aqueous solution. The Adsorption increased with increase in pH. The adsorption of cationic metal ion is mainly influenced by the amount of negative charges in the solution. Maximum adsorption has been observed at a pH of 5 for Cd^{+2} ions and at a pH of 6 for Pb^{+2} , Zn^{+2} , Hg^{+2} ions. By comparing the correlation coefficient (R2 value) for pseudo first-order kinetic and pseudo second-order kinetics, experimental q_e value favored pseudo second -order and thus, the adsorption is assumed to be pseudo second order.

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الخلاصة:

استخدمت في هذه الدراسة تحضير بوليمر متشابك جديد (٨-هايدروكسي فورفورال - هايدروكينون) لامتزاز وإزالة العناصر الثقيلة (الزئبق, الرصاص, الزنك وكاديوم الثنائي) في محاليلها المائية. وتم استخدام تقنية فوق البنفسجية لمعرفة كمية الامتزاز والعوامل المؤثرة على عملية الامتزاز من الدالة الحامضية ومعامل التوزيع وتأثير درجة الحرارة ودراسة الدوال الترموديناميكية. اوضحت النتائج ان اعلى كمية امتزاز كانت في ايون (Zn^{+2}) والتي كانت $98,9\%$ و $80,2\%$ و $75,3\%$ (Cd^{+2}) و $67,9\%$ (Hg^{+2}). وبينت النتائج الترموديناميكية ان التفاعل باعث للحرارة (exothermic) وقيمة الطاقة الحرة لكبس سالبة وتلقائي. ومن خلال الدراسة وجد ان حركية الامتزاز تتبع مرتبة التفاعل من الرتبة الثانية وذلك من ملاحظة القيمة العالية لمعامل الارتباط والتوافق بين النتائج التجريبية والمحسوبة لقيم كمية الامتزاز (qe) عند الاتزان. واثبت النتائج انطباق معادلة ايزوثيرم فريندلش لامتزاز بمعامل ارتباط اعلى من معادلة لانكماير.