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Effect of Chain Length of Aliphatic Amines Immobilized on a Magnetic Glycidyl Methacrylate Resin towards the Uptake Behavior of Hg(II) from Aqueous Solutions

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Abstract: Magnetic resin particles with magnetite (Fe_3O_4) core and glycidyl methacrylate/divinylbenzene resin shell were prepared. The core-shell particles obtained were immobilized with ethylenediamine, diethylenetriamine, or tetraethylenepentamine to give resins with names, R-1, R-2 and R-3, respectively. These resins showed good magnetic properties and could be easily retrieved from their suspensions using an external magnetic field. The uptake values of resins towards Hg(II) were found to be 2.1, 3.2, and 4.8 mmol/g for resins R-1, R-2, and R-3, respectively. The effect of chain length as well as the content of amine sites on the uptake behavior was studied at different temperatures. The values of kinetic and thermodynamic parameters of the uptake process were reported. These values reflected the effective role of amine type and chain length on both the rate of uptake and the maximum capacity of the resins. Moreover, the column studies showed that the longer the amine chain was the shorter was the critical bed height.

Keywords: Magnetic resins, mercury, adsorption, glycidyl methacrylate, amine resins

INTRODUCTION

Polymer micro spheres with core-shell morphology combining an organic polymer matrix and inorganic nanoparticles possess unique properties of

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both systems (1, 2). Growing attention is being given to magnetic nano- and micro spheres due to their potential applications as magnetic inks and fluids, recording media, isolation of environmentally hazardous materials from waste streams, or in biology and medicine (3, 4). Extensive efforts have recently been made towards the development of new technologies for separation of health hazards such as radioactive and toxic heavy metal ions from waste streams. One of the most effective and economic methods is the use of magnetic chelating resins. These types of resins have high capability and selectivity in the removal of contaminants due to their chemical nature. They can also be easily removed after loading from the waste stream using a magnet (5). Glycidyl methacrylate resins and their magnetic forms have various applications in the field of separation of hazardous species from waste stream, microbiology, and medicine (3, 6–10). In our lab, modified glycidyl methacrylate resins with different hydrophilic/hydrophobic cross-linker, different functionalities, and different embedded metal oxide were prepared and investigated towards the interaction with different metal ions in their aqueous solutions (11–19). On continuity of our program of modification, the present work is directed to immobilization of magnetic glycidyl methacrylate resin by different aliphatic amines with different chain length and amino group content. The effect of amine type (different chain length and amine group content) on the kinetics of the adsorption process as well as the maximum capacity of the resins towards Hg(II) in aqueous solution has also been investigated.

EXPERIMENTAL

Chemicals

Glycidyl methacrylate (GMA), divinylbenzene (DVB, 55%), benzoyl peroxide (Bz_2O_2), ethylenediamine, diethylenetriamine, and tetraethylenepentamine were Aldrich products. All the other chemicals were of analytical grade and were used as received. $FeSO_4 \cdot 7H_2O$, $FeCl_3 \cdot 6H_2O$, and $HgCl_2$ were used as sources for Fe(II), Fe(III), and Hg(II), respectively.

Preparation of Magnetite

Magnetite was prepared following the modified Massart method (20). A 250 mL (0.2 M) of Fe(III) solution was added with stirring to a freshly prepared 250 mL (1.2 M) of Fe(II) solution. A 200 mL of ammonia solution (30%) was suddenly poured to the previously prepared Fe(III)/Fe(II) solution while vigorous stirring was going on. A black precipitate was formed and was allowed to crystallize for 30 min with stirring. The precipitate was washed with deoxygenated water (water was boiled to repeat any gases, then bubbled with nitrogen gas) under magnetic decantation until the acidity

of suspension became below pH 7.5. The precipitate was dried at ambient temperature to give a black powder.

Preparation of Magnetic GMA/DVB Resin

Resin was prepared through polymerization of GMA in the presence of 55% DVB as a cross-linking agent with a molar ratio of 9.5:0.5. The requisite volumes of DVB and GMA were mixed well with 0.1 g Bz_2O_2 (initiator) until complete dissolution of the Bz_2O_2 . One milliliter isopropyl alcohol and 12.6 mL cyclohexane were mixed and added to the former solution. The contents were then poured into a flask containing 73 mL of 1% polyvinyl alcohol and 1 g of the dried magnetite. The mixture was refluxed for 6 h on a water bath at 75–80°C with continuous stirring. A heavy grayish precipitate was formed, filtered off, subsequently washed with distilled water and methanol, and dried in air.

Immobilization of Magnetic Resin with Amine

The resin obtained in the previous step was loaded by ethylenediamine, diethylenetriamine, or tetraethylenepentamine to give R-1, R-2, and R-3 resins, respectively. One gram of the resin and 4 g of the corresponding amine were dissolved in 12 mL DMF. The reaction mixture was heated for 72 h at 75–80°C in an oil bath. A heavy yellowish gray product was formed, filtered off, washed with distilled water and then methanol, and dried in air.

Estimation of Amino Group Content

The concentration of the amino group was estimated using the volumetric method (11). Forty milliliters of 0.05 M HCl solution was added to 0.1 g resin and conditioned for 15 hrs on a shaker. The residual concentration of HCl was estimated through the titration against 0.05 M NaOH solution and phenolphthalein as indicator. The number of moles of HCl interacted with amino groups and consequently the amino group concentration (in mmol/g of resin) was calculated by the following equation

$$\text{Concentration of } (-\text{NH}_2) \text{ group} = \frac{(M_1 - M_2) \times 40}{0.1} \text{ (mmol/g of resin)} \quad (1)$$

where M_1 and M_2 are the initial and final concentrations of HCl in mol/L.

Adsorption Measurements using the Batch Method

A stock solution (0.01 M) of Hg(II) was prepared by dissolving an appropriate weight of pure metal salt in distilled water. A stock solution (0.01 M) of EDTA was prepared in distilled water and was standardized against a standard solution of $MgSO_4 \cdot 7H_2O$ using Eriochrome Black-T (EBT) as an indicator.

The variation of uptake of Hg(II) by resins as a function of time was carried out by shaking 0.1 g of resin with 100 mL of Hg(II) solutions of concentrations of 2, 5 or 7 mM at $pH = 5.1$ (natural) and $22^\circ C \pm 2$. Five milliliters of the supernatant (free of the suspended solid) were taken at different time intervals where the concentration of the metal ion in the supernatant was determined using 5×10^{-3} M EDTA in a buffered solution of pH 4 (acetic acid/sodium acetate) using 4-(2-pyridylazo) resorcinol indicator (PAR).

Adsorption isotherms were obtained at $pH = 5.1$ (natural) and at different temperatures. A 0.1 g resin was conditioned with 100 mL Hg(II) solution with different concentrations for 3 h at $pH = 5.1$ and at 25, 35, 45, or $55^\circ C$ using a Vibromatic-384 shaker at 300 rpm. Five milliliters of the solution were taken at the end of the experiment where the residual concentration of metal ion was determined.

Column Experiments

Column experiments were performed in a plastic column (length 3.6 cm, diameter 1 cm). A small piece of filter paper was placed at the bottom of the column, then a known quantity of the resin under study was placed in the column. A metal ion solution with initial concentration of 5 mM was allowed to flow downward through the column at a specific flow rate. Samples were collected from the outlet of the column at different time intervals and analyzed for metal ion concentration. The experiment was terminated when the concentration of the metal ion at the outlet of the column equals the initial concentration ($M_1 = M_2$).

Elution Experiments

The column was charged with the resin (bed height = 2.4 cm) and resin particles were loaded with Hg(II) ions of initial concentration $C_o = 5$ mM at a flow rate of 2 mL/min. The elution studies were carried out using 0.1 M EDTA or 10% acidified thiourea solution obtained by dissolving 10% thiourea by weight in 0.1 N HNO_3 . The Hg(II) ions were eluted from resin particles following by washing with distilled water till the down flow becomes free of Hg(II) ions. The efficiency of the elution was calculated from the ratio of the uptake capacity in the second run to that obtained in the first run.

RESULTS AND DISCUSSION

The stability of the investigated resins against acid and alkali was examined by soaking in 0.5 M HNO₃ or 0.1 M NaOH for 24 h. The stability of the resins was checked by measuring the total uptake of Hg(II) by the resin before and after the acid or base treatment. The resins showed good stability in HNO₃ or NaOH. They also showed a good durability towards successive runs of adsorption/desorption of Hg(II). The resins were easily collected from aqueous media using an external magnetic field.

Batch Experiments

Figure 1 shows the adsorption isotherms of the uptake of Hg(II) at pH 5.1 by resins R-1, R-2, and R-3 at 25, 35, 45, and 55°C, respectively. Clearly, at all temperatures the uptake increases as the chain length of the amine increases, i.e. R-3 > R-2 > R-1. For example, at 25°C, the values of the maximum uptake were 2.1, 3.2, and 4.8 mmol/g for R-1, R-2, and R-3, respectively. Moreover, the values of the maximum uptake of resins are related to their amine group content of 7.5, 11.2, and 17.1 mmol/g for R-1, R-2, and R-3, respectively. The uptake mechanism may be explained by a complex formation between Hg(II) and amine sites on the resin (12, 17).

The adsorption data of Hg(II) on the three resins at different temperatures were analyzed according to the Langmuir adsorption model (14)

$$\frac{C_e}{q_e} = \frac{C_e}{q_s} + \frac{1}{K_{ads}q_s} \quad (2)$$

where C_e is the equilibrium concentration of the metal ion (mM), q_e is the adsorbing capacity at equilibrium (mmol/g), q_s is the maximum capacity (mmol/g), and K_{ads} is the binding constant. Plotting of C_e/q_e vs C_e gave straight lines, indicating that the adsorption complies with the Langmuir isotherm as shown in Fig. 2. Each straight line has intercept and slope values equal to 1/K_{ads}q_s and 1/q_s, respectively. The values of q_s and K_{ads} for the three resins at different temperatures were reported in Table 1. The calculated values of q_s are comparable with those obtained experimentally. At the same temperature, the binding constant (K_{ads} value) follows the order R-3 > R-2 > R-1. This sequence may be attributed to the increased chelating power towards Hg(II). Table 1, also shows that both q_s and K_{ads} values decrease as the solution temperature increases. The observed decrease of K_{ads} values may be attributed to the bond weakening due to the increased vibrational energy with temperature (15, 18, 19). On the other hand, the observed decrease in q_s values may be related to the exothermic nature (negative ΔH° values) of adsorption reaction (Table 2). Plotting of binding constant (K_{ads}) against the temperature was done according to the

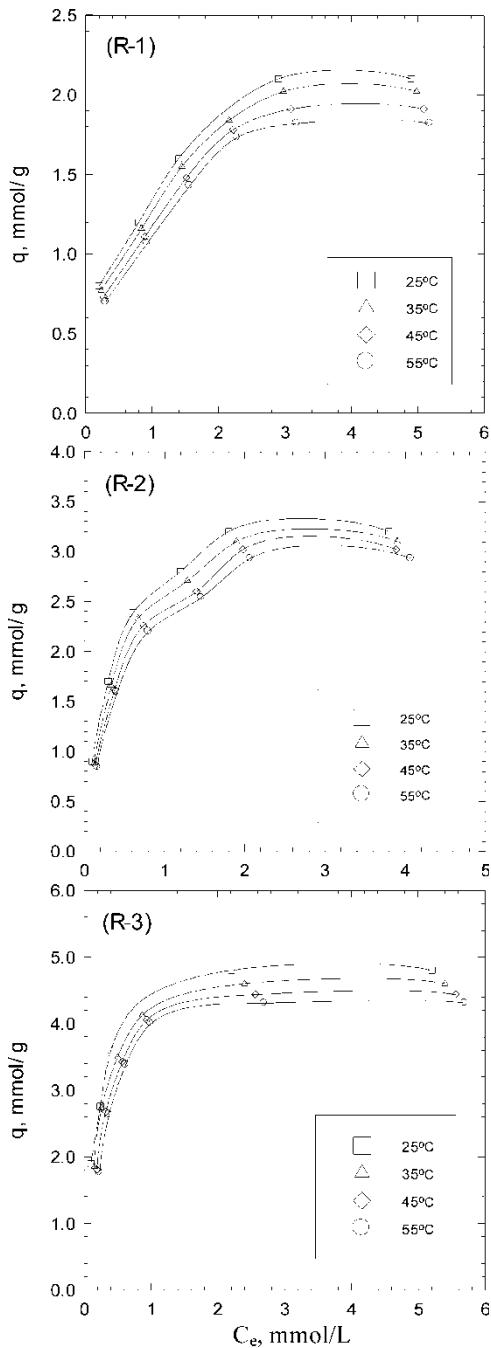


Figure 1. Adsorption isotherms of Hg(II) using the resins R-1, R-2, and R-3 at different temperatures.

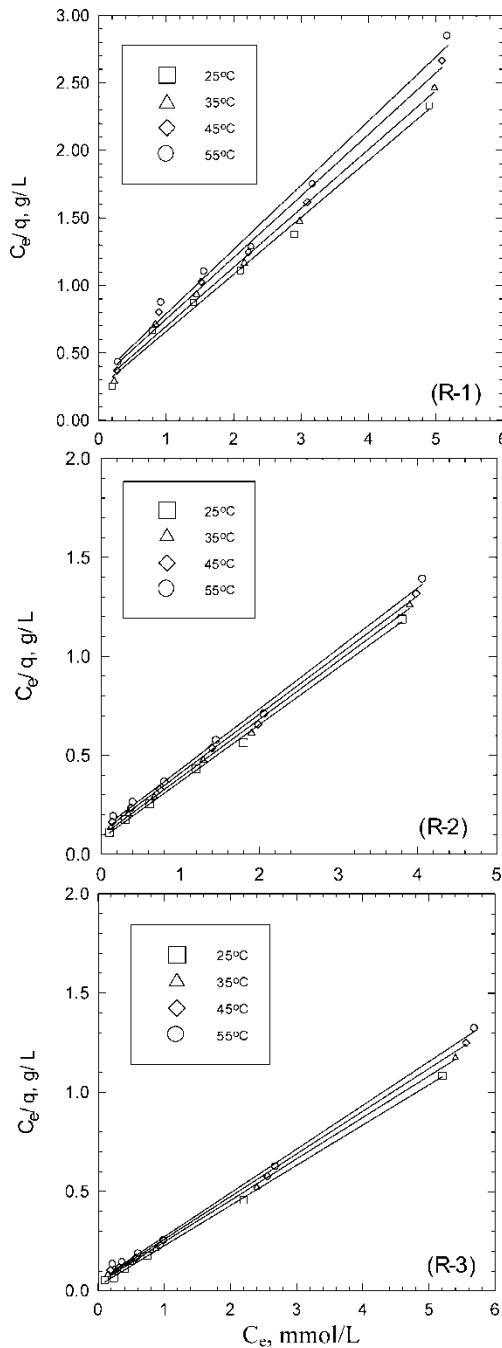


Figure 2. Langmuir model for Hg(II) using R-1, R-2, and R-3 at different temperatures.

Table 1. Langmuir parameters for the adsorption of Hg(II) on resins at different temperatures

Temp., (°C)	K_{ads} (L/mmol)	R-1			R-2			R-3		
		q_s , mmol/g.		R^2	q_s , mmol/g.		R^2	q_s , mmol/g.		R^2
		Calc ^a	Exp.		Calc ^a	Exp.		Calc ^a	Exp.	
25	1.78	2.37	2.10	0.991	3.68	3.47	0.998	7.64	4.95	0.999
35	1.67	2.29	2.02	0.992	3.20	3.39	0.997	5.82	4.78	0.999
45	1.55	2.19	1.91	0.991	2.71	3.35	0.997	4.83	4.65	0.998
55	1.51	2.10	1.82	0.989	2.46	3.28	0.996	4.14	4.53	0.998

^aCalculated from Langmuir plots.

Table 2. Thermodynamic parameters for the adsorption of Hg(II) on resins

Resin	ΔH° (kJ/mol)	ΔS° (J/mol·K)	ΔG° (kJ/mol)
R-1	-4.60	47.00	-18.56
R-2	-11.33	30.00	-20.36
R-3	-15.50	22.00	-22.06

van't Hoff equation

$$\log K_{\text{ads}} = \frac{-\Delta H^\circ}{2.303RT} + \frac{\Delta S^\circ}{2.303R} \quad (3)$$

where R is the universal gas constant (8.314 J/mol·K), T is the absolute temperature (Kelvin), and ΔH° and ΔS° are enthalpy and entropy change of adsorption process. Plotting $\log K_{\text{ads}}$ against $1/T$ gives a straight line with slope and intercept equal $-\Delta H^\circ/2.303 R$ and $\Delta S^\circ/2.303 R$, respectively. The values of ΔH° and ΔS° obtained were used to calculate the Gibbs free energy of adsorption (ΔG°)

$$\Delta G^\circ = \Delta H^\circ - T\Delta S^\circ \quad (4)$$

The values of ΔH° , ΔS° , and ΔG° are given in Table 2. The negative values of both ΔH° and ΔG° indicate an exothermic nature and spontaneity of the adsorption process, respectively. The observed positive value of ΔS° indicates more randomness state due to the liberation of water molecules during the chelation process. The values of ΔG° follow the sequence R-3 > R-2 > R-1. This order may be attributed to the increased efficiency of interaction of Hg(II) with the active sites.

The kinetics of the uptake process was examined by studying the change of uptake as a function of time. Figure 3 shows the uptake of Hg(II) by resins as a function of time at pH 5.1 and 25°C. At all time intervals, the rate of the uptake follows the order R-3 > R-2 > R-1. For all resins the uptake increased sharply over 30 min, and after that it increased slowly till reaching an equilibrium within 2 h. To study the effect of the chain length of amine on the rate of adsorption, the time/adsorption data obtained in Fig. 3 were treated according to pseudo first order kinetics model

$$\log(q_e - q_t) = \log q_e - \left(\frac{K}{2.303} \right) t \quad (5)$$

where q_e is the concentration of Hg(II) at equilibrium, q_t is the concentration of Hg(II) at time, t, K is the overall rate constant (min^{-1}). Figure 4 shows the plotting of $\log (q_e - q_t)$ versus t which gives straight lines with good fitting to the pseudo first order model for the three resins. From the values of slope

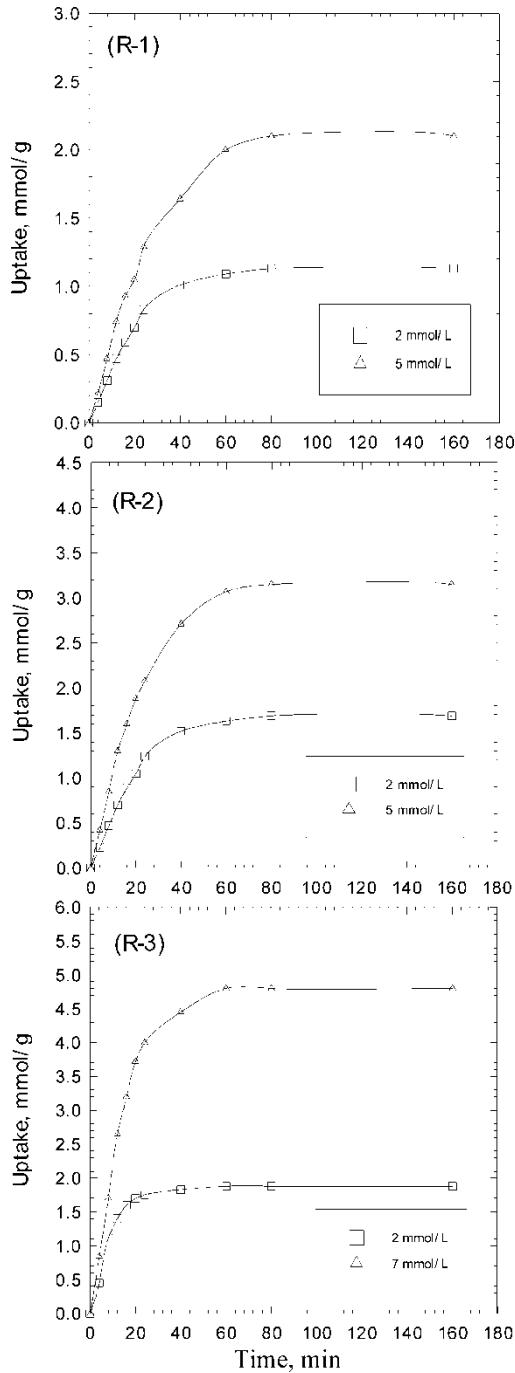


Figure 3. Uptake of Hg(II) as a function of time using R-1, R-2, and R-3.

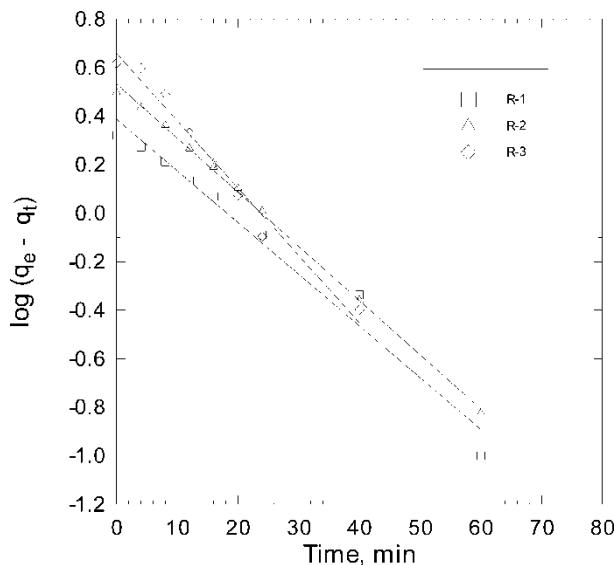


Figure 4. Pseudo first order data for the adsorption of Hg(II) on resins R-1, R-2, and R-3.

($K/2.303$) and intercept ($\log q_e$) of these lines, the values of overall adsorption rate constant (K) and equilibrium capacities (q_e) were calculated and reported in Table 3. The values of the forward and backward rate constants were also calculated from the combination of overall rate constant ($K = k_1 + k_2$) and the equilibrium constant ($K_{ads} = k_1/k_2$) as indicated in Table 3. On the other hand, the order of the adsorption reaction was experimentally determined from the determination of the reaction rate at two different initial concentrations (C_o) of Hg(II) for each resin. The values were found to be 1.31, 1.13, and 1.05 for R-1, R-2, and R-3, respectively. From the kinetic data given in Table 3 along with the reaction orders, the following points can be abstracted:

1. As the chain length of amine increases the reaction order better fits the first order kinetics ($n = 1$). This implies that the diffusion factor related to the textural properties of the resins becomes less effective on the reaction rate.
2. The values of q_e are generally comparable to those experimentally obtained. This indicates that the adsorption reaction proceeds mainly according to pseudo first order kinetics. It was also seen that the difference between experimental and calculated values of q_e for the same resin follows the order R-1 > R-2 > R-3. This order again confirms the effective role of the chain length on the reaction rate.

Table 3. Kinetic parameters for the adsorption of Hg(II) on resins

Resin	C _o (mM)	R ^{2a}	K = k ₁ + k ₂ (min ⁻¹) 10 ⁻²	K _{ads} = k ₁ /k ₂ , (L/mol)	k ₁ (min ⁻¹) 10 ⁻²	k ₂ (min ⁻¹) 10 ⁻⁵	q _e , mmol/g	
							Calc ^a	Exp.
R-1	5	0.974	4.90	1776	4.90	2.76	2.44	2.10
R-2	5	0.998	5.14	3683	5.14	1.40	3.40	3.20
R-3	7	0.978	6.66	7635	6.59	0.86	4.95	4.80

^aCalculated from first order model plotting.

3. The forward rate constant (k_1) follows the order R-3 > R-2 > R-1. This is also another evidence for the effective role of the chain length of the amine on increasing the rate of the adsorption reaction.
4. q_e value obtained of R-1 (2.1 mmol/g) is higher than that of the same resin free from metal oxide (1.73 mmol/g) (12). This indicates that the modified resin with embedded oxide has better adsorption properties.

Column Experiments

Effect of Flow Rate

The variation of relative concentration (C/C_o) of Hg(II) as a function of flow time for resins R-1, R-2, and R-3 at different flow rates and a fixed bed volume of 2 mL was investigated and the data obtained are reported in Table 4. For all resins and at the same bed height the total adsorption capacity (q_s) as well as the breakthrough point was reached within a shorter time at the higher flow rate compared to the lower one. In addition, at the same flow rate, the breakthrough points of Hg(II) was obtained at 95, 150, and 240 bed volumes for R-1, R-2, and R-3, respectively (Fig. 5). As indicated in Table 4, the efficiency of the removal follows the order R-3 > R-2 > R-1. This efficiency behavior is attributed to both the active site content (amine group) as well as the chain length.

Effect of Bed Height

Table 4 also reports the effect of the bed height on the breakthrough points of Hg(II). The bed height was chosen to be 1.2, 2.4, and 3.6 cm and a flow rate of 3 mL/min was used. The breakthrough time (t_b) and saturation time (t_s) were found to increase with increasing the bed height. This may be attributed to the availability of more binding sites for sorption of the metal ions. A bed depth service time (BDST) model is a simple model which verifies the linear relationship between the bed height (Z) and saturation time (t_s) of the column (21)

$$t_s = \frac{N_o Z}{C_o v} - \frac{1}{K_a C_o} \ln \left(\frac{C_o}{C_t} - 1 \right) \quad (6)$$

where C_t is the metal ion concentration at ($C_t/C_o = 99/100$), N_o is the total adsorption capacity (moles of adsorbate per liter of adsorbent), v is the linear velocity (cm/min), and K_a is the rate constant of adsorption (L/mole · min). The values of N_o were calculated from the slope of BDST plots. The values of both C_o and v should be kept constant during the column operation. The calculated values of N_o for the three resins were found to be comparable with the experimental values of q_s (Table 4). This

Table 4. Data of column studies for the uptake of Hg(II) on the resins at different bed heights and flow rates

Resin	Bed height, cm	Flow rate, ml/min	t_b , min	t_s , min	q_s , mmol/g ^a	Break-through capacity, mmol/g	Hg(II) removal efficiency, %	K_a , 1/mol · min	N_o , mmol/g	Z_o , cm	R^2
R-1	2.40	1	200	270	2.14	1.82	85.12	68.94	2.18	0.42	0.999
	2.40	2	95	140	2.13	1.73	80.94				
	2.40	3	57	95	2.09	1.55	73.88				
	1.20	3	27	55	2.15	1.45	67.59				
	3.60	3	94	135	2.12	1.70	80.04				
	2.40	1	310	400	3.23	2.82	87.30	75.15	3.18	0.25	0.999
R-2	2.40	2	150	200	3.23	2.73	84.51				
	2.40	3	103	134	3.23	2.46	76.06				
	1.20	3	44	73	3.12	2.36	75.73				
	3.60	3	155	190	3.22	2.79	86.50				
	2.40	1	490	570	4.77	4.36	91.42	82.71	5.00	0.15	0.999
R-3	2.40	2	240	280	4.73	4.27	90.37				
	2.40	3	160	190	4.73	4.18	88.47				
	1.20	3	73	103	4.73	3.82	80.72				
	3.60	3	243	283	4.89	4.42	91.83				

^aUsing the equation: $q_s = 0.5 (v_b + v_t) C_{o(Hg)}/g$ of resin; v_b and v_t are measured in L; $C_{o(Hg)}$ in mmol/L (22).

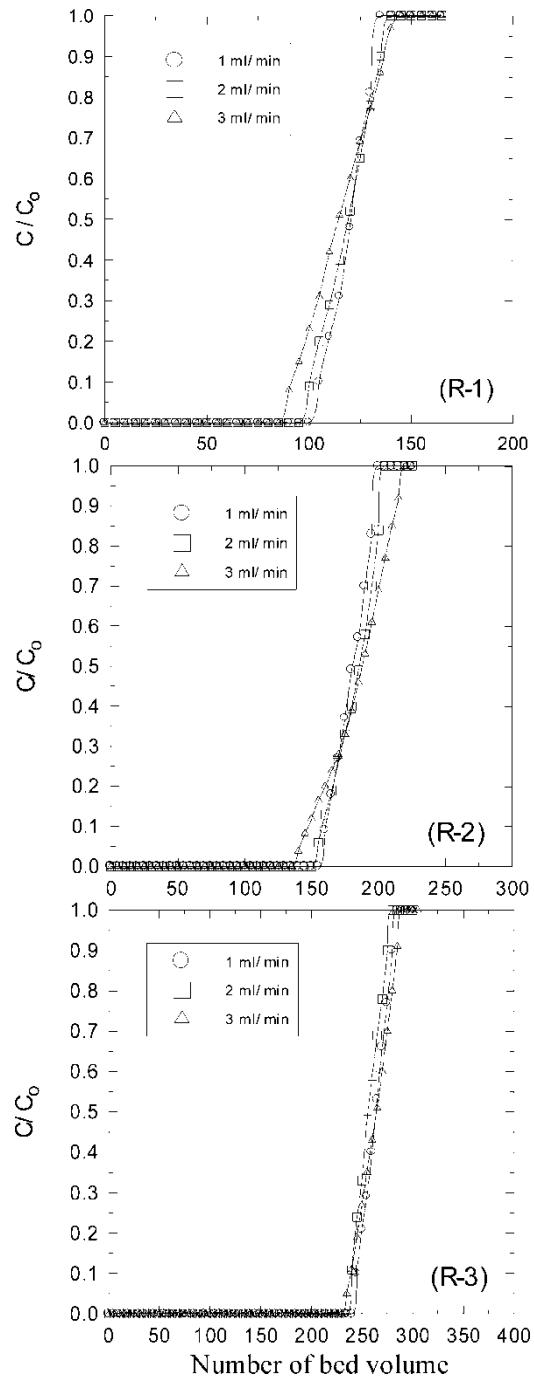


Figure 5. Effect of flow rate on the uptake of Hg(II) by resins R-1, R-2, and R-3.

indicates the validity of the BDST model for the investigated resins. K_a values were also calculated from the intercept of BDST plots and were given in Table 4. The values follow the order R-3 > R-2 > R-1 confirming the efficiency of the resins in the same order. On the other hand, the critical bed height (Z_o) can be calculated by setting $t_s=0$ in Eq. 6 and rearranging to get

$$Z_o = \frac{v}{K_a N_o} \ln \left(\frac{C_o}{C_b} - 1 \right) \quad (7)$$

where C_b is the breakthrough metal ion concentration (mol/L). The value of (C_o/C_b) is taken as ratio of (100/1). The value of Z_o was found to follow the order R-3 < R-2 < R-1 (Table 4). This trend may be related to the concentration of active sites as well as the chain length of the resin. The above arguments indicate that R-3 has a higher efficiency of Hg(II) removal relative to that of R-1 and R-2.

Elution Measurements

The data of elution studies were reported in Table 5. The efficiency of elution was verified using the following equation:

$$\text{Efficiency of elution}(\%) = \frac{\text{Total adsorption capacity in the second run}}{\text{Total absorption capacity in the first run}} \times 100\% \quad (8)$$

Generally, elution of Hg(II) from resins with EDTA was found to be more efficient than acidified thiourea. For the same type and amount of eluent, the elution efficiency follows the order R-1 > R-2 > R-3. This behavior is

Table 5. Parameters of elution process

Eluent	Volume, mL	Efficiency of elution, %		
		R-1	R-2	R-3
0.1 M EDTA	50	93.00	86.00	81.20
	75	97.60	92.00	87.30
	100	97.60	96.90	93.75
	150	97.60	96.90	96.25
	200	97.60	96.90	96.25
Acidified thiourea	50	83.00	80.00	77.00
	75	92.00	85.00	81.30
	100	95.23	91.60	87.30
	150	95.23	95.00	93.96
	200	95.23	95.00	94.79
	300	—	95.00	94.79

consistent with the values of the binding constant (K_{ads}) obtained and reflects the higher chelating power of R-3 relative to R-1 and R-2. All resins have a higher durability against sorption/desorption process for more than 5 cycles.

CONCLUSIONS

Glycidyl methacrylate resin with finely divided embedded magnetic Fe_3O_4 was prepared. The magnetic resin obtained was anchored with ethylenediamine, diethylenetriamine, or tetraethylenepentamine. The resins were tested towards the recovery of $\text{Hg}(\text{II})$ from its aqueous solutions at natural pH (5.1). Uptake values of 2.1, 3.2, and 4.8 mmol/g were recorded for R-1, R-2, and R-3, respectively. It is also seen that the textural properties of the resin becomes more pronounced on the rate of adsorption as the chain length of the amine moiety decreases. The column studies showed that R-3 with long chain length and high amine content has a short effective bed height and a higher rate of adsorption and therefore higher efficiency. The elution studies indicated that both EDTA or 10% acidified thiourea are efficient eluents. The elution efficiency for all resins is related to the binding constant of metal ion with the active sites on the resin.

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