

Mercuric Ions (II) Uptake From Aqueous Solutions by Chelating Resin Containing Pendant Multidentate Ligand

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ABSTRACT

The toxic nature and other adverse of the heavy metals lead to pollution and make it one of the most serious problems that affect certain sides of the environment. There are many heavy metal ions like the mercuric ions detected in industrial wastewaters originating from several factors such as from metal plating, mining activities, paint manufacture, etc. The above-mentioned ions isn't biodegradable and tends to be accumulated in living organisms, causing various diseases and disorders. Therefore, it must be removed from aqueous solution before discharge. In this paper, an experimental work it has been done regarding the optimal conditions of removing Hg (II) ions from water using chelating ion exchange resin namely XAD- & Pendant Multidentate Ligand Resin. The adsorption behaviour of mercury ions on XAD-& Pendant Multidentate Ligand Resin was studied as a function of the following variables: contact time, pH solutions, initial concentration of metal ions and resin dosage the adsorbent's maximum total adsorption capacity of for mercuric ions was: 0.82 mmol/g dry resin. The resin low affinity towards alkali and alkaline earth metals shows its use for samples that related to environment. The concluded results state that, XAD- & Pendant Multidentate Ligand Resin holds great potential for the purpose of removing mercuric ions from polluted wastewater

Introduction

No doubt that pollution is one of the most serious environmental problems which is caused by heavy metals, due to their toxic nature and other adverse effects [1]. Several heavy metal ions, such as cadmium, copper, nickel, lead, and mercury cause toxicity or death with less than a gram. These heavy metal ions can be detected in industrial wastewaters originating from so many factors like metal plating, mining activities, paint manufacture, etc.

The above-mentioned heavy metals are not biodegradable and tend to accumulate in living organisms, causing various diseases and disorders [2]. Their toxic effects include damage to the brain, kidneys and lungs [3]. Therefore, removing them from aqueous solution before discharge is highly recommended to avoid their damage. Poisoning by Mercury (hydrargyria or mercurialism) is a type of metal poisoning and a medical condition caused by exposure to mercury or its compounds. Mercury is a heavy metal occurring in several forms. Less than a

gram of these cause death. At zero oxidation of mercury Hg^0 exists as steam(vapor) or as liquid metal, its mercurial state Hg^+ exists as inorganic salts, and its mercuric state Hg^{2+} may form either inorganic salts or organo-mercury compounds; the three groups vary in toxic effects. Mercury is well-known for its extremely high toxicity and the serious threat to human life and natural environment. Mercury poisoning causes several diseases, including acrodynia (pink disease), Hunter-Russell disease, and Minamata syndrome [4-6]. Internationally, industry is forced to lessen down to tolerable level contents of heavy metal in both water and industrial wastewaters. United States Environmental Protection Agency (USEPA) established that the drinking water criterion for mercury is 2 $\mu g/L$ and the permitted discharge limit of wastewater for total mercury is 10 $\mu g/L$. Therefore, it is very important to diminish the residual mercury concentration at a level less than the safety limit for mercury ions from industrial wastewater, especially from drinking water [7]. For the removal of heavy metal ions from waste water, many conventional methods were employed like evaporation, electro-deposition, solvent extraction, reverse osmosis, membrane separation process and activated carbon

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adsorption [8]. However, these techniques have certain disadvantages such as higher operational cost, requiring additional chemical, high energy consumption and residual metal sludge disposable. Due to forbidding cost of these processes, Compared with other usual methods ion exchange provides advantages [9]. The uses of ion exchangers have been received with considerable attention [10]. Recently, a number of commercial ion exchangers like Amberlite IR-120, Amberlite IRC-748, Amberlite IRC-718, Lewatit TP-207 and a wide range of anion exchange resins with styrenic structures and chelating groups have been used to remove heavy metals from wastewater [11-18]. In this paper, an experimental work has been carried out regarding the optimal conditions of removing mercuric ions (II) from aqueous solutions using Chelating Resin Containing Pendant Multidentate Ligand.

Experimental

Reagents, Materials and Apparatus

Bis (salicylideneimiate) diethylenetriamin was prepared by the described procedure in the literature [19]. Dioxane and carbon disulfide were purchased from Merck Chemical Company (Germany). All used chemicals were of analytical grade. Mercury stock solution $1000 \mu\text{g mL}^{-1}$ was prepared by dissolving 1.6195 g of mercury nitrate (Analar grade) in deionized water acidified with 5mL of 5N HNO_3 to prevent hydrolysis and diluting to 1000 mL. A working solution for the experiments was freshly prepared from the stock solution. The working solutions with different concentrations of Hg (II) ions were prepared by appropriate dilutions of the stock solution immediately prior to their use. pH (3.5–6.5) was controlled by using acetic acid/sodium acetate buffer solutions below 3.0 with hydrochloric acid. The concentration of Hg (II) ions was determined spectrophotometrically by using diphenylthiocarbazone as spectrophotometric reagent. [20] ChromTech UV-1100) UV-Visible spectrophotometer was used for the spectrophotometric determination of metal ions in solutions. The pH measurements were made with (D-82362 digital pH meter, Weiheim, Germany). A mechanical shaker (SGM 3000100, Gallenkamp, Orbital Shaker) with an incubator, which had a speed of $200 \text{ strokes min}^{-1}$, was used for batch equilibration.

Preparation of the Chelating Resin(XAD-& Pendant Multidentate Ligand Resin)

The chelating resin was synthesized and purified by adapting the method as described in the literature.[21] Reaction of bis(salicylideneimiate) diethylenetriamine (46.7 g) with resin (1) (13.0 g) in refluxed dioxane (300 mL) for 48 h gave yellow resin (2). Produce: 24.4 g. Hydrolysis of (2) (23.1 g) with 6M HCl (500 mL) at 60°C for 12 h produced (3) in hydrochloride form. in the free amine form the resin was achieved by treatment with 2 M aqueous ammonia. Produce: 14.5 g. Resin (4) was obtained by stirring resin (3) (5.0 g) with carbon disulfide (15.2 g) and sodium ethoxide (13.4 g) in ethanol (100 mL) at 50°C for 12 h. Produce: 7.9 g. The resin was washed with deionized water, acetone, and vacuum dried. As a result the resin showed the spectroscopic and the analytical data identical to the literature [21].

Determination of Water Regain

Weighed amounts of the resin were kept soaked in deionized water for 24 h. Then, the resin was filtered by suction and dried by pressing between filter paper folds. Weighed amounts of the swollen resin were then dried at 90°C for 72 h and reweighed. The water taken up (in gram) by one gram of the dry resin was calculated. Water regain (%w/w) = Weight of water absorbed/ weight of dry resin x100.

Adsorption Studies

In all the adsorption studies, a 0.1 gm sample of the synthesized chelating resin was shaken with 25 mL of Hg (II) using 100 mL polyethylene bottles with a magnetic stirrer at room temperature. Determination of the metal ion concentration was carried out by allowing the insoluble complex to settle down, withdrawing an appropriate volume of the supernatant using a micropipette and then diluting to the linear range of the calibration curve for each metal. The adsorption capacities Q (mg/g XAD- & Pendant Multidentate Ligand Resin) were calculated by using the following equation:

$$Q = (C_0 - C_f) V/m$$

where C_0 and C_f are the initial and final concentrations (mg/L) of Hg(II) ion in the aqueous solution, respectively, V (L) is the volume of metal ion solution, and m is the added weight of XAD- & Pendant Multidentate Ligand Resin (g). The metal ion uptake was calculated as mole of Hg(II) ions/g dry

resin. Each study was performed at least in a triplicate. Metal uptake was examined at different time intervals and pH values.

Effect of Alkali and Alkaline Earth Metal Ions

To determine the interference of alkali and alkaline earth metal ions, diverse ions viz. Sodium, Potassium, Magnesium and Calcium ions influence on Hg (II) ions recovery from aqueous solution was studied. For this purpose, standard solutions of 50 mL of Hg (II) ions ($50 \mu\text{g mL}^{-1}$) containing manifold amounts of the foreign ions as interferences were analysed.

Preparation of Synthetic Seawater

The method was used to prepare synthetic seawater suggested by Muroi and Hamaguzhi. [22] The composition synthetic seawater used in this study is given below.

Salt	Concentration (g L^{-1})
NaCl	23.476
MgCl ₂	4.981
Na ₂ SO ₄	3.917
CaCl ₂	1.102
KCl	0.066
NaHCO ₃	0.192
KBr	0.096
H ₃ B ₃	0.026
SrCl ₂	0.024
NaF	0.003

Deionized water was used to dissolve the salts and diluted to 1 liter. The seawater thus prepared was spiked with aliquots of standard mercury nitrate solution in order to get an appropriate concentration of Hg (II) ions.

Concentration and Separation of Metal Ions

To concentrate the trace metal ions we used the batch equilibration process. Sample solutions (250-1000 mL) containing $0.1 \mu\text{g mL}^{-1}$ metal ion were adjusted to the optimum pH of adsorption with buffer solution. The solution was then shaken with 0.1 gm of resin for about 60 min. The sorbed Hg (II) ions were eluted with 3M hydrochloric acid (10 mL) and the concentration of Hg (II) ions in the eluent was determined. Hg (II) ions Extraction from other metal ions that are divalent in the dual blends were achieved. A 25 mL solution containing Hg (II) ions ($5 \mu\text{g mL}^{-1}$) and these ions was shaken with 0.1 gm of resin at pH 5.5. Separation of Hg (II) ion was carried out by a column method. A glass column with a length of 6 cm

and an id of 0.5 cm was used for separation purposes. The dry resin (0.5 g) was loaded into the glass column, soaked in the buffer solution and the metal ion solution was passed through the column at a flow rate of 0.5 mL min^{-1} . The adsorbed metal ion was eluted from the resin as mention above. Pre-concentration of Hg (II) ions from synthetic seawater using XAD- & Pendant Multidentate Ligand Resin was also carried out by column method. Pre-concentration was done at pH 5.5, flow - rate 0.5 mL/min .

Hg (II) ions that are selectively sorbed, were eluted with several bed-volumes of 3M hydrochloric acid at 1 mL/min flow-rate.

Results and Discussion

As it is mentioned above, the chelating ion exchanger with multidentate ligands based on diethylenetriamine into polystyrene beads was synthesised according to the procedure as described by Suzuki, *et al.* [21] in Figure 1. There is a description to the preparative scheme of the chelating resin. Also in the same figure there is a description of the structure that the Schiff base bound resin **2** is assumed to have.

In the above-mentioned structure direct N-alkylation has occurred. Resins **2**, **3**, and **4** gave all the spectroscopic and the analytical data identical to the literature. [21].

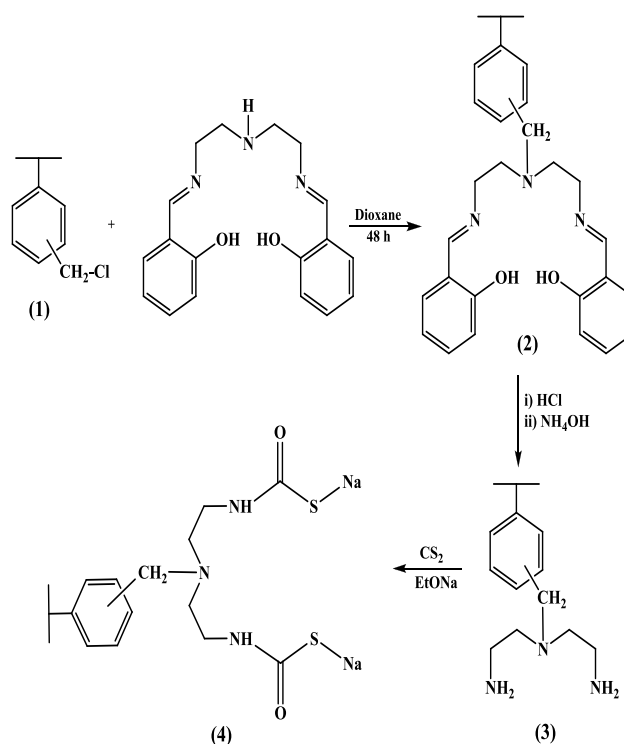


Figure 1. Synthesis of Resin

The water-regain was 0.40 g water/g dry resin. The conclusion states that the resin was very steady on the sequential treatment with 2M HCl and 2M NaOH solutions. The stability was examined by change of the hydrogen-metal exchanged capacity and IR spectrum, which indicated that there was no change in the bands of the synthesised resin after treatment with acid and base solutions. The total adsorption capacity of the resin (millimoles of metal ion taken up by 1 g of dry resin) is an important factor to determine and is the quantity of resin required to remove quantitatively a specific metal ion from the solution. also, one important factor in metal ion removal from aqueous solutions has the most significant effect on the affinity of a resin for a particular ion is the pH, which can affect the adsorbent surface charge and the degree of ionization. In a preliminary experiment, the adsorption behavior of Hg (II) ions on XAD- & Pendant Multidentate Ligand Resin at different pH values in the range of 1 to 7 pH, has been examined by the batch method and the results are shown in Figure 2. This range of pH was selected to avoid the precipitation of hydroxides. At pH values below two, hydrogen ions are likely to compete with Hg (II) ions and at pH values above six, Hg (II) ions might precipitate.

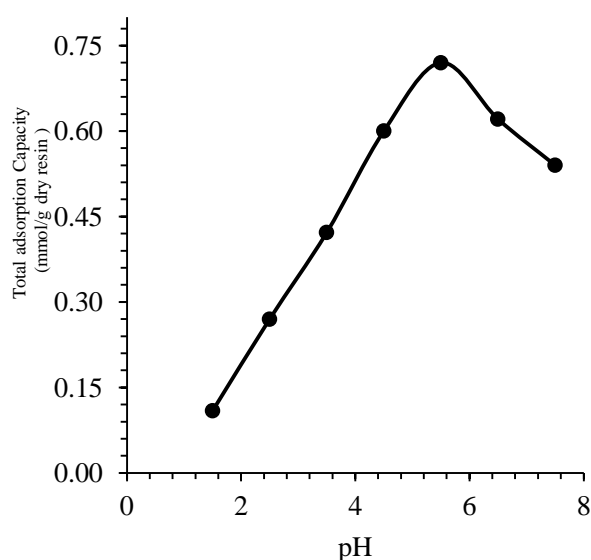


Figure 2. The Total adsorption capacity (T.C) for Hg (II) ions on XAD- & Pendant Multidentate Ligand Resin as a function of pH. Amount of resin, 0.1 g; temperature, 25 °C

At higher pH values (greater than six), the existence of OH⁻ ions produces the hydroxyl complexes of mercury Hg(OH)₂ [23]. It can be shown

from Figure 2, that in most of the cases the total adsorption capacity Hg (II) ions increases with increasing pH of aqueous solution from (1.5-5.5), reaching a limiting value, followed by a decrease in sorption, beyond the limiting value. At low pH, in the strong acidic media like pH=2, some of chelating atoms in the chelating site in the resin might be ionized with protons, so it will not be able to make coordination bonds with Hg (II) ions that lead to decreasing in chelating efficiency. Therefore, the total adsorption capacity of XAD- & Pendant Multidentate Ligand Resin will be decreased. As the pH of the solution increases, the number of protons dissociated from functional groups on the surface of XAD- & Pendant Multidentate Ligand Resin increases and thus more negative groups for complexation of mercury cations will be provided. Therefore, the electrostatic attraction increases between the mercury ions and the surface of adsorbent, which leads to get a higher total adsorption capacity. The optimum pH value, which corresponds to the maximum total adsorption capacity of Hg (II) ions, was observed at pH 5.5. From theoretical attitude, at pH = 6 the total adsorption capacity must be the best or it has the maximum value. This may be revealed that, pH=6 is very close to neutralization pH, therefore the other atoms which are close to the chelating site will be available to chelate with Hg (II) ions. At pH=4, the chelating site in the resin may be in its best steric shape or the chelating atoms in the resin may be more available to make more coordination bonds with Hg (II) ions that will lead to make the resin more effective or more able to remove the Hg(II) ions from the solution.

The effect of treatment time on the adsorption capacity is an important factor in evaluating the adsorption efficiency, which helps to determine the rate of maximum removal of solutes. For achieving the process of resin as being applied in a crammed pillar(column), the kinetics of the resin-metal interaction must be hasty. If the complexation is not adequately fast, concentration of the metal ion on a packed pillar is improbable because of the shortness of contact time between the resin and the solution. For cases like what is mentioned above, a batch method must be used, taking a large excess of resin and shaking it with metal solution for a longer period. For determining the time of equilibrium for Hg (II) ions under investigation. Kinetic experiments were carried out at optimum pH of adsorption for Hg (II) ions and

the results are plotted in Figure 3. It reveals that the adsorption percent of Hg(II) ions increases with contact time from 5 to 65 min. and then becomes almost constant up to the end of the experiment. It can be concluded that the binding of Hg(II) with XAD- & Pendant Multidentate Ligand Resin is high at initial stages and becomes almost constant after an optimum contact time of 65 min. The equilibrium total adsorption capacity was about 0.82 mmol/g dry resin for Hg(II). The optimum contact time correspond to the maximum total adsorption capacity of Hg(II) was observed to be about 65 min. Thus, equilibration of the resin with Hg (II) ions is sufficiently rapid, allowing the resin to be used in a packed column.

It's supposed that, the total adsorption capacity of mercury ions on XAD- & Pendant Multidentate Ligand Resin occurred according to two mechanisms: ionic exchange between hydrogen of chelating groups of resin and Hg(II) ions in solutions, and by internal diffusion via resin moieties. The first mechanism may be supported with the fact that, a considerable increase of acidity was noticed in solution after establishment of equilibrium. The other fact of ionic internal diffusion was confirmed by the rapid equilibrium between ions and resin, the chelating atoms in the chelating resin will be able to make coordination bonds with Hg(II) ions, and this ability will increase with the treatment time increasing. In addition, titration of released hydrogen ions in comparison with concentration of adsorbed ions by resin reveals that, the latter ions are in excess. The difference was attributed to presence of other adsorbed mechanisms than ionic exchange, such as internal diffusion and physical adsorption and so on.

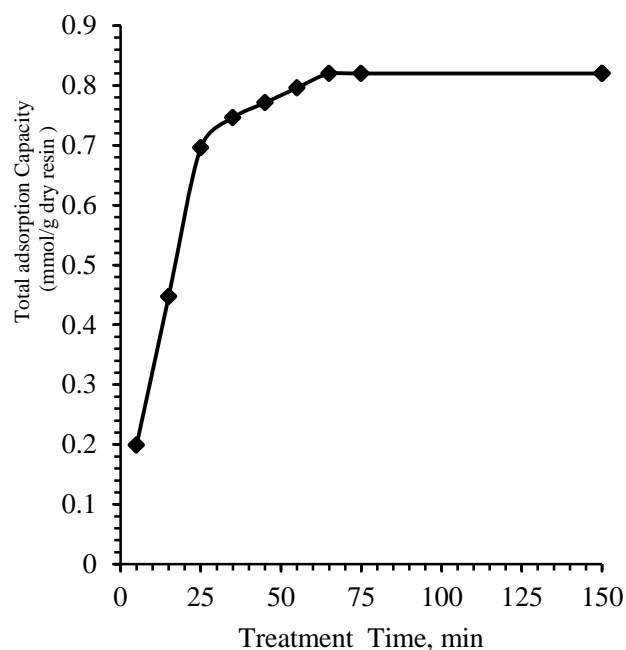


Figure 3. The Total adsorption capacity of Hg(II) ions on XAD- & Pendant Multidentate Ligand Resin as a function of time at pH 5.5. Amount of resin, 0.1 g; temperature, 25 °C

The effects of variation of resin dosage on Hg (II) ions removal by XAD- & Pendant Multidentate Ligand Resin was studied by using batch method at optimum pH. Resin amount was ranged from 0.015 to 0.150 g and equilibrated for 60 min at an initial Hg (II) ions concentration of 200 $\mu\text{g mL}^{-1}$. Fig.4 shows variation of Hg (II) ions percentage removal with resin dosage. It was found that the equilibrium concentration in solution phase decreases with increasing resin doses for a given initial Hg (II) ions concentration up to 0.100 g then adsorption capacity reaches almost a constant value. This concluded result was anticipated because for a fixed primary solute concentration, increasing adsorbent doses provides larger surface area and adsorption sites of the adsorbent. [17] For the quantitative removal of 200 $\mu\text{g mL}^{-1}$ Hg (II) ions in 25 mL, a minimum resin dosage of 0.12 g of XAD- & Pendant Multidentate Ligand Resin is required.

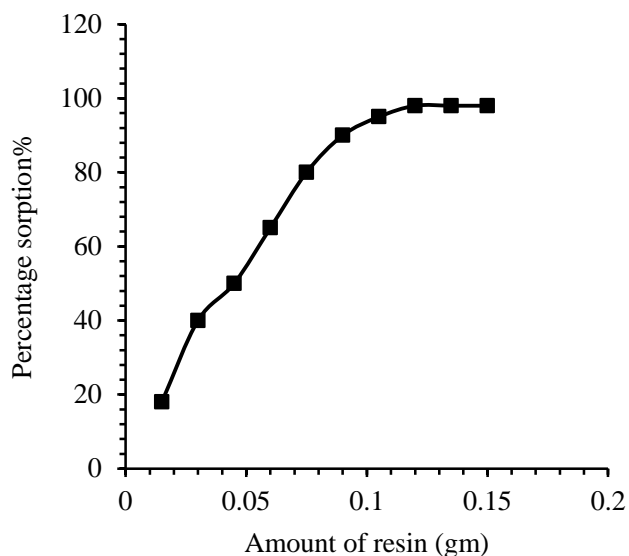


Figure 4. Effect of resin dosage on the adsorption of Hg (II) ions on XAD- & Pendant Multidentate Ligand Resin at pH 5.5. Hg (II) ions solution $200 \mu\text{g mL}^{-1}$, temperature, 25°C

Describing how solutes interact with the resin is not possible due to the crucial relation between the total adsorption capacity (T.C) and the primary concentration and so it is critical in optimizing the use of the resin. [24] The total adsorption capacity of Hg (II) ions on XAD- & Pendant Multidentate Ligand Resin is presented in Fig.5 as a function of the equilibrium concentration of Hg (II) ions at optimum pH for 60 min of contact time. per unit Hg (II) ions amount adsorbed mass of the resin that increased first with the concentration of the increasing primary metal ion as anticipated. Then reached a plateau value that represents saturation of the active groups that are available for interaction with interested metal ions, the resin was saturated at relatively low concentrations indicating strong binding for Hg(II) ions. When the initial concentrations of Hg (II) ions were less than $700 \mu\text{g mL}^{-1}$, the quantity of adsorption sites in XAD- & Pendant Multidentate Ligand Resin were vastly more than the amount of metal ion in an experienced solution. The isotherm of adsorption capacity deviates at high concentrations of Hg (II) ions. As the concentrations of metal ion increased, XAD- & Pendant Multidentate Ligand Resin adsorbed more metal ions and the polymer chains shortened. Hg (II) ions would be hard to diffuse toward the free chelating sites within XAD- & Pendant Multidentate Ligand Resin. in Figure 5 The graph plot clearly states a

vertical slope in the primary solution phase concentration range for pH 5.5. This is a desirable feature of a sorption system as it shows well-organized Hg (II) ions removal by XAD- & Pendant Multidentate Ligand Resin in mercury diluted solution.

The recovery of metal ion from the solution is not affected by the presence of manifold amounts of alkali and alkaline earth metals. The concluded results that shown in Table 1 reveals Hg (II) ions recovery rates in the existence of excess these foreign ions. This makes the suitability of this resin for trace concentration of Hg (II) ions from natural samples, which contain large concentrations of alkali and alkaline earth metals, e.g., seawater, pond water, etc. As well, the separation of trace amount of Hg (II) ions in various divalent metal ions existence was performed by both batch and column methods. Mercuric ions are extractable from the two elements Nickel and Copper as they are not retained on the resin at pH 5.5 (Tables 2 and 3). The resin thus reacts selectively with Hg (II) ions.

The selectivity can best be explained by noticing that Hg(II) ion is a very soft acid and the sulfur atom as terminal thiol or as thioether, is a very soft base, and a coordinate bond should form between them readily other metal ions are considerably "harder" and don't interact so strongly with soft bases. [25] The pillar (column) method was favoured over the batch method for the XAD- & Pendant Multidentate Ligand Resin because it is better for enrichment of the metal ions as the sorption is more complete and subsequent elution is easier.

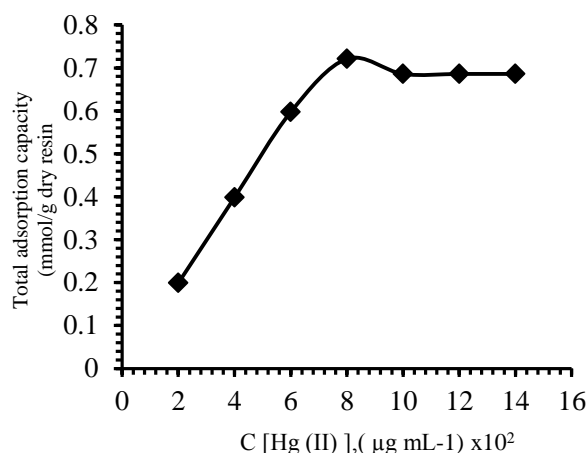


Figure 5. The total adsorption capacity of Hg (II) ions on XAD- & Pendant Multidentate Ligand Resin as a function of initial metal concentration at pH 5.5. Amount of resin, 0.1 g; temperature, 25°C , time=60min

Table 1. Effect of diverse ions

Interfering ions (50 µg mL ⁻¹)	Recovery (%)
	Hg(II) ions
Ca ⁺²	99.2
Mg ⁺²	98.6
K ⁺¹	100.2
Na ⁺¹	99.7
Sr	98.2

Table 2. Extraction of Hg(II) ions from other divalent metal ions performed at pH 5.5 [amount of Hg (II) ions taken, 5 µg mL⁻¹]

Metal ion	Amount of metal added/ µg mL ⁻¹	Amount of Hg (II) recovered*/ µg mL ⁻¹	Metal ion recovered* µg mL ⁻¹
Ni(II)	5.0	4.98	4.88
	10.0	5.01	9.65
	20.0	4.90	19.80
Cu(II)	5.0	4.96	4.94
	10.0	4.92	9.26
	20.0	4.85	20.02

* Batch method

Table 3. Extraction of Hg (II) ions from other divalent metal ions performed at pH 5.5 [amount of Hg(II) taken, 5 µg mL⁻¹]

Metal ion	Amount of metal added/µg mL ⁻¹	Amount of Hg (II) recovered*/µg mL ⁻¹	Metal ion recovered* µg mL ⁻¹
Ni (II)	20	4.96	19.85
Cu (II)	20	4.89	19.72

*Column

But the column method is more time consuming than the batch method. Preconcentration of Hg(II) ions on the resin was also carried out and the results are shown in (Table 4). Hg(II) ions could be enhanced up to 100 times with XAD- & Pendant Multidentate Ligand Resin.

Synthetic sea water spiked to different concentration of Hg(II) ions was used i.e., 0.02, 0.2 and 1.0 µg mL⁻¹ and 100 mL of the spiked test solutions of pH 5.5 were separately passed through the column conditioned as described above at a flow rate of 0.5 mL min⁻¹. The sorbed Hg(II) ions on the column was desorbed with 3M HCl. At lower concentrations, the recovery became less quantitative perhaps due to relatively high losses due to adsorption on the glass surface [26].

Table (5) gives the results of pre-concentration of Hg(II) ions with quantitative recovery. Satisfactory recovery was obtained using 3M HCl as eluent.

Table 4. Preconcentration of Hg (II) ions (amount of metal ion taken 0.1 µg mL⁻¹)

Sample volume/ mL	Amount of Hg (II) added/µg mL ⁻¹ (influent)	Amount of Hg (II) ions (effluent)	Hg (II) ions recovered* µg mL ⁻¹ (Eluent)
250	0.1	0.00	2.48
500	0.1	0.00	4.94
1000	0.1	0.00	9.89

*Column

Table 5. Pre-concentration of Hg(II) ions from synthetic seawater using XAD- & Pendant Multidentate Ligand Resin

Concentration of Hg (II) ions in sea water (µg mL ⁻¹)	Amount of Hg(II) ions loaded (µg) *	Eluent volume (mL) **	Amount recovered (µg)	Percentage recovery
1.000	100	30	97.80	97.80
0.200	20	30	19.20	96.00
0.020	2.0	20	1.9	95.00

* 100 ml of seawater passed through the column

** Mean of three values

An investigation on the adsorption properties of XAD- & Pendant Multidentate Ligand Resin towards Hg(II) in aqueous solutions, as a function of the following variables: contact time, pH solutions, initial concentration of metal ions, resin dosage show that the synthesised chelating resin (XAD- & Pendant Multidentate Ligand Resin) has strong complexing properties forming heterocyclic chelates with Hg (II) ions depending on the pH of the mobile phase. Equilibrium studies showed that, the maximum total adsorption capacity of adsorbent for Hg (II) ions was: 0.82 mmol/g dry resin, which shows that, XAD-& Pendant Multidentate Ligand Resin holds great potential for removing Hg (II) species from several sources. XAD- & Pendant Multidentate Ligand Resin has been found beneficial for concentration of trace metals and their separation from aqueous solutions. Fast equilibration of Hg (II) ions on XAD- & Pendant Multidentate Ligand Resin makes it applicable for the conventional column method. The resin low affinity towards alkali and alkaline earth metals shows its use for samples that related to environment. The resin also has the ability of retaining quantitatively Hg (II) ions from replicated seawater.

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