



# Removal of Pb(II) from aqueous solution using ethylene diamine tetra acetic acid-Zr(IV) iodate composite cation exchanger: Kinetics, isotherms and thermodynamic studies



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## ABSTRACT

EDTA-Zr(IV) iodate was prepared via the incorporation of aqueous solution of EDTA into the inorganic precipitate of Zr(IV) iodate. Distribution coefficient values for different metals showed that EDTA-Zr(IV) iodate had the highest adsorption capacity for Pb(II). The practical applicability of EDTA-Zr(IV) iodate was explored by separating Pb(II) metal from a synthetic mixture of metal ions. Dependence of adsorption on contact time, pH of the solution, exchanger dose and temperature was studied to achieve the optimum conditions. Kinetic studies showed better applicability for pseudo-second-order model. Thermodynamic parameters viz. – entropy change, enthalpy change and Gibb's free energy change were also calculated.

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## Introduction

The increasing concentration of heavy metals is unpleasantly affecting our ecosystem due to their toxicological and physiological effects in environment [1–3]. Pollution of natural water by metal ions has become a major issue all over the world because metal concentrations in water often exceed the admissible values. The main heavy metals which cause metal ion pollution are Hg, Cu, Th, Cd, Pb, Cr, As and Ni [4,5]. These metals, if present beyond certain concentration can be a serious health hazard which can cause many disorders in normal functioning of human beings and animals [1–3]. Among these toxic heavy metal ions, Pb(II) ion is one of the most toxic and longstanding environmental contaminant [4]. The permissible levels of Pb(II) in drinking and waste water are 0.05 mg L<sup>-1</sup> and 0.005 mg L<sup>-1</sup>, respectively [6,7]. Lead can affect almost every organ and system in the body. Exposure to high lead levels can severely damage the brain and kidneys in adults or children and ultimately cause death. Hence the removal of lead ions from water and waste water is important in terms of protection of public health and environment [8]. Various approaches have been

applied for the removal of these types of toxic metals from aqueous solution such as chemical precipitation, ultra-filtration, reverse osmosis, oxidation, electro dialysis, ion-exchange and adsorption on several low-cost adsorbents [9–12]. But, adsorption is one of the most effective and widely used methods particularly in the treatment of wastewater from industrial and domestic sources.

From last few years, a large number of composite cation exchangers have been synthesized [13–21]. These materials have been widely used in metal recovery process, water softening, removal of interfering ions, fabrication of ion-selective electrodes and fuel cells. Inconsideration of the advantages associated with composite materials, efforts were therefore made in our laboratory to synthesize composite cation exchange material EDTA-Zr(IV) iodate. The physicochemical properties of this material were examined by instrumental analyses, viz. FTIR spectroscopy, X-ray diffraction studies and TGA-DTA investigations [22]. In order to explore the potentiality of EDTA-Zr(IV) iodate composite cation exchange material in the separation of metal ions, distribution studies for various metal ions were performed in acidic mediums. On the basis of distribution coefficient values, EDTA-Zr(IV) iodate was found to be highly selective for Pb(II) which is one of the most toxic metal ions. The practical applicability of EDTA-Zr(IV) iodate was explored by separating Pb(II) metal from a synthetic mixture of metal ions viz. Pb<sup>2+</sup>, Hg<sup>2+</sup>, Zn<sup>2+</sup>, Cd<sup>2+</sup>, Mg<sup>2+</sup> and Al<sup>3+</sup>. The effects of

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several operating parameters such as contact time, pH, initial Pb(II) ion concentration, dose and temperature were investigated to arrive at optimum conditions for the adsorption of Pb(II) on EDTA-Zr(IV) iodate composite cation exchange material. Various kinetic models as well as isotherm models have been studied for their usefulness in correlating the experimental data. Adsorption-desorption and regeneration studies of EDTA-Zr(IV) iodate were also performed.

## Experimental

### Reagents and instrumentation

The main reagents used for the synthesis of the material were zirconium oxychloride, potassium iodate and EDTA. These reagents were purchased from Sigma-Aldrich, Germany. All other chemicals and reagents used were of analytical reagent grade. The standard stock solutions of metals were prepared by dissolving appropriate amounts of their corresponding nitrate and chloride salts in Milli-Q water.

The main instruments used during the study were single electrode pH meter (Orion 2 star, Thermo Scientific, USA), FTIR spectrophotometer (Perkin-Elmer Spectrum-BX, USA), an automatic thermal analyzer (V2.2A Du Pont 9900), a PW 1148/89 based X-ray diffractometer (Phillips, Holland) and a water bath incubator shaker (SW22/9550322, Julabo, Germany). Water was purified with a Milli-Q water purification system (Millipore, Bedford, MA, USA).

### Synthesis of EDTA-Zr(IV) iodate composite cation exchanger

EDTA-Zr(IV) iodate composite cation exchanger was synthesized by the same method as given in our previous paper [22]. Initially, 0.25 M aqueous solution of potassium iodate was added into 0.25 M Zr(IV) oxychloride solution gradually with continuous shaking. The pH was kept 1.0 by using 1 M nitric acid or 1 M ammonia solutions. The white precipitate of Zr(IV) iodate was obtained. Subsequently, 0.01 M EDTA solution was added into the inorganic precipitate of Zr(IV) iodate and mixed thoroughly with constant stirring for 1 h. The gelatinous precipitate so formed, was allowed to stand for 24 h in the mother liquor for digestion. The supernatant liquid was removed and the precipitate was washed with demineralized water several times to remove excess reagents. The products were dried at  $40 \pm 2$  °C in an oven. The dried product was then kept in demineralized water for cracking and converted to H<sup>+</sup> form by placing it in 1 M HNO<sub>3</sub> solution and washed with demineralized water to remove excess acid and finally dried at  $40 \pm 2$  °C.

### Distribution studies

In order to get the idea of partition behavior of the EDTA-Zr(IV) iodate exchanger toward the separation of metal ions of analytical interest, distribution coefficient ( $K_d$ ) values for different metal ions were determined in acidic mediums. 0.4 g of EDTA-Zr(IV) iodate composite cation exchanger in H<sup>+</sup> form was treated with 40 mL solution of metal ions in required solvent medium in a 100 mL Erlenmeyer flask. The mixture was shaken for 6 h at  $25 \pm 2$  °C in a temperature controlled incubator shaker. The amount of metal ions before and after adsorption was determined by titration against a standard solution of 0.01 M di-sodium salt of EDTA. The distribution coefficients were calculated using the following equation:

$$K_d = \frac{\text{milli equivalent of metal ions/gm of ion-exchanger}}{\text{milli equivalent of metal ions/mL of solution}} \times (\text{mL g}^{-1})$$

$$K_d = \frac{I - F}{F} \times \frac{V}{M} (\text{mL g}^{-1}) \quad (1)$$

where  $I$  is the initial amount of the metal ion in the solution phase,  $F$  is final amount of metal ion in the solution phase after treatment with the exchanger,  $V$  is the volume of the solution (mL) and  $M$ , the amount of ion exchanger taken (g).

### Selective separation of Pb(II) from a synthetic mixture of metal ions

For this study, 0.4 g of EDTA-Zr(IV) iodate composite cation exchanger (H<sup>+</sup> form) was packed in a glass column with glass wool support. Different sets of synthetic mixtures of metal ion solutions were taken in the columns in which the amount of Pb(II) was varied and the amount of rest metal ions (Hg<sup>2+</sup>, Zn<sup>2+</sup>, Cd<sup>2+</sup>, Mg<sup>2+</sup> and Al<sup>3+</sup>) was kept constant. The solution was allowed to flow down at the rate of 0.5 mL min<sup>-1</sup>. The mixture of metal ions namely Hg<sup>2+</sup>, Zn<sup>2+</sup>, Cd<sup>2+</sup>, Mg<sup>2+</sup> and Al<sup>3+</sup> were eluted together first and Pb(II), which was retained strongly by the EDTA-Zr(IV) iodate, eluted at last by using 0.1 M nitric acid as an eluent. The effluent was collected in 10 mL fractions and titrated with standard 0.01 M EDTA solutions.

### Adsorption studies

The adsorption of Pb(II) onto EDTA-Zr(IV) iodate was carried out by batch method. The adsorption experiments were carried out in 250 mL glass conical flasks. 0.4 g of EDTA-Zr(IV) iodate was added to 40 mL of Pb(II) solution of known concentration in conical flask which was placed in thermostat cum shaking assembly. The solution was stirred continuously at constant temperature for 1 h to achieve the equilibration time. After equilibration time, EDTA-Zr(IV) iodate was filtered off using Whatman filter No. 41 and the equilibrium concentration of Pb(II) was determined by EDTA titration. A number of parameters such as contact time, pH, EDTA-Zr(IV) iodate dose and initial Pb(II) concentration were changed in order to optimize the adsorption process.

The amount of Pb(II) per unit weight of adsorbent,  $q_e$  (mg g<sup>-1</sup>) was calculated by the following equation:

$$q_e = \frac{V(C_o - C_e)}{W \times 1000} \quad (2)$$

where  $V$  is the volume of Pb(II) solution in liter,  $C_o$  and  $C_e$  are the initial and final concentrations (mg L<sup>-1</sup>) of Pb(II) in solution, respectively,  $W$  is the weight (g) of EDTA-Zr(IV) iodate.

Kinetics studies were performed by varying the Pb(II) ion initial concentration ( $C_o$ , 10–60 mg L<sup>-1</sup>). The samples were collected at specified time intervals until equilibrium attained. Isotherm studies were performed by varying the reaction temperature (25–50 °C) and initial concentration of Pb(II) solution (10–60 mg L<sup>-1</sup>).

### Desorption and regeneration studies

Desorption and regeneration studies were also carried out by batch process. 100 mL of 20 mg L<sup>-1</sup> Pb<sup>2+</sup> solution was treated with 0.4 g of EDTA-Zr(IV) iodate in conical flask in a temperature controlled shaker incubator for 1 h. After 1 h, the EDTA-Zr(IV) iodate was washed several times with Milli-Q water to remove the excess of Pb<sup>2+</sup>. Then, EDTA-Zr(IV) iodate was treated with 100 mL of 0.1 M HNO<sub>3</sub> solution in other flask. The flask was again shaken in temperature controlled shaker incubator (to desorb Pb<sup>2+</sup>) for 1 h. The solution was then filtered with Whatman filter No. 41 and the filtrate was analyzed to check the desorbed Pb<sup>2+</sup> using EDTA titration as given above.

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