

Influence of the support base on the sorption of Co(II) with mixed solvents

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Abstract

Theonyltrifluoroacetone (TTA) mixed with trioctyl phosphine oxide (TOPO) have been supported on Amberlite XAD-4, silica gel and wood powder. The resulting resins have been used to study the sorption of cobalt(II) from acetate buffer and their sorption capacities were determined and were found to be 12.38, 11.61 and 6.51 mg g⁻¹ for TTA + TOPO/Amberlite XAD4, TTA + TOPO/silica gel and TTA + TOPO/wood, respectively. The experimental results showed that the sorption of Co(II) increases with its initial concentration. The results can be fitted by Freundlich isotherm in the studied cobalt concentration range [10⁻⁴–10⁻³ M] and 1/n values are 1.07, 1.17 and 1.73 for Amberlite XAD4, silica gel and wood powder, respectively. The sorption of Co(II) onto the prepared resins was found to follow the pseudo-second order model and the sorption rate have the values 8.79 × 10⁻³, 10 × 10⁻³ and 16 × 10⁻³ g mg⁻¹ min⁻¹ for Amberlite XAD4, silica gel and wood powder, respectively. Thermodynamic parameters have also been calculated for the three systems and sorption process was found to be spontaneous and endothermic one.

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1. Introduction

The removal of intermediate and low radionuclides from radioactive aqueous waste is a matter of great interest in the field of waste management. Cobalt (⁵⁸Co and ⁶⁰Co), cesium (¹³⁴Cs and ¹³⁷Cs) and strontium (⁸⁵Sr and ⁹⁰Sr) are considered from the most serious nuclides in this waste due to their relatively long half lives and their quantities. In addition, ⁶⁰Co is characterized by emission of high gamma energy (1.17 and 1.34 Mev) which is employed widely in medicine and nuclear industries. So, the separation of cobalt from the radio waste is an important step. Different techniques such as precipitation, oxidation/reduction [1], ion exchange [2], solvent extraction [3] and sorption [4] have been applied to recover the metal ions from aqueous solutions but in the recent years, the solid–liquid technique has come at the forefront. This is due to the superior characteristics of this technique since it is considered as a link between solvent extraction and ion exchange and characterized by its high binding capacity, selectivity towards different elements and enhancement of the

mobility of extractant in the resin surface. Valuable researches on the sorption and separation of metal ions with solid–liquid or solvent impregnated resin technique have been carried out [5–7].

The most common extractants used are organophosphorous compounds as tri-butyl phosphate (TBP) [8], di (2-ethyl hexyl) phosphoric acid (DEHPA) [9–12], bifunction carbamoylmethyl phosphine oxide [13] and organophosphonate [14]. Also, some extractants as tri-octyl amine [15], quaternary ammonium salts [16] and hydroxy quinoline [17] have rarely been used. Silica gel, Amberlite XAD series and cellulose powder have been used as supporting materials in such techniques. Until now, only a few studies have shown that the impregnated resin with mixed solvents exhibit good efficiency for the separation of metal ions [18].

Sekine and Dyrssen [19] and Ramadan [20] have used a mixture of TTA and TOPO to study the extraction of some divalent ions as Sr(II), Ca(II) and Co(II) from aqueous solutions. They found that the logarithmic values of equilibrium constant for the complexes Sr(TTA)₂.TOPO, Ca(TTA)₂TOPO and Co(TTA)₂TOPO are –9.91, –6.76 and –3.68, respectively. So, it could be observed that Co-complex has the highest stability with respect to the other complexes.

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Thus in this work, TTA and TOPO mixture supported on different bases viz—Amberlite XAD4, silica gel and wood powder in a trial to prepare impregnated resin with mixed solvents and using them to study the sorption of $^{60}\text{Co}^{2+}$ from aqueous solution. Different parameters were studied to optimize the sorption process. The effect of the presence of some interfering ions like Cs^+ and Sr^{2+} on Co^{2+} separation was studied.

2. Experimental

2.1. Reagents

TTA and TOPO were supported from Merck and Eastman Organic Chemicals and used as extractants. Amberlite XAD4 resin with mesh number (20–60), surface area $725\text{ m}^2/\text{g}$, porosity% 45, true weight density 1.02 and average pore diameter 4–9 nm. Silica gel is an analytical grade with mesh number (70–230) and having pore diameter 60 \AA . Both resins were obtained from Fluka while wood powder was obtained from Inshas region. Wood was grinded and sieved to give the particle diameter 0.1–0.4 mm.

2.2. Purification of bases

The three bases were washed several times with acetone and water to eliminate any foreign materials. The materials were filtrated, dried at $40\text{ }^\circ\text{C}$ for 48 h and kept in a desecator for further usage.

2.3. Preparation of impregnated resins

The dry method is applied for the preparation of mixed solvent impregnated resins. In this method, a fixed weights 2.77 and 0.20 g of TTA and TOPO, respectively were dissolved in 125 ml benzene then 40 ml of the solvents in benzene were mixed with 3 g of either support base Amberlite XAD4, silica gel or wood powder until all organic solution was absorbed. Then the impregnated resins were dried at $40 \pm 2\text{ }^\circ\text{C}$ for 48 h.

In order to detect the fixation of solvents into the supported base, an amount of 0.1 g of either impregnated resin TTA + TOPO/Amberlite XAD4 or TTA + TOPO/silica gel or TTA + TOPO/wood was shaken with 10 ml of acetate buffer for 48 h. Then TTA was determined spectrophotometrically in aqueous solution at wave length 325 nm. The librated percentage of TTA and TOPO into aqueous solution did not exceed 5%. The surface area of untreated silica gel and wood powder was measured using BET technique and found to be 422 and $7.1\text{ m}^2/\text{g}$ for silica gel and wood, respectively and these values were decreased to 322.49 and $6.91\text{ m}^2/\text{g}$ after impregnation with TTA + TOPO and this decrease was also observed for surface area of TTA + TOPO/Amberlite XAD4 and found to be $466.73\text{ m}^2/\text{g}$. This behavior could be attributed to the coating of pores of support bases by organic solvents which decrease the penetration of N_2 into some pores. The measurements of surface area and pore diameter were performed using pore size micrometer-9320, USA.

2.4. Preparation of $^{60}\text{Co}^{2+}$

The radioactive cobalt is prepared by irradiation of cobalt oxide for two days in Egyptian Et-RR-1 nuclear reactor.

2.5. Procedure

The sorption of cobalt(II) was studied by batch equilibration experiments. 0.05 g of either prepared resins was shaken well with 5 ml of acetate buffer spiked with radioactive cobalt in thermo-stated shaker of the type SW-20C/2 from Julobo laboratories GmbH, Germany. After equilibration and centrifugation, 1 ml of aqueous solution was withdrawn for gamma-ray estimation using Nucleus 500 connected to NaI crystal. The distribution coefficient (K_d) and uptake % ($U\%$) of cobalt were calculated from following equations.

$$K_d = \frac{A_o - A}{A} \times \frac{V}{m} \text{ (ml/g)} \quad (1)$$

$$U\% = \frac{A_o - A}{A_o} \times 100 \text{ (%) } \quad (2)$$

where, A_o and A are the activities expressed in counts per minute of 1 ml solution for the radioisotope before and after contacting the resin, V is the volume of the aqueous phase in milliliter and m is the weight of dry resin in gram.

3. Results and discussion

3.1. I.R. spectra

I.R. spectra of free support bases and impregnated resins are given in Fig. 1a–c. Fig. 1a represents I.R. spectra for untreated Amberlite XAD4 and TTA + TOPO/Amberlite XAD4. The spectra for free Amberlite XAD4 show characteristic peak at 3458 cm^{-1} that is related to O–H stretching vibration of H_2O molecules. The absorption bands at $2851\text{--}3015\text{ cm}^{-1}$ are ascribed to aromatic and aliphatic C–H stretching and the peaks at $1444\text{--}1640\text{ cm}^{-1}$ for C=C ring stretching. The spectra for TTA + TOPO/Amberlite XAD4 shows the shift and decrease in the intensity of stretching C–H and C=C bands at 2928 and $1444\text{--}1640\text{ cm}^{-1}$, respectively. This change may be referred to the role of π -electrons of C=C in immobilization of TTA and TOPO on Amberlite XAD4. Also, the appearance of new peaks at 1702, $1110.3\text{--}1290.64\text{ cm}^{-1}$ which are characteristic for keto C=O, phosphoryl group P=O and P–O–C stretching vibration confirms the incorporation of solvents into Amberlite XAD4. Fig. 1b represents I.R. spectra for TTA + TOPO/silica gel. There are intense peaks at 1656.36 and 1079 cm^{-1} which are characterized for keto and phosphoryl groups, respectively. Peaks at 955, 790 and 723 cm^{-1} which are assigned for C–H and C=C out of plane (flexion). In addition to sharp absorption peak at 481 cm^{-1} which indicates the presence of oxygenated inorganic constituents, I.R. spectra of wood and TTA + TOPO/wood are represented in Fig. 1c. The spectra of untreated wood show peak at 3433.38 cm^{-1} which is related to O–H stretching from water or hydroxyl groups of cellulose. Also, peak at 2918 cm^{-1} is

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