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Separation of Iron, Cobalt, Nickel, and Cadmium in Aqueous Acid Halide Solution Using Aliquat-Foam Columns

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NOTE

Separation of Iron, Cobalt, Nickel, and Cadmium in Aqueous Acid Halide Solution Using Aliquat-Foam Columns

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Abstract

Polyurethane foam loaded with Aliquat 336 was prepared. The suitability of the prepared reagent foam for the separation of various relative concentrations of iron and cobalt in acidic aqueous solutions was confirmed using the static technique. The separation of other metal couples was achieved on the basis of similar ideas. Columns packed with the Aliquat foam were successfully used in the separation of iron or cadmium from cobalt and nickel. The foam column performance proved quite reasonable, and the application of relatively high flow rates (10 mL/min) was found to be possible.

INTRODUCTION

The recent introduction of cellular plastics (1) as a support in reversed phase extraction chromatography is a useful addition to the subject. Polyurethane foam was proved (2) to retain a considerable amount of various organic reagents, and columns packed with the foam material have good hydrodynamic properties. Experience with different reagent-loaded foam columns (3) showed that relatively high flow rates could be used without causing any considerable leaching of the organic reagents from the foam material.

The aim of the present work was to prepare polyurethane foam loaded with methyl tricaprylyl ammonium chloride (Aliquat 336) and to examine the possibility of using this reagent foam for the separation and preconcentration of various metal ions in aqueous halide solutions using batch and column techniques.

EXPERIMENTAL

Reagents and Materials

Unless otherwise specified, all the reagents used were of analytical reagent grade. Bidistilled water was used. Methyl tricaprylyl ammonium chloride (Aliquat 336), pure grade, was used without further purification.

Polyurethane foam, a polyether of the open-cell type, was supplied by Greiner K. G. Schaumstoffwerk, Kremsmunster, Austria. The volume weight of the foam was 30 g/m³. The foam (cubes of about 3–4 mm edge) was washed with 1 *M* hydrochloric acid followed by bidistilled water until the washings were free from chloride ion. Then the foam material was washed with acetone and dried at 80°C.

Stock solutions containing 10 mg/mL of iron(III), cobalt(II), cadmium(II), and nickel(II) ions were prepared by dissolving the corresponding metal salt in bidistilled water. A series of standard iron, cobalt, cadmium, and nickel solutions was prepared by diluting the stock solutions of these elements with bidistilled water or 3 *M* hydrochloric acid.

The loaded foam material was prepared using the previously recommended procedure (4). The chromatographic columns were packed using the vacuum method (5) of foam column packing.

Apparatus

Glass columns of 15 mm diameter and 15 cm length were used in the dynamic experiments. In the batch extraction experiments, stoppered flasks were employed.

RESULTS AND DISCUSSION

Polyurethane foam loaded with tri-*n*-octyl amine has been previously employed (4) for the chromatographic separation of some metal halide complexes in aqueous solution. A detailed study for the application of polyurethane foam treated with methyl tricaprylyl ammonium chloride in the extraction and separation of iron, cobalt, nickel, and cadmium in aqueous acid halide solutions has been undertaken in the present work.

The effect of hydrochloric and hydrobromic acid concentrations on the extraction of iron(III), cobalt(II), cadmium(II), and nickel(II) has been investigated in the batch extraction system using the amine-loaded foam. In this experiment, 2 mg amounts of the above-mentioned ions in 10 mL aqueous solutions having various acid concentrations were shaken separately with 0.1 g loaded foam for 3 h in a mechanical shaker. The unextracted metal

ions were determined at each acid concentration after equilibrium and the extracted amounts were then calculated. Nickel(II) and cobalt(II) show no extraction in the range of acidity examined (1–8 *M* HCl or HBr). On the other hand, cadmium(II) and iron(III) were extracted on the foam. The extraction of cadmium(II) was found to be complete at all the acidities studied, while the extraction of iron(III) depended on the acid concentration. Quantitative extraction of iron was noticed at acidities above 5.8 and 8 *M* in hydrobromic and hydrochloric acids, respectively.

On the basis of these results, it was possible to separate iron from cobalt in aqueous solution using the static extraction system. Iron(III) (1–5 mg) is quantitatively retained on the amine-loaded foam even in the presence of as high as 1000 mg of cobalt(II) ion in 6 *M* hydrochloric acid solution. In all cases cobalt is not absorbed by the foam and remained in the aqueous solution. The separation of other metal couples was achieved on the basis of a similar method.

In separate flow experiments, quantitative retention and elution of iron(III) and cadmium(II) ions were examined. Thus, when 5 mg of iron or cadmium in 3 *M* hydrochloric acid solution was applied to the foam column, both metal ions were retained on the foam bed. The column was then washed with 3 *M* hydrochloric acid solution. In the case of iron, elution was quantitatively effected by passing 0.1 *M* L-ascorbic acid solution, while

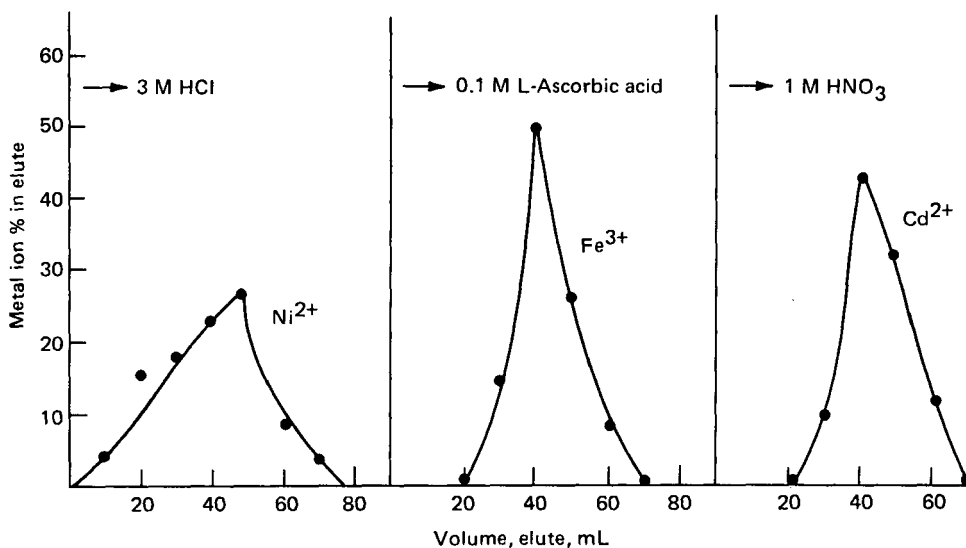


FIG. 1. Separation of Ni^{2+} – Fe^{3+} – Cd^{2+} in 3 *M* hydrochloric acid solution using Aliquat-foam column.

cadmium needs 1 *M* nitric acid solution at a 1–2 mL/min flow rate. The HETP as calculated from the elution curves was found to be 3.0 and 1.8 mm for iron and cadmium, respectively. Higher flow rates were tried, and the elution curves were found to be quite sharp and symmetric up to a 10 mL/min flow rate which reflects the relatively high efficiency of the proposed foam columns.

The breakthrough and total capacities of Aliquat foam were calculated from the breakthrough capacity curve and found to be 48 and 61 mg Fe^{3+} /g loaded foam, respectively. It is worth mentioning that the total capacity of 1 g loaded foam was also determined in batch experiments and found to be 64 mg Fe^{3+} , which is quite close to that obtained in the dynamic experiment.

The amine foam packed in chromatographic columns has been tested for the separation of iron(III), cobalt(II), nickel(II), and cadmium(II). Separations of iron(III)–cobalt(II); iron(III)–nickel(II); iron(III)–cadmium(II); cadmium–cobalt(II); cadmium(II)–nickel(II); iron(III)–nickel(II)–cadmium(II); iron(III)–cobalt(II)–cadmium(II); cadmium(II)–nickel(II)–cobalt(II); and iron(III)–cobalt(II)–nickel(II)–cadmium(II) were successfully carried out using a feeding solution of 5 mL containing 5 mg of each ion investigated in 3 *M* hydrochloric acid solution. At this acid concentration both cadmium(II) and iron(III) were retained quantitatively by the foam column. The elution of iron(III) is attained by washing the column with either 0.1 *M* HNO_3 , 1 *M* NaF, or 0.1 *M* L-ascorbic acid, followed by 1 *M* HNO_3 as the eluting agent for cadmium(II). Hydrochloric acid (3 *M*) is

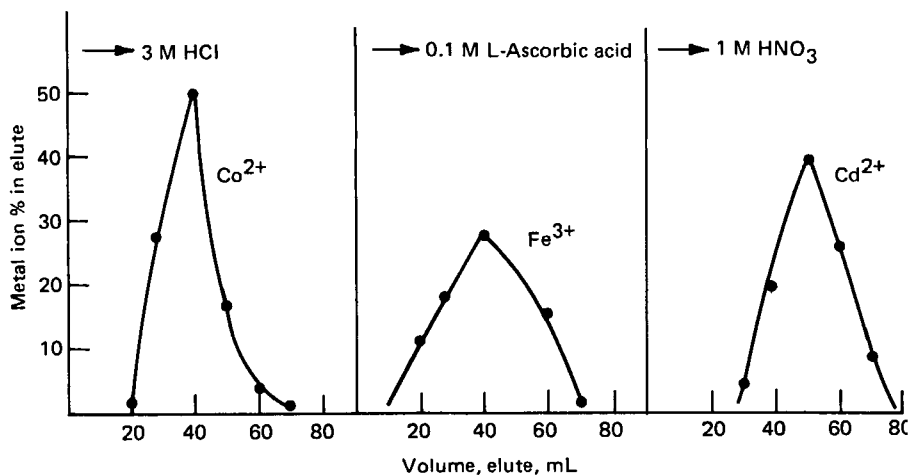


FIG. 2. Separation of Co^{2+} – Fe^{3+} – Cd^{2+} in 3 *M* hydrochloric acid solution using Aliquat–foam column.

the most proper eluting agent for both cobalt(II) and nickel(II). Representative elution curves for mixtures of iron(III)–nickel(II)–cadmium(II) and iron(III)–cadmium(II)–cobalt(II) are shown in Figs. 1 and 2, respectively.

The quantitative separation of various relative concentrations of these metal ions has been carried out using 2–4 mL/min flow rates. The proposed foam columns have been used in at least 50 chromatographic cycles without any detectable change in their performance and efficiency.

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