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Solid-Phase Extraction of Cd(II) as Diethyldithiocarbamate Complex by Polyurethane Foam: Kinetic and Thermodynamic Characterization

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ABSTRACT

This paper presents a study about the sorption of the cadmium–diethyldithiocarbamate [Cd(II)–DDTC] complex onto polyurethane foam (PUF). It was observed that the maximum sorption of Cd(II) (80 or 150 $\mu g\,L^{-1}$) was verified at pH 6.8 in the presence of $4.5 \times 10^{-5}\,mol\,L^{-1}$ DDTC. The shaking time needed to achieve the equilibrium

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was 40 min. From these data, a kinetic characterization was performed by applying three models, which revealed that a film-diffusion process was a rate-determining mechanism. Results also indicated that an ether-like solvent extraction was the sorption mechanism. The investigation of many metallic ions as concomitants showed that the sorption by foam is relatively selective and it can be enhanced by using a suitable masking agent or incrementing the foam mass.

Key Words: Solid-phase extraction; Polyurethane foam; Diethyldithiocarbamate; Cadmium.

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INTRODUCTION

The use of solid-phase extraction (SPE) procedures has occupied a remarkable position in the last few years in the analytical chemistry field due to its efficiency and low requirements in terms of cost.[1] In this context, several materials have been utilised for this purpose.[2–5] Among many solid phases used, the use of polyurethane foam (PUF) has grown since the first publication of the pioneering work of Bowen.[6]

Various research papers have been published reporting the use of unloaded foams for analytical preconcentration and separation of organic and inorganic species in aqueous medium.[7–11] Moreover, the use of PUF as a solid support for specific reagents[12–14] enhancing the selectivity and analytical throughput of SPE procedures has been described.[15] Additionally, some papers have focused the characterization of the sorption process by studying the kinetics and thermodynamics aspects related to the extraction.[16–18]

It is well known that the reaction between diethyldithiocarbamate (DDTC) and several metal ions sparingly yields water-soluble metal complexes that can be adequately extracted from aqueous medium with a wide variety of nonpolar organic solvents. Some works have reported the use of PUF loaded with DDTC for collection of Co(II), Hg(II), and Sb(III) from aqueous solution.[19–21]

Besides demonstrating the high potential of PUF to retain and remove Cd(II) from aqueous solution as DDTC complex, the goal of this work was to characterize the processes involved on the sorption of the Cd(II)–DDTC complex onto unloaded PUF.

EXPERIMENTAL

Apparatus

Cadmium solutions were analyzed on an Hitachi (Tokyo, Japan) polarized Zeeman atomic absorption spectrometer, model Z-8200, equipped with a cadmium hollow cathode lamp and an autosampler Hitachi model SSC-300. Integrated absorbance measurements were carried out by using an Hitachi Pyro tube-type cuvette. The instrumental operating conditions and the temperature program used are summarized in Tables 1 and 2, respectively.

Reagents

All reagents were of analytical grade and used without further purification. The solutions always were prepared with water purified in a Simplicity Milli-Q System (Millipore, Bedford, MA).

A $1000\,\mathrm{mg\,L^{-1}}$ cadmium stock solution was prepared by dissolving 1 g of metallic cadmium (Merck, Darmstadt, Germany) in a heated mixture of 15 mL of concentrated HNO_3 (Merck, Darmstadt, Germany) and 50 mL of water. After cooling, the volume was made up to 1000 mL in a volumetric flask.

A 0.1% w/v ($4.3 \times 10^{-3}\,\mathrm{mol\,L^{-1}}$) DDTC stock solution was prepared by dissolving 0.1 g sodium DDTC reagent in approximately 80 mL heated (60°C) water. Afterward, the volume was made up to 100 mL in a volumetric flask. This solution was stable for at least 2 weeks.

A $0.5\,\mathrm{mol\,L^{-1}}$ acetic buffer solutions with pH 4.0, 5.0, and 6.0 were prepared by dissolving 34 g of tri-hydrated sodium acetate (Vetec, Rio de Janeiro, Brazil) and 4.4 mL of glacial acetic acid (Merck, Darmstadt, Germany) in approximately 800 mL of water. Afterward $0.1\,\mathrm{mol\,L^{-1}}$ NaOH (Merck, Darmstadt, Germany) or $0.1\,\mathrm{mol\,L^{-1}}$ HCl (Merck, Darmstadt, Germany) was added to attain the desirable pH, and the volume was made up to 1000 mL.

A $0.5\,\mathrm{mol\,L^{-1}}$ phosphate buffer solution with pH 6.8 was prepared by dissolving 57 g of di-potassium hydrogen phosphate tri-hydrate (Merck, Darmstadt, Germany) and 34 g of potassium di-hydrogen phosphate (Merck, Rio de Janeiro, Brazil) in water to complete 1000 mL just after to adjust the pH to 6.8 with $0.1\,\mathrm{mol\,L^{-1}}$ NaOH.

Table 1. Operational conditions used in the determination of Cd by (ETAAS).

Parameter	Set value
Lamp current	7.5 mA
Wavelength	228.8 nm
Spectral bandwidth	1.30 nm
Purge gas	Argon
Background correction mode	Polarized Zeeman
Graphite furnace	Pyrolytical coated tube-type

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Table 2. Temperature program used for the determination of Cd by (ETAAS).

Stage	Temperature (°C)	Time (sec)	Ar flow rate (mL min^{-1})
Dry[a]	80–140	40	200
Pyrolysis	600	30	200
Atomization	2,500	10	0
Clean	2,550	4	200

[a] Addition of 10 μL of Pd 1000 mg L^{-1} after injection of 20 μL of solution.

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A 0.5 mol L^{-1} borate buffer solutions with pH 9.0 and 10 were prepared by dissolving 30.9 g of boric acid (Merck, Rio de Janeiro, Brazil) in exactly 1000 mL of water. Before volume adjustment, the pH solutions were settled to 9.0 and 10 with 0.1 mol L^{-1} NaOH solution.

The Triton X-100 used was supplied by Vetec (Rio de Janeiro, Brazil).

A commercial open cell, polyether-type PUF (Vulcan do Brasil—VCON 202, 42% resilience and 10–12 cells per linear cm) was pulverized in a blender and used as previously described.[11]

General Procedure

The experiments were carried out by agitating 25 mL of a solution containing known concentrations of Cd(II) (usually 100 μg L^{-1}) and DDTC (50 mg L^{-1}), in a mechanical shaker, with 50 mg of PUF for 40 min. A phosphate buffer solution was used to keep the system in pH 6.8. All these parameters were maintained constant in all experiments except otherwise mentioned.

After reaching the equilibrium, the mixture was filtered (polyethylene filtration unit with 0.45-μm pore size and 13-mm outer diameter (Millipore, Bedford, MA), and the concentration of cadmium was determined in the remained solution by electrothermal atomic absorption spectrometry (ETAAS). The percentage of Cd–DDTC complex sorbed by foam was calculated from these data using the following equation:

$$\mathrm{Cd}_{\mathrm{sorbed}}\ (\%) = \frac{C_0 - C_{\mathrm{f}}}{C_0} \times 100$$

where C_0 and C_{f} are the concentrations of Cd(II) before and after to perform the extraction procedure. The percent sorption was the average of at least three measurements. The measurements precision as relative standard deviation, in this case, was always around 5%.

RESULTS AND DISCUSSION

Chemical Characterization of Extraction

The pH of the medium is directly associated to the formation of Cd(II)–DDTC and its extraction by PUF. Therefore, experiments were carried out to evaluate the influence of this parameter on the sorption process. For this purpose, a set of tests was done by preparing aliquots of 25 mL of solution containing 50 and 100 μg L^{-1} Cd(II) and 100 mg L^{-1} DDTC, and varying the pH by using different buffer systems, covering the pH range between 4.0 and 10. The buffer solutions used are listed in the Reagents sub section. A PUF mass of 50 mg was added to the solutions, and the system was agitated for 1 hr. The obtained results (Fig. 1) showed that at pH 4.0 only 25% of the Cd (II)–DDTC complex is sorbed by PUF. On the other hand, quantitative sorption is achieved at a pH higher than 6.0. Based on these data, the pH of the solution was always buffered to 6.8 by using a phosphate system.

Extraction of cadmium as a DDTC complex is dependent on both the complex formation and the occurrence of effective contact between solid (PUF) and analyte present in the aqueous phase. This fact suggests that special attention should be given on a study of optimum ratio between metal and complexing agent concentrations. This study was carried out by fixing both the Cd(II) concentration and pH in 6.8 and by varying the

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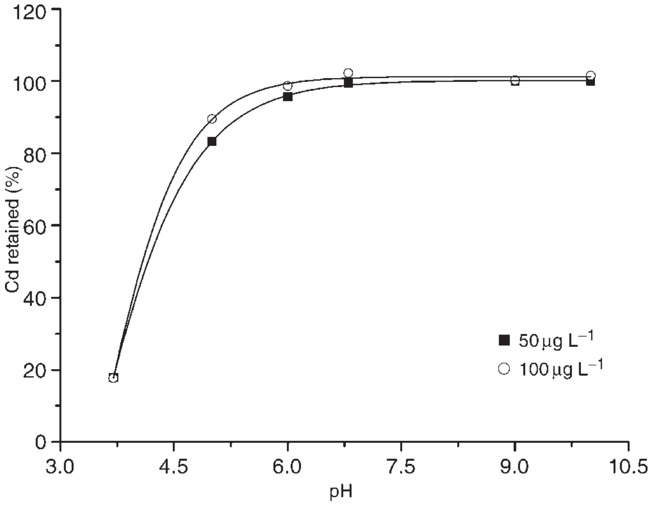

Figure 1. Effect of the pH on the Cd(II)–DDTC complex sorption; PUF mass = 50 mg, [DDTC] = 100 mg L^{-1}, [Cd(II)] = 50 and 100 μg L^{-1}, shaking time = 1 hr.

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DDTC concentration in solution from 2.5–200 mg L^{-1}. As can be seen in Fig. 2, when the DDTC concentration was 25 mg L^{-1}, a quantitative sorption of Cd(II) was achieved for the two situations tested. In conclusion, in the established conditions, the minimum concentration of DDTC for total extraction of cadmium concentration up to 200 μg L^{-1} should be 25 mg L^{-1}. For all further experiments a DDTC concentration of 50 mg L^{-1} was used.

Diethyldithiocarbomate (DDTC) is a sulfur-containing organic ligand that forms a strong hydrophobic complex with heavy metals.[22] This feature favors the use of the SPE process for performing preconcentration of metals capable of reacting with this chelating agent. In this procedure, the presence of a substance that enhances the hydrophilic character of the complex formed causes a decrease in the extraction efficiency. In the same way, this phenomenon occurs in the extraction process in the solid phase based on the reverse-phase mechanism. Thus, to verify it on the extraction of Cd–DDTC by PUF, an experiment was carried out, whereby the concentrations of Cd(II) and DDTC were 100 μg L^{-1} and 50 mg L^{-1}, respectively, in a solution containing Triton X-100, varied between 0% and 8% m/v. As can be seen in Fig. 3, the efficiency of the extraction process decreased, the higher the concentration of surfactant in solution. It suggests that the hypothesis of the mechanism of the sorption of cadmium, as a DDTC complex, is probably regulated by an ether-like solvent extraction.

Kinetic Characterization of the Extraction Process

The kinetic of the sorption of Cd–DDTC onto PUF was assessed by agitating aliquots of 25 mL of solution containing 50 mg L^{-1} DDTC and 10 and 50 μg L^{-1} Cd(II), in pH 6.8 with 50 mg PUF, between 0.5 and 120 min. The sorption percentage was achieved by determining the amount of cadmium remained in the solution. The obtained results, shown in Fig. 4, evidenced that the sorption equilibrium was attained near to 40 min.

To kinetically characterize the extraction of Cd(II)–DDTC by PUF, evaluating the regulator mechanism of the process and quantifying the changes in the sorption occurred as a function of time, it must be used as a suitable kinetic model. Besides several kinetic models, one of the simplest kinetic analyses is the Lagergren equation:[23]

$$\log(q_e - q_t) = \log q_e - \frac{kt}{2.303}$$

where q_e is the concentration of the Cd–DDTC complex sorbed at equilibrium (mol g^{-1}), q_t is the concentration of the Cd–DDTC complex sorbed at time t (mol g^{-1}) and k is the overall rate constant. As applying this model did not

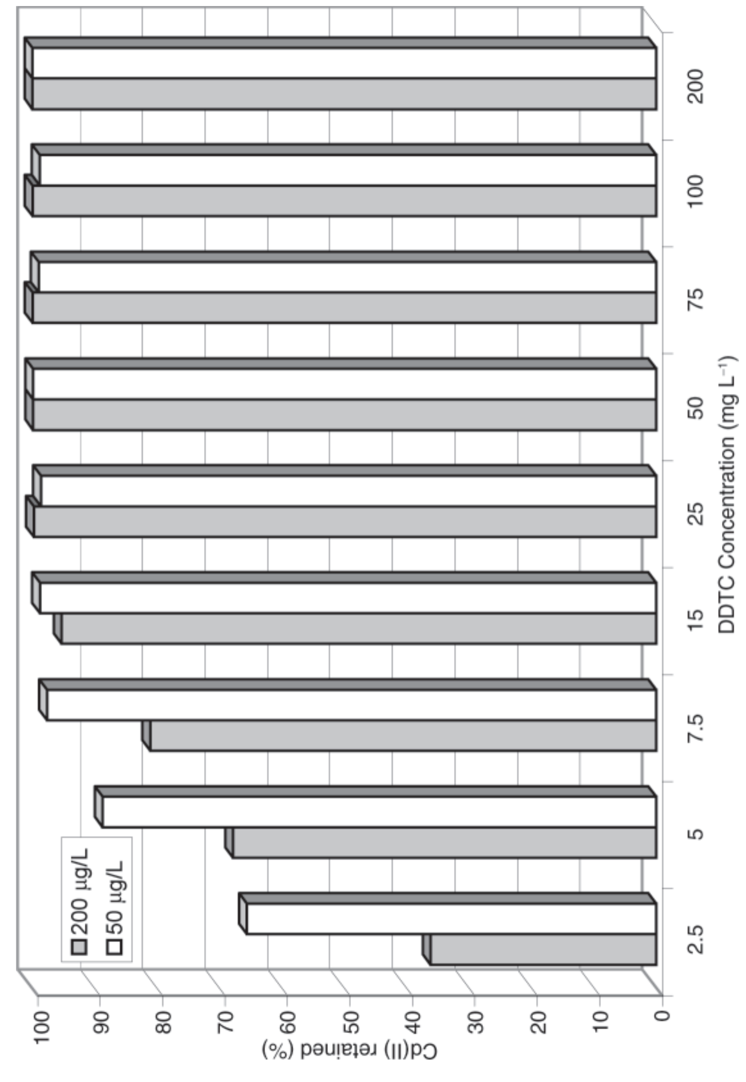

Figure 2. Influence of the DDTC concentration on the Cd(II) (50 and 200 µg L^{-1}) extraction by PUF; PUF mass = 50 mg, pH = 6.8, shaking time = 1 hr.

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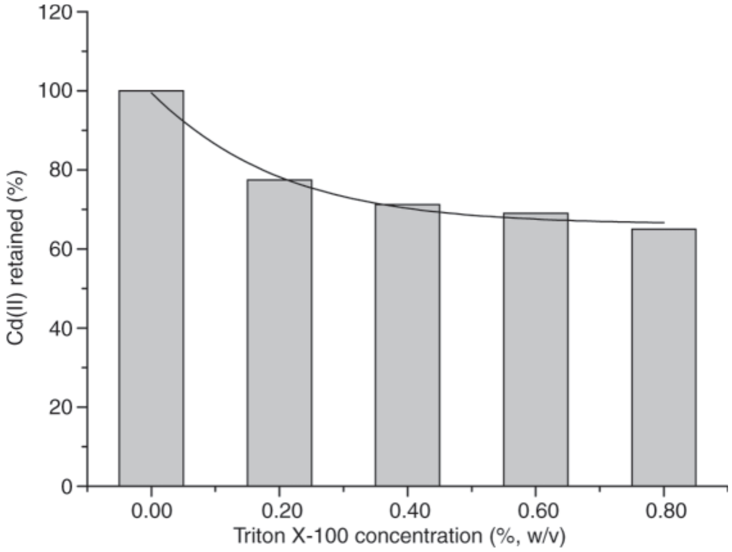

Figure 3. Influence of Triton X-100 concentration (surfactant agent) on Cd(II) metal ion retention; PUF mass = 50 mg, pH = 6.8, [DDTC] = 50 mg L^{-1}, [Cd(II)] = 100 μg L^{-1}, shaking time = 1 hr.

generate a straight line with a good correlation coefficient, a pseudosecond-order equation[24] was applied to the sorption process:

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e} t$$

The straight line obtained ($y = 0.0567 + 0.189,\ R = 0.9985$) is evidence that the kinetic sorption is regulated by a pseudosecond-order mechanism. The pseudosecond-order kinetic, constant k_2, then was evaluated as 0.017 g mol^{-1} min^{-1}.

Another tested simplified kinetic model to the sorption process was the intraparticle transport:[16,25,26]

$$q_t = K_d \sqrt{t}$$

where the K_d is the ratio between the amount of retained species and the amount of the remained species in solution at equilibrium. By applying this kinetic model to the sorption process by a plot of q_t vs. $\sqrt{t}$, shown in Fig. 5, it was possible to observe that the more external adsorptive sites were saturated in the first 15 min. From this point on, it succeeded in the retention of the adsorbate through the intraparticle transport, which presented slower

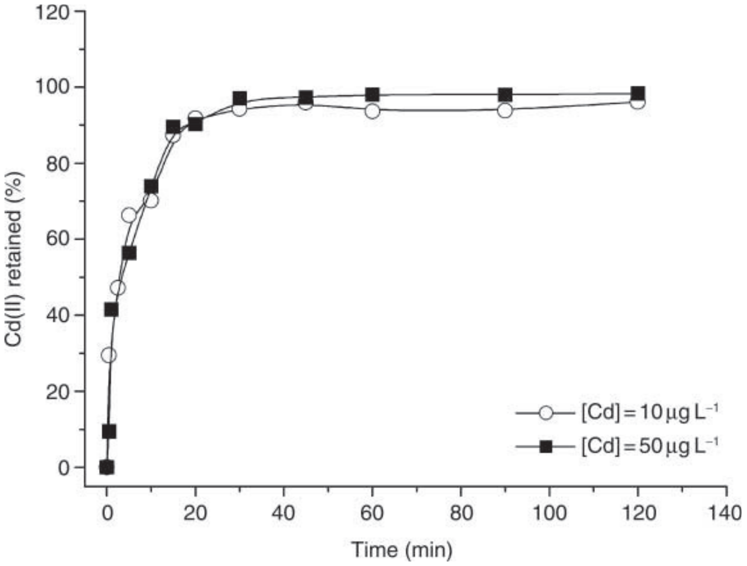

Figure 4. Effect of the shaking time on the retention of Cd(II) (10 and 50 µg L^{-1}); PUF mass = 50 mg, [DDTC] = 50 mg L^{-1}, pH = 6.8.

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kinetics than the film diffusion. The intraparticle constant was attained from the slope of the curve in the first 15 min as 0.031 µmol g^{-1} $min^{-1/2}$.

Isotherm Modeling

The evaluation of the sorption capacity of the solid phase was carried out by agitating, during 40 min, 50 mg of PUF with 25 mL of solution containing 50 mg L^{-1} DDTC and Cd(II) in the range of 200–3000 µg L^{-1} in pH 6.8. The sorption data was subjected to the Langmuir isotherm according to the following equation:

$$\frac{C_e}{C_{ads}} = \frac{1}{Q_b} + \frac{C_e}{Q}$$

where C_e is the concentration of Cd(II) in the equilibrium (mol L^{-1}), C_{ads} is the sorbed concentration (mol g^{-1}), Q_b is the Langmuir constant representing the maximum amount of the cadmium retained on the solid phase, and b is a coefficient relating the affinity between Cd(II)–DDTC and PUF. The plot of C_e/C_{ads} vs. C_e yielded a straight line ($y = 2796x + 0.011$, $R = 0.9921$),

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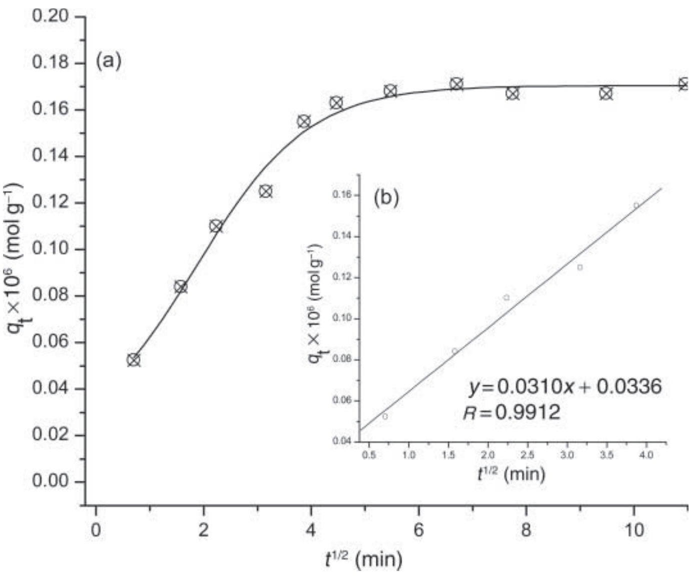

Figure 5. Morris–Weber plot for the kinetic modeling of Cd(II)–DDTC complex sorption onto PUF: (a) plot for overall range time tested and (b) linear plot for the first 15 min shaking time.

meaning that Langmuir sorption closely followed the sorption data. From the slope and intercept of the equation, the value of $Q = 3.6 \times 10^{-4}$ mol g^{-1} was obtained.

Thermodynamic Characterization

To thermodynamically characterize the sorption process, the efficiency of the extraction of Cd(II)–DDTC by PUF was tested in different temperatures. The study consisted in agitating, during 40 min, aliquots of 25 mL of solution containing 250 μg L^{-1} Cd and 1 mg DDTC in pH 6.8 with 50 mg PUF between 25°C and 50°C. The equilibrium constant, K_c, was calculated from the following form:

$$K_c = \frac{F_e}{1 - F_e}$$

where F_e is the fraction of Cd(II) sorbed at equilibrium. From this, $\log K_c$ was plotted vs. $1/T$ (T in K). A straight line ($y = 3.288x - 10.31$, $R = 0.9920$) was obtained over the entire range of temperature investigated. The following relationships were used to calculate the values of enthalpy, entropy, and Gibbs free energy for the sorption process:

$$\log K_c = -\frac{H}{2.303RT} + \frac{S}{2.303R}$$

$$G = -RT \ln K_c$$

From the slope and intercept of the linear fit obtained, the values of ΔG, ΔH and ΔS have been estimated, respectively, in -4.40 ± 0.89 kJ, -62.95 ± 4.02 kJ, and -197.4 ± 12.96 J. The negative values of ΔG and ΔH indicated, respectively, the spontaneous and exothermic characters of the sorption of Cd(II)–DDTC on PUF.

Presence of Metallic Concomitants

It is well recognized that the formation of a metal complex can be affected by the presence of another ion, which reacts with the complexing agent or metallic cation. Therefore, to verify the effect caused by the presence of concomitant metallic ions on the Cd(II)–DDTC sorption by PUF, known concentrations of the possible interferents were added in aliquots of 25 mL of solution containing 100 μg L^{-1} Cd(II), 50 mg L^{-1} DDTC, with pH 6.8. As previously done, the final solution was agitated with 50 mg of PUF during 40 min, and the Cd(II) that remained in solution was determined by ETAAS. The presence of Pb(II), Hg(II), Ag(I), Fe(III), Mn(II), and Zn(II) did not affect the extraction of Cd(II)–DDTC by PUF, in the established conditions, even when they were 100 times higher than the analyte. However, when Cu(II), Co(II), and Ni(II) were present in concentrations 10 times higher than Cd(II), a decreasing of the efficiency of sorption was observed. This behavior can be verified, in detail, in Table 3.

CONCLUSIONS

PUF in solid phase presented a high capacity and selectivity to extract Cd(II) ions complexed by DDTC, thus, opening new possibilities to preconcentrate and/or separate this cation in aqueous medium, with a low process cost. In addition, taking into account the hydrophobic characteristic of the complex, it was possible to conclude that a solvent extraction mechanism is the most probable controller of the whole sorption process, with the foam acting in a similar way as a nonpolar organic solvent. This hypothesis is highlighted when a surfactant agent (such as Triton X-100) was added to the extraction medium, which strongly decreased the amount of Cd(II)–DDTC

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Table 3. Study of the effect of concomitants on the extraction efficency of Cd(II)–DDTC complex by polyurathane foam 9 polyurethane foam.

Ratio metal/Cd	Cu(II)	Co(II)	Ni(II)	Pb(II)	Hg(II)	Ag(I)	Fe(III)	Mn(II)	Zn(II)
1	100	98.9	102	100	97.9	104	99.2	99.2	98.0
5	100	90.6	104	97.8	100	100	101	101	100
10	99.0	101	104	98.9	90.0	101	99.2	99.2	99.0
50	15.3	70.6	32.1	96.8	100	102	99.2	99.2	101
100	16.3	55.3	26.4	97.8	97.9	101	99.2	99.2	100

Note: Results are expressed as cadmium extraction efficiency (%).

retained by foam, thus, indicating that in a medium where the complex is more soluble, the sorption is diminished due to the lower affinity between the solid phase and Cd(II)–DDTC. This process usually is observed when a solvent extraction mechanism is involved and can be considered as a reverse salting-out effect.

Considering the high sorption capacity of PUF, the determination of cadmium remaining in the solution after Cd(II)–DDTC retention by foam must be carried out by using a sensitive analytical technique. In the present situation, ETAAS showed adequate characteristics to accomplish all sorption studies, making possible cadmium trace determinations.

From the analytical point of view, this work gives some address to perform cadmium preconcentration onto PUF from complex matrices. In addition, the possibility of carrying out the direct determination of cadmium concentration from the loaded PUF could be explored by its introduction into the graphite cuvette.[8]

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