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### Sorption of Co(II) onto chelating pyrocatechol violet-Amberlite XAD-16 resin

Zakia Bouariche<sup>a</sup>; Kamel Belhamel<sup>a</sup>; Mohamed Benamor<sup>a</sup>; Chafika Meziti<sup>a</sup>; Khedidja Younsi<sup>a</sup>; Idir Hassaini<sup>a</sup>

<sup>a</sup> Faculty of Technology, Laboratory of Organic Materials, Process Engineering Department, University of Bejaia, DZ-06000 Bejaia, Algeria

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## Sorption of Co(II) onto chelating pyrocatechol violet–Amberlite XAD-16 resin

ZAKIA BOUARICHE, KAMEL BELHAMEL\*,  
MOHAMED BENAMOR, CHAFIKA MEZITI,  
KHEDIDJA YOUNSI and IDIR HASSAINI

Faculty of Technology, Laboratory of Organic Materials,  
Process Engineering Department, University of Bejaia,  
DZ-06000 Bejaia, Algeria

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In this study, a chelating resin was synthesized by covalently linking pyrocatechol violet (PV) with the benzene ring of Amberlite XAD-16 through an azo ( $-N=N-$ ) spacer group and the resulting resin was characterized by infrared (IR) spectra. It has been used for the sorption of Co(II) in aqueous solutions at batch system by flame atomic absorption spectrometry (FAAS). Co(II) ions were extracted quantitatively in acidic nitric media at pH 4.15–5.10. The sorption capacity of modified resin was  $99.36 \text{ mg g}^{-1}$  of resin. The influences of pH of aqueous solutions, the amount of PV-XAD-16 resin, and the electrolyte salt concentration on the sorption of Co(II) ions were examined. The proposed separation-enrichment method was applied for the atomic absorption spectrometric determinations of Co(II) in river water with satisfactory results (recovery greater than 96.77%, relative standard deviation lower than 5%).

*Keywords:* Amberlite XAD-16; Pyrocatechol violet; Atomic absorption spectrometry; Sorption; Co(II)

### 1. Introduction

Heavy metal releases to the environment have been increasing continuously as a result of industrial activities and technological development, posing a significant threat to the environment and public health because of their toxicity, accumulation in the food chain, and persistence in nature. It is therefore necessary to remove heavy metals from wastewater. For this purpose, several methods have been suggested and used for the preconcentration and separation of trace elements according to the nature of the samples, the concentrations of the analytes, and the measurement techniques. These methods include solvent extraction, precipitation, adsorption, coagulation, electrolysis, membrane separation, ion exchange, and chelating resins [1–6]. Of all the preconcentration methods, chelating resins are good choices as they offer superior selectivity [7–10]. Therefore, the design of stable chelating resins having a high stability value,

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\*Corresponding author. Email: kbelhamel@yahoo.fr

binding constant, selectivity, and sorption capacity for metal ions is important [11–15]. Several chelating matrices have been developed using resins of Amberlite XAD series as a support [16–22]. Promising ones include Amberlite XAD-2 modified with 2-aminoacetylthiophenol [23], pyrocatechol [24], alizarin red-S [25], pyrocatechol violet (PV) [26], *o*-vanillinthiosemicarbazone [27], pyrogallol [28], *o*-aminophenol [29], tiron [30], Amberlite XAD-4 modified with an acetyl group [31], Amberlite XAD-7 modified with 2-hydroxybenzaldehyde-5-nitro pyridylhydrazone [32] and Amberlite XAD-16 modified with xylenol orange [33], 4-[(2-hydroxyphenyl)imino]methyl-1,2-benzenediol [34], and nitrosonaphthol [35]. In our previous study, a new chelating Amberlite XAD-4 resin was modified and used for Zn(II) extraction [36].

In a search for environment-friendly metal chelating ligands for industrial application, spectroscopic techniques were applied to the determination of Co(II) in different media. Hyvönen and Aksela [37] studied the complex formation equilibria of *n*-bis[2-(1,2-dicarboxyethoxy)ethyl]aspartic acid (BCA6) with Co(II). The complex formation was dominated by stable  $ML^{4-}$ . Co(II) chloro complexes were studied in aprotic solvents, namely, dimethylsulfoxide (DMSO), dimethylformamide (DMF), and propylene carbonate (PC) [38]. The measurements were performed spectrophotometrically in UV-Vis-IR (IR, infrared) region. It was found that the stability constants of the identified complexes increased inversely with the Gutmann's donor number of the solvents and the symmetry of tetrachlorocobaltate was strictly  $T_d$ . The isolation of two unsymmetrical isomeric Schiff bases with ethylenediamine and their VO(IV), Co(II), Ni(II), and Cu(II) complexes were studied by Kolawole and Osowole [39]. Further reaction of each complex with triphenylphosphine resulted in the formation of 1:1 adducts, with water occupying the sixth position in some, except Cu(II) and Co(II) which formed 1:2 adducts with [ML].

Conventional extractants were also used for Co(II) extraction, in which emphasis was placed on the functional group of extractants. The extraction of Co(II) was carried out by using bis(2,4,4-trimethylpentyl)phosphinic acid [40], bis(2-ethylhexyl)phosphinic acid (PIA-8) [41], the commercial extractant Cyanex 302 (bis(2,4,4-trimethylpentyl)-monothiophosphinic acid) [42], tri-*n*-butylphosphate [43], alamine 336 [44], octanal oxime (OCOX) [45], and di-*n*-pentyl sulfoxide [46].

In this article, a chelating resin was synthesized by covalently linking PV on the benzene ring of Amberlite XAD-16 and used for the sorption of Co(II) from aqueous solutions at batch system by flame atomic absorption spectrometry (FAAS).

## 2. Experimental

### 2.1. Reagents

Stock standard solutions of Co(II) were prepared by dissolving appropriate amounts of analytical reagent grade Co(II) nitrate in doubly distilled water. The pH adjustments were made with drops of  $1 \text{ mol L}^{-1}$   $\text{HNO}_3$  and  $\text{NH}_3$ . Amberlite XAD-16 resin was obtained from Rohm and Haas Co. Before use, it was thoroughly purified with a 50% methanol–water solution containing  $1 \text{ mol L}^{-1}$  HCl in order to remove the inorganic impurities and monomeric material, and dried in air.

## 2.2. Experimental procedure

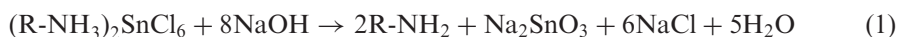
Atomic absorption spectrometry (AAS) measurements were taken with a Shimadzu model AA6500 atomic absorption spectrophotometer using air–acetylene flame. The pH measurements were made on a digital pH meter WTW Inolab pH Level 1. IR spectra (4000–400  $\text{cm}^{-1}$ ) in KBr were recorded on a Perkin Elmer FT-IR Spectrum 100. The operating conditions of the AAS instrument for the determination of Co(II) are listed in table 1.

Batch technique was used to extract Co(II). A volume of 100 mL of solution containing 10  $\text{mg L}^{-1}$  of Co(II) was used after adjusting its pH to optimum value. PV-loaded Amberlite XAD-16 (0.1 g) was added to the Co(II) solution and the heterogeneous mixture was shaken with a mechanical shaker model BS (VELP SCIENTIFICA) for 120 min at 60 rpm. The resin was filtered and the absorbed metal ion was eluted with 100 mL 0.2 mol  $\text{HNO}_3$ . The concentration of Co(II) was determined by FAAS.

## 3. Results and discussion

### 3.1. Synthesis of PV–Amberlite XAD-16 resin

Amberlite XAD-16 bead (5 g) reacted with a nitrating mixture containing 10 mL of concentrated  $\text{HNO}_3$  and 25 mL of concentrated  $\text{H}_2\text{SO}_4$  for 1 h at 60°C under stirring on an oil bath. Thereafter, the reaction mixture was poured into an ice–water mixture. The nitrated resin was filtered, washed repeatedly with distilled water until free from acid, and reduced thereafter by refluxing for 12 h at 90°C with a mixture of 40 g of  $\text{SnCl}_2$ , 45 mL of concentrated HCl, and 50 mL of ethanol. The amino-polymer was filtered off and washed with distilled water and 1 mol  $\text{L}^{-1}$  NaOH to release amino resin (R-NH<sub>2</sub>) from (R-NH<sub>3</sub>)<sub>2</sub>SnCl<sub>6</sub> (R = resin matrix) according to the following equation:



The amino resin was treated with 100 mL of 1 mol  $\text{L}^{-1}$  HCl, and washed with water to remove the excess HCl. It was suspended in 350 mL of ice–water and then diazotized with an equimolar mixture of 1 mol HCl and  $\text{NaNO}_2$  (added in small aliquots of 1 mL) below 0–5°C until the reaction mixture began to give a permanent blue color with starch-iodide paper according to the following equation:

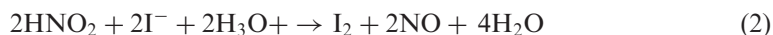


Table 1. The operating conditions of the atomic absorption spectrophotometer for determination of Co(II).

Parameters	Co(II)
HC lamp current (mA)	12
Wavelength (nm)	240.7
Sensibility ( $\text{mg L}^{-1}$ )	0.001
Flame fuel ( $\text{L min}^{-1}$ )	Air–C <sub>2</sub> H <sub>2</sub>
Oxidant ( $\text{L min}^{-1}$ )	Air
Burner (cm)	7
Debit ( $\text{L min}^{-1}$ )	2.2

The diazotized resin was filtered, washed with ice-cold water and made to react with PV (1.5 g in 400 mL of water and 100 mL of glacial acetic acid) at  $-5^{\circ}\text{C}$  for 24 h. The black beads were filtered, washed with distilled water, dried in air, and stored. The schematic reaction sequence is given in figure 1. During the various stages of the synthesis, we observed visual changes in the XAD-16 resin; the virgin resin was white, but after nitration the resin appeared orange, and then black during the grafting of the PV.

### 3.2. Analysis of IR spectra of Amberlite XAD-16 before and after grafting with the PV

Figure 2(a) shows the IR spectrum of the modified PV–Amberlite XAD-16 resin compared with the spectrum of unmodified Amberlite XAD-16 (figure 2b) and the PV (figure 2c). When considering the combined effect of two *ortho*- and *para*-directing OH groups, the coupling position of PV with the polymer matrix, as shown in figure 1, arises from electronic effects and steric reasons. There are three additional bands at 1661, 1669 ( $-\text{C}=\text{C}-\text{N}-$ ), and  $1575\text{ cm}^{-1}$  ( $-\text{N}=\text{N}-$ ), respectively, which do not appear in the spectra of Amberlite XAD-16 and PV. The appearance of these bands indicates chemical modification of XAD-16 by grafting of the PV on the benzene cores of XAD-16 polymeric support. Saxena and Singh [26] proposed the same formula during the grafting of the PV on the Amberlite XAD-2 resin.

### 3.3. Effect of pH of aqueous solutions on the extraction rate of Co(II)

The degree of Co(II) absorption at different pH values was determined by batch equilibration. A set of solutions (100 mL) containing  $10\text{ mg L}^{-1}$  of Co(II) were adjusted in the pH range 1.0–6.0 by adding drops of  $1\text{ mol L}^{-1}$  HCl or  $\text{NH}_3$  solutions.

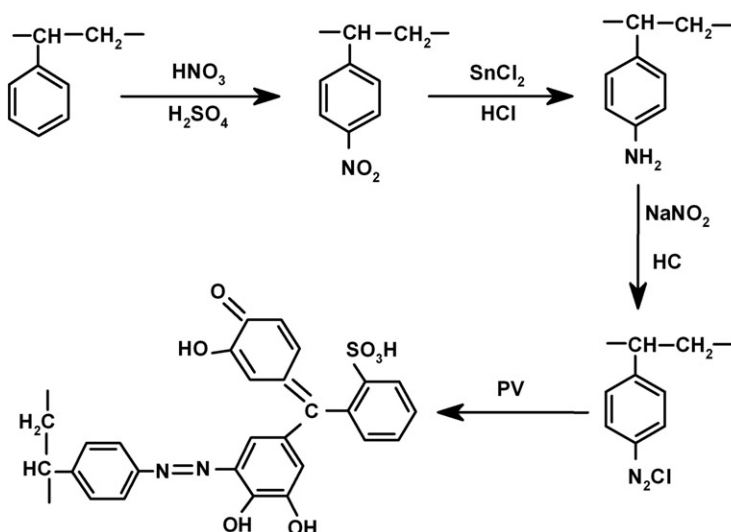


Figure 1. Scheme of the synthesis of PV-loaded Amberlite XAD-16 resin.

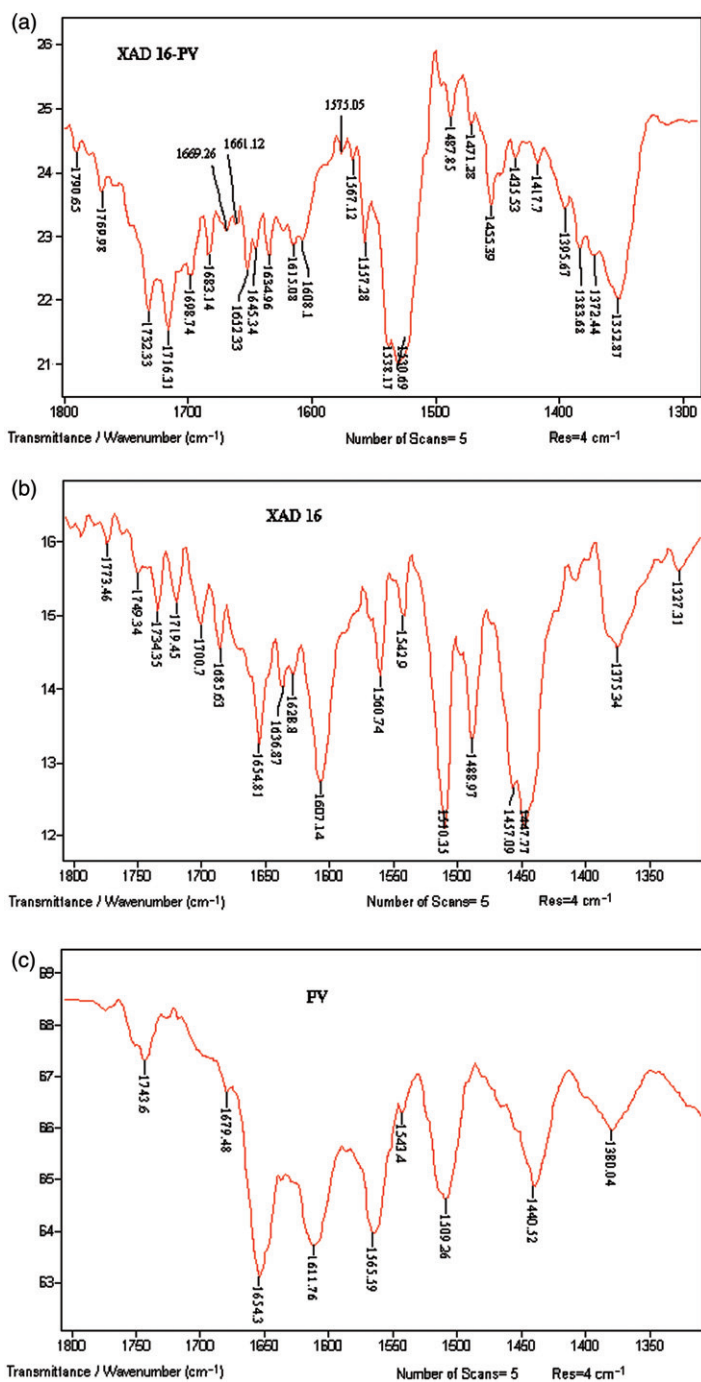


Figure 2. IR spectra. (a) Spectrum of PV-loaded Amberlite XAD-16 resin. (b) Spectrum of virgin resin XAD-16. (c) Spectrum of PV.

Modified Amberlite XAD-16 resin (0.1 g) was added to each solution and the mixture was shaken for 2 h. The uptake of Co(II) was ascertained by the determination of the metal content in the supernatant liquid by FAAS. As shown in figure 3, the extraction efficiency of Co(II) increased with decreasing pH of the aqueous solution. The extraction is indeed slower for pH less than 1.0 because cation exchange becomes more difficult when the acidity increases. As a result, maximum recovery of cobalt in the pH range 4.15–5.10 was 99.36%.

### 3.4. Kinetics of Co(II) absorption on PV functionalized Amberlite XAD-16

The absorption rates of Co(II) on virgin resin and on the violet functionalized Amberlite XAD-16 were determined by batch experiments. Therefore, 0.1 g of resin beads were stirred with 100 mL solution containing  $10 \text{ mg L}^{-1}$  of Co(II) for different equilibration times: 5, 10, 15, 20, 30, 40, 50, 60, 80, 100, and 120 min under optimum conditions. The concentration of Co(II) absorbed on functionalized Amberlite XAD-16 as well as that present in the supernatant solution was determined with FAAS. The absorption rate of Co(II) as a function of time is shown in figure 4. Co(II) ions were extracted by the virgin resin XAD-16 at very low efficiency, although there was a slight shoulder of the absorption rate at the beginning of contact, which dissipated with time. The kinetics of the PV–Amberlite XAD-16 resin and metal ion interaction is sufficiently rapid at pH less than 3. The faster uptake of Co(II) onto PV–Amberlite XAD-16 reflects good accessibility of the site of the chelating resin.

### 3.5. The absorption and desorption of Co(II) onto chelating PV–Amberlite XAD-16 resin

The absorption of Co(II) onto PV-XAD-16 resin was determined by batch method. Chelating resin (0.1 g) was mixed with 100 mL of solutions containing Co(II) at various

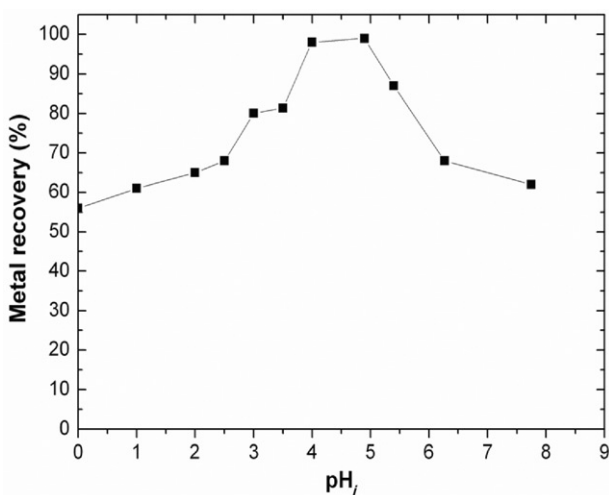


Figure 3. Effect of pH on the extraction rate of Co(II), stirring time: 120 min,  $[\text{Co(II)}] = 10 \text{ mg L}^{-1}$ , resin amount: 0.1 g, sample volume: 100 mL.

concentrations and shaken for 1 h under optimum conditions. The solid matrix was filtered, washed with distilled water, and subjected to the recommended batch procedure. The filtrate was diluted suitably and also subjected to FAAS determination. As shown in figure 5, the sorption capacity of the chelating resin increases with increasing concentration of Co(II) in aqueous phase during the first minutes of contact. High efficiency was found by using a solution containing  $10 \text{ mg L}^{-1}$  of Co(II). The optimum sorption capacity is  $99.36 \text{ mg g}^{-1}$  of resin.

The absorption of Co(II) on the resin is always followed by a desorption, which is very important because it allows the recovery of metal ions adsorbed and the recycling of the resin. The desorption of the retained Co(II) from the PV-Amberlite XAD-16 resin was tested using nitric acid ( $\text{HNO}_3$ ) at 0.1, 0.2, 0.3, and  $0.5 \text{ mol L}^{-1}$ . The variation of desorption with acid concentration was investigated and the optimum acid concentration for maximum recovery was  $0.2 \text{ mol L}^{-1}$ .

The effect of the amount of PV-XAD-16 resin on the recovery of Co(II) was studied by varying the quantity of chelating resin in the range 50–500 mg. The concentration of Co(II) in the aqueous phase was  $10 \text{ mg L}^{-1}$  at  $\text{pH} = 4.80$  (figure 6). Recovery of Co(II) gradually increased to 0.1 g of adsorbent and then remained unchanged. As a result, 0.1 g of chelating resin was found to be optimum and used for subsequent experiments.

To test the PV-XAD-16 resin recycling, it was subjected to several loading and elution by batch method. The resin beads (0.1 g) were stirred with 100 mL solution containing  $10 \text{ mg L}^{-1}$  of Co(II) for 1 h under optimum conditions (table 2). The chelating resin was filtered off and treated with 50 mL of  $0.2 \text{ mol L}^{-1} \text{ HNO}_3$ . The desorbed Co(II) content was estimated as described in the procedure. The absorption and desorption were repeated on the same resin beads and the absorption capacity was estimated after each cycle of operation. The capacity of the resin was also found to be practically constant after use for more than five times, as shown in figure 7.

The effect of various electrolytes like  $\text{NaNO}_3$ ,  $\text{KNO}_3$ , and  $\text{Ca}(\text{NO}_3)_2$  on the absorption of Co(II) using PV-Amberlite XAD-16 resin matrix was studied with

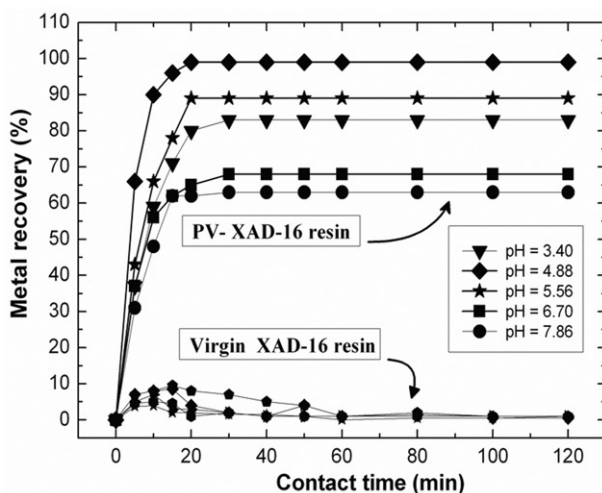


Figure 4. Kinetics of absorption of Co(II) on PV-functionalized Amberlite XAD-16,  $\text{pH}$ : (▼) 3.40, (◆) 4.88, (★) 5.56, (■) 6.70, (●) 7.86,  $[\text{Co}(\text{II})] = 10 \text{ mg L}^{-1}$ , resin amount: 0.1 g, sample volume: 100 mL.



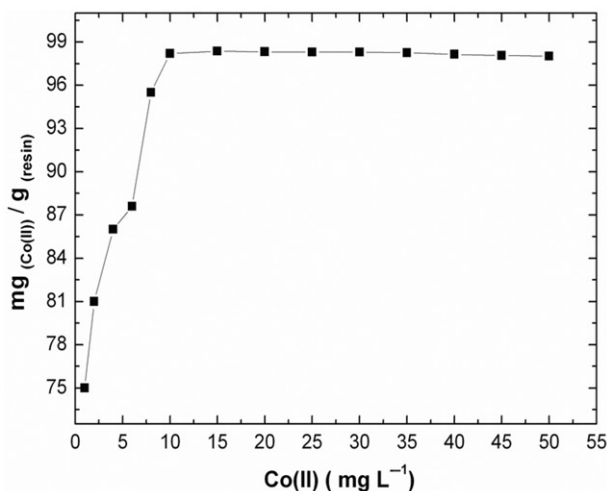


Figure 5. Absorption capacity of the chelating resin at various concentrations of Co(II).

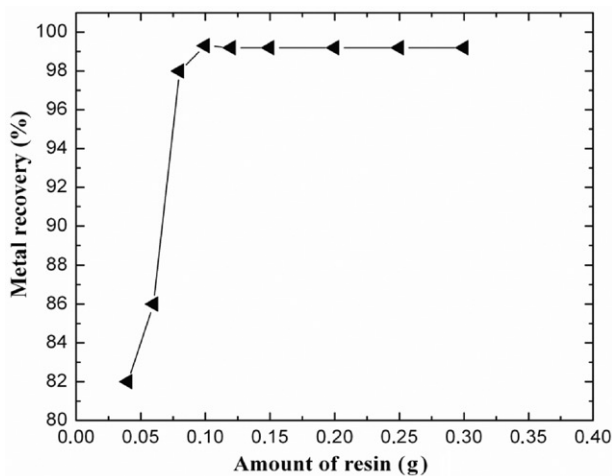


Figure 6. Effect of the amount of the chelating PV-XAD-16 resin on the recovery of Co(II), pH=4.5: [Co(II)]=10 mg L<sup>-1</sup>, sample volume 100 mL.

100 mL of solutions containing 10 mg L<sup>-1</sup> of Co(II) and the electrolyte salt at various concentrations. Co(II) was absorbed on the chelating resin, desorbed with HNO<sub>3</sub>, and determined by the recommended procedure. The results are reported in table 3. The rate of absorption decreases with increase in the concentration of salts, suggesting that there may be a competitive extraction between cations of these salts and Co(II) ions. These electrolytes are tolerable up to 10<sup>-3</sup> mol L<sup>-1</sup> for the absorption of Co(II) at pH 4.80.

Modified Amberlite XAD-16 was used to preconcentrate and determine Co(II) in a river water sample from Bejaia city. The main origin of Co(II) in river water is probably from the geological structure of the region of Bejaia with the river crossing

Table 2. Optimum conditions for absorption and desorption of Co(II) on PV-loaded Amberlite XAD-16.

Experimental parameters for absorption and desorption of Co(II)	
pH range	4.15–5.10
HNO <sub>3</sub> concentration for desorption (mol)	0.2
Capacity of absorption (mg g <sup>-1</sup> resin)	99.36
SD <sup>a</sup> (mg g <sup>-1</sup> )	0.72

<sup>a</sup>For five determinations.

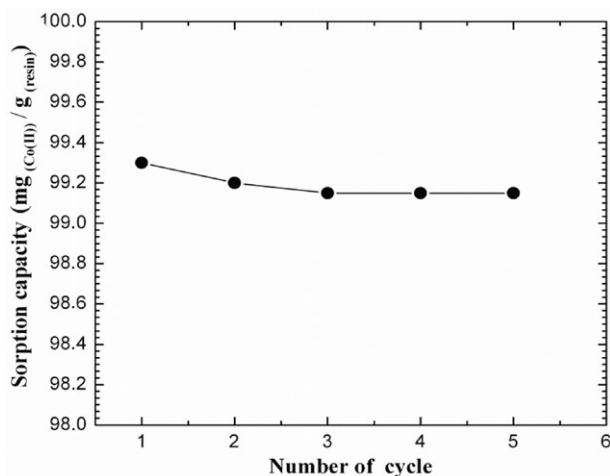


Figure 7. Effect of reprocessing of PV-loaded Amberlite XAD-16 resin on Co(II) absorption, stirring time: 60 min, [Co(II)] = 10 mg L<sup>-1</sup>, resin amount: 0.1 g, sample volume: 100 mL.

Table 3. The effect of electrolytes salts, NaNO<sub>3</sub>, KNO<sub>3</sub>, and Ca(NO<sub>3</sub>)<sub>2</sub>, on absorption capacity of Co(II) using PV-Amberlite XAD-16 resin.

Electrolyte	Absorption efficiency of Co(II)	
	(mol L <sup>-1</sup> )	(%)
NaNO <sub>3</sub>	10 <sup>-3</sup>	91.63
	10 <sup>-2</sup>	89.53
	0.1	86.39
KNO <sub>3</sub>	10 <sup>-3</sup>	89.98
	10 <sup>-2</sup>	81.15
	0.1	79.12
Ca(NO <sub>3</sub> ) <sub>2</sub>	10 <sup>-3</sup>	79.67
	10 <sup>-2</sup>	76.94
	0.1	71.23
Without ionic strength		99.36

a lead–zinc mine. The water sample was filtered to remove suspended particles. Co(II) concentration of the river water was 9.29 μg g<sup>-1</sup>. To understand the sorption ability of PV-Amberlite XAD-16 resin, the estimation of Co(II) ions were made by passing 1000 mL of water sample through the column packed with 2 g of PV-Amberlite

XAD-16 resin and determining the metal ions by FAAS after elution, as described in Section 2.2. The amount of Co(II) ions found was  $0.30 \mu\text{g g}^{-1} \pm 0.5\%$  and the recovery was 96.77%, with relative standard deviation lower than 5%.

### 3.6. Optimum conditions

The optimum conditions were experimentally determined from the effects of the parameters studied as follows: pH range: 4.15–5.10; sorption capacity of chelating resin:  $99.36 \text{ mg g}^{-1}$  of resin; stripping solution; concentration of nitric acid used for desorption:  $0.2 \text{ mol L}^{-1} \text{ HNO}_3$ ; time of Co(II) sorption: 40 min; concentration of electrolyte salts:  $10^{-3} \text{ mol L}^{-1}$ .

## 4. Conclusion

A chelating resin was synthesized by covalently linking PV with the benzene ring of Amberlite XAD-16 through an azo ( $-\text{N}=\text{N}-$ ) spacer. The grafting of the PV on the benzene cores of XAD-16 polymeric support was characterized by IR spectra. The sorption of Co(II) from nitrate solution on chelating PV–Amberlite XAD-16 resin is simple, fast, and economical. The extraction of Co(II) ions was slower for pH less than 1.0, with a maximum recovery of 99.36% of cobalt at pH of 4.15–5.10. The chelating resin has fast rate of equilibrium and high stability with lower matrix interference compared to analogous resins reported in [47, 48]. The proposed separation-enrichment method was applied for atomic absorption spectrometric determinations of Co(II) in river water with satisfactory results (recovery greater than 96.77%, relative standard deviations lower than 5%).

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