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The Removal of Cd and Cu from Aqueous Solution Using Sorbents Siirt Peanut Shells Immobilized on Amberlite XAD-4

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ABSTRACT

In this study, sorbents as Siirt peanut shell with Amberlite XAD-4 immobilized on polymers of Cd (II) and Cu(II) ions were investigated conditions of enrichment by solid phase extraction method. The effect of the recovery yield of the ambient pH, eluent type and concentration, solution flow rate, the effect of solution volume, salt effect, column repeatability, examined the application of certified reference material and environmental samples. Improved method has provided 60 times enrichment for Cu and Cd. Under optimum conditions, Cu and Cd recovery yields were found to be Cu 98.43 ± 3.76 and Cd 98.76 ± 4.11 for the 95% confidence level. Besides, the relative standard deviation of Cu and Cd were determined to be 6.51 and 7.11 % for 3 repeat experiments.

Keywords: Amberlite XAD-4, solid phase extraction, trace element, Cu, Cd

INTRODUCTION

Heavy metals have been excessively released into the environment due to rapid industrialization and have created a major global concern. Cadmium, zinc, copper, nickel, lead, mercury and chromium are often detected in industrial wastewaters, which originate from metal plating, mining activities, smelting, battery manufacture, tanneries, petroleum refining, paint manufacture, pesticides, pigment manufacture, printing and photographic industries, etc., (Wan Ngah & Hanafiah 2008). Heavy metal pollution in the developing world, is a significant risk to public health and natural life everyday expand coverage, the effects are increasing. The existence of these elements even in very small quantities is important for the environment and human health, it is known that excessive amounts cause toxicity acute and chronic (Nazlı et al. 2014). Trace elements to the enrichment method used in the analysis, solid phase extraction (Chen et al. 2009), the liquid-liquid extraction (Abkenar et al. 2010), co-precipitation (Feist & Mikula 2014), ion exchange and cloud point extraction can be mentioned. This method of solid phase extraction, use of less organic solvents, having high enrichment factor, fast and easy implementation, can be run with larger volumes, it is widely used because of no possibility of solid phase selection in very different properties (Kara et al. 2009; Chen et al. 2009). XAD resins (Ghaedi et al. 2009; Karadaş & Kara 2013; Ahmad et al. 2015; Ahmad et al. 2013; Islam et al. 2013), activated carbon (Gil et al. 2006), silica gel (Alan et al. 2007), ion exchange resins (Tejero et al. 2016), chelating resins (Martins et al. 2005; Islam et al. 2012; Ahmad 2012). The different characteristics and types the extraction materials, such as solid phase it is possible to encounter.

Flame atomic absorption spectrometry (FAAS) (Martins et al. 2005; Bermejo-Barrera et al. 2003; Islam, Aminul; Ahmad, Akil; Laskar 2015), electrothermal atomic absorption spectroscopy (ETAAS), inductively coupled plasma-optical emission spectroscopy (ICP-OES), inductively coupled plasma-mass spectroscopy (ICP-MS), are the analytical techniques commonly used in the analysis of heavy metals.

The aim of this study, sorbents as Siirt peanut shell immobilized on Amberlite XAD-4 on polymers of Cd (II) and Cu (II) ions were investigated conditions of enrichment by solid phase extraction method. The pH of the yield of recovery of the worked metal ion environment, eluent type and concentration, solution flow rate, the effect of solution volume, salt effect, column reproducibility, certified reference materials (Aquatic Plant Sample) and examined its application to environmental samples.

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Table 1. The operating conditions of atomic absorption spectrophotometer

	Cd^2	**Cu^{2+}**
Wavelength (nm)	228.8-326.1	216.5-327.4
Acetylene Flow Rate (L/min)	1.8	2.0
Relative Noise	0.9-1.0	1.0-7.2
Concentration (mg/L)	0.028-11. 0	0.077-5.80

Table 2. The study presented Analytical Characteristics of the metal ions

Method	**Element**	**Linear**	**Slope**	**Intercept**	**R^2**	**Regression Equation**
AAS	Cd^{2+}	0.028-11	0.09157	0.001137	0.9975	A=0.1841x + 0.0023
AAS	Cu^{2+}	0.077-5.8	0.06119	-0.00191	0.9998	A=0.1339x + 0.0119

A= Absorbance, BSS= Relative Standard Deviation, C=Concentration

Table 3. Microwave oven heating program for the decomposition of BCR-670 Aquatic Plant Sample certified standard reference material

Step	**1**	**2**
T (°C)	150	190
P (bar)	50	50
Power (%)	70	90
Ta (min)	5	1
Time (min)	10	10

EXPERIMENTAL

Used Equipment and Working Conditions

Analyst 700 Perkin Elmer atomic absorption spectrometry was used to determine the concentration of metal ions. HANNA Instruments HI 2211 pH / ORP Meter pH meters were used to adjust the pH of the solutions. Filtration columns (1.0 cm x10.0 cm) equipped with polypropylene frites were used for solid-phase extraction (SPE). A Watson Marlow 120 S model peristaltic pump was used to enable the solutions to pass through the column. In the study, the operating conditions of atomic absorption spectrophotometer and analytical characteristics of the metal ions are given in **Table 1** and **Table 2**.

During the study, cadmium and copper solutions on a daily, was prepared by diluting from the stock standard solution (Merck 1000 mg/L). BCR-670 Aquatic Plant Sample certified standard reference material (SRM) sample was solubilized using Berghof Speed Wave (MWS-3) microwave device. Operating conditions of microwave digestion system are given in **Table 3**. For acid digestion, approximately 0.50 g of BCR-670 Aquatic Plant Sample certified standard reference material was weighed accurately into a PTFE digestion vessel. 10 mL of concentrated HNO_3 and 2 mL of concentrated H_2O_2 were added to the vessel and waited for about 25 minutes before the vessel is closed. Decomposition of the sample was carried out in a microwave digestion system. As shown in **Table 3**, microwave oven heating program was applied to the Aquatic Plant Sample certified standard reference material. Then the residue was dissolved in Milli-Q water and filtered, and the filtrate was diluted to 50 mL.

The glassware used in the studies respectively washed by detergent and tap water, 1: 1 HNO_3 and tap water, 1: 1 HCl and tap water and finally washed thoroughly with deionized water After rinsed and dried in the oven was ready processing.

Preparation of Adsorbent

Amberlite XAD-4 (polystyrene divinyl benzene) was obtained from Sigma Aldrich. This resin was treated with 4 M HCl for resin modifier. Then, the resin was rinsed with distilled water until its pH level was neutral, followed with a rinsing in an ethanol–water (1:1) solution. Finally, it was washed with distilled water again and was then stored in a polyethylene bottle (Özdemir et al. 2012).

Preparation of Sorbents

Siirt peanut shells used as sorbents milled with memert brand mill. It passed through a -60 mesh screen. Sorbents were washed with distilled water, then dried in oven at 80 ^{0}C. It passed again to -60 mesh sieve.

Table 4. BET image of the colon filler material

Surface area

Single point surface area, P/Po = 0.349447984: 603.4616 m²/g

BET Surface area: 394.2622 m²/g

Pore Volume

Less Porous Single Point in Adsorption Total Pore Volume: 1176.537 Å

Less Porous Single Point in Adsorption Total Width P/Po = 0.983265783: 0.992526 cm³/g

Pore Size

Adsorption mean pore width (4V/A BET) : 100.6971 Å

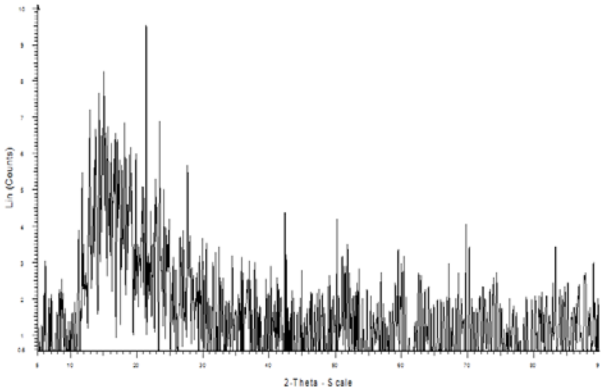

Figure 1. XRD image of the colon filler material

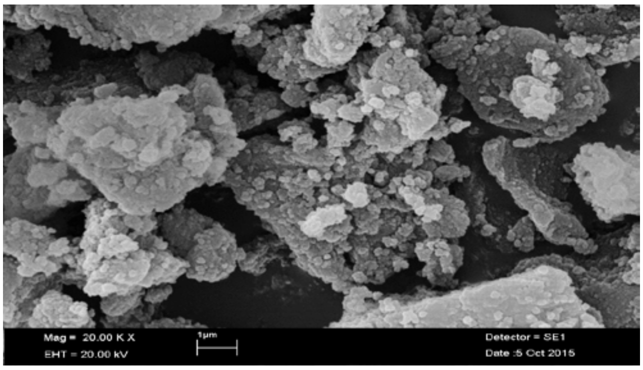

Figure 2. SEM image of the colon filler material

Preparation of SPE Columns

A Siirt peanut shell as sorbents was collected from Siirt area. Siirt peanut shells powder 1.00 g of Amberlite XAD-4 and 0.250 g Peanut was stirred with a magnetic stirrer IKA Werke RT-10P brand for 60 minutes with 10 ml of pure water. The resulting mixture is dried in an oven at 105 °C. The dried mixture was passed through a -60 mesh screen again and then was packed in a solid-phase column (1.0 cm x10.0 cm).

Column Characterization of Filler Material

XRD, SEM, FTIR, TGA and BET analysis of filler material were carried out and the obtained results are given in Table 4 and Figures 1-5.

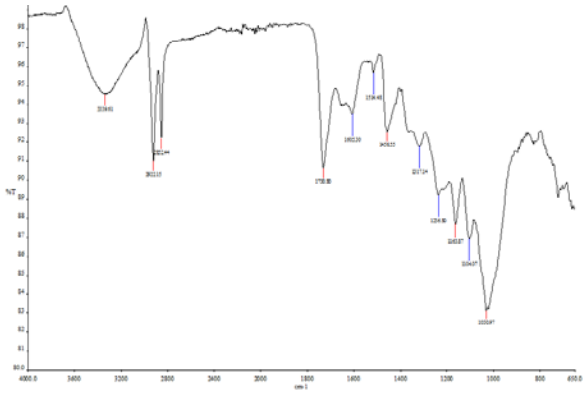

Figure 3. FT IR image of Siirt peanut shell

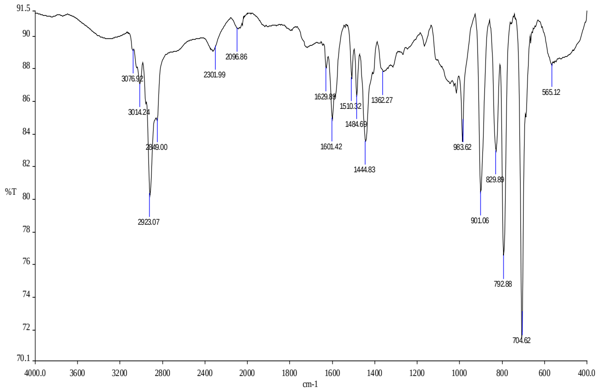

Figure 4. FT IR image of Siirt peanut shells immobilized on Amberlite XAD-4

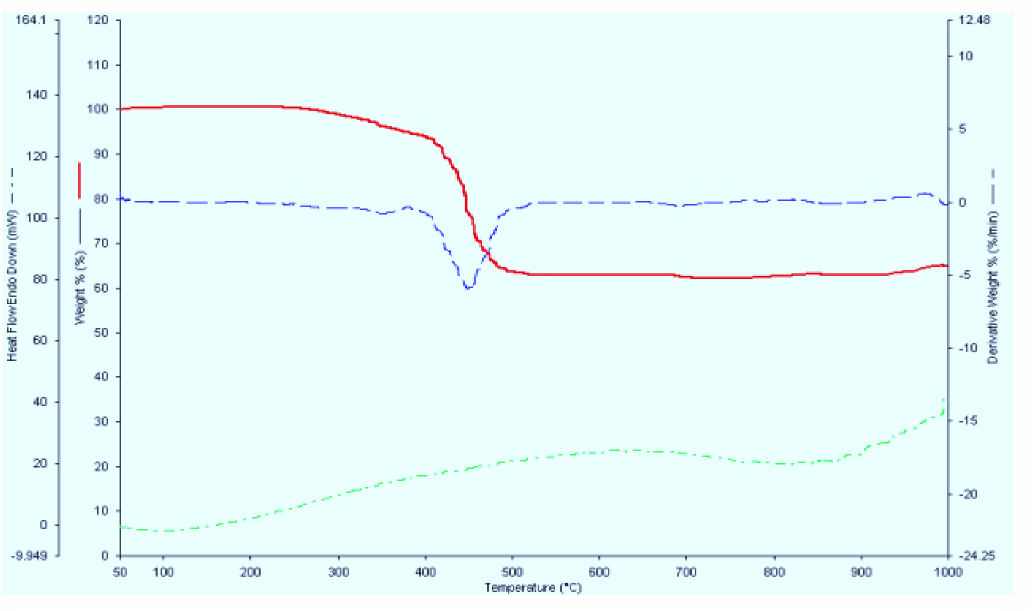

Figure 5. TGA image of the colon filler material

General sorption studies for Cd and Cu

The 50.0 mL model solutions containing 2.0 mg mL^{-1} of Cd^{2+}and Co^{2+} were prepared by diluting the stock solution of metals. The pH of the model solution was adjusted to the desired value, and then, this solution was passed through the solid-phase extraction column by a peristaltic pump. Then, about 10.0 mL of distilled water was passed through the column. The retained Cd^{2+} and Cu^{2+} ions on the biosorbents were then eluted from the column

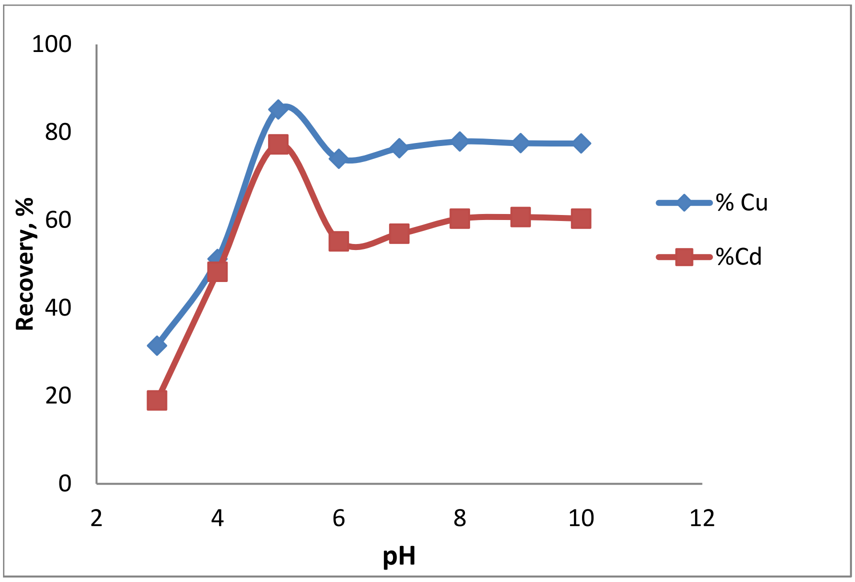

Figure 6. Effect of pH on solid phase extractions of Cu^{2+} and Cd^{2+}

with 5.0 mL of 1.0 mol L^{-1} HCl or HNO_3. The concentrations amount of the Cu^{2+} and Cd^{2+} ions in solution were simultaneously determined by AAS (Özdemir et al. 2012).

RESULTS AND DISCUSSION

Effect of pH on Recovery

The effect of pH on the recovery of Cu^{2+} and Cd^{2+} onto Siirt peanut shells immobilized on Amberlite XAD-4 was examined in the pH range of 3.0–10.0. For this purpose, column experiments were performed by using 50.0 mL of test solutions containing 2.0 mg/mL Cu^{2+} and Cd^{2+} ions. The effects of the initial pH on the sorbents of Cu^{2+} and Cd^{2+} ions in aqueous solution are given **Figure 6**. The removal efficiencies of Cu^{2+} and Cd^{2+} ions in the solution at a pH of 3.0 were found to be 31.45% and 18.96%, respectively, and they increased when the solution pH increased from 2.0 to 5.0. At a pH of 5.0, the removal rates of Cu^{2+} and Cd^{2+} ions were 85.20% and 77.24%, respectively. Thus, a pH of 5.0 was selected for Cu^{2+} and Cd^{2+}. Maximum biosorption capacities were also calculated at a pH of 5.0 Cu^{2+} and Cd^{2+}.

Effect of Flow Rate of Sample Solution on Recovery

The influence of the flow rate of the sample solution on Cu^{2+} and Cd^{2+} biosorption onto Siirt peanut shells immobilized on Amberlite XAD-4 was investigated in the range of 1.0– 8.0 mL min^{-1}. Recovery results depend on the flow rate of the solution and are displayed in **Figure 7**. At a flow rate of 2.90 mL min^{-1} for Cu^{2+} ions and At a flow rate of 4.55 mL min^{-1} for Cd^{2+} ions, recoveries of Cu^{2+} and Cd^{2+} ions were found to be 82.5 % and 100.0%, respectively.

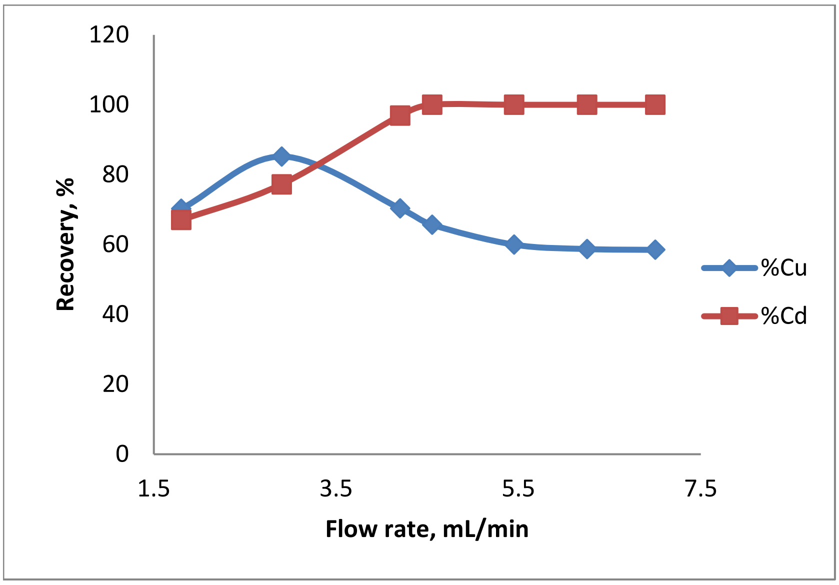

Figure 7. Effect of flow rate of sample solution on solid phase extractions of Cu^{2+} and Cd^{2+}

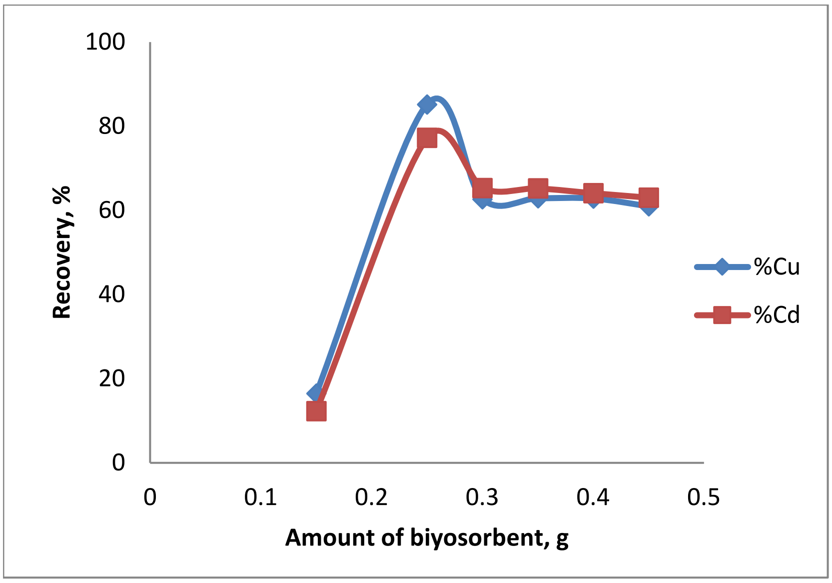

Figure 8. Effect of amount of biosorbent on solid phase extractions of Cu^{2+} and Cd^{2+}

Effect of Amounts of Siirt Peanut Shells Immobilized on Amberlite XAD-4 on Metal Ion Recoveries

The effects of the amounts of Siirt peanut shells on the biosorption of Cu^{2+} and Cd^{2+} ions were studied at a sample flow rate of 2.9 mL $min^{-1}$for Cu^{2+} and 5.55 mL min^{-1} for Cd^{2+} and at their individual pH values, respectively. The different amounts of Siirt peanut shells (100.0–450.0 mg) for the solid-phase extraction process were examined in the recoveries of Cu^{2+} and Cd^{2+} ions. As seen in **Figure 8**, the recovery values of Cu^{2+} and Cd^{2+} ions increased by increasing the amounts of Siirt peanut shells added and reached a constant value with at least 300.0 mg of the biosorbents. **Figure 7** is examined it will be seen as adequate for the amount of 250 mg sorbents. Recoveries of Cu^{2+} and Cd^{2+} ions were found to be 82.5 % and 77.24%, respectively.

The Effects of the Amounts of Amberlite XAD-4 on Solid-Phase Extractions

The effects of the amounts of Amberlite XAD-4 on solid-phase extraction were also examined. The results obtained are given in **Figure 9**. The amount of Amberlite XAD-4 was increased from 250.0 mg to 1000.0 mg and

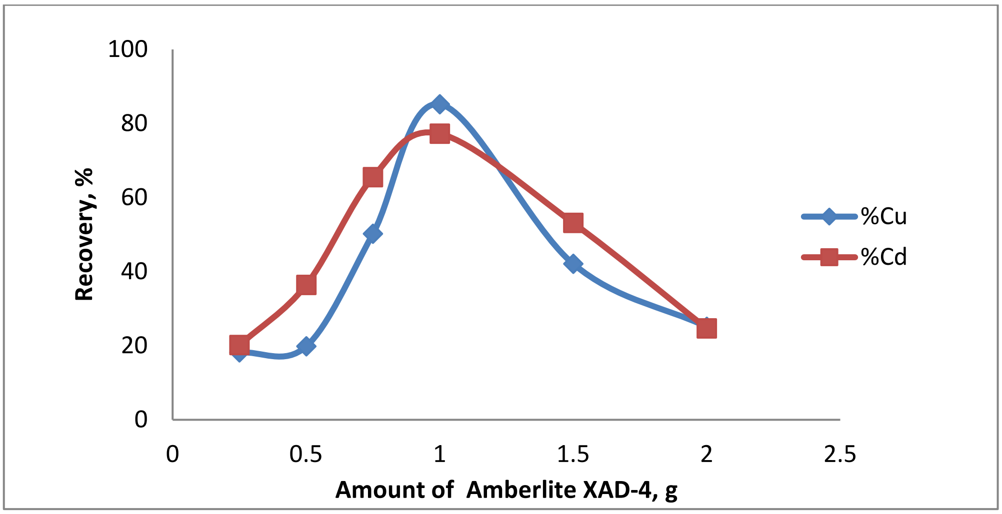

Figure 9. Effect of amount of Amberlite XAD-4 on solid phase extractions of Cu^{2+} and Cd^{2+}

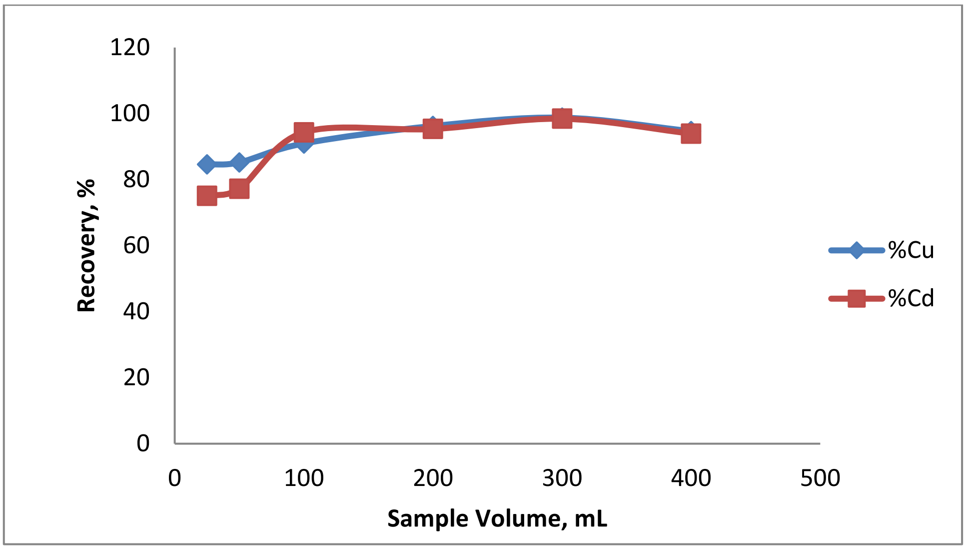

Figure 10. Effect of volume of sample solution on solid phase extractions of Cu^{2+} and Cd^{2+}

then decreased. The optimal recoveries were 1000.0 mg resin. Hence, 1000.0 mg of Amberlite XAD-4 was used for further SPE experiments.

Effect of Sample Volume on the Solid-Phase Extraction (SPE) Procedure

The effects of sample volume on the retention of Cu^{2+} and Cd^{2+} ions onto Siirt peanut shells immobilized on Amberlite XAD-4 were studied by changing the sample volume from 25.0 mL to 500.0 mL. The concentrations of Cu^{2+} and Cd^{2+} ions were kept constant at 2.0 µg mL^{-1}. The results are given in **Figure 10**. In approximately 300.0 mL of the sample solution, recoveries of Cu^{2+} and Cd^{2+} ions were found to be 98.76 % and 98.43%, respectively. When sample volume was increased, the recoveries of Cu^{2+} and Cd^{2+} ions were decreased.

Effect of Type and Volume of Eluent on Recovery

HCI and HNO_3 were tested for optimal eluent species for desorption of Cu^{2+} and Cd^{2+} ions from the solid-phase extraction column. As can be seen in **Table 5**, it is seen that 5.0 mL of 0.5 mol L^{-1} HNO_3 is sufficient.

Table 5. Effect of type and volume of elution solutions on the recovery of Cu^{2+} and Cd^{2+}(N=3)

Type of elution solution	Concentration (molL^{-1})	Elution Volume (mL)	Recovery (%)	
			Cu^{2+}	Cd^{2+}
HCL	0.5	5	91.17	75.57
	1.0	5	92.81	94.78
	1.5	5	94.66	94.56
	2.0	5	94.51	93.77
HNO_3	0.5	5	95.57	95.82
	1.0	5	93.76	89.71
	1.5	5	93.24	85.81
	2.0	5	90.6	85.45

Table 6. The effect of foreign ions on recovery metal ions

Ions	Added Salts	Concentration (mgL^{-1})	Recovery (%)	
			Cu^{2+}	Cd^{2+}
Na^+	NaCl	10000	96.33 ± 1.85	95.67 ± 1.78
Cl^-	$BaCl_2$. $2H_2O$	20	96.22 ± 1.91	85.54 ± 1.96
SO_4^{-2}	$MnS0_4.H_2O$	50	96.13 ± 1.97	88.42 ± 1.17
Mg^{2+}	$Mg(NO_3)_3.6\ H_2O$	5000	95.95 ± 1.89	89.62 ± 1.45
Ca^{2+}	$CaCl_2.2H_2O$	3000	95.65 ± 1.96	87.95 ± 1.13
F^-	KF	1000	95.44 ± 1.83	85.25 ± 1.78

The Effect of Foreign Ions

The determination of trace metals in real sample is difficult due to the matrix effect of foreign ions. The effect of foreign ions which interfere with the determination of analytes by the presented method often accompany them was examined with the optimized conditions to assess the possible applications of the procedure. Various salts and metal ions were added individually to a solution containing Cu^{2+} and Cd^{2+} and the results are summarized in **Table 6**. The recoveries of analytes were higher than >95% for $Cu^{2+}$and>85% for Cd^{2+}. Tolerable limit was defined as the highest amount of foreign ions that produced an error not exceeding 2% in the determination of investigated analytes ions by the combination of presented procedure and the flame atomic absorption spectrometric determination methods (Saygi et al. 2008).

Repeatability Column

The stability of the solid-phase extraction column was tested using 2.0 mg Cu^{2+} and Cd^{2+} ions, maintaining a sample volume of 50.0 mL. The biosorbed Cu^{2+} and Cd^{2+} ions were eluted with 5.0 mL of 5.5 mol L^{-1} HNO_3 solution. It can be seen from **Figure 11** that the column was stable for approximately 10 cycles and could be used with good, susceptible, and quantitative recoveries (85%) for 10 cycles.

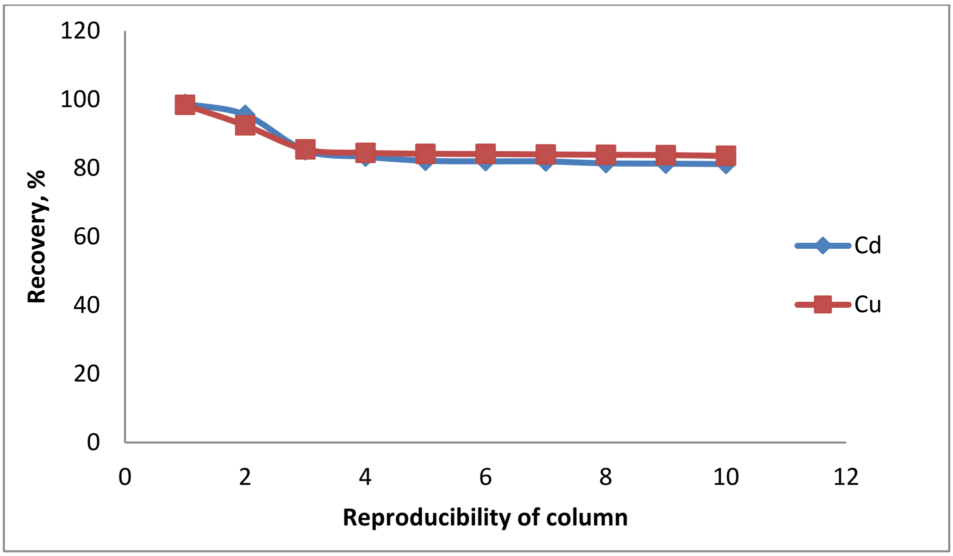

Figure 11. Reproducibility of column for solid phase extraction of Cu^{2+} and Cd^{2+}

Table 7. Accuracy Results

Element	(%), R± t s/√N	Relative Standard Deviation %
Cd	98.76 ± 4.11	7.11
Cu	98.43 ± 3.76	6.51

95% confidence Level N= 3 RSD: Relative Standard Deviation

Table 8. Application of the method to Certified Reference Material (BCR-670 Aquatic Plant Sample)

Metal ions	Certified ($\mu g g^{-1}$)	Found ($\mu g g^{-1}$)
Cu^{2+}	75.5 ± 2.50	74.9 ± 0.34
Cd^{2+}	1.82 ± 0.30	1.76 ± 0.46

95% confidence N= 3 (x ± ts/√N)

Limit of Detection and Qualification

The limit of detection (LOD) and limit of qualification (LOQ) of the present work were calculated under optimal experimental conditions after application of the preconcentration procedure to blank solutions. The observable limit values based on three times the standard deviation of the blind values (k=3, N=12) were calculated by dividing by the enrichment factor (Erdoğan 2005). The limit of detection for Cu^{2+} and Cd^{2+} were 3.20 ngL^{-1} and 2.80 ngL^{-1}, respectively. Limit of qualification (LOQ) values of Cu^{2+} and Cd^{2+}were found as 9.70 ngL^{-1} and 8.60 ngL^{-1}. It was observed that this study performed similar to previous studies (Bermejo-Barrera et al. 2003).

Analytical Features and Applicability of Methods

After determining optimum conditions for enrichment, the accuracy of the method were investigated. The results obtained are given in **Table 7**. Relative standard deviations (RSD) were found to be 7.11 and 6.51%, respectively, for Cu^{2+} and Cd^{2+}. Regression coefficients were found to be higher than 0.9975. The preconcentration factor was calculated to be 60 from the ratio of the initial to the final sample volume.

The method was applied to certified reference BCR-670 Aquatic Plant samples. The results were given in **Table 8**. The results were in good agreement with the certified values for Cu^{2+} and Cd^{2+}.

The presented method was applied to the waste water of Siirt, tap water, Tuzkuyusu salty water and Billoris spa water. The results were given in **Table 9**. These results confirmed the validity of the proposed method.

Table 9. Application of the method to real samples

Samples	Recovery (%)	
	Cd^{2+}	Cu^{2+}
Waste water	n.d.	14.95 ± 1.4
Tap water	n.d.	99.92 ± 1.2
Salty water	n.d.	34.81 ± 1.6
Biloris spa water	n.d.	28.61 ± 1.8

95% confidence N= 3 (x ± ts/√N), n.d: Not detected.

CONCLUSIONS

This preconcentration method based on the use of Siirt peanut shells immobilized on Amberlite XAD-4 as a biosorbents for solid-phase extraction was developed for Cu2+ and Cd2+ ions. It was found that both Cu^{2+} and Cd^{2+} ions were sorbed on a column at a pH of 5.0. In addition, the experimental parameters such as the effect of the recovery yield of the ambient pH, eluent type and concentration, solution flow rate, the effect of solution volume, salt effect, column repeatability were also optimized. The applicability of the method was validated through the analysis of certified reference BCR-670 Aquatic Plant. Results showed that founded concentrations for Cu2+ and Cd2+is similar with certified values. The values were found to be parallel to the studies in the literature (Bermejo-Barrera et al. 2003).

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