



*Anal. Bioanal. Chem. Res., Vol. 9, No. 2, 133-140, April 2022.*

## A Green Approach for Micro Determination of Silver(I) in Water and Soil Samples Using Vitamin C

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(Received 16 March 2021 Accepted 28 October 2021)

A cloud point extraction/solvation method (CPE-SM) based on the use of vitamin C as a chelating agent was developed for the separation and determination of silver in environmental samples. TritonX-100 was used to extract silver ions in aqueous solutions after forming solvation species with vitamin C in the presence of salting-out (0.5 M KNO<sub>3</sub>) at 90 °C for 10 min. This study evaluated the main parameters affecting the CPE-SM method, such as the kind and concentration of the salting-out, organic reagents, silver ion, temperature, heating time, and volume of surfactant. Under the optimized experimental factors, the calibration curve was linear in the range (0.1-10 µg l<sup>-1</sup>). The relative standard deviation (RSD) was 1.1-2.9% for n:5 at 1,4,9 µg l<sup>-1</sup>, a low limit of detection (LOD), and a limit of quantification (LOQ) of 0.035, 0.116 µg l<sup>-1</sup>, respectively. The accuracy of the method was evaluated by two independent techniques (flame atomic absorption spectrometry-FAAS, and spectrophotometry-using dithizone as a complexing agent). The CPE-SM technique was used to preconcentrate and determine the silver ions in river water, tap water, and soil samples, and the results were favorable and good.

**Keywords:** Cloud point extraction, Silver, Solvation method, Solvent extraction, Vitamin C

### INTRODUCTION

Scientists experience significant difficulty in developing a non-polluting, accurate, economical, and simple approach for the separation of different analytes, like trace elements in real samples [1,2]. Many extraction techniques have been used to separate and determine heavy and trace metals [3,4], such as column extraction [5], solid-phase extraction [6,7], solvent extraction [8-12], solvation method [13] and cloud point extraction (CPE) [14-16]. CPE has gotten the greatest interest from researchers because it agrees with all principles of green chemistry, as the amount of surfactant is

compared with organic solvent in liquid extraction and the surfactant is a non-toxic compound. Moreover, it is easy, low-cost, sensitive, highly efficient, and fast [17-22]. The optimum temperature for the cloud point is particularly important for generating micelles and achieving the critical micellar concentration (CMC). Two phases are generated: a cloud point phase (CPP) (more hydrophobic, with high micelle concentration and small volume) and an aqueous phase (more hydrophilic, with low surfactant quantity and larger volume). Hydrophobic complexes are produced by reacting the targeted elements with a complexing reagent, which is transferred into the micelles and then separated [23-25].

Solvation species have great hydrophobic characteristics

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and easy transfer to CPP. It contains metal nitrate and organic reagents in the presence of salting-out phenomena. Many reagents, such as ketones, aldehydes, esters, and others, make it simple to synthesize them [13]. When the solvation method is combined with CPE, the analysis becomes more sensitive and selective. Aspirin and Cu(II) nitrate after the formation of solvation species were used for extraction of Cu(II) from food samples in a successful, green, simple procedure in the presence of triton X-100. Salting-out included 0.1 M of KNO<sub>3</sub> and low LOD = 0.018 µg l<sup>-1</sup> [26]. Also, the Solvation method was applied to separate micro amounts of Ce(III) by using 2, 4-dimethyl pentan-3-one as an extractant with triton X-100 as a surfactant. In addition, 0.6 M from KNO<sub>3</sub> [27].

Vitamins were utilized as one of the green extraction reagents in CPE to preconcentrate metal ions. They are hydrophobic compounds, so, extracting them from the CPE requires two methods. The first method involves the extraction of vitamins directly. Forming non-polar complexes with other molecules is the second method, which is based on coordination bonds. Vitamins are ligands, which means they attract other molecules [41]. For extraction, nickel(II) was applied to CPE coupled with an onium system [H<sup>+</sup>(H<sub>2</sub>O) vitamin C; NiCl<sub>3</sub><sup>-</sup>] in the presence of vitamin C as a ligand and TritonX-114 [28].

Silver has bacteriostatic, thermal, and electrical conductivity properties. Therefore, it is a useful element in our lives. Silver compounds are widely used in the chemical industry as filters for pure drinking water and swimming pools, and are also used in the processing of medical materials, food, photographic industry, and. Natural water contains a trace quantity of silver because it has a low concentration in the crustal earth and is difficult to move in the water. For the determination of silver concentration in water and finding the toxicity effect on bio-organisms, very sensitive and accurate techniques should be used [30,31]. The ultrasound assisted-cloud point extraction approach was used to analyze the slivers. Copolymers and nanoparticles (Fe<sub>3</sub>O<sub>4</sub>) are used to separate slivers from samples. The process relies on charge transport between silver and agents at pH 5.0, as well as UV-Vis spectrophotometry used for the determination of the micro-volume of samples. With a LOD of = 0.85 µg l<sup>-1</sup>, the process is precise and sensitive [32].

The applied procedure CPE-SM, compared to other CPE

methods arranged in the studies [25,33-43], used different organic reagents and surfactants for the extraction of silver in different samples. Table 1 contains important results from many studies that were compared with this work, which are some of the best of the ones reported. Vitamin C appears to have a higher extraction efficiency and is a good reagent for extracting Ag(I) because of its chemical structure.

The main purpose of the present work was the separation and determination of silver in environmental

**Table 1.** Comparing the CPE-SM Technique for Ag(I) Pre-Concentration with the Previous Studies CPE Methods

Organic reagents	Surfactant	LOD	Ref.
4-( <i>p</i> -Chlorophenyl)-1-(pyridin-2-yl)thiosemicarbazide	TritonX-114	0.08 µg l <sup>-1</sup>	[25]
MBT	TritonX-114	2.200 ng ml <sup>-1</sup>	[33]
BMAA	TritonX-114	0.430 ng ml <sup>-1</sup>	[34]
APDC	TritonX-114	0.420 µg l <sup>-1</sup>	[35]
DDTC	TritonX-114	0.300 ng ml <sup>-1</sup>	[36]
Dithiazone	TritonX-114	0.700 µg l <sup>-1</sup>	[37]
Ligand less	PONPE 7.5	1.200 µg l <sup>-1</sup>	[38]
PAR	TritonX-114	6.000 µg l <sup>-1</sup>	[39]
Dithiazone	TritonX-114	0.560 ng ml <sup>-1</sup>	[40]
BIES	TritonX-114	1.4000 ng ml <sup>-1</sup>	[43]
Vitamin C	TritonX-100	0.035 µg l <sup>-1</sup>	This work

Abbreviations: MBT: 2-mercaptobenzothiazole, BMAA: bis(2-mercaptoanil) acetylacetone, APDC: ammonium pyrrolidine dithiocarbamate, DDTC: diethyl dithiocarbamate, PAR: 4,2 pyridylazo resorsinol.

samples using CPE. TritonX-100, vitamin C, and (KNO<sub>3</sub>) were used as a surfactant, complexing and salting-out (KNO<sub>3</sub>) agents, respectively. Conditions that affect CPE were evaluated.

## EXPERIMENTAL

### Chemicals and Solutions

Deionized water has been used in each solution in this paper. All used chemicals from Merck, Darmstadt, Germany have high purity (99.10-99.52%). The standard solution of 1mg ml<sup>-1</sup> of silver ion is prepared from AgNO<sub>3</sub> which prepares other dilution solutions in a suitable volumetric flask. The vitamin C was prepared (1 × 10<sup>-2</sup> M) in distilled water. CCl<sub>4</sub> was used to prepare a dithizone solution (1 × 10<sup>-2</sup> M).

### Instrumentation

A water bath for controlling cloud point temperature (CPT) (England, WNB7-45), A balance for weighting chemicals from Japan's A & D company (±0.0001 g) (Limited, Dool, CE, HR 200). A UV-Vis Biochrom spectrophotometer uses a quartz cell (1.00 cm) type (80-7000-11), the Libra S60 Cambridge CB40FJ. A hollow cathode lamp operating at 12 mA is used in a Shimadzu model (AA-6800 pattern, Kyoto, Japan) as a Flame atomic absorption spectrophotometer.

### Applied Procedure

Aqueous solutions (10 ml) contain the optimum quantity of silver(I), so the salting-out KNO<sub>3</sub> concentration is 0.5 M, with (1 × 10<sup>-4</sup> M) vitamin C and surfactant TritonX-100 (0.5 ml). Heating these solutions at optimum temperature for a suitable time in the electrical bath to form CPL in a small volume then separated it and added (5 ml) ethanol. Afterward, the absorbance was measured at λ<sub>max</sub> = 405 nm for CPL, which contains solvation species. A blank solution was created in a similar manner but without the silver ion. The aqueous layer was treated using the dithizone method, Ag was removed five times using tiny amounts of dithizone in CCl<sub>4</sub> (1 ml of 0.001% H<sub>2</sub>Dz solution). Ag and HNO<sub>3</sub> (1-2 M) are combined in an aqueous solution. The color of the last dithizone addition should not change from green to

yellow. The combined orange-yellow CCl<sub>4</sub> extracts were shaken for 20 s with 1 M HCl. The color of the last dithizone addition should not change from green to yellow. The combined orange-yellow CCl<sub>4</sub> extracts were shaken for 20 s with 1 M HCl. Separation of the aqueous layer (which contains Ag), the addition of a small amount of EDTA, ammonia pH adjustment to 4-5, and extraction of Ag from CCl<sub>4</sub> with a fraction of a 0.001% dithizone solution Remove any unattached dithizone from the extract with a dilute ammonia solution (2 drops of conc. NH<sub>3</sub> solution in 25 ml of water). Dissolve the AgHDz solution in 25 ml of CCl<sub>4</sub> and measure the absorbance at 462 nm using CCl<sub>4</sub> as a blank [42]. Plot the calibration curve, which is used to compute the amount of silver ion in the aqueous solution after extraction, and then use equation 1 to get the distribution ratio (D). LogD values are drawn in all of the graphics to provide more organized and broad values [25,33].

$$D = \frac{[Ag(I)]_{CPL}}{[Ag(I)]_{aq}} \quad (1)$$

### Real samples Preparation

**Water samples.** The tap and river water samples were filtered through a 0.5 μm filter membrane (Millipore) and stored in a polyethylene bottle for later use after being acidified to pH 2.0 with pure HNO<sub>3</sub> to minimize metal ion adsorption on the beaker surfaces.

**Soil samples.** Soil samples have been obtained in Najaf city, Iraq, from diverse areas. At 95 °C for 3 h, the tests were dried and homogenized, passed through a 130 grit filter. 5 ml of each (HNO<sub>3</sub>, H<sub>2</sub>O<sub>2</sub> and H<sub>2</sub>O) of the sampling soil in a Teflon container has been added to 1 g and digested by the application of a heating program in the microwave digestion oven and then 20 ml of H<sub>3</sub>BO<sub>3</sub> (5% w/v) was added to neutralize excess H<sub>2</sub>O<sub>2</sub>. Finally, the solution was filtered to a final volume of 50 ml using water. The concentration of masking agents (KCN, K<sub>2</sub>S<sub>2</sub>O<sub>3</sub>) was 0.1 M added to the samples. A CPE-SM technique uses 10 ml samples produced from the final clear solution [25].

## RESULTS AND DISCUSSION

The Spectrophotometric study included aqueous

solutions (10 ml) containing  $10 \mu\text{g l}^{-1}$  of silver ions and 0.5 M of  $\text{KNO}_3$  as salting-out in the existence of 0.5 ml of TritonX-100 and  $1 \times 10^{-4}$  M of vitamin C. The heating temperature was  $85^\circ\text{C}$  for 10 min later applied the general method. The UV-Vis absorption spectrum appears at  $\lambda_{\text{max}} = 405 \text{ nm}$  for solvation species. All of the influencing factors were investigated and determined.

### Effect of Salting-out Agent Concentration

The effect of  $\text{KNO}_3$  concentration, used for salting out, is shown in Figs. 1 and 2. Aqueous solutions (10 ml) containing  $10 \mu\text{g l}^{-1}$  silver ions were treated in the applied procedure with varied concentrations of salting-out ( $\text{KNO}_3$ ) in the range (0.1-1 M). The best  $\text{KNO}_3$  concentration for high extraction efficiency was 0.5 M. This concentration helps in achieving the highest possible rate of formation equilibrium with the formation of solvation species. Any concentration of less than or more than the ideal value does not do this. Explain the structure of solvation species in Eq. (1)

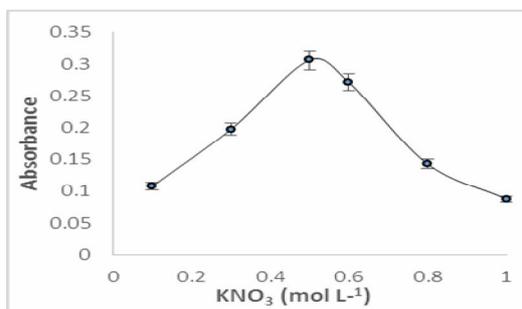
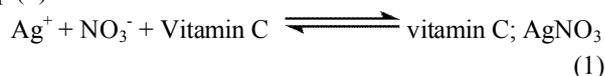


Fig. 1. Absorbance =  $f[\text{KNO}_3]$ .

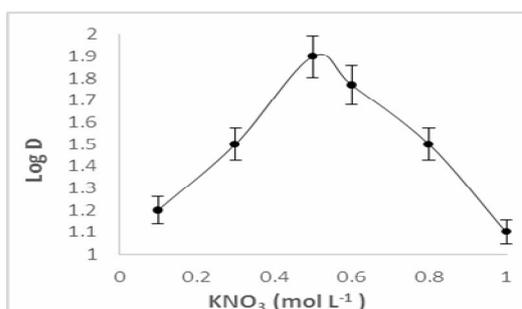


Fig. 2.  $D = f[\text{KNO}_3]$ .

### Effect of Temperature

The results of the comprehensive method, which extracted silver ions at different temperatures in the range ( $70\text{-}95^\circ\text{C}$ ) under optimal conditions, are shown in Figs. 3 and 4. The results show the optimum temperature was  $90^\circ\text{C}$ . According to the applied method, it was suitable for forming the CPL layer and provided higher extraction efficiency for the extraction of silver ions. Table 2 contains thermodynamic functions (enthalpy, entropy and free Gibbs energy) for the extraction of silver ions [33,34].

### Effect of Heating Time

To investigate heating time, a comprehensive method

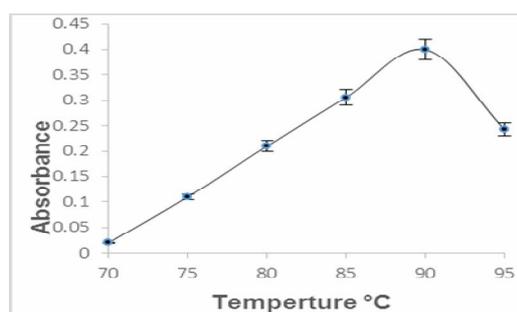


Fig. 3. Temperature effect on absorbance.

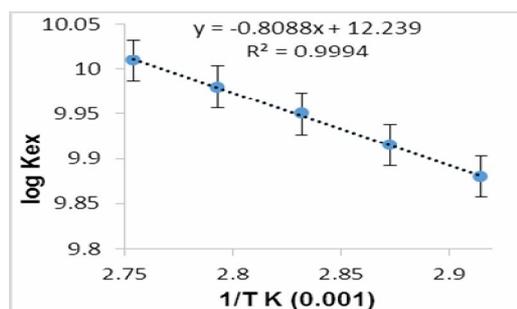


Fig. 4. Temperature effect on extraction constant.

Table 2. Thermodynamic Calculations for Silver Extraction in The CPE-SM Technique

$\Delta H_{\text{ex}}$ (kJ mol <sup>-1</sup> )	$\Delta G_{\text{ex}}$ (kJ mol <sup>-1</sup> )	$\Delta S_{\text{ex}}$ (J mol <sup>-1</sup> K <sup>-1</sup> )
0.0159	-69.655	191.668

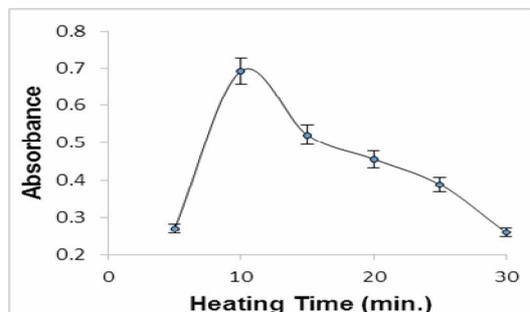


Fig. 5. Absorbance =  $f$  (heating time min).

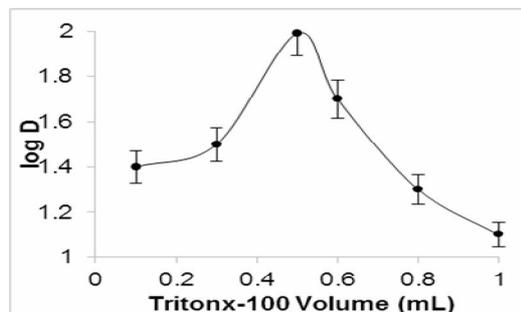


Fig. 8. TritonX-100 volume effect on D.

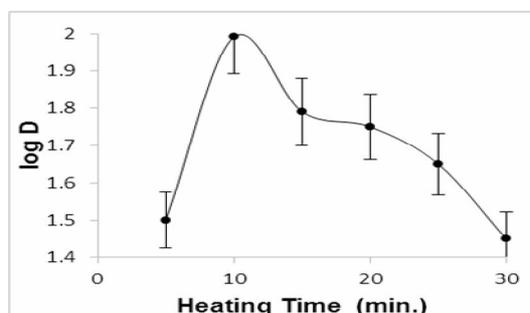


Fig. 6.  $D = f$  (heating time min).

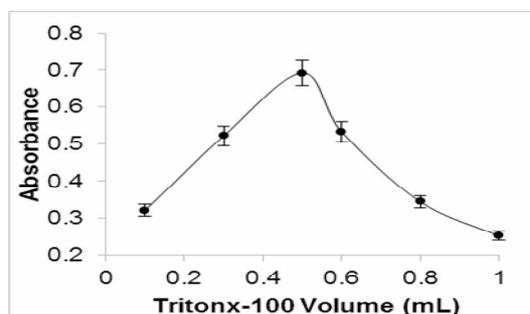


Fig. 7. TritonX-100 volume effect on the absorbance of extracted species.

was used with optimum conditions for all chemical concentrations and temperatures, with different heating times ranging from 5 to 30 min. The results are shown in Figs. 5 and 6. The optimal heating time was 10 min to give maximum extraction efficiency, whereas heating time provides the necessary energy to give the best rate of thermodynamic equilibrium for the formation of solvation species [14,15].

### Effect of Surfactant Volume

According to a comprehensive method in optimal conditions by using different volumes of surfactant TritonX-100 (0.1-1 ml) extracted silver ion, the results are in Figs. 7 and 8. The best value of TritonX-100 was 0.5 ml. This volume ensured a critical micelle concentration of CMC, which resulted in increased extraction efficiency. The properties of a non-ionic surfactant (TritonX-100) of lipophilicity and its capacity to bear the electrolytes brought about by the coinciding metal ions make it possible for CPE and a specific silver ion [33,34].

### Effect of Salting Out and Concentration

The applied method for extracting silver ions was used to test each state's different salting-out with increasing concentrations (0.1-1 M), and the experiment provided the results indicated in Figs. 9 and 10. So, each salting-out behaves differently in an aqueous solution. The experiment demonstrates that different salting-outs have different extraction efficiency for silver ions because the salting-out eliminates the silver ion's hydration shell. Storage species must be created and transferred to the CPL. Over the other five salts,  $\text{KNO}_3$  was chosen. It has a higher extraction coefficient, and it also has the best CPL, so the hydration shell of the potassium ion is greater in aqueous solutions [33].

### Effect of Organic Reagents

Different organic reagents are used for the extraction of silver according to the applied method in optimum conditions. The results in Table 3 show different extraction efficiency by using different reagents. Vitamin C has the

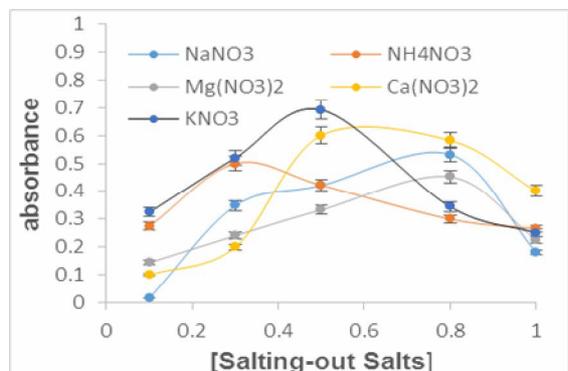


Fig. 9. Kind and concentrated salting-out effect on the CPL.

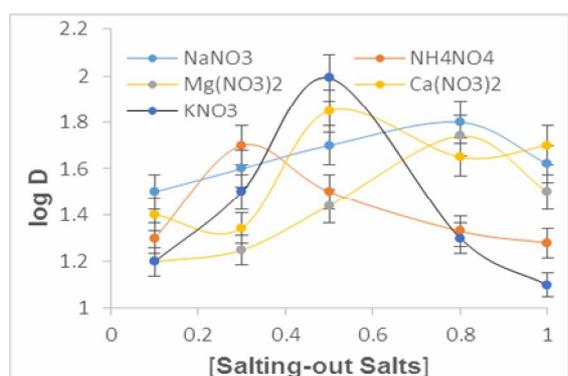


Fig. 10. Kind and concentration salting-out effect on D.

Table 3. Different Organic Reagents Have Different Extraction Efficiencies

Organic reagents	$\lambda_{\max}$ (nm)	Absorbance	D
Acetophenone	291	0.251	54.621
Methyl iso butyl ketone	292	0.347	66.752
Rhodamine-6G	556	0.405	83.461
2,4-Dimethyl pentanone	293	0.542	91.460
Vitamin C	405	0.692	98.000

best value for D and absorbance depending on its chemical structure [34]. Vitamin C increases the lipophilic properties of solvation species, allowing them to be transferred to the

cloud point layer and separated easily. It permits preconcentration at a specific temperature in the presence of a salting-out impact.

#### Analytical Parameters of the CPE-SM Technique.

The CPE-SM technique's analytical parameters, including the limit of detection, accuracy, and calibration curve linearity, were also analyzed [44]. Table 4 summarizes the findings, which show that silver recovery was quantitative (>99.3%) and that the method's accuracy was excellent. Also (RSD = 1.2% for  $C = 4 \mu\text{g l}^{-1}$ ,  $R^2 = 1$ ) for 10 ml of solution, the linearity measurements were  $0.1\text{-}10 \mu\text{g l}^{-1}$  under perfect conditions. Based on three times the standard deviation of the blank sample (3S/b), the detection limit was reported to be  $0.035 \mu\text{g l}^{-1}$ . However, the ratio of the slopes of the calibration curve after and before the enhancement process was used to calculate a preconcentration factor of 250.

The developed CPE-MS approach can quantitatively recover the additional spiking analytes (2, 4 or,  $6 \mu\text{g l}^{-1}$ ) from water samples. The recovery rate (R%) was estimated by use of Eq. (2). Where  $C_m$  is the silver content in a spiked sample,  $C_0$  is the metal value in a sample, and  $m$  is the silver content spiked up.

$$R\% = \{(C_m - C_0)/m\} \times 100 \quad (2)$$

Table 4. Analytical Figures of Merit for the CPE-SM Technique

Parameters	Ag(I)
Regression equation	$y = 66.712x + 0.0019$
Correlation coefficient	$R^2 = 1$
Linear range ( $\mu\text{g l}^{-1}$ )	0.5-10
precision (RSD%) $C = 4 \mu\text{g l}^{-1}$ , $n = 5$	1.2
Limit of quantification ( $\mu\text{g l}^{-1}$ )	0.116
Limit of detection ( $\mu\text{g l}^{-1}$ )	0.035
Preconcentration factor	250

CPE-SM conditions: 10 ml sample solution, 1% (w/v) TritonX-100 (0.5 ml), 0.5 M  $\text{KNO}_3$ ,  $1 \times 10^{-4}$  M vitamin C, temperature  $90^\circ\text{C}$  and heating time 10 min.

#### Applications for Real Samples

It was used to calculate silver in various water and soil

**Table 5.** Determination of Silver Ions in River and Tap Water Tests by Three Methods

Tests	CPE-SM method				FAAS method ( $\mu\text{g l}^{-1}$ )	Dithizone method ( $\mu\text{g l}^{-1}$ )
	Added ( $\mu\text{g l}^{-1}$ )	Found ( $\mu\text{g l}^{-1}$ )	RSD (%)	Recovery (%)		
River water	-	N.D.	-	-		
	2	2.03	1.1	102.2	2.12	2.15
	4	4.05	1.3	103.1	4.15	4.12
	6	5.08	1.2	99.3	6.12	6.12
Tap water	-	N.D.	-	-	-	-
	2	2.02	1.1	101.5	2	1.99
	4	4.05	1.2	102.5	4.89	4.82
	6	6.06	1.5	101.6	6.99	6.91

samples from Najaf city to test the accuracy of the applied method for assaying silver ions.

The recoveries of familiar concentrations of silver ions added to the water tests were studied by the CPE-SM procedure. The results shown in Table 5 show that the recovery of water tests is very acceptable and reasonable, demonstrating the capability of the CPE-SM procedure in the separation and determination of silver ions in water tests containing various materials under suitable conditions. Furthermore, silvers were determined using two other standard methods (flame atomic absorption spectrometry (FAAS) and the dithiazone method). To prove the authenticity of the CPE-SM technique, the described procedure was used for preconcentration and determination of silver in environmental samples [33-35]. The acquired results were summed up in Table 6.

**Table 6.** Determination of Silver Soil Samples by The CPE-SM Technique

Soil samples	Silver amount ( $\mu\text{g l}^{-1}$ )
1	$9.02 \pm 0.4$
2	$8.88 \pm 1.6$
3	$8.38 \pm 1.4$
4	$7.25 \pm 2.9$
5	$9.01 \pm 0.9$
6	$7.33 \pm 2.0$

\*All water and soil samples gotten from different position in Al-Najaf city. \*Mean  $\pm$  standard deviation, All value repeat for five time.

## CONCLUSIONS

A green, cheap, and sensitive analytical methodology CPE-SM was applied to separate and determine silver ions in water and soil samples. The chemicals used were green (vitamin C, TritonX-100, and others). Also, a low limit of detection and quantification, as well as recovery, was quantitative (>99.3%) and had good precision. The separation and preconcentration time of the applied technique was short (10 min). The coupled merits of the cloud point extraction technology (simple, safe, quick, and economical) and the solvation approach using a lipophilic reagent (selectivity and sensitivity) for silver ions were applied for the analysis in various concentrations. The created system yielded great outcomes regarding selectivity, cleanliness, ease, and accuracy for the quick separation of silver in water and soil tests.

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