Simultaneous Determination of Lead and cadmium in Environmental Samples Using Zinc-diethyldithiocarbamates

*Bashar Qasim** ⓘD ✉

Applied Chemistry Branch, Department of Applied Sciences, University of Technology, Baghdad, Iraq.
*Corresponding Author.

Abstract

Toxic metals have a serious impact on the ecological environment. The determination of these metal concentrations provides more information about their toxicity. In this study, a simple, rapid and selective spectrophotometric method with solid phase extraction method has been described for the simultaneous determination of lead (Pb) and cadmium (Cd) in environmental samples using a freshly precipitated zinc-diethyldithiocarbamate ($Zn-(DDTC)_2$) as a reagent. The pre-concentration of Pb and Cd contents was performed according to solid phase extraction process. The determination of Pb^{+2} and Cd^{+2} based on the replacement of Zn^{+2} from the freshly precipitated $Zn-(DDTC)_2$ by Pb^{+2} and Cd^{+2} in aqueous solution.

The proposed method presented a linear range of $0.1 – 16$ µgml^{-1} and $0.1 – 14$ µgml^{-1} for Pb and Cd respectively. The recoveries of the studied metals were ranged between $90 – 96\%$ with correlation coefficients (R^2) of 0.9985 and 0.9989 for Pb and Cd respectively. The optimum conditions of various parameters (pH, extraction period and aqueous phase volume) were experimentally investigated, and the effect of various salt and or ion solutions as interferences was also studied. The proposed method was also applied for a number of real environmental samples to assess metal ion contents. The obtained results showed a good precision and accuracy indicating that the current method can apply for routine measurement of Pb and Cd contents and also can be applied to more fields.

Keywords: Contaminated soil, Cadmium, Diethyldithiocarbamate, Lead, Quantitative determination.

Introduction

Soil contamination with potentially toxic metals has serious environmental problems in the last decade[1]. The releasing of heavy metals into the ecosystem induced their entering to food chain which finally results in potential risk for animals and human health[2-4]. In the eco-system and based on the biological role, the heavy metals can be classified as essential heavy metals including copper, zinc, and ferric[5], and also non-essential heavy metals such as mercury, lead, and cadmium[6]. These toxic metals exist in low content, but with the increasing of

pollutant sources, its concentrations were increased above the acceptable values[7].

Anthropogenic activities including mining and smelting process, industrial and agricultural activities and coal combustion residues are considered as the major sources for toxic metals in agricultural soils and water[8, 9]. Since these metals are not degraded or decomposed as the organic pollutants[10], therefore, the impact and incorporation of the toxic metal in soil and water resulted in transformation in its properties and alteration the soil

biological cycles which finally affect the microbiological communities[11, 12]. For this reason, it is important to find simple, rapid and sensitive method for the determination of Pb and Cd levels in the environmental samples.

Several analytical methods were already used or developed for the quantitative measurement of heavy metals. These methods include: ultraviolet-visible spectrophotometer[13], atomic fluorescence spectrometry[14], flame atomic absorption spectrometry[15], inductively coupled plasma mass spectrometry[16] and X-ray fluorescence technique[17]. Some of these techniques are still unavailable in most laboratories due to its high cost of the instruments. Among these methods, the atomic absorption spectrophotometry (AAS) has been used as common method for metal ions determination due to its sensitivity, selectivity, and accuracy[18-20].

Materials and Methods

Apparatus, Chemical Reagents and Solutions

A flame atomic absorption spectrometer (FAAS) (Perkin–Elmer 2380) is used to determine the metal ion concentrations for the studied samples. The pH-meter (ProfiLine 1970i) is used for the measurement of pH values of the solutions.

High purity chemical reagents of diethyldithiocarbamic acid (Na-DDTC), zinc sulfate ($ZnSO_4$), lead sulfate ($PbSO_4$) and cadmium sulfate ($CdSO_4$) with an analytical reagent grade were used in the current study. All glassware was cleaned using acidified HNO_3 solution and then rinsed with doubly deionized water.

Stock solutions of 1000µg ml^{-1} metal ion salts were prepared from their salts by dissolving of an appropriate amount in doubly deionized water. The stock metal ion solutions were prepared and used as standard solutions. The stock solutions for the other used solutions were prepared using their analytical grade. The solution of the acetate buffer was prepared from a mixture of acetic acid and sodium acetate in double distilled water.

The solid phase extraction method is an emerging process which has been used for the pre-concentration of heavy metal ions of interest in matrix[21]. Dithiocarbamates which derived from dithiocarbamic acid has the ability for pre-concentration of heavy metal ions from the environmental matrix via forming strong and stable complexes with metal ions[22]. Many researchers have used precipitated metal diethyldithiocarbamates reagents as co-precipitating agents for the extraction of metal ions prior to their determination with a spectrophotometric analysis[23-26].

Since the stability of zinc complex is lower than lead and cadmium, the objectives of this study was therefore to: *i*) use of freshly precipitated Zn-(DDTC)$_2$ as a pre-concentration agent for simultaneous quantitative determination of lead and cadmium prior to their measurement by AAS, and *ii*) assessment of the optimal conditions for the Pb and Cd extraction from environmental samples.

Preparing Zinc-(DDTC)₂

The Zn-(DDTC)$_2$ solution was prepared by mixing 0.1mmol of Na-DDTC solution with 0.1mmol of Zn(II) solution, heated to boil for 2min, and then cooled to room temperature. When the precipitate of Zn-(DDTC)$_2$ was formed, it was filtered using filter paper, washed using doubly deionized water, it then was dried at 120°C using laboratory oven for 24 hrs.

General Procedure

To determine the concentration of Pb and Cd ions, 100ml of solution containing 25 – 250µg of Pb^{+2} and Cd^{+2} was placed in a 50ml Erlenmeyer flask. For this solution, 0.1g of precipitated Zn-(DDTC)$_2$, and acetate buffer were added, it then was stirred for 10min. The solution was filtered and washed carefully with doubly deionized water.

The complexes of, Cd-(DDTC)$_2$ and Pb-(DDTC)$_2$ was formed as solid products by exchanging Zn^{+2} by Pb^{+2} and Cd^{+2} on the precipitated Zn-(DDTC)$_2$. The formed complexes were then dissolved by concentrated HNO_3, and the volumes of solutions were made to up of 10ml with doubly deionized water. The metal ion contents in the final solutions were measured by FAAS. The optimum experimental conditions represented by pH,

extraction time, volume of aqueous phase and reagent concentration were also investigated.

Sample Collection and Preparation
Soil Samples

Soil samples at 20 cm depth were manually collected from house garden using a spade. The collected samples were transferred to laboratory by using polyethylene bags. The collected soil was dried for 72 hrs at room temperature, manually homogenized and sieved with 2mm sieve mesh. The metal ion concentrations in the collected samples were measured after metal ion digestion with aqua regia/HF acid and determined using FAAS.

Water Samples

Water samples such as distilled water, tap water, river water, rain water and drain water were collected by using clean polyethylene bottles. These samples were transferred to the laboratory and drops of nitric acid were added to it as preservative.

Results and Discussion

Optimization of the Reaction Conditions

Effect of pH Value

The pH values play an important role by controlling the acidity or basicity of solutions. The effect of the pH values on the metal ions exchange process and therefore the replacement of Zn by Pb and Cd were performed using different pH values in the presence of acetate buffer solution, where the other parameters were kept constants. The effect of pH was assessed in the range of 1 – 10, and adjusted by using diluted HNO_3 and NaOH solutions. The experiment was performed by taking 25ml of solution containing 2µg of Zn and Cd. The general procedure was performed and the metal ion contents were determined using FAAS. The results of the pH effect are presented in Fig. 1, it clear that when the pH values increased, the recovery increased for both metal ions, attained the maximum value at pH=5, and then it remained constant at 7. On the other hand, when pH increased up to 7, a decrease in the recovery of the metal ions occurred. The using of pH lower than 4 might cause a decomposition for the dithiocarbama²tes, while the higher value would induce a precipitation of Pb and Cd as metal hydroxide which therefore reduces the efficiency of the metal ions extraction. Therefore, a pH value of 5 was therefore chosen for further analysis.
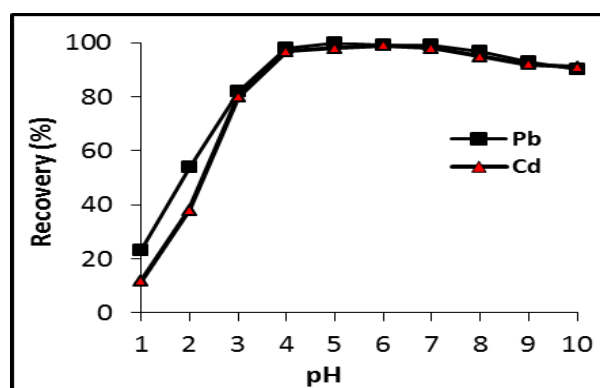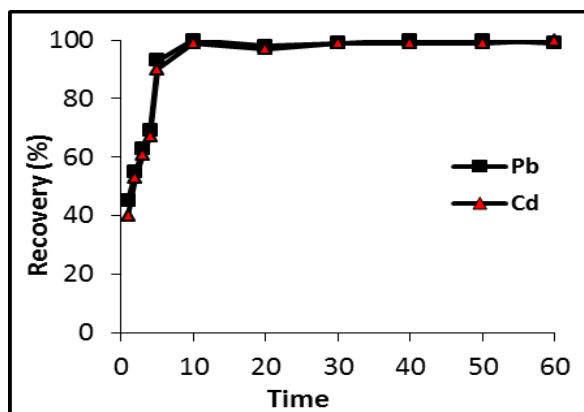

Figure 1. **Effect of pH values on the pre-concentration of Pb and Cd.**

Effect of Extraction Period

The effect of the extraction time method on the extraction efficiency of the metal ions was investigated. A solution of 5µg ml^{-1} of both Pb and Cd was taken and extracted at various periods of time (1 – 60 min) according to the previous general procedure. Fig. 2 shows that the recovery percentages increased at initial stages of the contact time, and the efficiency of extraction increased with the increasing of time till 10 min. It then remained constant with the time increase. Therefore, according to obtained data, a 10min was the best period of extraction to reach optimal metal ions extraction.

Figure 2. The effect of extraction period on the pre-concentration of Pb and Cd.

Effect of Aqueous Phase Volume

To investigate the effect of aqueous phase volume on the extraction efficiency for the studied metal ions, a solution of 5µg ml⁻¹ of the Cd and Pb ions has been taken and then diluted from 50 to 500ml using deionized water. The contents of Pb and Cd were determined according to the general procedure, and the recoveries were also calculated as shown in Fig. 3. The results have shown that the recovery percentages for Pb and Cd increased along aqueous phase volume from 50 to 800ml. Afterwards, the recovery percentages decreased considerably when the aqueous phase volume increased. Therefore, the optimal aqueous phase volume of 100ml was chosen for confidence.
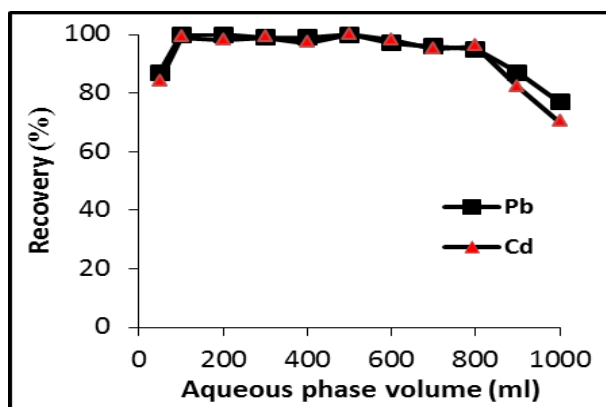
Figure 3. The effect of aqueous phase volume on the pre-concentration of Pb and Cd.

Effect of Diverse Ions

It is well known that soil and water contains various cations and anions such as sulfate, nitrate, phosphate, chloride as well as other different metal ions which might influence the determination of the studied metal ions. Therefore, this interfering effect as well as the tolerance limits of the foreign ions that might cause an error on the absorbance was investigated. A

solution of 5µg ml⁻¹ of Pb and Cd has been taken and then mixed with various amount of anions and cations salt solutions. The Pb and Cd concentrations were determined with optimal conditions as summarized in Table1. The obtained results showed that most anions and cations can be tolerated without interference even when they were at saturated levels as they exceeded 100-fold.

Table 1. The effect of diverse ions on Pb and Cd concentration

| Diverse ion	Limit of tolerance (µg/mL⁻¹)	Diverse ion	Limit of tolerance (µg/mL⁻¹)
Nitrate	1000	Mn^{+2}	100
Citrate	1000	Mg^{+2}	134
Phosphate	400	Fe^{+3}	170
Sulfate	800	Ni^{+2}	127
Tartrate	800	Cu^{+2}	240
Oxalate	600	Ca^{+2}	140

Validation of the Method
Calibration Curves

Calibration graphs for the studied metal ions were constructed using ten standard solutions that containing 0.05 – 20µg ml⁻¹ of Pb and Cd by plotting absorbance values against metal ion concentrations according to the used general procedure. The method of least squares was used to calculate the linearly ranges, equation of regression and correlation coefficient. Figs. 4 and 5 show that the straight line between metal contents and their absorptions obeyed the equations $y = 0.1027x + 0.0071$, and $y = 0.1327x - 0.0035$ for Pb and Cd respectively. The correlation coefficients (R^2) were 0.9985 and 0.9989 for Pb and Cd respectively. The linearity ranges were 0.1 – 16 µg ml⁻¹ and 0.1 – 14 µg ml⁻¹ for Pb and Cd respectively as summarized in Table 2.

$$y = 0.1027x + 0.0071$$
$$R^2 = 0.9985$$
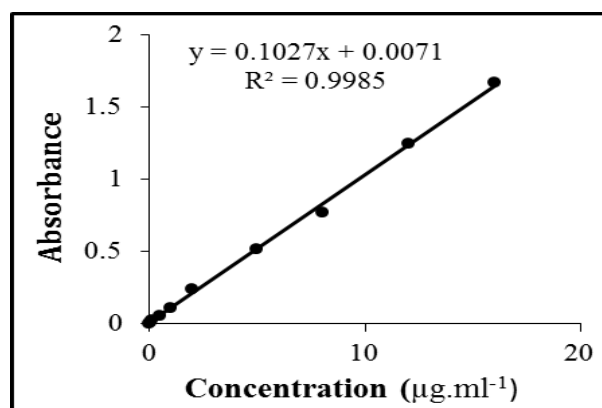
Figure 4. The calibration curves for Pb, constructed by plotting concentrations of metal ions against absorbance (µg ml⁻¹).
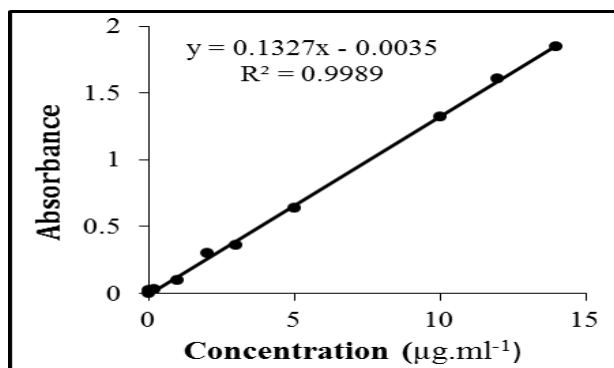
Figure 5. The calibration curves for Cd, constructed by plotting concentrations of metal ions against absorbance (µg ml⁻¹).

Table 2. Linear ranges, calibration equations and correlation coefficient.

Metal	Equation	Linearity	R^2
Pb	y = 0.1027x + 0.0071	0.1 – 16	0.9985
Cd	y = 0.1327x - 0.0035	0.1 – 14	0.9989

Recovery

The recovery of the current method which represents the accuracy was performed in five replicates by taking three different concentrations of 10, 15, and 25 µg ml⁻¹ for Pb and Cd standard solutions. The soil and water samples were previously analyzed to ensure its absence from Pb and Cd. The soil and water then spiked with 10ml of the corresponding metal ion standard solutions. The recovery values were ranged between 93 – 96 % for Pb and 90 – 96 % for Cd as summarized in Table 3. The obtained results indicated that the proposed method was capable and suitable for the determination of the studied metal ions at all their concentration levels with good recovery values.

Precision

The precision for the proposed method was performed in five replicates in term of inter-day and intra-day by spiking the soil and water with three different metal ion solutions of 10, 15 and 25 µg ml⁻¹. The precision which expressed as the relative standard deviation (RSD) was determined by analyzing the samples in same day and in five consecutive days. The RSD values were 2.3 for Pb and 3.9 for Cd as summarized in Table 3. These RSD values confirmed the repeatability and reproducibility of the current method for the routine analysis for Pb and Cd.

Sensitivity

The sensitivity of the current method was calculated according to the results that obtained from the calibration graph including the slope of the calibration graph (a) and the value of standard deviation (S). The limit of detection, (LOD) and also limit of quantification, (LOQ) were calculated according to the calibration graphs from the formula: LOD = 3S/a, and LOQ = 10S/a respectively. The obtained results (Table 3) showed that LOD values were 0.23µg.ml⁻¹ and 0.18µg ml⁻¹ for Pb and Cd respectively. Whilst, LOQ values were 0.71µg ml⁻¹ and 0.56µg ml⁻¹ for Pb and Cd respectively. According to the obtained results, it can be considered that the proposed method is satisfactory when compared both LOD and LOQ values were compared with other researcher's studies.

Table 3. Limit of detection, limit of quantification, precision (relative standard deviation, RSD %) and recovery values for spiked metal levels (µg g⁻¹).

Metal	LOD	LOQ	Precision (RSD)		Recovery (%)		
			Intra-day	Inter-day	10	15	25
Pb	0.23	0.71	1.6	2.3	100.7	99.2	99.3
Cd	0.18	0.56	1.7	1.9	99.1	101.4	101.8

Application:

Determination of Pb and Cd in Soil Samples

The current method was applied for five real soil samples to determine Pb^{+2}, and Cd^{+2} (S_1, S_2, S_3, S_4 and S_5) which were collected from different areas as identified in Table 4. The soil samples were digested with aqua regia/hydrofluoric acid and filtered, the filtrate was mixed with 0.1g of freshly precipitated $Zn-(DDTC)_2$. The procedure of the optimum conditions was applied for the determination of metal ion concentrations.

Table 4. Determination of Pb and Cd in soil samples (*n=3*; ±standard deviation)

Soil	Pb ($\mu g\ g^{-1}$)	Cd ($\mu g\ g^{-1}$)
S_1	34 ± 1.3	1.08 ± 0.12
S_2	27 ± 2.3	0.83 ± 0.05
S_3	46 ± 3.7	1.65 ± 0.35
S_4	32 ± 1.7	0.23 ± 0.07
S_5	21 ± 2.4	0.76 ± 0.2

Table 5. Determination of Pb and Cd in water samples (*n=3*; ±standard deviation)

Water sample	Pb ($mg\ L^{-1}$)	Cd ($mg\ L^{-1}$)
Distilled water	0.01± 0.002	<LOD
Tap water	1± 1.3	0.5± 0.09
River water	46 ± 3.7	2.65 ± 0.45
Rain water	3.2 ± 1.7	0.17 ± 0.24
Drain water	121 ± 2.4	18.76 ± 0.32

Determination of Pb and Cd in Water Samples

The Pb and Cd contents in water samples were determined according to the general procedure by taking 2ml of water for analysis. The obtained results are presented in Table 5.

Determination of Pb and Cd in Synthetic Mixture Samples

Synthetic mixtures containing Pb and Cd ($5\mu g\ mL^{-1}$ for each ion) with varying compositions of different metal ions were prepared and analyzed for Pb and Cd contents using the proposed method using EDTA as a masking agent. Table 6 show that the obtained results were highly reproducible, and the recovery values achieved for all sample solutions with RSD values did not exceed 1.27.

Table 6. Determination of Pb and Cd in synthetic mixtures (*n=3*; ±standard deviation)

Sample	Composition of mixture ($\mu g/mL$)	Added	Pb ($\mu g/mL$) Found	R(%)±S_d	Added	Cd ($\mu g/mL$) Found	R(%)±S_d
1	$Pb^{+2} - Cd^{+2}$	5	4.98	99.6±0.97	5	4.91	96.8±0.85
2	$Pb^{+2} - Cd^{+2} - Ni^{+2} - Mg^{+2} + EDTA$	5	5.02	100.4±1.03	5	5.23	101.2±1.1
3	$Pb^{+2} - Cd^{+2} - Al^{+3} - K^{+} - Mn^{+2} + EDTA$	5	5.1	102±1.27	5	4.95	99±0.91
4	$Pb^{+2} - Cd^{+2} - Ca^{+2} - Cr^{+3} - Cl^{-} + EDTA$	5	4.97	99.4±0.87	5	4.92	98.4±1.16
5	$Pb^{+2} - Cd^{+2} - SO_4^{-2} - Co^{+2} - Na^{+} + EDTA$	5	4.93	98.6±1.02	5	5.12	102.4±0.66

Conclusion

This study aimed to validate a simple and efficient method for simultaneous determination of Pb and Cd in environmental samples using freshly precipitated zinc-diethyldithiocarbamate as a reagent. In the current study, the solid phase extraction was applied as an efficient method for the pre-concentration of the studied metal ions before determination of their concentration by FAAS. The proposed method showed a good extraction for Pb and Cd in soil and water matrix. The selectivity of the proposed method came from the fact that the stability of Zn-(DDTC)$_2$ complex is lower than Pb and Cd dithiocarbamate complexes which therefore induces the replacement of Zn with Pb and Cd on DDTC solid phase. The obtained Pb-(DDTC)$_2$ and Cd-(DDTC)$_2$ as solid phases dissolve easily in the nitric acid, and the FAAS was used to measure their absorbance. The proposed method showed a good precision and accuracy which proves its suitability for the routine determination of Pb and Cd in environmental samples.

Authors' Declaration

- Conflicts of Interest: None.
- I hereby confirm that all the Figures and Tables in the manuscript are mine. Furthermore, any Figures and images, that are not mine, have been included with the necessary permission for re-publication, which is attached to the manuscript.

- Authors sign on ethical consideration's approval.
- Ethical Clearance: The project was approved by the local ethical committee at University of Technology.

References

1. Nawab J, Ghani J, Khan S, Xiaoping W. Minimizing the risk to human health due to the ingestion of arsenic and toxic metals in vegetables by the application of biochar, farmyard manure and peat moss. J Environ Manage. 2018; 214: 172–83. https://doi.org/10.1016/j.jenvman.2018.02.093.

2. Qin G, Niu Z, Yu J, Li Z, Ma J, Xiang P. Soil heavy metal pollution and food safety in China: Effects, sources and removing technology. Chemosphere. 2021; 267: 129205. https://doi.org/10.1016/j.chemosphere.2020.129205.

3. Ran H, Guo Z, Yi L, Xiao X, Zhang L, Hu Z, et al. Pollution characteristics and source identification of soil metal(loid)s at an abandoned arsenic-containing mine, China. J Hazard Mater. 2021; 413: 125382–2. https://doi.org/10.1016/j.jhazmat.2021.125382.

4. Taleb R, Qasim B. Potassium Hydroxide Activated Peanut Shell as an Effective Adsorbent for the Removal of Zinc, Lead and Cadmium from Wastewater. J Ecol Eng. 2023; 24(1): 66–78. https://doi.org/10.12911/22998993/156006.

5. Khoshgoftarmanesh AH, Afyuni M, Norouzi M, Ghiasi S, Schulin R. Fractionation and bioavailability of zinc (Zn) in the rhizosphere of two wheat cultivars with different Zn deficiency tolerance. Geoderma. 2018; 309: 1–6. https://dx.doi.org/10.1016/j.geoderma.2017.08.019.

6. Khan AM, Nawaz I, Yousaf S, Ammar Sabir Cheema, Iqbal M. Soil amendments enhanced the growth of Nicotiana alata L. and Petunia hydrida L. by stabilizing heavy metals from wastewater. J Environ Manage. 2019; 242: 46–55. https://doi.org/10.1016/j.jenvman.2019.04.040.

7. Taleb R, Qasim B. Adsorptive Removal of Potentially Toxic Elements from Wastewater Using Peanut Shells Biochar. IOP Conf Ser.: Earth Environ Sci. 2023; 1158(3): 032012. https://doi.org/10.1088/1755-1315/1158/3/032012

8. Liu M, Yang Y, Yun X, Zhang M, Wang J. Concentrations, distribution, sources, and ecological risk assessment of heavy metals in agricultural topsoil of the Three Gorges Dam region, China Environ Monit Assess. 2015; 187(3): 147. https://doi.org/10.1007/s10661-015-4360-6

9. Gascó G, Álvarez ML, Paz-Ferreiro J, Méndez A. Combining phytoextraction by Brassica napus and biochar amendment for the remediation of a mining soil in Riotinto (Spain). Chemosphere. 2019; 231: 562–70. https://doi.org/10.1016/j.chemosphere.2019.05.168

10. Tregubova P, Koptsik G, Stepanov A, Koptsik S, Spiers G. Organic amendments potentially stabilize metals in smelter contaminated Arctic soils: An incubation study. Heliyon. 2021; 7(1): e06022. https://doi.org/10.1016/j.heliyon.2021.e06022

11. Qasim B, Razzak AA, Rasheed RT. Effect of biochar amendment on mobility and plant uptake of Zn, Pb and Cd in contaminated soil. IOP Conf Ser.: Earth Environ Sci. 2021; 779(1): 012082. https://doi.org/10.1088/1755-1315/779/1/012082

12. Ding K, Wu Q, Wei H, Yang W, Séré G, Wang S, et al. Ecosystem services provided by heavy metal-contaminated soils in China. J Soils Sediments. 2016; 18(2): 380–90. https://doi.org/10.1007/s11368-016-1547-6

13. Alzahrani L, El-Ghamry HA, Saber AL, Mohammed GI. Spectrophotometric determination of Mercury(II) ions in laboratory and Zamzam water using bis Schiff base ligand based on 1,2,4-Triazole-3,5-diamine and o-Vaniline. Arab J chem. 2022; 16(5): 104418–8. https://doi.org/10.1016/j.arabjc.2022.104418

14. Zeng W, Hu Z, Luo J, Hou X , Jiang X. Highly sensitive determination of trace antimony in water samples by cobalt ion enhanced photochemical vapor generation coupled with atomic fluorescence spectrometry or ICP-MS. Anal Chim Acta. 2022; 1191: 339361 https://doi.org/10.1016/j.aca.2021.339361.

15. Cui T, Zhu X, Wu L, Tan X. Ultrasonic assisted dispersive liquid-liquid microextraction combined with flame atomic absorption spectrometry for determination of trace gallium in vanadium titanium magnetite. Microchem J. 2020; 157: 104993–3. https://doi.org/10.1016/j.microc.2020.104993.

16. Hamad AA, Alamer KH, Alrabie HS. The accumulation risk of heavy metals in vegetables which grown in contaminated soil. Baghdad Sci J. 2021; 18(3): 471-479. https://dx.doi.org/10.21123/bsj.2021.18.3.0471.

17. Hasan NM. A comparative study of heavy metals and trace elements concentration in milk samples consumed in Iraq. Baghdad Sci J. 2020; 17(1(Suppl.):

0310.https://doi.org/10.21123/bsj.2020.17.1(Suppl.).0310.

18. Naiema Vakili Saatloo, Boshra Ebrahiminejad, Parisa Sadighara, Manafi L, Najmeh Yazdanfar, Saeid Fallahizadeh. Quantification and human health risk assessment of cadmium and lead in wheat flour on the Iranian market by atomic absorption spectrometry. Case stud Chem Environ Eng. 2023; 8: 100438–8. https://doi.org/10.1016/j.cscee.2023.100438

19. Şaylan M, Zaman BT, Gülhan Bakırdere E, Bakırdere S. Determination of trace nickel in chamomile tea and coffee samples by slotted quartz tube-flame atomic absorption spectrometry after preconcentration with dispersive liquid-liquid microextraction method using a Schiff base ligand. J Food Compos Anal. 2020; 88: 103454. https://doi.org/10.1016/j.jfca.2020.103454.

20. Altunay N, Elik A, Gürkan R. Monitoring of some trace metals in honeys by flame atomic absorption spectrometry after ultrasound assisted-dispersive liquid liquid microextraction using natural deep eutectic solvent. Microchem J. 2019; 147: 49–59. https://doi.org/10.1016/j.microc.2019.03.003.

21. Anjos SL, Almeida JS, Sena L, Cristina A, Santos AP, Antonio F S Queiroz, et al. Determination of Cu, Ni, Mn and Zn in diesel oil samples using energy dispersive X-ray fluorescence spectrometry after solid phase extraction using sisal fiber. Talanta. 2021; 225: 121910–0.https://doi.org/10.1016/j.talanta.2020.121910.

22. de la Calle I, Fernández-Rodríguez D, Lavilla I, Bendicho C. Silver nanoparticle-cellulose composite for thin-film microextraction of Cd and Pb as dithiocarbamate derivatives followed by inductively-coupled plasma mass spectrometry determination. Advances in Sample Preparation. 2022; 4: 100041. https://doi.org/10.1016/j.sampre.2022.100041.

23. Hasan Cesur, Aksu Ç. Determination of Cadmium and Zinc in Fertilizer Samples by FAAS after Solid-Phase Extraction with Freshly Precipitated Manganese-diethyldithiocarbamate. Anal Sci. 2006; 22(5): 727–30. https://doi.org/10.2116/analsci.22.727

24. Cesur H. Selective solid-phase extraction of Cu(II) using freshly precipitated lead diethyldithiocarbamate and its spectrophotometric determination. Chem Pap. 2007; 61(5): 342–347. https://doi.org/10.2478/s11696-007-0045-0.

25. Uddin MN, Abdus Salam Md, Hossain MA. Spectrophotometric measurement of Cu(DDTC)2 for the simultaneous determination of zinc and copper. Chemosphere. 2013; 90(2): 366–73. https://dx.doi.org/10.1016/j.chemosphere.2012.07.029

26. Pournara AD, Rapti S, Lazarides T, Manos MJ. A dithiocarbamate-functionalized Zr_4^+ MOF with exceptional capability for sorption of Pb2+ in aqueous media. J Environ Chem Eng. 2021; 9(4): 105474. https://doi.org/10.1016/j.jece.2021.105474

التقدير المتزامن للرصاص والكادميوم في النماذج البيئية باستخدام الزنك ـ ثنائي إيثيل ثيوكاربامات

بشار حسين قاسم

فرع الكيمياء التطبيقية ، قسم العلوم التطبيقية ، الجامعة التكنولوجية، بغداد، العراق.

الخلاصة

تمتلك المعادن الثقيلة السامة تأثيرا خطيرا على البيئة ، وان عملية التقدير الكمي لتركيز هذه المعادن يعطي معلومات مهمة تخص سمية المعادن. في هذه الدراسة تم استخدام طريقة تحليلية طيفية سهلة ، سريعة وانتقائية مع طريقة الاستخلاص بالطور الصلب للتقدير المتزامن للرصاص والكادميوم في النماذج البيئية باستخدام الزنك ـ ثنائي إيثيل ثيوكاربامات المترسب ككاشف. تم استخدام طريقة الاستخلاص بالطور الصلب لعملية لتركيز المسبق لمحتوى الرصاص والكادميوم في النماذج البيئية. أظهرت الطريقة المقترحة مدى خطية يتراوح بين 0,1 – 16 مايكروغرام/مل و 0,1 – 14 مايكروغرام/مل لكل من الرصاص والكادميوم على التوالي. وكانت قيمة الاستردادية تتراوح بين 90 – 96 % مع معامل ارتباط (ر2) مقداره 0,9985 و 0,9989 لكل من الرصاص والكادميوم على التوالي. تم دراسة الظروف المثلى مثل (قيمة الاس الهيدروجيني ، وقت الاستخلاص و حجم الطور المائي) على كفاءة عملية الاستخلاص للمعادن بالاضافة الى دراسة تأثير وجود الاملاح والايونات الاخرى كمتداخلات. تم تطبيق الطريقة المقترحة في الدراسة على عدد من النماذج البيئية (التربة والماء) لتقدير كمية العناصر الموجودة فيها. اظهرت النتائج المستحصلة دقة وانتقائية جيدة تدل على امكانية استخدام الطريقة المقترحة في تقدير الرصاص والكادميوم وامكانية تطبيقها في مجالات اخرى.

الكلمات المفتاحية: التربة الملوثة، الكادميوم، داي اثيل داي ثايوكاربامات، الرصاص، التقدير الكمي.