

Separation and Preconcentration of Trace Amounts of Lead Ion by Coacervative Extraction Method and Determination by Flame Atomic Absorption Spectroscopy

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Abstract

In the present work, an efficient method was developed for the determination of lead in natural water samples. In this method, lead was extracted by octanoic acid reverse micelles (bulk phase) and measured by flame atomic absorption spectroscopy (FAAS). Ligand 1-(2-Pyridylazo)-2-naphthol was used as a chelating agent. In order to obtain the best extraction results, some experimental parameters (such as pH, ligand concentration, ionic strength, etc.) affecting the extraction efficiency were investigated and optimized. Under optimal conditions, the calibration curve was linear in the concentration range of 2.5–200.0 $\mu\text{g L}^{-1}$ with a correlation coefficient of 0.9998. The detection limit was 0.8 $\mu\text{g L}^{-1}$ and the preconcentration factor was 50. This method was successfully used to measure lead in natural water samples.

Keywords

Lead; Flame atomic absorption spectroscopy; Octanoic acid; Natural water samples.

1. INTRODUCTION

Heavy metals are environmental pollutants that human exposure to some of them through water and food can cause chronic and sometimes acute and dangerous poisoning. The term heavy metals refers to metals that have a relatively high density and atomic mass greater than iron, which are toxic in low concentrations [1,2]. Heavy metals such as lead, mercury, copper, cadmium, nickel, and chromium enter the body of living organisms in small amounts through food, drinking water, or air, and are deposited and accumulated in tissues such as fat, muscles, bones, and joints. This causes many diseases and complications such as neurological disorders, types of cancers, nutrient deficiency, hormonal imbalance, obesity, abortion, allergies and asthma, endocrine disorders, chronic viral infections, changes in fuel and it causes infertility, anemia, fatigue, headache and dizziness, loss of memory and hair loss, osteoporosis, insomnia and death, etc. in the body. In this research, the measurement and determination of lead ion is considered. Among the wide applications of lead, it can be used in the construction of building structures, pigments used in ceramic glaze and water pipes, decorative items, batteries and electronic components, cable coating, glass and ceramics, paints, etc. There is naturally lead in different concentrations in rocks, and during weathering and chemical erosion of

minerals, it enters the soil, water and air, and finally enters the food chain by entering plants. Lead is highly toxic and can cause chronic diseases such as headache, dizziness, tooth decay, reduced IQ, hearing loss, irritability, abdominal pain, nerve damage, kidney damage, blood pressure, lung and stomach cancer, etc. [3, 4]. So far, various methods have been used to measure this cation in different tissues, including inductively coupled plasma mass spectrometry [5], graphite furnace atomic absorption spectrometry [6], inductively coupled plasma atomic emission spectrometry [7] and flame atomic absorption spectrometry [8]. Among the mentioned methods, the flame atomic absorption spectrometry has received more attention and use due to its low cost and simplicity of operation. However, it is difficult to measure small amounts of lead in natural samples due to factors such as the low concentration of this element in the samples and matrix effects. To solve this problem, the use of a separation and preconcentration method is required. The most common preconcentration methods that have been used to measure this element in different samples are solid phase extraction [9], liquid-liquid extraction [10], precipitation and co-precipitation methods [11] and cloud point extraction [12]. Extensive efforts are always made to provide simple, efficient and environmentally friendly methods. One of these efforts is the introduction

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and development of coaservative extraction method. In this method, which is very similar to cloud point extraction, mass accumulations of micelles and admicelles formed by anionic or cationic surfactants act as an extracting phase in the solution and extract hydrophobic analytes into themselves [13,14]. The use of surfactants in the extraction process, due to being non-toxic and environmentally friendly, achieving a high extraction speed and preconcentration factor for the analysis of all kinds of analytes in environmental samples and the low amount of surfactant required for the separation of analytes, is of interest [15]. The agglomeration phenomenon was first reported in the early 20th century by Tiebackx, who worked on gelatin and gum arabic [16]. Following him, Bungenberg conducted a comprehensive study on the complex mass, which proposed the term coaservate [17]. In coaservative extraction method, by adding surfactant to the original sample with a concentration higher than the critical micelle concentration, a new phase called coaservate is formed. The bulk phase is the result of joining together 40 to 100 surfactant molecules and forming three-dimensional spherical clumps called micelles, with colloidal dimensions. After micelle formation, by an external factor such as temperature changes, pH changes, increase of an electrolyte or increase of a secondary solvent, these micelles, which are uniformly dispersed throughout the solution and are hydrated, dehydrated and placed next to each other and form a new phase inside the initial analyte solution [18]. As a result, the system becomes two-phase. The two existing phases are immiscible and in equilibrium with each other. By transferring the analyte from the aqueous solution to the extraction phase, separation and preconcentration and finally its measurement can be done [19-23].

In this research, coaservative extraction method was used to extract and preconcentrate lead ion from water samples and measure it with flame atomic absorption spectroscopy. In order to convert the lead cation into a hydrophobic complex, the ligand 1-(2-Pyridylazo)-2-naphthol has been used. In order to achieve the best extraction and measurement conditions, some experimental parameters affecting the extraction efficiency were investigated and optimized, which will be explained below.

2. EXPERIMENTAL

2.1. Reagents

All the chemicals used in this research were of analytical grade and deionized water was used to prepare the solutions. All materials used and ligand 1-(2-Pyridylazo)-2-naphthol were purchased from Merck, Germany.

2.2. Instruments

A flame atomic absorption spectrometer (GBC model Sens AA) with a lead hollow cathode lamp was used to determine the lead concentration. A German Hettich centrifuge (EBA20) was used to separate phases. A Japanese A&D analytical balance with an accuracy of one ten thousandth of a gram was used to weigh the materials. In order to measure the pH of the solutions, a pH meter made by Metrohem, model 827 was used.

2.3. Coaservative extraction of lead ions from water samples

For this procedure, 50 mL of aqueous sample containing lead ($2.5\text{-}200.0\ \mu\text{gL}^{-1}$) and 4×10^{-4} mol/L ligand 1-(2-Pyridylazo)-2-naphthol were transferred to a glass tube. Then 8 ml of 20% octanoic acid solution in THF was added to it. The solution was stirred and the pH of the solution was adjusted to 7 by adding nitric acid and sodium hydroxide. After adjusting the pH, the lid of the tube was closed and the solution was shaken for a few seconds. At this stage, in order to separate the aqueous and organic phase, centrifugation was done for 7 min at a speed of 3000 rpm. After centrifugation, the organic layer containing the extracted ions was above the aqueous layer in the test tube. Then the aqueous phase, which was completely clear, was removed using a syringe. After removing the aqueous phase, the volume of the organic phase was increased to 1 mL by adding $0.1\ \text{molL}^{-1}$ methanolic HNO_3 . Finally, this volume of the solution was transferred to the flame atomic absorption spectrometer and its absorption was measured.

3. RESULTS AND DISCUSSION

3.1. The effect of pH

pH plays an important role in the formation of the metal-ligand complex and as a result its extraction. Therefore, it should be checked and optimized. The results of this investigation are shown in Fig 1. Based on the obtained results, pH = 7 was chosen as the optimal value. The low extraction efficiency at low pH is due to the competition of H^+ ions with lead cation in the interaction with the ligand and the decrease in the extraction efficiency at pH higher than 7 is due to the competition of hydroxide ions with the ligand in the interaction with the lead cation.

3.2. The effect of chelating agent concentration

In the extraction of metal ions, the ligand is added to the solution containing the metal ion in order to form a hydrophobic complex between the metal ion and the ligand. The amount of ligand should be enough to form a complex with all the metal ions in the solution. For this purpose, the effect of the

amount of ligand was investigated in this experiment. The results of investigating the effect of ligand on extraction efficiency are presented in Fig 2. As can be seen, by increasing the amount of ligand and forming a stable complex with lead, the extraction efficiency increases quickly and the extraction is complete.

Therefore, the amount of $4 \times 10^{-4} \text{ molL}^{-1}$ of ligand was chosen as optimal.

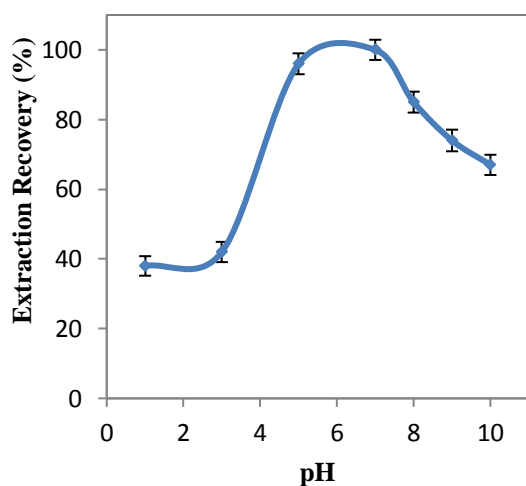


Fig. 1. The effect of pH on extraction recovery of lead

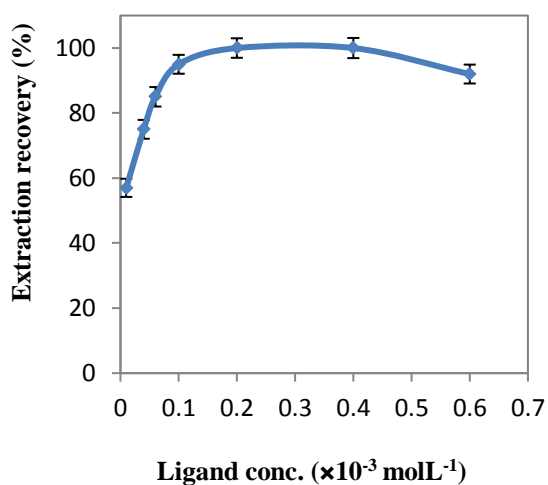


Fig. 2. The effect of ligand concentration on extraction recovery of lead

3.3. The effect of type of surfactant solvent on the extraction recovery

In order to investigate the effect of surfactant solvent, methanol, ethanol, acetone, acetonitrile and tetrahydrofuran were investigated. The solvent that mixes well with the sample matrix, has the ability to dissolve the surfactant and does not interfere in the extraction of the desired analyte, is suitable.

The test results for the mentioned solvents are shown in Fig 3. According to the results shown, THF was chosen as the optimal surfactant solvent.

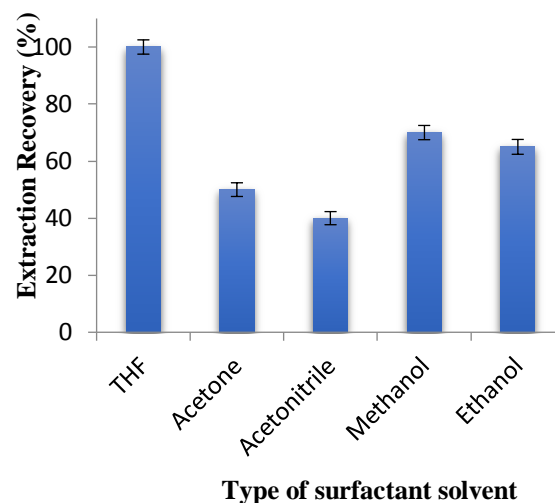


Fig. 3. The effect of surfactant solvent on extraction recovery of lead

3.4. The effect of amount of octanoic acid

In order to optimize the amount of surfactant, 2 to 8 ml of 20% octanoic acid solution in THF was investigated. The presented results are shown in Fig 4. As can be seen, the extraction efficiency increases up to 2.4% and reaches to full value and remains constant after that. Low amounts of extraction efficiency in low amounts of octanoic acid are due to its insufficiency for the extraction of lead complex. Therefore, 3.2% (8 ml of 20% solution) was chosen as the optimal amount.

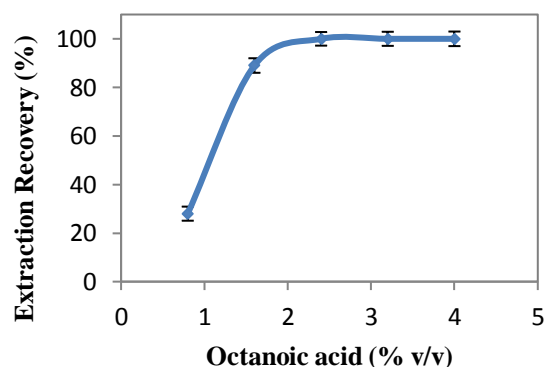


Fig. 4. The effect of amount of octanoic acid on extraction recovery of lead

3.5. The effect of ionic strength

In order to investigate the effect of salt on the efficiency of the method in the extraction of lead ions, different amounts of sodium chloride salt (0-25 % w/v) were added to 50 ml of a solution containing $80 \mu\text{gL}^{-1}$ of lead, and the extraction and measurement steps were carried out. The extraction efficiency was calculated. The results

showed that increasing the ionic strength does not affect the efficiency of lead extraction. This proves that this method is very resistant to high salt concentration.

3.6. The effects of centrifuge speed and time

The speed of the centrifuge should be enough to complete the phase separation. This parameter was investigated in the range of 2000 to 6000 rpm. According to the obtained results, at speeds higher than 3000 rpm, the two phases are completely separated. Therefore, the speed of the centrifuge in this method was considered 3000 rpm.

The centrifugation time should be chosen in such a way that the phases are separated and the test time is not prolonged. This parameter was examined in the range of 1 to 10 min. The results showed that by increasing the centrifugation time up to 5 min, the extraction efficiency increased and longer time has no effect on the extraction efficiency of lead. As a result, a time of 7 minutes has been considered to ensure the complete separation of the phases in the next tests.

3.7. Matrix effects

To investigate the interference of different ions on lead ion extraction, different concentrations of interfering ions were added to the solution containing 80 μgL^{-1} of lead cation and extraction was performed. The results of this study are given in Table 1. In this extraction, an interfering ion is said to cause a change of $\pm 5\%$ in the extraction efficiency. It can be seen that the interfering ions investigated in this research with the shown concentrations do not cause serious disturbance in the preconcentration and determination of lead.

Table 1. Effect of interfering ions on the extraction of lead

Interfering ions	Ion/Pb(II) ratio (w/w)	Recovery (%)
K ⁺ , Na ⁺ , Li ⁺ , Ca ²⁺ , Ba ²⁺ , Mg ²⁺ , Br ⁻ , F ⁻ , I ⁻ , Cl ⁻ , NO ₃ ⁻ , Fe ³⁺ , PO ₄ ³⁻ , Mn ²⁺ , Ag ⁺ , Zn ²⁺ , Cu ²⁺ , Cr ³⁺ , Hg ²⁺ , As ³⁺ , Cd ²⁺ , Cr (VI), CO ₃ ²⁻ , SO ₄ ²⁻ , Fe ²⁺	100	99.5

Table 3. Determination of lead in water samples

Sample	Lead amount (μgL^{-1})		Recovery (%)
	Added	Found	
Tap water (Drinking water system of Sari, Iran)	0.0	—	—
	100.0	99.4 (3.4)*	99.4
	150.0	149.8 (3.0)	99.9
Mineral water (Damavand mineral water, Iran)	0.0	—	—
	100.0	98.9 (3.2)	98.9
	150.0	148.8 (3.3)	99.2

Al ³⁺ , Ni ²⁺ , Co ²⁺	100	99.0
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3.8. Figures of merit

Table 2 presents the figures of merit obtained for coextractive extraction of lead. The results are: Dynamic linear range for Pb determination varies from 2.5 to 200.0 μgL^{-1} ($A = 0.0012C + 0.0287$, $R^2 = 0.9998$). The relative standard deviation (RSD) was obtained as 3.0 % ($n = 10$, $C = 80 \mu\text{gL}^{-1}$); while limit of detection (LOD) ($3S_b/m$) and limit of quantification (LOQ) were measured as 0.8 and 2.5 μgL^{-1} , respectively. The preconcentration factor was obtained as 50 for 50 mL of sample solution.

Table 2. Analytical characteristics of the method.

Parameter	Analytical feature
Linear range, μgL^{-1}	2.5-200.0
Limit of detection, μgL^{-1} ($n = 10$)	0.8
Limit of quantification, μgL^{-1} ($n = 10$)	2.5
RSD* (%)	3.0
Preconcentration factor	50

* Pb concentration was 80 μgL^{-1} for which RSD was obtained

3.9. Lead determination in real samples

To check the effectiveness of the method in extracting and measuring lead from water samples, water samples with different matrices were examined. Water samples were selected from Sari city water system, Tajan river water, Caspian Sea water, Damavand mineral water, rain water and NonNel Amol spring water. The results of the analysis of the above samples are shown in Table 3. As the results show, except for the river water, the rest of the samples did not show lead contamination. The amount of lead in the river water was 12 μgL^{-1} . In the following, the accuracy of the method was checked and evaluated by adding different amounts of lead to the samples and extracting and measuring. The values of relative efficiencies show that the present method has a good efficiency in extracting and measuring lead in water samples.

River water (Tajan river, Sari, Iran)	0.0 100.0 150.0	12.0 111.0 (3.2) 159.9 (3.5)	– 99.0 98.6
Spring water (NonNel Amol spring water)	0.0 100.0 150.0	– 99.5 (3.0) 148.6 (3.8)	– 99.5 99.1
Rain water	0.0 100.0 150.0	– 98.8 (3.4) 148.6 (3.2)	– 98.8 99.1
Sea water (Caspian Sea water, Sari, Iran)	0.0 100.0 150.0	– 101.1 (3.2) 151.0 (3.1)	– 101.1 100.7

* RSD of three replicate experiments

Table 4. Comparison of the presented method with other preconcentration methods (determination by FAAS)

Method	LOD ^a (μgL^{-1})	RSD. ^b (%)	Linear range (μgL^{-1})	PF ^c	Sample volume (mL)	Ref.
Solid phase extraction	1.8	< 10%	27*	100	500	[24]
Solid phase extraction	7.5	0.27	5-50	93	15	[25]
Cloud point extraction	8.0	2.7	20-300	35	35	[26]
Dispersive liquid liquid microextraction	8.3	5.9	27.4*	30	15	[27]
Dispersive liquid liquid microextraction	12.0	6.5	-	10	5	[28]
Coacervative extraction	0.8	3.0	2.5-200.0	50	50	This work

^a Limit of detection, ^b Relative standard deviation, ^c Preconcentration factor, * Low limit

4. CONCLUSION

In the present work, coacervative extraction method was used to extract lead from water samples. Effective parameters in this method were checked and optimized. In Table 4, merit figures of the proposed method are compared with other preconcentration methods. This table shows that the proposed method is superior to other methods in many parameters. The coacervative extraction method has many advantages, which can be attributed to things such as convenience and simplicity of the method, high safety, low cost, short operating time, energy saving, low amount of surfactant used, improvement of analysis conditions, no need for expensive laboratory tools and equipment, environmentally friendly and wide range of analyzed analytes with different polarities. The results of this research show that this method has a very good ability to measure lead in water samples.

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جداسازی و پیش تغلیظ مقادیر بسیار کم یون سرب به روش استخراج توده ای و اندازه گیری با طیف سنجی جذب اتمی شعله ای

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چکیده

در کار حاضر، یک روش کارآمد برای تعیین سرب در نمونه های آب طبیعی توسعه داده شد. در این روش سرب توسط میسل های معکوس اکتانوتیک اسید (فاز توده ای) استخراج و توسط طیف سنجی جذب اتمی شعله ای (FAAS) اندازه گیری شد. لیگاند ۲و۱ - پیریدیل آزو-۲- نفتول به عنوان عامل کیلیت کننده استفاده شد. برای به دست آوردن بهترین نتایج استخراج، برخی از پارامترهای تجربی (مانند pH، غلظت لیگاند، قدرت یونی و...) موثر بر بازده استخراج بررسی و بهینه سازی شدند. تحت شرایط بهینه، منحنی کالیبراسیون در محدوده غلظت ۲/۵-۲۰۰ میکروگرم بر لیتر با ضریب همبستگی ۰/۹۹۹۸ خطی شد. حد تشخیص ۰/۸ میکروگرم بر لیتر و فاکتور پیش تغلیظ ۵۰ به دست آمد. این روش با موفقیت برای اندازه گیری سرب در نمونه آب های طبیعی مورد استفاده قرار گرفت.

کلید واژه ها

سرب؛ دستگاه جذب اتمی شعله ای؛ اکتانوتیک اسید؛ نمونه آب های طبیعی.