

## An On-Line Preconcentration System Based on Graphitic Carbon Nitride Coated Membrane Filter for the Fast Determination of Trace Levels of Lead in Water Samples

Ehsan Zolfonoun\*, Seyed Reza Yousefi

1. Nuclear Fuel Cycle Research School, Nuclear Science & Technology Research Institute, End of North Karegar Ave., P.O. Box 14395-836, Tehran, Iran

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### Abstract

A ligandless on-line solid phase extraction method was developed for the preconcentration of lead prior to quantitation by inductively coupled plasma-optical emission spectrometry. In this method, the sample solution was passed through a syringe membrane filter coated with graphitic carbon nitride and Pb(II) ions were directly adsorbed on the surface of g-C<sub>3</sub>N<sub>4</sub> particles. The adsorbed Pb(II) ions were subsequently eluted from the membrane and transferred directly into the ICP-OES nebulizer with nitric acid solution. Under the optimized conditions and preconcentration of 25 mL of sample, the enhancement factor of 110 and the detection limit of 0.12 µg L<sup>-1</sup> were obtained. The proposed procedure was applied for the preconcentration and determination of lead in environmental water samples.

### Keywords

On-Line Preconcentration; Solid Phase Extraction; Graphitic Carbon Nitride; Lead; ICP-OES.

## 1. INTRODUCTION

The increase in the use of heavy metals in various industries over the past decades has resulted in an increased amount of metals in the environment [1]. Most of the heavy metals are not only environmentally destructive but also healthily hazardous even at trace levels. Lead is one of the most hazardous elements to human health. Metabolic poisoning, enzyme inhibition and nervous connection damages are the most important toxic effects of lead on the human body [2]. Various analytical techniques have been developed for the accurate determination of trace levels of lead in environmental and biological samples [3-5].

In most of the reported analytical techniques for the determination of trace amounts of lead, sample preparation (separation and preconcentration) step is needed prior to instrumental analysis. Among the different preconcentration methods, solid phase extraction (SPE) is a most widely used technique because of its advantages including high enrichment factor, quick phase separation and low consumption of organic solvents [6,7]. The SPE technique not only can be performed in the off-line mode but also provides the possibility of on-line coupling to analytical instruments. It enables partial or total automation of the analytical process, reduces analysis time, decreases analyte loss, increases sensitivity, and improves accuracy and

precision [8,9]. In recent years, great attention has been devoted to the application of nano- and micro-size materials, as sorbent in sample preparation techniques [10-12]. Unique size and desirable physicochemical properties of these materials makes them ideal sorbents in SPE for separation and preconcentration of trace analytes [13-15].

Graphitic carbon nitride (g-C<sub>3</sub>N<sub>4</sub>) is a new form of organic polymer-like materials that has received a great deal of interest for a wide range of applications, because of its unique structure and physicochemical properties [16-18]. It had been reported as a new sorbent for the extraction or removal of metal ions and aromatic compounds with high sorption capacity [19,20]. g-C<sub>3</sub>N<sub>4</sub> possesses N-functional groups (-NH<sub>2</sub>/-NH-/=N-), which are regarded as suitable sites for capturing metal ions. The previous studies demonstrated that the functional groups of C-N and N-H, could interact with metal ions by forming complexes. The adsorption of metal ions on the g-C<sub>3</sub>N<sub>4</sub> is ascribed to the strong surface complexation between the nitrogen-containing functional groups of the g-C<sub>3</sub>N<sub>4</sub> and metal ions [20]. In the current study, we propose a simple, fast and ligandless on-line preconcentration technique using graphitic carbon nitride coated cellulose acetate membrane for the determination of lead by inductively coupled plasma-optical emission spectrometry.

\*Corresponding Author: ezolfonoun@aeoi.org.ir

## 2. EXPERIMENTAL

### 2.1. Reagents

All chemicals used in this work were of analytical grade. All aqueous solutions were prepared in double-distilled deionized water (Milli-Q system, Millipore, USA). Pb(II) stock solution was supplied by Merck (Darmstadt, Germany). Nylon membrane filters (pore size, 0.45  $\mu\text{m}$ ; diameter, 13 mm) were purchased from Merck Millipore (Burlington, MA, USA). The  $g\text{-C}_3\text{N}_4$  was synthesized according to the previously reported method [16].

### 2.2. Instrumentation

A Perkin Elmer Optima 7300 DV optical emission spectrometer equipped with a charge-coupled device detector (CCD) and a cyclonic spray chamber with a concentric nebulizer was used for the determination of the target element. The detection wavelength for lead was 220.353 nm. The argon gas with 99.999 % purity for ICP-OES was purchased from Roham Gas Company (Tehran, Iran). A Metrohm model 744 digital pH meter, equipped with a combined glass-calomel electrode, was employed for the pH measurements. An ultrasonic water bath with temperature control (Tecno-Gaz SpA, Italy) was applied to disperse of adsorbent particles in aqueous solution. A scanning electron microscope (SEM) (TESCAN, MIRA III, Czech Republic) was used to examine the morphology and surface texture of the prepared  $g\text{-C}_3\text{N}_4$ .

### 2.3. On-line extraction procedure

The  $g\text{-C}_3\text{N}_4$  coated membrane filter was fabricated in the laboratory from dispersed  $g\text{-C}_3\text{N}_4$  particles in water. 25.0 mg of  $g\text{-C}_3\text{N}_4$  was added to 10 mL water and sonicated by an ultrasonic bath for 2 min. After that, while the mixture was sonicated, it was passed through a nylon syringe membrane filter. The on-line preconcentration system coupled to the ICP-OES spectrometer is illustrated in Fig. 1. Aliquot of 25 mL of the sample or standard solution containing Pb(II) was pumped through the sample line and fed onto the membrane filter. The sample line was washed with 5 mL deionized water for the removal of the matrix components before elution. Finally, by switching the three-way valve, the eluent solution was fed onto the membrane filter to desorb the target analyte from the sorbent particles. The eluted solution was directly introduced into the ICP-OES nebulizer and the emission signal of lead was measured against the time. The results were expressed as peak-height emissions, which were corrected against the blank signal.

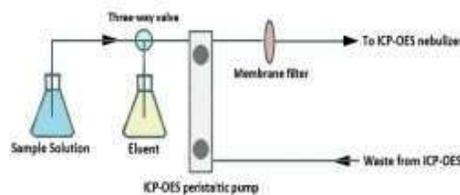


Fig. 1. Set-up of on-line extraction system.

## 3. RESULT AND DISCUSSION

### 3.1. Characterization of the sorbent

Fig. 2 shows SEM image of the prepared  $g\text{-C}_3\text{N}_4$ . According to this image, the synthesized  $g\text{-C}_3\text{N}_4$  has folded lamellar flakes texture. Also as can be seen from Fig. 2, the diameters of most particles are around 1  $\mu\text{m}$ .

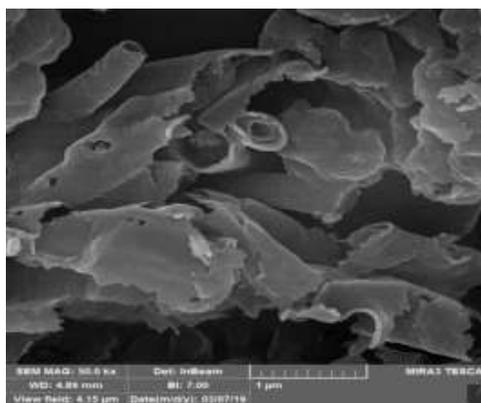


Fig. 2. SEM image of the  $g\text{-C}_3\text{N}_4$ .

### 3.2. Effect of pH

The pH of the sample solution is one of the most important factors in metal-adsorbent interaction in the SPE procedure. The effect of pH on the extraction of Pb(II) by  $g\text{-C}_3\text{N}_4$  was studied in the range of 3.0–8.0 using nitric acid or sodium hydroxide. The results in Fig. 3 show that the adsorption of Pb(II) is maximum in the pH range of 6.0 to 7.5.

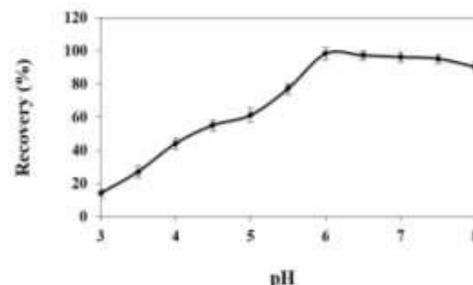
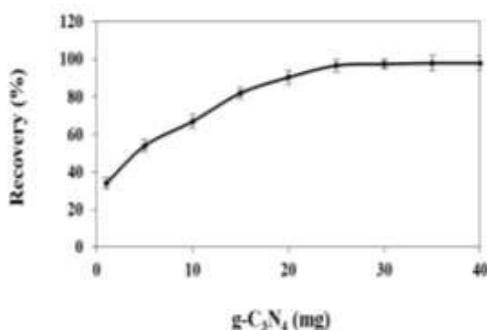


Fig. 3. Effect of pH on the recovery of Pb(II). Conditions:  $g\text{-C}_3\text{N}_4$  amount, 25 mg; concentration of analyte, 50  $\mu\text{g L}^{-1}$ .

The decrease in the extraction efficiency of lead at the lower pH values could be due to the competition between protons and the analyte for the surface functional groups of the g-C<sub>3</sub>N<sub>4</sub>. So, pH 6.0 was chosen as the optimum value.

### 3.3. Effect of g-C<sub>3</sub>N<sub>4</sub> amount

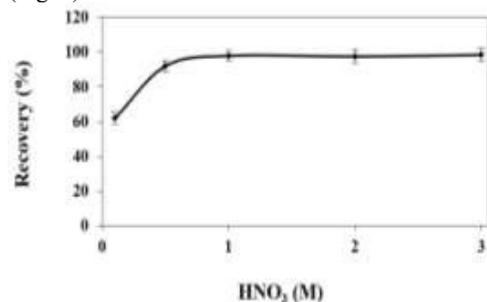
The effect of amount of g-C<sub>3</sub>N<sub>4</sub> on the quantitative extraction of Pb(II) was examined in the range of 1–40 mg. The results are shown in Fig. 4. The obtained results revealed that by increasing the sorbent amounts from 1 up to 25 mg, due to increasing accessible sites, the extraction efficiency increased and after that remained constant. Hence, the subsequent extraction experiments were carried out with 25 mg of g-C<sub>3</sub>N<sub>4</sub>.



**Fig. 4.** Effect of the g-C<sub>3</sub>N<sub>4</sub> amount on the recovery of Pb(II). Conditions: pH, 6.0; concentration of analyte, 50  $\mu\text{g L}^{-1}$ .

### 3.4. Effect of eluent type and flow rate

In order to find the best eluent, different mineral acids (HCl, H<sub>2</sub>SO<sub>4</sub>, HNO<sub>3</sub>) were examined as stripping agents. The results revealed that among the tested eluents, nitric acid was the superior stripping agent for the quantitative elution of Pb(II) from the membrane filter. Therefore, HNO<sub>3</sub> solution was selected for Pb(II) desorption. The effect of nitric acid concentration on the recovery of the adsorbed analyte was studied in the range of 0.1 to 3 mol L<sup>-1</sup> (Fig. 5).



**Fig. 5.** Effect of HNO<sub>3</sub> concentration on the recovery of Pb(II). Conditions: pH, 6.0; g-C<sub>3</sub>N<sub>4</sub> amount, 25 mg; concentration of analyte, 50  $\mu\text{g L}^{-1}$ .

Based on the obtained results, 1.0 mol L<sup>-1</sup> HNO<sub>3</sub> was sufficient for complete desorption of the target analyte from the sorbent surface. The effect of the eluent flow rate on the recovery of Pb(II) was examined in the range of 1.0–5.0 mL min<sup>-1</sup> and the flow rate of 2.0 mL min<sup>-1</sup> was selected as the optimum value for further experiments.

### 3.5. Effect of sample flow rate

The effect of flow rate of the sample solution through the membrane filter on the recovery of Pb(II) was studied. It was found that, in the range of 1–3.0 mL min<sup>-1</sup>, the retention of lead by the membrane is not affected by the flow rate of the sample solution. Consequently, for reduction of total analysis time, sample flow rate of 3.0 mL min<sup>-1</sup> was selected as the optimum sample flow rate.

### 3.6. Effect of diverse ions on the recovery

In order to evaluate the analytical applicability of the developed method, the effect of commonly occurring ions in natural water samples on the extraction and determination of lead was studied. In these experiments, 25 mL of sample solutions containing 50.0  $\mu\text{g L}^{-1}$  of Pb(II) and various amounts of interfering ions were treated according to the recommended procedure. Tolerable limit was set as the highest amount of foreign ions which cause an approximately  $\pm 5\%$  relative error in the determination of the analyte. The results showed that 40,000-fold Li<sup>+</sup>, Na<sup>+</sup>, K<sup>+</sup>, Cl<sup>-</sup>, F<sup>-</sup>, NO<sub>3</sub><sup>-</sup>, 20,000-fold Ca<sup>2+</sup>, Mg<sup>2+</sup>, Ba<sup>2+</sup>, Sr<sup>2+</sup>, SO<sub>4</sub><sup>2-</sup>, 400-fold Ag<sup>+</sup>, Cd<sup>2+</sup>, Co<sup>2+</sup>, Zn<sup>2+</sup>, Mn<sup>2+</sup>, 200-fold Fe<sup>3+</sup>, Ni<sup>2+</sup>, Cr<sup>3+</sup>, Ce<sup>3+</sup>, and 100-fold Al<sup>3+</sup>, Cu<sup>2+</sup>, Hg<sup>2+</sup> ions had no significant influence on the extraction and determination of Pb(II).

### 3.7. Analytical figures of merit

The analytical parameters of the proposed are summarised in Table 1. Linear working range of the method for determination of Pb(II) was found to be 0.5–500  $\mu\text{g L}^{-1}$ . The enhancement factor for the proposed method is 110, as obtained from the ratio of the slopes of the calibration curves with and without preconcentration.

**Table 1.** Analytical parameters of the proposed method.

Parameter	Analytical feature
Linear range ( $\mu\text{g L}^{-1}$ )	0.5–500
r <sup>2</sup>	0.996
LOD (ng L <sup>-1</sup> )	0.12
R.S.D. % (n = 10)	2.6
Sample volume (mL)	25
Enhancement factor	110

The limit of detection (LOD) of the proposed method was calculated as three times the standard deviation of 10 measurements of the blank solution over the slope of the calibration curve. The LOD

for the determination of Pb(II) was found to be  $0.12 \mu\text{g L}^{-1}$ . The relative standard deviation (R.S.D) of the proposed method for determination of  $50.0 \mu\text{g L}^{-1}$  Pb(II) (n= 10) was 2.6 %.

### 3.8. Application

The accuracy of the proposed method was evaluated, by the analysis of water certified reference materials, TMDA-53.3 (environmental water) and ERM-CA-713 (waste water), for the determination of Pb(II). The results are given in Table 2.

**Table 2.** Determination of lead certified reference materials.

Sample	Found value ( $\mu\text{g L}^{-1}$ )	Certified value ( $\mu\text{g L}^{-1}$ )
TMDA-53.3	358 (2.5) <sup>a</sup>	349
ERM-CA-713	45.5 (3.0)	49.7

<sup>a</sup> Values in parentheses are R.S.D.s based on three replicate analyses.

As can be seen, the obtained results are in good agreement with the certified values. Also the developed method was applied to find the amount of Pb(II) in tap water, well water and river water samples. The analytical results, along with the recovery for the spiked samples, are given in Table 3.

**Table 3.** Recovery of lead from water samples.

Sample	Added ( $\mu\text{g L}^{-1}$ )	Found ( $\mu\text{g L}^{-1}$ )	Recovery (%)
Tap water	0.0	<LOD	–
	10.0	9.6 (2.6) <sup>a</sup>	96
Well water	0.0	7.2 (3.1)	–
	10.0	17.9 (2.5)	107
River water	0.0	3.5 (3.6)	–
	10.0	13.0 (2.2)	95

<sup>a</sup> Values in parentheses are R.S.D.s based on three replicate analyses.

The recovery values calculated for the spiked samples were in the range of 95–107 %. The results demonstrated that the proposed on-line SPE method can be used as a reliable sample treatment

technique for extraction and determination of Pb(II) in real samples.

### 3.9. Comparison with other methods

The presented on-line procedure is compared with the some reported methods for extraction and determination of Pb(II) at Table 4. The proposed procedure shows low detection limit, low relative standard deviation and a high preconcentration factor. In comparison with the conventional SPE methods, the proposed on-line technique offers more simplicity and low sample consumption. Also, the proposed extraction method is ligandless and Pb(II) ions were directly adsorbed on the surface of g-C<sub>3</sub>N<sub>4</sub> particles without using any chelating agent.

## 4. CONCLUSION

An on-line preconcentration technique based on graphitic carbon nitride coated membrane filter was developed for the extraction and preconcentration of Pb(II) from aqueous samples, prior to ICP-OES determination. The developed on-line method facilitates the traditional SPE method, with high efficiency. Also, in this method, there is no need to use any chelating agent. The obtained results indicate that the proposed method gives a high enhancement factor and low LOD and can be used for the preconcentration and determination of lead in real water samples.

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**Table 4.** Comparative data from some reported methods for the preconcentration of lead.

Method	Detection technique	EF	Detection limit ( $\mu\text{g L}^{-1}$ )	R.S.D. (%)	Reference
Cloud point extraction	AAS	50	1.15	-	21
Magnetic solid-phase extractions	AAS	15	1.5	6.0	22
Dispersive liquid-liquid microextraction	AAS	125	1.1	-	23
Flow injection-Solid phase extraction	Spectro-photometry	50	0.9	7.8	24
Solid phase extraction	ICP-OES	7	4	8	25
Co-precipitation	ICP-OES	60	3.2	2.9	26
On-line ultrasound-assisted dispersive micro-solid phase extraction	ICP-OES	110	0.12	2.6	Present work

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## یک سیستم پیش تغلیظ آنالین بر اساس کربن نیتريد گرافیتی نشانده شده بر روی فیلتر غشائی برای اندازه گیری سریع مقادیر جزئی سرب در نمونه های آب

احسان ذوالنون<sup>\*</sup>، سیدرضا یوسفی

پژوهشکده چرخه سوخت هسته ای، پژوهشگاه علوم و فنون هسته ای، انتهای خیابان کارگر شمالی، صندوق پستی ۸۳۶-۱۴۳۹۵ تهران، ایران

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### چکیده

یک روش استخراج با فاز جامد آنالین و بدون لیگاند برای پیش تغلیظ سرب پیش از اندازه گیری با روش طیف سنجی نشری اتمی-پلاسمای جفت شده القائی توسعه داده شد. در این روش محلول نمونه از یک فیلتر غشایی سرسرنگی که بر روی آن کربن نیتريد گرافیتی نشانده شده است، عبور داده شده و یون های سرب مستقیماً بر روی سطح آن جذب می شوند. سپس یون های جذب شده بوسیله محلول اسید نیتريك واجذب شده و مستقیماً به داخل مه پاش طیف سنج وارد می شوند. تحت شرایط بهینه و پیش تغلیظ ۲۵ میلی لیتر نمونه، فاکتور تغلیظ ۱۱۰ و حد تشخیص ۰/۱۲ میکروگرم بر لیتر بدست آمدند. این روش برای پیش تغلیظ و اندازه گیری سرب در نمونه های آب زیست محیطی بکار برده شد.

### واژه های کلیدی

پیش تغلیظ آنالین؛ استخراج با فاز جامد؛ کربن نیتريد گرافیتی، سرب؛ ICP-OES.