

*Full Paper*

## **Development and Application of a Potentiometric Hg<sup>2+</sup>-Imprinted Polymer/graphitic Carbon Nitride/Carbon Paste Electrode**

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**Abstract-** A potentiometric electrode was developed for Hg<sup>2+</sup> through modifying a carbon paste electrode using Hg<sup>2+</sup>-ion imprinted polymer and graphitic carbon nitride (g-C<sub>3</sub>N<sub>4</sub>). The optimal composition of the developed electrode (Hg<sup>2+</sup>-IP/g-C<sub>3</sub>N<sub>4</sub>/ CPE) was observed for a composition of Hg<sup>2+</sup>-IP: g-C<sub>3</sub>N<sub>4</sub> nano-sheets: nujol oil: graphite powder at a weight ratio of 5:5:25:65 (w:w). The sensor's response had a Nernstian slope (30.5±0.4 mVdecade<sup>-1</sup>) and a short response time of around 10 s, and produced a linear response over a broad concentration window extending from 1.0×10<sup>-9</sup> M to 1.0×10<sup>-3</sup> M and a detection limit of 4.3×10<sup>-10</sup> M was determined for it. Effects of the presence of Na<sup>+</sup>, K<sup>+</sup>, Ag<sup>+</sup>, Ca<sup>2+</sup>, Mg<sup>2+</sup>, Cu<sup>2+</sup>, Ni<sup>2+</sup>, Zn<sup>2+</sup>, Cd<sup>2+</sup> and Co<sup>2+</sup> in the test solution, on the response of Hg<sup>2+</sup>-IP/g-C<sub>3</sub>N<sub>4</sub>/ CPE was evaluated and the sensor was found to be high selectivity. The developed sensor was also evaluated through analyzing the concentration of Hg<sup>2+</sup> in different water samples.

**Keywords -** Ion-imprinted polymers (IIP), Potentiometric sensors, Mercury determination, Graphitic carbon nitride (g-C<sub>3</sub>N<sub>4</sub>), Carbon paste electrode

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## 1. INTRODUCTION

Imprinting organic polymers has with different ions for developing new selective polymeric sorbents for the template ions has been a hot topic during the past decade [1].

As a useful method for preparing agent selective polymers, ion imprinting is a technique, based on allowing functional monomers to coordinate with a template (e.g. metal ion) and form a complex. This structure is next fixed through polymerization reactions involving initiating and cross-linking agents. As a final step the metal ions (template) are leached from the polymer structure through a physical or chemical procedure [2]. Further to the considerable selectivity of the resulting IIPs, the polymers also offer great adsorption capacities. The materials also enjoy a high stability against harsh chemical conditions [3]. Accordingly, the application of these polymers for preparing potentiometric [4,5] and voltammetric electrode [6-10] has attracted many scientists IIP-modified electrodes offer many of the advantages of various modifying collectively.

Mercury a very hazardous heavy metal [11], with considerable cellular toxicity, and bio-accumulative qualities in vital tissues, which lead to respiratory, renal, and reproductive conditions and event to death [12-14].

Consequently developing techniques and materials for use in a fast and sensitive devices for monitoring of Hg(II) has been very important. Classically, different approaches have been used to this end. These include cold vapor atomic absorption spectrometry [15,16], surface-enhanced Raman scattering [17], atomic fluorescence spectrometry [18,19], atomic absorption spectroscopy [20], and x-ray fluorescence spectrometry [21]. However the majority of these techniques are complex and costly.

Electrochemical methods have also been used in the analysis of heavy metal ions [22-26]. These tools offer advantages like high sensitivity, low response times and operation costs, and ease of miniaturization. One very interesting family is ion-selective electrodes (ISEs). ISEs have been successfully used for trace analysis in environmental and biological systems [27, 30]. Various ISEs based on different imprinted polymer have been developed for Ca(II) [31], Ag(I) [32], Cd(II) [33], Pb(II)[34], Hg(II) [35] and K(I) [36].

The advances in the area of preparing a wide range of nanomaterials have rather recently provided the chances for further improvements through developing new highly sensitive and trace analyses of heavy metals [37].

Carbon nanostructures, like single/double/multi-walled carbon nanotubes, fullerenes and graphene have been very attractive in this regard, given their high specific surface area, physico-chemical stability, wide range of applicable electrochemical potentials, and negligible charge-transfer resistance [36]. One recent step was the work on graphitic carbon nitride (g-C<sub>3</sub>N<sub>4</sub>) sheets, as a novel class of carbon nanomaterials, in the area of photo(electro) chemical applications including sensing, solar energy, photocatalysis, and regeneration of hydrogen. In this case, the free electron pairs of the nitrogen atoms present on the surface of nanosheets,

gives rise to the adsorption of metal ions and hence boosts the sensitivity of the corresponding tools.

The easy synthesis of g-C<sub>3</sub>N<sub>4</sub> nanosheets is a great advantage for its application in various areas. Large amounts of g-C<sub>3</sub>N<sub>4</sub> can be processed through bulk condensation of nitrogen-rich precursors like cyanimide, dicyandiamide, melamine, ammonium thiocyanate, urea, and thiourea [38-43].

The aim of this work was to develop a potentiometric Hg<sup>2+</sup>-selective electrode using an Hg<sup>2+</sup>-imprinted polymer (Hg<sup>2+</sup>-IP), and g-C<sub>3</sub>N<sub>4</sub>. After developing the sensor its potentiometric behavior in terms of detection limit, selectivity, linear range, slope, response time, pH range were evaluated and its applicability in the analysis of Hg<sup>2+</sup> concentration in water samples was also evaluated.

## 2. EXPERIMENTAL

**Chemicals** The nanoparticles of the Hg<sup>2+</sup>-imprinted polymer were prepared through reacting either methylene succinic or itaconic acid (ITA) (Merck) as the functional monomer, ethylene glycol dimethacrylate (EGDMA) as the cross-linker, and 2,2'-azobisisobutyronitrile (AIBN) (Sigma-Aldrich) as the initiator. g-C<sub>3</sub>N<sub>4</sub> was prepared using melamine obtained from Sigma-Aldrich. The CPEs were prepared using 1–2 μm graphite powder (Merck) and nujol oil (Aldrich). HgCl<sub>2</sub> (Merck) was used to prepare template solutions in double distilled water (DDW). The remaining chemicals were of analytical grade purity.

### 2.1. Instruments

The potentiometric measurements were performed using an Ag/AgCl/KCl reference electrode (Azar electrode, Iran) and the Hg<sup>2+</sup>-IP/g-C<sub>3</sub>N<sub>4</sub>/CPE as an indicator. The reference and indicator electrodes were connected through a mili-voltmeter, forming a cell as below:

Carbon paste electrode | sample Hg<sup>2+</sup> ion solution | Ag/AgCl/KCl (satd.)

The pH values of the solutions was set by adding 0.1 M solution of HNO<sub>3</sub> or NaOH by a Bante210 Benchtop pH Meter.

### 2.2. Preparation of g-C<sub>3</sub>N<sub>4</sub> nano-sheets

*Graphitic carbon nitride nano sheets were prepared as described elsewhere* [44]. To this end, bulk C<sub>3</sub>N<sub>4</sub> (b-C<sub>3</sub>N<sub>4</sub>) was first prepared through thermally treating 5 g of melamine at 550 °C in an sealed alumina crucible for 5 h. Next 1 g of the treated powder was subjected to refluxing in 100 mL of 5 M nitric acid solution at 125 °C for 12 h. The resulting mixture was then cooled to ambient temperature and the solid product was separated by centrifuging at 10,000 rpm for half an hour, and repeatedly rinsed with DDW to remove the acid residues. The supernatant was collected and sonicated for 10 h. The resulting suspension of g-C<sub>3</sub>N<sub>4</sub> nano-

sheets then filtered through a 45  $\mu\text{m}$  water phase microporous membrane and g-C<sub>3</sub>N<sub>4</sub> nano-sheets were stored as 300  $\mu\text{g mL}^{-1}$  dispersions.

### 2.3. The imprinted polymer nanoparticles

Nanoparticles of the Hg<sup>2+</sup> imprinted polymer were formed as described in a later work [45], through a two-step thermal precipitation polymerization approach. In the initial step, to 30 ml of acetonitrile 2 and 1 mmol of ITA and HgCl<sub>2</sub> were added under stirring and the solution was kept stirring for 5 h to facilitate the interaction of the species and the formation of an Hg<sup>2+</sup>-ITA self-assembly complex. The next step involved adding 8 mmol and 0.08 g of EGDMA and 2,2'-azobisisobutyronitrile to the reaction mixture, followed by purging the mixture with N<sub>2</sub> gas for 10 min, and sealing the reaction system. The polymerization reaction started at 65 °C and was allowed to continue for 24 h while stirring at 400 rpm. After the ending of the reaction, the polymer particles were repeatedly washed with methanol, and next the template (Hg(II) ions) were leached from the polymer through washing with a 2 M solution of hydrochloric acid. This was continued until no trace of the template ion could be detected in the supernatant by voltammetry. Eventually the particles were rinsed using DDW until the pH became neutral.

### 2.4. Preparation the sensor

The Hg<sup>2+</sup>-IP/g-C<sub>3</sub>N<sub>4</sub>/CPEs were prepared by thoroughly mixing different quantities of Hg<sup>2+</sup>-IP with different amounts of graphite powder, nojol oil, and g-C<sub>3</sub>N<sub>4</sub> nano-sheets to obtain a homogenous paste which was then filled in a plastic tube (2.0 mm in diameter). The packing was performed carefully, so as to avoid the formation of air gaps can stop the flow of currents through the sensor. From the opposite opening of the tube, a copper wire was carefully inserted into the paste to serve as an electrical contact. The outer surfaces of the CPEs were cleaned and smoothed using abrasive paper. The finalized electrodes were conditioned by being dipped in a 1.0 $\times$ 10<sup>-3</sup> mol L<sup>-1</sup> of Hg<sup>2+</sup> solution for one day, prior to use.

## 3. RESULTS AND DISCUSSION

### 3.1. Composition of the carbon paste

For the sake of comparison, two sets of modified and unmodified CPEs were prepared at various of compositions, the details of which are summarized in Table 1. The unmodified CPE, with all other ingredients similar to the optimized composition (electrode No. 1) revealed a slope of 10.2 $\pm$ 0.6 mV decade<sup>-1</sup>, while adding 5% of the IIP to the paste greatly improved the response (electrode No. 2) reflecting the modifying effect of the IIP. This effect could be attributed to the specific interactions of Hg<sup>2+</sup> with the empty sites of the IP.

Studies on the effect of incorporating g-C<sub>3</sub>N<sub>4</sub> in the CPE composition indicated that the presence of 5% of g-C<sub>3</sub>N<sub>4</sub> further improves the response. This could be due to the fact that the presence of g-C<sub>3</sub>N<sub>4</sub> enhances the conductivity of the electrode, and hence improves the conversion of the chemical signal to an electrical signal.

Based on all observations, electrode No. 5 composed of 65% graphite, 25% nujol oil, 5% Hg<sup>2+</sup>-IP and 5% g-C<sub>3</sub>N<sub>4</sub> had the best performance and hence this composition was used in the rest of the experiments.

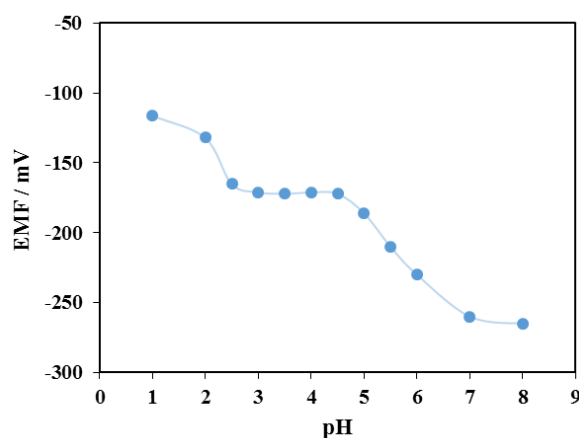
**Table 1.** The optimization of the Hg<sup>2+</sup>-IP/g-C<sub>3</sub>N<sub>4</sub>/CPE

No.	Graphite (wt%)	Nujol (wt%)	IIP (wt%)	g-C <sub>3</sub> N <sub>4</sub> (wt%)	Linear Range (mol L <sup>-1</sup> )	Slope (mV decade <sup>-1</sup> )
1	75	25	---	---	1.0×10 <sup>-2</sup> - 5.0×10 <sup>-3</sup>	10.2±0.6
2	70	25	5	---	1.0×10 <sup>-3</sup> - 1.0×10 <sup>-6</sup>	28.6±0.3
3	70	25	----	5	1.0×10 <sup>-2</sup> - 1.0×10 <sup>-4</sup>	15.1±0.7
4	67	25	3	5	1.0×10 <sup>-3</sup> - 5.0×10 <sup>-7</sup>	27.2±0.8
5 <sup>a</sup>	65	25	5	5	1.0×10 <sup>-3</sup> - 1.0×10 <sup>-9</sup>	30.5±0.4
6	63	25	7	5	1.0×10 <sup>-3</sup> - 1.0×10 <sup>-9</sup>	28.9±0.2
7	63	25	5	7	1.0×10 <sup>-3</sup> - 5.0×10 <sup>-8</sup>	29.2±0.3

**a** an optimized composition

### 3.2. Effect of pH

The influence of test solution pH on the potentiometric response of  $\text{Hg}^{2+}$ -IP/g- $\text{C}_3\text{N}_4$ /CPE with the optimal composition (electrode No. 5) was monitored by varying the pH of a  $10^{-5}$  M solution of  $\text{Hg}^{2+}$  from 1-8. This was achieved by adding proper amounts of concentrated  $\text{HNO}_3$  or  $\text{NaOH}$ . Based on the results (Figure 1), the response of the sensor remained almost constant in the pH window of 3.0–4.5. Yet the decrease in the potential signal at pH values higher than 4.5, is attributed to the formation of  $\text{Hg}^{2+}/\text{OH}^-$  complexes, decreasing the amount of free  $\text{Hg}^{2+}$  ions. Fluctuations of the response at pH values  $<3.0$ , on the other hand, arise from the protonation of the active sites of IIP nanopartilces.



**Fig. 1.** Effect of pH on the sensor response in  $1.0 \times 10^{-5}$  M  $\text{Hg}^{2+}$

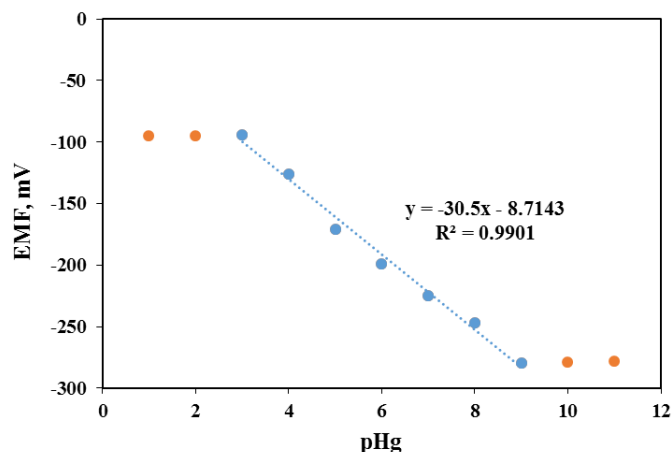
### 3.3. Response time

Response time, i.e. the time required for reaching at least 95% of the corresponding equilibrium potential from the time the electrode comes in contact with a solution [46-49] was examined through recording the potential readings every 2 s over 4 min. For the optimized, the response time was determined to be about 10 seconds.

### 3.4. Performance of $\text{Hg(II)}$ -IP/g- $\text{C}_3\text{N}_4$ /CPE

The potential response of the optimal electrode over a concentration range of  $1.0 \times 10^{-11}$ - $1.0 \times 10^{-1}$  mol  $\text{L}^{-1}$  was recorded and the calibration curve is illustrated in Figure 2. As it is clear, the potential response of the electrode decreased with decreasing the  $\text{Hg(II)}$  concentration from  $1.0 \times 10^{-3}$  mol  $\text{L}^{-1}$  to  $1.0 \times 10^{-9}$  mol  $\text{L}^{-1}$  with a slope of  $30.5 \pm 0.4$  mV decade $^{-1}$  (according to the Debye-Hukel equation, when the concentration is  $<0.1$  mol  $\text{L}^{-1}$ , the activity and concentration are considered to be approximately equal, so the concentration was used here for plotting).

The lower detection limit of the sensor was determined through extrapolating the linear portion of the electrode's calibration plot at low concentrations. This value was determined to be  $4.3 \times 10^{-10}$  M.



**Fig. 2.** The calibration curve of the  $\text{Hg}^{2+}$  selective electrode based on  $\text{Hg}^{2+}$ -IP/g- $\text{C}_3\text{N}_4$ /CPE

### 3.5. Selectivity evaluations

As the most critical quality of any sensor, selectivity describes the tendency of an ion selective electrode to the target ion while other ions are present. Using the matched potential method (MPM) [50-54], the selectivity coefficients of the optimal electrode were determined (Table 2) using a  $1.0 \times 10^{-7}$  mol  $\text{L}^{-1}$  solution of  $\text{Hg}(\text{II})$ , in the presence of varying concentrations of interfering ions in the range of  $1 \times 10^{-6}$  to  $1.0 \times 10^{-1}$  mol  $\text{L}^{-1}$ .

**Table 2.** The selectivity coefficients of various interfering cations for electrode

Interfering ion	Selectivity Coefficients	Interfering ion	Selectivity Coefficients
$\text{Na}^+$	$<10^{-4}$	$\text{Cd}^{2+}$	$3.6 \times 10^{-4}$
$\text{K}^+$	$<10^{-4}$	$\text{Ni}^{2+}$	$6.2 \times 10^{-4}$
$\text{Ag}^+$	$2.6 \times 10^{-4}$	$\text{Cu}^{2+}$	$2.5 \times 10^{-4}$
$\text{Ca}^{2+}$	$<10^{-4}$	$\text{Co}^{2+}$	$3.1 \times 10^{-4}$
$\text{Mg}^{2+}$	$1.1 \times 10^{-4}$	$\text{Zn}^{2+}$	$4.7 \times 10^{-4}$

### 3.6. Analytical application

To evaluate the applicability of the sensor for environmental monitoring applications, the  $\text{Hg}^{2+}$ -IP/g- $\text{C}_3\text{N}_4$ /CPE was used for the analysis of different water samples, spiked with 2.5, 12.5, and 20  $\text{nmol L}^{-1}$  of  $\text{Hg}^{2+}$  were spiked into the water samples. The summary of the data in Table 3 show that the recovery values are acceptable. Hence the  $\text{Hg}^{2+}$ -IP/g- $\text{C}_3\text{N}_4$ /CPE was concluded as a simple and accurate tool for the analysis of real samples.

**Table 3.**  $\text{Hg}^{2+}$  contents in two water samples

sample	Added ( $\text{nmol l}^{-1}$ )	Found ( $\text{nmol l}^{-1}$ )	Recovery (%)
<b>Tap water</b>	0.0	<LOD	----
	2.5	2.43	97.2
	12.5	12.16	97.28
	20.0	20.09	100.4
<b>Sea water</b>	0.0	<LOD	----
	2.5	2.53	101.2
	12.5	12.84	102.72
	20.0	19.87	99.35

## 4. CONCLUSION

Nanoparticles of an ion-imprinted polymer and graphitic carbon nitride were used to develop a modified CPE as a potentiometric electrode for the determination of  $\text{Hg}^{2+}$ . The IIP was used as the sensing element of the sensor. Given the specific interactions of  $\text{Hg}^{2+}$  with the recognition sites of  $\text{Hg}^{2+}$ -IP, was expected and found to be highly selective to  $\text{Hg}^{2+}$ . Advantages of the CPE include a wide dynamic range, low detection limit, and short response time. The potentiometric sensor was found to be applicable for simpler, cheaper, more specific analysis of mercury ions in aqueous media.

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