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A New Potentiometric Sensor Based on Carbon Paste Electrode Modified with Methacrylic Acid Based-Ion Imprinted Polymer for Nickel Ion Determination

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Abstract- A novel ion-imprinted polymer (IIP)-based potentiometric sensor is introduced for precise determination of Ni²⁺ ions. The imprinted polymer was prepared using methacrylic acid and ethylene glycol dimethacrylate as functional monomer and cross-linker. This potentiometric sensor is a carbon paste electrode, composed of IIP particles (4%), graphite (64%), n-eicosane (25%), and sodium tetra phenyl borate (5%) in its optimum condition. The sensor exhibited a Nernstian slope of 29.1 ± 0.4 mVdecade⁻¹ in the concentration range of 1.0×10^{-6} to 1.0×10^{-1} mol L⁻¹ with a detection limit of 5×10^{-7} mol L⁻¹. The sensor demonstrated a stable potential response in the pH range of 4–7 and fast response times below 1 minute. Thanks to the high selectivity of the IIP-based probe, common interfering species had almost no effect on its response toward Ni²⁺ ions. The proposed sensor was prosperously applied to analyze the Ni²⁺ content of real sea, lake, and industrial water samples.

Keywords- Carbon paste; Ion imprinted polymer; Potentiometry; Nickel ion; Methacrylic acid

1. INTRODUCTION

Nickel is a toxic heavy metal that can be found as a contaminant in vegetables and dairy products. The source of this contamination can be industries that use nickel in their production lines. Studying the hazardous effects of nickel on human health showed that its high concentrations could cause acute pneumonitis, dermatitis, asthma, eczema, disorders of the

respiratory system, and cancer of the lungs and nasal cavity [1-6]. According to the *World Health Organization (WHO)*, the permissible limit of nickel in drinking water is 0.2 mg/l. Therefore, accurate determination of trace levels of nickel in environmental samples is of high importance [7-11]. Several different methods, such as atomic absorption spectrometry (AAS) [12, 13], flame atomic absorption spectrometry-electro thermal atomization, ICP-AES and flame photometry [14, 15] have been used for the determination of nickel. However, these methods are usually accompanied by sample pretreatment steps and therefore they are not very convenient methods to be used in the routine analysis of a large number of environmental samples. So there is a critical need for the development of a selective, portable, inexpensive diagnostic tool for the determination of nickel [16-19].

Electrochemical sensors especially ion-selective electrodes (ISEs) based on plasticized PVC membranes have found a wide application in the determination of various species. For nickle ions, various ionophores including crown ethers [20], porphyrins [3], pentacyclooctaaza, cyclams, [16, 21, 22] and Schiff's bases [20, 23-25] have been used in the development of ISEs. Some of these ionophores suffer from drawbacks such as poor selectivity, non-Nernstian response, long response time, and poor reproducibility. Recently, many of these problems are overcomed by replacing the old sensing agents with a novel material called ion-imprinted polymer (IIPs) [26-31].

An IIP is a synthetic polymer with selective ionic recognition properties. This characteristic is assigned to the nature and positioning of functional groups, situated within the recognition cavities of the polymer matrix that are complementary to the analyte in size and shape [27, 29, 31-33].

In general, preparation process of an IIP involves mixing the template and a proper monomer followed by polymerization in presence of a cross-linker. Finally, removal of the template from the polymer matrix deposits selective binding sites which can be selectively occupied with the target molecule [4, 34, 35].

Potentiometric carbon paste electrodes (CPEs) have several privileges including ease of fabrication, renewability of the surface, low cost, chemical inertness, robustness, stability of response, low ohmic resistance, no need for internal solution and compatibility with various types of modifiers. Hence, using IIPs as sensing agents in CPEs can result in robust assays for potentiometric determination of various analyses [36-46].

In this study, an IIP was synthesized for Ni^{2+} using methacrylic acid as monomer and ethylene glycol dimethacrylate as cross-linker. The IIP was then used for modifying a simple carbon paste electrode. After optimization of the electrode composition, it was used for the determination of Ni^{2+} . In the next step, all effective parameters on the electrode function were optimized to reach the best analytical performance. Finally, the proposed assay was used for the determination of Ni^{2+} in water samples from different sources.

2. EXPERIMENTAL SECTION

2.1. Chemicals and Instruments

Chemicals of analytical reagent grade were used during the synthesis and analysis. Nickel nitrate (Ni(NO₃)₂.6H₂O), Methacrylic acid (MAA) Acetonitrile, ethylene glycol dimethacrylate (EGDMA), 2, 2-azobisisobutyronitrile (AIBN), sodium tetraphenyl borate (NaTPB), hydrochloric acid (HCl) and sodium hydroxide (NaOH) were all purchased from Merck company (Germany).

Analysis of functional groups of the IIP were done using a thermo scientific (Nicolet IS 10) Fourier Transform Infrared (FTIR) spectrophotometer. A digital ion analyzer model pHS-3C was applied in measurement of potentiometric responses of the electrode. The surface morphology of the ion imprinted polymer was investigated on JSM-7800F scanning electron microscope (JEOL, Japan).

2. 2. Synthesis of the Ni²⁺-imprinted polymer

To prepare nickel imprinted polymers, 2 mmol of MMA, 1 mmol of Ni(NO₃)₂.6H₂O and 30 mL of acetonitrile were transferred to 100mL flask. Then, the resulting mixture was stirred up to 0.5 h for establishment of the equilibrium in complexion reaction. Following that, 8 mmol of EGDMA and 0.06 g of the initiator (AIBN) were added to the above solution and it was purged with N₂ gas for 10 minutes to remove O₂ and create inert atmosphere. The final mixture was sealed and immersed in an oil bath, stirred for 24 hours at 60 ° C. Then, the IIP was washed with ethanol 3 times to eliminate the unreacted monomers. To remove the template, the polymer was also washed six times with hydrochloric acid (2 mol L⁻¹). Finally, the particles were washed with distilled water to give a neutral pH and dried at 70 °C to remove the solvent. The synthesis route of the non-imprinted polymer (NIP) was similar to the IIPs except that it was done in the absence of Ni²⁺ ion.

2.3. Fabrication Ni²⁺-IIP sensor

To make a carbon paste electrode based on Ni²⁺ ion imprinted polymer, 65 mg of graphite, 5 mg of sodium tetra phenyl borate and 5 mg of powdered IIP were mixed in a mortar for about 15 min. Subsequently, 25 mg of n-eicosane was melted in a dish at 50 °C and blended with the previously prepared mixture using a stainless-steel spatula to achieve to a homogenous paste. The resultant paste was then packed into a plastic tube. Electrical contact was made by pushing a copper wire down the plastic tube into the back of the mixture. Renewing surface of the electrode was done by pushing an excess of the paste out of the tube and polishing it on a piece of paper. The unmodified carbon paste electrode was prepared by the same method except that the NIP particles was added to the mixture instead of IIP.

2.4. Potentiometric measurements for plotting the calibration curve

The IIP-based electrode was first conditioned by submerging it in nickel nitrate solution (0.01 mol L⁻¹, pH= 5.5) for 12 h followed by rinsing several times with double distilled water. Then, the potentiometric responses of the sensor toward Ni²⁺ solutions with different concentrations ranging from 1.0×10^{-7} - 1.0×10^{-1} mol L⁻¹ were recorded and plotted to obtain the calibration curve. All the potential responses of the sensor were recorded in room temperature and after waiting for 50 s, regarded as the electrode response time. The potentiometric measurements were performed by the following electrochemical cell:

Hg, Hg₂Cl₂, KCl(satd)||test solution| IIP based carbon paste electrode

2.5. Real sample preparation and determination

In order to evaluate potential of IIP based carbon paste electrode for determination of Ni^{2+} in real samples, three different real samples including sea water (collected from the south Caspian see), river water (collected from the Chitgar river) and industrial water were analyzed by the fabricated sensor. All samples were analyzed freshly right after the collection. Samples were filtered firstly to remove any suspended material and then diluted 10 times with double distilled water. The pH of these solutions was adjusted to 5.5 using PBS buffer. To check the accuracy of the method two different concentrations of Ni²⁺ was spiked to each real samples and the recorded potentials was inserted in the calibration curve equation formerly established for estimating the concentration of Ni²⁺ ions in real water samples.

3. RESULTS AND DISCUSSION

3.1. Characterization of the synthesized IIP

FT-IR analysis of the NIP, washed and unwashed IIP show that there was no considerable difference among the spectra of the examined polymers in all recorded regions except the region of~1500–1600cm⁻¹. These spectra are shown in Figure 1. Presence of strong stretching vibration band at ~1720 cm⁻¹ is related to the -C=O band, situated mainly at the surface of polymeric particles. The carboxylic acid -C-O band stretch and the -O-H bend vibrations appear at 1250 cm⁻¹,1450 cm⁻¹ and 1147 cm⁻¹. This bands can be seen in all examined polymers. There is a new band at 1554 cm⁻¹ in the FT-IR spectrum of unwashed IIP which can be assigned to the -C=O band linked to Ni²⁺ via coordination bonding. It can be seen that the washing of the IIP and removal of Ni²⁺ leads to vanishing this vibration band of the –C=O coordinated to Ni²⁺. These observations can be reliable evidence for the presence of a proper interaction among selective recognition sites in the IIP nanoparticles which are created during imprinting process. The similarity between the FT-IR spectra of the washed IIP and the corresponding NIP indicated that chemical structures of both polymers are alike.



Figure 1. FT-IR spectra of the unleached IIP, leached IIP, and NIP

The synthesized imprinted polymer was also characterized by Scanning electron microscopy image (SEM). Figure 2 shows the SEM image of the synthesized IIP. The image clearly shows that the synthesized IIP is a highly porous spherical structure which possesses pores in nanometer size regime.



Figure 2. Scanning electron microscopy image of synthesized IIP

3.2. Comparative evaluation responses of electrodes based on IIP and NIP

Initial potential response curves of the IIP- and NIP-based electrodes are displayed in Figure 3. It can be seen that the IIP-based electrode shows a near-Nernstian response of 23.837 mVdecade⁻¹ over the concentration ranges of 1.0×10^{-6} - 1.0×10^{-1} mol L⁻¹; but the electrode

modified with the NIP shows a sub-Nernstian slope equal with 0.8679 mV decade⁻¹ which means that the NIP-based electrode is almost insensitive to Ni²⁺ions. The IIP-based electrode demonstrates a meaningful sensitivity for the detection of target ions in comparison with the NIP based electrode. The better performance of IIP-based electrode is due to the selective interaction of the IIP sites with Ni²⁺ cations.



Figure 3. Comparison of the potentiometric response behaviors of the IIP and NIP-based electrodes to Ni^{2+} , fabricated and tested in the same conditions

3.3. Optimization

Asserting the IIP capability in selective recognition of Ni²⁺, the effect of different components of the electrode including the content of the IIP, n- icosane and sodium tetra phenyl borate on its performance was examined in the next step. For this aim, different electrodes with various electrode compositions were prepared and their potentiometric responses were recorded as a function of Ni(II) concentration. The results obtained are summarized in Table 1.

The IIP-based electrode, containing no sodium tetra phenyl borate, exhibits a sub-Nernstian response toward Ni²⁺ ions; whereas, addition of the sodium tetra phenyl borate in the IIP-less electrode results in no significant sensitivity to Ni²⁺ ions. This means that simultaneously presence of the IIP and sodium tetra phenyl borate is an essential requirement to see a considerable sensitivity and Nernstian response. It can be assumed that the sodium tetra phenyl borate, as a cation exchanger, participates in the exchange process of Ni²⁺ ions and creates such a synergetic effect with the IIP which results in a Nernstian response to Ni²⁺ ions. The sensitivity and linearity of the electrode response also depend on the amount of IIP, which determines the number of provided binding sites. However, when the IIP amount exceeds 4% in the electrode composition, the response slope decreases, probably due to the accumulation

of the IIP particles on the electrode surface and interfering the ions to electron transduction process. This phenomenon related to the fact that the IIP is intrinsically an insulating material. In general, the optimum potentiometric response was obtained with a 4% IIP, 5% sodium tetra phenyl borate, 66% graphite, and 25% n-eicosane. The electrode showed a Nernstian slope of 29.1 mV decade⁻¹ over the concentration range of 1×10^{-6} to 1×10^{-1} M with an LOD of 5×10^{-7} M.

Electrode number	Graphite	IIP	n-icosane	NaTPB	Linear range (mol L ⁻¹)	Slope
1	68%	2%	30%		10-6-10-3	y = 15.812x - 4.940
2	65%	2%	30%	3%	10-5-10-1	y =24.291x + 12.385
3	67%		30%	3%	10-4-10-1	y= 11.134x - 10.765
4	63%	4%	30%	3%	10-5-10-1	y = 26.123x - 161.08
5	61%	6%	30%	3%	10-6-10-3	y = 20.223x - 1.9867
6	59%	8%	30%	3%	10-4-10-1	y = 19.523x - 11.553
7	63%	4%	30%	3%	10-5-10-1	y = 26.123x - 161.08
8	68%	4%	25%	3%	10-6-10-1	y = 27.22x - 163.25
9	73%	4%	20%	3%	10-6-10-1	y = 22.223x - 153.18
10	56%	4%	35%	3%	10-6-10-2	y = 19.117x - 147.86
11	68%	4%	25%	3%	10-6-10-1	y = 27.22x - 163.25
12	70%	4%	25%	1%	10-6-10-3	y = 18.954x - 150.37
13	66%	4%	25%	5%	10-6-10-1	y = 29.117x - 163.63
14	64%	4%	25%	7%	10-6-10-1	y = 26.634x - 164.95

Table 1. The effect of IIP-based electrode composition on its response to Ni(II)



Figure 4. Solution pH effect on the potential response of optimized electrode to 1×10^{-5} mol L⁻¹ of Ni²⁺

pH dependent response of the IIP based carbon paste electrode was investigated by varying the pH of the 10^{-5} M of Ni²⁺ solution in range of 2 to 8 through adding the concentrated HCl

acid and NaOH base solutions and the results are illustrated in Figure 4. As can be seen, the potential of the electrode was nearly constant in the range of 4.0–7.0 which indicates the applicability of this electrode in the specified pH range; beyond this range, a considerable change occurs in the potential of the electrode. At pH values below 4.0, the changes in the potential can be attributed to the competition of the H⁺ with Ni²⁺ for occupying the binding sites in the IIP; while, at pH values above 7, the fluctuations can happen as a result of hydrolysis of the analyte (Ni²⁺) and formation of hydroxyl adduct of Ni²⁺ which leads to reduce the solubility of the ions in aqueous solution.

3.4. Analytical performance of the sensor

The potential responses of the sensor, assembled in optimized conditions (composition 13, Table 1), were used to obtain a calibration curve. The result is shown in Figure 5 (triplicate measurements was done at each concentration). As can be seen, the calibration curve demonstrates a linear response in the concentration range of $1 \times 10^{-6} - 1 \times 10^{-1}$ mol L⁻¹. The slope of the plot is about 29.1 (±0.5 mV decade⁻¹) with R²=0.998 which indicates that the sensor obeys the Nernstian law and also confirms that Ni²⁺ species are liable for the observed potential responses. Moreover, the limit of detection of the sensor was calculated to be 5×10^{-7} mol L⁻¹, based on the IUPAC definition.



Figure 5. Calibration curve plotted for Ni²⁺ determination using the optimized sensor

3.5. Study of response time and memory effect

Based on the IUPAC definition, the practical response time of an ISE is the time required for the response of an ion selective electrode to reach ± 1 mV of its equilibrium value after a tenfold change in the concentration of the sample solution. The response time was recorded by changing the Ni²⁺ concentration in solution over a concentration range of 1×10^{-6} to 1×10^{-1} mol L⁻¹. The actual potential versus time traces is shown in Figure 6. As it is apparent from the figure, the Ni²⁺ IIP based ISE reach to the equilibrium response within about 50 s. The rather quick response time was attributed to the fast exchange kinetics of the interaction between the analyte ions and the active sites on the IIP at the solution/IIP interface.



Figure 6. The dynamic response time of the sensor for step changes in the concentration of Ni²⁺: (a) 1.0×10^{-6} mol L⁻¹, (b) 1.0×10^{-5} mol L⁻¹, (c) 1.0×10^{-4} mol L⁻¹, (d) 1.0×10^{-3} mol L⁻¹, (e) 1.0×10^{-2} mol L⁻¹ and (f) 1.0×10^{-1} mol L⁻¹



Figure 7. Reversibility of sensor response as a result of a change in Ni^{2+} concentration from low-to-high and then high-to-low

The hysteretic effect was studied by quick changing of the concentration of nickel from 1×10^{-5} to 1×10^{-3} mol L⁻¹ (partially raising the curve) and subsequently reducing it from 1×10^{-3} to 1×10^{-5} mol L⁻¹ (descending part of the curve) in Figure 7. Obviously, the potentiometric response of the electrode, prepared with IIP particles, is fast (about 50 seconds) and reversible. In addition, it is evident that despite longer recovery times (70-80s) in the dilution phase, no unwanted memory or hysteretic effects are observable for the sensor.

3.6. Interference study

The matched potential method (MPM) was used to determine the potentiometric selectivity coefficients of possible interfering species. These coefficients demonstrate the preference of the developed ISE to an interfering ion (X) in comparison to analyte ions [47-51]. To determine the MPM coefficients, a specified concentration of Ni²⁺ ions (1.0×10^{-5} mol L⁻¹) was added to a reference solution and the equilibrium potential response was recorded. In other experiments, the interfering ions (X) were added stepwise to a similar reference solution until its potential reached the level matched with that obtained before the addition of the primary ions. Gathering these data, MPM selectivity coefficient (*K*Ni,X) was calculated using the equation below: K Ni,x = a_{Ni} / a_x

(1)

In this equation a_{Ni} and a_x are the activities of nickel and the interfering ions, respectively. The experimentally determined MPM selectivity coefficients, listed in Table 2, verify that none of the tested interfering ions has a serious effect on the response of the sensor towards the analyte.

Interference cation	Selectivity coefficients $(K^{\text{pot}}{}_{Ni}{}^{2+}; X^{Z+})$	Interference cation	$\begin{array}{c} \text{Selectivity coefficients} \\ (K^{\text{pot}}{Ni}^{2+};X^{Z+}) \end{array}$
Co ²⁺	3.7×10 ⁻²	$\mathrm{NH_{4}^{+}}$	7.9×10 ⁻³
Cu^{2+}	5.3×10 ⁻²	Fe^{2+}	3.8× 10 ⁻³
$\mathrm{Ag}^{\scriptscriptstyle +}$	8.3×10 ⁻²	Cd^{2+}	2.5×10 ⁻²
Mg^{2+}	9.7×10 ⁻³	Ca^{2+}	8.4×10 ⁻³
Zn^{2+}	5.8×10 ⁻³	Al^{3+}	3.1×10 ⁻³

Table 2. Selectivity coefficients ($K^{\text{pot}}_{Ni}^{2+}$; X^{Z+}) of the electrode for various interfering cations

3.7. Real sample analysis

Finally, the designed sensor was applied for determination of Ni²⁺ ions in river, industrial, and seawater samples to demonstrate the capability of the method for analyzing complex real samples. The validity of the results was checked by comparing them to those obtained from the atomic absorption spectrometry analysis of the samples, as a gold standard method. The results are summarized in Table 3. As can be seen, the proposed sensor acts sufficiently acceptable for

the determination of Ni(II) ions in real samples. Moreover, there was no significant difference between the determination results of the proposed sensor and those of the reference method (at a confidence level of 95%).

Water sample	Ni(II) added (ppm)	Ni(II) found (ppm)	RSD% (n = 4)	Flame-AAS method (ppm)	RSD% (n = 4)	Recovery (%)
Caspian sea						
		0.038	2.12	0.023	1.09	
	1	0.963	1.34	1.02	0.98	92.5
	2	1.95	2.33	2.15	0.74	95.6
Chitgar lake						
		0.041	1.81	0.029	1.14	
	1	0.975	1.96	1.012	0.83	93.4
	2	1.989	1.53	2.051	1.38	97.4
Industrial water						
		0.059	3.27	0.044	2.04	
	1	1.014	1.41	1.035	1.76	95.5
	2	2.012	2.5	2.041	1.31	97.65

Table 3. Determination of Ni^{2+} in different water sample sources by the IIP-based carbon paste electrode and a standard analytical method (n=3)

4. CONCLUSION

In this work, a nanostructured Ni^{2+} -imprinted polymer was introduced as a recognition element of an all-solid-state potentiometric sensor. The proposed sensor showed a Nernstian response toward Ni^{2+} species in a extensive concentration and pH range. Since the related blank electrode, holding the NIP material as the ionophore element, showed no sensitivity to Ni^{2+} , it can be concluded that the recognition capability of the electrode to Ni^{2+} is assigned to the Nickel ion compatible cavities, created during the imprinting process, in the polymeric matrix of the IIP. The proposed assay, exhibits high selectivity, stability, reversibility, and long shelf life, which make it an appropriate candidate to be applied in many analytical applications, particularly in the analysis of Ni^{2+} content of environmental samples. The suggested sensor introduces a promising and low-cost tool for Ni^{2+} analysis.

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Declarations of interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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