

FULL PAPER

Design and fabrication of carbon paste electrode for determination of Cr(III) ion in real water samples using a new synthesis Schiff base as selective ionophore

Esmat Shojaei  | Mahboubeh Masrournia*  | Ali Beyramabadi  | Hossein Behmadi *Department of Chemistry, Mashhad Branch, Islamic Azad University, Mashhad, Iran*

Chromium has been widely applied in industrial of pigment, stainless steel and electroplating and tanning procedure that enters the water sources. In this work, a simple and reproducible potentiometric sensor was developed for the determination of Cr(III) ions in real solid samples. For this purpose, a new Schiff base (4-((E)-(2-amino-4 chlorophenylimino) methyl) -5-(hydroxymethyl)-2-methylpyridin-3-ol) were synthesized based on a chemical methods and utilized as a novel and sensitive ionophore for the preparation of Cr(III)-carbon paste electrode. Percentage of carbon paste electrode composition including, graphite powder, ionophore, paraffin oil as a binder, and graphene oxide(GO), was optimized with a chemometric approach using a central composite design method. The optimum percentage of ionophore and nanomaterial in the electrode was obtained 15%w/w. The predicted slope by the computational method and the obtained slope by the experimental method for the proposed sensor was not significantly different. The sensor was linear in the range of 1×10^{-10} - 1×10^{-2} M for the determination of Cr(III). The effects of other parameters such as pH, response time, lifetime, and interfering ions were studied with one factor at the time method in the optimal composition of the sensor. Proper range of pH, lifetime and response time were acquired 3.0-9.0, 8 weeks and 10 s, respectively. The essential advantages of the proposed sensor were the suitable linear range, a wide range of pH, proper sensor stability, and fast response for the Cr(III) determination. Finally, the analytical application of prepared sensor evaluated to determine Cr(III) in waste and tap water samples.

***Corresponding Author:**

Mahboubeh Masrournia

Email: masrour@mshdiau.ac.ir

Tel.: +98 (51) 18414182

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Carbon paste electrode; Cr(III); Schiff base; potentiometric sensor; central composite design(CCD).

Introduction

Chromium is an important heavy metal with a hazardous impact on the environment. Chromium has been widely applied in industrial of pigment, stainless steel and electroplating and tanning procedure that enters to the water sources [1,2]. Also Cr (III) ion is an essential nutrient that affects in the

functioning of living organisms in humans [1-3]. Although Cr(III) ion is considered a best-selling supplement, further studies on the harmful effects of Cr (III) ion should be conducted in light of the newly obtained evidence[4]. According to the World Health Organization (WHO) and US Environmental Protection Agency(EPA), the permissible values of Cr(III) in drinking water are 0.05 mg

L⁻¹, [5]. So far, many methods have been developed to Cr (III) ion measurement with analytical instruments [6]. Among them, electrochemical techniques are suitable methods due to many advantages such as convenience, availability, simple operation and automation, low cost, fast response, and good selectivity [7-17].

Due to simple modification and easy regeneration of carbon paste electrodes (CPEs) [18-28], this type of electrodes were successfully applied for the fabrication of electroactive, drug, food and determination of various cations and organic compounds [29-37]. Although these sensors are very sensitive and suitable for measuring ions, there is a high tendency to provide highly selective and sensitive sensors especially for Cr(III) ion determination in real samples with complex matrices. For this purpose, it is essential to modify the sensors with new synthetic compounds to increase the sensitivity and selectivity and reduce the cost of producing the sensors. Among various electrochemical methods that can be applied to measure an analyte, the potentiometric method is a convenient procedure that is often chosen because of its cheapness, rapidity, availability, and no need for sophisticated tools for environmental sample analysis [38-40].

Schiff bases include the imine functional groups (-RC=N-) that are synthesized using a condensation reaction between amine functional groups in primary amine (R-NH₂) and carbonyl functional groups in ketones (RCOR') to form the azomethine bond [41, 42]. In the reaction, an aryl or alkyl group can be utilized as R and R'. These compounds have found great importance in the field of pharmaceuticals and medicinal to prepare various drugs and in other industries as pigments, catalysts, polymer stabilizers, and corrosion inhibitors [43]. The presence of functional groups, including nitrogen and oxygen atoms in a Schiff base, contributes to the formation of complexes between them with metal cations [44-47].

In this work, a potentiometric method was applied for determination of Cr(III) ion using a modified electrode, including graphite powder, paraffin oil as a binder, graphene oxide, and a new synthetic Schiff bases as ionophore 4- ((E)- (2-amino-4-chlorophenylimino) methyl) -5- (hydroxymethyl) -2- methylpyridin -3 -ol. (Figure1) The percentage of carbon paste electrode composition was optimized in one step with a chemometric approach to reduce the number of trials. Besides, the effects of other parameters such as pH, response time, lifetime, and interfering ions were studied with one factor at the time method in the optimal composition of the sensor. Finally, the sensors were successfully applied to measure Cr(III) Ion in raticide, toothpaste, and depilatory powder samples.

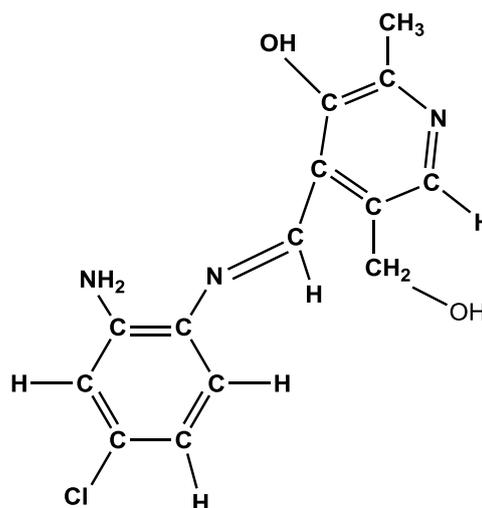


FIGURE1 Chemical structure of new synthetic ligand of 4- ((E)- (2-amino-4-chlorophenylimino) methyl) -5- (hydroxymethyl) -2- methylpyridin -3 -ol (C₁₄H₁₄ClN₃O₂).

Experimental

Reagents and instrumentals

All materials were purchased with a high purity grade such as graphite powder with a particle size of 1–2 μm from Merck, Paraffin oil from Aldrich, and Graphene oxide nanoplates

($\geq 99\%$ purity, 3.4-7 nm thickness, and 6-10 Layers) from US Research Nanomaterials, Inc(USA). Other inorganic compounds and high purity solvents were obtained from Aldrich or Merck companies, respectively. Schiff base 4-((E)-(2-amino-4-chlorophenylimino) methyl)-5-(hydroxymethyl)-2-methylpyridin-3-ol were sensitized according Ref.[47]. The stock solution of Cr(III) and others cations such as Na(I), Pb(II), Cu(II), Cd(II), Ni(II) in a concentration of 10^{-1} M were prepared and diluted from 10^{-2} to 10^{-10} M by serial dilution. All aqueous solutions of Cr(III) were prepared using double distilled water.

The measurements of the analyte were carried out by Metrohm 691 pH/potentiometer along with a saturated calomel electrode (SCE) as an external reference electrode and the prepared CPEs as an indicator electrode. ICP-OES (Spectro Arcos, model 76004555, Germany) was used to determine the Cr(III) ion in the real samples. All computations were accomplished on a computer with 8 GB memory and an Intel Pentium 73.07 GHz CPU.

Preparation of new sensor and analytical process

First, a certain amount of graphite powder, paraffin oil, graphene oxide nanoplates, and ionophore were poured into a mortar and stirred for 5 min to form a perfectly uniform mixture. A metal tube (with an inner diameter of 5.0 mm and a height of 3.0 cm) was chosen as a body of indicator electrode and filled with the obtained mixture (paste) for the preparation of Cr (III) ion carbon paste electrode. A copper wire was inserted through the other end of the tube into the carbon paste for electrical connection. A new surface was produced by scraping out the old surface and replaced by a new carbon paste surface. The condition-stage of the electrode was acquired for 24 h by immersing in a 1.0×10^{-2} M of a standard solution of Cr(III) ion.

In the present work, all experiments were accomplished at a stable temperature of 25 ± 1 °C. The used cell is as follows:

CPE of Cr(III) ion /test solution of As(III)/ SCE

Results and discussion

To investigate the ability of the electrode to determine different cations, the CPE with a specific percentage of compositions, including graphite powder, paraffin oil, graphene oxide nanoplates, and ionophore was fabricated and applied to determine various metal ions. The results indicating the sensor have a suitable response for determination of Cr(III) ion in comparison with other metal ions. This may be related to the presence of functional groups containing nitrogen and oxygen on the ligand with an appropriate interaction with Cr(III) ion.

Optimization of CPE composition

Nowadays, the use of the experimental design method to optimize the effective factors in a phenomenon is very much considered because it significantly reduces the number of experiments. It can also well evaluate the effects of factor interactions and their impact on response. Creating a random design can reduce the effects of uncontrolled variables as well [48]. To optimize the compositions of CPEs for determination of Cr(III) ion, an experimental design based on a matrix design was created, including fifteen runs. The selected level of each factor and the experimental runs and the obtained response presented in Tables 1 and 2, respectively. Each run was performed three times in the same conditions and slope (response) was calculated from the mean of the results.

TABLE 1 introduction of the factors and their levels

Factors	Symbol	Sign/unit	Lowest level (-)	Highest level (+)
Ligand	A	A/ %w/w	0	20
Graphene oxide	B	B/ %w/w	0	20
Paraffin oil	C	C/ %w/w	15	25
Graphite powder	D	D/ %w/w	50	72.5

Here, the response is the slope of potential diagram versus various concentrations of Cr(III) ion in which the ideal response is the Nernstian slope. Therefore, the slopes of the curve obtained for the percentage of each component in runs of 5 and 13 were closer to the Nernstian slope, indicating these

compositions are proper to prepare the Cr(III) ion CPE. However, the obtained data of the experimental runs were fitted with a quadratic model to evaluate more precisely and obtain the equation of correlation between the

TABLE 2 Experimental matrix and responses

Std Order	Run Order	A	B	C	D	Slope
7	1	10	0	20	70	10
6	2	20	10	20	50	17.98
9	3	10	10	15	65	14.99
12	4	10	10	20	60	15.37
8	5	10	20	20	50	19.1
4	6	5	5	17.5	72.5	9.49
15	7	10	10	20	60	15.5
5	8	0	10	20	70	8
3	9	5	15	22.5	57.5	10.42
11	10	10	10	20	60	15.6
10	11	10	10	25	55	13.2
13	12	10	10	20	60	15.7
1	13	15	15	17.5	52.5	20.98
2	14	15	5	22.5	57.5	15.65
14	15	10	10	20	60	15.61

Percentages of the electrode compositions with the response. The polynomial equation was determined and shown as Eq.1.

$$\text{Slope} = +15.53 + 2.50 * A + 2.27 * B - 0.45 * C + 0.65 * A * B + 0.71 * A * C - 1.68 * B * C - 0.65 * A^2 - 0.26 * B^2 - 0.37 * C^2 \quad \text{Eq. 1}$$

TABLE 3 The optimum percentage of each component in Cr(III) CPE

Factors	A	B	C	D	Predicted slope
Optimum compositions (%)	15	15	20	50	20.05

The analysis of variance was applied to evaluate the results of the experiments at a 95% confidence level. Obviously, a factor or their interactions have a significant effect on the slope obtained when the P-value of factor or their interactions were less than α -level (0.05) in the ANOVA Table. The results

The optimum percentage of each component in Cr(III) CPE predicted with the polynomial equation is presented in Table 3.

indicated that the proposed model with this design, all factors, and their interaction were significant variables with a positive or negative effect on the slope (Table 4). The significance of the model and no significance of the lack of fit (LOF) indicate that the data fit the model well, and the obtained equation can

well describe the results. Also, high R- square (0.9993) and adjusted R- square (0.9980) obtained with this model can confirm that the proposed model is significant, and the data fit the model well. Besides, the model can well predict future results because the predicted R- square of the model (0.9365) is high.

Nowadays, there is a high tendency to use carbon-based nanomaterials in many fields due to its unique features such as high surface to volume ratio, thermal and chemical stability, low price and simple method preparation[49-53]. Usage of carbon-based nanomaterials in

TABLE 4 ANOVA Table for obtained experimental data in present work

Source	Sum of Squares	df	Mean Square	F Value	p-value	Significant
Model	188.26	9	20.92	759.7	< 0.0001	+
A-ligand	49.8	1	49.8	1808.67	< 0.0001	+
B-nano	41.41	1	41.41	1503.77	< 0.0001	+
C-paraffin	1.6	1	1.6	58.18	0.0006	+
AB	1.14	1	1.14	41.23	0.0014	+
AC	1.34	1	1.34	48.82	0.0009	+
BC	7.57	1	7.57	274.98	< 0.0001	+
A ²	9.88	1	9.88	358.9	< 0.0001	+
B ²	1.58	1	1.58	57.44	0.0006	+
C ²	3.27	1	3.27	118.68	0.0001	+
Residual	0.14	5	0.028			
Lack of Fit	0.074	1	0.074	4.7	0.0961	-
Pure Error	0.063	4	0.016			
Cor Total	188.4	14				

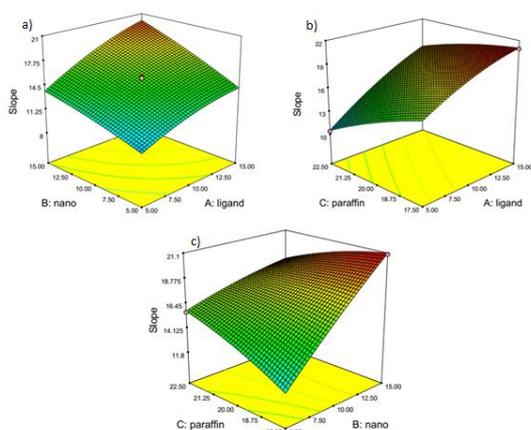


FIGURE 2 Effect of interactions between the factors on the sensor response

The electrode composition can increase the response of the electrode through increasing the contact surface [54-60]. However, the electrode response was reduced for a further increase of the material due to the saturation of the electrode surface. Therefore, the electrode response for the determination of Cr(III) was increased with increasing

graphene oxide up to 15% and reduced afterwards.

Ionophore or ligand is an essential ingredient in the ion-selective electrode composition because the functional group on the ligand can interact with the analyte and enhance the selectivity of the sensor toward the analyte. The effects of the ligand percentage change on the electrode response showed that the slope was increased by increasing the ligand percentage in the electrode compositions to 15% and reduced for further increasing. The factor of selectivity relates to this material. Active sites on the structure of ligand are reduced by increasing excess amounts of ligand, so the response of the electrode is decreased.

The effects of interactions between the factors are shown in Figure 2. As seen in Figure 2, the electrode response increases with the simultaneous increase of the percentage of two compositions in the electrode and then decreases. These changes

in the graph (Figure 2a) are more severe than the other two graphs because of the slope of this curve is highest, indicating the effect of the interaction between two factors (graphene oxide and ligand percentages) on the electrode response is more significant than other interactions.

Effect of pH

pH is the effective factor in efficiency of ion selective electrode in various media. For this purpose, pH range was studied from 1 to 12 by optimal sensor. The solution of HNO₃ or NaOH 1.0 M was applied for adjusting the pH of cation solution. In pH range of 3.5 - 9.0 (Figure 3), potential is independent of pH.

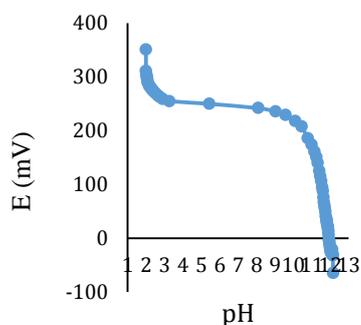


FIGURE 3 pH effect on the sensor potential for the determination of Cr(III) ion

In low pH, potentials increased which is because the possibility of attracting them at the electrode surface, the electrode surface was response to hydronium ions. In high pH, the possibility be attributed to the formation of some Cr (III) hydroxyl in the solution and decreased Cr (III) ions concentration and electrode response. Thus, these sensors can utilize in the pH ranges of 3.5 - 9.0 for the measurement of Cr(III).

Response time effect

Time of response is a significant and substantial factor with a high effect in the practical application of sensors, especially when there are too many samples to measure the analyte. The average time of response was

defined as the required time for electrodes reaches 90% of their final potential after settling in a sample solution [61-63]. For this sensor, the time of response was investigated in various concentrations of Cr(III) solutions in a range of 1×10^{-10} to 1×10^{-2} mol L⁻¹. In the results eventuate that the response time of the proposed sensor is about 10 s for determination of Cr(III) ion.

Selectivity

The potentiometric selectivity coefficients, defined as the capability of the discrimination of CPE for an interfering ion, X, concerning the analyte. A Matched potential (MPM) method was used in the study to investigate the sensor selectivity. This method was carried out in two-stage. In the first stage, the determined concentration of the primary ion is added to a reference solution of the analyte, and the potential of the proposed sensor is determined. In the second stage, interfering ions(X) are added to the reference solution of the analyte step by step until the potential changes of the proposed sensor were comparable to the previous solution. In the study, an Cr(III) ion concentration of 1.0×10^{-5} M was selected as the reference solution of the analyte and $K_{MPM} Cr, X$ was calculated to evaluate the sensor selectivity by the following equation :

$$K_{MPM} Cr(III), X = \frac{a_{Cr(III)}}{a_X}$$

Where $\alpha_{Cr(III)}$ and α_x were the activity of Cr(III) ion and interfering ion, respectively. According to the results presented in Table 5, none of the ions added to the standard analyte solution interfered with the analyte measurement in the concentration range used. Therefore, the proposed sensor displayed a high sensitivity toward Cr(III) ion in present other ions in the sample solution.

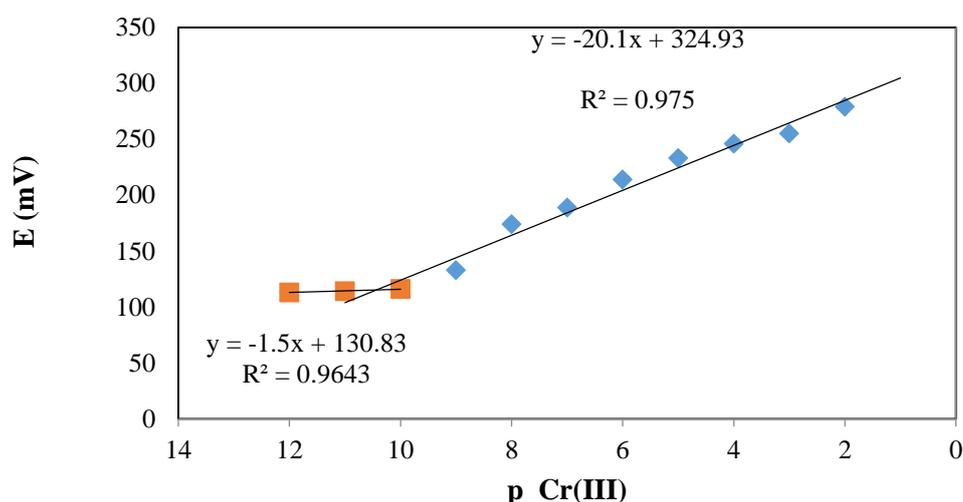
TABLE 5 Potentiometric selectivity coefficients for the prepared sensor

Interfering ion	K_{MPM}
Zn ²⁺	4.9×10^{-4}
Pb ²⁺	2.3×10^{-2}
Cd ²⁺	2.3×10^{-2}
Mg ²⁺	1.2×10^{-2}
Ni ²⁺	2.3×10^{-2}
K ⁺	6.9×10^{-3}
Al ³⁺	4.8×10^{-3}
Fe ³⁺	8.0×10^{-2}
Fe ²⁺	8.1×10^{-2}

Figure of merits

The response of CPE was experimented in a concentration range of 1×10^{-12} - 1×10^{-1} M for the determination of Cr(III) ion to draw the

calibration curve. According to Fig 4, the sensor response is linear in the concentration range of 1.0×10^{-10} to 1.0×10^{-2} M with an R-square of 0.9759 and a slope is 20.1 mVdecade⁻¹. Detection limit is the minimum concentration of analyte that sensor can respond to it and calculated from the ratio of three times the standard deviation of the blank solution to the calibration curve slope obtained for the analyte determination ($3S_b/m$). For this sensor, the detection limit was acquired 3.71×10^{-11} M. Relative standard deviation for the determination of Cr(III) ion was obtained from the measurement of the standard solution of Cr(III) ion (1.0×10^{-6} M) for five times in the same conditions and was 5.6%.

**FIGURE 4** Calibration curve for the determination of Cr(III) with the proposed sensor**Analytical application***Real sample analysis*

To evaluate the capability of the proposed sensor for determination of Cr(III) ion, two real samples, wastewater of chromium electroplating industries and river water (Torgabeh river in Mashhad) were applied. The results were compared with the data obtained from ICP-OES (Table 6). All real samples were analyzed without any sample preparation method with standard addition

method which is comparable to the value of ICP method. The recovery method considered with the spiked of standards of solution Cr(III). As it can be derived from Table 6, there was a satisfactory agreement between the results of the developed sensor and those obtained by ICP-OES.

Conclusion

In this study, a potentiometric sensor was developed to determine Cr(III) ion in two real samples. The sensor compositions, including graphite powder, paraffin oil, graphene oxide,

and ionophore was optimized with a chemometric approach based on a mixture design to reduce the experimental runs and determine the effect of interactions between factors. A synesthetic Schiff base as an ionophore in the sensor composition was selected and applied due to suitable interaction with Cr(III) ion. Other effective factors in the determination of Cr(III) with the proposed sensor, such as the response time of sensor,

pH and sensor selectivity, were studied with one factor at the time procedure. Finally, the prepared sensor was successfully applied to determine Cr(III) ion in the waste water and river water samples with a good recovery.

The sensor has a wide linear range (1×10^{-10} – 1×10^{-2} M), suitable slope (20.1 ± 0.3), low LOD (3.71×10^{-11} M) with relative standard deviation 5.6%.

TABLE 6 The obtained results for real samples analysis by the prepared sensor (n=5)

Sample	Added (ppm)	CPE sensor		ICP	
		Founded (ppm)	Recovery%	Founded (ppm)	Recovery%
Waste water	0	0.75	---	0.79	---
	1	1.73 ± 0.3	99.0	1.80 ± 0.1	100.1
	2	2.74 ± 0.2	99.6	2.81 ± 0.2	100.7
River water	0	ND	---	ND	---
	0.9	0.89 ± 0.4	98.9	0.91 ± 0.3	101.1
	1.5	1.4 ± 0.5	93.3	1.54 ± 0.1	102.6

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Orcid:

Esmat Shojaei: <http://orcid.org/0000-0003-3345-3674>

Mahboubeh Masrournia:

<https://orcid.org/0000-0003-2895-8096>

Ali Beyramabadi: <http://orcid.org/0000-0001-9616-8550>

Hossein Behmadi: <http://orcid.org/0000-0002-9970-9831>

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