

# **Chemical Methodologies**

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# Original Research Article

# Design and Construction of a Novel and an Efficient Potentiometric Sensor for Determination of Sodium Ion in Urban Water Samples

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#### ARTICLE INFO

#### **Article history**

Submitted: 2022-04-23 Revised: 2022-07-15 Accepted: 2022-08-15

Manuscript ID: CHEMM-2206-1567 Checked for Plagiarism: **Yes** 

Language Editor:

Dr. Ermia Aghaie

Editor who approved publication:

Dr. Zeinab Arzehgar

# **DOI**:10.22034/CHEMM.2022.348712.1567

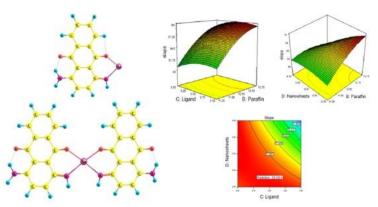
# KEYWORDS

Sodium-ion Modified sensor Graphene oxide Ionic Liquid 1, 4 Diaminoanthraquinone Experimental design

#### ABSTRACT

Sodium ions are one of the essential cations for various activities in the human body to control fluid levels, blood pressure, and nerve and muscle functions. Sodium-ion is a highly soluble chemical that small amounts of it can be absorbed by the body through water intake. However, consuming an excess amount of sodium ions can cause problems in the body. Therefore, it is vital to measure sodium ions in water samples. A novel and cheap potentiometric sensor was developed to measure trace amounts of sodium ions in real water samples. For the purpose, four effective components in the Nernstian response of the sensor, including 1-Hexyl-3-methyl imidazolium hexafluorophosphate as an ionic liquid, 1, 4 Diaminoanthraquinone (DAQ) as an ionophore, graphene oxide nanosheets, and paraffin oil as a binder were optimized using a response surface methodology (RSM) based on central composite design. The optimum percentages of paraffin, ionic liquid, ionophore, and graphene oxide to prepare the sensor were 13.34, 11.40, 3.21, and 2.16 %, respectively. Under the best percentage of electrode components, the potentiometric sensor showed a suitable slope of 59.2 mV decade-1 over a wide Na<sup>+</sup> concentration range (10<sup>-6</sup>-10<sup>-2</sup> mol L<sup>-1</sup>) with a proper detection limit of 8.97×10<sup>-7</sup> mol L<sup>-1</sup>. The sensor can be applied to measure sodium ions in a pH range of 4 to 8. The optimized geometry of the complex formed between sodium ion and the ionophore was investigated using the density functional theory (DFT) methods.

#### GRAPHICAL ABSTRACT



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#### Introduction

Carbon paste electrodes (CPEs) have been extensively utilized in electroanalysis because of possessing unique properties such as low cost, wide potential window, and proper sense diversity [1]. Other advantages of CPE to determine an analyte include simple and fast fabrication, rapid response, low residual current, renewable surface, simple maintenance, and suitable selectivity [2, 3]. The performance of CPE<sub>s</sub> for an analyte determination is contingent upon the properties of the modifier materials present in its components [4-9]. One type of new working electrode is the ionic liquid-carbon paste electrode (IL-CPEs), which is fabricated using an ionic liquid as the modifier and binder in the traditional CPE<sub>s</sub> [10, 11]. The unique properties of ionic liquids such as low vapor pressure, high ionic conductivity, negligible toxicity, wide electrochemical windows, and high thermal stability have resulted, leading to an intrinsic electrocatalytic ability and wide electrochemical windows in IL-CPEs [12-15].

Sodium-ion is known as one of the main extracellular fluid cations [16]. These ions are involved in the regulation of regulate the acidbase balance in the kidneys by replacing hydrogen ions with sodium ions in the renal tubule [17, 18]. Besides, the sodium ion is a vital electrolyte in the body, and its balance dramatically impacts human health. Obviously. Electrolyte plays an essential role in most metabolic processes. Increasing levels of this electrolyte in the body can cause excess fluid, tissue swelling, and reduced urine output [19]. Age, stress, sodium intake, and water mineralization can contribute to the development of primary hypertension. Among the factors mentioned, sodium intake is a significant parameter with a high effect on primary hypertension [20]. In the diet, a small amount of sodium can be absorbed into the body through water intake [21, 22]. Thus, determining Na+ ions in real water samples is critical for evaluating their effects on the human body.

Meanwhile, graphene oxide (GO) is a twodimensional nanosheet synthesized from the oxidation of graphite powder using a chemical procedure based on the Hummers' method [23]. GO sheets are used as a modifier in the preparation of electrochemical sensors due to their interesting physicochemical properties such as high thermal and chemical stability, large surface area, and good electron mobility [24-26]. The response surface is a plot in two or three dimensions, describing the relationship between the response and one or more factor levels. Response surface methodology (RSM) describes the response surfaces of experimental design with information about systems. RSM is a collection of mathematical methods that investigates investigating the relationship between one or several response variables and several independent variables [27, 28]. Design of Experiments (DOE) is a branch of statistics that investigates the effect of changes in input variables (Xs) on the outputs response variable (Y). Another traditional method to study these effects is one factor at a time (OFAT), in which the effect of one factor on the response is investigated while. In contrast, the other factors are fixed in certain values. Disadvantages of the strategy are the a large number of experiments and the absence of the possibility of simultaneous examination of variables and their interactions [29]. Nevertheless, in the DOE method, the effect of changing all the factors is simultaneously studied in the response, which leads to a reduction in the number of experiments and the study of the effects of the interaction between the factors [30]. In the present study, a new selective and sensitive developed CPE was using 1-Hexyl-3methylimidazolium hexafluorophosphate and graphene oxide nanosheets to determine the trace amounts of sodium ions in real water samples. Also, the percentage of the CPE components, 1-Hexyl-3methyl imidazolium including hexafluorophosphate as an ionic liquid, 1, 4 Diaminoanthraquinone (DAQ) as an ionophore, graphene oxide nanosheets, and paraffin oil as a binder were optimized using a response surface methodology (RSM) based on central composite design. Using a proper ionic liquid and a suitable ligand in the sensor construction leads to an

increase in the method's selectivity, a reduction in the response time, and an increase in the sensor's lifetime for measuring sodium ions. Besides, the application applying graphene of nanosheets as a sensor component can enhance the sensor quality with its convenient properties such as high thermal and chemical stability, large surface area, and good electron mobility. 1, 4 Diaminoanthraquinone is a low price compound used in cosmetics for hair dyeing. A simulation strategy using DFT was performed to check the possibility of interaction between sodium ions and ionophores before preparing the sensor. It exhibits a suitable ability as an ionophore with proper selectivity toward sodium ions in the sensor composite.

#### **Materials and Methods**

The stock solution of 0.1 mol L-1 of sodium ion was prepared by dissolving a calculated amount of sodium nitrate (0.8499 g) in 100 mL of deionized (DI) water. The working solutions were made up daily by diluting the stock solution of the sodium ion with DI water. DI water was obtained using a Milli-Q purification system (Milford, MA, USA) and was used to prepare all solutions. Graphene oxide nanosheets were purchased from the Research Institute of Petroleum Industry (Tehran, Iran). 1-hexyl-3-methyl Also, imidazolium hexafluorophosphate [C<sub>6</sub>MIM] [PF<sub>6</sub>] (Purity of 99 %) was purchased from Green Compounds Co. Iran. Further, 1, 4 Diaminoanthraquinone (DAQ) (Purity of ≥88.0%) was obtained from Sigma-Aldrich (USA). Finally, other required chemicals such as Graphite powder (Purity of 99.99 %) and nitrate salts of cations (Purity of ≥99 % were purchased from Merck (Darmstadt, Germany).

#### Fabrication of the electrode

Different percentages of graphite powder, paraffin oil, ionic liquid, ionophore, and graphene oxide nanosheets were utterly mixed in a mortar for 30 minutes to prepare the CPE. Once the material became utterly homogeneous, the obtained paste was carefully inserted into a polypropylene tube. A stainless-steel rod was inserted from the other end of the propylene tube to create an electrical

contact. The outer surface of the electrode was flattened by a special glossy paper. The prepared electrode was placed in the air for 24 hours. The CPE was placed in a solution of  $1 \times 10^{-3}$  mol L  $^{-1}$  sodium ions for 24 hours to replace the active regions of the ionophore in the electrode with sodium ions. Then, the electrode surface was washed with DI water. After several times of use, it was placed in  $10^{-3}$  mol L $^{-1}$  of ethylene diamine tetraacetic acid (EDTA) for 30 min.

### Apparatus and potentiometric measurements

All potentiometric measurements were performed using a pH meter 827 (Metrohm, Switzerland) at  $25 \pm 0.1$  °C. In this work, the potentiometric system consisted of two electrodes, including a saturated calomel reference electrode (Azar electrode, Iran) as the reference electrode and sodium modified carbon paste electrode (MCPE) as the indicator electrode as the following cell:

(Hg|Hg<sub>2</sub>Cl<sub>2</sub>, KCl (sat d.) || Na<sup>+</sup>ion sample solution | ion-selective electrode (CPE).

For this purpose, the solutions of sodium nitrate were prepared within the concentration range  $10^{-10} \cdot 10^{-1}$  mol L-1 at  $25 \pm 0.1$  °C. Subsequently, two of the electrodes, as mentioned above, were placed together in the solutions to measure the potential, where after stabilization  $\pm 1.0$  mV, the potential was recorded at the ambient temperature. Then, the calibration curve was plotted by the potential function versus the concentration logarithm of sodium ions.

#### Experimental design

Four components, including ionophore (1-Hexyl-3-methyl imidazolium hexafluorophosphate), ionic liquid (1-Hexyl-3-methyl imidazolium hexafluorophosphate), paraffin oil, graphene oxide nanosheets were selected to prepare the CPE sensor. Then, a random design was generated using a central composite design to optimize and examine the effect of the percentage of electrode components as independent variables at two levels in the response (The CPE slope). The design

includes 21 random tests with five repetitions at the central point (Tables 1 and 2).

**Table 1**: The levels of independent variables considered in the central composite design (CCD)

Code	Factors (w/w %)	-a	Low actual	Center	High actual	+a
A	Ionic Liquid	10	11.25	12.5	13.75	15
В	Paraffin	10	11.25	12.5	13.75	15
С	Ligand	2	2.5	3	3.5	4
D	nanosheets	1	2	3	4	5

<sup>&</sup>lt;sup>a</sup>Percents are in the electrode

Table 2: The central composite design (uncoded values) and the obtained experimental results

Run	A (w/w %)	B (w/w %)	C (w/w %)	D (w/w %)	Graphite	Slope	Linear range (log [Na+])
1	11.25	11.25	2.5	2	73	51.6	-3 to -8
2	13.75	13.75	3.5	2	67	55.8	-2 to -9
3	13.75	11.25	3.5	4	67.5	53.9	-4 to -8
4	12.5	15	3	3	66.5	56.1	-3 to -6
5	13.75	11.25	2.5	4	68.5	58.8	-1 to -5
6a	12.5	12.5	3	3	69	55.6	-1 to -7
7a	12.5	12.5	3	3	69	55.9	-1 to -7
8a	12.5	12.5	3	3	69	55.2	-1 to -7
9	15	12.5	3	3	66.5	53.8	-2 to -5
10	12.5	12.5	4	3	68	49.23	-1 to -6
11 <sup>a</sup>	12.5	12.5	3	3	69	55.6	-1 to -7
12	12.5	12.5	3	1	71	46	-1 to -4
13	11.25	11.25	3.5	2	72	50.3	-1 to -5
14 <sup>a</sup>	12.5	12.5	3	3	69	55.6	-1 to -7
15	12.5	10	3	3	71.5	43.1	-3 to -7
16	10	12.5	3	3	71.5	59.643	-2 to -5
17	11.25	13.75	2.5	4	68.5	57.8	-1 to -6
18	12.5	12.5	3	5	67	58.2	-1 to -6
19	13.75	13.75	2.5	2	68	45.4	-4 to -7
20	11.25	13.75	3.5	4	67.5	43.4	-2 to -5
21	12.5	12.5	2	3	70	54.5	-3 to -6

A: Amount of Ionic Liquid; B: Amount of Paraffin; C: Amount of Ligand; D: Amount of nanoparticles

# Computational details

Herein, all of the calculations were carried out using the B3LYP functional [31] and the 6-311+G(d,p) basis set as implemented in the Gaussian 03 program [32]. The optimized geometries did not show any imaginary frequencies. Thermal corrections were considered in the evaluation of the energies. All structures were visualized using the Chemcraft 1.7 program [33].

# **Results and Discussion**

CPE optimization and analysis of variance

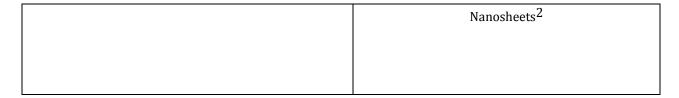
The obtained responses in Table 2 were evaluated with an analysis of variance (ANOVA) at a 95% confidence limit. The effects of variables and their interactions on the slope based on the ANOVA test are presented in Table 3. P-value is a suitable parameter in the ANOVA table to determine the significant factor in the response (Slope) [34]. According to the p-value, the proposed model is a

significant variable because its p-value is lower than 0.05 and the p-value lack of fit is higher than 0.05 at a 95% confidence limit. However, the lack of fit confirms the diversity of data around the fitted model. Lack of fit is a clear criterion for better data matching and model accuracy. If its p-value is greater than 0.05, then it is not significant

and the model fitted with the results well. Besides, the p-value of all factors and interactions except for the interaction between the percentage of paraffin and ligand (BC) was less than 0.05, indicating the significant effects of these factors and interactions on the obtained slope.

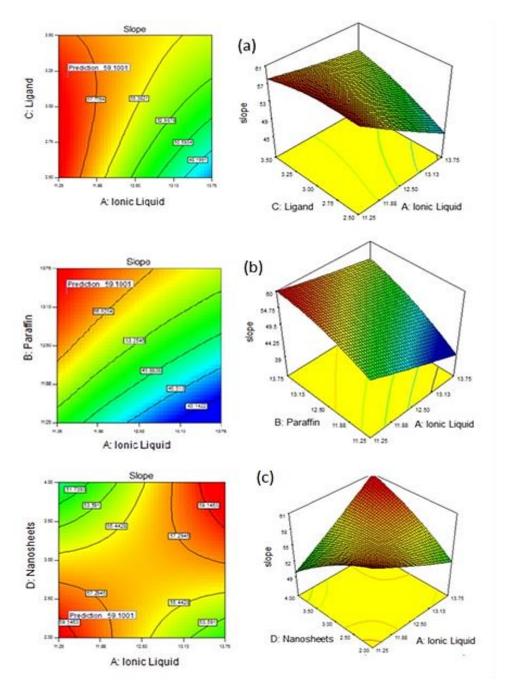
Table 3: Analysis of Variance (ANOVA)

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Source	Sum of Squares	Df	Mean Square	F Value	p-value	Type of effect	
Model	503.07	14	35.93	189.6	< 0.0001	Significant	
A-Ionic Liquid	17.07	1	17.07	90.07	< 0.0001	Significant	
B-Paraffin	84.5	1	84.5	445.86	< 0.0001	Significant	
C-Ligand	26.88	1	26.88	141.85	< 0.0001	Significant	
D-Nanosheets	74.42	1	74.42	392.68	< 0.0001	Significant	
AB	11.56	1	11.56	61	0.0002	Significant	
AC	56.18	1	56.18	296.43	< 0.0001	Significant	
AD	91.2	1	91.2	481.23	< 0.0001	Significant	
BC	0.61	1	0.61	3.19	0.1242	not significant	
BD	31.6	1	31.6	166.74	< 0.0001	Significant	
CD	100.82	1	100.82	531.98	< 0.0001	Significant	
A^2	1.16	1	1.16	6.1	0.0485	Significant	
B^2	61.54	1	61.54	324.69	< 0.0001	Significant	
C^2	25.08	1	25.08	132.32	< 0.0001	Significant	
D^2	22.22	1	22.22	117.22	< 0.0001	Significant	
Residual	1.14	6	0.19				
Lack of Fit	0.81	2	0.4	4.85	0.0852	not significant	
Pure Error	0.33	4	0.083				
Cor Total	504.21	20					
Std. Dev.					0.44		
	Mean				53.13		
	C.V.%				0.82		
	PRESS				103.94		
	R-Squared				0.9977		
	Adj R-Squared				0.9925		
	Pred R-Squared				0.7939		
				Slope =+55.64 -1.46* A +3.25* B -1.30 * C +3.05*			
				D +1.70* A * B +2.65* A * C +4.78 * A * D +0.28 * B *			
Final Equ	ation in Terms of Co	ded Fa	ctors:	C -2.81* B * D -3.55 * C * D +0.21* A <sup>2</sup> -1.57 * B <sup>2</sup> -			
				$1.00 * C^2 - 0.94 * D^2$			
				Slope = +197.32211 -42.38135 * Ionic Liquid			
				+19.47905* Paraffin -15.80263* Ligand			
Final Equation in Terms of Actual Factors:				+10.35247* Nanosheets +1.08800* Ionic Liquid *			
				Paraffin +4.24000* Ionic Liquid * Ligand +3.82000			
				* Ionic Liquid * Nanosheets +0.44000* Paraffin *			
				Ligand -2.24860* Paraffin * Nanosheets -7.10000*			
				Ligand * Nanosheets +0.13731* Ionic Liquid <sup>2</sup> -			
				1.00213* Paraffin <sup>2</sup> -3.99831* Ligand <sup>2</sup> -0.94083*			
L			1.00210 Furumin 0.77001 Diguna 0.74000				

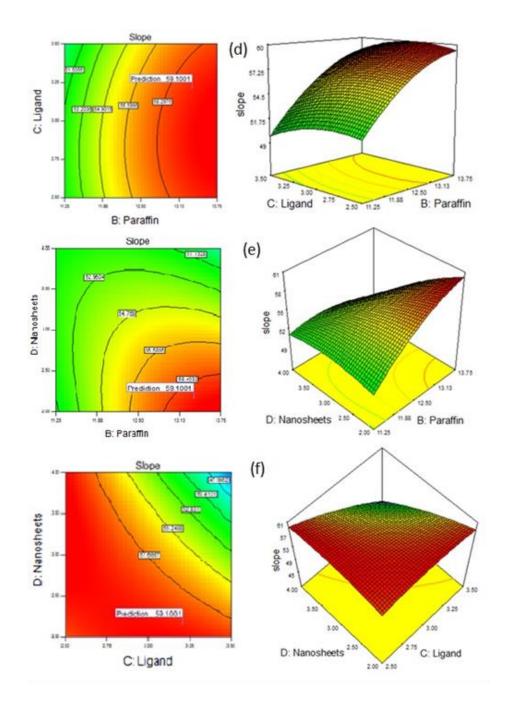


To study the effect of the interaction in the slope, the 3D graphical plots are presented in Figure 1, showing that the responses change dramatically with the simultaneous change of factors. Changes in the CPE slope is the greatest in Figure 1a, which confirms that the interaction between the ligand and the ionic liquid has the highest effect on the slope. However, the slope increases simultaneously with an increasing percentage of the ligand (C) and a decreasing percentage of the ionic liquid (A) (Figure 1a). Similarly, the slope

value improved with a low percentage of liquid ionic (A) and a high percentage of paraffin (B) (Figure 1b). As shown in Figure 1d, the interaction between paraffin (B) and ligand (C) is not significant, and both parameters with the maximum value show better CPE slopes [16]. Figure 1f demonstrates the interaction between graphene oxide nanosheets (D) and ligand (C). The slope value approaches the value of the Nernstian slope with increasing ligand percentage [35].



**Figure 1a-1c:** The response 3D surface plots for interaction effects of variables on the slope: a) AC (interaction of ligand and ionic liquid); b) AB (interaction of paraffin and ionic liquid); c) AD (interaction of nanoparticle and ionic liquid



**Figure 1d-1f:** d) BC (interaction of ligand and paraffin); e) BD (Interaction of paraffin and nanoparticle); f) CD (interaction of nanoparticle and ligand

Figure 1e illustrates the interaction between graphene oxide nanosheets (D) and paraffin (B). It indicates that the CPE slope approaches the Nernstian slope with decreasing graphene oxide and increasing paraffin percentage. The quality of the fitting results with the model is described by the coefficient of determination ( $R^2$ ) and the adjusted coefficient of determination ( $R^2$ <sub>adj</sub>).

The values of R<sup>2</sup> and adjusted R<sup>2</sup> were obtained as 0.9977and 0.9925, respectively (Table 3), showing the model fitted with the responses well. Figure 2 illustrates the diagram of the predicted value of slope versus its actual values. According to the Nernst equation, the target Nernstian slope response for the sodium ion is 59.1 mV decade<sup>-1</sup>. The optimum values with the best desirability for the Nernstian slope were obtained as percentage

of paraffin, ionic liquid, ionophore (ligand), and graphene oxide were 13.34, 11.40, 3.21, and 2.16 %, respectively (Table 4).

Also, the best corresponding electrode response was obtained as  $59.2 \pm 0.16$  mV decade<sup>-1</sup> under the optimized conditions (Figure 3). Also, linear

dynamic range (Figure 4), response time, and detection limit were obtained as  $1\times10^{-6}-1\times10^{-2}$  mol L-1, 20 Sec, and  $8.97\times10^{-7}$  mol L-1, respectively. The reproducibility of the electrode was investigated by the electrode.

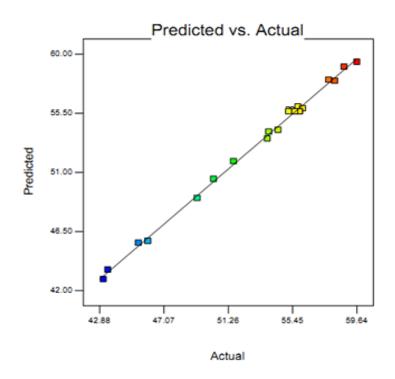
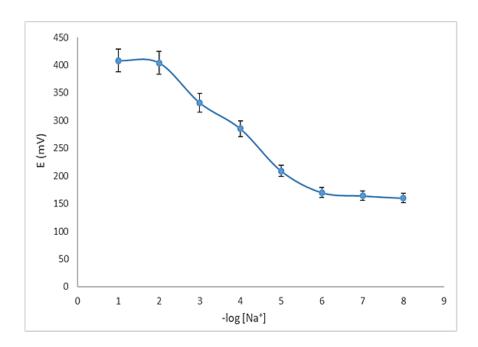


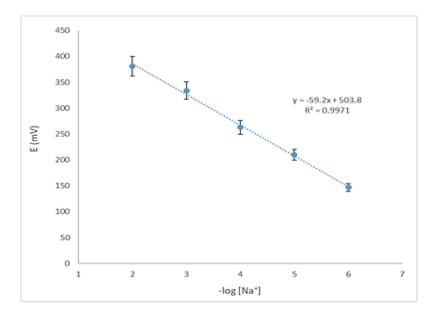
Figure 2: Relationship between predicted versus real values



**Figure 3:** Description of slope performance for the target value Na<sup>+</sup> cation carbon paste electrode under optimized condition

**Table 4:** The obtained optimum values based on experimental design (design expert software) for desired factors

Factors	Optimum amount
Ionic Liquid	11.40
Paraffin	13.34
Ligand (Ionophore)	3.21
Nanosheets	2.16
Graphite	69.9007
Slope	50.1001



**Figure 4:** Description of slope performance for the target value Na<sup>+</sup> cation carbon paste electrode under optimized conditions

#### Reproducibility of the fabricated electrode

Three similar fabricated electrodes under were optimization composite utilized investigate the reproducibility of the electrode. Then, the calibration curve was plotted for the standard solution of sodium ion within the range of  $10^{-6}$ - $10^{-2}$  mol L<sup>-1</sup> for the CPE electrode with the optimum percentage of components. Finally, the average slope of the calibration curve was obtained as  $59.23 \pm 0.16$  mV decade<sup>-1</sup> (Figure 5). The results confirm a Nernstian response of the prepared electrodes under the optimized conditions obtained from the response surface design strategy.

# Effect of pH on the electrode response

One of the critical factors in the potentiometric response of the modified electrodes is the pH measurement since there is a competition between the proton and the metal to bind to carrier receptors in the carbon paste structure. The effect of PH on the Nernstian response of a modified and optimized CPE was investigated by applying two concentrations of 10<sup>-2</sup> mol L<sup>-1</sup> and 10<sup>-</sup> <sup>3</sup> mol L<sup>-1</sup> sodium nitrate. The pH of the sample solution was changed within a range of 2 to 11 via hydrochloric acid and potassium hydroxide with a concentration of 0.1 mol L-1. The obtained results are shown in Figure 6, showing that the optimum electrode potential response was constant within the pH range of 4-8; as a result, OH- and H<sub>3</sub>O+ ions did not interfere. Therefore, the desired electrode can be applied within this constant pH range. Out of this range, there is a deviation of potential. It can be related to the competition of H<sup>+</sup> ions, the formation of sodium hydride at pH lower than 4, the instability of the CPE, and the formation of sodium hydroxide complex in the solution at pH higher than 8.

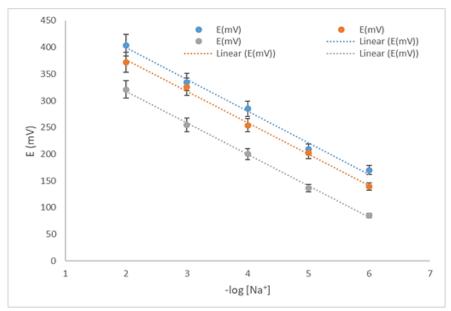
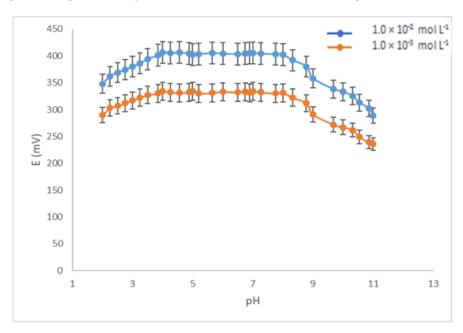


Figure 5: Reproducibility of three similar Na<sup>+</sup> ion CPEs under optimized conditions



**Figure 6:** Effect of pH on the potential response of the Na<sup>+</sup> cation CPE at 1×10<sup>-3</sup> mol·L<sup>-1</sup> and 1×10<sup>-2</sup> mol·L<sup>-1</sup> Na<sup>+</sup> ion under optimized conditions

# Electrode selectivity

One of the essential properties of an electrode is its selectivity behaviour which represents the ability to measure the target sample accurately. Selectivity is the efficiency of an electrode in determining the ion in the presence of other disturbing ions. In this study, the selectivity coefficient for the corresponding electrode was determined using the separate solution method (SSM) [36]. The effect of interfering ions on the response of the electrodes is determined by the selectivity coefficient (Kpot<sub>A, B</sub>). The selectivity coefficients of electrodes are investigated using

which different methods, each of has characteristic constraints. For measuring the selection coefficients, a concentration of  $1.0 \times 10^{-3}$ mol L<sup>-1</sup> of Na<sup>+</sup> ion and a concentration of  $1.0 \times 10^{-3}$ mol L-1 of interfering ions were considered. The results are shown in Table 5. The result indicates that other cations have a non-significant effect on performance of optimum CPEs determining sodium ions. Therefore, the optimum electrode has a high selectivity toward sodium ions.

# Dynamic response time and lifetime of fabricated electrode

The dynamic response time is the time required for the electrode to reach the equilibrium potential and stable potential response within the range of ±1 mV of the final potential. When the electrode is consecutively placed into the sample solution from the lower concentration to the higher concentration solution. The rate of electrode potential response stabilization is correlated with the time required to balance the

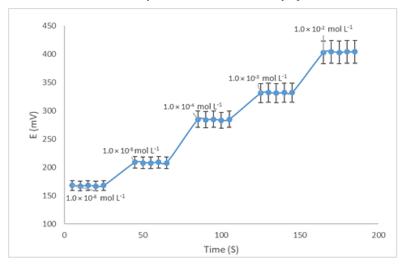
sodium ions in the sample solution and the sodium ions at specific locations in the ligand used in the electrode composition. In the present work, the response time of the corresponding electrode was tested against Na $^+$  ion with a concentration in the range of  $1\times10^{-6}$ - $1\times10^{-2}$  mol L $^{-1}$ . Sodium solutions, each with a difference of ten times of concentration, were studied sequentially. The response time was obtained as 20 Sec (Figure 7). Also, the lifetime of the sensor was monitored for eight consecutive weeks. Until six consecutive weeks, no significant changes were observed in the Nernstian response of the electrode, and it can be used for analytical applications.

# Response time of the electrode

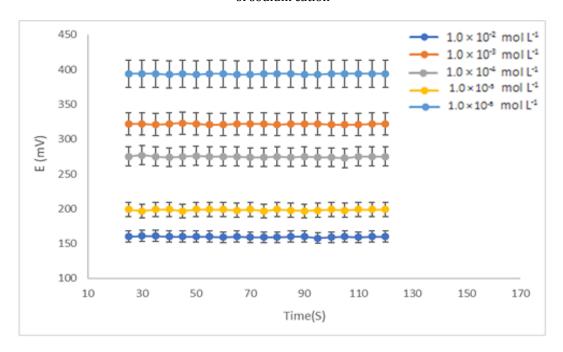
The response time is one of the essential parameters for the analytical application of CPEs. The present work investigated the time required to achieve a stable response for sodium solution at concentrations  $10^{-6}$ - $10^{-2}$  mol L<sup>-1</sup>. The results in Figure 8 indicate that the electrode response is fixed after 20 Sec.

Table 5: Potentiometric selectivity coefficient values of the Na+ cation CPE under optimized conditions

interference ions	K SSM	interference ions	K SSM
k+	5.35×10 <sup>-5</sup>	Li+	8.64×10 <sup>-2</sup>
NH <sub>4</sub> +	5.49×10 <sup>-3</sup>	Ba <sup>2+</sup>	2.98×10 <sup>-4</sup>
Sr <sup>2+</sup>	6.83×10 <sup>-4</sup>	Zn <sup>2+</sup>	3.76×10 <sup>-7</sup>
Mn <sup>2+</sup>	1.63×10 <sup>-5</sup>	Cd <sup>2+</sup>	4.60×10 <sup>-4</sup>
Cs <sup>1+</sup>	3.27×10 <sup>-6</sup>	Sn <sup>2+</sup>	1.91×10 <sup>-3</sup>
Co <sup>2+</sup>	5.20×10 <sup>-7</sup>	Fe <sup>3+</sup>	4.98×10 <sup>-5</sup>
Ni <sup>2+</sup>	2.46×10 <sup>-6</sup>	Cr <sup>3+</sup>	5.71×10 <sup>-5</sup>
AL <sup>3+</sup>	3.20×10 <sup>-3</sup>	Ca <sup>2+</sup>	$1.2 \times 10^{-6}$



**Figure 7:** Dynamic response time plot for the Na<sup>+</sup> cation CPE in optimized conditions for different concentrations of sodium cation



**Figure 8:** Static potential time plots for the Na<sup>+</sup> cation CPE in the optimized conditions from different concentrations of sodium cation

#### **Application**

Several urban water samples were tested to investigate the efficiency of the optimized carbon paste electrode. Urban water samples were obtained from different areas in Mashhad and its suburbs (Mashhad, Iran). All samples were analyzed using the prepared sensor without any sample preparation procedure. The concentration of Na<sup>+</sup> was determined using the calibration method. The results were compared with those

obtained by the atomic absorption spectroscopy method for more assurance. The working urban solutions were prepared by diluting the urban sample in distilled water. All experiments were carried out at 22±0.5 °C and repeated triplicate under the same conditions (Table 6). The results confirmed that the electrode could be successfully used to measure sodium ions in real water samples, which are not significantly different from the results obtained with AAS.

**Table 6:** Results of sodium analysis in urban water samples by the Na<sup>+</sup> cation CPE and reference method (n=3)

Sample	ISE <sup>a</sup> (mg L <sup>-1</sup> )	AAS <sup>a</sup> (mg L <sup>-1</sup> )	
urban water 1	2.35	2.40	
urban water 2	2.41	2.39	
urban water 3	3.20	3.28	

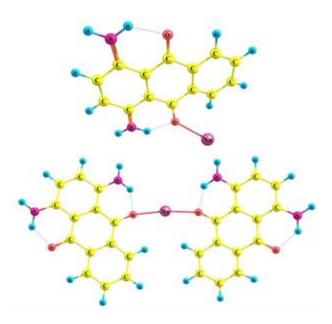
 $<sup>^{</sup>a}$  Results are average of triplicate measurements

#### DFT calculations

One or two neutral molecules of 1, 4 Diaminoanthraquinone (DAQ) can coordinate with the Na+ion, whose optimized geometries are shown in Figure 9. Meanwhile, the 1, 4 Diaminoanthraquinone (DAQ) can be deprotonated firstly to produce its anionic form. Geometries of the neutral molecule of the 1, 4 Diaminoanthraquinone (DAQ) and its anionic form are shown in Figure 10.

One or two anionic molecules of the deprotonated-1,4 Diaminoanthraquinone (DAQ)

can link to the Na<sup>+</sup> ion too. The optimized geometries for coordination of the anionic moieties to the Na<sup>+</sup> cation is shown in Figure 11. As seen, the anion is coordinated to the metal ion via the oxygen atom of the carbonyl group and nitrogen atom of the deprotonated amine group, as the deprotonated-1, 4 Diaminoanthraquinone (DAQ) acts as a bidentate ligand. This model has been proposed for the interaction of the deprotonated-1, 4 Diaminoanthraquinone (DAQ) molecule with several metal ions [37].



**Figure 9:** Optimized geometries for coordination of one or two neutral molecules of the 1, 4 Diaminoanthraquinone (DAQ) to the Na<sup>+</sup> cation

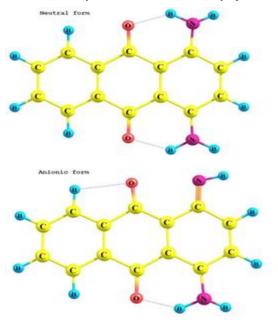
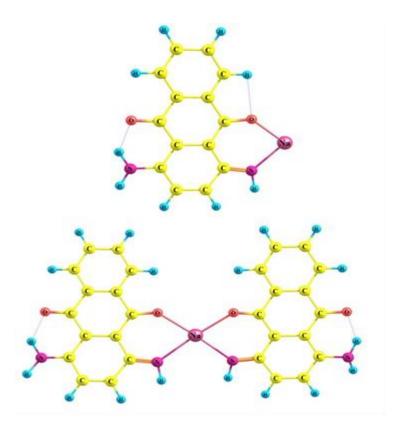


Figure 10: Optimized geometries of the neutral and anionic forms of the 1, 4 Diaminoanthraquinone (DAQ)

The difference in the Gibbs free energies between the products and reactants is known as  $\Delta G$ . The calculated  $\Delta Gs$  for the investigated-coordination reactions are reported in Table 7. Based on the calculated  $\Delta Gs$ , coordination of two deprotonated-1, 4 Diaminoanthraquinone (DAQ) (its anionic form) to the sodium cation is the most favorable interaction. This complexation involves the most negative value of the  $\Delta G$ . In the neutral form, the 1, Diaminoanthraquinone (DAQ) molecule coordinates weakly to the Na+ ion via the oxygen atom of the carbonyl group. However, in the anionic form, the investigated ligand is strongly coordinated to the Na+ cation via two donor atoms.

Coordination of the deprotonated-amine nitrogen and carbonyl oxygen to the Na+ ion results in chelation. The chelating effect causes a very negative  $\Delta G$ . The chelate formation increases the entropy of the reaction, thereby causing very negative  $\Delta G$ of the complex formation. Coordination of two deprotonated-1, Diaminoanthraquinone (DAQ) and formation of two chelate rings result in more negative  $\Delta G$  of the complex formation. Note that the formed complex has a square planar geometry. Two deprotonated nitrogens and two oxygen atoms of the carbonyl groups occupy four positions of the square structure.



**Figure 11:** Optimized geometries for coordination of one or two deprotonated 1, 4 Diaminoanthraquinone (DAQ) ligand to the Na<sup>+</sup> cation

Comparison the sensor with other prepared sensors

Several sensors were selected to evaluate the prepared sensor's ability to determine the sodium ion. The results are shown in Table 8. The important advantages of this modified sensor with

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the previously published paper for sodium-ion determination include the reduction of detection limit, the improvement of dynamic linear range, and the proximity of the slope to the theoretical Nernstian slope. Besides, the sensor has a wider linear range than the other prepared sensors.

**Table 7:** The  $\Delta G$  (kJ.mol<sup>-1</sup>) calculated for complexation of the 1, 4 Diaminoanthraquinone (DAQ) and Na<sup>+</sup> ion in four different positions

Complexation reaction	$\Delta G$ complexation
Na+ - one 1,4-diaminoanthraquinone neutral molecule	-174.65
Na+ - two 1,4-diaminoanthraquinone neutral molecule	-283.18
Na+ - one 1,4-diaminoanthraquinone anionic form	-658.10
Na+ - two 1,4-diaminoanthraquinone anionic form	-888.72

**Table 8:** Comparison of the sensor ability with other prepared sensors

No	Method	Slope mV decade <sup>-1</sup>	Linear range mol L <sup>-1</sup>	Detection limit mol L <sup>-1</sup>	Reference
1	POT-ISE <sub>S</sub>	59.2 ±0.16	10-6-10-2	8.97 ×10 <sup>-7</sup>	This work
2	POT-CCF	59.2 ± 0.6	10 <sup>-3</sup> -10 <sup>-1</sup>	-	[38]
3	POT-ISEs	56.2 ± 1.4	10 <sup>-4</sup> - 10 <sup>0</sup>	$4.0 \times 10^{-4}$	[39]
4	POT-ISE <sub>S</sub>	55.1 ± 0.7	10-4 - 10-1	-	[40]
5	POT(SS-ISE)	58.68	10 <sup>-6</sup> -10 <sup>-1</sup>	-	[41]
6	POT(SS-ISE)	58 ± 3	7.08×10 <sup>-7</sup> -1	$3.16 \times 10^{-6}$	[42]
7	Wireless- ISE	56.1	10-4-1	=	[43]
8	Wireless- ISE	63.75	10-4 - 10-1	-	[44]
9	Wireless- ISE	54.85 ±5.8	10-4-10-1	-	[45]
10	POT(SS-ISE)	52.4	10-4-1	-	[46]
11	EPADs	54.8±1.4	10-3-1	-	[47]

#### Conclusion

The present study developed an efficient and fast potentiometric sensor to determine sodium ions in real water samples. The experimental design approach was used to optimize the percentage of components for constructing a new carbon paste electrode instead of one factor at the time strategy. Usage of this procedure offers several important advantages, such as saving cost and time, reducing the number of experiments, getting an accurate response, and examining the effect of independent variables. The optimal conditions, maximum Nernstian slope of the calibration curve, and significant interactions between factors were obtained using the central composite design method based on RSM. The proposed carbon paste electrode was constructed with 1-Hexyl-3methyl imidazolium hexafluorophosphate as an ionic liquid, 1, 4 Diaminoanthraquinone (DAQ) as an ionophore, graphene oxide nanosheets, and paraffin oil as a binder and provided an attractive choice for the determination of Na+ cation using the potentiometric method. The optimum percentages of paraffin, ionic liquid, ionophore, and graphene oxide to prepare the sensor were 13.34, 11.40, 3.21, and 2.16 %, respectively. The sensor displays a stable response time, wide dynamic concentration range (10-6-10-2 mol L-1), good sensitivity, and low detection limit (8.97×10-7 mol L-1) to determine sodium ions. Therefore, the sensor is appropriate for measuring the concentration of Na+ cation in water samples without the effect of interfering with other cations and without the need for any sample preparation procedure such as extraction, derivatization, or filtration steps for real water analysis. This electrode has a fast response time (~20 Sec) over the whole concentration range within the pH range of 4-8 and excellent selectivity coefficients over a range of interfering cations. The electrode could also be used for six consecutive weeks without altering the response. The reproducibility of the sensor under optimized conditions was examined, and it was successfully used as an indicator electrode to measure trace amounts of sodium ions in real water samples.

#### Acknowledgements

The authors would like to thank the Islamic Azad University of Mashhad, Mashhad, Iran, for financial support.

# **Funding**

This research did not receive any specific grant from funding agencies in the public, commercial, or not-for-profit sectors.

#### **Authors' contributions**

All authors contributed to data analysis, drafting, and revising of the paper and agreed to be responsible for all the aspects of this work.

# **Conflict of Interest**

There are no conflicts of interest in this study.

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# **HOW TO CITE THIS ARTICLE**

Shiva Ariavand, Mahmoud Ebrahimi, Ebrahim Foladi. Design and Construction of a Novel and an Efficient Potentiometric Sensor for Determination of Sodium ion in Urban Water Samples. *Chem. Methodol.*, 2022, 6(11) 886-904 <a href="https://doi.org/10.22034/CHEMM.2022.348712.1567">https://doi.org/10.22034/CHEMM.2022.348712.1567</a>

URL: http://www.chemmethod.com/article 155298.html