Application of 1, 4-diaminoanthraquinone as a new selectophore material for construction of a potentiometric iron (III)-selective electrode

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A new Fe^{+3} selective electrode based on PVC membrane was prepared with a view to its application as an indicator electrode in the potentiometric cell. 1,4-Diaminoanthraquinone(DAQ) as a lipophilic selective element, sodium tetraphenylborate (NaTPB) as an anionic additive, nitrobenzene (NB) as a solvent mediator, and polyvinyl chloride (PVC) as a polymeric matrix were employed to form the membrane mixture. The best electrode response had a slope of 19.6 ± 0.2 mV per decade over a wide iron(III) concentration range (1.0×10^{-6} to 1.0×10^{-2} M) with a detection limit of 8.0×10^{-7} M. This electrode showed a fast response time (~12 s) over the whole concentration range in the pH range of 1.6-3.5. In order to study the selectivity of the electrode toward Fe^{+3} ions in presence of many common alkaline, alkaline earth, transition, lanthanide and heavy metal ions the matched potential method was applied. According to the obtained results the interference of all mentioned cations on the proposed iron(III) electrode was negligible. The sensor was successfully employed as an indicator electrode in the potentiometric titration of iron(III) with EDTA at pH 6.0. The applicability of the sensor was studied in determination of Fe^{3+} ions in mixtures with different ions.

Keywords: Sensor, Potentiometry, Ion-Selective Electrode, PVC Membrane

INTRODUCTION

Among different analytical methods, the potentiometric ion selective electrodes are mostly applied to determine the amount of various ions due to the wide linear range, low detection limit, good Nernstian slope, high sensitivity and selectivity, low cost, portability, easy maintenance, simplicity and speed of the usage, etc. [1-5].

Lately, a large number of PVC membrane electrodes for diverse ions such as iron were reported by us and other researchers [4-44].

Iron is a metal of the first transition series with atomic number 26. It is a mineral element which our body needs for numerous functions including complexation with oxygen in hemoglobin and myoglobin which are two prevalent proteins to transport oxygen in thebody. Moreover, the iron could be found in many other proteins and enzymes. The human body needs an appropriate amount of iron, otherwise the lack of iron causes iron-deficiency anemia. On the other hand, a high amount of iron in our body leads to iron poisoning [45].

In this paper the preparation of a new Fe^{+3} ion selective electrode was described, based on PVC membrane with DAQ as a selector element (Fig.1) with a special interaction between its heteroatoms such as nitrogen and iron(III) ion.
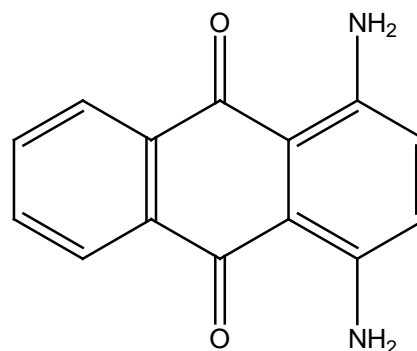

Fig. 1.1, 4-Diaminoanthraquinone (DAQ) structure

EXPERIMENTAL

Reagents

1, 4-Diaminoanthraquinone (DAQ) was purchased from Fluka. The nitrate and chloride salts of all used cations, reagent grade nitrobenzene (NB), acetophenone (AP), benzyl acetate (BA), dibutyl phthalate (DBP), sodium tetraphenyl borate (NaTPB), high molecular weight PVC,and tetrahydrofuran (THF) were purchasedfromMerck and Aldrich and used without modification or further treatments, except for the vacuum drying of the salts over P_2O_5. Doubly distilled deionized water was used in all experiments.

The EMF measurements

To potential measurement a Corning ion analyzer 250 pH/mV meter was used at 25.0 °C. The activities were calculated by the Debye–Hückel procedure [46]. The electrochemical cell was assembled to

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EMF measurements according to the following system:

Ag–AgCl | internal solution, 1.0×10^{-3} M Fe$(NO_3)_3$| PVC membrane | test solution | Hg–Hg$_2$Cl$_2$, KC1 (satd.)

PVC membrane electrode preparation

In order to prepare the membrane solution by the dipping method, specific amounts of PVC, ionophore (DAQ), plasticizer (NB), and anionic additive (NaTPB) were blended and dissolved in 3-5mL THF. AfterevaporatingTHF from the obtained solution an oily concentrated mixturewas formed. A pipet tip (3-5 mm o.d. on top) was dipped into the blend for about 5s in order to form a transparent membrane of about 0.3 mm thickness[47-55]. In the next stage the pipet tip was removed from the mixture and left at room temperature for about 12 h.At last the pipet tip was filled with 1.0×10^{-3} M Fe(NO$_3$)$_3$ solution as an internal filling solution and the pipet tip was soaked in the same solution for 24 has a pretreatment. A silver/silver chloride electrode was used as an internal reference electrode.

RESULTS AND DISCUSSION

Potential response of the electrode

In order to investigate the electrode response over a wide concentration range of various cationsnumerous polymeric liquid membrane sensors based on DAQ were fabricated. Due to the special structure of DAQ and its ability to form complexes with iron (III), the Fe^{+3} selective electrode showed the most sensitive response with a slope of 19.6 ± 0.2 mV per decade (Fig. 2).

Effect of membrane composition

As the type and nature of membrane composition have a substantial impact on the ion selective electrodes response, to obtain the best Nernstian response with steady, noiseless and repeatable potentials, the membrane composition was optimized by employing various proportions of additives, ionophore, plasticizer and PVC [56-61].
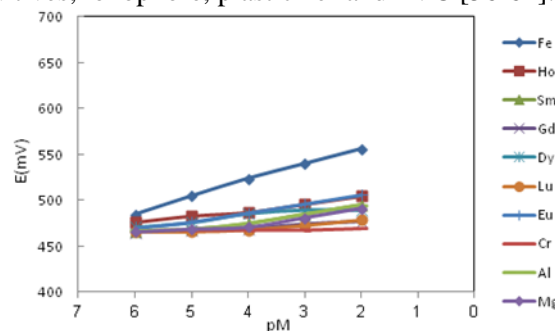

Fig. 2. Potential responses of various ion-selective electrodes based on DAQ.

According to the obtained results listed in Table 1, among four different plasticizers, NB showed the best sensitivity. Essentially the plasticizer acts as a solvent mediator to ameliorate the performance of the sensor via facilitating the movement of the ionophore in the polymeric membrane matrix. By changing the amount of NATPB the proper rate of ionic additives was achievedwhich could impress on the workability of the electrode by increasing the sensitivity of the membrane via reducing the ohmic resistance and speeding upthe process of ion extraction into the membrane [62-70]. Hence, the best Nernstian potential response was obtainedwith amembrane composition of (w/w):30% PVC, 5% DAQ, 2% NaTPB and 63% NB (electrode no.11).

Calibration curve

The potential response of the iron(III) selective electrode based on DAQ,which is demonstrated in terms of calibration curve (Fig. 3) shows the wide working range (1.0×10^{-6} to 1.0×10^{-2} M) fortheoptimizedDAQ-based iron (III) ion selective electrode. The slope of the linear part of the calibration curve is 19.6 ± 0.2 mV per decade with a detection limit of 8.0×10^{-7} M. According to the IUPAC recommendations, the detection limit is calculated bycrossing of two extrapolated segments of the calibration curve.

Table 1. Composition of membrane ingredients

| Electrode No.	Composition of Carbon Paste (wt. %)							Slope (mV/ decade)	Dynamic Linear Range (M)
	PVC	NB	DBP	AP	BA	NaTPB	DAQ		
1	30	66	-	-	-	2	2	16.9 ± 0.4	1.0×10^{-6}-1.0×10^{-2}
2	30	-	66	-	-	2	2	14.1 ± 0.4	1.0×10^{-5}-1.0×10^{-2}
3	30	-	-	66	-	2	2	15.7 ± 0.5	1.0×10^{-5}-1.0×10^{-2}
4	30	-	-	-	66	2	2	14.8 ± 0.3	1.0×10^{-5}-1.0×10^{-2}
5	30	68	-	-	-	0	2	12.8 ± 0.3	1.0×10^{-4}-1.0×10^{-2}
6	30	67	-	-	-	1	2	16.1 ± 0.3	1.0×10^{-6}-1.0×10^{-2}
7	30	65	-	-	-	3	2	15.5 ± 0.4	1.0×10^{-6}-1.0×10^{-2}
8	30	67	-	-	-	2	1	14.9 ± 0.3	1.0×10^{-6}-1.0×10^{-2}
9	30	65	-	-	-	2	3	17.6 ± 0.2	1.0×10^{-6}-1.0×10^{-2}
10	30	64	-	-	-	2	4	18.4 ± 0.3	1.0×10^{-6}-1.0×10^{-2}
11	30	63	-	-	-	2	5	19.6 ± 0.2	1.0×10^{-6}-1.0×10^{-2}
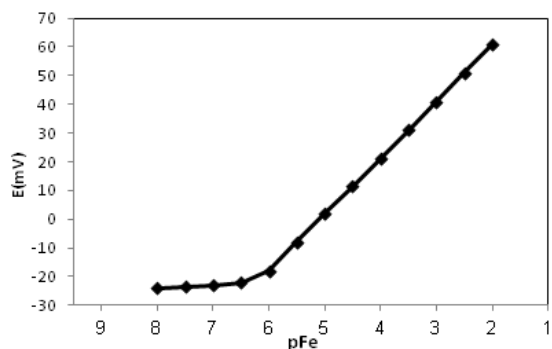
Fig. 3. Calibration curve of the DAQ-based Fe^{3+} sensor.

pH Effect

The profile of the designed electrode potential response versus the pH changes was investigated by employing 1.0×10^{-3}M Fe^{+3} solutionand adding very small drops of HNO_3or NaOH over the pH range 1.0-11.0. Pursuant to the obtained graph (Fig. 4) it is obvious that the working pH range of the electrode, where the potentials stay constant, is from 1.6 up to 3.5.
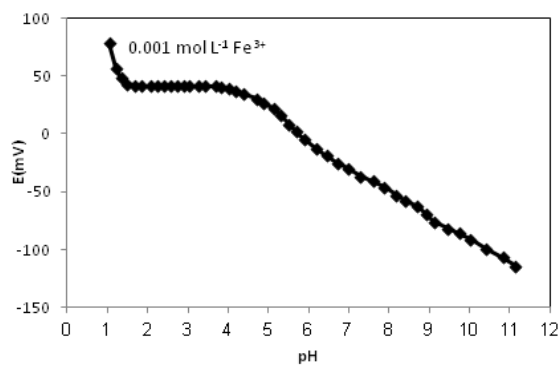

Fig. 4. The pH effect of the test solution on the potential response of the iron (III) sensor.

The potential drift at pH values lower than 1.6 could be attributed to the protonation of the heteroatoms of the ionophore, as a result of whichDAQ loses the ability of forming complexes with the iron ions. On the other hand, the potential drift at higher pH values could be due to the formation of some hydroxyl complexes of Fe^{3+} ions in solution, which results in reduced amount of free iron (III) ions in the solution [71].

Dynamic response time

One of the most important factors in analytical applications is the response time of the ion selective electrode,whichis defined as the average time neededto reach a stable potential value of the ion selective electrode after sequential submerging of electrodes in a series of primary ion solutions with ten-fold difference in concentration. In order to measure the dynamic response time of the Fe^{+3} ion selective electrode, various concentrations of Fe^{+3} over the range from 1.0×10^{-6} to 1.0×10^{-2} Mwere employed. According to the obtained results illustrated in figure 5, the average response time required for the iron(III) electrode is about 12 s.
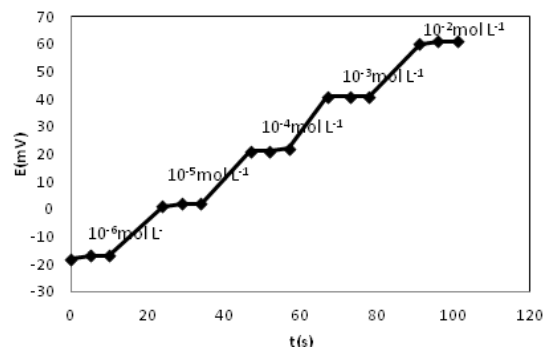

Fig. 5. Dynamic response time of Fe^{3+} sensor

Selectivity of the sensor

To assess the effect of interfering ions including lanthanides, alkali and alkaline earth elements, transition and heavy metals on the potential response of the proposed electrode, the selectivity coefficients were determined graphically by the match potential method (MPM). In order to determine the selectivity coefficients by MPM, the potentialofadding the specificactivity (concentration) ofa Fe^{+3}solution to areferencesolutionwas measured. On the other hand, in a separate experiment, a certain amount of various interfering ions was sequentially added to the same reference solution, as long as the obtained potential response matches the initial one. Eventually, by the ratio of primary ion (A) activity changes to the interfering ion (B) according to the following equation, the matched potential method selectivity coefficient (K_{MPM}) was determined for each cation [72]:

$$K_{ij}^{MPM} = \frac{\Delta a_A}{a_B}$$

Pursuant tothe resulting values,which are summarized in table 2, the interference of other cations on the proposed electrode was negligible. Thispoints to a very high performance of the designed electrode toward Fe^{+3} ions and special interaction between Fe^{+3} ions and DAQ.

Analytical applications

To verify the practical utility of the proposed electrode, it was applied as an indicator electrode in order to determine iron (III) in a potentiometric titration of 25 mL of 1.0×10^{-4} M Fe^{3+} solution with 1.0×10^{-2} M EDTAat pH=6adjusted by acetate buffer. As figure 6 shows, by increasing the amount

Table 2. Selectivity coefficients ($K_{Fe^{3+}}^{MPM}$) of the proposed Fe^{3+} sensor.

Interfering Ion	$K_{Fe^{3+},B}^{MPM}$	Interfering Ion	$K_{Fe^{3+},B}^{MPM}$
Ho^{3+}	8.0×10^{-4}	Dy^{3+}	1.0×10^{-3}
La^{3+}	8.0×10^{-4}	Ca^{2+}	2.0×10^{-3}
Tm^{3+}	1.5×10^{-3}	Co^{2+}	1.0×10^{-3}
Nd^{3+}	1.0×10^{-3}	K^{+}	1.0×10^{-3}
Eu^{3+}	8.5×10^{-4}	Pb^{2+}	8.0×10^{-4}
Pr^{3+}	8.5×10^{-4}	Ni^{2+}	1.0×10^{-3}
Yb^{3+}	2.0×10^{-3}	Mg^{2+}	8.0×10^{-4}
Lu^{3+}	1.0×10^{-3}	Cd^{2+}	1.0×10^{-3}
Er^{3+}	8.0×10^{-4}	Na^{+}	8.0×10^{-4}
Tb^{3+}	1.5×10^{-3}	Cr^{3+}	2.5×10^{-3}
Sm^{3+}	8.5×10^{-4}		

Table 3. Determination of Fe^{3+} in presence of a mixture of metal ions.

Fe^{3+}(M)	Added cations (M)	Found[a](M)	Recovery (%)
1.0×10^{-6}	$(0.001)Eu(NO_3)_3$ & $(0.001)Er(NO_3)_3$	1.03×10^{-6}	103
1.0×10^{-6}	$(0.001)Gd(NO_3)_3$ & (0.001) $Pr(NO_3)_3$	1.03×10^{-6}	103
1.0×10^{-6}	$(0.001)La(NO_3)_3$ & $(0.001)Ho(NO_3)_3$	1.03×10^{-6}	103
1.0×10^{-6}	$(0.001)Dy(NO_3)_3$ & $(0.001)Yb(NO_3)_3$	1.00×10^{-6}	100
1.0×10^{-6}	$(0.001)Tb(NO_3)_3$ & $(0.001)Nd(NO_3)_3$	1.03×10^{-6}	103
1.0×10^{-6}	$(0.001)Pb(NO_3)_2$ & $(0.001)Ni(NO_3)_2$	1.00×10^{-6}	100
1.0×10^{-6}	$(0.001)Cr(NO_3)_3$ & $(0.001)Cd(NO_3)_3$	1.03×10^{-6}	103
1.0×10^{-6}	$(0.001)KNO_3$ & $(0.001)Mg(NO_3)_2$	1.00×10^{-6}	100
1.0×10^{-6}	$(0.001)NaNO_3$ & $(0.001)Ca(NO_3)_2$	1.03×10^{-6}	103
1.0×10^{-6}	$(0.001)Pb(NO_3)_2$ & $(0.001)Ca(NO_3)_2$ & $(0.001)KNO_3$	1.00×10^{-6}	100
1.0×10^{-6}	$(0.001)Cr(NO_3)_3$ & $(0.001)Na(NO_3)$ & $(0.001)Ca(NO_3)_2$	1.03×10^{-6}	103

[a]Results are based on three measurements

of EDTA, a reduction in the potential values is seen, which is attributed to a decrease in free iron(III) concentration due to complexation with EDTA. As a result, the sharp end point in the titration curve reflects the fact that this electrode could successfully play the role of an indicator electrode. Furthermore, the designed Fe^{+3} electrode was employed to determine Fe^{+3} ions in presence of binary mixtures containing various cations, the result of which, listed in table 3, demonstrates the acceptable recovery for Fe^{+3} ion in all mixtures.
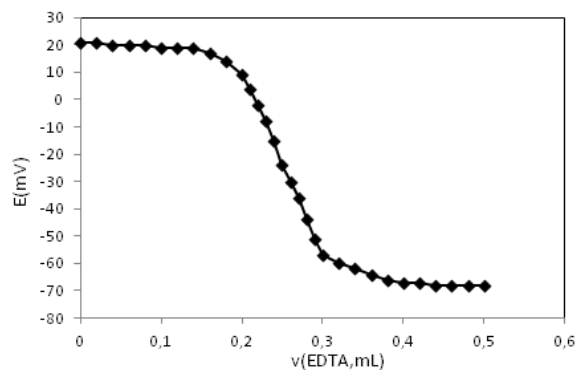

Fig. 6. Potential titration curves of 25 mL of 1.0×10^{-4} M Fe^{3+} solution with 1.0×10^{-2} M of EDTA.

CONCLUSION

In the present work, a new iron-selective PVC membrane electrode based on DAQ as a carrier ion and NB as a solvent mediator was developed. The best membrane composition (5% DAQ, 2% NaTPB, 30% PVC, 63% NB) showed a slope of 19.6 ± 0.2 mV decade^{-1} in the linear range of 1.0×10^{-6} -1.0×10^{-2} M and a limit of detection of 8.0×10^{-7} M. This electrode is independent of pH changes in the range of 1.6-3.5. It was successfully employed in titration and direct potentiometric methods.

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ПРИЛОЖЕНИЕ НА 1, 4-ДИАМИНОАНТРАХИНОН КАТО НОВ СЕЛЕКТОФОРЕН МАТЕРИАЛ ЗА ПРОИЗВОДСТВОТО НА ПОТЕНЦИОМЕТРИЧЕН Fe(III)-СЕЛЕКТИВЕН ЕЛЕКТРОД

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(Резюме)

Приготвен е нов, йон-селективен електрод за определянето на Fe^{+3} , базиран на PVC-мембрана. Индикаторният електрод е предназначен за потенциометрични определяния. Използват се 1,4-диаминоантрахинон (DAQ) като липолитичен селективен агент, натриев тетрафенилборат (NaTPB) като анионна добавка, нитробензен (NB) като разтворител-медиатор и поливинилхлорид (PVC) като полимерна матрица. Най-добър отговор на електрода се при наклон 19.6 ± 0.2 mV за декада за широк интервал на концентрации на желязо (III) от 1.0×10^{-6} до 1.0×10^{-2} M при чувствителност от 8.0×10^{-7} M. Този електрод показва бърз отговор (~12 s) за широк интервал от концентрации на Fe^{+3} (1.0×10^{-6} M до 1.0×10^{-2} M) за pH в обхвата 1.6-3.5. Селективността на електрода спрямо Fe^{+3} е изследвана в присъствие на много метални йони (алкални, алкалоземни, тежки метали, преходни елементи и лантаниди). Според получените резултати смущенията от страна на изследваните метали са пренебрежими. Така направеният сензор е използван успешно при потенциометричното титруване на разтвори на желязо (III) с EDTA при pH 6.0. Приложимостта на сензора е потвърдена и в присъствие на смеси от различни йони.