Al(III) Coated Wire Selective Electrode Based on 5-Bromo(salicylidene-2-aminothiophenol) Schiff Base as a New Ionophore

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A new Al(III) selective membrane based on 5-bromo(salicylidene-2-amino thiophenol) as a neutral carrier in a poly(vinyl chloride) (PVC) matrix is coated on platinum wire to determine trace amounts of Al(III) ion in various samples. The sensor exhibits a good response for Al(III) ion over a wide concentration range of 5.20×10^{-7} to 1.00×10^{-2} M with Nernstian slope of 19.64 ± 0.15 mV decade^{-1} and low detection limit of 5.01×10^{-7} M. The performance of the sensor is best in the pH range of 4.00-6.50 without any divergence in potential and it also works well in partially non-aqueous medium. The coated wire electrode has a short response time of about 5 s and is stable for at least 2 months. The proposed membrane electrode revealed good selectivity for Al(III) ion over a wide variety of other metal ions. It was successfully applied for determination of Al(III) ion in different real and environmental samples and also as an indicator electrode for potentiometric titration of Al(III) ion with EDTA.

Keywords: Al(III) selective electrode, Potentiometric sensors, Coated platinum electrode, Schiff base

INTRODUCTION

Ion-selective electrodes (ISEs) are well established analytical tools that are used routinely to measure a wide variety of different ions selectively and directly in complex samples. In recent decades, a large variety of mainly cation-selective carriers that induce extremely high selectivity of many orders of magnitude over other potentially interfering sample ions [1,2] have been synthesized by various research groups. Nowadays, many published articles on ionophore-based ISEs are available [3,4]. In spite of successful progresses in the design of highly selective ionophores for various metal ions, there are only a limited number of reports on the development of highly selective ionophores for trivalent ions such as Cr(III) [5], Al(III) [1,6,7,8], Ga(III) [9], and Ce(III) [10]. The increasing use of ion selective sensors in the fields of environmental, agricultural and medical analysis has stimulated analytical chemists to

develop new sensors for fast, accurate, reproducible and selective determination of various species.

An exciting advance in ISEs was made by Cattrall and Freiser [11] when they developed coated wire ISEs. Coated wire electrodes may suffer from reproducibility and long-term stability (drifting potential) problems, resulting from the poorly defined contact and mechanism of charge transfer between the membrane coating and the conducting transducer. Coated electrodes in which an electroactive species is incorporated into a thin polymeric film coated directly on a metallic or graphite conductor has been shown to be very effective for a wide variety of inorganic and organic cations and anions [12-14]. Electrodes of this sort are simple, inexpensive, durable, capable of reliable response in a wide concentration range for a wide variety of both organic and inorganic ions and suitable for measurements in small volumes of sample or for the desired in vivo applications of ISEs that biomedical researchers have long awaited.

Al(III) ion, despite its wide applications in our daily life,

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has been found to be a non-essential element for living systems. During the last two decades, many studies have focused on the toxicity of Al(III) ion on living system, especially on human being and it is believed that Al(III) ion plays important roles in the pathology of dialysis diseases. Aluminum is also known to cause dementia, anemia, bone and Parkinson disease [15-17]. Further, the detection of aluminum ion has always been a challenge due to the lack of spectroscopic characteristics, weak coordination ability in comparison with transition metal ions [18]. Therefore, this is very necessary to catch on alternate options for detecting the trace amount of aluminum ion. In search of new metal ion quantification techniques, the potentiometric membrane sensor is an inexpensive and convenient method, which provides acceptable sensitivity and selectivity for the target metal ion.

EXPERIMENTAL

Reagents

Analytical-reagent grade dibutyl phthalate (DBP), diethyl sebacate (DES), and dimethyl sebacate (DMS), were obtained from Fluka. Poly(vinylchloride) (PVC), sodium tetraphenyl borate (NaTPB) and tetrahydrofuran (THF) were purchased from Merck. The used ionophore is, 5-bromo(salicylidene-2-amino thiophenol) (ionophore) (Fig. 1). Nitrate and chloride salts of all cations used were of the highest purity available (all from Merck) and used without any further purification. Doubly distilled deionized water was used throughout.

Preparation of Ligand

The tridentate Schiff base ligand was prepared according to the literature [19,20] by condensation between 5-bromo salicylaldehyde (0.20 g, 0.001 mol), 2-amino thiophenol (0.11 g, 0.001 mol), in methanol and crystallized by dichloromethane/methanol mixed solvent through the partial evaporation of dichloromethane. The characteristics of the product are as follows: IR (KBr): cm^{-1}:3460(b), 1624(s), 1591(m), 1210(m). UV-Vis (C_2H_5OH), λ (ϵ, M^{-1} cm^{-1}) (167)(8400), 334(6200), 374(8100)

Apparatus and emf Measurements

All emf measurements were made at 25 °C by the use of
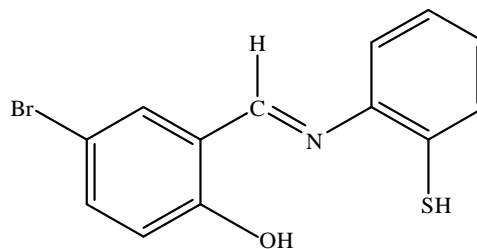

Fig. 1. Structural formula of ionophore.

a Metrohm pH-Ion Meter (Model 780) with a Ag.AgCl, KCl(sat,d) reference electrode in a cell assembly as follows: Ag.AgCl.Al(III) ion soln. membrane. Pt wire. Activities were calculated in accordance with the Debye-Huckel procedure [21].

Membrane Preparation

The schematic setup and general protocol for preparation of this sensor used in this work was similar to the previous studies [1,6,22] except that this electrode was a coated wire electrode with a Al(III) ion selective ionophore. In order to reach to a higher sensitivity, selectivity, and a wider dynamic linear range for the potential response of the aluminum-selective electrode, the effect of different proportions of the membrane component were investigated. The result show that the electrode with 5% w/w ionophore; 61.81% w/w DBP; 30.91% w/w PVC and 2.28% w/w NaTPB has a better potential response than that of the electrodes with other ratios. Total weight of this mixture (100.00 mg) was dissolved in dry freshly THF and was homogenized by ultrasound. The solvent was evaporated slowly to make one oily concentrated mixture. A platinum wire (0.5 cm) was dipped into the mixture for ~5 s until a thin film of membrane of ~0.1 mm thickness was formed. After each coating, the membrane was air-dried for 24 h by using the technique as literature [23]. The electrode was finally conditioned for 1 h in a 0.001 M of Al(NO₃)₃ solution.

Preparation of Real Sample

For the preparation of samples, 8 ml of Al-Mg syrup was heated in a furnace to dryness. The remainder was dissolved in 5 ml of concentrated nitric acid and diluted to 250 ml with pH setting on 4.5. Preparation of another samples for determination of its Al(III) ion content was

carried out by adding known amounts of concentrated nitric acid. After adjustment of pH to 4.5, content of Al(III) ion was estimated by proposed electrode using standard addition method.

RESULTS AND DISCUSSION

Electrode Response Characteristics

The 5-bromo(salicylidene-2-amino thiophenol) has three coordinating sites; *i.e.*, one hydroxyl group, one soulphonyl group and a ternary nitrogen atom, which in this research is used to detect of Al(III) ion. Further, its sufficient solubility in water and ability to complex with Al(III) ion [24,25] encourage us to use this ligand as ionophore in the PVC membrane.

In preliminary investigation, the suitability of the ionophore as an ion carrier was checked in the fabrication of the PVC-membrane- ISEs for a wide variety of metal ions including alkali, alkaline earth and transition metal ions. The potential response of the membrane electrode to various metal ions is shown in Fig. 2. As it is obvious among different cations that tested, Al(III) ion has the most sensitive response and is suitable for membrane of this electrode. This is due to the selective behavior of the PVC membrane system against Al(III) ion in aqueous solvent in comparison to the other metal ions tested. The Al(III) ion

selective coated platinum wire electrode at optimum composition showed a linear Nernstian response with a slope of 19.61 ± 0.73 mV decade^{-1} over the range of 5.2×10^{-7} to 1.0×10^{-2} M for $Al(NO_3)_3$ with a correlation coefficient of 0.998 (n = 7).

Optimization of Membrane Composition

Besides the critical role of the nature of ionophore in preparing PVC membrane electrodes, it is well known that some important features of the incorporated PVC membranes, such as the nature and amount of ionophore, the properties of plasticizer, the plasticizer/PVC ratio, and specially influence the sensitivity and selectivity of the ion-selective electrodes [26,27]. Thus, different aspects of the composition of membranes based on ionophore for Al(III) ion were optimized, and the results are summarized in Table 1. It is obvious that number 5 has better behavior with a good Nernstian response over a wide linear range. The potentiometric response of the membrane ion-selective electrodes based on the neutral ionophores is greatly influenced by the polarity of the membrane medium, which is defined by the dielectric constants of the major components of the membrane.

Some important features of PVC composite such as the properties and amounts of the plasticizer are investigated as one of significant influence on sensitivity and selectivity of
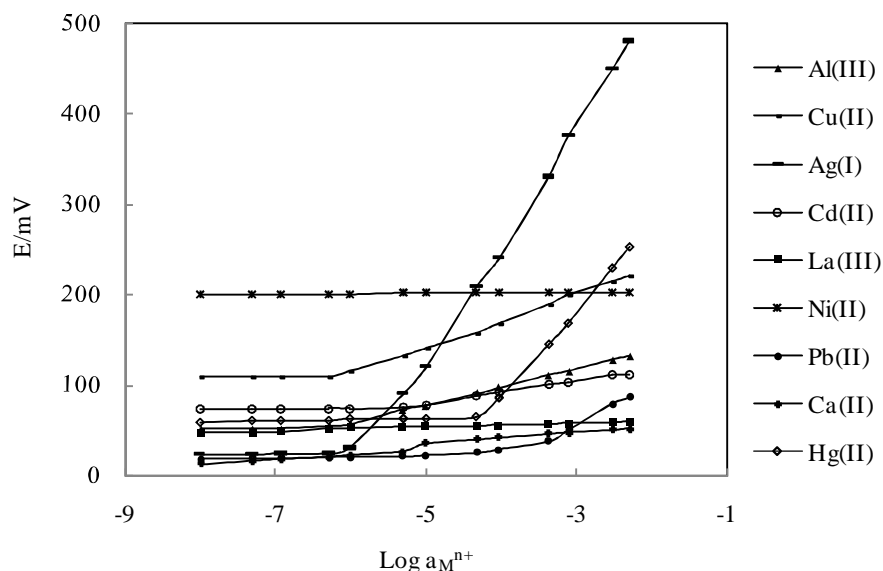

Fig. 2. The potential response of various cations on coated wire electrode.

this PVC composite. It should be noted that the nature of the plasticizer affects not only the dielectric constant of membrane, but also the mobility of ionophore molecules [26-29]. For this purpose, the influence of the nature of plasticizer on the Al(III) ion response was studied, several electrodes containing different types of plasticizers, namely DES, DMS, DBP were tested. ISE based on DBP exhibits a better Nernstian slope than that of DMS and DES (with slopes of 26.51, 33.30 mV decade^{-1}, respectively), as given in Table 2. Therefore, DBP was chosen as a plasticizer in the rest of experiments.

There are several reports on the effect of ionic additives in ISEs [29-31]. The presence of additives also may catalyze the exchange kinetics at the sample-membrane interface [32]. In this work, we examined the influence of NaTPB, as a suitable lipophilic additive, on the response

characteristics of the proposed PVC membrane, and the results are also included in Table 3. As Table 3 shows, in the absence of NaTPB the electrode exhibits near Nernstian, while adding more amount of the anionic additives to the membrane produces a super Nernstian. The obtained optimum amount of NaTPB was 1.2% and the experimental slope for this coated wire electrode at optimum condition of additive and ligand became 19.60 ± 0.42 mV decade^{-1} with a correlation coefficient of 0.990.

Effect of the pH of the Test Solution on the Electrode Potential

The pH response profile of the proposed electrode for 1.0×10^{-3} and 1.0×10^{-4} M Al(III) ion solutions was investigated over the pH range of 1.5-11.0. The pH was adjusted by using HNO$_3$ and NaOH solutions. The results

Table 1. Optimization the Ionophore of Membrane Composition Platinum Wire Electrodes for Al^{3+} Ion Detecting

Electrode No.	Ionophore (mg)	PVC (mg)	DBP (mg)	NaTPB (mg)	Slope (mV decade^{-1})	L.R. (μ M-M)	R^2	D.L. (μ M)
1	0.0	32.60	65.15	2.28	8.19	0.52-0.1	0.993	0.50
2	2.0	32.30	64.59	1.11	27.14	1.0-0.001	0.990	1.0
3	3.0	31.80	63.60	1.67	32.78	5.0-0.1	0.987	1.0
4	4.0	31.26	62.52	2.22	31.31	5.0-0.1	0.981	5.0
5	**5.0**	**30.19**	**61.81**	**2.28**	**19.87**	**0.52-0.01**	**0.991**	**0.50**
6	6.0	30.22	60.45	3.33	31.99	100.0-0.1	0.992	100.0
7	7.0	29.70	59.41	3.89	26.85	5.0-0.1	0.990	5.0
8	9.0	28.67	57.34	4.99	34.43	50.0-0.1	0.988	10.0
9	10.0	28.15	56.29	5.55	32.37	500.0-0.1	0.990	500.0

Table 2. Effect of type of plasticizer

Electrode No.	Type of plasticizer	Slope (mV decade^{-1})	L.R. (μ M-M)	R^2	D.L (μ M)
1	Without plasticizer	10.90	5.0-0.01	0.987	5.00
2	DBP	19.71	0.52-0.01	0.998	0.50
3	DMS	26.51	5.0-0.10	0.990	1.00
4	DES	33.30	50.0-0.10	0.995	50.00

obtained are given in Fig. 3. As seen, the potential remained constant in the pH range of 4.0 to 6.5. Therefore, this range was chosen as the working pH range of the proposed assembly. At lower pH (<4.0), the proposed electrode responds to hydrogen ions, while at higher PHs this response is reduced. This reduction might be attributed to the formation of some hydroxyl complexes of Al(III) .

Detection Limit, Reproducibility, Repeatability, Response Time and Life Time

Before determinig the analytical parameter in primary experiments, we investigated the applicability of this electrode in different media. Since, ionophore of this membrane composition may be soluble in non-aqueous solvents and reduce the stability (life time) and reproducibility of the electrode by leaching out of the membrane, therefore the applicability of this membrane composition electrode in mixed (water-organic) solutions is investigated. For this goal, linear range of electrode is tested in several mixed solvent with different %V/V such as ethanol/water. As shown in Table 4, linear range remains constant for below of %40 V/V ethanol/water, clearly suggested that electrode can be applied to estimate the Al(III) ion in partially nonaqueous solutions without any

Table 3. Optimization of Anionic Additive

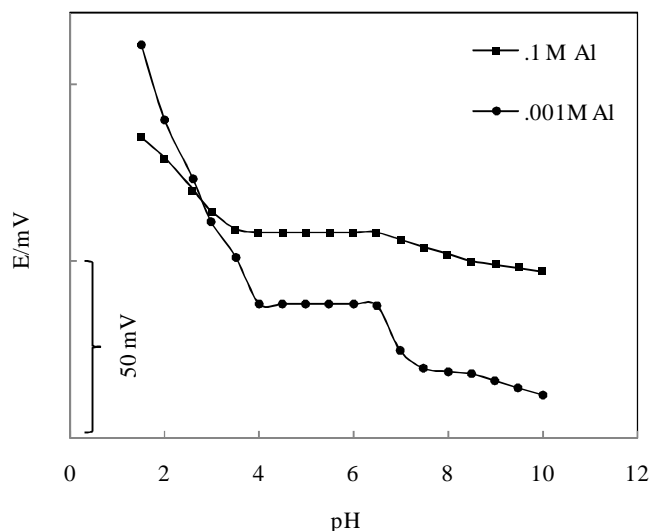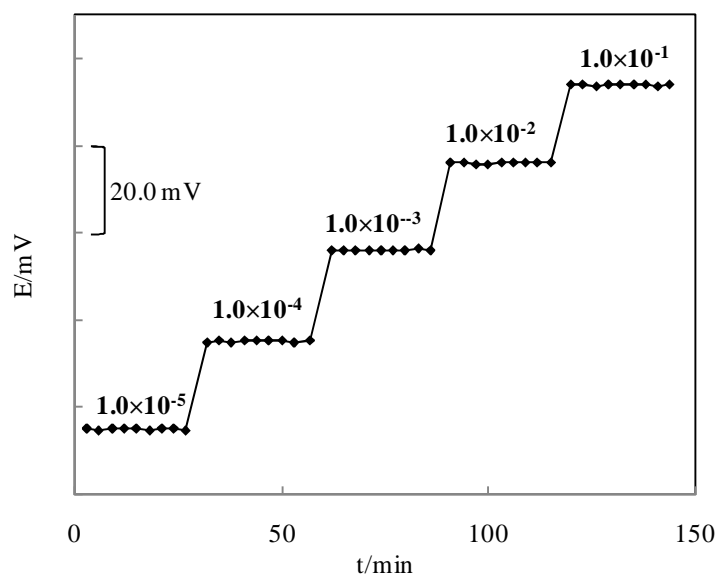| NaTPB	Slope (mV decade^{-1})	L.R. (µ M-M)	R^2	D.L. (µ M)
0.0	9.19	5.0-0.01	0.990	5.00
1.0	35.69	5.0-0.001	0.990	3.20
2.0	47.13	5.0-0.01	0.990	4.70
3.0	42.77	50.0-0.1	0.990	50.00
4.0	42.63	50.0-0.01	0.980	50.00
6.0	19.60	0.52-0.01	0.990	0.52
9.0	29.87	5.0-0.005	0.980	5.00
10.0	36.11	5.0-0.1	0.970	5.00

Fig. 3. Effect of pH on the response of the proposed composite electrode in two different concentrations of Al^{3+} ion in the pH range of 1.5-11.0.

Table 4. Effect of Ethanol as Nonaqeouos Solvent on the Electrode Response

%V/V Ethanol/Water	Slope (mV decade^{-1})	R^2	L.R. (μ M-M)
5	20.28	0.996	0.52-0.01
10	19.40	0.992	0.52-0.01
20	19.54	0.994	0.52-0.01
30	19.67	0.993	0.52-0.01
40	19.69	0.998	0.52-0.01
50	12.38	0.967	50.0-0.1

Fig. 4. Dynamic response of the proposed Al^{3+} ion selective electrode for step changes in concentration of Al^{3+} ion at pH 5.0, 1.0×10^{-5} to 1.0×10^{-1} M.

interference. It is necessary to mention that in the mixed solvent, up to %40 V/V ethanol/water, by leaching out of the membrane into solvent, the structure of electrode was destroyed. The limit of detection was 5.01×10^{-7} M, as determined from the intersection of the two extrapolated segments of the calibration plots. The time of contact and concentration of equilibrating solution were optimized, so that the membrane electrode was stable and potentials were reproducible at relatively short response times.

The optimum equilibration time in a 1.0×10^{-3} M Al(NO$_3$)$_3$ for the membrane electrode was about 24 h. The reproducibility was investigated by preparing seven similar electrodes at optimum membrane composition, then

the slope of each electrode was determined. The average slope with standard deviation was 19.43 ± 0.16 mV decade^{-1}. In the repeatability study, the calibration curves of one electrode in five times were obtained, the calibration curves had been taken every other day. The average slope with standard deviation was 19.26 ± 0.07 mV decade^{-1}. The response time of the electrode was evaluated (according to IUPAC definition) by measuring the time required to achieve a 90% value of steady potential for an Al(III) ion solution. A response time of 5 s was obtained for this PVC membrane (Fig. 4). To investigate the life time of the electrode the calibration curves of Al(III) ion selective electrode at its optimized composition were periodically

obtained for 2 months. The results showed that the potential response did not change significantly in this time duration. Regard to mentioned properties, we are going to estimate the ability of this electrode in a complex matrix such as AL-Mg syrup as a drug.

Selectivity Coefficients

The selectivity behavior is obviously one of the most essential characteristics of an ISE, determining the feasibility of a reliable measurement in a target sample. The experimental selectivity coefficients depend on the activity and the method of their determination. Different methods of selectivity determination are available in the literature. In the present study, the selectivity coefficients were determined using the fixed interference method (FIM).

The fixed interference method is based on the semi-empirical Nikolsky-Eisenman equation. The emf of a cell comprising an ion-selective electrode and a reference electrode were measured for solutions of constant activity of the interfering ion (1.0×10^{-2} M), a_J, and varying activity of the primary ion, a_I. The emf values obtained are plotted *vs.* the logarithm of the activity of the interfering ion. The intersection of the extrapolated linear portions of this plot indicates the value of a_I used to calculate $K_{I,J}^{pot}$ from the following equation:

$$K_{I,J}^{pot} = \frac{a_I}{a_J^{(z_I/z_J)}}$$

where "z" is the charge of ion. This equation, regardless of its simplicity and ease of use for mono-valance ions, gives the results higher than those expected. The selectivity coefficients were evaluated and the results are summarized in Table 5.

Analytical Applications

Potentiometric titration. The practical utility of the proposed membrane electrode was investigated by using as an indicator electrode for the titration of 25 ml of 1.0×10^{-2} M Al(III) ion solution against a standard 1.0×10^{-1} M EDTA solution at pH 5.0. The titration curve is shown in Fig. 5. End point was in 2.5 ml EDTA. Therefore, the amount of Al(III) ion in the solution at pH 5.0 can be determined with
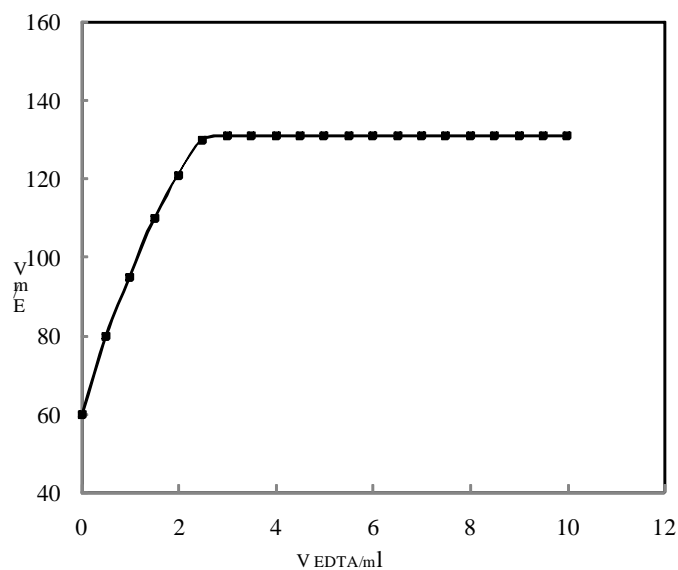
Table 5. Selectivity Coefficients of Various Ions for Al^{3+} ISE Calculated by FIM

Interfering ion	$\log K_{Al^{3+},M^{n+}}$
Ce^{3+}	-3.20
Cr^{3+}	-2.75
Fe^{3+}	-1.70
La^{3+}	-3.11
Ca^{2+}	-2.76
Co^{2+}	-4.29
Zn^{2+}	-4.79
Pb^{2+}	-3.10
Mn^{2+}	-4.15
Ba^{2+}	-2.18
Ni^{2+}	-3.49
Hg^{2+}	-2.58
Cu^{2+}	-1.90
Na^{+}	-4.83
K^{+}	-4.54

this electrode. Consequently, Al(III) ion selective sensor can be successfully used as an indicator electrode in the potentiometric titration.

Determination of Al(III) Ion in Al-Mg Syrup, Tap Water and Mineral Water

The proposed electrode was applied to the determination of Al(III) ion in AL-Mg syrup (a drug from SohaHelal Company), tap water and mineral water (from Satarap Company) under laboratory conditions. For the preparation of samples, 8ml of Al-Mg syrup was heated in a furnace to dryness. The remainder was dissolved in nitric acid and pH was set on 4.50, then diluted to 50 ml. The Al(III) ion concentration of the samples was determined using proposed electrode by the standard addition method. Al(III) ion was also determined by flame atomic absorption spectrometry (AAS). The results are shown in Table 6.

Table 6. Determination of Al^{3+} Ion Concentration in Real Samples

Sample	AAS method	Proposed electrode
Al-Mg Syrup	-	$7.19 \times 10^{-3} \pm 0.01 \times 10^{-3}$
Tap water (M)	$4.09 \times 10^{-3} \pm 0.01 \times 10^{-3}$	$4.29 \times 10^{-3} \pm 0.03 \times 10^{-3}$
Mineral water (M)	$4.90 \times 10^{-3} \pm 0.01 \times 10^{-3}$	$5.10 \times 10^{-3} \pm 0.04 \times 10^{-3}$

Table 7. Comparison of Proposed ISE with Previous Reported Al^{3+} Ion Sensors

Work No.	L.R. (μ M-M)	D.L. (μ M)	Response time	Life time	pH range	Ref.
1	0.30-0.05	0.250	10 s	45 days	3.1-5.5	[1]
2	5.00-0.01	2.500	10 s	10 weeks	2.9-5.0	[6]
3	1.00-0.16	0.600	<5 s	3 months	3.0-8.5	[7]
4	0.10-0.10	0.086	~10 s	3 months	3.0-6.0	[33]
5	18.00-0.10	8.700	10 s	1 month	2.5-4.5	[34]
6	0.52-0.01	0.500	5 s	2 months	4.0-6.0	Proposed ISE

Results from proposed electrode and AAS were found in agreement. Thus, it can be concluded that the membrane electrode may have applications in the drug analysis and environmental monitoring of Al(III) ion.

CONCLUSIONS

Based on the results obtained in this work, the proposed electrode has a simple operation, sufficient stability, precision, low detection limit, fast response time and direct application to determination of Al(III) ion in concentration range of 5.20×10^{-7} to 1.00×10^{-2} M. The electrode was found to perform satisfactorily over the pH range of 4.00-6.50 and can be successfully employed for the estimation of Al(III) ion in real samples. This electrode can be used not only in aqueous media, but also in detection of Al(III) ion in binary water-alcohol systems, containing 40.0% of ethanol.

Comparison of this sensor with previous reported sensors [1,6,7,33,34], shown in Table 7, indicate that our sensor has low detection limit, response time and partially good life time. Besides, the proposed sensor not only can be used in aqueous media and, but also it is applicable for the determination of aluminum ion in binary water-alcohol system this method in comparison with other reported methods in Al(III) detection, such as high performance chelation ion chromatography [35], and fluorescence spectrometry [36] is independent from temperature effects. Although other methods [37,38] might show more precise responses than those of this proposed electrode, advantages of the coated wire electrode make other methods superiority insignificant.

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