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Fabrication of a membrane Electrode by using bis (5-phenylazosalicylaldehyde)-1, 2-benzenediimine

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Abstract
A membrane electrode that has a specific response for a particular ion is known as an ion-selective electrode. Membrane electrodes are used to determine the concentration of ions. Fabrication of an ISE that has a rapid, sensitive, and accurate response was the objective of this work. In this work, a membrane electrode based on bis (5-phenylazosalicylaldehyde)-1, 2-benzenediimine) and DES was constructed. The membrane electrode exhibited a linear response to the concentration of Pb^{2+} within the interval of $6.7\times10^{-6} – 5.0\times10^{-3}$ with a slope of 35.5mV per decade and the appropriate pH was 3.5-5.0. The response time was 9 sec and the detection limit was 3.3×10^{-6}M. The proposed Pb^{2+} membrane sensor was used for complexometric titration of Pb^{2+} ion. The results were accurate and this electrode was used to determine Pb^{2+} in real samples.

Keywords: Electrochemical cells, Potentiometry, Ion-selective electrode, Lead-selective Electrode

Introduction

The first ISE was the “pH electrode”. It is used to measure the amount of H^+ or pH. Nowadays, pH Electrodes are widely used. Membrane electrodes based on Ionophores are used to determine the concentration of inorganic ions (Morf, 1981) [16]. The polymeric membrane is the essential constituent of a carrier-based membrane electrode. (Umezawa, 1990) [22]. It consists of an ionophore and lipophilic salt. The electrode reacts to the activity of the ion and typically has a large dynamic interval of concentration (Bakker, *et al*., 1997) [3]. Its selectivity depends on the equilibrium constant of the exchange reaction of the analyte and other ions between the two phases. It is related to the complex formation constant (k_f) of the interfering ions with the ionophore in the membrane phase. Ionophores are either charged or neutral in their unassociated form. Ionophores are macromolecules (typically macrocyclic) which bond to ions to form a complex (Buck *et al*., 1994) [5]. No important exchange happens within the membrane when the membrane is conditioned with a solution of the ion (Buck and Cosofert, 1993) [9]. Nowadays, we have hundreds of different types of ISE for the determination of the many ions (Korita, 1975; Manca *et al*., 1989; Sane, and Upadhyay, 1990) [13, 15, 20].

Reagents and Chemicals

Diethylsebacate (DES), Dibutyl phthalate (DBP), Benzyl acetate (BA), Poly (vinyl chloride) (PVC), Tetrahydrofuran (THF), and Sodium Tetraphenylborate (NaTPB) were used from Fluka or Merck Chemical Companies. Also, other chemicals provided from either Merck or Fluka.

A laboratory-synthesized Schiff’s base, bis (5-phenylazosalicylaldehyde)-1, 2-benzenediimine, was used as a neutral carrier (Kia, 2002) [12].

Membrane preparation

To prepare the PVC membrane, combine the specific amounts of PVC, ionophore (bis (5-phenylazosalicylaldehyde)-1, 2-benzenediimine) and plasticizer as a solvent mediator (DES). Then the solution was stirred, and this composition was dissolved in THF. A narrow tube (3.0 mm i.d. and 5.0 cm long) was dipped into the resulted solution for nearly 10 sec. The narrow tube was stored for 24 h at 25.0 °C. Then the tube was filled with a standard solution. Finally, the membrane was conditioned by the cation solution. The Ag/AgCl was utilized as an internal reference electrode.

Apparatus

The schematic representation of the constructed membrane electrode is:

Ag-AgCl | KCl (3M) | the internal solution, $Pb(NO_3)_2$ 1×10^{-2} M | PVC membrane | Examination solution | $Hg\text{-}Hg_2Cl_2$, KCl (sat'd).

A HIOKI3256 digital high tester was performed for potential measurement at 25 °C. Emf determinations were made with a SCE electrode (Philips). A Metrohm pH (model 780) meter was applied for pH determinations.

Results and discussion

The responses of ISE to various cations

The Pb^{2+} membrane electrode showed a linear response to the concentration of Pb^{2+} within the interval of 6.7×10^{-6} M – 5.0×10^{-3} M.

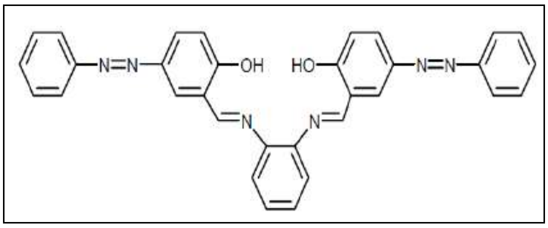

Fig 1: Structure of bis (5-phenylazosalicylaldehyde)-1, 2-benzenediimine (ionophore)

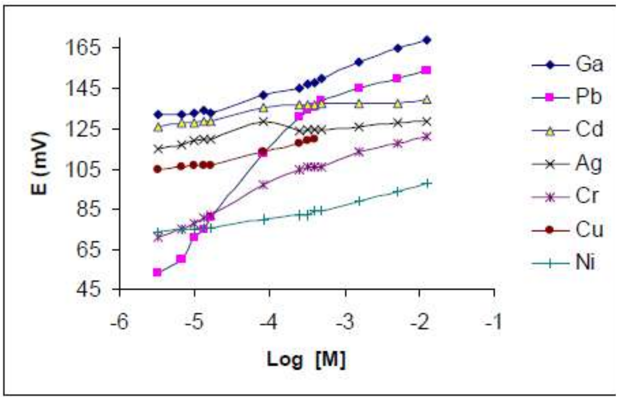

Fig 2: Potential responses of different cations

Choose of the solvent mediator

Several solvent mediators (plasticizer) were examined to modify the characteristics of the membrane. The membrane-based on DES had shown good potential response based on bis (5-phenylazosalicylaldehyde)-1, 2-benzenediimine (Figure 3).

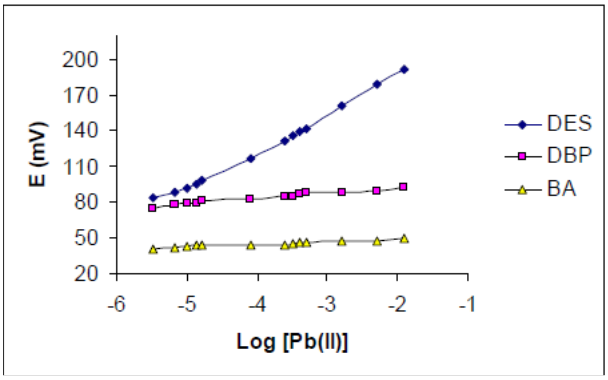

Fig 3: The effect of different solvent mediators on the potential response of ISE

The effect of internal solution concentration on the potential response of the ISE

Internal solution concentration has only a slight effect on the response of the ISE. However, the internal solution concentration was 0.010 M Pb $(NO_3)_2$.

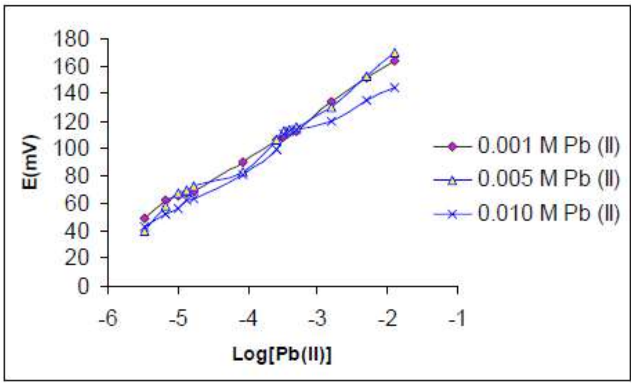

Fig 4: The effect of internal solution concentration on the potential response of ISE

The effect of the various ratio of DES/PVC on the potential response of ISE

Four electrodes with various weight ratio of DES/PVC (1, 1.5, 2.0, and 2.5) were prepared. The membrane with the DES/PVC ratios of 2.0 had shown a Nernstian response (Table 1 and Figure 5).

Table 1: Composition of Pb^{2+} ISE with a different weight ratio of DES/PVC

Electrode	1	2	3	4
DES/PVC	1.0	1.5	2.0	2.5
PVC%	47.7	38.5	31.8	27.3
DES%	47.8	57.0	63.7	68.2
Ionophore%	2.0	2.0	2.0	2.0
NaTPB%	2.5	2.5	2.5	2.5

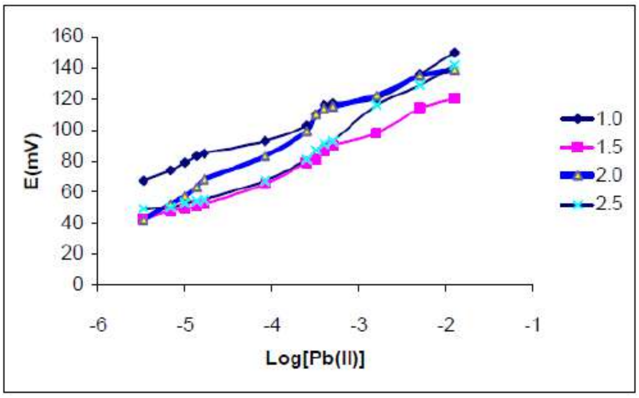

Fig 5: The effect of various ratios of DES/PVC on the membrane electrode response

The optimum amount of ionophore in the electrode membrane

Table 2: Composition of the membrane with different ratios of ionophore

Electrode	1	2	3	4
Ionophore%	0.0	1.0	2.0	3.0
PVC%	32.5	32.1	31.8	31.5
DES%	65.0	64.3	63.6	63.0
NaTPB%	2.5	2.5	2.5	2.5

To optimize the amount of ionophore in the electrode membrane, four electrodes were made. The membrane with 2% of the ionophore had shown a Nernstian slope (Table 2 and Figure 6).

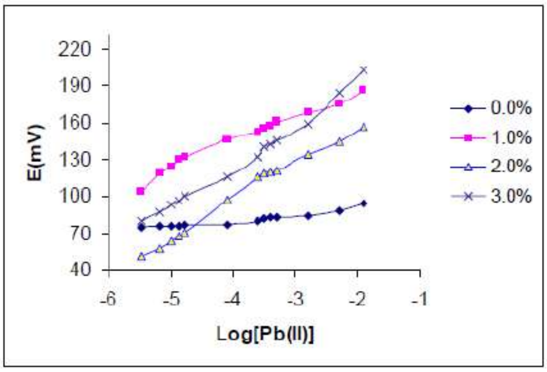

Fig 6: Responses of the electrode with various ratios of ionophore.

Optimizing the amount of additive (NaTPB) on the membrane of Pb^{2+} ISE

Different amounts of NaTPB were tested. The compositions of electrodes are given in Table 3 and their corresponding responses are given in Figure 7. As results show, the membrane with 2.5% of the NaTPB has a better response.

Table 3: Potential responses (in mV) of Pb^{2+} ISE at different amounts of NaTPB

Electrode	1	2	3	4
Ionophore%	2.0	2.0	2.0	2.0
PVC%	32.7	32.3	31.8	32.1
DES%	65.3	64.7	63.7	64.3
NaTPB%	0.0	1.0	2.5	3.5

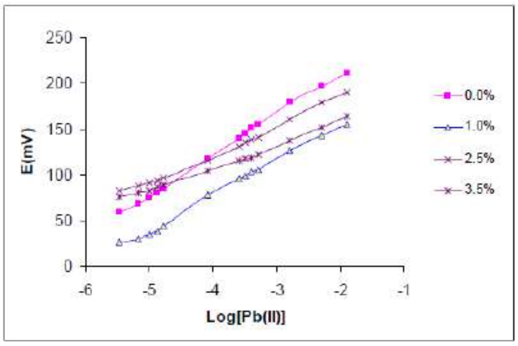

Fig 7: Responses of Pb^{2+} membrane with various ratios of NaTPB

Repeatability

The responses of six electrodes under the optimum conditions were measured (Figure 8).

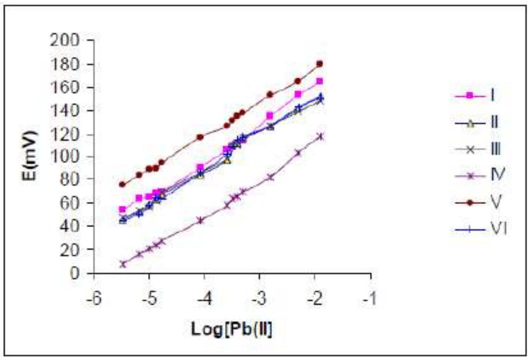

Fig 8: Repeatability of the Pb^{2+} ISE

Response Time

According to Figure 9, the time needed to reach the equilibrium was 9 seconds.

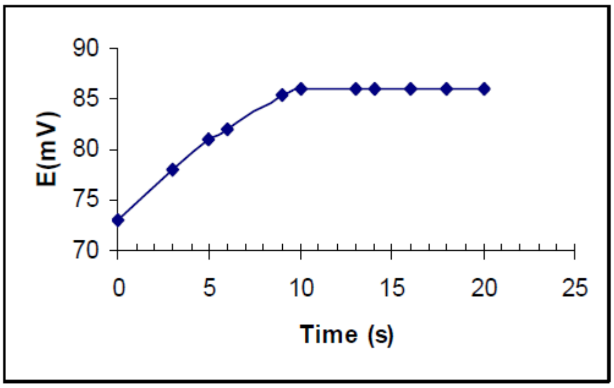

Fig 9: Response time of lead electrode

Detection limit

As figure 10 shows, the detection limit was 3.3×10^{-6} M. Potentials were constant at the pH interval 3.5-5.0

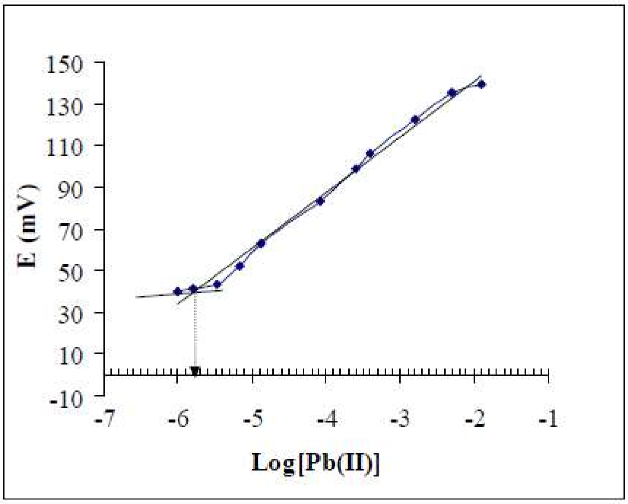

Fig 10: Lower detection limit of the lead electrode

Effect of the pH of the sample solution on the membrane response

The effect of the pH of the sample solution on the potential response of the membrane was studied over the pH interval of 2.5 −11.5. Potentials were constant at the pH interval of 3.5-5.0 (Figure 11). The drift could be due to the protonation of the ionophore and formation of hydroxy complexes of Pb^{2+} ion.

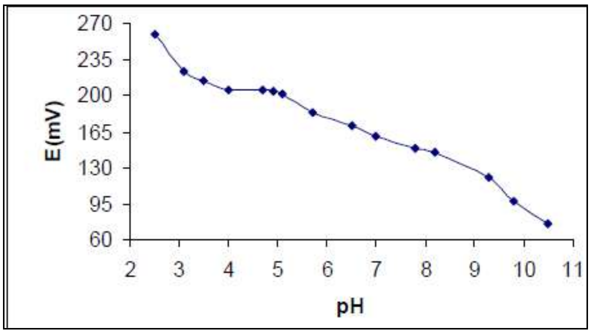

Fig 11: Effect of pH of the sample on the potential response of the lead electrode

Selectivity coefficients of the Pb^{2+} ISE

Match potential method (MPM) was used to determine the value of the selectivity coefficient. In Table 4, the selectivity coefficients for some ions are presented.

Table 4: The Effect of pH of the sample on the membrane responses

M^{n+}	Selectivity coefficient
Ag^{+}	3.3×10^{-2}
Ba^{2+}	4.6×10^{-3}
Cd^{2+}	7.4×10^{-2}
Ce^{3+}	2.9×10^{-3}
Co^{2+}	5.7×10^{-2}
Cr^{3+}	6.7×10^{-2}
Cu^{2+}	8.7×10^{-2}
La^{3+}	6.7×10^{-3}
Li^{+}	1.8×10^{-3}
Mg^{2+}	4.5×10^{-3}
Mn^{2+}	6.2×10^{-2}
Ni^{2+}	7.5×10^{-3}
Na^{+}	8.4×10^{-3}
Sr^{2+}	3.6×10^{-3}
Zn^{2+}	2.4×10^{-2}

Analytical application of Pb^{2+} ISE

The constructed Pb^{2+} sensor was applied for complexometric titration of 30 ml 1.0×10^{-4}M Pb^{2+}solution with EDTA (Figure 12).

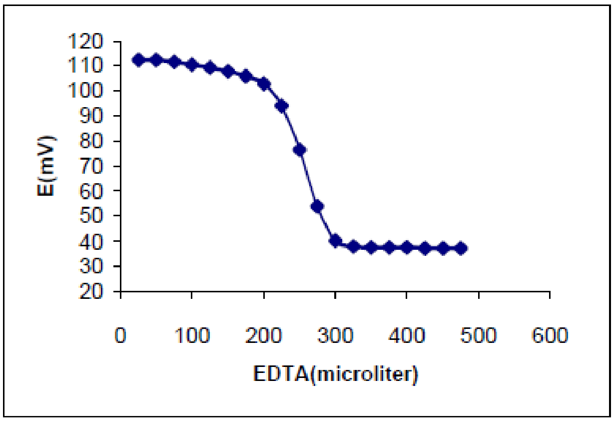

Fig 12: Titration curve for titration of 30 ml of 1.0×10^{-4} M Pb^{2+} with 0.01 M EDTA using Pb^{2+} ISE

Conclusions

In this work, we construct a membrane electrode based on a laboratory-synthesized Schiff's base, bis (5-phenylazosalicylaldehyde)-1, 2-benzenediimine) was utilized as a neutral carrier and DES as a plasticizer, that has a fast, sensitive and accurate response for Pb^{2+} ion. The proposed Pb^{2+} membrane sensor was applied for complexometric titration of Pb^{2+} ion with EDTA. The results were accurate and this electrode could determine Pb^{2+} in real samples.

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