

Full Paper

Fabrication of a Lead All Solid State Sensor for ppb Level Monitoring of Aqueous Lead Samples

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Abstract- A novel all-solid-state ion selective electrode (ASS-ISE) has been constructed for the determination of Pb(II) ions in complex aqueous media. The ASS-ISE is constructed of a layer of a conductive graphite-epoxy resin composite on a copper wire, further coated with a Pb(II) selective PVC membrane. The optimal PVC membrane is composed of 31% PVC, 62% nitrobenzen (NB), 2% of sodium tetraphenyl borate, and 5% of N,N'-dimethylcyanodiaz-18-crown-6 as a selectophore (L). The studies proved the device to have a Nernstian response (i.e. 29.4 ± 0.3 mV per decade) in a concentration range from 1.0×10^{-8} mol L⁻¹ to 1.0×10^{-3} mol L⁻¹, with a lower detection limit of 4.0×10^{-9} mol L⁻¹. The proposed ASS-ISE was also found to possess a good Pb(II)-selectivity as opposed to various interfering cations. The device was further practically tested in the analysis of lead content of some water samples with complex matrices.

Keywords- Lead, Sensor, All solid state, Potentiometry, Water

1. INTRODUCTION

Lead is a widely used element with varied industrial applications in alloys, storage batteries, paints, high quality glass, solder for electronic applications, and also for shielding radiation. The element is, however, very toxic and accumulates in liver and kidneys, as well as the central nervous system of mammals, if they are exposed to it [1]. The WHO and the US Environmental

Protection Agency (EPA) have set a maximum tolerable amount of 0.05 mg or lead in 1L of drinking water, and the maximum tolerable concentration of lead in waste waters is in the same order [1]. Evidently, the determination of trace amounts of the element in different samples is an attractive area for research.

Among the conventional methods used for the determination of Pb(II) ion in various samples, spectrophotometric methods [2], atomic absorption spectroscopy–electro thermal atomization (AAS–ETS) [3,4], inductively coupled plasma–atomic emission spectroscopy (ICP–AES), inductively coupled plasma–optical emission spectroscopy (ICP–OES) or the inductively coupled plasma–mass spectroscopy (ICP–MS) [5,6], chromatography [7] and photometry [8–10] are the most well-known. Yet, the majority of these techniques need cumbersome sample pre-treatment; and require infrastructural back-up, and are consequently not convenient for routine analytical applications.

Potentiometric sensors, on the one hand could be good alternatives for such applications since they offer merits like high selectivity, sensitivity, good precision, simplicity and reasonable analysis costs. These versatile analytical devices [11] have hence studied as an alternative for the analysis of Pb(II), based on several neutral compounds with oxygen, nitrogen and sulphur donor atoms [12–16].

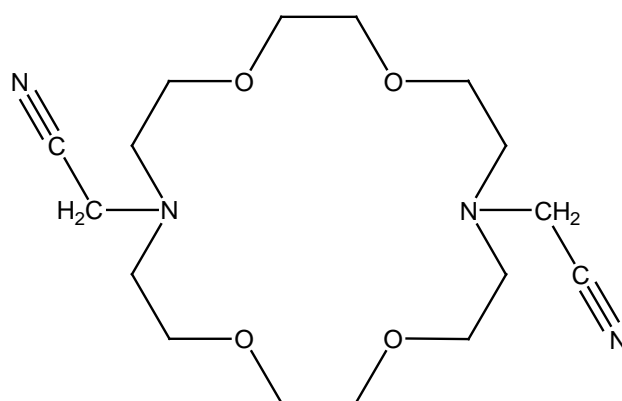
All solid-state ion-selective electrodes (ASS-ISEs), on the other hand, constitute a sub-category of ISEs, with advantages of freedom from the limitations of imposed by the presence of internal solutions, and hence enjoy improved limits of detection, life times and more variable applicable positions further to the fact that these devices can be based on various intermediary materials [17-21]. Additionally, ASS-ISEs offer better mechanical stability [22-28], which make researchers and producer able to construct them in various mechanical designs and even produce them as disposable electrodes.

Given these advantages and the need for precise, accurate and selective devices for the analysis Pb(II), different ionophores have been evaluated for the construction of polymeric lead-selective electrodes, and since the development of a portable devices for on- and off-line monitoring of Pb(II) in different samples, has long been very interesting, the present work was focused on the design and construction of a Pb(II) selective ASS-ISE. The all-solid-state element of the electrode was based on a conductive polymeric composite (CPC) composed of multi-walled carbon nanotubes (MWCNTs) and an epoxy resin. This element was next coated on a copper wire and after drying the whole ASS element was then covered with a layer of a selective PVC membrane.

2. MATERIALS

Tetrahydrofuran (THF), dibutyl phthalate (DBP), benzyl acetate (BA), nitrobenzen (NB), potassium tetrakis (p-chlorophenyl) borate (KpClTPB) and sodium tetrapenyl borate (NaTPB), graphite powder (1–2 μm particle size), the MWCNTs and all salts (nitrate or chloride form)

were of analytical reagent grade and were obtained from Merck Co., high-molecular weight polyvinylchloride (PVC) was from Fluka Co. and the ion-carrier L, N,N'-dimethylcyanodiaza-18-crown-6 (Scheme 1) was previously introduced as a novel selectophore for Pb(II) ions and its synthesis has been described earlier [29]. Macroplast Su 2227 epoxy was from Henkel Co. and the desmodur RFE hardener was obtained from Bayer Ag.



Scheme 1. Chemical structure of N,N'-dimethylcyanodiaza-18-crown-6 (L) as the used selectophore for Pb(II) ion

2.1. Preparing the ASS-ISE

The first step for preparing the ASS element included preparing the composite. This was achieved through add-mixing various amounts of graphite powder, MWCNTs, the epoxy resin and the hardener in a small amount of THF. This mixture was left in the air to allow some of its content to evaporate and next a shielded copper wire (0.5 mm diameter and 15 cm length) was polished and dipped into the resulting viscose mixture to be coated with a layer of the composite. The so-prepared ASS was left to dry in the air for 12 h.

After the ASS element was prepared, it was dipped into a cocktail of the selective PVC membrane 3-5 times (so that a polymeric membrane formed on its surface) and left to dry in air for one day. Experiments proved that the optimal results could be obtained with a PVC membrane composed of 5% wt. of L, 61% wt. of NB, 31% wt. of PVC, and 2% wt. of NaTPB, prepared through add-mixing the ingredients in a small volume of THF and aging the resulting homogenous mixture in air so as to viscosity.

The final step included conditioning the ASS-ISEs in a 10^{-3} mol L⁻¹ Pb(II) solution and then using them in a cell assembly as below:



The electronic connection of the two electrodes was established through an ion analyzer with a 250 pH/mV ± 0.1 mV. The measurements were performed using the calibration method.

3. RESULTS AND DISCUSSION

3.1. Membrane composition

Reviewing the literature revealed that the majority of Pb(II)-ISEs have been symmetrical devices (i.e. the polymeric ISE acts as the interface of the internal and external solutions), which suffer certain limitations in terms of robustness and miniaturization of the devices. Naturally, asymmetrical devices, in which the internal solution is eliminated, enjoy the advantages required for medicinal, biological, environmental and even in vivo applications.

With ASS-ISEs, the presence of the CPC helps combine the merits of PVC membranes, which originate from their variable and adjustable compositions, with those of solid state devices. Clearly the nature and amount of the ingredients of the PVC membranes, leave critical effects on the response and behavior of the sensors [27-36], and hence the effect of the membrane ingredients on the ASS-ISE response, were evaluated by preparing and evaluating different PVC membrane compositions (Table 1). Former experiments have revealed a plasticizer/PVC ratio of around 2.0 generally to be optimal, and consequently this ratio was maintained in all compositions. To further simplify the expression of the results in all compositions the amount of PVC was kept at 31% wt.

Table 1. Various membrane ingredients used in making ASS-PVC membrane sensor

No.	Composition (%)				Slope* (mV/decade)	DL (mol L ⁻¹)
	PVC	Plasticizer	Ion-carrier	Additive		
1	31	66 NB	3	-	16.1 \pm 0.4	1.7 \times 10 ⁻⁶
2	31	65 NB	4	-	17.4 \pm 0.3	1.1 \times 10 ⁻⁶
3	31	64 NB	5	-	18.4 \pm 0.2	1.0 \times 10 ⁻⁶
4	31	63 NB	6	-	18.4 \pm 0.3	1.0 \times 10 ⁻⁶
5	31	63 NB	5	1 NaTPB	25.8 \pm 0.2	1.2 \times 10 ⁻⁸
6	31	62 NB	5	2 NaTPB	29.4 \pm 0.3	4.0 \times 10 ⁻⁹
7	31	62 NB	5	2 KpClTPB	29.3 \pm 0.3	6.5 \times 10 ⁻⁹
8	31	62 DBP	5	2 NaTPB	25.8 \pm 0.2	8.5 \times 10 ⁻⁹
9	31	62 BA	5	2 NaTPB	26.2 \pm 0.3	7.3 \times 10 ⁻⁹
10	31	67 NB	-	2 NaTPB	6.7 \pm 0.2	4.4 \times 10 ⁻⁴

* The results are based on five replicate measurements

A plasticizing agent, which is an inert and generally non-volatile compound capable of dissolving PVC, was also used in PVC membranes. This is to enhance the mobility of the free and complexed species. The compounds were chosen from conventional plasticizers, and included dibutyl phthalate (DBP), nitrobenzen (NB) and benzylacetate (BA). The experiments revealed NB lead to the best responses, most probably due to the rather high polarity of Pb(II) ions, which prefer a high polarity media, such as NB.

Further critical ingredient of a PVC ISE membranes is its ionic additive since the incorporation of small amounts of such compounds can significantly enhance the exchange mechanism of ionic species and hence lower the Ohmic resistance of an ISE membrane [30-35]. Here, 2% NaTPB improved the sensor response and decreased the Ohmic resistance of the membrane.

It was also found through the study of the PVC membrane compositions (Table 1) that increasing the amount of the ionophore up to 5% wt. increased the sensor response. Above this amount, however, the response did not change and consequently 5% wt. was chosen as the optimal value for the concentration of the ligand.

By testing the response behavior of a membrane lacking the ion carrier (membrane no. 10), it was found that a blank membrane shows very poor sensing behaviors, while further studies indicated that a membrane with the composition of no. 6, led to the best Nernstian slope (29.4 ± 0.3 mV/decade).

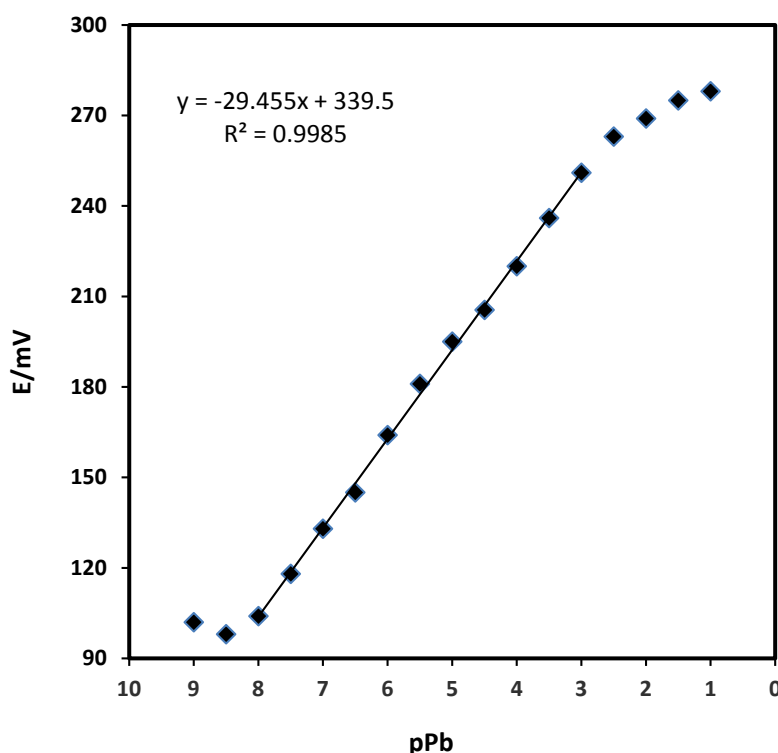


Fig. 1. Calibration curve of Pb(II)-ASS-ISE; the results are based on 5 replicate measurements

3.2. Figures of merit for the Pb(II) ASS-ISE

Evaluation of the response of the optimal ASS-ISEs at different Pb(II) concentration ranging from 1.0×10^{-9} to 1.0×10^{-1} mol L⁻¹ and plotting the results against $-\log [\text{Pb(II)}]$ (Figure 1), indicated that the response of the developed ASS-ISE with the optimal composition is linear from 1.0×10^{-8} - 1.0×10^{-3} mol L⁻¹. This shows a considerable improvement as compared to the results of symmetrical devices, which commonly range from 0.1 to 10^{-5} or even 10^{-6} mol L⁻¹.

The lower detection limit of the device, acquired from the calibration curve data, was found to be 4.0×10^{-9} mol L⁻¹, through extrapolating the two linear parts at the lower concentrations of the calibration curve, while the upper detection limit was 10^{-3} mol L⁻¹.

Further, the dynamic response time of the proposed ASS-ISE [36-44], was evaluated by successively immersing the device into different sample solutions from 1.0×10^{-8} to 1.0×10^{-3} mol L⁻¹ and recording the potential time behavior. The results revealed that the response time of the sensor to be only around 10 s.

The potential-pH behavior of the ASS-ISE was further assessed by recording the response behavior of the optimal electrode, in a solution with a fixed and known concentration of Pb(II) (i.e. 1.0×10^{-5} mol L⁻¹), upon continuously varying the pH of the solution (from 2.0 to 10.0). The changes in the pH were the created by adding small volumes of concentrated NaOH or HCl solutions so as to avoid considerable concentration changes.

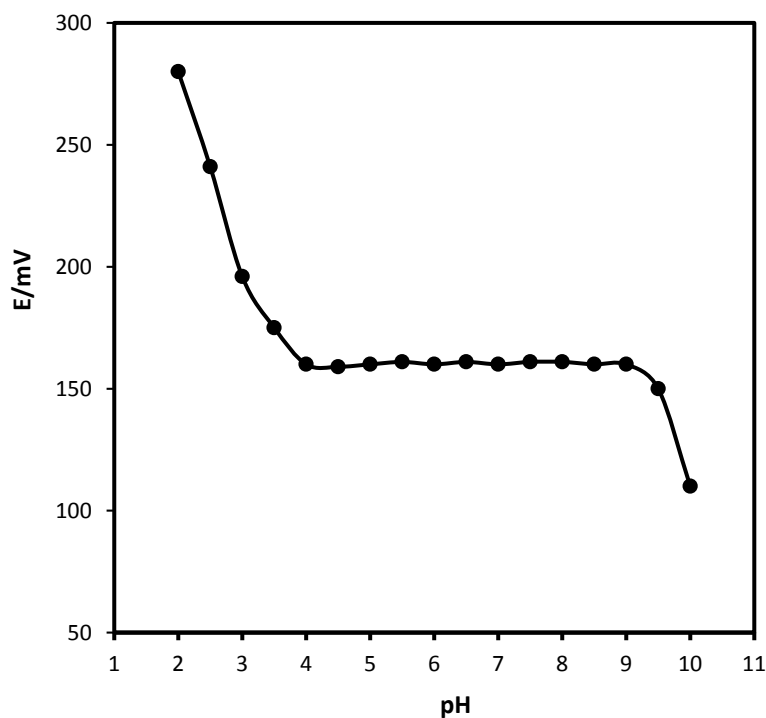


Fig. 2. pH effect on the potential response of the ASS-ISE in a solution of Pb(II) (1.0×10^{-5} mol L⁻¹)

The resulting plot (Figure 2) revealed the response of the sensor to be independent from pH values in the range of 4.0 to 9.0. The considerable potential changes above pH=9.0, were the result of the formation of $\text{Pb}(\text{OH})^+$ or $\text{Pb}(\text{OH})_2$ species, which lower the activity of the free Pb(II) ion. At pH values less than 4.0, the protonation of the N atoms in the structure of the ionophore leads to the interference of protons leading to drifts towards more positive values [45-49].

The evaluation of the lifetime of the sensor was made by daily recording the calibration curve and potential response of 3 sensors for 1 hour/day for 12 weeks. The life time of ASS-ISEs are expected to be in the range of 4–10 weeks [50-57]. The experiments showed that after a period of 7 weeks, the potential slope and lower detection limit decreased and increased respectively. This has most probably been the result of the gradual leakage and loss of the membrane ingredients (i.e, plasticizer, the ion-carrier, or the ionic additive).

The selectivity coefficients of the ASS-ISE, described as an ion selective electrode's tendency to specifically respond to the target ion, in the presence of interfering ions, is the most important characteristic of any ion selective electrode, as indicated by its name. These values were also evaluated by the well-established matched potential method (MPM) [58-63] and the results are given in Table 2.

Table 2. The selectivity coefficients of various interfering cations for Pb(II) ASS-ISE

Cation	Selectivity Coefficients
Na^+	$<10^{-6}$
K^+	$<10^{-6}$
Mg^{2+}	$<10^{-6}$
Ca^{2+}	$<10^{-6}$
Hg^{2+}	7.5×10^{-4}
Zn^{2+}	2.3×10^{-4}
Co^{2+}	1.9×10^{-4}
Cd^{2+}	8.9×10^{-4}
Al^{3+}	$<10^{-6}$
Ni^{2+}	3.9×10^{-4}
Fe^{3+}	7.2×10^{-6}
NH_4^+	1.1×10^{-6}
Cu^{2+}	2.5×10^{-4}

The content of the table revealed that none of the evaluated interfering ions can lead to significant errors in the response of the proposed sensor.

Finally, to assess the possibility of using the ASS-ISE in the analysis of real samples, the optimal electrode was used for the determination of Pb(II) concentration in some aqueous waste water samples, acidified with HNO₃ (pH about 5) prior to the analysis.

Each sample was tested 5 times, through the calibration curve method and the results are summarized in Table 3. To validate the method, the results (Table 3) were compared with those of obtained through inductively coupled plasma mass spectrometry, which confirmed them to be consistent.

Table 3. Results of lead analysis in water samples by the proposed method and reference method

Sample	Found by the ASS-ISE* (ppb)	Found by ICP-Mass (ppb)
Sample 1	22.3±0.5	21.7±0.4
Sample 2	18.8±0.4	18.2±0.3
Sample 3	26.8±0.9	25.8±0.3
Sample 4	17.5±0.6	17.8±0.4

* The results are based on five replicate measurements

Results of the real sample analysis showed the applicability of the Pb(II) ASS-ISE in complex water matrixes.

4. CONCLUSION

Since lead is a widely used element in variety of industrial applications such as alloys, storage batteries, paints, high quality glass, solder for electronic applications, and also for shielding radiation, it can enter to environment and be a serious treat for human life. Thus, monitoring of lead content of water samples is of great importance. In this work, an all-solid-state ion selective potentiometric sensor for the determination of Pb(II) in waste water samples was prepared based on a conductive polymeric composite (CPC) of graphite powder, MWCNTs, and an epoxy resin and a PVC membrane. The CPC was coated on a copper wire and then coated with a thin layer of the PVC-based ion selective membrane. The best PVC membrane which contained 31% wt. of PVC, 62% wt. of NB, 2% wt. of an ionic additive, and 5% wt. of the ion-carrier L was selected after several experiments. N,N'-dimethylcyanodiaz-18-crown-6 (L) was used as the selectophore for Pb(II) ion. The final ASS-ISE proved to have

a dynamic linear range of 1.0×10^{-8} to 1.0×10^{-3} mol L⁻¹ of Pb(II), and a detection limit of 4.0×10^{-9} mol L⁻¹. The life time of ASS-ISEs was about 7 weeks. The response of the sensor was independent from pH values in the range of 4.0 to 9.0. Finally, the ASS-ISE was effectively applied to the analysis of Pb(II) content of some waste water samples.

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