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ORIGINAL PAPER

Synthesis and electroanalytical applications of a novel hydrazinecarbothioamide derivative molecule

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Abstract

Molecules containing multiple donor atoms such as nitrogen, oxygen and sulfur in their structure are more preferred as ionophores in the preparation of ion-selective electrodes. In this study, a new hydrazinecarbotioamide derivative molecule was synthesized as an ionophore and characterized using NMR, FT–IR and elemental analysis techniques. The potentiometric electrodes prepared with the synthesized new molecule (ionophore) exhibited selectivity towards lead(II) ions. This novel electrode had a low detection limit of 6.98×10^{-7} M over a wide concentration range of 1.0×10^{-6}–1.0×10^{-1} M. Obtained experimental data showed that the electrode has good repeatability, good selectivity and good stability. At the same time, it could work in a wide pH range without being affected by pH changes. The analytical applications of the developed electrode, whose potentiometric performance parameters were studied, were carried out in different water samples.

Keywords Lead(II) · Potentiometry · Hydrazinecarbothioamide · Ionophore · Synthesis

Introduction

Lead (Pb), which is an element widely used in various fields of industry, is one of the important heavy metals that cause environmental pollution (Kendüzler et al. 2020). Lead is a highly toxic metal and has a high accumulation potential in the body of organims (Zhai et al. 2013). In the human body, lead can easily accumulate in the blood, nervous system, kidney, reproductive system and brain, and can cause various health problems such as acute lead poisoning, organ dysfunctions and brain damage (Deepa et al. 2014; Khazal et al. 2022). Also, due to its toxicity, lead is considered as an inorganic pollutant (Esmailzadeh et al. 2022). Considering all these points, the determination of lead in various samples is very important for environmental and human health. So far, the determination of lead in various samples has been carried out by different analytical methods such as atomic absorption spectrometry (AAS) (Soylak and Yilmaz 2011; Tinas et al. 2018), spectrophotometry (Jankiewicz et al. 2001), inductively coupled plasma mass spectroscopy (ICP–MS) (Su et al. 2014) and high performance liquid chromatography (HPLC) (Liang et al. 2018). These proposed analytical methods are expensive, complex and require pre-treatment, as well as experienced personnel and a well-equipped laboratory (Gupta et al. 2012; Isildak et al. 2023).

Potentiometric ion-selective electrodes (ISEs), which are important alternatives to the known analytical methods used in the determination of various heavy metals, are an important option for on-site and rapid analysis (Saadeh et al. 2012a, b; Özbek and Isildak 2022a, b). In addition, potentiometric ISEs have a low detection limit over a wide range of concentrations and are very successful in the detection of various ions, organic and inorganic species (Fouad et al. 2023; Cetin et al. 2023) Ionophores are important agents that enable detection by interacting with the analyte in potentiometric ISEs (Özbek et al. 2022a). Crown ethers (Ghorbani et al. 2013), pyridinecarboximide derivatives (Hassan et al. 2003), calixarenes (Cadogan et al. 1999), porphyrins (Vlassici et al. 2008) and carbothioamide derivatives (Özbek et al. 2022b; Özbek 2022) were used as ionophores for Pb(II) determination.

In this study, (*E*)-2-(1-(2-oxodihydrofuran-3(2*H*)-ylidene) ethyl)hydrazinecarbothioamide **(3)** (Fig. 1) was used as an ionophore, and selective potentiometric sensors were

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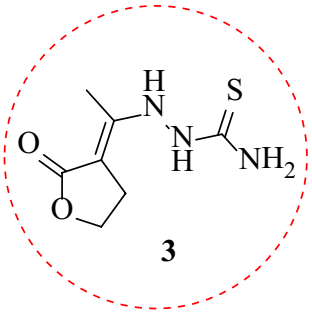


Fig. 1 The chemical structure of (*E*)-2-(1-(2-oxodihydrofuran-3(2*H*)-ylidene)ethyl) hydrazinecarbothioamide

prepared for Pb(II) ions. Linear working range, detection limit, response time, pH working range parameters and analytical applications of the prepared electrodes were investigated.

Experimental

Reagents and chemicals

All reagents used in the (*E*)-2-(1-(2-oxodihydrofuran-3(2*H*)-ylidene)ethyl) hydrazinecarbothioamide (ionophore) synthesis were obtained from Sigma Aldrich. Graphite (Sigma Aldrich), epoxy (Macroplast Su 2227; Henkel) and hardener (Desmodur RFE; Bayer AG), were used to prepare the solid contact electrodes. Plasticizers [bis(2-ethylhexyl)adipate (DEHA), bis(2-ethylhexyl)sebacate (BEHS) and *o*-nitrophenyloctyl ether (*o*-NPOE)], poly(vinyl chloride) (PVC), potassium tetrakis(*p*-chlorophenyl)borate (KT*p*ClPB) and tetrahydrofuran (THF) purchased from Sigma Aldrich. The nitrate salts of cations used for the preparation of stock solutions were also obtained from Merck and Sigma Aldrich. The nitric acid and sodium hydroxide used in the pH studies were obtained from Sigma Aldrich and Merck, respectively.

Apparatus

^{1}H– and ^{13}C–NMR spectra were recorded on a Brucker Advance III instrument (400 MHz). FT–IR spectra was obtained by PerkinElmer Frontier spectrometer. The melting point of the ionophore was measured in Electrothermal 9100. Surface images of prepared electrodes were carried out with a scanning electron microscope (SEM) (Quanta FEG 450-FEI). Potentiometric data were recorded by using a computer–controlled multichannel potentiometric system (Medisen Medical Ltd. Sti., Turkey). A lab-made software program was used in the measurement system. Ag/AgCl electrode (Thermo–Orion) was used as reference electrode.

Method

Synthesis of (*E*)-2-(1-(2-oxodihydrofuran-3(2*H*)-ylidene) ethyl) hydrazinecarbothioamide

(*E*)-2-(1-(2-oxodihydrofuran-3(2*H*)-ylidene)ethyl)hydrazinecarbothioamide was synthesized using the method reported in our previous studies (Özbek et al. 2023; Doğan et al. 2022). The 2-acetyl-γ-butyrolactone **(1)** (1 mmol) was dissolved in warm ethanol (20 mL). Thiosemicarbazide **(2)** (1 mmol) was dissolved in warm water (20 mL) and then added to this solution. Acetic acid (5 drops) was added to this mixture and stirred at room conditions for 4 h. The precipitate formed at the end of the reaction was filtered and washed several times with ethanol. The synthesis scheme of (*E*)-2-(1-(2-oxodihydrofuran-3(2*H*)-ylidene)ethyl)hydrazinecarbothioamide **(3)** is given in Fig. 2.

Preparation of ion-selective electrodes

In this study, the ISEs were prepared in two stages as detailed in our previous studies (Özbek and Ölcenoglu 2023; Özbek 2023). Firstly, the solid contact mixture consisting of graphite (50.0%), epoxy (35.0%) and hardener (15.0%) was dissolved in THF (3 mL) and mixed until it becomes homogeneous. 20 cm long copper wires were coated with the mixture reaching a certain viscosity and left to dry for 24 h in the dark. This mixture was prepared because the membrane structure cannot be attached directly to the copper wire surface. Also, the solid contact mixture is to provide electrical conductivity. Then, PVC membrane ion selective electrodes were prepared. For this, PVC, plasticizer, KT*p*ClPB and ionophore were dissolved in THF (3 mL) in the ratios given in Table 1. After obtaining a homogeneous mixture, the surface of the previously prepared solid contact electrodes was

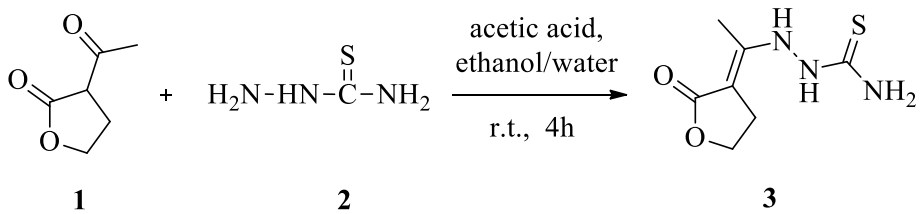


Fig. 2 Synthesis schema of (*E*)-2-(1-(2-oxodihydrofuran-3(2*H*)-ylidene)ethyl)hydrazinecarbothioamide

Table 1 Potentiometric performances of the prepared electrodes

No.	Membrane composition (*w*/*w*)						Potentiometric performance			
	PVC	Ionophore	Plasticizer			KT*p*ClPB	Linear concentration range (M)	Limit of detection (M)	Slope (mV/decade)	R^2
			o-NPOE	DEHA	BEHS					
1	31.0	4.0		64.0		1.0	1.0×10^{-2}–1.0×10^{-6}	9.35×10^{-7}	25.0 (±2.8)	0.9892
2	31.0	3.0		65.0		1.0	1.0×10^{-2}–1.0×10^{-6}	9.88×10^{-7}	26.0 (±3.8)	0.9784
3	31.0	4.0			64.0	1.0	1.0×10^{-2}–1.0×10^{-5}	9.68×10^{-6}	22.6 (±1.7)	0.9765
4	31.0	3.0			65.0	1.0	1.0×10^{-2}–1.0×10^{-6}	8.58×10^{-7}	24.7 (±2.4)	0.9855
5	31.0	2.0			66.0	1.0	1.0×10^{-1}–1.0×10^{-6}	9.71×10^{-7}	25.3 (±2.1)	0.990
6	31.0	2.0		66.0		1.0	1.0×10^{-1}–1.0×10^{-6}	6.98×10^{-7}	25.5 (±3.2)	0.9928
7	31.0	2.0	66.0			1.0	1.0×10^{-2}–1.0×10^{-5}	3.43×10^{-6}	32.6 (±3.5)	0.9738

coated with these mixtures and left to dry. Direct potentiometric measurements were taken with the prepared PVC membrane electrodes.

Results and discussion

Characterization of ionophore

Characterization of the synthesized ionophore was performed by NMR, FT–IR and Elemental analysis methods. The ^{1}H– and ^{13}C–NMR spectra of the synthesized ionophore are given in Fig. 3a–b. All spectral data are in good agreement with the expected structures.

The structure of compound **3** was explained on the basis of spectroscopic methods. In the ^{1}H–NMR spectrum, the –NH and –NH_2 protons are resonated at δ 1.62, 10.40, 9.64, 4.59 ppm as a singlet, respectively. The methylenic protons (–OCH_2CH_2–) are resonated as triplet at δ 3.43, 2.29 ppm, respectively. Addition methyl protons give a singlet at δ 2.17 ppm. In the ^{13}C–NMR spectrum, the signals of at δ 176.32 and 163.41 are belong to C=S and C=O groups. Olefinic protons resonated δ 151.40, 101.07 ppm, respectively. As expected, aliphatic protons are resonated at δ 60.23, 25.76, 11.21 ppm. All spectral data are good agreement to proposed structure.

(*E*)-2-(1-(2-oxodihydrofuran-3(2*H*)-ylidene)ethyl) hydrazinecarbothioamide

White solid, Yield: 80% M.p. 165–168 °C. **^{1}H–NMR** (400 MHz, *d*-DMSO, ppm): δ = 11.62 (s, 1H, –NH), 10.40 (s, 1H, –NH_2), 9.64 (s, 1H, –NH_2), 4.59 (s, 1H), 3.43 (t, *J* = 6.4 Hz, 2H), 2.29 (t, *J* = 6.8 Hz, 2H), 2.17 (s, 3H, –CH_3). **^{13}C–NMR** (100 MHz, *d*-DMSO, ppm): δ = 176.32, 163.41, 151.4, 101.07, 60.23, 25.76, 11.21. **FT–IR** (ATR, cm^{-1}): 3277, 3119, 1636, 1567, 817. **Anal. calc. for** $C_7H_{11}N_3O_2S$: C, 41.78; H, 5.51; N, 20.88. Found: C, 40.86; H, 5.41; N, 20.67.

Membrane composition

In this study, the ionophore properties of compound (*E*)-2-(1-(2-oxodihydrofuran-3(2*H*)-ylidene)ethyl)hydrazinecarbothioamide **(3)** were investigated. The synthesized ionophore was directly used in electrode production. The electrodes containing different amounts of plasticizer, PVC, KT*p*ClPB and ionophore in their composition were prepared, and their potentiometric performance properties were tested. KT*p*ClPB is a negatively charged ion exchanger and cation sensitive substance. The ratio of PVC and KT*p*ClPB was kept constant in all electrodes. Thus, the most ideal combination of plasticizer and ionophore was determined.

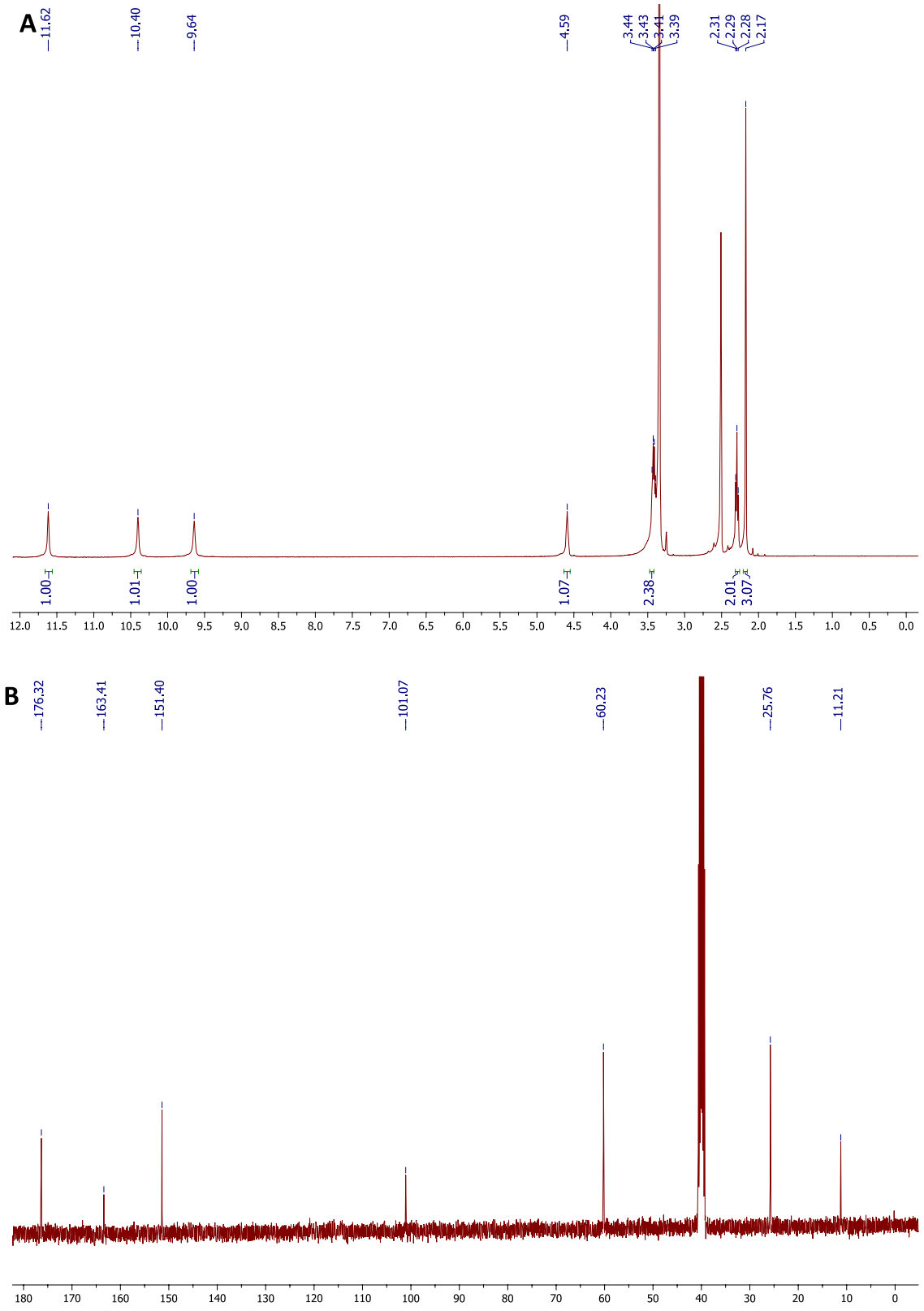


Fig. 3 **A** ^{1}H–NMR **B** ^{13}C–NMR spectrum of the synthesized ionophore

It was observed that electrodes containing lower amounts of ionophores worked in a wider linear range. Following the determination of the ionophore ratio, the appropriate plasticizer was selected. According to Table 1, it was seen that the electrodes prepared with DEHA have a wider working range and lower detection limit.

Potentiometric response, calibration curve and repeatability

The potentiometric response, calibration curve and repeatability of the prepared PVC membrane Pb(II)–selective electrode are given in Fig. 4a–c. As shown in Fig. 4a–b, the prepared electrode exhibited an ideal potentiometric response to Pb(II) ions in a wide concentration range of 1.0×10^{-1}–1.0×10^{-6} M. The electrode had a near–Nernstian behavior of 25.5 ± 3.2. Figure 4c shows the repeatability of the electrode for three different concentrations (10^{-2}, 10^{-3} and 10^{-4} M). As can be seen, the prepared electrode exhibited reproducible behavior for the three different concentration values. The response time of the prepared electrode was examined according to the rules recommended by IUPAC as shown in Fig. 4a (Buck and Lindner 1994). For this purpose, the time for the electrode to reach equilibrium was investigated at each 10-fold concentration change. As a result, it was determined that the electrode had a fast response time of 10 s.

Ionophore effect

In order to show the interaction of the ionophore used in the study with Pb(II) ions, the potentiometric response of the ionophore–free solid contact and PVC membrane electrodes to Pb(II) ions was investigated. As shown in Fig. 5, the electrodes without ionophore did not respond to Pb(II) ions. The optimum membrane (2.0% ionophore, 31.0% PVC, 66.0% DEHA and 1.0% KT*p*ClPB) containing the ionophore exhibited a linear response to Pb(II) ions.

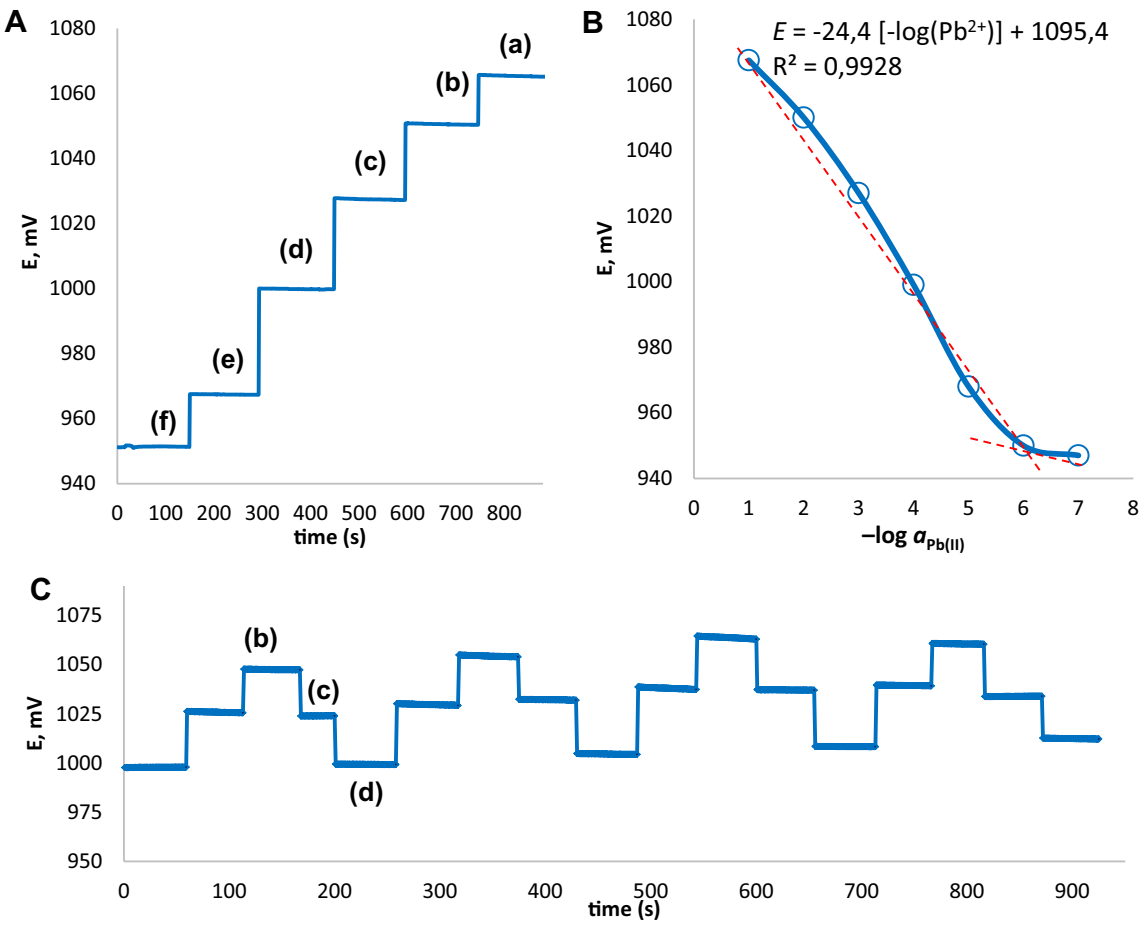


Fig. 4 **A** Potentiometric response **B** Calibration curve **C** Repeatability of lead(II)–selective electrode (**a**: 10^{-1} M, **b**: 10^{-2} M, **c**: 10^{-3} M, **d**: 10^{-4} M, **e**: 10^{-5} M, **f**: 10^{-6} M)

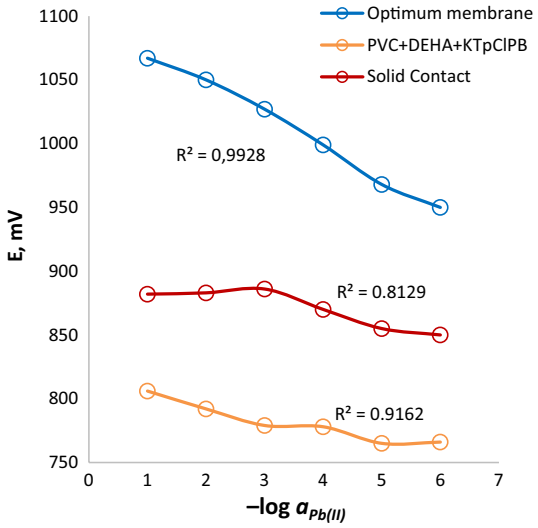


Fig. 5 Effect of ionophore on electrode behavior

Surface characterization

The surface images of the electrode with optimum components were examined by SEM (Fig. 6). Based on these SEM images, we found that the electrode surface is tight and consists of spaces suitable for ion entry.

Potentiometric selectivity

The selectivity of the ion selective electrodes is a very important parameter. Different methods for selectivity have been proposed by IUPAC (Umezawa et al. 2000). In this study, the selectivity coefficients were calculated using the separate solution method using the equation below.

$$logK_{A,B}^{pot} = \frac{(E_B - E_A)Z_A F}{RTln10} + \left(1 - \frac{Z_A}{Z_B}\right) loga_A$$

where, $K_{A,B}^{pot}$ = selectivity coefficient, a_A = activity of Pb(II), a_B = activity of interfering ion, z_A = charge of Pb(II), z_B = charge of interfering ion; R, T, and F have the usual meanings.

Selectivity coefficients for all ions were calculated using potential values corresponding to 1.0×10^{-2} M. The calculated selectivity coefficients are given in Table 2. The prepared electrode is selective towards Pb(II) ions according to the metals given in Table 2.

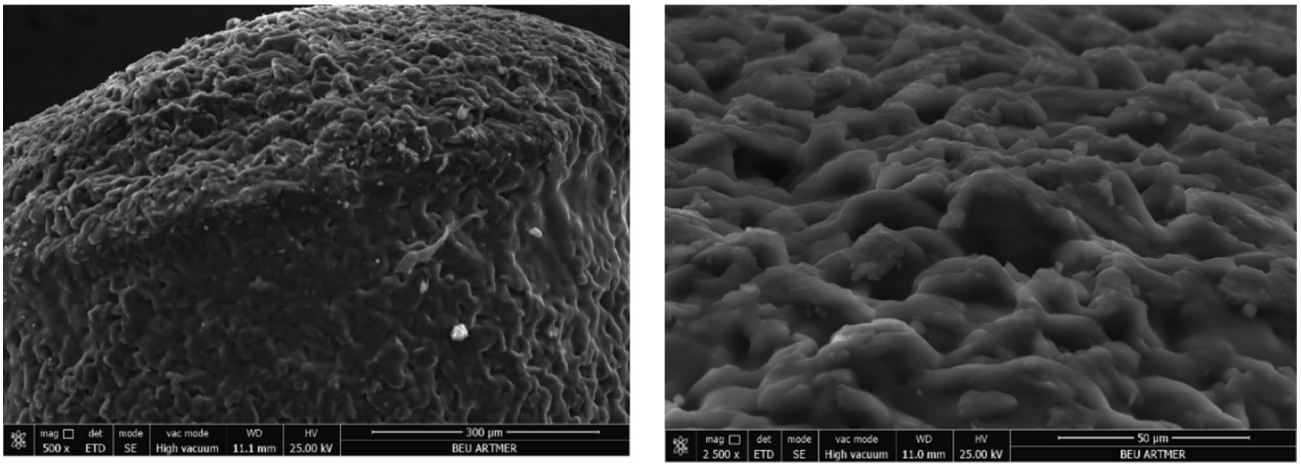


Fig. 6 SEM surface of lead(II)–selective electrode

Table 2 The calculated potentiometric selectivity coefficients

Interfering ions	Selectivity coefficient		Interfering ions	Selectivity coefficient	
	$log\ K_{Pb^{2+},M^{n+}}^{pot}$	$K_{Pb^{2+},M^{n+}}^{pot}$		$log\ K_{Pb^{2+},M^{n+}}^{pot}$	$K_{Pb^{2+},M^{n+}}^{pot}$
Cr^{3+}	−0.70	2.0×10^{-1}	Li^{+}	−2.94	1.2×10^{-3}
Cu^{2+}	−1.01	9.8×10^{-2}	Mg^{2+}	−3.31	4.9×10^{-4}
Zn^{2+}	−1.49	3.2×10^{-2}	Cd^{2+}	−3.42	3.8×10^{-4}
Ni^{2+}	−1.69	2.0×10^{-2}	Sr^{2+}	−5.37	4.3×10^{-6}
Na^{+}	−1.82	1.5×10^{-2}	Ca^{2+}	−5.39	4.1×10^{-6}

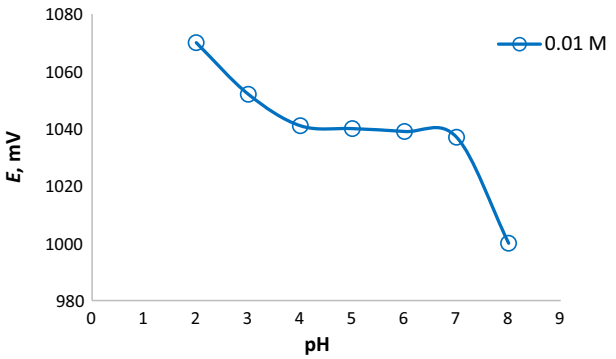


Fig. 7 pH working range of the novel lead(II)–selective electrode

Table 3 The lead(II) analysis in different water samples

Samples	Pb^{2+} quantity, (M)		
	Added Pb^{2+}	Found (±SD) with sensor*	% Recovery
Purification drinking water	1.0×10^{-2}	9.74×10^{-3}	97.4
Commercial drinking water	1.0×10^{-3}	9.12×10^{-4}	91.2
Tap water	1.0×10^{-3}	9.40×10^{-4}	94.0

*Average value ($n=3$)

pH working range

The pH working range of the developed lead(II)–selective electrode was investigated using nitric acid and sodium hydroxide solutions. 1.0×10^{-2} M Pb(II) was added to these solutions and then the potentiometric measurement was taken. The pH working range of the lead(II)–selective electrode is given in Fig. 7. As shown in this plot, the pH working range of the electrode was determined as 4.0–7.0. High mV values at pH < 4.0 may be due to hydronium ion, while low mV values at pH > 7.0 may be due to $Pb(OH)_2$ formation.

Analytical applications

The analytical applications of the prepared electrode were carried out using three different water samples. Pb(II) was added to the water samples in the amounts given in Table 3, and later potential values were measured with the prepared electrode. The obtained potential values were substituted in the linear equation in the calibration curve and the concentrations were calculated. Recovery was calculated based on added and found Pb(II) ions. As a result, we propose that the prepared electrode can detect Pb(II) ions with high recoveries.

Comparative study

Some potentiometric performance properties of this novel lead(II)–selective electrode were compared with the electrodes previously reported in the literature. When Table 4 is examined, it is clear that the prepared electrode has a wider concentration range and lower detection limit than its counterparts. It also has a faster response time and a wider pH working range than some of the electrodes shown in Table 4.

Table 4 Comparison of the novel lead(II)–selective electrode with those reported in the literature

Ionophore	Concentration range (M)	Limit of detection (M)	pH working range	Response time (s)	References
(*E*)-2-(1-(2-oxodihydrofuran-3(2*H*)-ylidene)ethyl) hydrazinecarbothioamide	1.0×10^{-6}–1.0×10^{-1}	6.98×10^{-7}	4.0–7.0	10	This work
1,4,8,11-tetrathiacyclotetradecane	1.0×10^{-5}–1.0×10^{-2}	2.2×10^{-6}	3.0–6.5	15	Elmosallamy et al. (2008)
N,N′-bis(2-hydroxy-1-napthalene)-2,6-pyridiamine	3.2×10^{-6}–1.0×10^{-1}	3.2×10^{-6}	3.5–7.5	10	Gupta et al. (2007)
poly(m-phenylenediamine) microparticles	3.16×10^{-6}–3.16×10^{-1}	6.31×10^{-6}	3.0–5.0	14	Huang et al. (2011)
acridono-crown ether	1.0×10^{-4}–1.0×10^{-2}	7.9×10^{-6}	4.0–7.0	5	Golcs et al. (2018)
PbS nanoparticles	1.0×10^{-5}–1.0×10^{-2}	Not reported	3.0–7.0	10	Song et al. (2008)
amide-linked diporphyrin xanthene	2.6×10^{-6}–1.0×10^{-1}	Not reported	4.5–7.5	< 30	Zhang et al. (2007)
(*E*)-2-(1-(4-(3-(4-chlorophenyl) ureido)phenyl)ethylidene)hydrazine carbothioamide	1.0×10^{-5}–1.0×10^{-1}	1.65×10^{-6}	5.0–11.0	5	Özbek et al. (2022b)
(*E*)-2-((1H-pyrrol-2-yl)methylene) hydrazinecarbothioamide	1.0×10^{-5}–1.0×10^{-1}	3.96×10^{-6}	5.0–9.0	5	Özbek (2022)

Conclusion

In this study, a hydrazinecarbothioamide derivative molecule **(3)**, different derivatives of which were used as sensor materials in previous studies, was used as an ionophore. The ionophore was synthesized for the first time in this study and its sensor properties were investigated. Potentiometric electrodes prepared with hydrazinecarbothioamide derivative molecule **(3)** work in a wide concentration range and have a low detection limit. The prepared electrodes have low cost, fast response time, good selectivity and stability. The results obtained from this study once again proved that hydrazinecarbothioamide derivatives have good ionophore properties. Consequently, we can state that hydrazinecarbothioamide derivatives are ideal sensor materials for ion-selective electrodes.

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Availability of data and materials Not applicable.

Declarations

Conflict of interest Authors do not have any conflicts of interest on this article.

Ethical approval The work is not applicable for both human and/or animal studies.

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