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Potentiometric PVC membrane ion–selective electrode for the determination of Sr(II) ions

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ABSTRACT

In this study, an ion–selective electrode was developed for the potentiometric determination of strontium ions. Membrane optimization studies showed that the most suitable membrane composition was 4.0% (*w/w*) synthesized ionophore, 63.0% (*w/w*) bis(2–ethylhexyl)sebacate (BEHS), 32.0% (*w/w*) poly(vinyl chloride; PVC) and 1.0% (*w/w*) potassium tetrakis(*p*–chlorophenyl)borate (KT*p*ClPB) in terms of potentiometric performance characteristics. The electrode, which exhibits a stable potentiometric behaviour, shows a linear response in the concentration range of $1.0 \times 10^{-5} - 1.0 \times 10^{-1}$ mol L^{-1} ($R^2 = 0.998$). The electrode has a detection limit of 7.94×10^{-6} mol L^{-1} and a fast response time of 7s. The electrode exhibits selectivity against Sr(II) ions in the presence of some cationic species and works without being affected by pH changes in the pH range of 6.0–9.0. The proposed electrode performs the determination of Sr(II) ions in water samples with high recoveries.

1. Introduction

Strontium is an important alkaline earth metal with high reactivity [1]. Strontium is used in the production of plastics, paints, bricks, glasses for colour television sets and some other glass materials, electrolytic zinc and also in energy storage [2,3]. Strontium, which is widely found in the environment, is also present in the diverse sites in human body, including bones and teeth. It plays an important role in biological systems, but its excess can cause a variety of physiological problems [4]. Some oxides, hydroxides and carbonates of strontium have a strong irritating effect on the skin and eyes [5]. The determination of strontium has been carried out using different analytical methods such as flame atomic absorption spectrometry [6], inductively coupled plasma atomic emission spectrometry [7] and spectrophotometric [8]. These methods provide highly accurate detection, but they are highly costly and time consuming, involve complex procedures and require sample pre-treatment and experienced personnel. Thus, ion–selective electrodes (ISEs) may be preferred as alternatives to these analytical methods. ISEs provide important advantages such as low cost, high selectivity, low detection limit, wide linear range, fast response time and ease of use [9–11]. Therefore, they are frequently used for the routine determination of various ions in different samples [12–15].

The polymer structure not only provides mechanical stability to the membrane, but also provides features such as biological compatibility and adhesion. Although there are polymers with these properties, PVC is preferred because it can easily trap the membrane system in the majority of ion–selective electrodes [16,17].

Ionophores, which are in the composition of ISEs and interact directly with the analyte ion, are very important in the production of electrodes. Today, commercially available ionophores have been replaced by organic molecules that can be synthesized more economically in the laboratory [18]. In the present work, a carbothioamide derivative molecule was synthesized, and PVC membrane ion–selective electrode was prepared. The potentiometric performance properties of this electrode, which was proposed for the determination of strontium ions, were investigated.

2. Experimental

2.1. Instruments

The characterization studies of the synthesized ionophore were performed using ^{1}H– and ^{13}C– NMR (Bruker Avance DPX–400 instrument), elemental analysis (LECO CHNS 932) and melting points (Electrothermal

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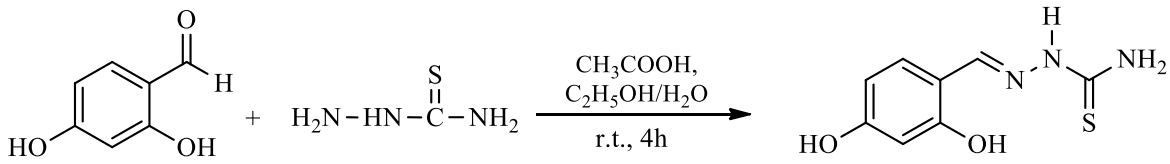

Fig. 1. Synthesis schema of ionophore.

Table 1
Potentiometric performance characteristics of prepared electrodes.

No	Membrane composition (*w/w*)						Potentiometric performance			
	PVC	Ionophore	Plasticizer			KT*p*ClPB				
			o–NPOE	DEHA	BEHS		Linear concentration range (mol L^{-1})	Limit of detection (mol L^{-1})	Slope (mV dec^{-1})	R^2
1	32.0	4.0			63.0	1.0	1.0×10^{-1}–1.0×10^{-5}	7.94×10^{-6}	23.0 (±2.2)	0.9980
2	32.0	4.0		63.0		1.0	1.0×10^{-1}–1.0×10^{-5}	9.12×10^{-6}	18.3 (±2.4)	0.9895
3	32.0	4.0	63.0			1.0	1.0×10^{-2}–1.0×10^{-5}	1.02×10^{-5}	15.8 (±1.8)	0.9679
4	32.0	3.0			64.0	1.0	1.0×10^{-1}–1.0×10^{-5}	1.18×10^{-5}	15.6 (±1.9)	0.9873
5	32.0	5.0			62.0	1.0	1.0×10^{-1}–1.0×10^{-5}	9.07×10^{-6}	20.0 (±2.0)	0.9882

9100 apparatus) measurement devices. Potentiometric measurements were performed using on a computer–controlled multi–channel potentiometric system (Medisen Medical Ltd. Sti., Turkey). Ag/AgCl reference electrode (Orion, Thermo Scientific no 900100) was used in this measurement system. pH measurements were performed with a digital pH meter (Mettler Toledo Model S220–K).

2.2. Reagents

All reagents used in the ionophore synthesis were obtained from Sigma Aldrich (Germany). High molecular weight PVC, BEHS, bis(2–ethylhexyl)adipate (DEHA), *o*–nitrophenyloctyl ether (*o*–NPOE), tetrahydrofuran (THF), nitric acid (HNO_3), sodium hydroxide (NaOH), KT*p*ClPB and nitrate salts of the cations used in selectivity were purchased from Sigma Aldrich (Germany). Graphite, epoxy (Macroplast Su 2227) and hardener (Desmodur RFE) used in the preparation of conductive solid contact electrodes were obtained from Sigma Aldrich (Germany), Henkel (Istanbul, Turkey), Bayer AG (Darmstadt, Germany), respectively.

2.3. Method

2.3.1. Synthesis of ionophore

The method previously reported in the literature was used for the ionophore synthesis [19]. In brief, the 2,4-dihydroxybenzaldehyde (1 mmol) was dissolved in warm ethanol (15 mL). Thiosemicarbazide (1 mmol) was dissolved in warm water (15 mL) and then added to this solution. Acetic acid (five drops) were subsequently added to the resulting mixture. The reaction was stirred magnetically for 4 h at room conditions. The precipitate formed at the end of the reaction was filtered off. After washing with ethanol several times, ionophore was ultimately synthesized (Fig. 1).

(*E*)-2-(2,4-dihydroxybenzylidene)hydrazinecarbothioamide: White solid. Yield 92%. M.p. 195–197 °C.^{1}H NMR (400 MHz, δ, ppm, DMSO-*d6*): δ 11.18 (s, 1H, –NHCS), 9.76 (s, 2H, –OH), 8.25 (s, 1H, –N═CH), 7.96 (s, 1H, –NH), 7.75 (s, 1H, –NH), 7.68 (d, J = 8.0 Hz, 1H, –Ar–H), 6.30–6.25 (m, 2H, Ar–H); ^{13}C NMR (101 MHz, δ, ppm, DMSO–*d6*): 177.52, 160.92, 158.46, 141.24, 128.81, 112.25, 108.22, 102.75. Anal. calc. for $C_8H_9N_3O_2S$: C, 45.49; H, 4.29; N, 19.89. Found: C, 46.01; H, 4.21; N, 19.70.

2.3.2. Preparation of polymeric membrane electrodes

The electrodes were prepared by the method we previously reported [20,21]. First, open–ended copper wires were coated with a solid contact mixture of graphite (50.0%), epoxy (35.0%), and hardener (15.0%) dissolved in THF (~3 mL). Then, PVC membrane mixtures with different components were prepared using various plasticizers (BEHS, *o*–NPOE and DEHA), ionophore and KT*p*ClPB (Table 1). The electrodes were left to dry for 24 h. Finally, the pre–prepared solid contact electrodes were coated with a PVC membrane mixture and the electrodes were made ready for the potentiometric measurement [22].

2.3.3. Preparation of water samples

Strontium(II) analysis was performed in three different water samples (purification drinking water, commercial drinking water and tap water), which did not contain strontium(II), according to the standard addition method. Strontium(II) was added to the water samples at known concentrations and the water samples were made ready for potentiometric measurement.

2.3.4. Potential measurements

Potentials were measured using an Ag/AgCl reference electrode. All potential studies were carried out at 25 ± 1.0 °C temperature by using the following cell assembly:

Ag/AgCl; KCl (saturated) $\|Sr^{2+}$ sample solution|PVC membrane|conductive solid contact|Cu wire.

3. Results and discussion

3.1. Characterization of the ionophore

In the ^{1}H–NMR spectrum of ionophore (Fig. 2a), –NH proton resonated as a singlet at 11.18 ppm. The –OH protons were observed as a singlet at 9.76 ppm. The –N═CH proton resonated as a singlet at 8.25 ppm. The amine protons resonated at 7.96 and 7.76 ppm. Aromatic protons in the benzene ring were observed as dublet at 7.68 ppm and multiplet at 6.30–6.25 ppm. In the ^{13}C–NMR spectrum of the synthesized molecule (Fig. 2b), the peaks of –C═S and –C═N carbons were observed at 177.52 ppm and 160.92 ppm, respectively. The peaks of –C–OH carbons in the benzene ring were observed at 158.46 and 141.24 ppm. In addition, the peaks at 128.81, 112.25, 108.22 and 102.75 ppm belong to the other carbons in the benzene ring. The spectroscopic data was found to be identical to the data reported in the literature [23].

3.2. Membrane optimization

The potentiometric performance of an ISE is directly related to the prepared membrane mixture and the proportions of the components in

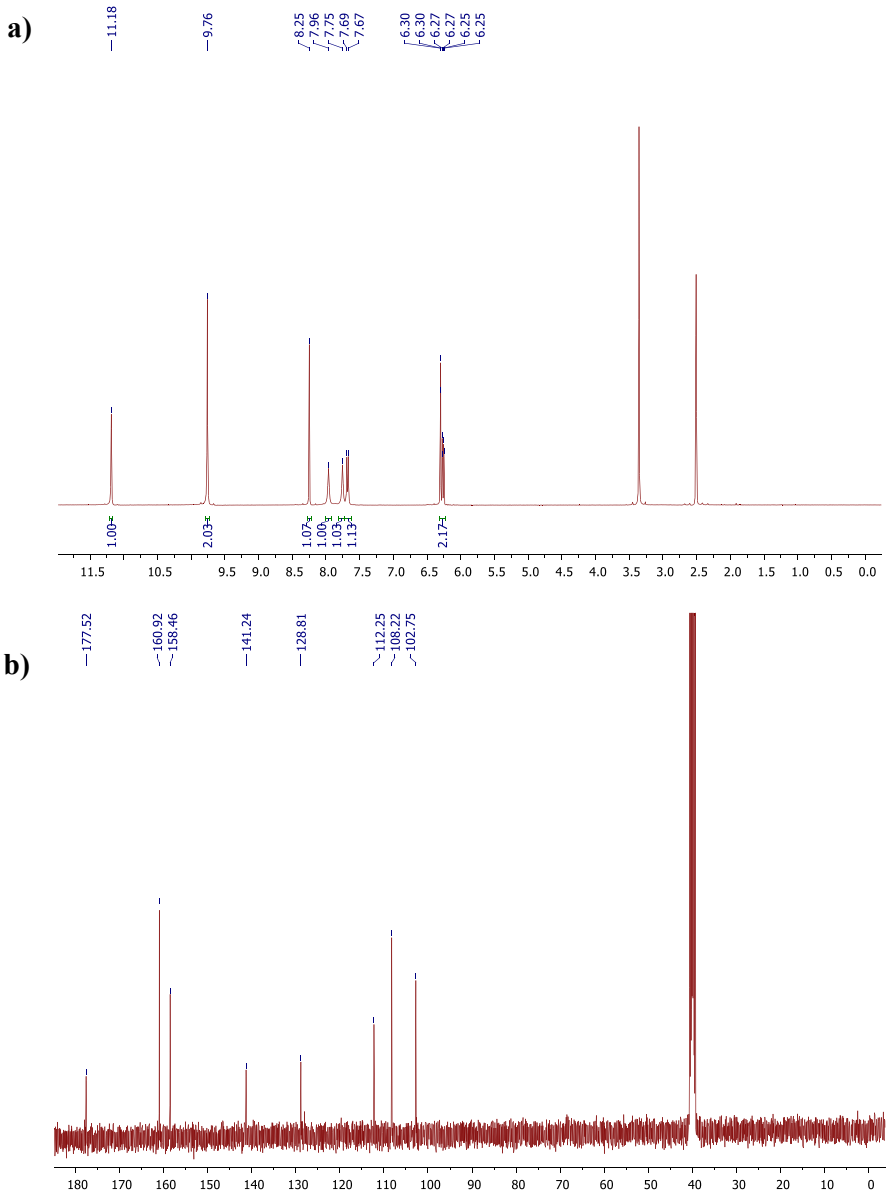

Fig. 2. a) ^{1}H NMR **b)** ^{13}C NMR spectrum of synthesized ionophore.

this mixture. Undoubtedly, ionophores in polymeric membranes are the most important components that provide direct interaction with the analyte and show selectivity towards a certain ionic species. Therefore, the ratios of the membrane components were investigated to obtain the electrode composition with the best potentiometric performance. Firstly, the ratios of ionophore, PVC and KT*p*ClPB were kept constant to see the plasticizer effect. Therefore, the most suitable plasticizer was selected for the membrane mixture. When the data in Fig. 3 and Table 1 are evaluated, it is seen that BEHS, one of the plasticizers used for the determination of Sr^{2+} ions, is more suitable. The electrode prepared with BEHS has a lower limit of detection value and a more sensitive potentiometric behaviour. Then, PVC, BEHS and KT*p*ClPB were kept constant and ionophore ratios were changed. Thus, the ideal ionophore ratio was determined. The calibration curves of the prepared electrodes are given in Fig. 4. As seen in Table 1, Electrode I (32.0% PVC, 4.0% ionophore, 63.0% BEHS and 1.0% KT*p*ClPB) is superior to other prepared PVC membrane electrodes in terms of detection limit, correlation coefficient (R^2) and slope.

The proposed interaction mechanism between ionophore and strontium(II) ions is shown in Fig. 5.

3.3. Potentiometric response, calibration curve, repeatability and response time

In this study, all potentiometric measurements were performed using the Electrode I. The linear working range of the electrode was determined using Sr^{2+} solutions. The potentiometric behaviour of the electrode are shown in Fig. 6, and the calibration curve is shown in Fig. 7. According to the obtained potentiometric measurements, the proposed electrode exhibited a linear response to Sr^{2+} ions in the concentration range of $1.0 \times 10^{-5} - 1.0 \times 10^{-1}$ mol L^{-1} ($R^2 = 0.9980$) with a slope of 23.0 ± 2.2 mV/decade. The detection limit of the proposed electrode was calculated using the calibration curve in Fig. 7 according to IUPAC recommendations [24]. The detection limit was determined as 7.94×10^{-6} mol L^{-1} using the potential value obtained from the intersection of two extrapolated regions of the calibration curve. The repeatability of the electrode was tested using solutions of three different concentrations. The obtained potentiometric measurements are shown in Fig. 8. As can be seen in this figure, the electrode exhibited a reproducible response to 1.0×10^{-2}, 1.0×10^{-3} and 1.0×10^{-4} mol L^{-1} Sr^{2+} ion solutions.

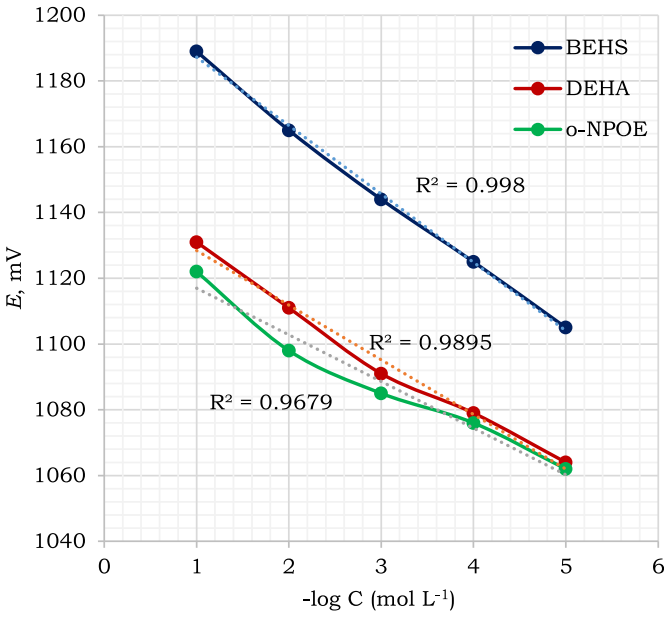

Fig. 3. Plasticizers effect on electrode behaviour.

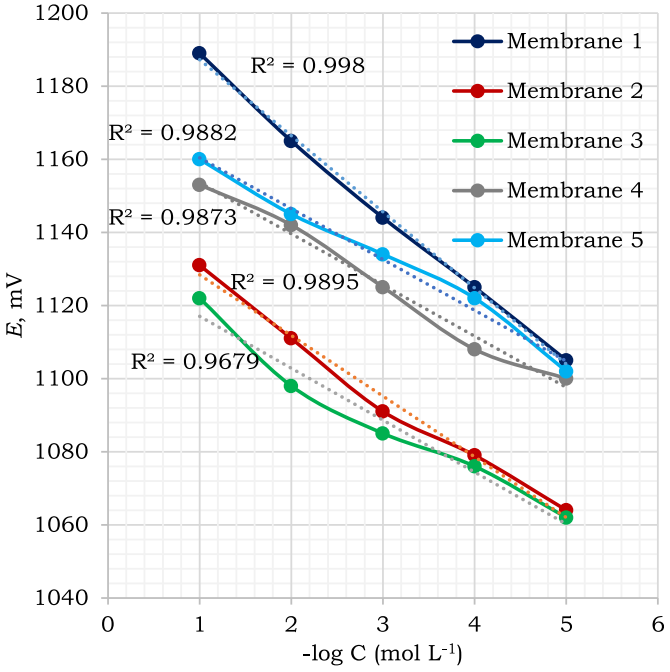

Fig. 4. Potential response of the prepared electrodes.

In this study, the response time was determined according to the IUPAC recommendations [25], and the mean time for the potentials to reach steady value as concentration changes was tested. As a result of the potentiometric measurements, it was determined that the developed electrode has a fast response time of just 7s.

3.4. Selectivity

Selectivity is one of the most important properties of ion–selective electrodes. The mathematical expression of the selectivity of an ion–selective electrode towards the main ion in the presence of foreign ions is determined by the selectivity coefficients. The potentiometric selectivity coefficients of the electrode for Sr^{2+} ions against some cationic

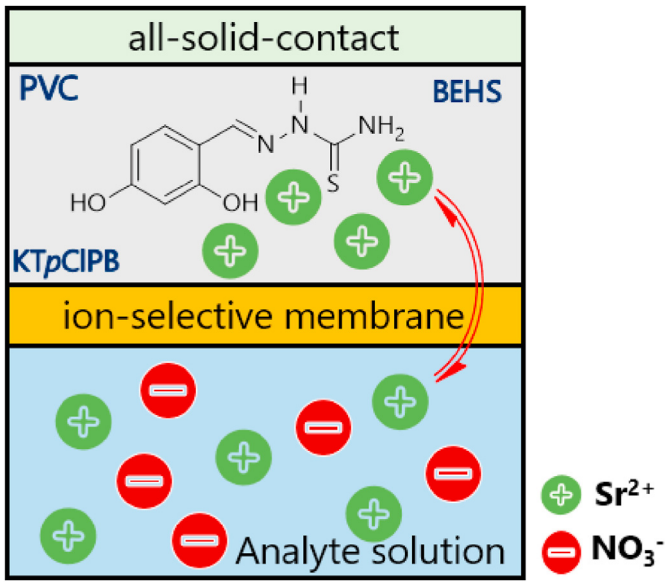

Fig. 5. Mechanism of interaction between ionophore and Sr^{2+} ions.

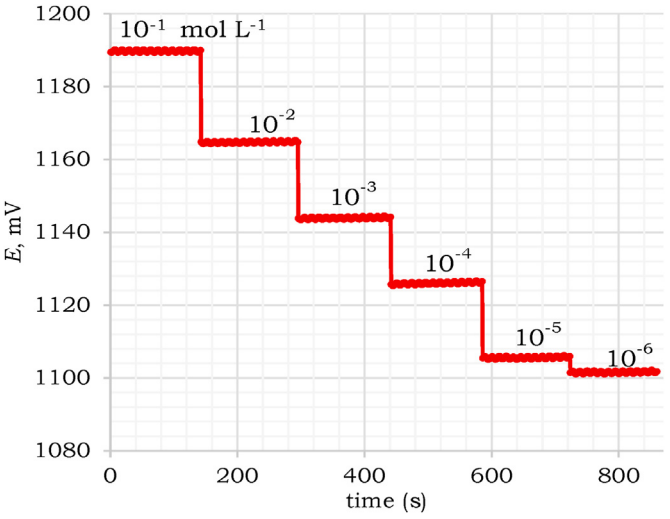

Fig. 6. The potentiometric response of the electrode to Sr^{2+} solutions.

species were calculated using the separate solution method (SSM) recommended by IUPAC [25], and the selectivity coefficients for these cations are shown in Table 2. If an electrode has equivalent responses to two ions, then $K_{A,B}^{pot} = 1.0$. The smaller the $K_{A,B}^{pot}$ values, the less impact the interfering ion will have on the measured potential. When $K_{A,B}^{pot}$ values are <1.0, the electrode is more responsive to the primary ion and when $K_{A,B}^{pot}$ values are >1.0, the electrode is more responsive to the foreign ions [26]. According to the selectivity coefficients given in Table 2, it can be stated that the proposed electrode is sufficiently selective towards strontium ions in the presence of various interfering ions.

3.5. pH effect

It is an important advantage that the ISEs remain unaffected by the pH of the medium in a wide pH range. In this study, the pH working range of the proposed electrode was tested in the pH 2.0–12.0 range using 1.0×10^{-2} mol L^{-1} Sr(II) solutions. The pH was adjusted using HNO_3 (pH: 2.0–7.0) and NaOH (pH: 8.0–12.0). The change of electrode potential depending on the pH change is shown in Fig. 9. When Fig. 9 is examined,

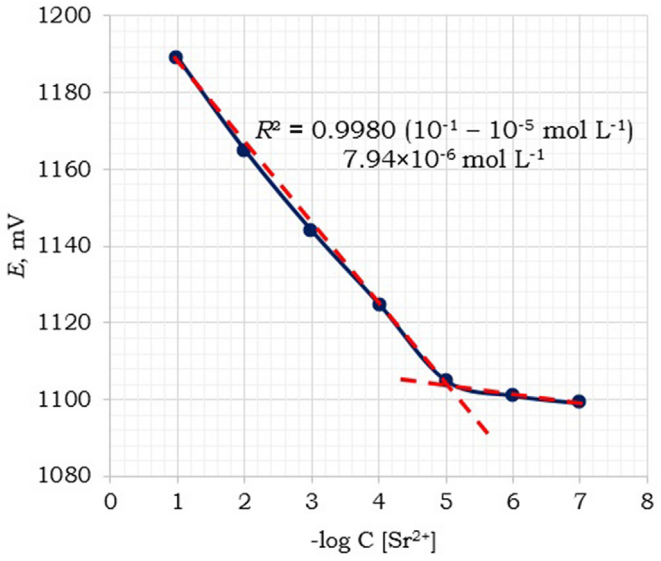

Fig. 7. The calibration curve of the electrode.

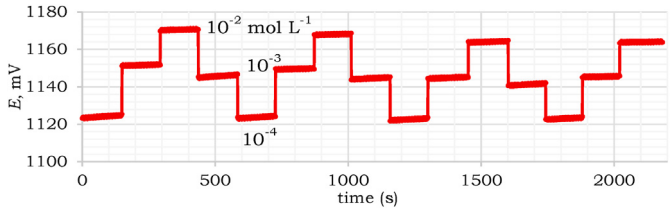

Fig. 8. The repeatability of the proposed electrode.

Table 2
The potentiometric selectivity coefficients of the developed electrode.

Interfering ions	Selectivity coefficient, $K^{pot}_{Sr^{2+},M^{n+}}$	Interfering ions	Selectivity coefficient, $K^{pot}_{Sr^{2+},M^{n+}}$
Cd^{2+}	8.9×10^{-2}	Cu^{2+}	3.7×10^{-2}
Co^{2+}	7.9×10^{-2}	Na^{+}	3.1×10^{-2}
Ni^{2+}	5.6×10^{-2}	Ca^{2+}	1.4×10^{-2}
Zn^{2+}	4.0×10^{-2}	Mg^{2+}	8.7×10^{-3}
K^{+}	3.8×10^{-2}		

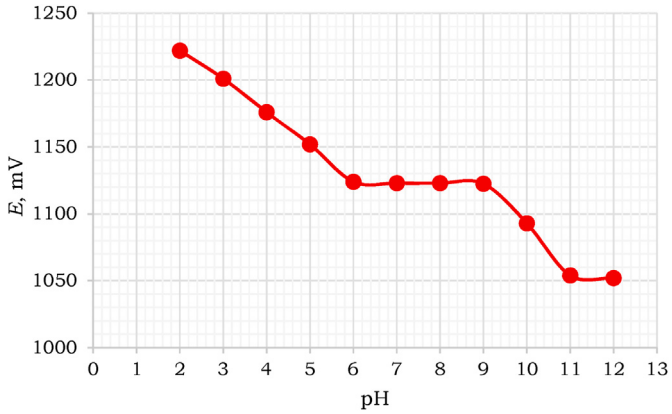

Fig. 9. Effect of pH on the potentiometric response of the electrode.

it can be observed that the potentiometric response of the proposed electrode in the pH range of 6.0–9.0 is not affected by the pH change.

Table 3
The strontium analysis in different water samples.

	Sr^{2+} quantity, (mol L^{-1})		
	Added Sr^{2+}	Mean (± SD) found with sensor[a]	% Recovery
Purification drinking water	1.00×10^{-3}	$9.74\ (\pm 0.2) \times 10^{-4}$	97.4
Commercial drinking water	1.00×10^{-3}	$9.66\ (\pm 0.1) \times 10^{-4}$	96.6
Tap water	1.00×10^{-3}	$8.91\ (\pm 0.7) \times 10^{-4}$	89.1

[a] Average value ($n = 3$).

Table 4
Potentiometric response characteristics of the proposed electrode.

Properties	Values/Range
Electrode type	PVC membrane (solid contact)
Optimized membrane composition	32.0% PVC, 4.0% ionophore, 63.0% BEHS and 1.0% KT*p*ClPB
Linear range (mol L^{-1})	$1.0 \times 10^{-5} - 1.0 \times 10^{-1}$
R^2	0.998
Slope (mV/decade)	23.0 ± 2.2
Detection limit (mol L^{-1})	7.94×10^{-6}
Response time (s)	7
pH range	6.0–9.0

Table 5
Comparison with reported strontium(II)–selective electrodes.

No	linear concentration range (mol L^{-1})	limit of detection (mol L^{-1})	pH working range	response time (s)	Ref.
1	$3.2 \times 10^{-5} - 1.0 \times 10^{-1}$	Not reported	3.0–10.0	10	[27]
2	$1.0 \times 10^{-5} - 1.0 \times 10^{-1}$	4.0×10^{-6}	3.0–10.6	<10	[28]
3	$9.0 \times 10^{-6} - 1.0 \times 10^{-1}$	5.0×10^{-6}	3.0–10.0	<10	[29]
4	$1.6 \times 10^{-6} - 3.0 \times 10^{-3}$	6.3×10^{-7}	4.3–9.4	<15	[30]
5	$1.9 \times 10^{-5} - 1.0 \times 10^{-1}$	Not reported	3.0–10.0	15	[31]
6	$1.0 \times 10^{-5} - 1.0$	1.0×10^{-5}	4.0–7.0	15	[32]
7	$1.0 \times 10^{-5} - 1.0 \times 10^{-1}$	7.94×10^{-6}	6.0–9.0	7	This work

There are potential changes in pH values (pH < 6.0 and pH > 9.0) outside of this range. The high potential at low pH (<6.0) values may be due to the response of the electrode to the proton in the medium, and the low potential at high pH (>9.0) values may be due to the interaction of Sr^{2+} ions with the hydroxide to form $Sr(OH)_2$.

3.6. Analytical applications

In order to demonstrate the analytical applicability of the developed electrode, Sr(II) was added to the water samples according to the standard addition method. Direct potentiometric measurements were performed in the prepared water samples. The Sr(II) values added to the water samples and those found with the sensor are given in Table 3. Based on the results obtained, it can be said that the proposed electrode can perform Sr(II) analysis on various samples.

3.7. Potentiometric response characteristics and comparison study

The characteristic performance of the proposed electrode is given in Table 4 and, its comparison with other potentiometric electrodes in the literature is given in Table 5. The electrode is better than the earlier

reported strontium(II) ion–selective electrodes regarding linear working concentration range [27,30,31] (Table 5). Also, it exhibits fast response time than the electrodes given in Table 5.

4. Conclusions

Potentiometric ISEs have successfully performed the detection of ionic species in various samples for a long time. In particular, their cost–effective, portable, selective and sensitive nature has made them the center of attention, and they have been widely used in routine analyses [33,34]. In this study, a novel potentiometric electrode is proposed to be used for the detection of strontium ions. Electroactive material was synthesized in the laboratory and a lower cost electrode was produced. This novel electrode, which can be an alternative to other analytical methods in the determination of strontium, has a wide linear working range, fast response time and stable potentiometric behaviour.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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