



Novel Solid Contact Ion Selective Sensor for Potentiometric Analysis of Barium Ions

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Abstract: Barium as an alkaline earth metal is widely used in multiple industries. Due to its widespread use, it is toxic to living things in high concentrations. In this study, polymer membrane barium-selective potentiometric sensors were prepared in which 4-aminobenzoic acid 2-diethylaminoethyl ester was used as an ionophore. The prepared sensors exhibited selective and stable behavior towards barium ions. The novel barium-selective potentiometric sensor had a low detection limit of 4.23×10^{-6} M over a wide linear concentration range from 1.0×10^{-5} to 1.0×10^{-1} M. The sensor proposed in the present study had good repeatability, short response time (8s) and could work over a wide range of pH. The sensors prepared simply and economically were able to determine barium ions in various samples with very high recoveries.

Keywords: Barium, sensor, potentiometry, ion-selective electrodes.

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1. INTRODUCTION

Barium occurs naturally in the food and groundwater, and its water-soluble salts are toxic (Kravchenko et al., 2014; Gad, 2014). Barium is commonly used in various industrial sectors such as the petroleum industry, electroplating, alloying, glass industry, and also in pigments and corrosion protection films (Grajeda et al., 2017; Modi et al., 2014). Due to its widespread use in industry, barium pollutes both soil and water. High concentrations of barium can be toxic to living things (Oliveira et al., 2012; Llugany et al., 2000). Especially in humans, it can cause a number of health problems such as respiratory failure, hypophosphatemia, loss of reflexes and rhabdomyolysis (Altunoluk et al., 2024). To date, inductively coupled plasma mass spectrometry (ICP-MS), atomic absorption spectroscopy (AAS) and spectroscopic methods have been proposed for the determination of barium in various samples (Ardianrama et al., 2020; Bisergaeva et al., 2020; Huelga-Suarez et al., 2013). These methods, which are not particularly suitable for on-site analysis, are complicated to use, time-consuming, costly, and require experienced personnel and a well-equipped laboratory (Özbek, 2023; Abu Shawish et al., 2020).

Potentiometric methodologies, which have a highly important place in electrochemistry, are a subject intensively studied by sensor researchers (Berkel and Özbek, 2024). Potentiometry is an electrochemical method in which the potential of an electrode system consisting of reference and working electrodes is measured against time (Isildak and Özbek, 2021). Potentiometric ion-selective electrodes (ISEs) have important advantages such as easy use, short response time, low limit of detection, wide range of concentration, low cost, low-energy consumption, good selectivity and repeatability (Abu Shawish et al., 2016; Özbek et al., 2024a; Wu et al., 2023; Verma et al., 2023). ISEs are applied in many fields, for instance in the analysis of food, pharmaceutical, agriculture, medicine and environmental samples (Özbek and Isildak, 2022; Özbek and Altunoluk 2024). ISEs can be prepared using a wide variety of materials (Özbek et al., 2022a).

Ionophores are molecules that are both commercially available and synthesized. These molecules are the most important sensor material that can selectively interact with the analyte (Özbek et al., 2022a; Ören et al., 2024). Ionophores are undoubtedly the most critical components that influence the electrode's selectivity towards a single ion as well as other commonly used ions. 3-deoxy-D-erythro-hexos-2-

ulose bis(thiosemicarbazone) (Zamani et al., 2010), 2,3,4-pyridine-1,3,5,7,12-pentaazacyclopentadeca-3-ene (Singh et al., 2005), 4-4'-methylene-diantipyrine (Abedi et al., 2008), and organophosphorus compounds (Saleh, 2000) have been previously used as ionophores in the selective determination of barium ions as reported in the literature. In the current study, we developed new ion-selective sensors that exhibit high selectivity to barium ions by using the 4-aminobenzoic acid 2-diethylaminoethyl ester (Figure 1) molecule as an ionophore. 4-aminobenzoic acid 2-diethylaminoethyl is also a local anesthetic drug (Özbek and Altunoluk, 2024). This study aimed to investigate the ionophore properties of its chemical form.

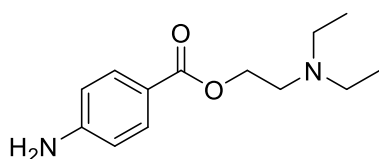


Figure 1. Chemical structure of the ionophore.

2. EXPERIMENTAL SECTION

2.1. Chemicals and Apparatus

Graphite, high molecular weight poly(vinyl chloride) (PVC), 4-aminobenzoic acid 2-diethylaminoethyl ester (ionophore; $\geq 97\%$), plasticizers, anion excluder, tetrahydrofuran (THF; $\geq 99.9\%$), sodium hydroxide (NaOH; $\geq 98.0\%$), nitric acid (HNO₃) and

metal nitrate salts were of the greatest analytical purity and were obtained from Sigma-Aldrich. All potential measurements were made with an Ag/AgCl reference electrode (Thermo Scientific) connected to a multi-channel potentiometer (Medisen Medical Ltd. Sti., Turkey) and the prepared sensors.

2.2. Method

In this study, the preparation of solid contact PVC membrane barium-selective sensors were performed in two stages, as in our previous studies (Özbek et al., 2024b; Özbek et al., 2022b). Initially, conductive solid contact mixtures were prepared by dissolving 50.0 mg graphite, 35.0 mg epoxy and 15.0 mg hardener in approximately 3 mL THF. Copper wires then were dipped into the homogeneous solid contact mixture 3–4 times and subsequently left to dry in the dark for 24 h. Then, polymer membrane mixtures were prepared by dissolving them in the solution of THF with the compositions in Table 1. Mixtures containing ionophore, PVC, plasticizer and anion excluder were dissolved in approximately 3 mL of THF. After a homogeneous mixture was provided, the mixtures reaching a certain viscosity were coated on the surface of previously prepared solid contact electrodes. The surface of pre-prepared conductive solid contact electrodes were covered with these mixtures and allowed to dry. Potentiometric measurements were conducted using barium ion solutions with the prepared sensors. The preparation steps and potentiometric measurement system of the new barium-selective sensors were summarized in Figure 2.

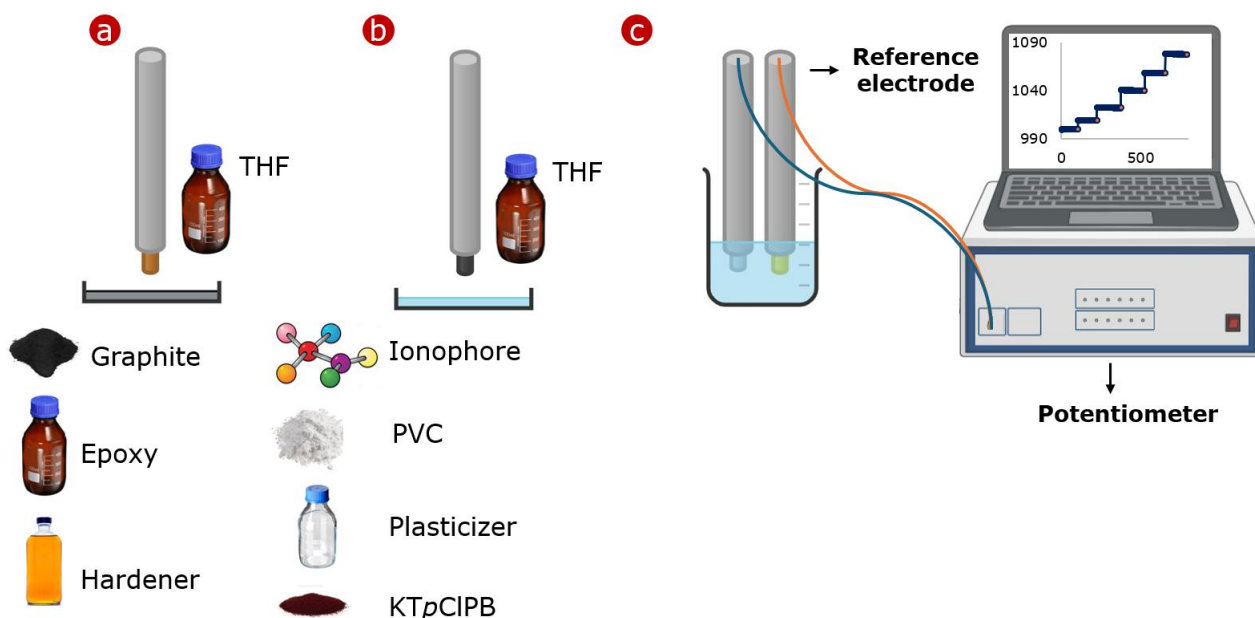


Figure 2. Preparation stages of barium-selective sensors.

3. RESULTS AND DISCUSSION

In the current study, sensors with a total of seven different compositions were designed using 3 different plasticizers (namely, dibutyl phthalate (DBP), bis(2-ethylhexyl)sebacate (BEHS) and *o*-

nitrophenyloctyl ether (*o*-NPOE)). Plasticizers reduce the viscosity of the polymer matrix and provide mobility of the components in the membrane structure. In the literature, the most commonly used composition of PVC membrane electrodes was reported as 1–7% ionophore, 28–33% PVC, 60–69%

plasticizer and 0.03–10% anion excluder (Erol et al., 2009). The potentiometric performance characteristics of the sensors prepared in these ranges were investigated using Ba²⁺ ion solutions. Barium ion solutions were prepared with Ba(NO₃)₂. The potentiometric performance features of the prepared sensors were given in Table 1. According to the obtained data, mixture number VII among the

prepared sensors had the lowest detection limit (4.23×10⁻⁶ M) in a wide concentration range of 1.0×10⁻⁵–1.0×10⁻¹ M. When the potentiometric performance properties of the prepared sensors were evaluated, the most ideal composition was determined to be 3.0% ionophore, 32.0% PVC, 64.0% BEHS and 1.0% KTpCIPB.

Table 1. Certain performance features of the prepared barium–selective potentiometric sensors.

No	Sensor composition (w/w)					Potentiometric performance			
	PVC	Ionophore	Plasticizers			Linear Range, M	Detection Limit, M	Slope (mV/decade)	R ²
		BEHS	DBP	O-NPOE	KTpCIPB				
1	32.0	3.0	64.0		1.0	1.0×10 ⁻⁵ –1.0×10 ⁻³	1.08×10 ⁻⁵	15.5 (±2.2)	0.9859
2	32.0	3.0		64.0	1.0	1.0×10 ⁻⁵ –1.0×10 ⁻³	9.22×10 ⁻⁶	10.1 (±1.5)	0.9860
3	32.0	4.0	63.0		1.0	1.0×10 ⁻⁵ –1.0×10 ⁻¹	9.97×10 ⁻⁶	11.0 (±1.1)	0.9866
4	32.0	5.0	62.0		1.0	1.0×10 ⁻⁴ –1.0×10 ⁻¹	1.0×10 ⁻⁴	21.0 (±1.2)	0.9779
5	32.0	4.0		63.0	1.0	1.0×10 ⁻⁴ –1.0×10 ⁻¹	9.77×10 ⁻⁵	16.5 (±2.5)	0.9518
6	32.0	5.0		62.0	1.0	1.0×10 ⁻⁴ –1.0×10 ⁻¹	2.15×10 ⁻⁵	24.0 (±3.4)	0.9825
7	32.0	3.0	64.0		1.0	1.0×10 ⁻⁵ –1.0×10 ⁻¹	4.23×10 ⁻⁶	19.0 (±0.2)	0.9995

In this study, the behavior of sensors with and without ionophores in their composition against barium ions was tested in order to determine the influence of the ionophore used on the behavior of the sensor. For this purpose, the behavior of solid contact, without any ionophore (BEHS + PVC + KTpCIPB) and optimum (Sensor VII) electrodes

against barium ion solutions is presented in Figure 3. Based on this data, electrodes without ionophores in their composition did not give a linear response to barium ions. Consequently, it can be stated that the ionophore in the proposed sensor composition is able to interact with barium ions.

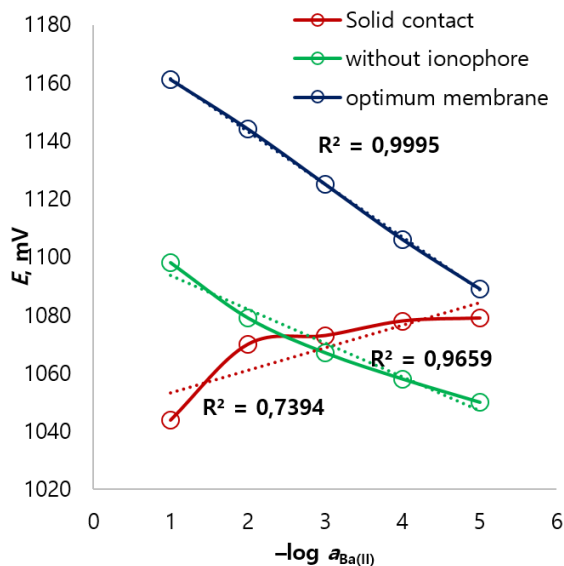


Figure 3. Response of electrodes with and without ionophore to barium ions.

The potential (mV)–time (s) behavior of this novel barium–selective sensor was presented in Figure 4a, which shows that the sensor displays a linear response in the concentration range from 1.0×10⁻⁵ to 1.0×10⁻¹ M. The detection limit of detection (LOD) the prepared sensor was calculated using the calibration curve in Figure 4b. The detection limit of the sensor

was calculated considering the recommendations reported by IUPAC (Buck et al., 1994). The potential value corresponding to the point where the horizontal and vertical axes intersect in Figure 3b was written in its place in the linear equation. The detection limit of the proposed sensor was calculated as 4.23×10⁻⁶ M. The repeatability performance of the prepared novel

barium-selective potentiometric sensor was tested with barium ion solutions in three different concentrations (namely, 10^{-1} , 10^{-2} and 10^{-3} M). Figure 4c shows that the sensor demonstrates repeatable and stable performance for three different concentration values. The results given in Figure 4

(a-c) also indicates that the proposed sensor has a near-Nernstian response in addition to a low detection limit over a wide concentration range. Additionally, the sensor has a repeatable and stable behavior.

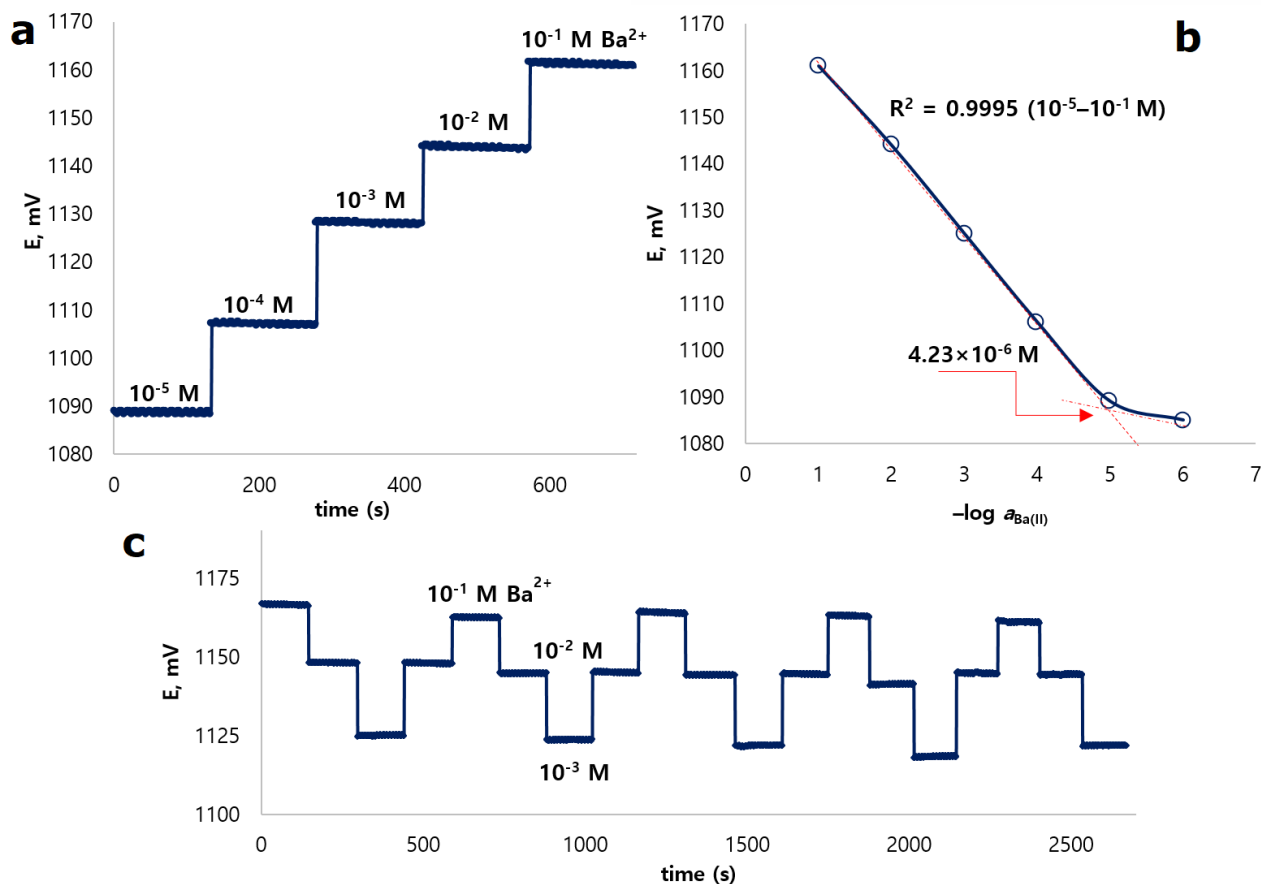


Figure 4. a-) The potentiometric response (E (mV)–time (s)), b-) calibration curve c-) repeatability of barium-selective sensor.

The response time of the newly developed barium-selective potentiometric sensor was investigated according to the guidelines recommended by IUPAC (Buck et al., 1994). For this purpose, the equilibrium time of the sensor from one concentration to another

was determined. The sensor developed according to Figure 5 reaches equilibrium in approximately 8 seconds for a 10-fold concentration change and has a very fast response time.

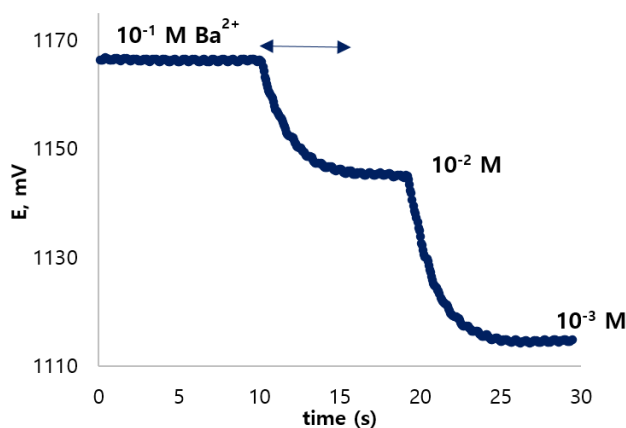


Figure 5. The dynamic response time of the sensor developed in the present study.

One of the most essential characterization parameters of ion-selective electrodes is considered

to be their selectivity. Selectivity is the behavior of a sensor towards a single species in the presence of

diverse ionic species. In this study, ten different cationic species were used to test the selectivity of the prepared sensor against barium ions. Figure 6 indicates the response of the barium-selective potentiometric sensor to barium ions relative to other ions. Based on this data, the prepared sensor exhibited the highest potential values against barium ions. Selectivity coefficients ($K_{A,B}^{pot}$) were calculated to mathematically express the selectivity of the ions used in the investigation of selectivity. Selectivity coefficients for different ions were calculated based on the separate solution method (SSM) defined by IUPAC (Umezawa et al., 2000). For this purpose, using the data obtained from potentiometric measurements, the potential value of every ion at the concentration of 1.0×10^{-2} M was written into the equation recommended by IUPAC, and the selectivity coefficients were then calculated for the ions in Table 2.

$$\log K_{A,B}^{pot} = \frac{(E_B - E_A)Z_A F}{RT \ln 10} + \left(1 - \frac{Z_A}{Z_B}\right) \log a_A$$

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

According to the data presented in the Table 2, the selectivity order of the examined cations varies as $K^+ > Sr^{2+} > Co^{2+} > Mn^{2+} > Ni^{2+} > Na^+ > Zn^{2+} > Mg^{2+} > Li^+ > Cd^{2+}$. The developed sensor is around 100 fold more selective towards barium ions than the closest interfering type. Based on the data given in Figure 6 and Table 2, we can state that the proposed novel sensor is highly selective towards barium ions than other species.

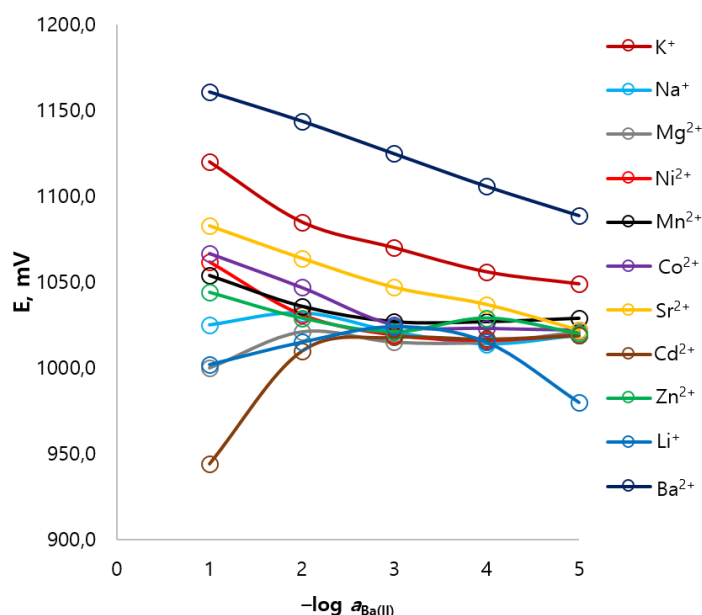


Figure 6. Selectivity of the newly developed barium-selective sensor towards other ions.

Table 2. The selectivity coefficient of barium-selective sensor.

Interfering ions	$\log K_{Ba(II),M^{+n}}$	$K_{Ba(II),M^{+n}}$
K ⁺	-2.02	9.55×10^{-2}
Sr ²⁺	-2.70	1.99×10^{-3}
Co ²⁺	-3.28	5.25×10^{-4}
Mn ²⁺	-3.65	2.24×10^{-4}
Ni ²⁺	-3.82	1.51×10^{-4}
Na ⁺	-3.79	1.62×10^{-4}
Zn ²⁺	-3.89	1.29×10^{-4}
Mg ²⁺	-4.16	1.92×10^{-5}
Li ⁺	-4.36	4.36×10^{-5}
Cd ²⁺	-4.53	2.95×10^{-5}

The working range of the prepared sensor in terms of pH was determined using nitric acid (for pH 2.0–7.0) and sodium hydroxide (for pH 8.0–12.0). The pH working range was studied for two different

concentration values, 1.0×10^{-2} and 1.0×10^{-3} M barium ion solution was added to the pH solutions previously prepared. Potentiometric measurements were taken with the prepared sensor. Potential-pH

graph was drawn with the obtained data. The pH working range of the barium-selective sensor is given in Figure 7 which points that pH remained constant between 5.0 and 11.0 at both concentration values. While the high potential values at pH values of less than 5.0 in 1.0×10^{-2} M concentration are due to the presence of hydronium ions, the low potential values at high pHs in both concentrations may be due

to the formation of $\text{Ba}(\text{OH})_2$. The low potential values at $\text{pH} < 5.0$ in 1.0×10^{-3} M concentration may suggest that the sensor exhibits an unstable behavior in this acidic environment. Combined, it can be suggested that the newly proposed sensor can work stably at different concentration values independently of pH changes.

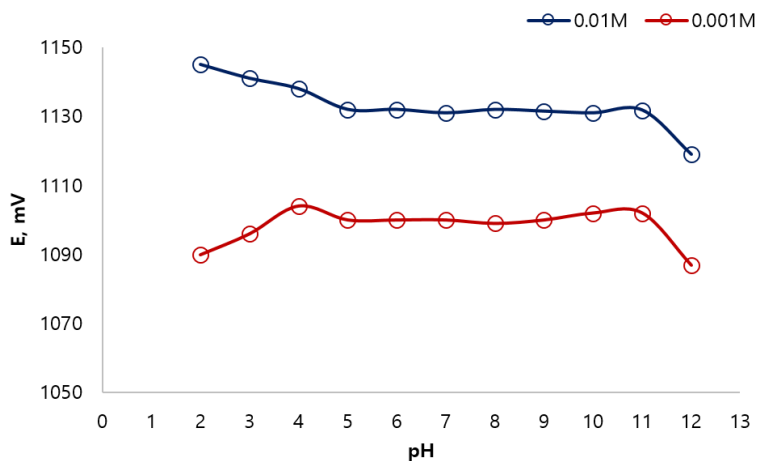


Figure 7. pH working range of the newly proposed barium-selective sensor.

Analytical applications of the produced sensors are an important indicator of sensor performance. In particular, applications in different matrices are necessary to demonstrate the selectivity and reliability of the sensor against a single ion. Therefore, applications of ISEs in real samples are of great importance. Analytical applications of the prepared novel barium-selective potentiometric sensor were conducted using different water and tea samples. Barium ions were added to these samples

in the amounts presented in Table 3, and subsequently potential values were measured with the newly developed sensor. The amount of barium added in the real samples was calculated by substitution of the potential values into the linear equation. Consequently, it was observed that the developed sensor can detect barium ions with high recoveries (>90.0%). The obtained results show that the sensor can give reliable results in the determination of barium ions in different matrices.

Table 3. Detection of Ba^{2+} ion in different samples.

Real Samples	Ba^{2+} quantity, (M)		
	Added Ba^{2+}	Found (\pm SD) with sensor*	% Recovery
Bottled water	1.0×10^{-3}	$9.57 (\pm 0.35) \times 10^{-4}$	95.7
Tea		$9.55 (\pm 0.25) \times 10^{-4}$	95.5
Pure water		$9.12 (\pm 0.10) \times 10^{-4}$	91.2
Tap water (Tokat, Turkey)		$9.02 (\pm 0.13) \times 10^{-4}$	90.2

*Average value ($n = 3$).

Comparison of the proposed new sensor with its counterparts in the literature is given in Table 4. As seen there, the sensor has a faster response time than previously proposed ones. It is relatively similar to the others in terms of linear working range and detection limit. When pH working ranges are examined, there is only one equivalent that can work at high pH levels independently of changes in the pH (Saleh, 2000).

Table 4. The potentiometric performance characteristics of sensor compared with other proposed barium ISEs.

Ionophore	Concentration range, M	Limit of detection, M	pH working range	Response time (s)	Reference
(<i>E</i>)-2-(2-((2-carbamothioylhydrazono)methyl)phenoxy)acetic acid	1.0×10 ⁻⁶ – 1.0×10 ⁻¹	5.54×10 ⁻⁷	6.0–9.0	<10	Altunoluk et al., 2024
2,3,4-pyridine-1,3,5,7,12-pentaazacyclopentadeca-3-ene	1.41×10 ⁻⁶ – 1.0×10 ⁻¹	Not reported	2.5–7.0	18	Singh et al., 2005
4-4'-methylenediantipyrine	1.0×10 ⁻⁶ – 1.0×10 ⁻²	5.2×10 ⁻⁷	3.4–10.6	15	Abedi et al., 2008
1,2-bis(diarylphosphine oxide) naphthalene	1.0×10 ⁻⁵ – 1.0×10 ⁻¹	5.0×10 ⁻⁶	3.0–11.0	60	Saleh, 2000
Barium–Rose Bengal	5.0×10 ⁻⁵ – 1.0×10 ⁻¹	2.5×10 ⁻⁶	4.5–10.0	20	Othman et al., 2006
4-aminobenzoic acid 2-diethylaminoethyl ester	1.0×10 ⁻⁵ – 1.0×10 ⁻¹	4.23×10 ⁻⁶	5.0–11.0	8	This work

4. CONCLUSION

ISEs have an essential place in electroanalytical chemistry due to their versatile applications. Features such as low cost, on-site analysis, ease of preparation and simple use distinguish ISEs from other costly analytical devices that require a laboratory. For many years, researchers have proposed sensors selective to many cationic and anionic species by taking advantage of these properties of potentiometric ISEs. They have significant advantages over other analytical techniques, especially in the detection of ionic species. In this study, 4-aminobenzoic acid 2-diethylaminoethyl ester was utilized as an ionophore. The proposed novel barium-selective potentiometric electrode was found to have a wide concentration range, near-Nernstian response, and lower limit of detection. Additionally, the sensor had a wide pH working range and fast analysis time. Therefore, we can state that the proposed sensor is rapid, selective, economical and reproducible for the on-situ detection of barium ions. This electrode which was successfully applied to different real samples can be utilized as an alternative to the various other analytical methods in the analysis of barium ions.

5. CONFLICT OF INTEREST

The authors state that they have no conflict of interest to declare.

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