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Novel electrochemical sensor for the determination of potassium ions based on silver diethyldithiocarbamate as sensor material

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ABSTRACT

Potassium is one of the macro minerals necessary for the cellular functions, obtained from foods. Excessive amounts of potassium can cause health problems. Determination of potassium in various samples, especially food and drug samples, is an important task. In this study, we developed a new potentiometric sensor selective to potassium ions. The sensor had a detection limit of 8.64×10^{-5} M in the concentration range of 1.0×10^{-1}–1.0×10^{-4} M and a near–Nernstian behavior of 49.0 ± 4.32 mV/decade. The sensor exhibited a response time of 10 s, as well as good selectivity, good repeatability, and a wide pH working range (4.0–11.0). The developed sensor was successfully applied to a drug sample and different water samples with very high recoveries (>91.20 %).

1. Introduction

Potassium is one of the essential macro minerals required for the physiological systems and, its intake is usually provided from foodstuffs [1,2]. Potassium has a wide range of biological functions, including enzyme activation, balancing pH and blood pressure, nerve transmission, muscle maintenance, and apoptosis [3]. The normal potassium ion level in blood serum is approximately 3.5–5.9 mM [4], and abnormal potassium levels can cause or may contribute to many disorders such as high blood pressure, diabetes, various cancers, kidney and heart diseases [5,6]. Therefore, the determination of potassium ions in various biological samples is important for the diagnosis and treatment of certain diseases. Additionally, it is important to detect and quantify potassium levels to evaluate the quality of particular foods such as infant foods, vegetables, fruits, etc. [7]. So far, numerous analytical methods for determination of potassium ions have been reported, including atomic absorption spectrometry (AAS) [8], inductively coupled plasma mass spectrometry (ICPMS) [9], high–performance liquid chromatography (HPLC) [10], X–ray fluorescence spectrometry (XRF) [11] and neutron activation analysis (NAA) [12]. However, these analytical methods are expensive, time consuming, require large amounts of solvents, experienced personnel, well–equipped laboratories and high energy consumption [13,14].

Potentiometry, one of the electrochemical analysis methods, has many versatile advantages. In particular, they are low cost, highly fast and selective, reproducible, and have wide concentration range, low detection limit, long lifetime and also very suitable for on–site analysis [15–17]. Therefore, they are widely preferred to be used for the analysis of various food, pharmaceutical, medical, agricultural, environmental and textile samples (Fig. 1) [18,19].

Ionophores found in the structure of ion–selective electrodes are special molecules that can selectively interact with the analyte ion. With developing technology, bio-renewable and green materials such as cellulose, chitosan and gelatin are also widely preferred in sensor research [20–22]. In previous studies, Valinomycin **(1)** [23,24], 4,4'-bis [4''-phenoxy(15-crown-5) methyl]benzyl **(2)** [25], 5,5'-(1,4-phenylene)bis(3-(4-methoxyphenyl)-4,5-dihydro-1*H*-pyrazole-1-carbothioamide) **(3)** [26] and 5,10,15,20–tetrakis (*p*–bromophenyl)porphyrin **(4)** [2] were used as ionophores. The interaction between potassium ions and ionophores occurs according to the ion exchange mechanism. Valinomycin, an antibiotic, is an ionophore that makes membranes particularly permeable to potassium ions. Many ion–selective sensors have been proposed for the determination of potassium ions [27]. Other ionophores exhibit a potential as a result of the interaction of functional groups in their structures with potassium ions. In this study, silver diethyldithiocarbamate **(5)** (Fig. 2) was used as an ionophore, and its sensor properties were investigated. Sensors with different components were prepared and the potentiometric performance properties of these sensors were tested under laboratory conditions. Finally, we successfully applied these sensors to various drug and water samples.

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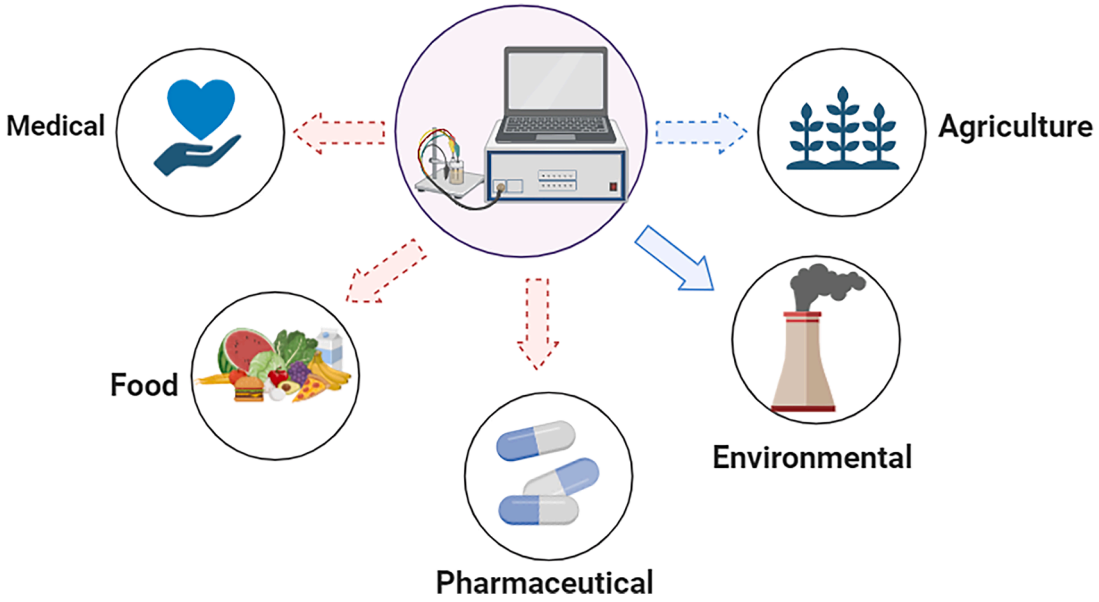

Fig. 1. Application areas of potentiometric sensors.

2. Experimental

In this study, graphite, silver diethyldithiocarbamate (ionophore; 99.9 %), plasticizers [bis(2–ethylhexyl)sebacate (BEHS; ≥97.0 %), bis (2–ethylhexyl) adipate (DEHA; ≥99.0 %), DBP (99.9 %)], KT*p*ClPB, PVC and tetrahydrofuran (THF; ≥99.9 %) used to prepare all–solid contact electrodes were purchased from Sigma–Aldrich. Epoxy (Macroplast Su 2227) and hardener (Desmodur RFE) were supplied from Henkel and Bayer AG, respectively. Cation nitrate salts used in selectivity studies were obtained from Merck, Fluka and Sigma–Aldrich. Nitric acid (HNO_3) and sodium hydroxide (NaOH; ≥98.0 %) used in pH studies were purchased from Sigma–Aldrich and Merck, respectively.

Potential measurements were achieved by a laboratory–made computer–controlled, multi–channel potentiometer (Medisen Medical Ltd. Sti., Turkey). A silver/silver chloride electrode (Thermo Scientific Orion 900100) was operated as a reference electrode throughout the potentiometric measurements. The ultrapure water used in the study was obtained from the MP Minipure water purification system (Dest up, 0513957).

In the preparation of conductive solid contact PVC membrane sensors, the method frequently used in the literature was also utilized in this study [28–30]. Firstly, conductive solid contact electrodes were prepared by mixing 50 % (*w/w*) graphite, 35 % (*w/w*) epoxy and 15 % (*w/w*) hardener in 3 mL THF. When the mixture was homogeneous, one of the open ends of approximately 15 cm copper wires was dipped into the mixture, their surfaces were covered and left to dry for 24 h in the dark. Then, PVC membrane mixtures consisting of PVC, plasticizer, ionophore and anion excluder at the mass ratios indicated in Table 1 (100 mg total) were prepared by dissolving them in 3 mL THF. Copper wires, previously coated with solid contact mixture, were then dipped into this mixture, their surfaces were coated and left to dry for approximately 24 h. These dried sensors were included in the potentiometric measurement system with the reference electrode, and subsequently potential measurements were performed.

Potential measurements were conducted at room temperature (25 ± 1.0 °C) by using the following electrochemical cell assembly:

Ag/AgCl; KCl (3M) || K^+ sample solution | PVC membrane | conductive solid contact | copper wire

Potentiometric data were recorded while the prepared sensor and reference electrode were immersed in 50 mL of test solution and each time rinsed with distilled water and wiped with a smooth adsorbent tissue paper.

3. Results and discussion

3.1. Sensor optimization and potentiometric response

In this study, sensors with five different compositions were prepared. The compositions and performance characteristics of the prepared sensors are given in Table 1. Three different plasticizers (DBP, BEHS and DEHA) were used in the composition of the sensors. The ionophore, polymer, plasticizer, and anion excluder compositions of the first three sensors were kept constant. Among these prepared sensors, the sensor containing DBP had the lowest detection limit and the highest R^2 value in the concentration range of 1.0×10^{-1}–1.0×10^{-4} M (Table 1, Fig. 3). Among the prepared sensors, sensor I had the most ideal composition (3.0 % ionophore, 64.0 % DBP, 32.0 % PVC and 1.0 % KT*p*ClPB), and potentiometric performance studies were completed using sensors with this composition in the other stages of the study. The potentiometric behavior of the prepared sensor was tested using potassium solutions. Fig. 4 shows the potentiometric response of the developed sensor. As this figure points, the sensor exhibited a very stable behavior in a reversible manner in the concentration range of 1.0×10^{-1}–1.0×10^{-4} M. The calibration curve of the prepared sensor III is given in Fig. 5. The determination limits of the sensors prepared using calibration curves were determined according to IUPAC [31]. The potential value corresponding to the intersection of the horizontal and vertical axes on the calibration curve was written in the linear equation $E = -47.77$ [(–log K^+)] + 1238.1). Thus, the detection limit of the sensor was calculated as 8.64×10^{-5} M. The response time of the proposed sensor was investigated according to IUPAC guidelines [32]. The time for the sensor to reach equilibrium for each 10–fold concentration change was determined. The sensor had a response time of less than 10 s.

3.2. Repeatability

The repeatability graph of the developed sensor is presented in Fig. 6, and the repeatability data is presented in Table 2. Successive measurements were taken at three different concentration values (10^{-1}, 10^{-2} and 10^{-3} M), and it was determined that the sensor exhibited repeatable

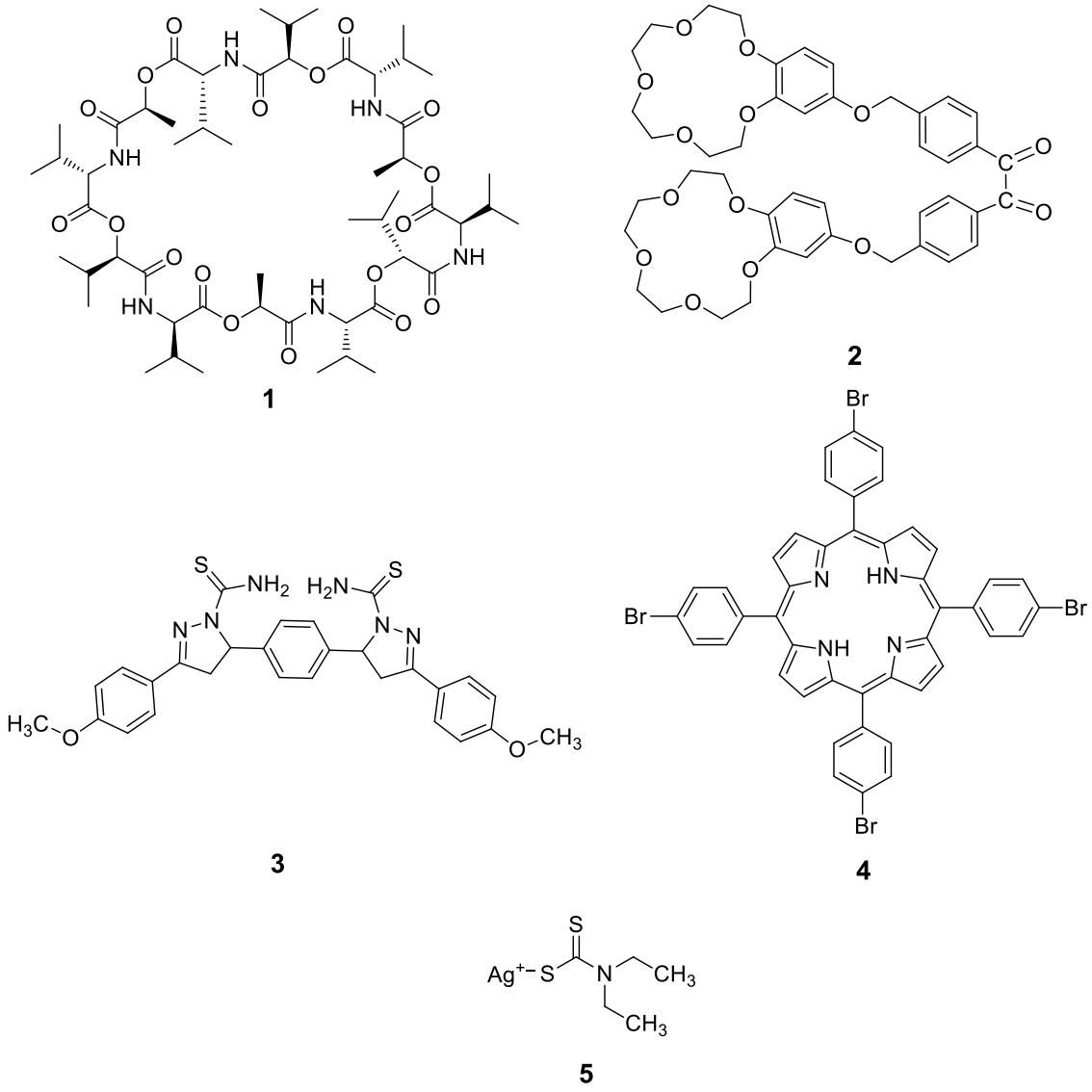

Fig. 2. The chemical structures of ionophores used in potassium-selective sensors.

Table 1
Composition and potentiometric characterization of the prepared sensors

No	Membrane composition (*w/w*)						Potentiometric performance			
	PVC	Ionophore	Plasticizer			KT*p*ClPB				
			DBP	BEHS	DEHA		Linear concentration range (M)	Limit of detection (M)	Slope (mV/decade)	R^2
1	32.0	3.0	64.0			1.0	1.0×10^{-1}–1.0×10^{-4}	8.64×10^{-5}	49.0 (±4.32)	0.9974
2	32.0	3.0		64.0		1.0	1.0×10^{-1}–1.0×10^{-4}	1.0×10^{-4}	44.3 (±4.70)	0.9883
3	32.0	3.0			64.0	1.0	1.0×10^{-1}–1.0×10^{-4}	9.75×10^{-5}	45.3 (±4.02)	0.9905
4	32.0	2.0			65.0	1.0	1.0×10^{-1}–1.0×10^{-4}	9.44×10^{-5}	43.3 (±2.01)	0.9910
5	32.0	4.0			63.0	1.0	1.0×10^{-1}–1.0×10^{-4}	9.89×10^{-5}	42.3 (±4.78)	0.9837

behavior. According to the repeated measurements taken for the three different concentration values in Table 2, the standard deviations are ± 1.20, ± 0.63 and ± 0.45, respectively. These results show that the proposed sensor exhibits repeatable and stable behavior. Based on the these data given in Fig. 6 and Table 2, the newly developed sensor was found to be reproducible.

3.3. Selectivity

It is very important for ion-selective electrodes to exhibit species-specific behavior. Prepared sensors must exhibit particular selectivity towards the main ion rather than other ions that may be potentially present in the environment. In this study, the selectivity of the sensor prepared using 15 different cationic species was also investigated. The potentiometric behavior of the sensor against potassium ions and other ions is given in Fig. 7. As can be seen, at high concentrations, the proposed sensor is highly selective towards potassium ions, while at low concentrations ($< 10^{-4}$ M), some ions may interfere. Since potassium is generally found in certain concentrations in food or drug samples, the sensor selectivity in the samples to be analyzed is acceptable. IUPAC has

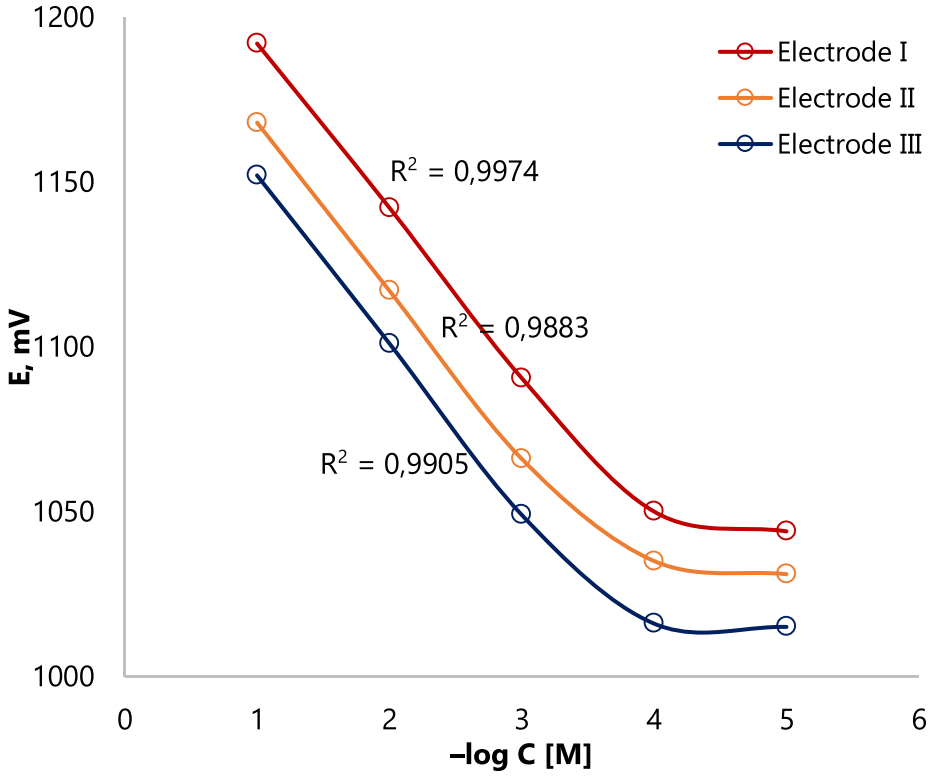

Fig. 3. Calibration curves of sensors prepared with different plasticizers (3.0 % ionophore, 64.0 % plasticizer, 32.0 % PVC and 1.0 % KT*p*ClPB).

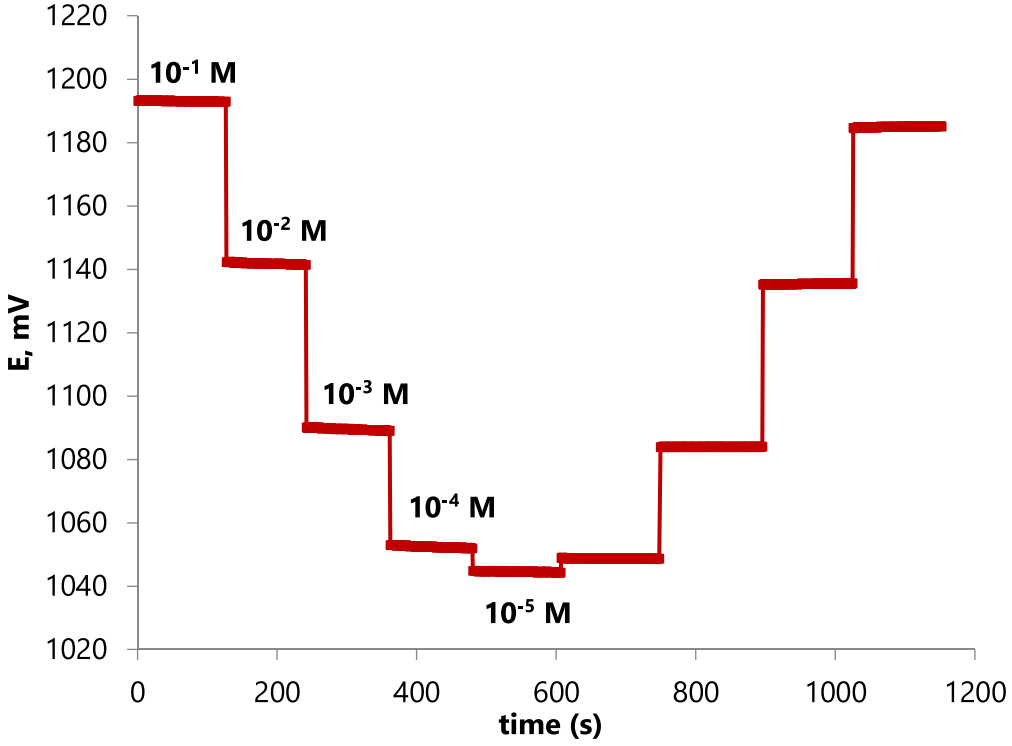

Fig. 4. Potentiometric behavior of potassium selective sensor.

recommended selectivity coefficients for ion selective electrodes. In this study, selectivity coefficients were calculated using the separate solution method (SSM) recommended by IUPAC [31]. Table 3 shows the selectivity coefficients calculated using the potential values of the ions corresponding to 10^{-1} M. As a result, Fig. 7 and Table 3 show that the proposed sensor is selective for potassium ions in the presence of other ions.

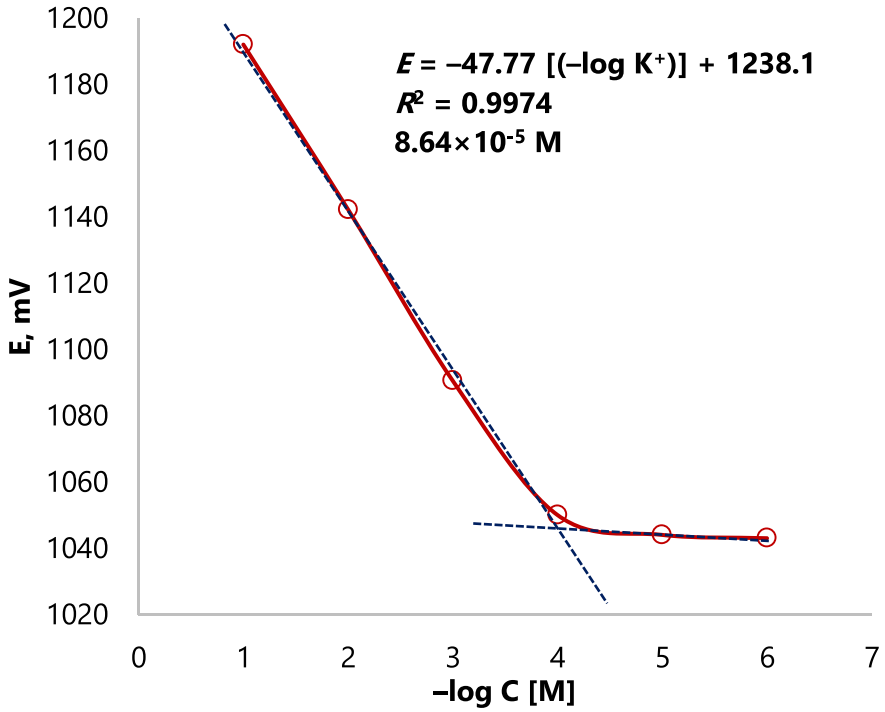

Fig. 5. The calibration curve of potassium selective sensor.

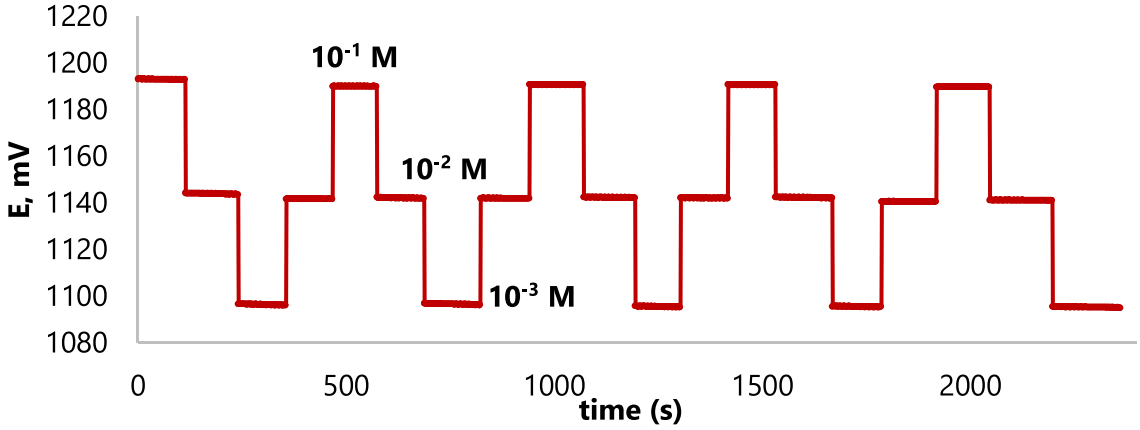

Fig. 6. The repeatability of potassium selective sensor.

Table 2
Repeatability data of potassium(II)–selective sensor.

Concentration (M)	Potentials (mV)					
	I	II	III	IV	V	Average (±SD)
1.0×10^{-1}	1193.2	1191.1	1191.0	1190.1	1189.7	1191.0 (±1.20)
1.0×10^{-2}	1141.0	1142.0	1142.0	1140.4	1141.0	1141.3 (±0.63)
1.0×10^{-3}	1096.0	1096.0	1095.0	1095.0	1095.4	1095.4 (±0.45)

3.4. pH working range

The pH working range shows the range in which the recommended ion selective electrodes can work independently of pH. The pH working range of the proposed sensor was determined by preparing acid–base solutions in the range of pH 2.0–12.0. While pH<7.0 solutions were prepared with HNO_3, pH>8.0 solutions were prepared with NaOH. 10^{-2} M KNO_3 was added to the prepared solutions. The developed sensor was immersed in these solutions sequentially and the potentials (mV) were recorded. The pH working range of the sensor is given in Fig. 8 which shows that it can work in the pH range of 4.0–11.0 without being affected by pH changes.

3.5. Analytical applications

Applications of each proposed ion selective electrode in real samples can be considered as an indicator of its reliability. In this study, applications of the developed sensor in different water samples and a drug sample were carried out. Potassium at a certain concentration was added to the water samples given in Table 4, and the amount of potassium was further determined with the sensor. Additionally, potassium determination was carried out in an analgesic drug sample with a particular potassium concentration in its composition. Finally, the developed

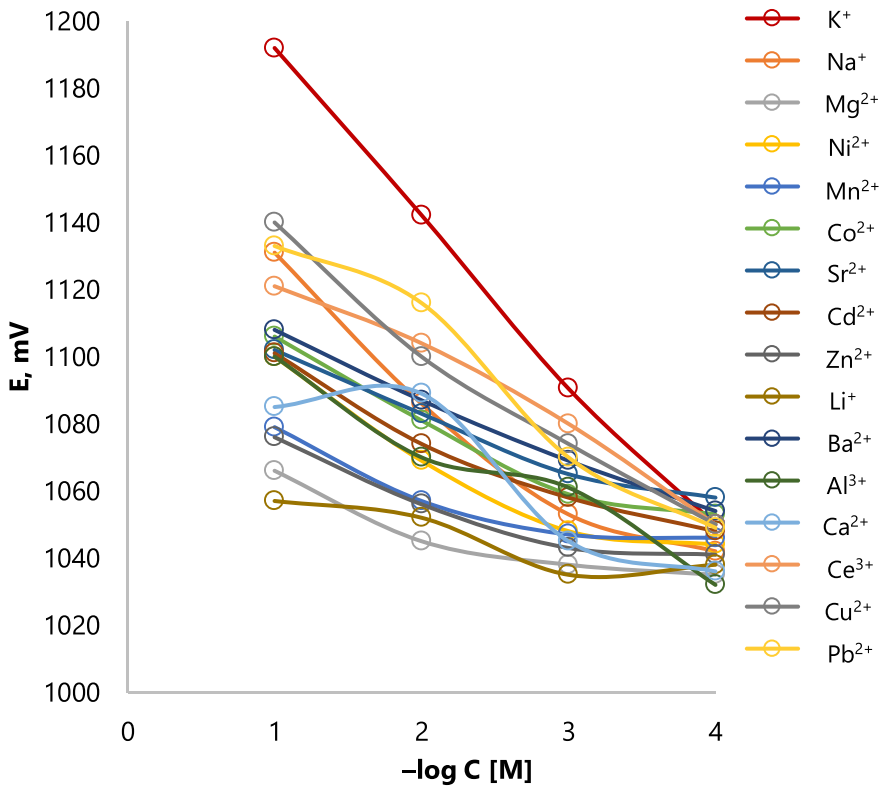

Fig. 7. Potentiometric selectivity of potassium selective sensor.

Table 3
Selectivity coefficient of potassium selective sensor.

Interfering ions	Selectivity coefficient		Interfering ions	Selectivity coefficient	
	$logK^{pot}_{K^+,M^{n+}}$	$K^{pot}_{K^+,M^{n+}}$		$logK^{pot}_{K^+,M^{n+}}$	$K^{pot}_{K^+,M^{n+}}$
Cu^{2+}	–1.01	0.098	Ni^{2+}	–1.55	0.028
Na^{+}	–1.03	0.093	Al^{3+}	–1.56	0.027
Ce^{3+}	–1.20	0.063	Ca^{2+}	–1.81	0.015
Ba^{2+}	–1.42	0.038	Mn^{2+}	–1.91	0.012
Co^{2+}	–1.46	0.035	Zn^{2+}	–1.96	0.011
Sr^{2+}	–1.52	0.030	Mg^{2+}	–2.13	7.4×10^{-3}
Cd^{2+}	–1.54	0.029	Li^{+}	–2.28	5.2×10^{-3}

sensor was found to be able to determine potassium in both water samples and drug samples with very high recoveries.

3.6. Comparison study

The proposed new potassium selective sensor was compared with other ISEs in the literature. According to Table 5, the proposed new sensor has the widest pH working range compared to its counterparts. In addition, it has a very fast response time like its counterparts. Valinomycin has been widely preferred over other ionophores in the preparation of selective sensors for potassium ions. Membrane structures prepared with valinomycin are permeable to potassium ions. This significantly affects the behavior of the sensors.

4. Conclusion

Ion selective electrodes have been widely used in many fields to date to determine various ionic species. The biggest advantages of ion selective electrodes are that they are fast, highly selective and low cost to manufacture. In this study, a new ion selective sensor selective to potassium ions was proposed. The proposed sensor had a low detection limit over a wide concentration range. The sensor, which can work in a wide pH range, has been applied quite successfully in the analysis of potassium ions in water and drug samples. It is clearly seen that the developed sensor can be used as an alternative to other analytical methods in potassium ion analysis in various food, drug and water samples.

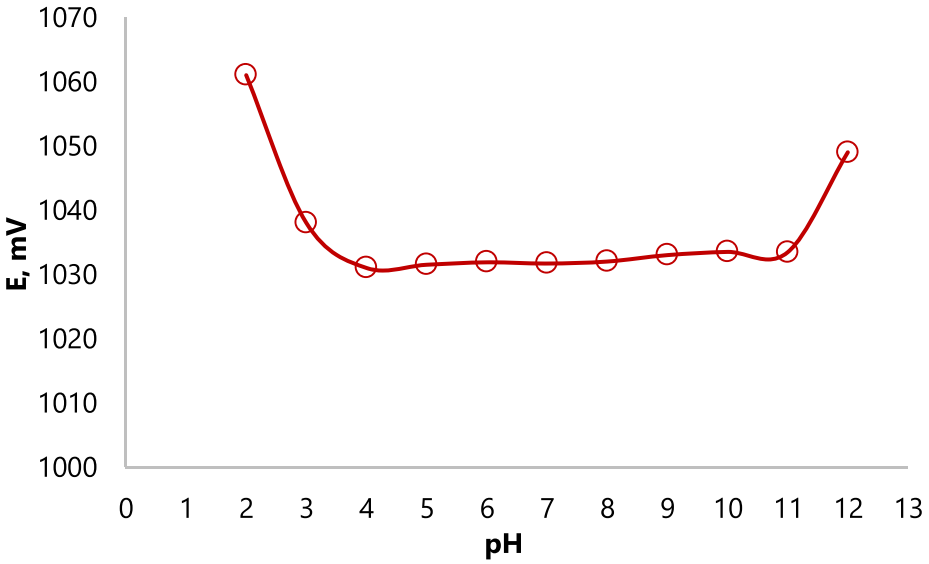

Fig. 8. pH working range of potassium selective sensor.

Table 4
Application of developed sensor in different real samples.

Water samples	K^+ quantity, (M)		% Recovery
	Added K^+	Found (±SD) with sensor*	
Purification water	1.0×10^{-3}	$0.986\ (\pm 0.50) \times 10^{-4}$	98.60
Bottled water	1.0×10^{-3}	$0.923\ (\pm 0.39) \times 10^{-4}$	92.30
Tap water	1.0×10^{-3}	$0.912\ (\pm 0.24) \times 10^{-4}$	91.20
Drug sample	K^+ quantity, (mg)		
	Drug content K^+	Found (± SD) with sensor*	% Recovery
Analgesic drug	5.85 mg	5.71 (± 0.10)	97.60

* Average value ($n = 3$)

Table 5
Comparison of the developed potassium selective sensor with ISEs in the literature.

Ionophore	Concentration range (M)	Limit of detection (M)	pH working range	Response time (s)	Refs.
1	$1.0 \times 10^{-5} - 1.0$	5.8×10^{-6}	Not reported	Not reported	[23]
1	$5.0 \times 10^{-5} - 1.0 \times 10^{-1}$	4.0×10^{-5}	5.0–9.0	< 15	[24]
2	$1.0 \times 10^{-5} - 1.5 \times 10^{-1}$	1.0×10^{-7}	5.0–7.0	< 10	[25]
3	$1.0 \times 10^{-5} - 1.0 \times 10^{-1}$	6.02×10^{-6}	6.0–10.0	10	[26]
4	$1.0 \times 10^{-5} - 1.0 \times 10^{-1}$	5.88×10^{-6}	6.0–10.0	< 5	[2]
1	$1.0 \times 10^{-6} - 1.0 \times 10^{-2}$	Not reported	Not reported	30	[33]
1	$1.0 \times 10^{-5} - 1.0 \times 10^{-1}$	4.0×10^{-5}	5.0–9.0	< 15	[34]
1	$1.0 \times 10^{-5} - 1.0 \times 10^{-2}$	6.3×10^{-6}	Not reported	Not reported	[35]
5	$1.0 \times 10^{-4} - 1.0 \times 10^{-1}$	8.64×10^{-5}	4.0–11.0	< 10	This work

CRediT authorship contribution statement

Oguz Özbek: Writing – original draft, Resources, Methodology, Investigation, Formal analysis. **Mehmet Alperen Şahin:** Investigation, Formal analysis.

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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