IRAQI JOURNAL OF APPLIED PHYSICS
Vol. 22, No. 3, July-September 2026, pp. 485-492

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Synthesis and Analysis of PVC Selective Membrane of Heavy Metal Ions for Water Pollution Detection and Selectivity Study via Separation Solution Method (SSM)

Fabrication of electrodes made from Al^{3+}, Zn^{2+}, Pb^{2+}, and Na^{1+} ions were used as a potentiometric electrochemical sensors for water pollution detection by heavy metals. In this work, the sensor device consisted of Zn, Pb, Na, and Al ions as ion exchangers and polyvinyl chloride PVC and tris (2-ethylhexyl) phosphate as plasticisers. The response properties of these electrodes, calibration curve, lifetime, and limit of detection were studied. Interactions against monovalent, divalent, and trivalent ions like Al^{3+}, Zn^{2+}, Pb^{2+}, and Na^{1+} were also investigated through selectivity. The limits of detection of Al, Zn, Pb, and Na were 0.0894, 0.819, 0.0037, and 16.73, and the correlation coefficients were about 0.9595, 0.9627, 0.9744, and 0.9595, and the lifetimes were approximately 24, 25, 25, and 30 days, respectively. A short time was required to reach the equilibrium potential. The system's small size, inexpensive materials, and ability to measure speed and accuracy make it ideal. Therefore, the electrochemical sensor can be applied to quantitatively determine heavy metal ions in river water samples with satisfactory results.

Keywords: Ion-selective electrodes; PVC membrane; Metal ions detection; Membrane selectivity
Received: 27 November 2025; Revised: 2 February 2026; Accepted: 9 February 2026; Published: 1 July 2026

1. Introduction

The ISE is an electrochemical cell that consists of an operating electrode supported by an ion-selective membrane and a reference electrode. Ion-selective electrodes (ISEs) aim to transform the activity of a specific ion into a measurable electric potential (E_{cell}) as the output signal [1].

Ion-selective electrodes (ISEs) are among the most frequently used potentiometric sensors in laboratory analysis, and they are also employed in various applications, including physiological measurements, industrial process control, drug analysis, and environmental monitoring [2,3].

The most significant advantage of ISE is that it measures activity directly, rather than concentration. The linear working range of many electrodes was quite extensive. Electrodes functioned well in colored or turbid samples, making them more effective than spectrophotometric measurements. For most ISEs, the equilibrium state was reached in under a minute, making it suitable for kinetic studies and monitoring changes in flowing process streams. The equipment used was simple and quite inexpensive [4-6].

Electrochemical analyses are commonly used due to their simplicity, ease of use, and lower costs. For a potentiometric study, the potential is determined between the operating and reference electrodes, so that the electrical current (with a high voltage) between both electrodes is typically maintained at almost zero. According to the selective reordering of ionic particles between the two layers, the pulse is produced by all the voltage at the junction between both membranes, as well as the solution. A potentiometric cell consists of an ISE, which is extremely sensitive to a particular ion to be analyzed and is less sensitive to many other external interferences, referring to reference electrodes (typically Ag/AgCl), which are constant and known as the target potential [7].

Environmental pollution is defined as the contamination of the biological and physical components of the atmosphere and Earth's system to such an extent that normal environmental processes are adversely affected [8,9]. Since a large amount of chemical pollutants infiltrate rivers, lakes, and wetlands, water pollution is the primary type of pollution. When toxic chemicals are released into the environment, they can be transported by air, water, and living organisms. They can also accumulate in the food chain and become part of the natural biogeochemical cycle. All ecosystems' "trouble spot" is water because so many pollutants are carried by it. It also serves as a medium in the cycle and a solvent for a variety of substances: animals, plants, soil, and air [10]. Recently, numerous researchers have focused on detecting water pollution caused by heavy metals [11-14].

Heavy metals are metallic elements that have a high density and are toxic, even at low concentrations. Heavy metal is a general expression that characterizes the group of metals with an atomic density larger than 4 g/cm^3 or 5 times larger than water [15].

Heavy metals are divided into two classes: essential and non-essential according to their role in living systems: (a) basic heavy metals are required by living organisms for their growth and development, and (b) physiological processes, such as those that use manganese (Mn), iron (Fe), Nickel (Ni), copper (Cu) and zinc (Zn), and living organisms do not need non-essential heavy metals, including arsenic (As), Cadmium (Cd), lead (Pb) and mercury (Hg), for pathological functions [9].

Since numerous pollutants are carried by water, water is the "trouble spot" of all ecosystems. It also plays a crucial role in the cycle (air-soil-plants-animals) as a medium and a solvent for various substances [16].

Water pollution is one of the problems that threatens the lives of humans and animals alike. The pollution of ecosystems by toxic heavy metals and organic pollutants is constantly increasing due to human activities [17].

Selectivity, defined in ISE, is the ability to distinguish between both the primary ions and many other sampling ions, and thus is one of the most important parameters describing the ISE [18,19].

The selectivity function depends on both the mobility of the ion in the membrane and the ion partitioning coefficients between all the aqueous layers, membrane layers, and the ionophore complex formation constants [20].

Selectivity is regarded as the most essential characteristic that identifies an ion-selective electrode, confirming the potential of a reliable intended sample. There are numerous methods for measuring selectivity; the separated solution method (SSM) is one of them. Measurement of SSM was dependent on the equation of Nickolsky-Eisenman, which is an IUPAC-recommended method for determining the selectivity coefficient of ion-selective electrodes [21].

Tris (2-ethylhexyl) phosphate (TEHP) is a commonly used plasticiser, particularly in polyvinyl chloride (PVC) and other polymers, due to its ability to improve flexibility and processability. It also serves as a solvent and a fire retardant. As such, it is present in various environmental matrices, including sediment [22].

The separation solution method (SSM) was used to calculate the measurement of the selectivity coefficient. This is the simplest and most widely used method of obtaining quantitative results using ion-selective electrodes. Standard solutions are prepared by serial dilution of a concentrated standard. This method is suitable for analyzing all samples in which the analyte of interest is present in its free, uncompleted state [23-25].

This work aims to measure water pollution by heavy metal ions, and to measure the selectivity of ISE electrodes with interfering ions using the Separation Solution Method (SSM).

2. Materials and Methods

To prepare the ion-selective membrane, the following materials were used. Metal ions (Zn, Pb, and Al) and Na salt ions of concentration 1000 ppm from Belgium. Tetrahydrofuran (THF) for HPLC, SRL-INDIA, Polyvinyl chloride (PVC) (Fluka AG, 98%). The plasticizers of the composition and viscosities are Tris(2-ethylhexyl) phosphate, from Fluka AG, hydrochloric acid (HCl) (SIGMA; 99.9%), and ammonium hydroxide (NH_4OH) (Laboratory Reagent: approximately 25% NH_3).

Electrode membranes for water pollution were prepared using metal ions (Al, Zn, Pb, and Na) ions, along with a plasticizer. The immobilization of the heavy metal ions into the matrix membrane was done using the procedure outlined by Davis et al. [26], as shown in Fig. (1). Mix 0.25 g PVC with 0.04 g of heavy metal ions and 0.4 g plasticizer, then add 7-9 mL of tetrahydrofuran (THF) to mix until the solution is homogenized. The precise proportions of the electrode fabrication and specific ion exchanger used are shown in table (1). The mixtures are placed into a 30-35 mm diameter glass ring and allowed to relax on a glass plate. The two sections were then cemented together using a viscous PVC-THF mixture (to ensure there is no loss of the membrane mixture), and a filter ribbon was fixed on top of the glass. The solvent was then allowed to evaporate for at least 24 to 48 hours, depending on the room temperature [27,28].

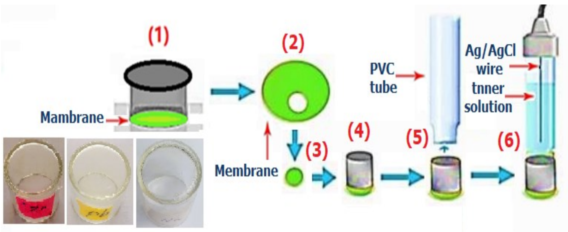


Fig. (1) Formation of the ion-selective membrane

To assemble the ion-selective electrode (ISE), the membranes were carefully removed from the glass plate. The thickness of the extracted membranes varied from one membrane to another, with thicknesses ranging approximately from 0.4 to 0.7 mm. The membrane size was sufficient for electrode preparation see Fig. (2). The PVC tube was plunged into THF solution from one of its sides and positioned vertically on the prepared membrane, so that the membrane portion was cut with a diameter equal to the PVC tube and carefully pressed to the end of the PVC tube. Connected the other end of a glass container assembly to a plastic cover, and an Ag/AgCl wire was pushed through it. The electrode was then activated by immersing it in a water solution containing (Al, Zn, Pb, and Na) ions for at least 2 hours before use, as in the Emad and Al-Byati method [29].

An ion-selective electrode (ISE) cell is made up of a reference electrode (RE) and an ion-selective electrode (ISE). In general, the cell consists of two "internal" and "external" reference electrodes, as well as a selective thin-film membrane that serves as the recognition element.

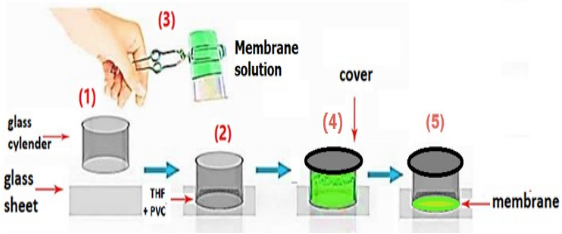


Fig. (2) Assembling the Ion-Selective Electrode

As shown in Fig. (3), design the cell under the fundamental rule for creating electrolytic cells, as long as the current flowing through the cell is equal to zero. This cell consists mainly of two electrodes, a working electrode (WE) and a reference electrode (RE) [26].

The working electrode (WE) is the central electrode in the electrochemical cell, which can have different configurations and compositions that provide different potential ranges. It can be made from various materials, including titanium (Ti), platinum (Pt), mercury (Hg), carbon (C), zinc oxide (ZnO), and nickel (Ni), among others [17].

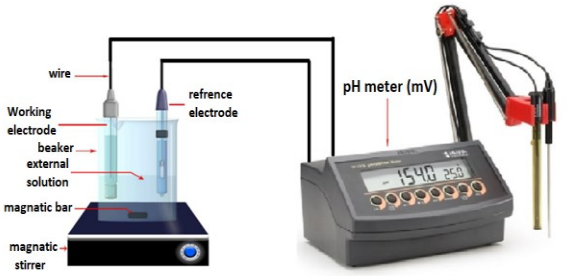
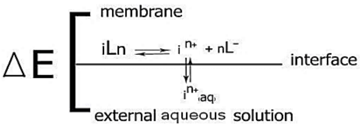


Fig. (3) Basic components of an ion-selective electrode sensor

The most popular reference electrodes (RE) are the Ag/AgCl and calomel electrodes, which have a constant potential that does not change with the applied potential in WE and are not sensitive to the composition of the target analyte.

The potential of the working electrode (WE) varies in response to the ion concentration in the field water. In contrast, the potential of the reference electrode remains constant regardless of the characteristics of the target analyte. In potentiometric measurements, the potential difference (measured at a constant current) is determined when both immersed electrodes are used in an electrochemical cell containing a solution. The change in potential is proportional to the concentration of the specific analyte in the solution (in our case, water). The exchange between internal and external solutions across the membrane is determined by ionic exchange.

The exchange between internal and external solutions across the membrane is determined by ionic exchange. The relation below represents the ionic exchange process of a cation through a membrane [30]



(1)

3. Results and Discussion

The standard solutions of (Al, Zn, Pb, and Na) ions were diluted from a standard of about 1000 ppm in the proportions shown in table (2), using the equivalent law in Eq. (2)

For acid and base, $N_1V_1 = N_2V_1$ (2)

Table (2) ISE sensor response (Potential) at different ion concentrations (C) (Al, Zn, Pb, and Na), for standard samples

Concentration of Al (ppm)	Calculated concentration (M)	e.m.f (mV)
0.1	0.370×10^{-5}	-358
0.2	0.741×10^{-5}	-349
0.3	1.111×10^{-5}	-346
0.4	1.482×10^{-5}	-344
0.5	1.853×10^{-5}	-336

Concentration of Zn (ppm)	Calculated concentration (M)	e.m.f (mV)
1	0.153×10^{-4}	-448
3	0.4588×10^{-4}	-439
5	0.765×10^{-4}	-430
7	1.07×10^{-4}	-423
9	1.3766×10^{-4}	-417

Concentration of Pb (ppm)	Calculated concentration (M)	e.m.f (mV)
0.01	0.483×10^{-7}	-447
0.02	0.965×10^{-7}	-440
0.03	1.447×10^{-7}	-435
0.04	1.930×10^{-7}	-430
0.05	2.413×10^{-6}	-424

Concentration of Na (ppm)	Calculated concentration (M)	e.m.f (mV)
100	0.43497×10^{-2}	-243
150	0.6525×10^{-2}	-236
200	0.8699×10^{-2}	-230
250	1.087×10^{-2}	-222
300	1.3049×10^{-2}	-214

The concentrations were converted from parts per million to molar using Eq. (2) to use in the calibration curve of ion electrode using (TEHP) as a plasticizer to (Al, Zn, Pb, and Na), as shown in table (1).

$$M = \frac{ppm}{A.wt \times 1000} \quad (3)$$

where $A.wt$ represents the atomic molecular weight of specific ions.

More than one concentration was chosen to determine the electrode's responsiveness to different concentrations.

The sensor response to metals and salts was measured using the system in Fig. (4), through which the e.m.f. (eV) was measured at different concentrations (C). Table (3) summarizes the results of these measurements.

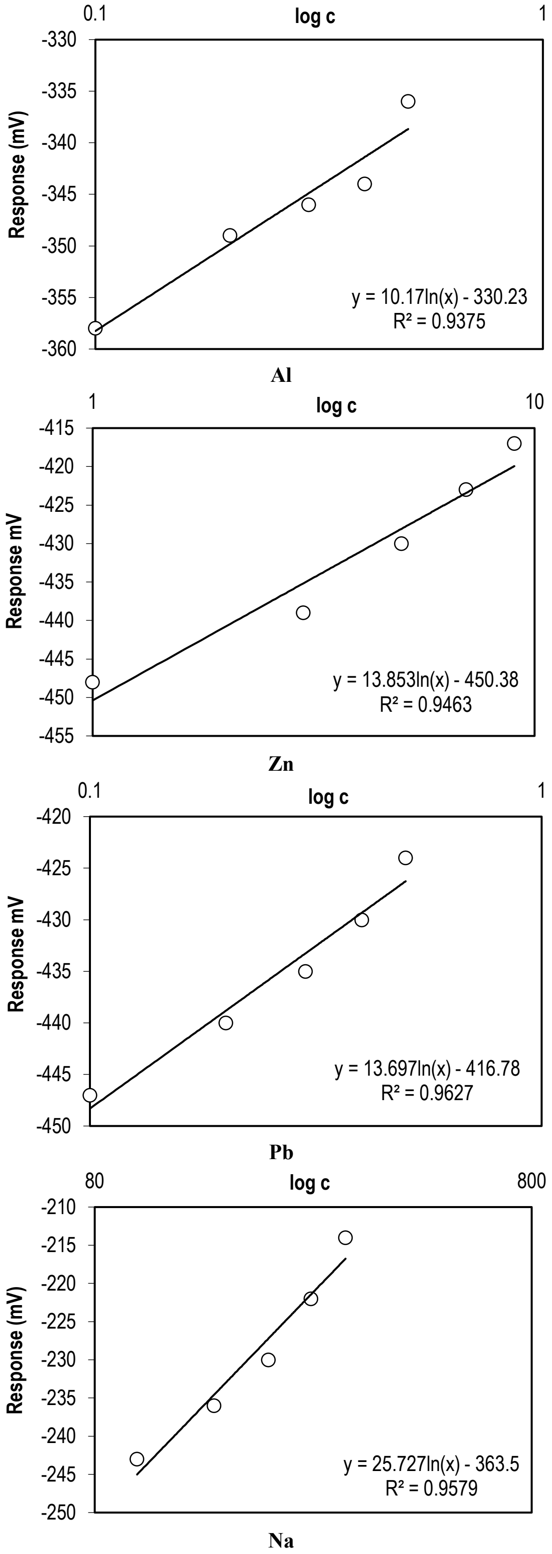


Fig. (4) Calibration curves of (Al, Zn, Pb, and Na) ions sensor for standard samples

The limit of detection (LOD) is the lowest concentration or quantity of a substance that can be reliably identified and distinguished [31].

To calculate the detection limit value, we use the following equation:

$$\frac{3\,SD}{Slope} \qquad (4)$$

where SD is the standard deviation

Table (3) The potentiometric properties for (Al, Zn, Pb, and Na) electrode

Properties of Al	Electrode values
Linear range	$0.37\times10^{-5}\ to\ 1.18\times10^{-5}$ M
Slope	**23.4**
Limit of detection	0.0894 ppm
correlation coefficient	**0.9375**
Lifetime (day)	**24**

Properties of Zn	Electrode values
Linear range	$0.15\times10^{-4}\ to\ 1.37\times10^{-4}$ M
Slope	**31.8**
Limit of detection	0.819 ppm
correlation coefficient	**0.9463**
Lifetime (day)	**25**

Properties of Pb	Electrode values
Linear range	$0.15\times10^{-6}\ to\ 0.76\times10^{-6}$ M
Slope	**31.5**
Limit of detection	0.0037 ppm
correlation coefficient	**0.9627**
Lifetime (day)	**25**

Properties of Na	Electrode values
Linear range	$0.43\times10^{-2}\ to\ 1.3\times10^{-2}$ M
Slope	**59.2**
Limit of detection	16.73 ppm
correlation coefficient	**0.9579**
Lifetime (day)	**30**

The potential response of Al, Zn, Pb, and Na electrodes at varying concentrations of the material was displayed in a linear range from (0.153×10^{-4} M to 1.3766×10^{-4} M), (0.153×10^{-6} M to 0.765×10^{-6} M), (0.43497×10^{-2} M to 1.3049×10^{-2} M), and (0.370×10^{-5} M to 1.185×10^{-5} M), respectively. The Correlation coefficient was 0.9595, 0.9627, 0.9744, and 0.9595, respectively, as shown in Fig. (4), which displays the linear relation between sensor potential (e.m.f.) resulting from the fabricated electrochemical sensors and the concentration of metals and salt ions.

The influence of interfering ions on the response behaviors of ion-selective membrane electrodes is usually described in terms of selectivity coefficients. Different methods were suggested for measuring the selectivity coefficients [32,33]. The K values of heavy metal sensors were calculated using the separate solutions method (SSM). An electrode is calibrated in the main ion solutions when using the SSM.

The variation in the electrode potential obtained when attracted with the electrolytes, including both interfering ions (J) and primary ions (I) at the same activity, is used to estimate the selectivity coefficient (K).

A cell's potential contains an ion-selective electrode, as well as two different solutions being used to calculate a reference electrode, one of which includes ions A with activity aA, and the other is found to contain B ions with equal A activity (aA=aB). The Kpot A, B value is determined according to the following Eq. (3), respectively, after recording EA and EB values [29].

$$Log\left(K_{pot}\right) = \frac{EB-EA}{\frac{2.303RT}{zF}} + \left(1-\frac{Z_A}{Z_B}\right) log(aA) \qquad (5)$$

where EA and EB are potentials of the electrodes, Z_A and Z_B are charge numbers of ions, and Aa is activities of the principal A and interfering B ions when $a_A = a_B$

Separate Solution Method (SSM) Works The electrode's potential is measured in a series of solutions with varying concentrations of the primary ion and, separately, in solutions with varying concentrations of each interfering ion. Using the measured electrode potentials and the known concentrations of the ions, the potentiometric selectivity coefficient (K) is calculated.

The selectivity coefficient (K) is a measure of the relative response of the electrode to the interfering ion compared to the primary ion. A lower K value indicates better selectivity [34].

The results indicated that the selectivity coefficients depended on the concentration and composition of the electrodes, which in turn depended on the charges of the ions or substances. All values are listed in table (4). The selectivity headed for the species measured is described in Fig. (5).

The results given in table (4), and Fig. (5) showed that in case of selectivity coefficient less than one, the electrodes will provide a good response, while if the selectivity coefficient is more than one, it will lead to idea that the response of the electrode will convert towards the interfering ion rather than the analytic. The electrodes (acids and salts) showed excellent selectivity for the interfering ions.

Selectivity is quantitatively demonstrated by the selectivity factor (K). It is usually offered in a logarithmic notation, as log (K). In this investigation, the results of primary ion selectivity coefficients and interfering ions such as Al^{3+}, Zn^{2+}, Pb^{2+}, and Na^{1+}, have been obtained. In addition, the concentration, composition of the electrodes, and the charges of both the principal ion and the interfering ions. According to the results, Pb was the element that selected the interfering ions the best, followed by Na, Al, and Zn.

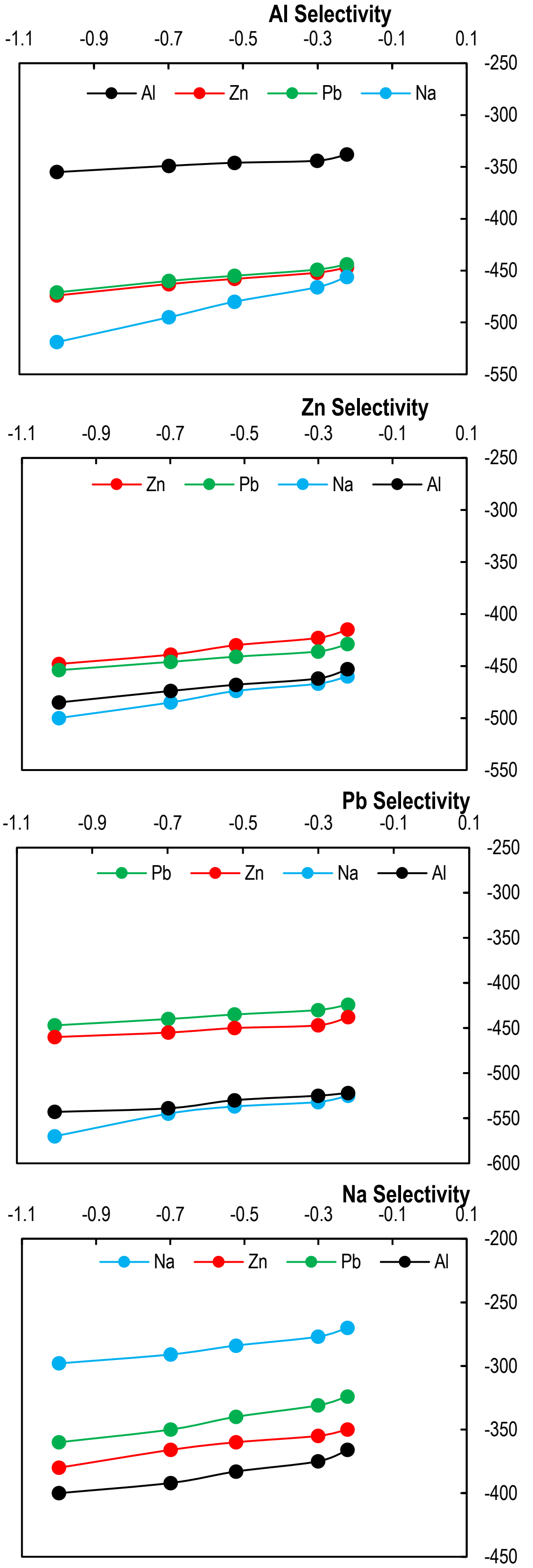


Fig. (5) Selectivity of (Al, Zn, Pb, and Na) ion electrodes with interfering ions via the Separation Solution Method (SSM)

4. Conclusions

This work demonstrated designed membranes for ISEs of (Al, Zn, Pb, and Na) ions to fabricate electrochemical (ISE) sensors for the detection of water pollution with heavy metals for the first time. It has been shown to have high selectivity in detecting heavy metals in water. The limits of detection of Al, Zn, Pb, and Na were 0.0894, 0.819, 0.0037, and 16.73, and the correlation coefficients were about 0.9595, 0.9627, 0.9744, and 0.9595, and the lifetimes were approximately 24, 25, 25, and 30 days, respectively. An interesting characteristic of the sensor was the system's small size, inexpensive materials, and ability to measure speed and accuracy. Therefore, the electrochemical sensor can be applied to quantitatively determine heavy metal ions in river water samples with satisfactory results.

Acknowledgments

The authors are grateful to Professor Dr. Yehya Kamal AL-Bayati from the Department of Physics in the College of Science, University of Baghdad for their fruitful help during the work.

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Table (1) The precise proportions of the electrode fabrication

Ions	Ions size (g)	Plasticizer (TEHP) (g)	PVC (g)	THF (mL)	specific ion exchanger used
Al^{+1}	0.04	0.4	0.25	7 - 9	Al-selective ionophore
Zn^{+2}	0.04	0.4	0.25	7 - 9	Zn-selective ionophore
Pb^{+2}	0.04	0.4	0.25	7 - 9	Pb-selective ionophore
Na^{+1}	0.04	0.4	0.25	7 - 9	Na-selective ionophore

Table (4) Selectivity coefficients for (Al, Zn, Pb, and Na) ions at the electrode at different concentrations of interfering ions

Concentration (M)	Concentration of Al ions (M): Concentration of interference ions (M)						
	Al^{+3}	Interfering ions					
		Zn^{+2}		Pb^{+2}		Na^{+1}	
	E_b (mV)	E_b (mV)	$K_{A,B}$	E_b (mV)	$K_{A,B}$	E_b (mV)	$K_{A,B}$
0.370×10^{-5}	-358	-474	0.000458448	-471	0.000572881	-519	0.000512481
0.741×10^{-5}	-349	-463	0.000469965	-460	0.000587273	-495	0.000487823
1.111×10^{-5}	-346	-458	0.000445181	-455	0.000556302	-480	0.00052866
1.483×10^{-5}	-344	-452	0.000464135	-449	0.000579987	-466	0.000464062
1.853×10^{-5}	-336	-447	0.000393365	-444	0.000491552	-456	0.000433756

Concentration (M)	Concentration of Zn ions (M): Concentration of interference ions (M)						
	Zn^{+2}	Interfering ions					
		Pb^{+2}		Na^{+1}		Al^{+3}	
	E_b (mV)	E_b (mV)	$K_{A,B}$	E_b (mV)	E_b (mV)	$K_{A,B}$	E_b (mV)
$0.\ 153\times 10^{-4}$	-448	-450	0.861953566	-490	0.044173447	-485	0.064040043
0.4588×10^{-4}	-439	-445	0.640400427	-480	0.015859814	-474	0.107153944
0.765×10^{-4}	-430	-441	0.44173447	-470	0.010249612	-468	0.101667829
1.07×10^{-4}	-423	-436	0.380754602	-461	0.008493672	-464	0.0910162
1.3766×10^{-4}	-417	-429	0.353498111	-454	0.006133283	-457	0.091884473

Concentration (M)	Concentration of Pb ions (M) : Concentration of interference ions (M)						
	Pb^{+2}	Interfering ions					
		Zn^{+2}		Na^{+1}		Al^{+3}	
	E_b (mV)	E_b (mV)	$K_{A,B}$	E_b (mV)	$K_{A,B}$	E_b (mV)	$K_{A,B}$
0.483×10^{-7}	-447	-460	0.367466194	-570	0.0076964	-543	0.000132627
0.965×10^{-7}	-440	-455	0.31501248	-545	0.015390911	-539	0.000132629
1.447×10^{-7}	-435	-450	0.291664078	-537	0.01292729	-530	0.000191281
1.930×10^{-7}	-430	-447	0.270046235	-532	0.009695468	-525	0.000227385
2.413×10^{-6}	-424	-438	0.250030685	-525	0.00837729	-522	0.000142874

Concentration (M)	Concentration of Na ions (M): Concentration of interference ions (M)						
	Na^{+1}	Interfering ions					
		Zn^{+2}		Pb^{+2}		Al^{+3}	
	E_b (mV)	E_b (mV)	$K_{A,B}$	E_b (mV)	$K_{A,B}$	E_b (mV)	$K_{A,B}$
0.4349×10^{-2}	-238	-380	0.000262636	-360	0.001160155	-400	0.000128093
0.6525×10^{-2}	-231	-366	0.000541012	-350	0.001775562	-392	0.000180792
0.8699×10^{-2}	-224	-360	0.000579987	-340	0.002562002	-383	0.00025409
1.087×10^{-2}	-217	-355	0.00055893	-331	0.003323155	-375	0.00031758
1.3049×10^{-2}	-210	-350	0.000527754	-324	0.003640334	-366	0.00041606