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Synthesis and Characterization of New Zn-ion Selective Membranes as Sensors for Environmental Monitoring

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ABSTRACT

Ion-selective electrodes (ISEs) made from Zn^{2+} ion-selective membranes were synthesized for use as potentiometric electrochemical sensors for water pollution detection with heavy metals for the first time. The electrode consisted of Zn^{2+} ions as an ion exchanger and polyvinyl chloride and tris (2-ethylhexyl) phosphate as a plasticizer. The response properties of this electrode, UV-visible spectra of the sample, calibration curve, lifetime, limit of detection, linear range, and pH effect were studied. The limit of detection (LOD) for Zn^{2+} was 0.891 ppm, the correlation coefficient was about 0.983, the lifetime was approximately 25 days, and the linear range was from $0.153 \times 10^{-4}\ to\ 1.07 \times 10^{-4}$. The best sensitivity was observed at the pH range of 5–10. It offers a robust, rapid potentiometric sensor for zinc in field water. The system's small size, inexpensive materials, and ability to measure speed and accuracy make it ideal. An interesting characteristic of the sensor was that a short time was required to reach the equilibrium potential.

Keywords: Ion-Selective electrode, PVC membrane, Environmental monitoring, Water pollution, Heavy metals.

1. INTRODUCTION

Ion-selective electrodes (ISEs) are among the most frequently used potentiometric sensors in laboratory analysis; they are also employed in various applications, including physiological measurements, industrial process control, drug analysis, and environmental monitoring **(AL-Bayati et al., 2015; Ahluwalia, 2015)**. ISEs are responsive ionic species, and they are divided into (a) crystalline membranes, which include two types, namely, single and poly or mixed crystal, and (b) nanocrystalline membranes, which comprise three types (glasses, neutral carriers, liquid ion exchangers, and immobilized liquid in a rigid polymer) **(Crespo, 2017)**.

ISEs and miniaturized sensors are chemical analytical devices that can provide information about the presence of a specific molecule or ion in complex samples. Typically, the

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recognition process analysis occurs, converting the chemical change into an electrical or optical signal **(Nakamura, 2009; Joo et al., 2008; Frag et al., 2011)**. Ion-selective membranes are classified into four types: glass membranes, crystalline membranes, enzyme electrodes, and polyvinyl chloride (PVC) membrane electrodes **(Mohan et al., 2021)**.

A sensor is a transducer that converts a physical quantity into an electrical quantity for measurement purposes. It transforms the input chemical quantity, which ranges from the concentration of components of a specific sample to an exhaustive analysis of composition, into an electrical signal. Chemical information is generated from a biomaterial chemical reaction, a chemical compound, or both, from the physical transducer's surface to the analyte. The study of chemical sensors is a new discipline that draws on multidisciplinary fields, including biology, chemistry, optics, electricity, acoustics, mechanics, semiconductors, microelectronics, thermodynamics, and membrane technology **(Kukharchuk et al., 2021)**.

Recently, potentiometric membrane sensors have been widely employed in electrochemical analysis **(Abu-Shawish, 2011; Kormosh et al., 2008; Dimeski, 2010)**. This is primarily due to the selective electrodes' low cost, straightforward design, broad linear concentration range, low detection limit, sufficient selectivity, excellent precision, and suitability for use with turbid and colored solutions **(Bakhtiarzadeh et al., 2008)**. One of the most used potentiometric sensors is the ion-selective electrode (ISE), which is used in industry, process control, drug analysis, physiological measurement, environmental monitoring, and laboratory analysis **(Sayhood et al., 2010)**. The electrochemical sensor involves an electrochemical reaction between the target, a detectable analyte, and the receptor on the transducer surface, generating a measurable electrical signal, either potential (at constant current) or current (at constant potential). With high sensitivity, speed, and low cost, these sensors have gained considerable attention from analysts **(Banasiak et al., 2018; Al-Azzawi et al., 2023; Bi, 2019; Gautam et al., 2021)**.

Water pollution is one of the significant concerns that endangers the lives of both people and animals. The degradation of ecosystems by hazardous heavy metals and organic contaminants continues to rise due to human activities **(Sleimi et al., 2022; Kurniawan et al., 2023)**. Since water carries numerous pollutants, it is the "trouble spot" of all ecosystems. It also plays a crucial role in the air–plant–soil–animal cycle as a medium and a solvent for various substances **(Usiabulu et al., 2023; Mumbi et al., 2022)**.

Heavy metals are metallic elements with a high density and are poisonous at low and high concentrations. "Heavy metal" refers to a collection of metals with an atomic density greater than 4 g/cm^3, more than five times that of water **(Justino et al., 2017)**.

Heavy metals are divided into two classes, namely, essential and nonessential, based on their role in living systems: (a) Basic heavy metals are required by living organisms for their growth and development, (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 nonessential heavy metals, including arsenic (As), Cadmium (Cd), lead (Pb) and mercury (Hg), for pathological functions **(Al Azzawi et al., 2023)**.

The pH value is a numerical indicator of how basic or acidic aqueous or other liquid solutions are. The hydrogen ion concentration in pure water, which is neutral (neither acidic nor alkaline), is (10 - 7) gram equivalents per liter, or a pH of 7. A solution is deemed basic, or alkaline, if its pH is greater than 7 and acidic if its pH is less than 7. Tris(2-ethylhexyl) phosphate (TEHP) is a commonly used plasticizer, 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 **(He et al., 2023)**.
This study aimed to fabricate an electrochemical (ISE) sensor for detecting water pollution with heavy metals, using molecular polymers to produce membranes for ISEs of Zn ions.

2. CHEMICAL MATERIALS

The following materials were used to prepare the ion-selective membrane. Metal ions (Zn^{2+} standard solution 1000 µg/ml (Spectro ECON)) were obtained from Belgium. Tetrahydrofuran (THF) (C_4H_8O; 72.11 g/mol), BASF Company, and PVC (Fluka AG, 98%). The plasticizers used in the composition and viscosities were tris(2-ethylhexyl) phosphate (Fluka AG) & UV Spectroscopy Model (UV-1800 PC) SHIMADZU (Japan), hydrochloric acid (HCl) (SIGMA, 99.9%), and ammonium hydroxide (NH_4OH) (Laboratory Reagent: approximately 25% NH_3). Reference electrode single junction (Saturated calomel electrode (SCE) ORION, model 90-01.

3. EXPERIMENTAL WORKS

3.1. Casting of the Membrane

Electrode membranes for environmental monitoring were prepared using the electroactive complexes of metal ions (Zn^{2+}) with a plasticizer. Zn ions were immobilized in the matrix membrane using the procedure outlined by **(Smaeel et al., 2020)**, **Fig. 1.** A total of 0.04 g Zn^{2+} ions were combined with 0.4 g plasticizer and 0.25 g PVC powder. Afterward, 7–9 mL THF was added by stirring until a sticky solution was obtained. The solution was poured into a two-piece glass casting assembly consisting of a glass cylinder (30 mm high and 35 mm in diameter) to which a glass plate was attached. The two pieces were then glued together using a viscous PVC-THF mixture to prevent loss of the membrane mixture. The upper surface of the cylinder was covered with a filter paper pad, upon which a substantial weight (about 200 g) was positioned. The mixture was allowed to stand for 2–3 days to facilitate slow evaporation **(AL-Bayati et al., 2016; Abd Jaber et al., 2020)**.

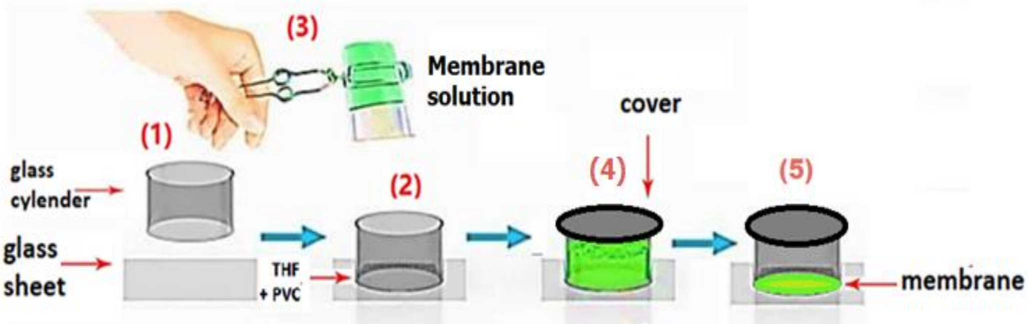

Figure 1. Formation of the ion-selective membrane.

3.2. Assembly of the ISE

To assemble the ISEs, we carefully removed the glass ring with the adherent membrane from the glass plate. The membrane was then separated from the border of the ring. A membrane disc was sliced to the same diameter as the exterior of the PVC tube. Vertical rotation on a glass plate soaked with THF-PVC was used to flatten and smooth one side of the PVC tube. The membrane disc was then put on the polished end, and its external edge was gently sealed to the end of the PVC tube. To link the plastic cap to the glass tube, we attached it to the opposite side of the tube and inserted a silver wire coated with silver chloride (Ag/AgCl) through it. The electrode was then activated by immersing it in a water solution containing 9 ppm Zn ions for at least two hours before use **Fig. 2**. **(Nassory et al., 2008)**.

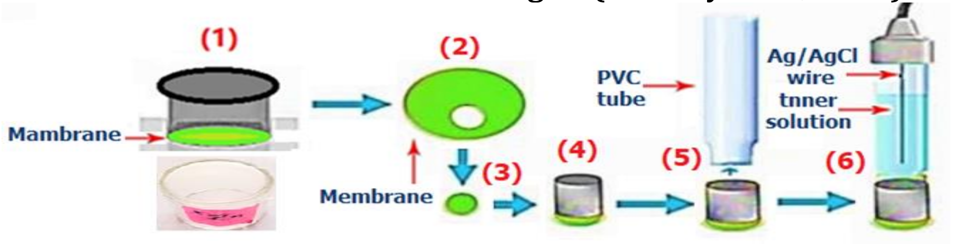

Figure 2. Assembly of the ISE.

The working electrode (WE) was the central electrode in the electrochemical cell. It can have different configurations and compositions, offering various potential ranges. It can be made from carbon, silver (Ag), Platinum (Pt), mercury (Hg), Nickel (Ni), zinc oxide (ZnO), etc. **(Ghosh, 2022)**. The silver wire coated with silver chloride (Ag/AgCl) and calomel electrodes are the most popular reference electrodes (RE); they have a constant potential that does not change with the applied potential in the WE and are not sensitive to the composition of the target analyte.

3.3. Cell Design of ISE

An ISE cell consists of a reference electrode (RE) and an ISE. The cell generally comprises two “internal” and “external” REs and a selective thin film or membrane as the recognition/transduction element. The cell was designed under the basic rule for designing electrolytic cells, provided that the current passing through the electrolytic cell was equal to zero **Fig. 3**. This cell consisted mainly of two electrodes: a working electrode (WE) and a reference electrode (RE) **(Ismaeel et al., 2020)**. The WE potential changed in response to the target sample, whereas the RE remained a fixed potential independent of the target analyte properties. In potentiometric measurement, the potential difference at a constant current) was measured when both electrodes were immersed in an electrochemical cell containing the solution. The change in the potential was proportional to the concentration of a specific analyte in the solution. The exchange between internal and external solutions across the membrane was determined through ionic exchange.

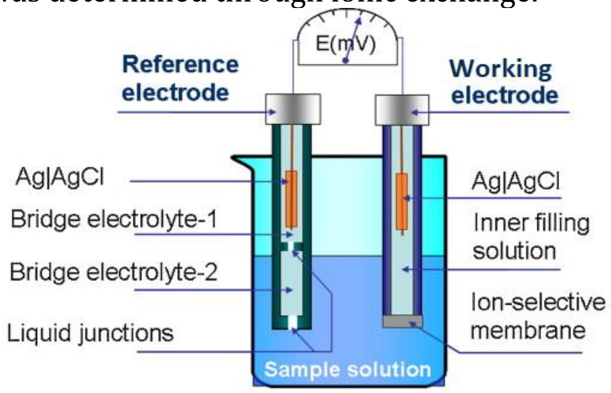

Figure 3. Basic components of the ISE cell **(Bobacka, 2024)**

4. RESULTS AND DISCUSSION

4.1. Standard Sample Analysis

4.1.1. Ultraviolet-Visible (UV-Vis.) Spectrophotometric Method

A dual-beam UV-Vis spectrometer (UV-1650 PC model, Shimadzu, Japan) connected to a computer via Shimadzu UV Probe Data System software (version 1.10) with 1.00 cm quartz cells was used to record the UV-Vis spectrum of the synthesized film. The wavelength of the UV-Vis ranged from 100 nm to 400 nm. UV radiation has sufficient energy to elevate valence electrons in several atoms or molecules from stable to higher-energy states. The excited electrons shifted from a bonding to an antibonding orbital. Saturated compounds with unshared electron pairs, such as nitrogen, oxygen, Sulfur, and halogens, can absorb UV radiation at n-δ* transitions (150–250 nm) **(Khoshayand et al., 2008)**. A UV-visible spectrophotometer was used to test the material's capability to absorb Zn ions of different concentrations (ppm) to use it as a WE to test the membrane function, **Fig. 4.**

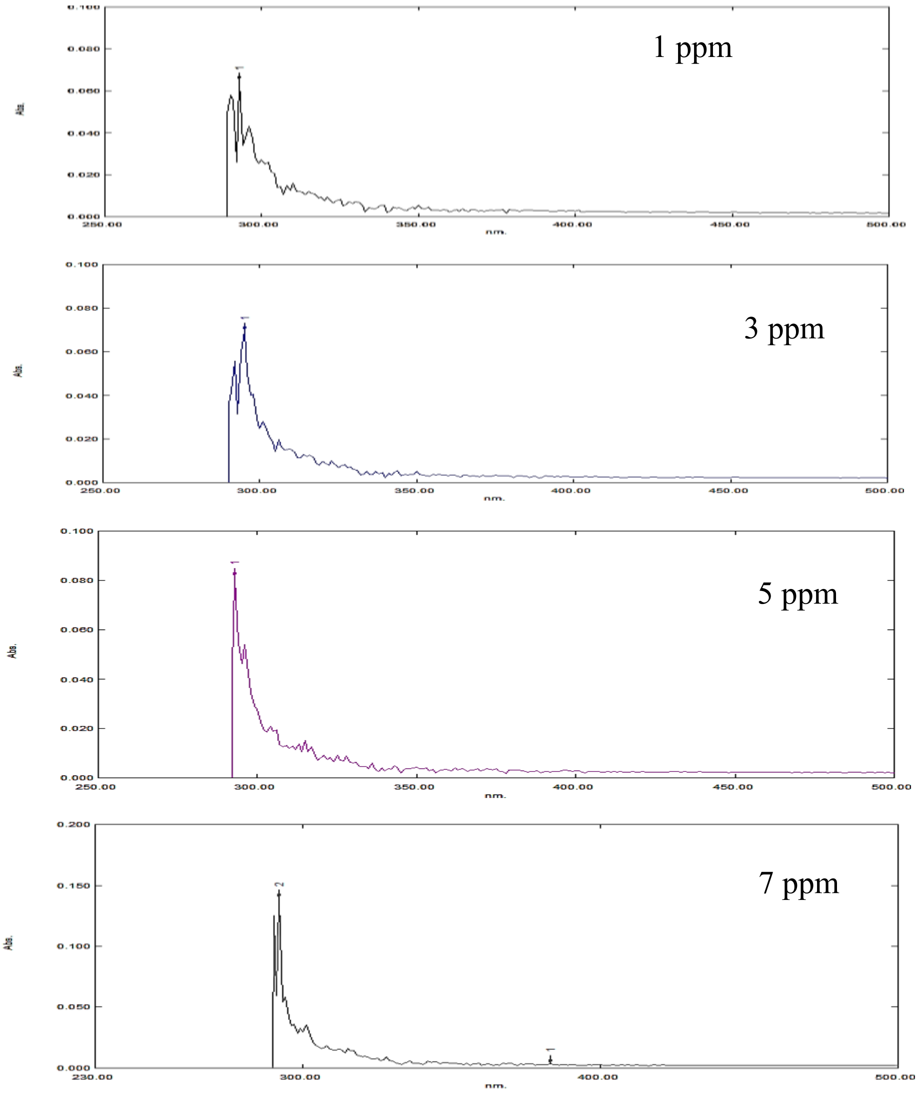

Figure 4. The capability of the material to absorb Zn^{2+} ions (ppm).

Fig. 4 reveals that the material's absorption capacity increased with the concentration of Zn ions, which helped us learn about the membrane's responsiveness. The Zn ions were prepared at (1, 3, 5, and 7 mg/L) from a stock solution of 1000 mg/L. The requisite dilution using distilled water was performed following Eq. (2) **(O'Connor, 2021)**.

For acids and bases, the following was used:

$$N_1V_1 = N_2V_2 \tag{1}$$

Where N_1 indicates the focus of Zn ion solution (1000 mg/L), V_1 the Zn ion solution (ml), N_2 means the focus of Zn ions (1, 3, 5, and 7) mg/L, and V_2 is the volume obtained of Zn ions (ml).

The concentrations were converted from ppm to molar using equations in the Zn ion electrode calibration curve, with TEHP as a plasticizer, **Table 1. Fig. 5** shows the calibration curve of the Zn ion electrode**.**

$$CapM = \frac{ppm}{A.wt\ \times 1000} \tag{2}$$

Table 1. ISE sensor for different Zn^{2+} ion concentration responses or potential.

Concentration (mg/L)	Concentration (Mole/L)	e.m.f (mv)
1	0.0000153	-440
3	0.00004588	-428
5	0.0000765	-418
7	0.000107	-413

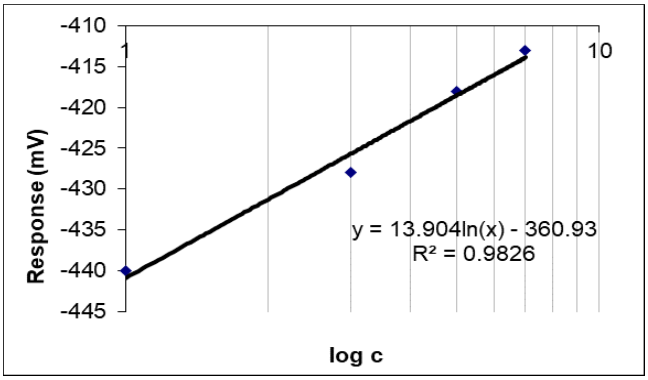

Figure 5. Calibration curve of Zn^{2+} ion electrode

The Potential (e.m.f.) is resulting from the fabricated electrochemical sensor and the concentration (C) of ions and salts. The limit of detection (LOD) is the lowest concentration or quantity of a substance that can be reliably identified and distinguished **(Lambarki et al., 2025)**.

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

$$SD = \frac{3\ SD}{Slope} \tag{3}$$

Where SD: Standard deviation

Table 2. The potentiometric properties of the Zn^{2+} electrode.

Properties of Zn Calibration	Electrode values
Linear range	$0.153 \times 10^{-4}\ to\ 1.07 \times 10^{-4}$
Slope	31.98
Limit of detection	0.891
correlation coefficient	0.983
Lifetime (day)	25

Fig. 5 and Table 2 illustrate the potentiometric properties for the (Cd, Pb, Na, and Ca) electrode. The sample concentration was calculated using Eq. (4), depending on the calibration curve plot between the electrode potential response (mV) versus the logarithmic concentration of the analyte (M). Consequently, any specified increase in the response was measured in millivolts, resulting from a corresponding rise in concentration. This condition allowed for the determination of concentration before and after the addition. The concentration of each sample was determined by extrapolating the x-axis of the calibration curve. **(Mirsky et al., 2011; Opik et al., 2009)**.

$$y = 13.904\ ln(x) - 360.93 \tag{4}$$

Eq. 4, which was obtained from the calibration curve in **Fig. 6**, was used to calculate the practical values recorded by the Zn ion electrode when applied to field water of the Tigris River at different zones and a depth of 4 m (in molarity unit). Eq. (5) was used to convert the experimental values from molarity units to ppm units, **Table 3**.

$$ppm = M \times A.wt \times 1000 \tag{5}$$

Table 3. The response of Zn^{2+} ions membranes to the used field water

(y) e.m.f (mv)	(X) Concentration (Mole/L)	Concentration (mg/L)
-521	1.002×10^{-5}	0.65
-528	0.608×10^{-5}	0.39
-530	0.524×10^{-5}	0.32
-540	0.249×10^{-5}	0.15

The values obtained from the calibration curve in **Fig. 5** show that the membrane responded well to detecting the presence of Zn ions, and the sensor stabilized after less than a minute of being inserted into the water to be measured. Compared with the international standard value for the percentage of Zn ions in water (3 ppm) **(Nigam et al., 2021; Hynes, 1999)**, the percentage of Zn ions in the water fell within permissible limits. All potentiometric measurements were conducted at a temperature of 23–25 °C. Temperature did not significantly alter the electrode response.

4.2. Effect of pH

pH plays a vital role in the response of the electrode membrane. The pH range is the range during which a change in pH won't significantly alter the potential that is being measured **(Slesinskl et al., 2022)**. The pH measurements were carried out, and the pH was adjusted by adding a few drops of standardized sodium hydroxide (NaOH) or hydrochloric acid (HCl) solution. The influence of pH on electrode responsiveness was investigated by measuring the potential variation in the electromotive force (e.m.f.) over a pH range of 1–11 for two distinct zinc ion concentrations (3 and 7 ppm). **Table 4** shows the results of the functioning pH ranges of the Zn ISEs. **Fig. 6** depicts a typical plot of the effect of pH on electrode responsiveness.

Table 4. Effective pH ranges used for Zn^{2+} ISEs.

pH	3 mg/L	7 mg/L
1	-313	-325
2	-368	-360
3	-388	-376
4	-394	-385
5	-394	-387
6	-394	-395
7	-394	-395
8	-395	-395
9	-397	-399
10	-399	-399
11	-400	-401

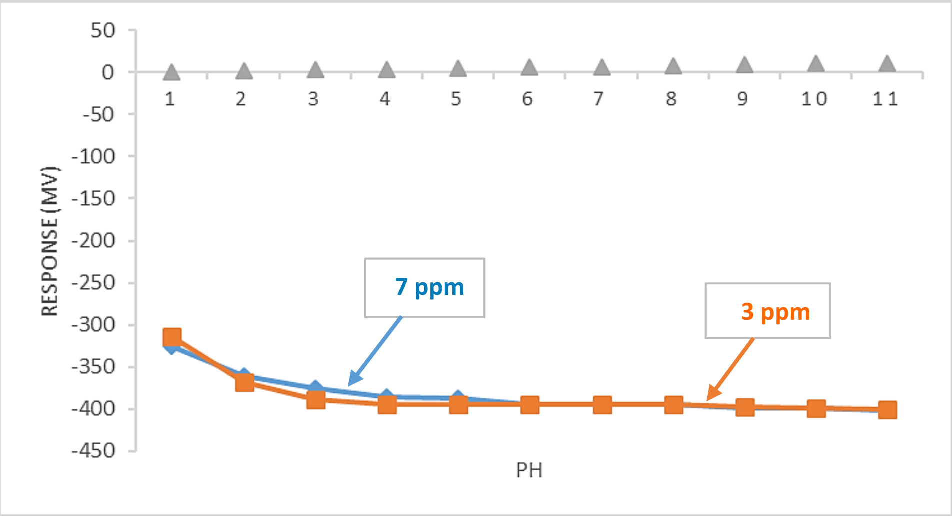

Figure 6. Effect of pH on the potential of Zn^{2+} ion electrodes at concentrations of 3 and 7 mg/L.

Fig. 6 shows that the Zn ion electrode works perfectly within the pH range of 5 to 10, while below pH 5, it works less efficiently. **Table 5** demonstrates a comparative analysis of this study with other studies **(AL-Azzawi et al., 2024)** based on their objectives, methodologies, materials, applications, and key findings. According to **Table 5**, combining the rapid response of PVC-based ISEs with the high sensitivity of biosensors could yield hybrid sensors for comprehensive environmental monitoring. Future work might explore integrating these technologies to leverage their respective strengths.

Table 5. Comparative analysis between ISE using a PVC member with an amperometric sensor.

Title	Zn-ion selective membrane sensor for environmental monitoring	Amperometric biosensor for Zn pollution detection
Objectives	Focused on developing a potentiometric ion-selective electrode (ISE) using a PVC membrane with Zn ions as the ion exchange for detecting Zn pollution in water.	Aimed to create an amperometric biosensor using alkaloids from (Catharanthus roseus) linked to chitosan nanoparticles (CSNPs) for Zn ion detection in water.
Methodologies	- Technique: Potentiometry. - Materials: PVC membrane with Zn ions, tris(2-ethylhexyl) phosphate (TEHP) plasticizer. - Process: Using UV-Vis spectroscopy and pH studies, membrane casting, electrode assembly, and calibration.	- Technique: Cyclic voltammetry (CV). -Materials: CSNPs-Linker-alkaloids compound, glassy carbon electrode (GCE). - Process: Synthesis of nanocomposite, electrode modification, and electrochemical characterization.
Sensor Type	Potentiometric ion-selective electrode (ISE) (synthetic membrane).	Amperometric biosensor (biological recognition element).
Environmental and Practical Implications	Synthetic PVC membrane (robust but non-biodegradable).	Eco-Materials: Uses plant-derived alkaloids (eco-friendly).
Applications	Practical for routine monitoring of Zn in rivers/industrial effluents due to broad pH tolerance and rapid response.	Suitable for trace Zn detection in polluted water, with potential for biocompatible environmental and biomedical applications.
Conclusion	It offers a robust, rapid potentiometric sensor for zinc with enhanced pH adaptability, making it ideal for industrial applications. The system's small size, inexpensive materials, and ability to measure speed and accuracy make it ideal.	It provides an eco-friendly, highly sensitive biosensor suited for trace detection and biocompatible uses, albeit with more complex preparation.

5. CONCLUSIONS

This work demonstrated the designed membrane for Ion-selective electrodes of Zn ions to successfully fabricate electrochemical (ISE) sensors for detecting water pollution with heavy metals for the first time. It was shown to have a high selectivity for detecting heavy metals in water. An interesting characteristic of the sensor was that a short time was required to reach the equilibrium potential. The effective pH of the Zn was investigated, with the best sensitivity in the pH range of 5 to 10. As a result, the electrochemical sensor can be applied to quantitatively determine heavy metal ions in river water samples with satisfactory results.

Credit Authorship Contribution Statement

Bilal A. Abdullah: Data Curation, Original Draft Preparation and Literature Review. Wasan R. Saleh: Manuscript review and editing,

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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تخليق وتوصيف أغشية انتقائية جديدة لأيونات الزنك كمستشعر لمراقبة البيئة

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الخلاصة

تم تصنيع أقطاب كهربائية انتقائية للأيونات (ISEs) مصنوعة من أغشية انتقائية للأيونات من Zn^{2+} لاستخدامها لأول مرة كمستشعرات كهروكيميائية جهدية للكشف عن تلوث المياه بالمعادن الثقيلة. يتكون القطب من أيونات Zn^{2+} كمبادل أيوني، وكلوريد البولي فينيل وفوسفات ثلاثي (2–إيثيل هكسيل) كمُلَيِّن. دُرست خصائص استجابة هذا القطب، وأطياف الأشعة فوق البنفسجية والمرئية للعينة، ومنحنى المعايرة، وعمره الافتراضي، وكشف الحد الأقصى، والمدى الخطي، وتأثير الرقم الهيدروجيني. بلغ حد الكشف 0.891 جزء في المليون، ومعامل الارتباط حوالي 0.983، وعمره الافتراضي حوالي 25 يومًا، وتراوح المدى الخطي بين 0.153×10^{-4} الى 1.07×10^{-4}. لوحظت أفضل حساسية عند نطاق الرقم الهيدروجيني(pH) بين 5 الى 10. يوفر هذا المستشعر مقياس جهد قوي وسريع للزنك في مياه الحقل. حجم النظام الصغير، ومواده منخفضة التكلفة، وقدرته على قياس السرعة والدقة تجعله مثاليًا. ومن الخصائص المميزة لهذا المستشعر قصر مدة الوصول إلى جهد التوازن.

الكلمات المفتاحية: القطب الانتقائي للأيونات، غشاء PVC، مراقبة البيئة، تلوث المياه، المعادن الثقيلة.