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Fabrication of Ion-Selective Electrodes (ISEs) for Detection of Some Metal Ions in Aqueous Solutions

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Ion-selective electrodes made from Cd, Pb, Na, and Ca ions were synthesized for use as a potentiometric electrochemical sensor of water pollution detection. The electrodes consisted of Cd, Pb, Na, and Ca ions as ion exchangers and polyvinyl chloride and tris(2-ethylhexyl) phosphate as plasticizers. The response properties of these electrodes, UV-visible spectra of samples, calibration curve, lifetime, limit detection, concentration range and pH effect were studied. The limit of detection of Cd, Pb, Na, and Ca were 0.000693, 0.007454, 36.5167, and 5.496729, respectively, and the correlation coefficients were 0.9482, 0.9803, 0.999, and 0.9861, and the life time approximately were 25, 25, 30, and 1 days, respectively. The best sensitivity was observed at a pH range of 6 to 10 for Cd and of 5 to 10 for Pb, while the electrodes of Na and Ca don't work within the specific pH range due to rapid electrode oxidation, which reduces their efficiency. The proposed electrodes were successfully applied in the detection of heavy metals in water.

Keyword: Ion-selective electrode; PVC membrane; Metals ions; Aqueous solution
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1. Introduction

Ion-selective electrodes (ISEs) are among the most frequently used potentiometric sensors in laboratory analysis; they are also used in applications such as physiological measurement, industrial process control, drug analysis, and environmental monitoring [1,2]. Recently, potentiometric membrane sensors have been widely employed in electrochemical analysis [3-5]. 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 [6]. 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 [7].

Potentiometric chemical sensors known as ion-selective electrodes work by creating an electrostatic potential difference across a membrane that divides into two phases when the electrode is submerged in an ion-containing solution that can be exchanged with the membrane. Therefore, a component that interacts chemically and reversibly with the determinant must be present in the membrane [8]. The logarithmic activities of these determinants in the solution determine the interfacial potential's linear magnitude [9].

An appropriate electrochemical cell can be used to detect the interfacial potential, which is impossible to do directly. The potential is measured at equilibrium when there is no current present. The development of ISEs has advanced quickly over the last four decades, and exciting new developments are currently in the works [10]. The most important advantage property of ISE was that they measure activity directly, not concentration. Electrodes would function well in colored or turbid samples so they were more effective than the spectrophotometric measurements. For most ISE the equilibrium state was reached in less than a minute, this rapidly made it suitable for kinetic studies and for monitoring change in flowing process streams. The equipment used were simple, and quite inexpensive [11-13].

The term "environmental pollution" refers to the contamination of the earth's and the atmosphere's biological and physical components to the point that it negatively impacts regular environmental processes [14,15].

Since a large amount of chemical pollutants infiltrate rivers, lakes, and wetlands, water pollution is the main type of pollution. When toxic chemicals are released into the environment, they can be carried by the air, water, and living things. They can also build up in the food chain and become a 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 [16]. Recently many researchers focused on the detection of water pollution by heavy metals. [17-20].

Heavy metals indicate that any metallic element has a high density and is 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 more, larger than water [21]. Heavy metals are divided into two classes: essential and non-essential according to their role in living systems: (a) Essential heavy metals are those living organisms need for their growth, development, and physiological functions like Mn, Fe, Ni, Cu, and Zn, (b) while living organisms do not need non-essential heavy metals like As, Cd, Pb, and Hg for pathological functions [22].

Due to the fact that 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 [23]. Water pollution is one of the problems that threaten the lives of the human body and animals alike. Because of human activity, the ecosystem is continuously becoming increasingly contaminated by organic pollutants and hazardous heavy metals [24].

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} g 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 [25].

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 [26]. Tris(2-ethylhexyl) phosphate (TEHP), dioctyl phthalate (DOP), and o-nitrophenyl octyl ether (NPOE) are all plasticizers, but they have different chemical structures, properties, and uses. TEHP is primarily used as a plasticizer and flame retardant in polyvinyl chloride (PVC) and other polymers, while DOP is a widely used plasticizer for PVC. NPOE is also used as a plasticizer, but it has specific applications in optodes and other specialized areas [26].

This work aims at the fabrication of ion-selective electrodes (ISEs) for the detection of some metal ions in aqueous solutions.

2. Experimental Works

The following materials were used: metal ions (Cd, Pb, Na, and Ca) of concentration 1000 ppm from Belgium, tetrahydrofuran (THF) for HPLC and UV spectroscopy SHIMADZU Model UV-1800 PC (Japan), 2.5 liters SRL, India, Polyvinyl chloride (PVC) (Fluka AG, 98%), reference electrode single junction (saturated calomel electrode (SCE) ORION, model 90-01). The plasticizers of composition and viscosities are Tris(2-ethylhexyl) phosphate, from Fluka AG, (HCl) (SIGMA; 99.9%), and ammonium hydroxide (NH_4OH) (Laboratory Reagent: about of 25% NH_3).

Electrode membranes for environmental monitoring were prepared using the electroactive complexes of metal ions (Cd, Pb, Na, and Ca) with a plasticizer. The immobilizing of the Cd, Pb, Na, and Ca ions into the matrix membrane was done using the procedure outlined by Davis et al. [27] as shown in Fig. (1). 0.04 g of Cd, Pb, Na, and Ca ions was combined with 0.4 g of plasticizer (TEHP) and 0.25 g of PVC powder. Thereafter, 7-9 mL of THF was added with a continuous stirrer until a sticky solution was obtained. The precise proportions of the electrode fabrication and specific ion exchanger used are shown in table (1). The solution was poured into two pieces of glass casting assembly. The first one (glass ring) was a glass cylinder (30 mm high and 35 mm in diameter), to which a glass plate (second one) was attached and the two pieces were glued together using a viscous (PVC-THF) mixture (to ensure there is no 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 allowed mixture to stand for 2-3 days to facilitate the slow evaporation of the mixture [28,29].

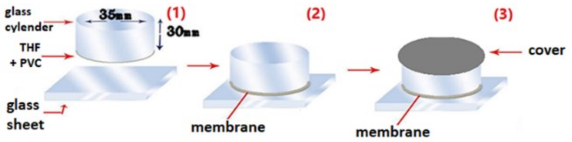


Fig. (1) Schematic explanation for the formation of the ion-selective membrane

Table (1) Precise proportions of the electrode fabrication

Ions	Ions Size (g)	Plasticizer (TEHP) (g)	PVC (g)	THF (mL)	Specific Ion Exchanger used
Cd^{2+}	0.04	0.4	0.25	7-9	Cd-selective ionophore
Pb^{2+}	0.04	0.4	0.25	7-9	Pb-selective ionophore
Ca^{2+}	0.04	0.4	0.25	7-9	Ca-selective ionophore
Na^{1+}	0.04	0.4	0.25	7-9	Na-selective ionophore

To assemble the ion-selective electrode (ISE); the glass ring with the adherent membrane were carefully removed from the glass plate. The membrane was then separated from the border of the ring. A disc of membrane was sliced to the same size as a PVC tube's exterior diameter. The membrane disc was then put on the polished end, and the disc membrane's outside edge was gently sealed to the end of the PVC tube, to link the plastic cap to the glass tube, a plastic cap was attached to the opposite side of the tube, and an Ag/AgCl wire was pushed through it. The electrode was then activated by immersing it in a water solution containing Cd, Pb, Na, and Ca ions for at least 2 hours before use, Emad and Al-Byati method [30], as shown in Fig. (2).

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

Design the cell according to the basic rule for designing electrolytic cells, provided that the current passing through the electrolytic cell is equal to (zero), as illustrated in Fig. (3). This cell consists mainly of two electrodes, working electrode (WE) (Ag/AgCl wire) and reference electrode (RE) (saturated calomel electrode) [27].

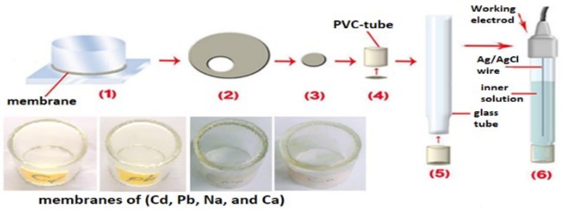


Fig. (2) Assembling the ion-selective electrode

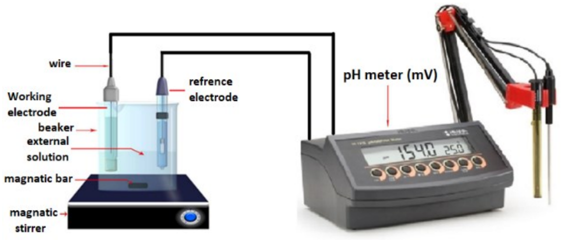


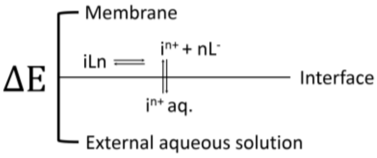
Fig. (3) Basic components of ion-selective electrode sensor

The main electrode in the electrochemical cell is the working electrode (WE), which can have different configurations and compositions that provide different potential ranges. It can be made from carbon (C), platinum (Pt), mercury (Hg), nickel (Ni), titanium (Ti), zinc oxide (ZnO), etc. [24].

The Ag/AgCl and calomel electrodes are the most popular reference electrode (RE), which has a constant potential that does not change with the applied potential in WE and is not sensitive to the composition of the target analyte. The internal filling solution of the working electrode (WE) contains the target ion at a known concentration.

The potential of the working electrode (WE) changes as a response to the concentration of ions in the field water, while the potential of the reference electrode is fixed independent of target analyte properties. In potentiometric measurements, the potential difference (at a constant current) is measured when both electrodes immersed used an electrochemical cell-containing solution. The change in the potential is proportional to the concentration of the specific analyte in the solution (water in our case), as shown in Fig. (3).

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 [31]



(1)

3. Results and Discussion

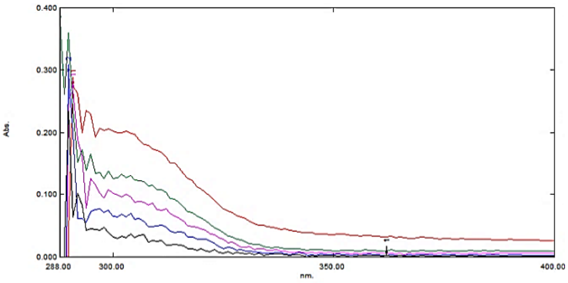
A SHIMADZU UV-1650 PC double-beam UV-visible spectrophotometer, interfaced with a computer via a SHIMADZU UV probe data system program (Version 1.10), was used with a 1cm quartz cells, to record the UV-visible spectra of the synthesized membranes. UV-visible spectroscopy was used to study the ability of the material under study to absorb Cd, Pb, Na, and Ca ions in different concentrations to use it as a working electrode to test the membrane function as shown in Fig. (4), which revealed that the absorption of the material increases with the increase in the concentration of Cd, Pb, Na, and Ca ions, and gives us an idea about the membrane's responsiveness.

The standard solutions of Cd, Pb, Na, and Ca ions were diluted from a standard of about 1000 ppm to the 0.001, 0.002, 0.003, 0.004, and 0.005 ppm for Cd, 0.01, 0.02, 0.03, 0.04, and 0.05 ppm for Pb, 100, 150, 200, 250, and 300 ppm for Na, 30, 40, 50, 60, and 70 ppm for Ca, using the equivalent law in Eq. (2). To completely neutralize the solution there must be equality between the normal distribution (N_1) of the acid and its volume (V_1) with the normal distribution (N_2) of the base and its volume (V_2) as in Eq. (2):

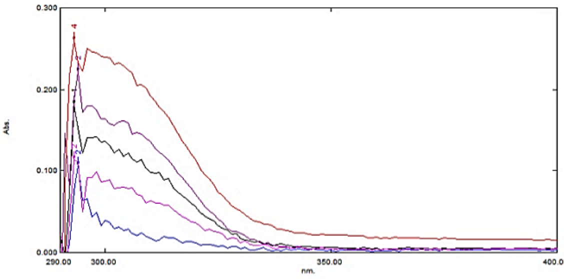
For acid and base

$$N_1V_1 = N_2V_1 \quad (2)$$

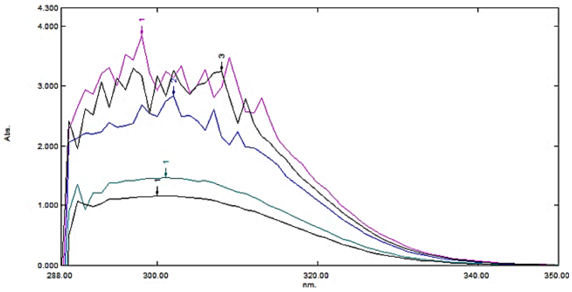
The sensor response to metals and salts was measured using the system in Fig. (3), through which the e.m.f. (eV) was measured at different concentrations. Table (2) summarizes the results of these measurements.



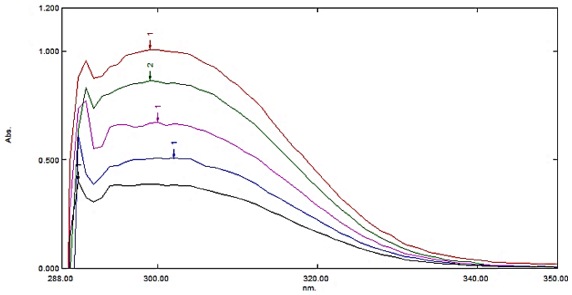
Cd



Pb



Na



Ca

Fig. (4) The absorption spectra of Cd, Pb, Na, and Ca ions in ppm unit

Table (2) ISE sensor response (potential) at different ion concentrations of Cd, Pb, Na, and Ca ions for standard samples

Concentration of Cd (ppm)	Calculated concentration (M)	e.m.f (mV)
0.001	0. 8896x10^{-8}	-428
0.002	1.779 x10^{-8}	-422
0.003	2.669 x10^{-8}	-417
0.004	3.558 x10^{-8}	-409
0.005	4.448 x10^{-8}	-406

Concentration of Pb (ppm)	Calculated concentration (M)	e.m.f (mV)
0.01	0.153x10^{-6}	-389
0.02	0.306 x10^{-6}	-382
0.03	0.459 x10^{-6}	-377
0.04	0.6118 x10^{-6}	-371
0.05	0.765 x10^{-6}	-368

Concentration of Na (ppm)	Calculated concentration (M)	e.m.f (mV)
100	0.43497x10^{-2}	-335
150	0.6525x10^{-2}	-326
200	0.8699x10^{-2}	-318
250	1.087x10^{-2}	-312
300	1.3049x10^{-2}	-307

Concentration of Ca (ppm)	Calculated concentration (M)	e.m.f (mV)
30	0.7485x10^{-3}	-341
40	0.998x10^{-3}	-337
50	1.2475x10^{-3}	-335
60	1.497x10^{-3}	-332
70	1.7465x10^{-3}	-327

Figure (5) demonstrates the calibration graphs which display the linear relation between sensor potential (e.m.f.) resulting from the fabricated electrochemical sensor and the concentration (C) of ions metal and salts.

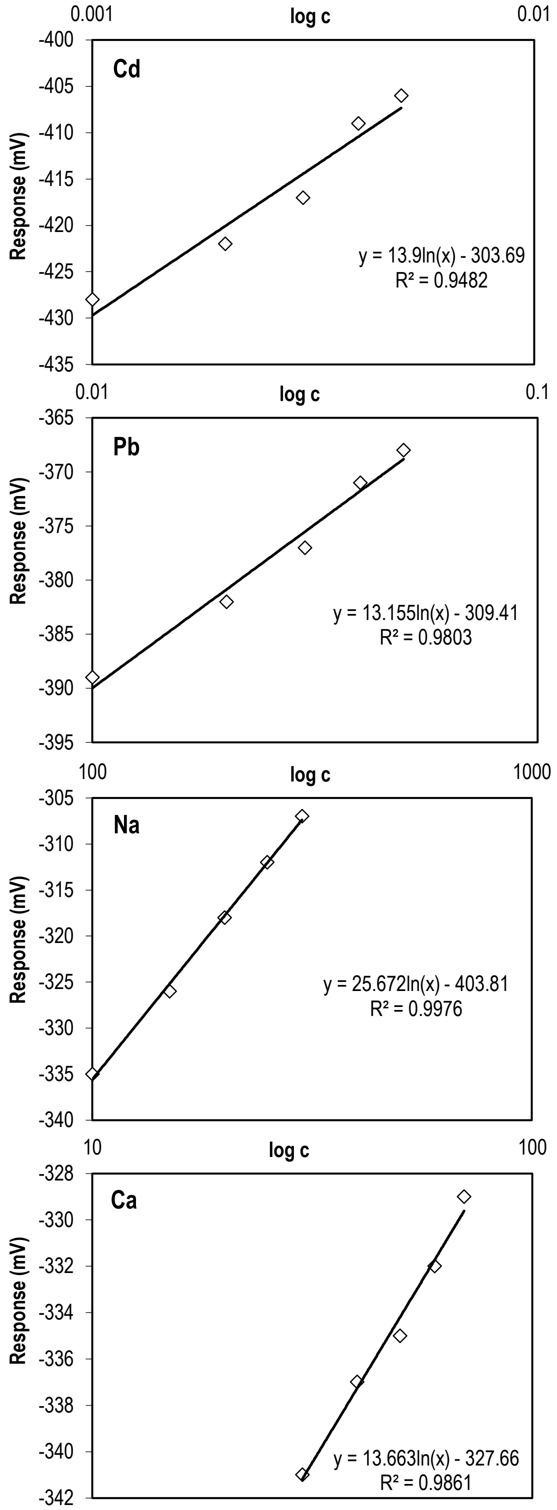


Fig. (5) Calibration curves of Cd, Pb, Na, and Ca ions sensor for standard samples

The limit of detection (LOD) is the lowest concentration or quantity of a substance that can be reliably identified and distinguished [32]. To calculate the detection limit value, we use the following equation: 3*SD*/*slope*, where *SD* is the standard deviation. Figure (5) and table (3) illustrate that the potentiometric properties for Cd, Pb, Na, and Ca electrode. Demonstrates the concentration of the ions field water is calculated using equations (4-7). Thus, any particular increase of response in mV due to a particular increase in the concentration (i.e., the slope of the curve) will only fit in one unique part of the curve and thus the concentration before and after addition can be determined. Each sample's concentration was determined by extrapolating the calibration line's x-axis [33,34].

For Cd ions: $y = 13.9\ ln(x) - 303.69$ (4)
For Pb ions: $y = 13.155\ ln(x) - 309.41$ (5)
For Na ions: $y = 25.672\ ln(x) - 403.81$ (6)
For Ca ions: $y = 13.663\ ln(x) - 327.66$ (7)

where y and x represent the e.m.f (eV), and concentration of ions (molarity), respectively

Table (3) Potentiometric properties for Cd, Pb, Na, and Ca electrodes

Properties of Cd Calibration	Electrode values
Linear range	$0.9x10^{-8}$ to $4.5x10^{-8}$
Slope	**31.97**
Limit of detection	0.000693
correlation coefficient	**0.9482**
Lifetime (days)	**25**

Properties of Pb Calibration	Electrode values
Linear range	$1x10^{-7}$ to $7.5x10^{-7}$
Slope	**30.2565**
Limit of detection	0.007454
correlation coefficient	**0.9803**
Lifetime (days)	**25**

Properties of Na Calibration	Electrode values
Linear range	$4x10^{-3}$ to $14x10^{-3}$
Slope	**59.0456**
Limit of detection	36.5167
correlation coefficient	**0.999**
Lifetime (days)	**30**

Properties of Ca Calibration	Electrode values
Linear range	$6.3x10^{-4}$ to $17.5x10^{-4}$
Slope	**31.4249**
Limit of detection	5.496729
correlation coefficient	**0.9861**
Lifetime (days)	**1**

The equations (4-7) were taken according to the calibration curves in Fig. (5), and used to calculate the practical values recorded by the Cd, Pb, Na, and Ca ions electrode when applied to field water of Tigris River at different zones and depth of 4 m in molarity unit. Equation (8) was used to convert the experimental values from the molarity unit to ppm unit, and the result is listed in table (4)

$ppm = M \times A.wt \times 100$ (8)

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

Table (4) The response of Cd, Pb, Na, and Ca ions membranes for the field water

e.m.f. (mV) (y)	Calculated Concentration (M) (x)	Concentration (ppm)
	Cd	
-483	$0.2498x10^{-5}$	0.225
-477	$0.358x10^{-5}$	0.402
-473	$0.4444x10^{-5}$	0.499
-468	$0.\ 6363x10^{-5}$	0.715
	Pb	
-381	$0.216x10^{-5}$	0.448
-378	$0.272x10^{-5}$	0.564
-375	$0.294x10^{-5}$	0.615
-370	$0.432x10^{-5}$	0.715
	Na	
-535	$0.783x10^{-2}$	180
-531	$0.919x10^{-2}$	211
-528	$0.975x10^{-2}$	224
-525	$1.1x10^{-2}$	253
	Ca	
-425	$0.806x10^{-3}$	32.3
-430	$0.559x10^{-3}$	22.4
-433	$0.448x10^{-3}$	17.95
-437	$0.335x10^{-3}$	13.42

According to the values obtained from the calibration curves in Fig. (5), the membrane responds well to detect the presence of Cd, Pb, Na, and Ca ions in the river water. When compared to the international standard value for the percentage of Cd, Pb, Na, and Ca ions in water 0.003, 0,01, 200, and 50 ppm [35,36], it becomes clear that the percentage of Cd, Pb, Na, and Ca ions in the water falls within the permissible limits. All potentiometric measurements were conducted at a temperature of 23-25 °C. Temperature did not significantly alter the electrode response.

The pH value plays an important role in the response of the electrode's membrane [34]. The pH range is the range during which a change in pH won't significantly alter the potential that is being measured.

The pH is measured using two pH meter devices, one recording the e.m.f. reading of the electrode and the other recording the pH reading. The pH measurement was carried out at room temperature and adjusted by introducing a few drops of normalized ammonia hydroxide (NaOH) and hydrochloric acid (HCl) solution to have basic and acidic solutions. A 10 ml of two different concentrations of standard solution ions are taken: 0.002 and 0.004 ppm for Cd, 0.02 and 0.04 ppm for Pb, 250 and 150 ppm for Na, and 40 and 60 ppm for Ca. The e.m.f. reading is recorded for each pH number from 1 to 11 for the first focus; the same process is repeated for the second concentration. Then the matching reading is known. This process determines the acidic range in which the electrode operates. The results of the working pH ranges of the Cd, Pb, Na, and Ca ion-selective electrodes are listed in table (5). Figure (6) shows the effect of pH on the electrode response. The listed concentrations were chosen with tolerance from the global concentration values.

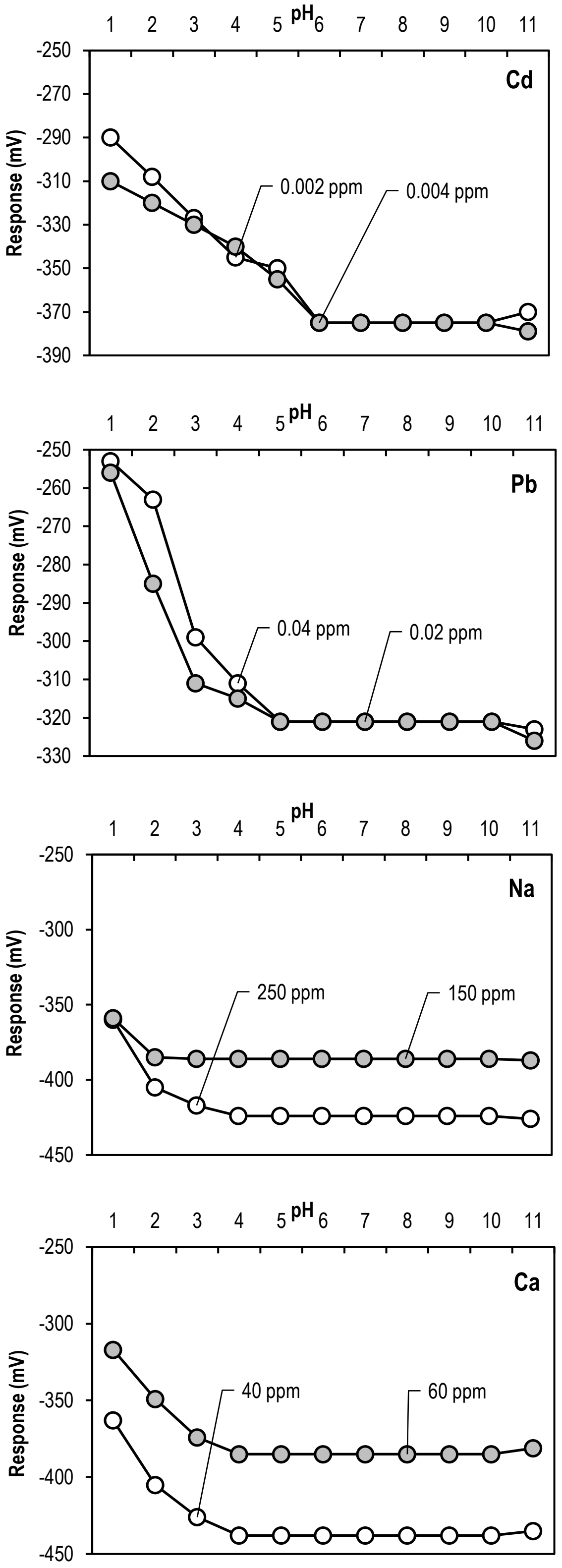


Fig. (6) Effect of pH on the response of the Cd, Pb, Na, and Ca ion selective electrodes

Table (5) Effective pH ranges on the response of Cd, Pb, Na, and Ca ion selective electrodes

pH	Response at a certain concentration of Cd (mV)	
	0.002 ppm	0.004 ppm
1	-290	-310
2	-308	-320
3	-327	-330
4	-345	-340
5	-350	-355
6	-375	-375
7	-375	-375
8	-375	-375
9	-375	-375
10	-375	-375
11	-370	-379

pH	Response at a certain concentration of Pb (mV)	
	0.02 ppm	0.04 ppm
1	-253	-256
2	-263	-285
3	-299	-311
4	-311	-315
5	-321	-321
6	-321	-321
7	-321	-321
8	-321	-321
9	-321	-321
10	-321	-321
11	-323	-326

pH	Response at a certain concentration of Na (mV)	
	150 ppm	250 ppm
1	-360	-359
2	-405	-385
3	-417	-386
4	-424	-386
5	-424	-386
6	-424	-386
7	-424	-386
8	-424	-386
9	-424	-386
10	-424	-386
11	-426	-387

pH	Response at a certain concentration of Ca (mV)	
	40 ppm	60 ppm
1	-363	-317
2	-405	-349
3	-426	-374
4	-438	-385
5	-438	-385
6	-438	-385
7	-438	-385
8	-438	-385
9	-438	-385
10	-438	-385
11	-435	-381

These figures demonstrate that the electrode of Cd ions works within the pH range of 6 to 10, and the electrode of Pb works within the pH range of 5 to 10. The electrodes of Na and Ca don't work within the specific pH range due to rapid electrode oxidation, which reduces their efficiency.

4. Conclusions

This work demonstrated designed membranes for ISEs of Cd, Pb, Na, and Ca ions to successfully fabricate electrochemical (ISEs) sensors for detecting water pollution with heavy metals for the first time. It was shown to have a high selectivity to detect 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 electrodes was investigated, with the best sensitivity of acids 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.

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