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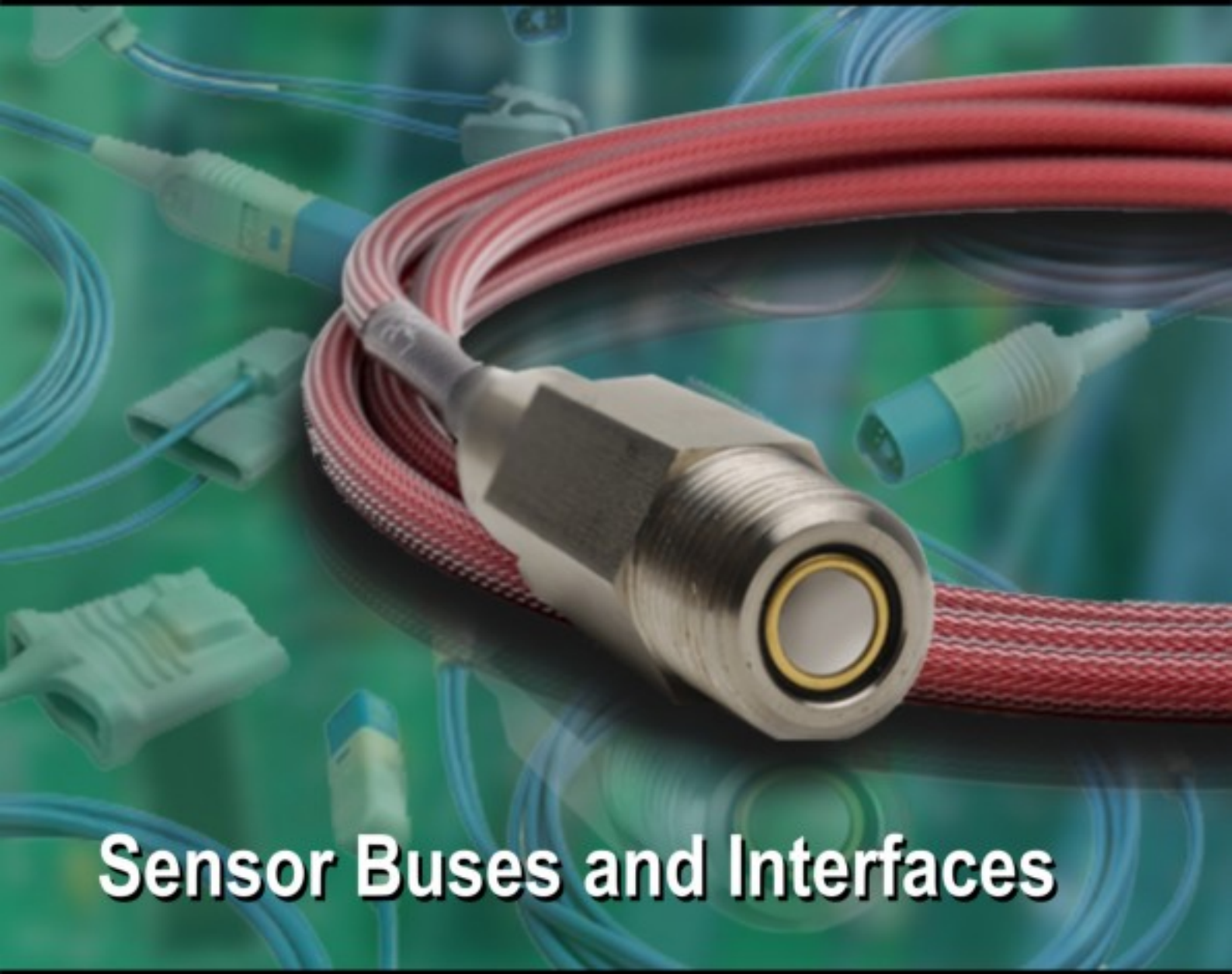

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A Cadmium Ion-selective Membrane Electrode Based on Strong Acidic Organic-Inorganic Composite Cation-Exchanger: Polyaniline Ce(IV) Molybdate

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Abstract: A cadmium ion-selective composite cation-exchanger polyaniline Ce(IV) molybdate was used as electroactive component for the construction of an ion-selective membrane electrode. The membrane electrode showed a Nerstian response for Cd(II) ions over a wide concentration range $5 \times 10^{-6} – 1 \times 10^{-1}$ with a sub-Nerstian slope of 27 mV per decade change in concentration of cadmium ions. The limit of detection was also ascertained to be 5×10^{-6} M. It has a fast response time 15 s and can be very well utilized for more than three months with out any appreciable divergence in potentials. The optimum pH for the smooth functioning of this electrode was found to be in the Ph range of 2.5 – 7.5. The electrode also showed better selectivity for Cd(II) ions over many other interfering ions. The practical utility of membrane electrode was demonstrated by using as indicator electrode for the potentiometric titration of Cd(II) with EDTA and determination of cadmium content in drain water.

Keywords: Composite cation-exchanger; Polyaniline Ce(IV) molybdate; Cd(II) ion-selective membrane electrode

1. Introduction

In modern world, the pollution of heavy metal ions have become more and more severe. Among many toxic metals cadmium is one of them that appear in the environment mainly due to industrial processes. Anthropogenic processes like combustion of coal and mineral oil, Ni-Cd batteries,

phosphate fertilizers, mining, alloy processing, paint industries and natural exposure from soils or earth crust with high content of cadmium are the major sources of cadmium exposure to the people. Human uptake of cadmium takes place through foods that are rich in cadmium e.g. liver, mushrooms, shellfish, cocoa powder and dried seaweed. Cadmium strongly adsorbs to organic matters in soil. The provisional tolerable input for cadmium recommended by the WHO on food additives is 1 μg kg^{-1} of body weight per day [1], while the permissible limits of cadmium discharge in wastewater and drinking water are 0.1 and 0.05 mg L^{-1}, respectively [2]. United States Environmental Protection Agency (EPA) has found cadmium to potentially cause the following health effects when people are exposed to it at levels above the minimum concentration level (MCL) for relatively short periods of time such as nausea, vomiting, diarrhea, muscle cramps, salivation, sensory disturbances, liver injury, convulsions, shock and renal failure and in long-term cadmium has the potential to cause the following effects from a lifetime exposure at levels above the MCL: inhibiting respiration for α-oxoglutarate and pyruvate [3], kidney, liver, bone and blood damage [4,5]. Several techniques including; flame atomic absorption spectrometry (FAAS), electro thermal atomic absorption spectrometry (ETAAS), inductively coupled plasma mass spectrometry (ICP-MS) and atomic fluorescence spectrometry (AFS) [6–13] for the determination of cadmium have been used. However, the wide utilization of these methods for the determination of trace elements in different samples is limited by the expensive equipment and sample pretreatment. At the same time, numerous voltametric methods especially using chemically modified electrodes (CME) have also been developed [14, 15]. Because of some imperfectness of these methods for application to routine analysis, there is a challenge for analytical chemists to develop new sensors for the fast, accurate, reproducible, and selective determination of various species in the fields of environmental, agricultural and medicinal analysis. Potentiometric ion-selective electrodes are considered reliable in the field of chemical analysis for direct or indirect measurement in complex samples without the knowledge about the color of the sample or turbidity and are inexpensive. This fact makes ion-selective electrodes attractive to scientist in many disciplines [16]. Therefore, there is a demand for suitable materials that can be used for preparation of sensitive and selective ion-sensors, chemical sensors or more commonly ion-selective electrodes (ISEs). Precipitate based ion-selective membrane electrodes are well known as they are successfully employed for the determination of several anions and cations [17-25]. There are some homogeneous ion-exchange membranes are coherent ion-exchanger gels in the shape of disks, ribbons, etc and heterogeneous precipitate ion-exchange membranes consisting of suitable colloidal ion-exchanger particles as electroactive materials embedded in a polymer (inert) binder, i.e., poly(vinyl chloride) (PVC), or epoxy resin (Araldite), or polystyrene, polyethylene, nylon, poly(methyl methacrylate) etc., have been extensively studied as potentiometric sensors [26-37]. Ion-exchange membranes also find application in diverse processes such as (electro-dialysis, diffusion dialysis, electro-deionization, membrane electrolysis, fuel cells, storage batteries, electro-chemical synthesis *etc.*), which are energy resource and environmental saving. Thus, the development of ion-exchange membranes of high chemical, mechanical, and thermal stabilities, which meet the growing demand of the previously mentioned processes, is of great importance. Now a days, the use of 'organic-inorganic' composite ion-exchange materials has generated widespread interest in developing new ion-selective electrodes (ISEs) for sensor applications [38-42], especially for the determination of heavy toxic metals [43-45].

Therefore, we have also made an effort to develop composite cation-exchanger through incorporation of electrically conducting organic polymer *i.e.* polyaniline into the matrices of inorganic precipitate of multivalent metal acid salt *i.e.* Ce(IV) molybdate. Since, newly developed polyaniline Ce(IV) molybdate composite cation-exchanger is highly selective for Cd(II) ions; the further research work was carried out to obtain new heterogeneous precipitate based membrane ion-selective electrode by means of this composite as electroactive phase for the determination of Cd(II) ions; present in the solutions.

2. Experimental

2.1. Reagents and Instruments

The main reagents used for the synthesis of the material were ceric ammonium nitrate [$(NH_4)_2Ce(NO_3)_6$] (BDH, India), ammonium molybdate, $(NH_4)_6Mo_7O_{24}.4H_2O$) (BDH, India) and aniline ($C_6H_5NH_2$) (Qualigens, India). All other chemicals and reagents used were of analytical reagent grade. A digital pH meter (Elico LI-10, India), a digital potentiometer (Equiptronics EQ 609, India) with a saturated calomel electrode as a reference electrode were used.

2.2. Preparation of Polyaniline Ce(IV) Molybdate and its Membranes

The composite cation-exchanger used as electroactive component for the preparation of membranes was prepared in the usual method as described in our previous studies [46]. For the preparation of the membrane material was grinded to fine powder and was mixed thoroughly with PVC, dissolved in 10 ml of tetrahydrofuran (THF) and finally mixed with 10 drops of dioctylphthalate used as a plastisizer. The mixing ratio of the electroactive component was varied with a fixed content of PVC to come across a composition, which gave the membrane showing the best performance and the resulting solutions were carefully poured into a glass-casting ring (diameter 10 mm) resting on a glass plate. These rings were left for slow evaporation of THF to obtain thin films. In this way four sheets of different thickness of master membranes were obtained.

2.3. Characterization of Membrane

Some physicochemical properties that affect the electrochemical behavior of the membranes were determined as given below.

2.3.1. Conditioning of the Membrane

The membranes were conditioned by equilibrating with 1 M sodium chloride; about 1 ml of sodium acetate was also added to adjust the pH 5-6.5 (to neutralize the acid present in the film).

2.3.2. Water Content (% Total Wet Weight)

The conditioned membranes were first soaked in water to elute diffusible salts, blotted quickly with Whatman filter paper to remove surface moisture, and immediately weighed. These were further dried to a constant weight in vacuum over P_2O_5 for 24 h. The water content (% total wet weight) was calculated as:

$$\% \, Total \, wet \, \, weight = \frac{W_w - W_d}{W_w} \times 100, \quad (1)$$

where W_w = weight of the soaked/wet membrane and W_d = weight of the dry membrane.

2.3.3. Porosity

Porosity (ε) was determined as the volume of water incorporated in the cavities per unit membrane volume from the water content data.

$$\varepsilon = \frac{W_w - W_d}{AL\rho_w}, \quad (2)$$

where A = area of the membrane, L = thickness of the membrane and ρ_w = density of water.

2.3.4. Thickness and Swelling

The thickness of the membrane was measured by taking the average thickness of the membrane by using screw gauze.

Swelling was measured as the difference between the average thicknesses of the membrane equilibrated with 1 M NaCl for 24 h and the dry membrane. The results of the membrane characterization are given in Table 1.

Table 1. Characterization of ion-exchanger membranes of polyaniline Ce(IV) molybdate composite cation-exchanger.

S. No.	Membrane composition			Thickness (mm)	Water content as % weight of wet membrane	Porosity	Swelling of membrane (mm)
	Polyaniline Ce(IV) molybdate (mg)	PVC (mg)	Plasticizer (drops)				
M-1	100	200	10	0.38	13.46	0.492	0.021
M-2	150	200	10	0.42	13.50	0.475	0.033
M-3	200	200	10	0.54	17.40	0.569	0.050
M-4	250	200	10	0.61	18.60	0.635	0.058

2.4. Ion-selective Electrode Preparation

On the basis of physicochemical characterization the membrane sample (M-1) Table 1 was assumed to possess good electrochemical properties. The concerned membrane was cut in the shape of disc and mounted at the lower end of a Pyrex glass tube (o.d. 1.6 cm, i.d. 0.8 cm) with Araldite. Finally, the assembly was allowed to dry in air for 24 h.

The tube was then filled with internal filling solution (0.1 M $Cd(NO_3)_2$. The electrode was finally conditioned by soaking in a 0.1 M $Cd(NO_3)_2$ solution for 5-7 days and for 1 h at least before use. A saturated calomel electrode was inserted in the tube for electrical contact and another saturated calomel electrode was used as an external reference electrode. The whole arrangement can be shown as:

Internal reference electrode (SCE)	Internal electrolyte 0.1 M Cd^{2+}	Membrane	Sample solution	External reference electrode (SCE)

The response of the electrode in terms of the electrode potential (at 25 ± 2 °C), corresponding to the concentration of a series of standard solutions of $Hg(NO_3)_2$ (10^{-9} –10^{-1} M), was determined at a constant ionic strength as described by IUPAC Commission for Analytical Nomenclature [47]. After performing, the experiment membrane electrode was removed from the test-solution and kept in a 0.1 M $Cd(NO_3)_2$ solution.

Potential measurements of the membrane electrode were plotted against the selected concentrations of the respective ions in an aqueous medium using the electrode assembly. The calibration graphs were plotted three times to check the reproducibility of the system. In order to study the characteristics of the electrode, the following parameters were evaluated: lower detection limit, slope response curve, response time and working pH range.

The response time was measured by recording the e.m.f. of the electrode as a function of time when it was immersed in the solution to be studied. The electrode was usually first dipped in a 1×10^{-3} M solution of the ion concerned and immediately shifted to another solution (pH ≈ 4.0) of 1×10^{-2} M ion concentration of the same ion (10 fold higher concentration). The potential of the solution was read at zero second, that is, just after immediate dipping of the electrode in the second solution and subsequently recorded at the intervals of 5 s. The potentials were then plotted against the time. A series of solutions of varying pH in the range of 1 to 10 were prepared, keeping the concentration of the relevant ion constant (1×10^{-3} M). The pH variations were brought out by the addition of dilute acid (HCl) or dilute alkali (NaOH) solutions. The value of the electrode potential at each pH was recorded and was plotted against pH.

To study the cationic interference due to other ions, the selectivity coefficients of various interfering cations for the ion-selective membrane electrode was determined by the mixed solution method as discussed elsewhere [48]. A beaker of constant volume contained a mixed solution having a fixed concentration of interfering ion (M^{n+}) (1×10^{-3} M) and varying concentrations (1×10^{-1} to 1×10^{-9} M) of the primary ion. Now the potential measurements were made by using the membrane electrode assembly.

3. Results and Discussion

The membranes were prepared by using polyaniline Ce(IV) molybdate electroactive material in PVC support as a binding martial. The membrane composition was varied in different mixing ratios of electroactive polyaniline Ce(IV) molybdate and PVC dissolved in tetrahydrofuran (THF) with a fixed amount (10 drops) of plastisizer dioctylphthalate to achieve membranes of good electrochemical properties. After that membrane were taken under the careful characterization to select a member with good electrochemical properties for the preparation of ion-selective membrane electrode. The results of membrane characterization are given in Table 1. It was found that on increasing the amount of electroactive component, the thickness, swelling, water content, porosity increased. Thus, the low orders of water content, swelling and porosity with less thickness of the membrane (M-1) suggest that interstices are negligible and diffusion across the membrane would occur mainly through the exchange sites. Hence, membrane sample M-1 (thickness 0.36 mm) was selected for the preparation of the ion-selective electrode for further studies.

Ion-selective membrane electrode prepared using membrane sample M-1 showed a liner Nernstian response in the concentration range of 1×10^{-1}–5×10^{-6} M at a fixed ionic strength by adding a suitable excess of electrolyte with sub-Nerstain slope of 27 mV/decade change in cadmium ion concentration as shown in Fig. 1. The selectivity of the membrane for the Cd(II) depends on the distribution coefficient of the composite cation-exchanger used [46]. The limit of detection for this

electrode calculated as recommended by IUPAC from the intersection of the two extrapolated segments of the calibration curve was found to be $1 \times 10^{-1} - 5 \times 10^{-7}$ M for Cd^{2+} ions.

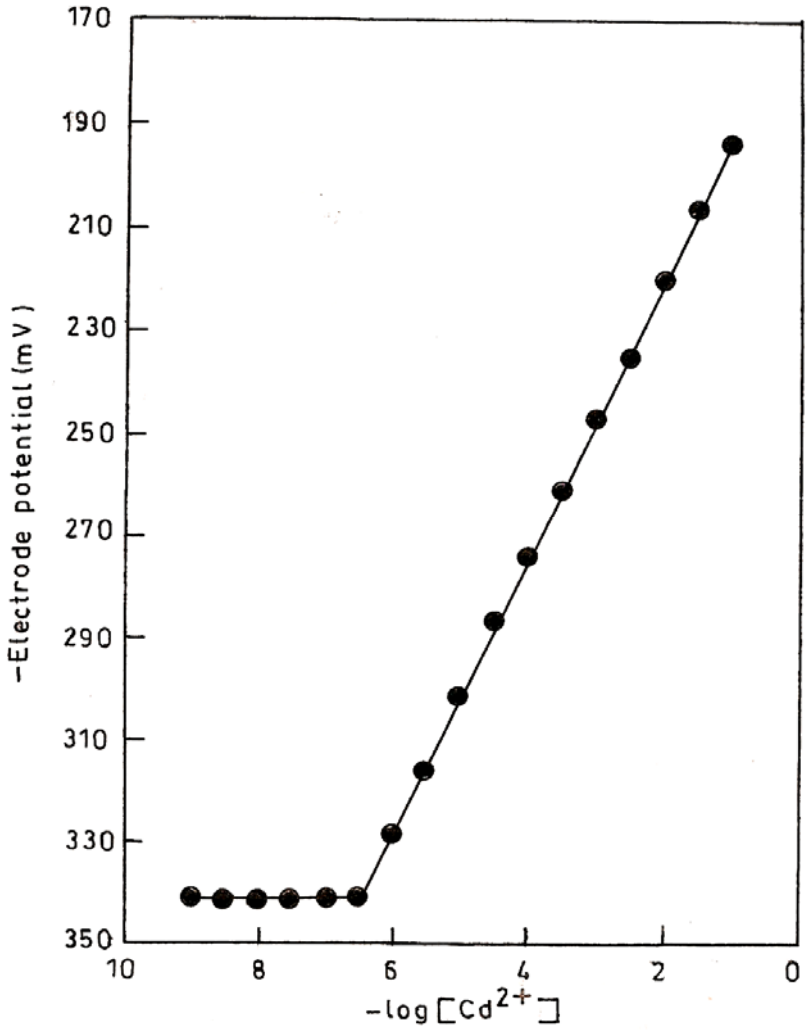

Fig. 1. Calibration curve for polyaniline Ce(IV) molybdate membrane electrode in aqueous solution of $Cd(NO_3)_2$.

The average time required for the Cd^{2+} selective electrode to reach a stable within ±2 mV of the final equilibrium value after successive immersion of a series of cadmium ion solutions, each having a 10 fold difference in concentration, was measured. The static response time thus obtained was 15 s for concentration $\leq 1 \times 10^{-2}$ M as evident from Fig. 2. The sensing behavior of the membrane electrode remained unchanged when the potentials recorded either from low to high concentration or vice versa. Although the solid contact electrodes are often criticized for their poor response and stability while the response of this electrode is quick and the lifetime is reasonable, at least 3 month. It is very important that the performance of any ion-selective electrode should be checked sooner every time before using it for any analytical purpose. In present case, it was observed that the measured potential of Cd^{2+} ions in a given concentration range of 10^{-1}–10^{-9} M was reproducible within ±3 mV and there was no significant change in the slope of the Nernst plot during the experiment over a time period of 3 months. This suggests a longer electrode life and a stable electrode performance.

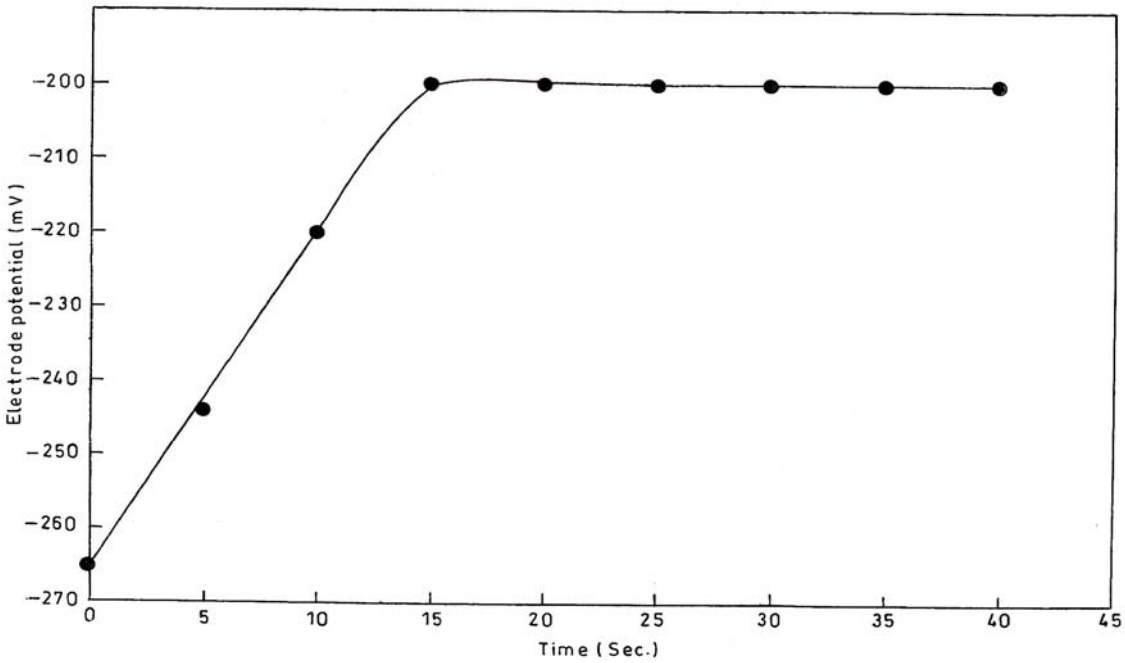

Fig. 2. Response of Cd^{2+} ion-selective of polyaniline Ce(IV) molybdate membrane electrode at different time interval.

One of the ions present in the aqueous solution is the hydrogen ion. It interferes with the functioning of the electrode. Consequently, it is necessary to find out the optimum pH range where the electrode functions without interference from the hydrogen ions. Therefore, the influence of pH on the potential response of the Cd^{2+} ion-selective membrane electrode was tested at 1×10^{-3} M Cd^{2+} concentration over the pH range 1-10. The results are shown in Fig. 3. It is observed that the potential remained constant from pH 2.5-7.5 and the ion-selective membrane electrode can be suitably used in this pH range. However, the potential decreased at higher pH values that may be due to the formation of hydroxyl complex of Cd^{2+} ion in the solution.

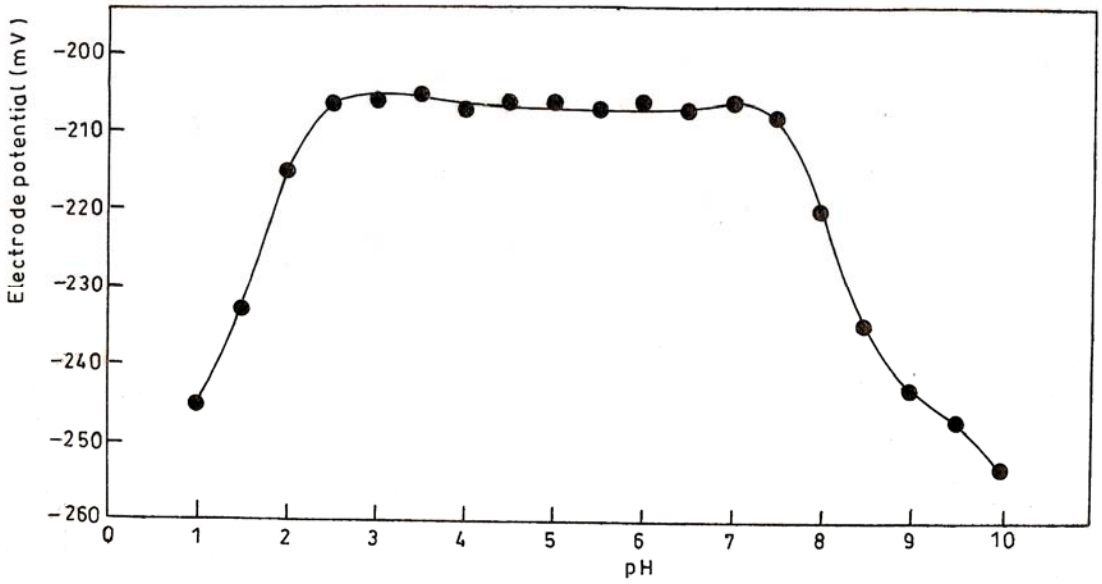

Fig. 3. Effect of pH on the electrode response of Cd^{2+} ion-selective the polyaniline Ce(IV) molybdate membrane electrode.

One of the most important characteristics of ion-selective membrane electrode is considered its selectivity because it often determines whether a reliable measurement in a targeted sample is possible for the primary ions in presence of secondary ions present in the sample solutions. This is measured in terms of selectivity coefficient ($K_{Cd.M}^{POT}$), which has been determined using mixed solutions method [48], and the results are summarized in Table 2. The potentiometric selectivity data of this membrane electrode for various metal ions is also shown in Fig. 4. It is seen from the Fig. 4 that alkali and alkaline earth metal ions do not interfere with the determination of mercury ions, while Cu(II), Zn(II), Pb(II) and Mn(II) interfere to a very little extent, and the interference of Fe(III) and Al(III) was found to be negligible. Thus, the results revealed that the electrode was selective for Hg(II) in the presence of interfering cations.

Table 2. Selectivity coefficient values ($K_{Cd.M}^{POT}$) for Cd^{2+} ion-selective polyaniline Ce(IV) molybdate membrane electrode for Cd(II) ions.

Interfering ions (M^{n+})	**Selectivity Coefficients $K_{Cd.M}^{POT}$ values**
Li^{+}	5
Na^{+}	5
K^{+}	5
Mg^{2+}	1×10^{-2}
Ca^{2+}	1×10^{-2}
Sr^{2+}	1×10^{-2}
Cu^{2+}	5×10^{-2}
Fe^{3+}	5×10^{-3}
Al^{3+}	5×10^{-3}
Mn^{2+}	1×10^{-1}
Hg^{2+}	1×10^{-1}
Pb^{2+}	1×10^{-1}

The proposed ion-selective membrane assembly was successfully applied for the potentiometric determination of 0.01 M $Cd(NO_3)_2$ solution against 0.01 M EDTA solution as titrant. For this purpose, 5 ml solution of cadmium nitrate was taken into a beaker and its volume was raised up to 20 ml by adding DMW. Resulting solution was titrated with EDTA and potential was recorded after each addition of 0.5 ml of EDTA. Typical results for the titration are shown in Fig. 5. It was observed that addition of EDTA causes decrease in potential as a result of decrease in cadmium ion concentration in the solution due to the complex formation of EDTA with Cd^{2+} ions. The amount of Cd(II) ions in unknown solutions can be accurately determined from the resulting neat titration curves providing a sharp end point. The potentiometric titration of Cd(II) were also successfully carried out in the presence of 1×10^{-5} M Hg(II), Pb(II) and Cu(II), hence demonstrating its applications as a sensor developed for potentiometric determination of Cd(II) in mixtures. The membrane electrodes was also applied to the direct measurement of Cd^{2+} in the drain water collected from Department of Chemistry, Aligarh Muslim University, Aligarh, India. The samples were collected from five different locations of drains and preserved with HNO_3, stored in glass bottles and analyzed within 12 h after collection. Since the samples contain particulate matters, they were centrifuged and the potentials were measured after adjusting the pH ≈ 4 with HNO_3 or NH_3. Three replicate measurements were done to obtain the Cd(II) contents in five samples with this electrode using the membrane sensor's calibration graph. The concentration of cadmium in the sample was 10^{-3} M and the reproducibility of the results was checked to three times. The amount of Cd(II) present in these samples was also determined by potentiometric titration with EDTA. It was observed that the concentration of Cd(II) determined using calibration curve and EDTA potentiometric curve are comparable.

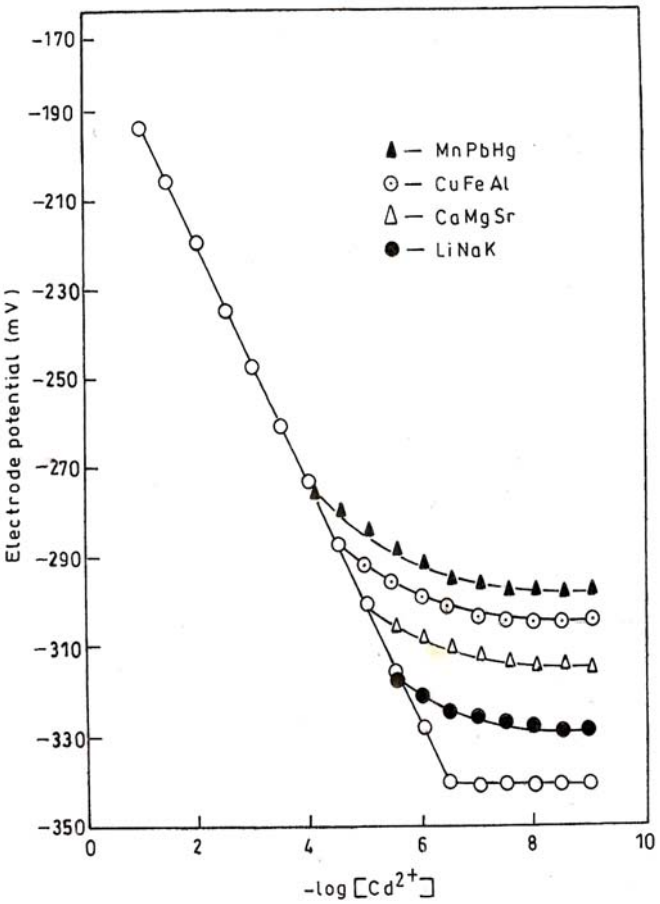

Fig. 4. Selectivity coefficients of various interfering ions for polyaniline Ce(IV) molybdate membrane electrode.

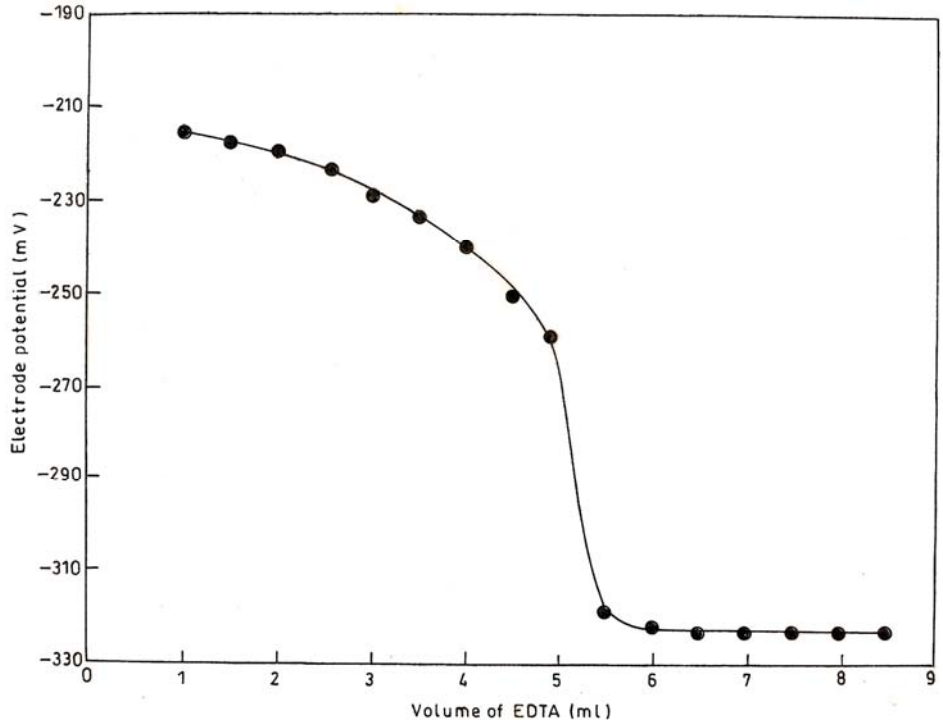

Fig. 5. Potentiometric titration of Cd(II) against EDTA solution.

4. Conclusions

Polyaniline Ce(IV) molybdate, a composite cation-exchanger was successfully utilized as electroactive component for the fabrication of ion-selective membrane electrode. The electrode exhibited a good detection limit, fast response time, good pH range, reasonable selectivity for Cd(II) in presence of other metal ions as well as it is easy to prepare and use. This electrode can be very well utilized for determination of Cd(II) in real samples as well as in drain water collected from the waste stream of the Chemistry Department, Aligarh Muslim University, Aligarh (India). Thus, the electrode provides inexpensive tool for the on spot direct determination of cadmium.

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Guide for Contributors

Aims and Scope

Sensors & Transducers Journal (ISSN 1726-5479) provides an advanced forum for the science and technology of physical, chemical sensors and biosensors. It publishes state-of-the-art reviews, regular research and application specific papers, short notes, letters to Editor and sensors related books reviews as well as academic, practical and commercial information of interest to its readership. Because it is an open access, peer review international journal, papers rapidly published in *Sensors & Transducers Journal* will receive a very high publicity. The journal is published monthly as twelve issues per annual by International Frequency Association (IFSA). In additional, some special sponsored and conference issues published annually.

Topics Covered

Contributions are invited on all aspects of research, development and application of the science and technology of sensors, transducers and sensor instrumentations. Topics include, but are not restricted to:

- Physical, chemical and biosensors;
- Digital, frequency, period, duty-cycle, time interval, PWM, pulse number output sensors and transducers;
- Theory, principles, effects, design, standardization and modeling;
- Smart sensors and systems;
- Sensor instrumentation;
- Virtual instruments;
- Sensors interfaces, buses and networks;
- Signal processing;
- Frequency (period, duty-cycle)-to-digital converters, ADC;
- Technologies and materials;
- Nanosensors;
- Microsystems;
- Applications.

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