Application of 1,2-Dimaleimidobenzene as a Neutral Ionophore in Construction of a Highly Selective Neodymium(III) Liquid Membrane Electrode

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Received: 11 August 2012 / Accepted: 7 September 2012 / Published: 1 February 2013

A highly selective neodymium(III) ion-selective membrane electrode has been fabricated in a poly(vinyl chloride) matrix based on 1,2-dimaleimidobenzene (DMB) as an ionophore, dibutyl phthalate (DBP) as a plasticizer, oleic acid (OA) and sodium tetraphenyl borate (NaTPB) as a good lipophilic additives. The electrode exhibits a Nernstian slope of 20.7 ± 0.3 mV decade⁻¹ over a wide concentration range $(1.0\times10^{-6}$ to 1.0×10^{-2} mol L⁻¹) for Nd³⁺ ions and a detection limit of 5.8×10^{-7} mol L⁻¹ between pH 2.3 and 9.1. It has a fast response time of 5 s. The proposed membrane sensor revealed good selectivity for Nd(III) over a wide variety of other metal ions. The electrode was successfully applied as an indicator electrode for the potentiometric titration of neodymium ions with EDTA as well as for the determination of the Nd³⁺ ion in different water samples.

Keywords: Sensor, Potentiometry, Ion-Selective Electrode, PVC Membrane

1. INTRODUCTION

Neodymium is one of the most abundant of lanthanides after cerium. It shows similar characteristics to the other trivalent lanthanides. Neodymium is included in many formulations of barium titanate, used as dielectric coatings and in multi-layer capacitors essential to electronic equipment. The most important application of neodymium is as the fundamental basis of neodymium-iron-boron permanent magnets. Neodymium and other lanthanide oxides are widely used in the preparation of optical glasses, glass fibers for optical purposes, gasoline-cracking catalysts, polishing compounds and carbon arcs, and in the iron and steel industries to remove sulfur, carbon, and other electronegative elements from iron and steel [1].

Different instrumental methods have been developed for its determination in different sample matrices such as: capillary electrophoresis, ICP-MS, ICP-AES, gravimetric determination, isotope dilution mass spectroscopy, absorption spectra of 4f electron transitions. Although these techniques provide an accurate measurement in trace amount of elements, they destroyed the samples. In comparison, potentiometric ion-selective sensors offer advantages of low cost, speed and ease of preparation and procedures, wide linear dynamic ranges, portability, selectivities, and simple operatory. They also provide an analysis method without destruction of analyte. There are a few reports on neodymium potentiometric sensors [2-9].

Several highly selective and sensitive polyvinyl chloride (PVC)-membrane sensors have recently been reported by our group and other researchers for different metal ions [10-36]. In this research, we wish to introduce a highly selective and sensitive Nd(III) sensor, based on 1,2-dimaleimidobenzene (DMB) (Figure 1) as an excellent ion carrier for the monitoring of Nd(III) ions.



Figure 1. The DMB structure.

2. EXPERIMENTAL

2.1. Reagents

The ionophore 1,2-dimaleimidobenzene was purchased from Aldrich. Merck Chemical (Germmany) and Aldrich Co. (USA) were the suppliers for the nitrate and chloride salts of all cations and the reagent grades of benzyl acetate (BA), nitrobenzene (NB), acetophenone (AP), dibutyl phthalate (DBP), oleic acid (OA), sodium tetraphenyl borate (NaTPB), tetrahydrofuran (THF), and high relative molecular weight PVC. The chloride and nitrate salts of the used cations were also purchased from Merck and Aldrich at the highest available purity and were submitted to no further modification. All solutions were prepared using doubly distilled deionized water.

2.2. The emf measurements

All emf measurements were carried out with the following assembly:

Ag–AgCl | internal solution, 1.0×10^{-3} mol L⁻¹ NdCl₃ | PVC membrane | sample solution | Hg–Hg₂Cl₂, KC1 (satd.)

Corning ion analyzer 250 pH/mV meter was used for the potential measurements at 25.0 ± 0.1 ⁰C. The activities were calculated according to the Debye–Huckel procedure [37].

2.3. Electrode preparation

The general procedure to prepare the PVC membrane was as followed: Different amounts of the ionophore (DMB) along with appropriate amounts of PVC, plasticizer and additive were dissolved in tetrahydrofuran (THF). The solution was mixed well. The resulting mixture was transferred into a glass dish of 2 cm diameter, and the solvent was evaporated slowly until an oily concentrated mixture remained. A Pyrex tube (3-5 mm o.d. on top) was dipped into the mixture for about 10 s, so that a transparent membrane of about 0.3 mm thickness was formed. The tube was pulled out from the mixture and kept at room temperature for 12 h. The tube was then filled with an internal solution $(1.0 \times 10^{-3} \text{ mol L}^{-1} \text{ NdCl}_3)$. The electrode was finally conditioned for 24 h by soaking in a $1.0 \times 10^{-3} \text{ mol L}^{-1} \text{ Nd}^{3+}$ ion solution [38-53]. A silver/silver chloride electrode was used as an internal reference electrode.

2.4. Selectivity coefficient measurement

Selectivity coefficients were calculated by match potential method (MPM) [54-61]. According to MPM, a specified activity (concentration) of the primary ions (A) is added to a reference solution and the potential is measured. In a separate experiment, interfering ions (B) are successively added to an identical reference solution, until the measured potentialmatches the one obtained before by adding primary ions. The matched potential method selectivity coefficient, K^{MPM} , is then given by the resulting primary ion to interfering ion activity (concentration) ratio, $K^{MPM} = \Delta a_A/a_B$.

3. RESULTS AND DISCISSION

3.1. The potential response of the sensor

To have a clear picture about the selectivity of the ligand for various metal ions, during the next experiments this carrier was used in the construction of membrane sensors for a wide variety of cations, including alkali, alkaline earth, transition and heavy metal ions, and the potential response of the most sensitive ion-selective electrodes based on DMB are shown in Figure 2a and 2b. As can be seen, with the exception of Nd³⁺ ions, all the tested cations showed relatively weak responses in the concentration range 1.0×10^{-6} to 1.0×10^{-2} mol L⁻¹, due to their weak interactions with the ionophore. This behavior may be considered to be the result of the selective tendency of the ionophore against Nd³⁺ in comparison to other metal ions, and the rapid exchange kinetics of the resulting DMB- Nd³⁺ complex.



Figure 2. Calibration curve of the DMB-based neodymium sensor.

3.2. The membrane composition influence

Taking into consideration that the sensitivity and selectivity of a given ionophore depend radically on the membrane ingredients as well as the nature of the solvent mediator and the nature of the used additives [62–72]. Table 1 shows the data obtained with membranes having various ratios of different constituents. As can be seen from Table 1, the increasing level of DMB up to 2% resulted in large slope of the membrane (numbers 2,6,7 and 10), that displays larger slopes. Since the nature of plasticizer influences the dielectric constant of the membrane phase, the mobility of the ionophore molecules, and the state of ligand, it was expected to play a key role in determining the selectivity, working concentration range, and response time of the membrane electrode. After the evaluation of four solvent mediators (NB, AP, BA and DBP), DBP was chosen to be employed in the sensor construction.

It is widely accepted that the addition of lipophilic anions to cation-selective membrane sensors not only diminishes the ohmic resistance and enhances the response behavior and selectivity, but also in cases where the extraction capability is poor, increases the sensitivity of the membrane electrodes [73-81]. As it can be seen from Table 1, the slope of the Nd³⁺ membrane sensor in the absence of additive is about half of the expected Nernstian value (number 1). However, the addition of 2% NaTPB and 5% OA increases the sensitivity of the electrode response considerably, so that the membrane electrode (number 10) demonstrates a nice Nernstian behavior. The obtained data revealed that, the membrane incorporating 61% DBP, 30% PVC, 2% DMB, in the presence of 2% sodium tetraphenyl borate (NaTPB) and 5% oleic acid (OA) as a suitable lipophilic additives considerably improved the sensitivity of the neodymium sensor and showed the best sensitivity, with a good Nernestian slope of 20.7 ± 0.3 mV decade⁻¹ of Nd³⁺ concentrations $(1.0 \times 10^{-6} - 1.0 \times 10^{-2} \text{ mol L}^{-1})$.

Sensor No.	Composition (w/w, %)				Slope (mV decade ⁻¹)	Dynamic Linear range $(mol L^{-1})$
	DMB	Plasticizer	Additive	PVC	(
1	2	DBP, 68	Na TPB,0	30	11.2 ± 0.3	1.0×10^{-4} -1.0 ×10 ⁻²
2	2	DBP, 66	NaTPB,2	30	21.5 ± 0.7	1.0×10^{-5} - 1.0×10^{-2}
3	2	BA, 66	NaTPB,2	30	15.1 ± 0.3	1.0×10^{-5} -1.0 $\times 10^{-2}$
4	2	AP, 66	NaTPB,2	30	15.3 ± 0.4	1.0×10^{-5} -5.0 $\times 10^{-2}$
5	2	NB, 66	NaTPB,2	30	14.8 ± 0.2	1.0×10^{-5} - 1.0×10^{-2}
6	2	DBP, 67	NaTPB,1	30	17.4 ± 0.5	$1.0 \times 10^{-5} - 1.0 \times 10^{-2}$
7	2	DBP, 65	NaTPB,3	30	18.2 ± 0.2	1.0×10^{-5} -6.4 $\times 10^{-2}$
8	1	DBP, 67	NaTPB,2	30	15.8 ± 0.6	$1.0 \times 10^{-5} - 1.0 \times 10^{-2}$
9	3	DBP, 63	NaTPB,2	30	17.9 ± 0.5	$1.0 \times 10^{-5} - 1.0 \times 10^{-2}$
10	2	DBP, 61	NaTPB,2; OA,5	30	20.7 ± 0.3	$1.0 \times 10^{-6} - 1.0 \times 10^{-2}$
11	2	DBP, 56	NaTPB,2; OA,10	0 30	16.5 ± 0.7	$1.0 \times 10^{-6} - 1.0 \times 10^{-2}$
12	2	DBP, 51	NaTPB,2; OA,1	5 30	18.4 ± 0.3	$1.0 \times 10^{-6} - 1.0 \times 10^{-2}$

Table 1. Optimization of the membrane ingredients.

3.3. Calibration graph

The EMF response of the PVC membrane at varying concentration of Nd³⁺ ions (Figure 3) indicated a linear range from 1.0×10^{-6} to 1.0×10^{-2} mol L⁻¹. The slope of the calibration curve was 20.7 ± 0.3 mV decade⁻¹ of Nd³⁺ ion concentration. The detection limit of the proposed membrane sensor was determined to be 5.8×10^{-7} mol L⁻¹ from the intersection of the two extrapolated segments of the calibration curve.



Figure 3. Calibration curve of the DMB based neodymium sensor.

3.4. The pH effect

The pH dependence on the membrane electrode was tested for the pH values from 2.0 up to 12.0 at a certain Nd³⁺ ion concentration $(1.0 \times 10^{-3} \text{ mol } \text{L}^{-1})$. The pH was adjusted by introducing small drops of hydrochloric acid (0.1 mol L⁻¹) or sodium hydroxide (0.1 mol L⁻¹) into the test solution. The

influence of the pH response on the PVC membrane electrode is depicted in Figure 4. Evidently, the potential remained constant from the pH value of 2.3 up to 9.1, beyond which some potential drifts took place. The observed drift at higher pH values can be due to the formation of insoluble of $Nd(OH)_3$ or other soluble intermediate products including $Nd(OH)^{2+}$, and $Nd(OH)_2^+$ which may not necessarily form stable complexes with the ionophore. At lower pH values, the potentials increased, indicating that the membrane sensor responded to protonium ions, as a result of the extent protonation of the ionophore nitrogen atoms.



Figure 4. The pH effect of the test solution $(1.0 \times 10^{-3} \text{ mol } \text{L}^{-1})$ on the potential response of the neodymium sensor.

3.5. Response time



Figure 5. Dynamic response time of the neodymium electrode for step changes in the Nd³⁺ concentration: A) $1.0 \times 10^{-6} \text{ mol } L^{-1}$, B) $1.0 \times 10^{-5} \text{ mol } L^{-1}$, C) $1.0 \times 10^{-4} \text{ mol } L^{-1}$, D) $1.0 \times 10^{-3} \text{ mol } L^{-1}$, E) $1.0 \times 10^{-2} \text{ mol } L^{-1}$.

Dynamic response time is an important factor, for the evaluation any sensor. The dynamic response time of the membrane was measured at various concentrations (0.000001, 0.00001, 0.0001, 0.0001, 0.001, 0.001, 0.001 and 0.1 mol L^{-1}) of the test solutions with different neodymium concentrations and results are shown in Figure 5. As can be seen, in the whole concentration range, the electrode reaches the equilibrium response in a very short time (~5 s).

3.6. Selectivity coefficients of the Nd(III) sensor

The selectivity behavior is obviously one of the most important characteristics of an PVC membrane ion-selective electrode. The potentiometric selectivity coefficients of the sensor, which are supposed to describe the preference of the DMB-based membrane electrode for the target ion in the presence of an interfering ion were determined through the matched potential method. The resulting selectivity coefficient values are summarized in Table 2. It is clear that the selectivity coefficients of the electrode for all ions were in the order of 6.2×10^{-3} or smaller. This means these ions would not significantly disturb the response of the Nd³⁺ selective membrane sensor, especially if their concentrations were low in the test solution.

Interfering	$K^{MPM}_{_{Nd}^{3+},B}$	Interfering	$K^{MPM}_{Nd^{3+},B}$
10115		10115	
Er ³⁺	6.3×10^{-4}	Fe ³⁺	2.8×10^{-3}
Gd ³⁺	7.5×10^{-4}	Na ⁺	5.1×10^{-4}
Sm ³⁺	6.8×10^{-4}	K ⁺	6.7×10^{-4}
La ³⁺	1.0×10^{-3}	Ca ²⁺	2.6×10^{-3}
Lu ³⁺	2.2×10^{-3}	Pb^{2+}	2.1×10^{-3}
Ho ³⁺	3.5×10^{-3}	Co ²⁺	1.0×10^{-3}
Dy ³⁺	2.8×10^{-3}	Cd^{2+}	4.4×10^{-3}
Tm ³⁺	1.0×10^{-4}	Ni ²⁺	3.4×10^{-3}
Cr ³⁺	6.2×10^{-3}	Cu ²⁺	1.0×10^{-3}

 Table 2. Selectivity coefficients of various interfering ions.

Table 3 compares the linearity range, detection limit, pH range, response time, slope and selectivity coefficients of the suggested sensor with those of the best previously Nd³⁺ electrodes reported in the literature by other researchers [3-8]. It is evident that the newly developed sensor is superior to the formerly reported Nd³⁺ sensors in terms of selectivity, detection limit and dynamic concentration range.

Parameter	Ref. 3	Ref. 4	Ref. 5	Ref. 6	Ref. 7	Ref. 8	This work
$LR \pmod{L^{-1}}$	5.0×10^{-7} -	1.0×10^{-6} -	1.0×10^{-6} -	1.0×10^{-5} -	1.0×10^{-6} -	1.0×10^{-6} -	1.0×10^{-6} -
	1.0×10^{-2}	1.0×10^{-1}	1.0×10^{-2}				
$DL \pmod{L^{-1}}$	1.0×10^{-7}	7.0×10^{-6}	7.9×10^{-7}	2.0×10^{-6}	6.2×10^{-7}	8.0×10^{-7}	5.8×10^{-7}
Response time (s)	10	<15	<5	<10	<10	<15	~5
pH range	4.0-8.0	4.0-8.0	4.0-6.5	3.5-8.5	3.7-8.3	3.0-7.0	2.3-9.1
Slope (mV decade ⁻¹)	19.8±0.3	19.6±0.3	20.1±0.2	19.6±0.3	19.7±0.4	19.4±0.3	20.7±0.3
Log K _{sel} >-2	Hg, Ni	La, Gd,	Ce, Yb,	La, Sm,	La, Dy	La, Gd,	-
-		Sm, Cu,	Gd, Sr,	Gd		Sm, Pr	
		Yb	Cu, La,				
			Ag				

Table 3. Comparison of different Nd³⁺ electrodes.

3.7. Analytical application

The electrode was found to function well under laboratory conditions and the sensor was used as an indicator electrode in the titration of a 1.0×10^{-4} mol L⁻¹ neodymium ion solution with a standard 1.0×10^{-2} mol L⁻¹ EDTA solution. The resulting titration curve is shown in Figure 6, which indicates that the sensor was capable of monitoring the amounts of neodymium ions in such measurements.



Figure 6. Potentiometric titration curve of 20.0 mL from a 1.0×10^{-4} mol L⁻¹ Nd³⁺ solution with 1.0×10^{-2} mol L⁻¹ of EDTA.

The developed Nd^{3+} sensor was applied for the determination of Nd^{3+} ions concentration in mixtures of different ions and the results are summarized in Table 4. As it is obvious, the recovery of Nd^{3+} ions is very good (96–104%).

Serial no.	Composition	Observed $(mol L^{-1})$	content
1	$0.00010 \text{ mol } L^{-1} \text{ Nd}(\text{NO}_3)_3 + 0.001 \text{ mol } L^{-1} \text{ Ho}(\text{NO}_3)_3 + 0.001 \text{ mol } L^{-1} \text{ Eu}(\text{NO}_3)_3$	0.000098	
2	$0.00010 \text{ mol } L^{-1} \text{ Nd}(\text{NO}_3)_3 + 0.001 \text{ mol } L^{-1} \text{ Tm}(\text{NO}_3)_3 + 0.001 \text{ mol } L^{-1} \text{ Lu}(\text{NO}_3)_3$	0.000104	
3	$0.00010 \text{ mol } L^{-1} \text{ Nd}(\text{NO}_3)_3 + 0.001 \text{ mol } L^{-1} \text{ Dy}(\text{NO}_3)_3 + 0.001 \text{ mol } L^{-1} \text{ Tb}(\text{NO}_3)_3$	0.000097	
4	$0.00010 \text{ mol } L^{-1} \text{ Nd}(\text{NO}_3)_3 + 0.001 \text{ mol } L^{-1} \text{ Sm}(\text{NO}_3)_3 + 0.001 \text{ mol } L^{-1} \text{ Gd}(\text{NO}_3)_3$	0.000096	
5	$0.00010 \text{ mol } L^{-1} \text{ Nd}(\text{NO}_3)_3 + 0.001 \text{ mol } L^{-1} \text{ Er}(\text{NO}_3)_3 + 0.001 \text{ mol } L^{-1} \text{ Pr}(\text{NO}_3)_3$	0.000103	
6	$0.00010 \text{ mol } L^{-1} \text{ Nd}(\text{NO}_3)_3 + 0.001 \text{ mol } L^{-1} \text{ La}(\text{NO}_3)_3 + 0.001 \text{ mol } L^{-1} \text{ Yb}(\text{NO}_3)_3$	0.000102	
7	$0.00010 \text{ molL}^{-1} \text{ Nd}(\text{NO}_3)_3 + 0.001 \text{ molL}^{-1} \text{ Cr}(\text{NO}_3)_3 + 0.001 \text{ molL}^{-1} \text{ Fe}(\text{NO}_3)_3$	0.000097	
8	$0.00010 \text{ mol } L^{-1} \text{ Nd}(\text{NO}_3)_3 + 0.001 \text{ mol } L^{-1} \text{ Pb}(\text{NO}_3)_2 + 0.001 \text{ mol } L^{-1} \text{ Co}(\text{NO}_3)_2$	0.000096	
9	$0.00010 \text{ mol } L^{-1} \text{ Nd}(\text{NO}_3)_3 + 0.001 \text{ mol } L^{-1} \text{ Ca}(\text{NO}_3)_2 + 0.001 \text{ mol } L^{-1} \text{ NaNO}_3$	0.000101	
10	$0.00010 \text{ mol } L^{-1} \text{ Nd}(\text{NO}_3)_3 + 0.001 \text{ mol } L^{-1} \text{ Ni}(\text{NO}_3)_2 + 0.001 \text{ mol } L^{-1} \text{ KNO}_3$	0.000096	

The proposed sensor was also used to the determination of Nd^{3+} ions in tap and river water samples. The results, after triplicate measurements, are summarized in Table 5. As it is seen, the accuracy of Nd^{3+} ions determination in different water samples is almost quantitative.

Table 5. Determination of Nd^{3+} spiked in tap and river water samples by use of the proposed electrode.

Sample	Nd^{3+} added (mg mL ⁻¹)	Found (mg mL ⁻¹)	Recovery (%)
River water	0.25	$(0.28^{a} \pm 0.04)$	112
	0.5	(0.55 ± 0.03)	110
Tap water	0.25	(0.29 ± 0.03)	116
	0.5	(0.54 ± 0.02)	108

^aResults are based on three measurements

4. CONCLUSION

An ion-selective electrode was constructed based on 1,2-dimaleimidobenzene (DMB) with a wide concentration range of 1.0×10^{-6} to 1.0×10^{-2} mol L⁻¹, good detection limit (5.8×10^{-7} mol L⁻¹) and a Nernstian slope (20.7 ± 0.3 mV decade⁻¹). This sensor exhibited neodymium selectivity from common alkali, alkaline earth, transition and heavy metal ions. The recommended sensor displayed a quick response time of 5 s and its potential responses were pH independent across the range of 2.3–9.1. The proposed electrode was successfully applied to the determination of neodymium in different sample solutions with good accuracy.

ACKNOWLEDGEMENT

The authors acknowledge the financial support kindly offered by the Research Council of Mashhad Branch Islamic Azad University.

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