

Full Paper

Oxime-2-(1-cyclohexenyl) Cyclohexanone: Synthesis and Application as a New Ion Carrier for Preparation of Cobalt Selective Potentiometric Electrode

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Abstract- In this paper, a new cobalt selective liquid membrane electrode has been constructed by using a new synthesized oxime-2-(1-cyclohexenyl) cyclohexanone in a poly (vinyl chloride) matrix. The electrode was shown a good Nernstian response for Co^{2+} (32.1 ± 0.5) over a concentration range of (3.0×10^{-2} to 7.0×10^{-7} mol L^{-1}). The electrode detection limit was 6.0×10^{-7} mol L^{-1} . The response time of electrode was fast (~ 10 s) and useful working pH range was 3.5-8.5. It can be used over a period of 15 weeks without any divergence in potential. The proposed electrode revealed good selectivity for Co^{2+} over a wide variety of other metal ions. The Co^{2+} ion sensor has been practically utilized as an indicator electrode in potentiometric titration of Co^{2+} with EDTA. Also, direct determination of Co^{2+} in water and battery wastewater samples was done to confirmation of analytical ability of electrode.

Keywords- Oxime-2-(1-cyclohexenyl) cyclohexanone, Co^{2+} selective sensor, Liquid membrane, Potentiometric

1. INTRODUCTION

Cobalt is widespread in nature in meteorites, fresh waters, soils, plants, and animals. It is well known as a component of Vitamin B12 [1]. Cobalt deficiency in animals may lead to retarded growth, loss of appetite and anemia, and rapid recovery from these symptoms occurs

upon feeding them with a cobalt-supplementary diet [2]. Therefore, determination of cobalt is very important. A number of sophisticated methods is applied for cobalt determination including flame atomic adsorption spectroscopy, stripping voltammetry, chemiluminescence [3-5]. These methods are characterized by good accuracy but they require time-consuming sample preparation and are relatively expensive [6]. Therefore, new methods are being developed, including potentiometric methods with ion selective electrodes (ISE). Portability, low cost, good sensitivity and selectivity, accuracy and ease of use are the most important advantages of ion selective electrodes (ISEs) which caused them an important analytical tool to measuring of ion in solution [7-16]. But there are limited reports on Co^{2+} selective electrodes in literature and most of these electrodes suffer a lack of stability, a narrow working concentration range, high response time and short life time. In accordance with, a spread attempt has been done in the synthesis and characterization of variety ionophores to preparation new electrodes for the measuring of Co^{2+} ions, in various samples [17-22].

Therefore, in this study, oxime-2-(1-cyclohexenyl) cyclohexanone (Fig. 1) was synthesized and used to fabricate a Co^{2+} selective liquid membrane potentiometric electrode as a new ionophore. Oximes are a very important intermediate in organic chemistry. They are applied in medical treatment [23], in preparation of amines [24] and lactams [25].

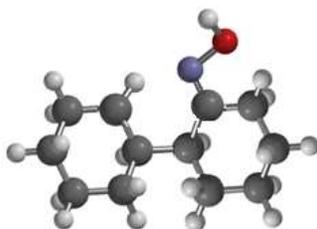


Fig. 1. The structure of oxime-2-(1-cyclohexenyl) cyclohexanone

2. EXPERIMENTAL SECTION

2.1. Reagents and Chemicals

Benzyl acetate (BA), nitrobenzene (NB) and dibutyl phthalate (DBP) as plasticizer, sodium tetraphenyl borate (NaTPB) as additive, polyvinyl chloride (PVC) and tetrahydrofuran (THF) were obtained from Merck. Cyclohexanone and hydroxylamine hydrochloride were purchased from Merck. 2-(1-chloro cyclohexyl) cyclohexanone was synthesized according to literature [26]. The nitrate salts of related cations used in measurements were all of the highest purity available and purchased from Aldrich or Merck. Throughout all experiments distilled deionized water was used.

2.2. Apparatus

The potential measurements were done at 25.0 ± 0.1 °C with a corning ion analyzer 250 pH/mV meters. Two Silver/silver chloride electrodes (purchased from Azar-Electrode, Iran) were used as reference electrodes.

^1H and ^{13}C NMR spectra were recorded with a Bruker Avance BRX-250 MHz in deuterated dimethyl sulfoxide with tetramethylsilane as internal standard. The IR spectra were recorded on a Bruker, Tensor 27 instrument. Ferigan-MAT Electron impact ionization-mass spectrometer (70 eV) (in m/z) was used to obtaining mass spectra. Melting point was determined with Bransstead Electro Thermal B.

2.3. Synthesis of Ionophore

At first, the 2-(1-chloro cyclohexyl) cyclohexanone (Fig. 2a) was synthesized from cyclohexanone as reported [32]. The melting point of product was 42-43 °C. Then, oxime-2-(1-cyclohexenyl) cyclohexanone (Fig. 2b) was prepared using 2-(1-chloro cyclohexyl) cyclohexanone and hydroxylamine hydrochloride in alkaline solution (pH=8~9). The oxime-2-(1-cyclohexenyl) cyclohexanone was synthesized with elimination of chlorine as hydrochloride in one pot reaction (Fig. 2). IR (CHCl_3): 3210.38; 2931.75; 1668.16; 1445.26; 1218.40; 1139.15; 1042.15; 963.02; 891.82; 759.14; 663.02; 599.85 cm^{-1} . ^1H NMR: (CDCl_3) δ ppm, 1.5- 2.2 (15 H, m); 2.62- 2.86 (2H, q), 5.5 (1H, s), 9.8 (1 H, s). ^{13}C NMR: (CDCl_3) δ ppm, 22.37, 22.76, 23.86, 24.55, 25.31, 25.74, 27.35, 30.79, 49.59, 123.46, 136.67, 160.94. MS: m/z (relative intensity): 193.0(12.9), M^+ ; 176(17.1); 163(23.8); 151(100, Base peak); 150(49.9); 134(33.3); 124(10.5); 91(4.7); 79(7.6).

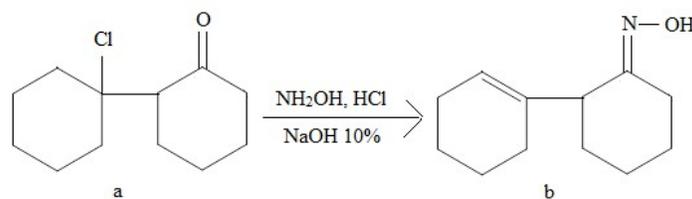


Fig. 2. The Schematic illustration of synthesis of oxime-2-(1-cyclohexenyl) cyclohexanone

2.4. Preparation of liquid membrane Electrode

The liquid membrane electrode was prepared as followed: Various amounts of the oxime-2-(1-cyclohexenyl) cyclohexanone, additive (NaTPB), PVC and plasticizer (DBP or BA or NB) were mixed and dissolved in 2 mL of tetrahydrofuran (THF). The solvent was evaporated slowly until an oily residue was obtained. Then, a plastic tube (3-5 mm o.d.) was dipped into the mixture for some seconds till a transparent membrane with 0.3 mm thickness was formed.

The membrane was stored under ambient conditions for 24 h. After the membrane lost its THF content, the tube was filled with $1.0 \times 10^{-3} \text{ mol L}^{-1} \text{ Co(NO}_3)_2$ as internal filling solution. Finally, the electrode was conditioned for 24 h by soaking in a $1.0 \times 10^{-3} \text{ mol L}^{-1} \text{ Co(NO}_3)_2$ solution.

2.5. Emf Measurements

The emf (electromotive force) was measured in following cell;
Ag–AgCl, KCl (satd.) | internal solution, $1.0 \times 10^{-3} \text{ mol L}^{-1} \text{ Co(NO}_3)_2$ | PVC membrane | sample solution | Ag–AgCl, KCl (satd.)

Activities were calculated according to the Debye- Huckel equation. The calibration curve with several standard solutions was draw for all of the measurements.

3. RESULTS AND DISCUSSION

3.1. Membrane Composition Effect on the Potential Response of the Sensor

Membrane composition and geometry of used ionophore affects the selectivity and linearity of electrode [27]. Therefore, some electrodes with various compositions were prepared (Table 1) and their potentiometric responses measured as function of Co^{2+} ion concentration. The slope of the potential response plots have been determined and given in Table 1. As can be seen, the prepared liquid membrane electrode with the 10% ionophore, 2% NaTPB, 29% PVC, and 59 % BA (no. 10) exhibited the best Nernstian slope ($32.1 \pm 0.5 \text{ mV/decade}$) and selected as optimum one for the constructing of this electrode.

The effect of ionophore amount on potentiometric response of the electrode was investigated. From Table 1, it was obvious that the response of the proposed electrode was too low (slope of 7.5 ± 0.6 and $9.1 \pm 0.4 \text{ mV per decade}$) in the absence of ionophore (no. 7, 8) and increases with increasing ionophore amount until a value of 10% (no. 10). It prove significant effect of the ionophore and affinity of the oxime-2-(1-cyclohexenyl) cyclohexanone toward Co^{2+} ion.

The other factor which helps the extraction of ions in liquid membrane electrode is plasticizer. Plasticizer is an important component for polyvinyl chloride-membrane electrode due to its high lipophilicity. It is such as a solvent and allow homogeneous dissolution and diffusional mobility of the ion-pair inside the membrane [28, 29]. After the evaluation of DBP, NB and BA as solvent mediators, as seen in Table 1, the prepared electrode (no. 10) with benzyl acetate (BA) exhibited the best potentiometric response and Nernstian behavior. Therefore, BA was chosen to be employed in the sensor construction. NB and DBP presented higher dielectric constant values than BA (with DC of 5), which leading to the extraction of the polar interfering ions, and poorer extraction of Co^{2+} ions (with high charge density). But this negative effect can be compensated by the selective complexation of oxime-2-(1-cyclohexenyl) cyclohexanone with the Co^{2+} ions.

Table 1. The optimization of the membrane ingredients

Membrane No.	Composition (%)				Slope (mV per decade)
	PVC (%wt.)	plastisizer (%wt.)	Ionophore (%wt.)	NaTPB (%wt.)	
1	30	(DBP)63	5	2	18.9±0.5
2	30	(DBP)61	7	2	21.7±0.4
3	29	(DBP)59	10	2	21.6±0.3
4	32	(DBP)59	7	2	19.3±0.3
5	30	(NB)63	5	2	19.6±0.5
6	29	(NB)59	10	2	26.4±0.4
7	35	(BA)65	0	0	7.5±0.6
8	35	(BA)63	0	2	9.1±0.4
9	30	(BA)60	10	0	22.5±0.3
10	29	(BA)59	10	2	32.1±0.5

The presence of ionic additive in an ion-selective membrane, decreases the Ohmic resistance and improves the response behavior. Also, when the extraction power is poor, it help the electrode to extract ionic species into hydrophobe membrane [30]. Therefore, the presence of lipophilic anions in the membrane was also tested. The data given in Table 1 revealed that addition of 2% NaTPB as an additive increase the slope of the membrane electrode response from the sub-Nernstian value of 22.5±0.3 mV per decade (no. 9) to the Nernstian value of 32.1±0.5 mV per decade (no. 10). This lipophilic anion helps to the ion-exchange of the Co^{2+} from aqueous solution to organic layer of the membrane.

3.2. Internal Solution

Three different $\text{Co}(\text{NO}_3)_2$ solutions (1.0×10^{-2} , 1.0×10^{-3} and 1.0×10^{-4} M) were prepared as the filing solutions and the potential response of the Co^{2+} selective electrode was evaluated (Fig. 3). As obtained results, there is just an expected change in the intercept of the resulting Nernstian plot and there is not significant differences in potential response with variation of internal solution concentration. Therefore, a 1.0×10^{-3} M concentration Co^{2+} solution was used as internal solution of proposed electrode.

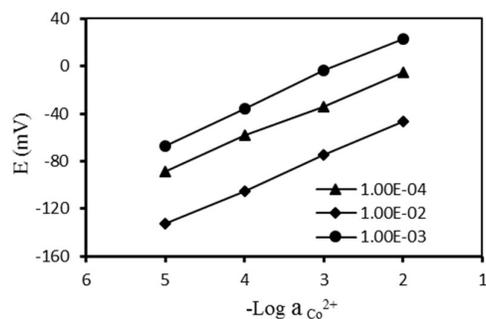


Fig. 3. The effect of filling solution concentration on the electrode response

3.3. Effect of pH

The influence of the solution pH on the Co^{2+} sensor response was tested at $1.0 \times 10^{-3} \text{ M Co}^{2+}$ solution. The pH of the solution was adjusted in the pH range of 2.0 up to 11 by spiking small volume of sodium hydroxide or hydrochloric acid. As can be seen in figure 4, the sensor showed a stable potentiometric response over the pH range of 3.5-8.5. Out of this range, there are fluctuations in potential.

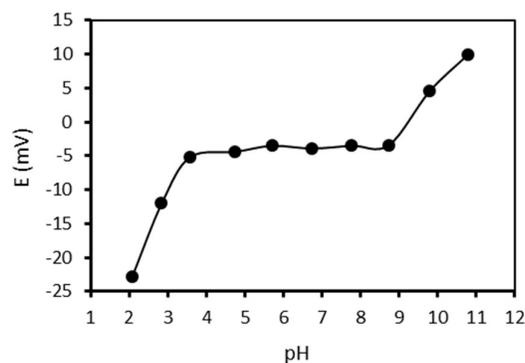


Fig. 4. pH effect on potential response of the Co^{2+} selective electrode

The variation in electrode response below the pH value of 3.5 may be attributed to the protonation of the used ionophore [15]. Charge-dipole interaction between hetero atoms of ionophore and Co^{2+} ions is stronger than interaction between these atoms and hydronium ions. In this pH range, hydronium ion concentration is enough to prevail on Co^{2+} in competition for making complex with ionophore. On the other hand, the increase in potentials in $\text{pH} > 8.5$ is due to the formation of the Co^{2+} ion hydroxy complexes in the solution. Therefore, the pH range 3.5 to 8.5 was considered as the working pH range of the proposed electrode.

3.4. Response Time

The average time to achieve a potential within ± 0.1 mV of the final steady-state potential while immerse the electrode in a series of solution with a tenfold different in concentration, is defined as response time. The experimental conditions like the ionic concentration, the stirring rate, composition of the test solution, any previous usages or preconditioning of the electrode, and the testing temperature can affect the experimental response time of an electrode [31].

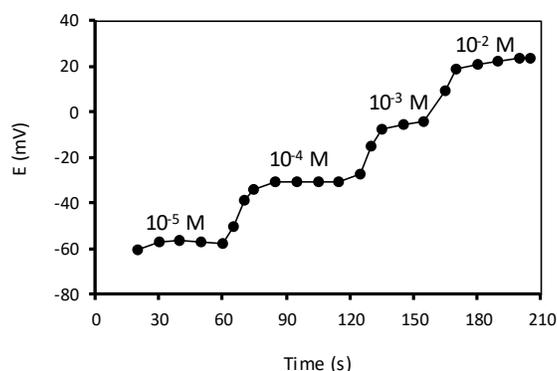


Fig. 5. Dynamic response time of Co^{2+} selective electrode for step changes in concentration of Co^{2+}

Dynamic response time of proposed electrode was determined over a concentration range 1.0×10^{-5} to 1.0×10^{-2} M Co^{2+} ions. The changes of actual potential versus response time is shown in Fig. 5. It is clear that the electrode has a relatively short response time (10 s) in whole concentration range. This is probably due to the fast Co^{2+} -ionophore exchange kinetics at the aqueous layer-membrane interface.

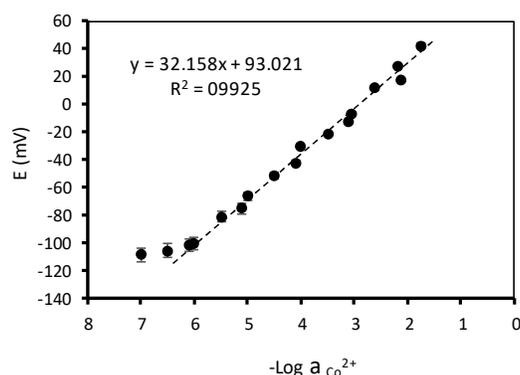


Fig. 6. The calibration curve of the Co^{2+} selective electrode

3.5. Working Range and Limit of Detection

The working range of an ISE includes the linear part of the calibration graph and detection limit is calculated extrapolating two linear segments of the calibration plot. For the most ISEs, detection limits are in the range of 10^{-5} – 10^{-6} mol L⁻¹. In this study, as shown in Fig. 6, the working range of the proposed electrode is between 7.0×10^{-7} and 3.0×10^{-2} M. Also, the limit of detection of the membrane electrode was 6.0×10^{-7} mol L⁻¹.

3.6. Selectivity

Selectivity is the relative response of the electrode to primary ion compared to secondary ion and expressed with selectivity coefficient. The smaller value of the selectivity coefficient means the higher selectivity of the electrode. In this study, selectivity coefficient (K_{MPM}) values were evaluated by the matched potential method (MPM) [32] and reported in Table 2. It is clearly seen that they are much smaller than 0.1 and electrode exhibit sufficient selectivity towards Co^{2+} over all the interfering ions studied.

Table 2. The selectivity coefficients of Co^{2+} selective electrode for some interfering cations

Ion	K_{MPM}
Zn^{2+}	1.2×10^{-2}
Ag^+	4.9×10^{-3}
Pb^{2+}	1.2×10^{-2}
Mg^{2+}	4.9×10^{-3}
Cd^{2+}	1.2×10^{-3}
Cr^{3+}	1.5×10^{-3}
Mn^{2+}	1.9×10^{-3}
Cu^{2+}	1.5×10^{-2}
Na^+	1.2×10^{-2}

3.7. Lifetime

The lifetime of proposed electrode was studied by monitoring the electrode slope changes with time. The Co^{2+} selective electrode were tested for 19 weeks. The electrodes were used one hour per day during this time and the electrode slope was recorded. The results have been shown in Fig. 7. A significant change in slope is observed after 15 weeks. Therefore, 15 weeks was considered as lifetime of this Co^{2+} selective electrode.

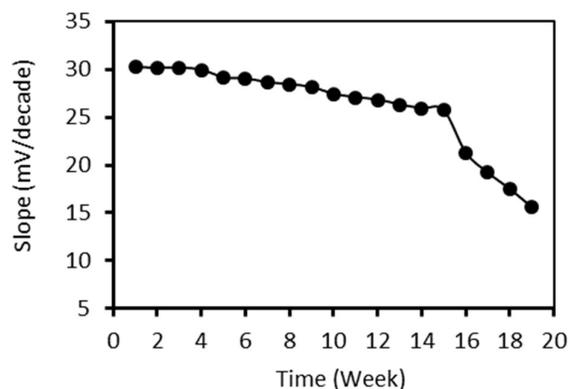


Fig. 7. The lifetime of the Co^{2+} selective electrode

Plasticizer, ionophore, or ionic additive can be leaching from the polymeric film into the sample during long time of usage and causes limited lifetimes of the sensors [33,34].

3.8. Repeatability and Reproducibility

The repeatability and reproducibility of the proposed electrode were investigated. The repeatability of the cobalt selective electrode was tested by potentiometric measurements in five Co^{2+} solution with 1.0×10^{-3} M concentration. The relative standard deviation of the five replicate measurements was 4.3% which indicates good repeatability.

Also, the reproducibility of the cobalt selective electrode was studied using four different optimized liquid membranes. All membranes were prepared using similar compositions and were used for potentiometric measurements of 1.0×10^{-4} M Co^{2+} solution. It was observed that the proposed electrodes demonstrated high reproducibility with less than 5.7% relative standard deviation.

3.9. Analytical Application

ISEs are an ideal choice for environmental measurements because they are simple to use and low in cost. They have low interferences from the matrix and easily automated for rapid sampling.

The applicability of the sensor was evaluated through its applications as an indicator electrode to determine the end point in the potentiometric titration of 25.0 mL of 1.0×10^{-4} M of Co^{2+} solution with EDTA 1.0×10^{-2} M. Fig. 8 shows the resulting titration curve. The obtained results indicated that the amount of Co^{2+} ions in solution can be accurately determined with the electrode.

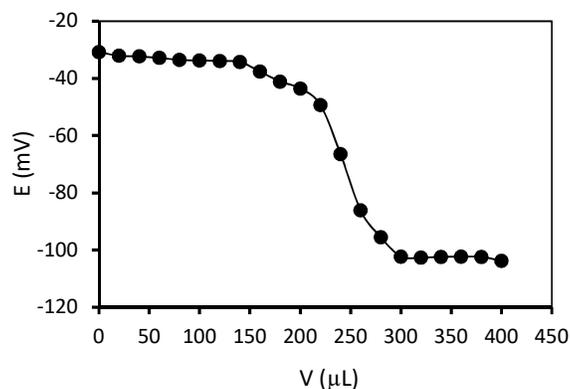


Fig. 8. Potentiometric titration curve of 1.0×10^{-4} M Co^{2+} solution with 1.0×10^{-2} M EDTA using the Co^{2+} selective electrode as an indicator electrode

The proposed electrode was also used in real samples to determine Co^{2+} ions in battery wastewater samples. After filtering of wastewater samples using a $0.45\text{-}\mu\text{m}$ pore size membrane filter, pH adjusted about 6. Co^{2+} ions were added to the some sample solutions.

Table 3. Determination of Co^{2+} ions in various battery wastewater samples

Sample	Spiked amount	Measured with proposed sensor (mol L^{-1})
battery wastewater	0	$3.7 (\pm 0.09)^a \times 10^{-4}$
Spiked battery wastewater	2.0×10^{-4}	$5.6 (\pm 0.11) \times 10^{-4}$
Spiked battery wastewater	4.0×10^{-4}	$7.5 (\pm 0.14) \times 10^{-4}$

^a S_d based on three replicate analysis

The electrode was applied to the direct measurement of Co^{2+} ion in battery wastewater sample and spiked battery wastewater samples. Potentials of the samples were measured by direct potentiometry method. As shown in Table 3, the proposed electrode could be used successfully for the direct determination of Co^{2+} ions in these samples.

3.10. Comparison of the proposed Co^{2+} selective electrode and some of the best previously reports

The Nernstian slope, detection limit, linearity range, response time and pH range of proposed Co^{2+} selective electrode are compared with some of the previously reported in literature [1, 22, 35-37]. The results are shown in Table 4 and clearly indicate that the proposed electrode show the better potentiometric performance characteristics such as low detection limit, wide linearity range, short response time and wide working pH range than the previously reported ones and is closed to the best of them.

Table 4. Comparison of the proposed Co^{2+} selective electrode and the some previously reports

Ionophore	Slope	Detection limit (M)	Linear range (M)	Response time (s)	pH	Ref.
1-(6-choloro quinoxaline-2-yl) hydrazine	29.4	1×10^{-6}	1×10^{-1} - 3×10^{-6}	10	3-8	[1]
4-tert-butyl thia calyx[4] arene	29.5	3.5×10^{-7}	1×10^{-1} - 1×10^{-6}	10	4-6.5	[22]
5,11,17,23-Tetra-tert-butyl-25,27-bis-[2-(8-oxyquinoline)ethoxy]-26,28-dihydroxy calyx[4] arene	30.2	8.5×10^{-7}	1×10^{-1} - 6×10^{-6}	15	2.5-6.5	[35]
Palladium (II) dicholoro acetyl thiophen fenhone azine	29.6	8×10^{-7}	1×10^{-1} - 1×10^{-6}	25	3-7	[36]
p-(4-n-butylphenylazo) calyx[4]arene (i)	29.0	4×10^{-6}	1×10^{-1} - 9.2×10^{-6}	25	4-7.2	[37]
oxime-2-(1- cyclohexenyl) cyclohexanone	32.1	6×10^{-7}	3×10^{-2} - 7×10^{-7}	10	3.5-8.5	This work

4. CONCLUSION

In this study, oxime-2-(1-cyclohexenyl) cyclohexanone was synthesized and utilized as a new and selective sensing element in fabrication of a PVC based potentiometric membrane electrode. The electrode reveals a remarkable Nernstian slope (32.1 ± 0.5 mV per decade) over a wide concentration range of 7×10^{-7} to 3×10^{-2} with detection limit of 6×10^{-7} mol L^{-1} for potentiometric determination of Co^{2+} ions. Wide pH range, rapid response time and low detection limit of the proposed electrode make it a good device for potentiometric determination of Co^{2+} content in water and battery wastewater samples.

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