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Full Paper

PVC Membrane Potentiometric Sensor based on (E)-2-Acetyl-3-(butyl-amino)-N-phenyl buten-2-thioamide for Selective Determination of Iron(II)

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Abstract- In this paper, the construction and performance characteristics of novel PVC membrane Fe(II) ion selective electrodes based on (E)-2-acetyl-3-(butyl amino)-N-phenyl buten-2-thioamide is described. The electrode exhibits linear responses with Nernstian slope of 30.0±0.2 mVdecade^{-1} of activity within the Fe(II) ion concentration range of 3.5×10^{-7} to 1.2×10^{-2} M. This sensor is applicable in a pH range of 5.0-7.0. The lower detection limit is 1.6×10^{-7} M. It has a response time less than 10 s and can be used practically for a period of at least 2 months without any measurable divergence in results. The isothermal temperature coefficient of the sensor was assessed 1.24×10^{-3} V/^{o}C. The electrode showed excellent selectivity towards Fe(II) ion over a wide range of alkali, alkaline earth, and transition metals ions. It was successfully applied for the direct determination of Fe(II) ions in real samples.

Keywords- Ion-Selective Electrode, Fe(II) Determination, PVC Membrane, Potentiometry, (E)-2-Acetyl-3-(butyl amino)-N-phenyl buten-2-thioamide

1. INTRODUCTION

The importance of iron (Fe) in environmental, industrial, and medical applications as well as human physiology and many other biological systems is very well known [1]. Fe is an

essential nutritional element for all life forms and biotic enzymes (such as catalase). It plays a central role in the biosphere and serves as the active center of proteins responsible for O_2 and electron transfer [2]. The environmental and biological effectiveness of Fe depends on its chemical properties, such as valence, solubility, and the degree of chelating characteristic [3]. Metallic iron and its compounds are widely used in various industrial processes and may enter into natural waters through the discharge of wastes. Also iron alloys are used as containers and pipelines for supplying water in many areas [4]. In comparison with other transition metals, iron is considered as being a moderately toxic element, but medical research studies have shown that the toxic doses of iron and its compounds can lead to serious problems [5]. On the one hand, if the iron concentration exceeds the normal level in the body, it may become a potential health hazard. Excess iron in the body causes liver and kidney damage (hemochromatosis). Some iron compounds are suspected to be carcinogens [6-9]. Dissolved Fe in the natural water is found as both Fe(II) and Fe(III) forms with transformations between these states, which is of great interest in both atmospheric chemistry and oceanography [10]. Therefore, it is very important to develop sensitive methods for quantitative determination of trace Fe in various matrices. Potentiometric detection based on ion-selective electrodes (ISEs), as a simple method, offers several advantages such as fast response, easy preparation, simple instrumentation, wide linear dynamic range, relatively low detection limit, reasonable selectivity, application in colored, turbid and viscous solutions and low cost [11]. The technique is often non-destructive, adaptable to small sample volume with possible applications in real-time analysis. Because of these merits, the number of available sensors is being increasingly realized over the last few years.

Electroanalytical methods have attracted more attention in recent years for environmental and biological compound determination due to their sensitivity, accuracy, lower cost, and simplicity [12-22].

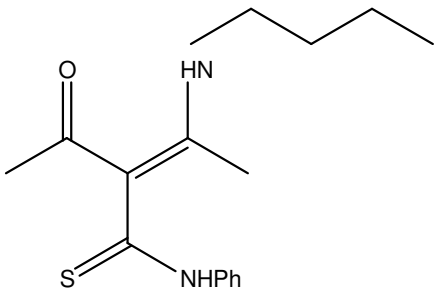

Fig. 1. Chemical structure of ionophore (E)-2-acetyl-3-(butyl amino)-N-phenyl buten-2-thioamide

The purpose of this work was development of membranes of (E)-2-acetyl-3-(butyl amino)-N-phenyl buten-2-thioamide (Fig. 1) as an electroactive material, sodium tetraphenyl

borate (NaTPB) as an anionic additive and dibutyl phthalate (DBP) as a solvent mediator in PVC matrix to construction of a Fe(II) selective sensor. The constructed sensor was applied for determination of Fe(II) in some real samples.

2. EXPERIMENTAL

2.1. Reagents

Analytical reagent grade chemicals and doubly distilled water were used for preparing all aqueous solutions. Chloronaphthalene (CN), dioctyl phthalate (DOP), dibutyl phthalate (DBP) and dimethyl sebacate (DMS) were obtained from Aldrich (Milwaukee, WI, USA). High molecular weight poly vinyl chloride (PVC) powder, tetrahydrofuran (THF), acetophenone (AP), oleic acid (OA), sodium tetraphenylborate (NaTPB) from Merck (Darmstadt, Germany) or Fluka (Buchs, Switzerland) were used as received. The nitrate or chloride soulfat salts of all cations used (all from Merck) without further purification except for vacuum drying over P_2O_5. Ligand (Fig. 1) was synthesized and purified as described elsewhere [23].

2.2. Apparatus and EMF measurements

Atomic absorption spectrophotometric measurements were made on AA220 spectrometer (Varian, Australia) under the recommended conditions based on the manufacturer's instructions. All the potential measurements were carried out using a 713 pH-mV meter (Metrohm, Swiss) at laboratory ambient temperature. The emf measurements with the polymeric membrane electrode (PME) were carried out with the following cell assemblies:
Ag/AgCl, KCl (3 M) | internal solution, 1.0×10^{-3} M Fe(II) | PVC membrane |Sample solution| Ag/AgCl, KCl (saturated)

All the emf observations were made relative to an Ag/AgCl double junction reference electrode (Azar electrode, Iran) under magnetic stirring. A silver/silver chloride electrode containing a 3 M solution of KCl was used as the internal reference electrode. Activities were calculated according to the Debye–Huckel procedure, using following equation:

$$\log f = -0.511\, z^2 \left[\mu^{1/2}/(1 + 1.5\mu^{1/2}) - 0.2\, \mu \right]$$

Where μ is the ionic strength and z the valence of ion.

2.3. Fabrication of electrode

For the PVC membrane preparation, 30 mg from the powdered PVC were blended thoroughly with 63 mg from the DBP plasticizer, 2 mg from the NaTPB additive and 2 mg from the ionophore in 2-4 mL of fresh THF. Then was shaken vigorously and the clear solution has been poured (so that bubbles are not formed) into glass rings (20 mm i.d.) placed

on a smooth glass plate. The master PVC membrane of about 0.4 mm thickness was sectioned with a cork borer (5 mm diameter) and attached to a polyethylene tubing (5 cm length and 4 mm i.d. on top) with PVC–THF slurry. Then the tube was kept at the room temperature for about 24 h, before being filled with an internal filling solution of 1.0×10^{-3} M of ($Fe(NH_4)_2(SO_4)_2.H_2O$) and being soaked in a 1.0×10^{-1} M ferric ammonium sulfate solution for 24 h. A silver/silver chloride electrode was used as an internal reference electrode.

3. RESULTS AND DISCUSSION

3.1. EMF measurements

The existence of donating nitrogen, sulphur and oxygen atoms in the ionophore structure enabled ligand to act as a suitable ion carrier for some metal ions, which could be used in the PVC membranes. In the preliminary experiments, the ionophore was used as a neutral carrier to prepare polymeric membrane (PME) selective electrodes under identical conditions for a variety of metal ions. Fig. 2 shows the potential response of the ion selective electrodes (ISEs) based on ionophore for the different ions with Nernstian slope as compared to other metal resulting that electrode is sensitive to Fe^{2+} ion.

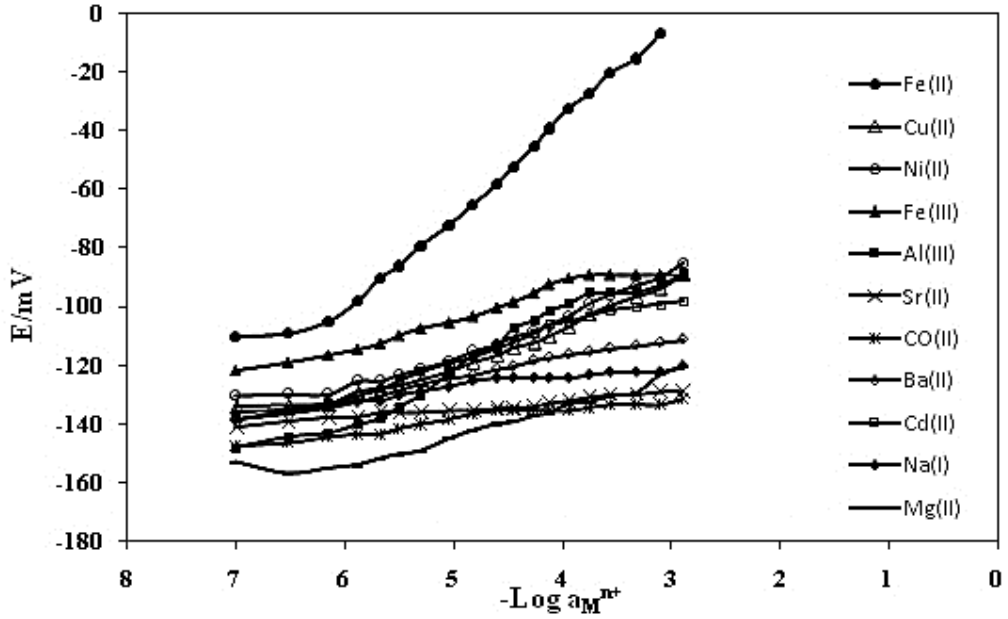

Fig. 2. Potential responses of different polymeric membrane electrodes

3.2. Effect of membrane composition

It is well known that the sensitivity and selectivity obtained for a given ionophore depend significantly on the membrane composition and the nature of solvent mediator and additives

used [24]. Thus some different compositions of the Fe^{3+}-selective membrane were optimized and the results are summarized in Table 1. It is generally accepted that the presence of lipophilic anions in a cation-selective membrane electrode diminishes the ohmic resistance, enhances the response behavior and selectivity, increasing also the sensitivity of the membrane electrodes [25,26]. Table 1 illustrates that the sensor slope in the absence of NaTPB is lower than the expected Nernstian value (membrane no.9). Nevertheless, the addition of 2% NaTPB will increase the sensitivity of the electrode response considerably, so that the membrane electrode can demonstrate a Nernstian behavior. It is reported that the response characteristics of ion-selective electrodes are largely affected by the nature and amount of plasticizer used. This is due to the influence of plasticizer on the dielectric constant of the membrane phase, the mobility of the ionophore molecules and the state of ligands [27]. Our experience on using several plasticizers for preparation of membrane (Table 1, Nos. 3–7) showed that plasticizer DBP perform best as it exhibits the widest working concentration range and near-Nernstian slope. However, the membrane sensor with a composition of 63% (w/w) DBP in the presence of 30% (w/w) PVC, 5% (w/w) ionophore, and 2% (w/w) NaTPB (No. 9, Table 1) exhibits the best performance.

Table 1. Optimization of the membrane ingredients

No.	PVC %	Plasticizer %	Ligand %	Anionic Additive %	Slope [a] (mV decade^{-1})	Linear Range (M)	Detection limit (M)
1	30	65(DBP)	5	0(NaTPB)	15.4±0.5	7×10^{-6}-1.6×10^{-3}	0.3×10^{-6}
2	30	64(DBP)	5	1(NaTPB)	25.8±0.6	4.4×10^{-6}-1.3×10^{-3}	1.6×10^{-6}
3	30	63.5(DBP)	5	1.5(NaTPB)	28.3±0.6	2.3×10^{-7}-1.2×10^{-3}	1.2×10^{-7}
4	30	63.5(DOP)	5	1.5(NaTPB)	21.5±0.3	3.0×10^{-7}-1.6×10^{-3}	1.4×10^{-7}
5	30	63.5(DMS)	5	1.5(NaTPB)	25.6±0.4	5.0×10^{-7}-1.0×10^{-3}	2.3×10^{-7}
6	30	63.5(AP)	5	1.5(NaTPB)	16.4±0.8	2.0×10^{-7}-1.8×10^{-3}	1.2×10^{-7}
7	30	63.5(CN)	5	1.5(NaTPB)	17.0±0.7	7.1×10^{-7}-9.4×10^{-4}	5.0×10^{-7}
8	30	63.5(DBP)	5	1.5(OA)	26.9±0.5	7.0×10^{-7}-9.4×10^{-4}	3.5×10^{-7}
9	**30**	**63(DBP)**	**5**	**2(NaTPB)**	**30.0±0.2**	**3.5×10^{-7}-1.2×10^{-2}**	**1.6×10^{-7}**
10	30	64(DBP)	4	2(NaTPB)	28.6±0.3	5.0×10^{-7}-1.2×10^{-2}	3.0×10^{-7}
11	30	64(DBP)	3	3(NaTPB)	27.5±0.6	3.5×10^{-7}-1.3×10^{-2}	1.3×10^{-7}
12	30	66(DBP)	2	2(NaTPB)	26.0±0.2	2.7×10^{-7}-1.9×10^{-3}	1.2×10^{-7}
13	30	66(DBP)	1	2(NaTPB)	19.1±0.6	3.2×10^{-7}-1.1×10^{-3}	1.3×10^{-7}

[a] Mean values of slopes for three replicate measurements ± SD

3.3. Effect of internal reference solution

The effect of the internal solution concentration of the membrane-selective electrode for Fe^{3+} ion based on ionophore was investigated. The concentration was varied from 1.0×10^{-4} to 1.0×10^{-2} M (Fig. 3) and the results show that the variation of the internal ($Fe(NH_4)_2(SO_4)_2.H_2O$) concentration did not cause any significant difference in potential response. Therefore, 1.0×10^{-3} M concentration of the reference solution that was quite appropriate for smooth functioning of the proposed sensor.

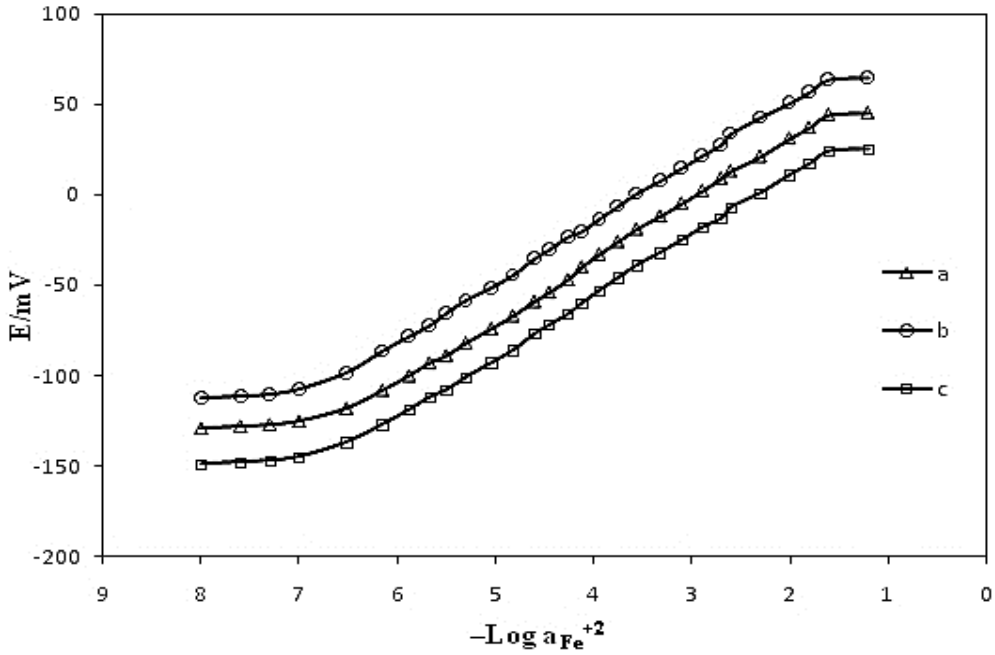

Fig. 3. Effect of concentration of an internal reference solution on electrode response at various concentrations of (a) 1×10^{-4} M, (b) 1×10^{-3} M, and (c) 1×10^{-2} M

3.4. Effect of pH of the test solution

The pH parameter was varied in the range of 2.0-9.0 at two certain Fe(II) ion concentration values (1.0×10^{-5} M) by adjusting the pH of the test solution with HCl and NaOH (Fig. 4). It is seen that the potentials remained constant in the pH range 5.0–7.0. Beyond this range (pH<5.0), a gradual reduction in the potential was observed may be due to the protonation of the ionophore, which results in an increased potential of the system by increasing concentration of protonium ions in solution.

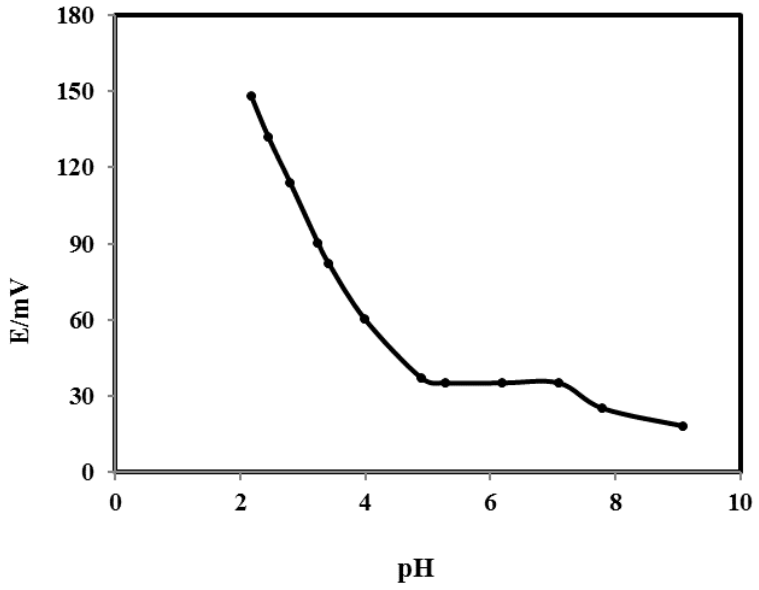

Fig. 4. Effect of pH of the test solution on the potential response of the Fe^{2+} selective electrode at 1×10^{-5} M

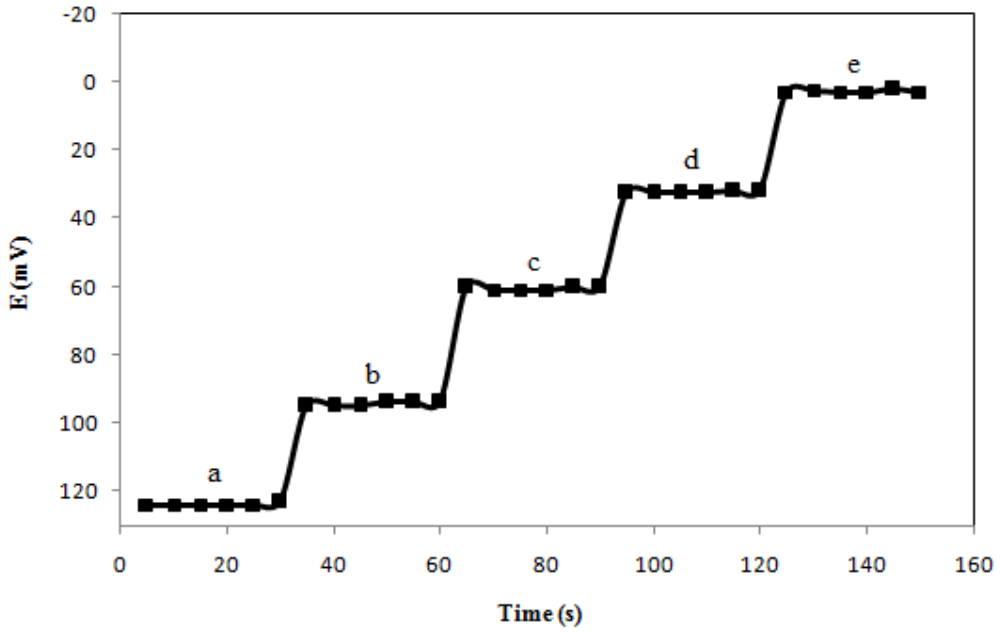

Fig. 5. Dynamic response of membrane electrode for step changes in concentration of Fe^{2+}: (a) 1×10^{-6} M,(b) 1×10^{-5} M, (c) 1×10^{-4} M, (d) 1×10^{-3} M, (e) 1×10^{-2} M

3.5. Response and life time

Dynamic response time is an important factor for an ion-selective electrode. The response time of electrode was determined by measuring the time required to achieve a 90%

of the steady potential [28]. In this study, the dynamic response time of the membrane was measured at various activities 1.0×10^{-6}–1.0×10^{-3} M of the test solutions (Fig. 5). The response time of sensors was found to be less than 10 s at various activities of the test solution. This is most probably because the fast exchange kinetics of complexation-decomplexation of Fe(II) ion with the electrode at the test solution–membrane interfaces.

3.6. Calibration plot and statistical data

The dynamic response time of the membrane was measured (prepared under optimal membrane ingredients) at varying concentrations from3.5×10^{-7} to 1.2×10^{-2} M of the test solutions as depicted in Fig. 6, where the stable potentials can be obtained within 10 s. whereas the slope of the calibration graph was 30.0±0.2 mV per decade. The detection limit, as determined from the intersection of the two extrapolated segments of the calibration graph, was 1.6×10^{-7} M.

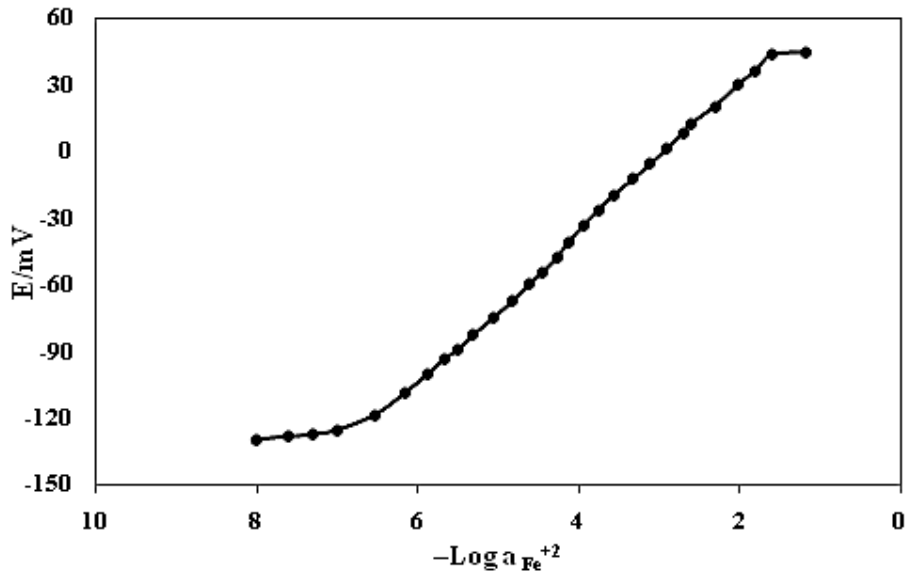

Fig. 6. Calibration graph for the PME, The PVC membrane composition used is that given for membrane No. 9

3.7. Selectivity coefficients

As it is deduced from the name of these devices, the selectivity behavior can be regarded as the most important characteristic of an ISE, determining whether a reliable measurement in the target sample is possible [29]. The potentiometric selectivity coefficients, $K^{Pot}_{Fe^{2+},M}$ of the iron sensor were evaluated by the matched potential method (MPM), which is recommended

to overcome the limitation associated with the methods based on the Nicolsky-Eisenman equation [30,31]. These limitations include non–Nernstian behavior of interfering ions and inequality of charges of primary and interfering ions. The obtained results for the $K^{Pot}_{Fe^{2+},M}$ of Fe^{2+} ion selective electrode are given in Table 2. As seen from Table 2, with the exception of Fe^{3+}, Mg^{2+} ions, the selectivity coefficients obtained for all other cations were in the order of 10^{-3}, indicating that these ions do not disturb the functioning of the Fe^{2+} ion-selective electrode significantly.

Table 2. Selectivity coefficients of the developed Fe(II) electrode according to MPM Method

M^{n+}	$K^{MPM}_{Fe^{2+},M}$	M^{n+}	$K^{MPM}_{Fe^{2+},M}$
Ca^{2+}	9.2×10^{-3}	Cu^{2+}	3.0×10^{-3}
Hg^{2+}	6.1×10^{-3}	Na^{+}	1.0×10^{-3}
Ni^{2+}	5.4×10^{-3}	Fe^{3+}	2.0×10^{-2}
Mn^{2+}	4.1×10^{-3}	Cr^{3+}	5.3×10^{-3}
Sr^{2+}	2.7×10^{-3}	Ag^{+}	3.1×10^{-3}
Co^{2+}	2.5×10^{-3}	Al^{3+}	3.8×10^{-3}
Pb^{2+}	2.5×10^{-3}	Mg^{2+}	1.2×10^{-2}

3.8. Effect of non-aqueous media

For investigation of electrode performance in non-aqueous media, it was used of three water-methanol, water-ethanol and water-acetone mixtures and the results obtained are represented in Table 3. The membrane works satisfactorily in non aqueous media up to 10% (v/v) content of methanol and ethanol and 5% (v/v) acetone so in these mixtures the working concentration range and slope remain unchanged. However, above 10%(v/v) methanol and ethanol and 5%(v/v) acetone, the slope and working concentration range reduced and potentials showed drift. Therefore, the electrode can be satisfactorily used in these media with the above-mentioned percentages.

3.9. Effect of temperature

The trends of changes of electrode performance with temperature are investigated at test solution temperatures 20, 30, 40, and 50 ^{o}C for the Fe^{2+}electrode and results are represented in Table 4. The electrode exhibits good Nernstian behavior in the temperature range (20-50 ^{o}C). The standard cell potentials (E°_{cell}) were determined at different temperatures from the respective calibration plots as the intercepts of these plots at pFe^{2+}=0, and were used to

determine the isothermal temperature coefficient $\left(\frac{dE^{\circ}}{dt}\right)$ of the cell with the aid of the following equation [32]:

$$E^{\circ}_{cell} = E_{25} + \left(\frac{dE^{\circ}}{dt}\right)(t - 25)$$

Table 3. Performance of PVC membrane electrode (No. 9) in partially non-aqueous medium

Non-aqueous content % (v/v)	Linear range (M)	Slope(mV decade^{-1})
0	3.0×10^{-7}-1.0×10^{-2}	29.2±0.2
Methanol		
5	7.0×10^{-7}-1.0×10^{-2}	29.2±0.2
10	7.0×10^{-7}-1.0×10^{-2}	29.2±0.4
15	3.0×10^{-7}-1.0×10^{-2}	26.6±0.5
20	3.0×10^{-7}-1.0×10^{-2}	25.4±0.5
Ethanol		
5	7.0×10^{-7}-1.0×10^{-2}	29.7±0.3
10	3.5×10^{-7}-1.0×10^{-2}	27.9±0.4
15	3.5×10^{-7}-1.0×10^{-2}	26.3±0.5
20	1.3×10^{-7}-1.0×10^{-2}	26.0±0.4
Acetone		
5	3.5×10^{-7}-1.0×10^{-2}	28.5±0.5
10	5.4×10^{-7}-1.0×10^{-2}	26.6±0.7
15	5.0×10^{-7}-1.0×10^{-2}	25.0±0.7
20	3.0×10^{-7}-1.0×10^{-2}	22.4±0.6

Table 4. Performance characteristics of the PVC membrane electrode at different temperature

Temperature (°C)	Slope (mV decade^{-1})	E°_{elec} (mV)	E°_{cell} (mV)	Linear range (M)
20	27.97	-145.07	-140.17	3.2×10^{-7} – 1.0×10^{-2}
30	29.98	-123.65	-128.55	3.2×10^{-7} – 1.0×10^{-2}
40	31.79	-102.58	-117.28	3.2×10^{-7} – 1.0×10^{-2}
50	33.60	-78.09	-102.59	3.2×10^{-7} – 1.0×10^{-2}

According to the equation the standard potential of electrode E°_{cell} *vs*. (t-25) gave a straight line, the slope of the straight line obtained (Fig. 7) represents the isothermal coefficient of the electrode. The value of $\left(\frac{dE^{\circ}}{dt}\right)$, obtained 1.24×10^{-3} V/°C for the polymeric membrane Fe^{2+} ion- selective electrode, revealing the high thermal stability of the electrode within the investigated temperature ranges.

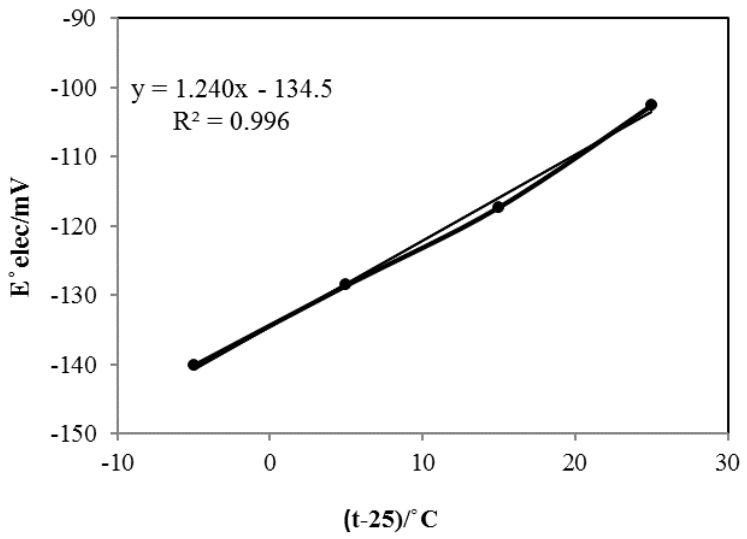

Fig. 7. Variation of standard electrode potential with temperature for PVC membrane electrode

Table 5. Determination of Fe(II) in different samples by proposed electrode

Sample	Added (μg mL^{-1})	Found by Present method	Found by AAS
Tap Water	0.0	0.34±0.04	0.35±0.02
	0.5	0.84±0.02	0.86±0.04
Skim cow's milk	0.0	0.62±0.02	0.65±0.01
	0.5	1.08±0.03	1.12±0.02
Ferrous sulfate syrup	0.0	80.08±0.5	82.32±0.1

3.10. Analytical Applications

The proposed electrode was successfully used to the determination of Fe(II) ions in the tap water, skim cow's milk and ferrous sulfate syrup by standard addition method. Determined results by the proposed electrodes were in good agreement with data gained by AAS method Table 5. Results indicate that the proposed sensor can be efficiently applied for estimation of iron content in real samples.

Table 6. Comparison of Fe(II) selective electrodes with those previously reported in literature

Carrier	Slop (mV decade^{-1})	Linear Range (M)	Detection Limit (M)	pH Range	Response Time (s)	Ref.
N-Phenylaza-15-Crown-5	29.2±0.2	1.0×10^{-6}-1.0×10^{-2}	-	5.0-7.0	15	[33]
tris- (1,1 0-phenanthroline)	30.0±0.2	1.0×10^{-6}-1.0×10^{-4}	4.0×10^{-7}	3.0-9.0	20	[34]
benzo-18-crown-6 crown ether	-	1.0×10^{-6}-1.0×10^{-4}	6.0×10^{-7}	8.3	-	[35]
4-(2-Pyridylazo)-resorcinol (PAR)	-	4.4×10^{-7}-3.0×10^{-6}	1.4×10^{-7}	9.11	-	[36]
E-2-acetyl-3-(butyl amino)-N-phenyl buten-2-thioamide	30.0±0.2	3.5×10^{-7}-1.2×10^{-2}	1.6×10^{-7}	5.0-7.0	<10	This work

3.11. Comparison of the characteristics of the Fe^{2+} electrodes

Table 6, lists slope, linear range, detection limit, pH range, response time and of some of the Fe(II)-selective electrodes based on a variety of different ionophores against proposed membrane electrodes for comparative purposes [33-36]. The proposed electrode has been found to be better in terms of its wide concentration range compare to most of the sensors. The proposed electrodes represent short response time and detection limit in comparison to those reported for other reported systems. It is seen, selectivity of the sensors improved with respect to the various secondary ions, especially Fe(III), which had been reported to be major interfering ions in many of the reports.

4. CONCLUSION

In the present study, electrode was prepared based on (E)-2-acetyl-3-(butyl amino)-N-phenyl buten-2-thioamide. The main virtue of the proposed potentiometric sensor are its simplicity of preparation, short conditioning time, fast response time, broad dynamic range, Nernstian behavior, low detection limit, low cost, and fairly good selectivity.

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