



Original Research Article

Determination of Magnesium (II) by a Coated Graphite Electrode Based on Risperidone as an Ionophore

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ABSTRACT

In this research, a coated graphite electrode was constructed for determination of Mg^{2+} in pharmaceutical supplements. The designed sensor demonstrates an ideal Nernstian slope ($30.1 \text{ mV. Decade}^{-1}$) over a wide concentration range (1×10^{-6} - $1 \times 10^{-1} \text{ Mol L}^{-1}$). The selectivity of the sensor was evaluated over 16 different cations by matched potential method and no serious interference was observed from them. The designed electrode could also be used in partially non aqueous mediums up to the presence of 20% of organic solvents without any tangible alteration in Nernstian slope and linearity domain. The response time and life span of the proposed electrode were 20s and 2 months respectively. The function of the electrode was inspected in solutions with different pH value and also as an indicator electrode in potentiometric titration with EDTA.

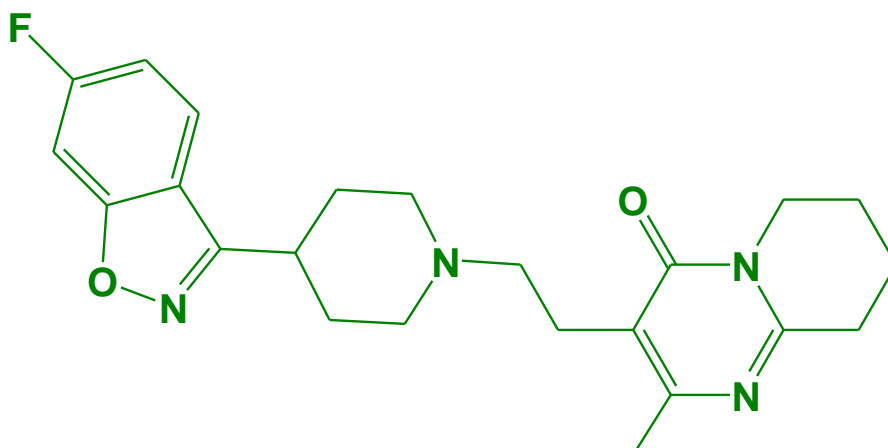
Keywords: Magnesium, Potentiometry, Ion selective electrode, Risperidone

Introduction

Today, the problem of counterfeit medicines has become a dilemma and the problem of global. Although these pharmaceutical products are very similar in appearance to the original products but it has a very low quality and they can endanger the lives of patients. Quality control of medications is very important and one of the main ways to control the quality of these products is to measure the amount of active ingredient in these products [1]. Magnesium metal is also used in many pharmaceutical supplements due to its unique biological properties. This dual capacity catalysis reduces the direct stimulating effect of acids by neutralizing gastric acid and forming magnesium chloride and water and by increasing the pH, paxine can be deactivated. Also, this metal hydroxide molluscan can enhance the components of the gastrointestinal mucous membrane and increase the tonicity of the sphincter esophagus and stomach. In addition to treating the reflux and reducing the acidity of the stomach, magnesium, by increasing the osmolarity of the contents of the intestine, causes water to enter the intestine, resulting in dilatation and stimulation of the movement of the intestines and the emptying of the intestine. In other words, it is a very effective medication for the treatment of constipation. Other therapeutic benefits of this cation include treatment of seizure attacks, muscle spasms, pregnancy toxicity, early delivery, barium toxicity, and stress relief. Therefore, this metal is available to patients in the form of sulfate, hydroxide and oxide in suspension form, tablets and injectables [2-5]. Measuring is important in order to control the quality of these pharmaceutical products. Several methods for measuring magnesium, including atomic absorption spectroscopy, plasma mass coupled with mass spectrometry and ion chromatography, have been designed [6-8] But these methods require expensive, expensive devices, which may not be easy to fund. These methods are time-consuming and require high-performance applications. Designing an ion selective electrode to measure this ion seems highly logical. Because these electrochemical sensors are very affordable and affordable, they are very selectable, accurate and accurate. Other advantages of ion selective membrane can be portability and applicability in opaque and colored samples [9-14]. So far, many selective ion electrodes have been made based on various ligands and ionophors for different analyte measurements, but the use of drug molecules as ion exchangers in the membrane structure is less attractive to chemists. But many of these molecules contain sulfur and nitrogen hetero atoms in their structure and can establish sustainability complexes with

different metals, this function can be used in extraction methods as well as selective electrodes for measuring cation minerals.

The structure and name of iopac, the Risperidone drug, shown in Fig. 1, is abundantly used to treat schizophrenia, autism and acute phase of mania in patients with bipolar disorder. It has little solubility in water and since it has nitrogen and oxygen heteroatoms, it is expected that it will interact well with metals as a ligand. In this study, Risperidone was first used as an ion exchanger to make a coated graphite electrode for measuring magnesium [15].



3-[2-[4-(6-fluoro-1,2-benzoxazol-3-yl)piperidin-1-yl]ethyl]-2-methyl-6,7,8,9-tetrahydropyrido[1,2-a]pyrimidin-4-one

Figure 1. The structure and name of iopac risperidone

Experimental section

Reactors and Raw Materials Required

Acetyl phthalate (DOP), Nitrobenzene (NB), potassium tetrakis (4-chlorophenyl) borate (KTY), sodium hydroxide, nitric acid, acetone and ethanol were purchased from Sigma Aldrich Company. The active ingredient of Risperidone was obtained from Sobhan Pharmacy Factory. Nitrate salt of various metals, polyvinyl chloride (PVC) and tetrahydrofuran (THF) from Merck and Sigma were purchased. Magnesium oxide supplement was purchased from Jaline and 21 Century pharmaceutical companies. All of the aforementioned materials were purchased in the same manner and were used without further purification steps. The solutions used for distilled water were deionised and their activity was calculated by the Hockel equation.

Devices

The electrode system potential was recorded by multimeter (Sairan, Iran) with a precision of ± 0.1 mV and at 25 ° C. A Saturation Calomel Electrode (SCE) was used as an external reference electrode, and a grate bar of 3 cm in diameter and 3 mm in diameter was used to make a polymeric membrane coated graphite electrode. The pH of the solutions was adjusted by conventional glass electrodes and the Shimadzu G / 670 atomic absorption device was used to analyze the actual samples.

Preparation of membrane

Different amounts of plasticizers (DOP, NB), ionophore (risperidone), ionic additive (KTY) and PVC were weighed and mixed with each other and dissolved in 3 ml THF. The resulting mixture is placed under the hood for some time to evaporate the tetrahydrofuran in it and create a thick, viscous mixture of like. At this time, the graphite rod is placed within the solution for 15 seconds to form a thin membrane with a thickness of 0.4 mm at its surface. In the next step, the graphite rod was placed at room temperature for one day to dry the membrane. The resulting membrane electrode was then placed in a solution of 1×10^{-3} Mol L⁻¹ magnesium nitrate for 24 hours.

Discussion and Conclusion

Investigating the interaction of ionophore with various metals

In order to understand what is ionophore (risperidone), which one of the metals interacts better. A membrane was made by combining 5% Risperidone, 30% PVC, 2% KTY and 63% DOP, and after drying in a solution of 1×10^{-3} Mol.L⁻¹, the nitrate content of chromium (III), magnesium (II), mercury (II), Silver (I), copper (II), zinc (II), cobalt (II), manganese (II), nickel (II) and iron (III) were prepared for 24 hours. Then, the potential response of the prepared electrode to these ions was recorded and the resulting calibration curves were plotted as shown in Fig. 2.

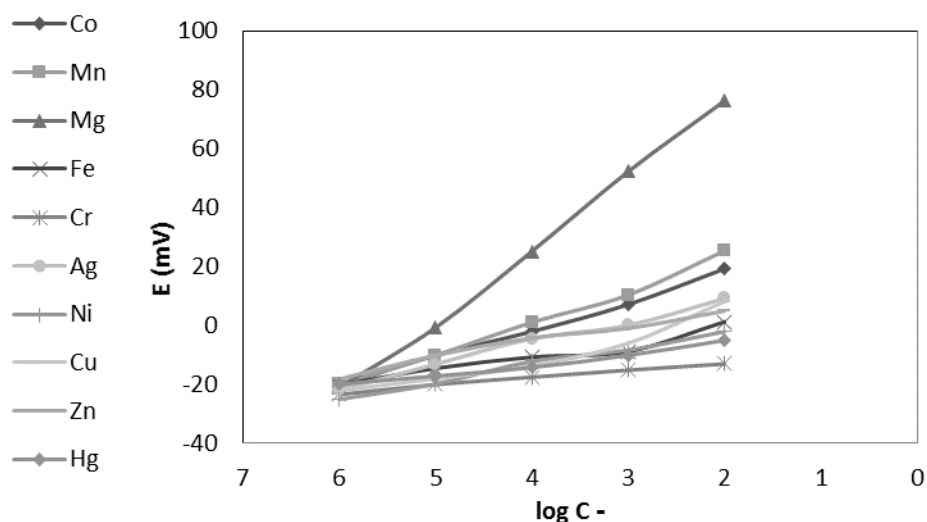


Figure 2. Investigating the interaction of ionophore with various metals

Optimization of the membrane and its effect on the linear range and sensitivity of the sensor

As shown in Table 1, the widest linear range, the highest Nernstian gradient and also the lowest detection limit of membrane No. 10 were found to have 7% ionophore, 2% ion additive (KTY), 31% polyvinyl chloride and 60% (DOP). The first variable studied was the amount of ion exchanger, as it is known that the potential response of the sensor has a direct relation to the ionophore value, so that the monomers 1 and 2 without ionophore have a very low linear velocity and Nernstian inclination, but with increasing the amount of ionophore up to 7% increases the linear range and slope of the calibration curve. However, the further addition of ionophore leads to a slope decrease. This phenomenon appears to be due to the saturation of the membrane surface and the localized non-uniformity [17]. Another factor that can affect the performance of the ion selective electrodes is astonishingly the nature of the plasticizer. In this study, two nitrobenzene and dioctyl phthalate solvents were examined (membranes 9 and 10), and as it is known, the DOP solvent significantly increased the sensitivity of the sensor, since the calibration curve slope from 28.3 to 30.1 mV/decade has reached. For this reason, dioctyl phthalate was selected as a plasticizer. It seems that dielectric constant and lower polarity DOP reduce the disturbance of ions and thus improve the performance of the designed sensor. The effect of the presence of KTY ion additive on sensor

performance was also investigated and it was found that the membrane sensor made in the absence of these lipophilic anions showed much less sensitivity and in fact the slope of the calibration curve from 1.30 millivolts per membrane per membrane No. 10 has been reduced by 26.2 mV to mercury in membrane No. 3 without ionic additive. It seems that these lipophilic anions reduce the ohmic resistance of the membrane and, when ionophore has little extraction ability, it can improve the analyte extraction process and enhance the selectivity of the sensor [18].

Table 1. Effect of membrane percent composition on sensor response designed for risperidone base

Membrane No.	Unofur (Risperidone)	Ionic additive (KTY)	Plasticizer	PVC	line slope (mV.Decade ⁻¹)	Linear range (Molar)	Limit detection (Molar)
1	0	0	(DOP)60	40	8/8	$1 \times 10^{-2} - 1 \times 10^{-3}$	-
2	0	2	(DOP)60	38	10/1	$1 \times 10^{-2} - 1 \times 10^{-4}$	-
3	7	0	(DOP)60	33	26/3	$1 \times 10^{-2} - 1 \times 10^{-6}$	9×10^{-7}
4	5	2	(DOP)63	30	20/7	$1 \times 10^{-2} - 1 \times 10^{-6}$	9×10^{-7}
5	3	2	(DOP)60	35	19/4	$1 \times 10^{-2} - 1 \times 10^{-5}$	7×10^{-6}
6	2	2	(DOP)60	36	18/8	$1 \times 10^{-2} - 1 \times 10^{-5}$	9×10^{-6}
7	8	2	(DOP)60	30	24/4	$1 \times 10^{-2} - 1 \times 10^{-6}$	9×10^{-7}
8	10	1	(DOP)59	30	25/7	$1 \times 10^{-2} - 1 \times 10^{-6}$	9×10^{-7}
9	7	2	(NB)60	31	28/3	$1 \times 10^{-2} - 1 \times 10^{-6}$	8×10^{-7}
10	7	2	(DOP)60	31	30/1	$1 \times 10^{-2} - 1 \times 10^{-6}$	8×10^{-7}

The performance of the electrode in the presence of organic solvents

In order to investigate the sensitivity and performance of the potentiometric sensor designed in non-aqueous environments, a number of magnesium nitrate solutions were prepared in a concentration range of 1×10^{-2} - 1×10^{-6} Mol.L⁻¹ which contained different amounts of organic solvents of ethanol and acetone. Then, the potential sensor response was evaluated and the linear range and slope of the calibration curves obtained in Table 2 were reported. This sensor is nerve-tilt and can maintain its linear range even in the presence of 20% organic solvents. However, a further increase in the proportion of these solvents leads to a sharp decrease in sensitivity and an increase in the detection limit. It seems that since ionophore is used for lipophilic, by adding organic solvents and reducing the polarization of ion exchange solution, it exits from the membrane tissue and leaks into the sample solution and shows its effect by decreasing the gradient and increasing the detection rate.

Table 2. The effect of organic solvents on the potential response of the sensor

Amount of non-blue content (%v/v)	Linear range (Molar)	line slope (mV.Decade⁻¹)
0	$1 \times 10^{-2} - 1 \times 10^{-6}$	30/1
ethanol		
5	$1 \times 10^{-2} - 1 \times 10^{-6}$	30/1
10	$1 \times 10^{-2} - 1 \times 10^{-6}$	29/5
15	$1 \times 10^{-2} - 1 \times 10^{-6}$	29/2
20	$1 \times 10^{-2} - 1 \times 10^{-6}$	28/7
25	$1 \times 10^{-2} - 3 \times 10^{-5}$	25/4
acetone		
5	$1 \times 10^{-2} - 1 \times 10^{-6}$	30/1
10	$1 \times 10^{-2} - 1 \times 10^{-6}$	29/7
15	$1 \times 10^{-2} - 1 \times 10^{-6}$	29/4
20	$1 \times 10^{-2} - 1 \times 10^{-6}$	28/3
25	$1 \times 10^{-2} - 9 \times 10^{-6}$	24/6
A mixture of acetone and ethanol with a ratio of 1:1		
5	$1 \times 10^{-2} - 1 \times 10^{-6}$	30/1
10	$1 \times 10^{-2} - 1 \times 10^{-6}$	29/6
15	$1 \times 10^{-2} - 1 \times 10^{-6}$	29/1
20	$1 \times 10^{-2} - 1 \times 10^{-6}$	28/2
25	$1 \times 10^{-2} - 6 \times 10^{-5}$	23/9

Effects of pH on potential response

In order to evaluate the performance of the sensor, designed at different pHs, a number of 1×10^{-4} molar solutions of magnesium nitrate were prepared and its pH was slightly increased in the amount of micro liters of very concentrated nitric acid and sodium hydroxide in the pH range of 1 to 11 was adjusted by glass electrode and at the next step, the solvent potential was recorded by membrane electrode. As shown in Fig. 3, the potential response of the electrode in the range of 4.3 to 7.8 is independent of pH. The potential oscillations in acidic pHs appear to be due to the competition of the proton ions with the analecte in the formation of the complex with ionophore (risperidone), and fluctuations in alkaline pHs can also be due to the reaction of the metal complex with the hydroxyl ion [19].

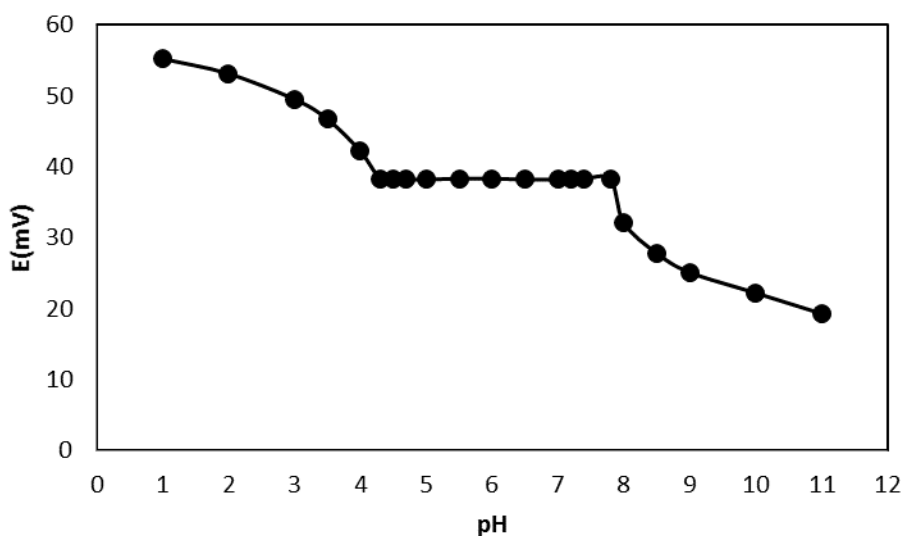


Figure 3. Effect of pH on magnesium sensor (II) function

Response time and sensor lifetime

The response time of an ion electrodes is defined as the moment the electrode is placed within the solution until it reaches 90% of the potential equilibrium or potential potential. In order to verify this parameter, the electrode was designed in solutions with a concentration difference of 10 times in a concentration range of 1×10^{-2} - 1×10^{-6} M and its potential was recorded per second.

Then the potential changes were plotted according to the time, the results are shown in Fig. 3. The proposed electrode takes 20 seconds to reach a stable and stable potential, which means the response time is 20 seconds. To estimate the lifetime of the electrode, three times the electrode calibration curve for magnesium ion was plotted, and the linear range, Narnis slope and detection limit were investigated. In Figure 4, the slope of the calibration curve is depicted in terms of the passage of time. As it is known, after a 2 month period, a significant drop in the slope of the Neristic is observed, in other words, the electrode loses its sensitivity. In addition, after this period, the detection limit of the sensor increased and the linear range narrowed [27-36].

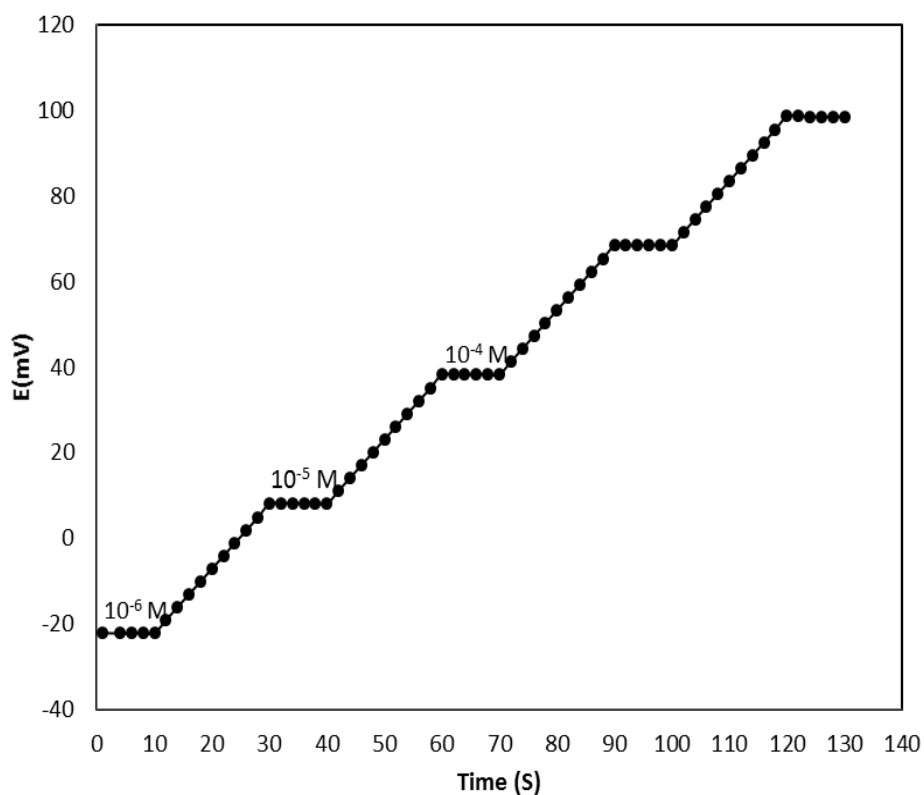


Figure 4. Dynamic Response Time Estimation of Risperidone-Based Graffiti-Coated Electrode

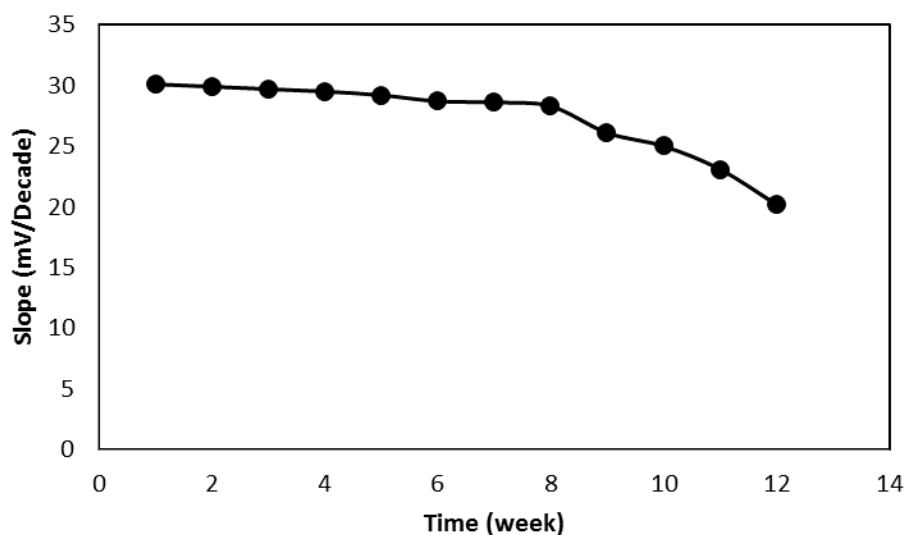


Figure 5. Examination of Magnesium Electrode (II) Lifetime Selection

Selectivity

The selectivity of a method in Analytical Chemistry is very important, since the more a parsing method is more selectable, its responses will be more correct and more repeatable. Hence, many methods have been designed to estimate the selectivity of selective electrodes, one of which is the most reliable and most commonly used potential. In this study, electrodes selectivity was investigated by a potentiality approach matched to 16 different ions. The closer the selectivity coefficient (KMPM) is to zero, however, indicates that the membrane electrode does not respond to it, and tends to be closer to the original analysis, but as far as this quantity is closer to 1, it indicates that The electrode cannot differentiate between the original ion and the intruder [20]. And as the data in Table 3 clearly shows that the calculated selectivity coefficients are much smaller than 1, and in some cases a membrane electrode is designed to be at least 100 to 1,000 times more likely to extract the main ion, magnesium (II).

Table 3. Designed Sensor Selectivity Examination

Annoying ions	MPM method selectivity	Annoying ions	MPM method selectivity
Cu ²⁺	2.1×10^{-2}	Ni ²⁺	7.2×10^{-3}
Cr ³⁺	5.4×10^{-2}	Mn ²⁺	6.6×10^{-2}
Cd ²⁺	1.3×10^{-3}	Zn ²⁺	9.7×10^{-4}
Hg ²⁺	4.1×10^{-3}	Ca ²⁺	8.6×10^{-3}
Ag ⁺	6.8×10^{-4}	K ⁺	1.9×10^{-2}
Fe ²⁺	1.6×10^{-3}	Na ⁺	3.6×10^{-3}
Pb ²⁺	2.8×10^{-2}	Al ³⁺	5.1×10^{-3}
Fe ³⁺	3.4×10^{-2}	Co ²⁺	3.3×10^{-2}

Potentiometric titration

Indirect potentiometric advantages have the advantage that it can sometimes be preferred to direct potentiometry. One of these benefits is the inability of ions and intruder species to respond to the sensor response, as well as the ability to measure concentration instead of activity. In this regard, the performance of the proposed coated graffiti electrode was investigated as a detector electrode in a potentiometric titration of 20 ml of a 1×10^{-5} molar solution of magnesium nitrate with EDTA of 1×10^{-3} m. The concentration of EDTA was deliberately chosen to be 100 times more concentrated, so that volume changes caused by the addition of titrand did not contribute to the potential for an analyte to change. As can be seen clearly in the resulting titration curve shown in Fig. 6, the equivalence volume (200 μ L) and the concentration of magnesium can be calculated with considerable accuracy on the titration curve.

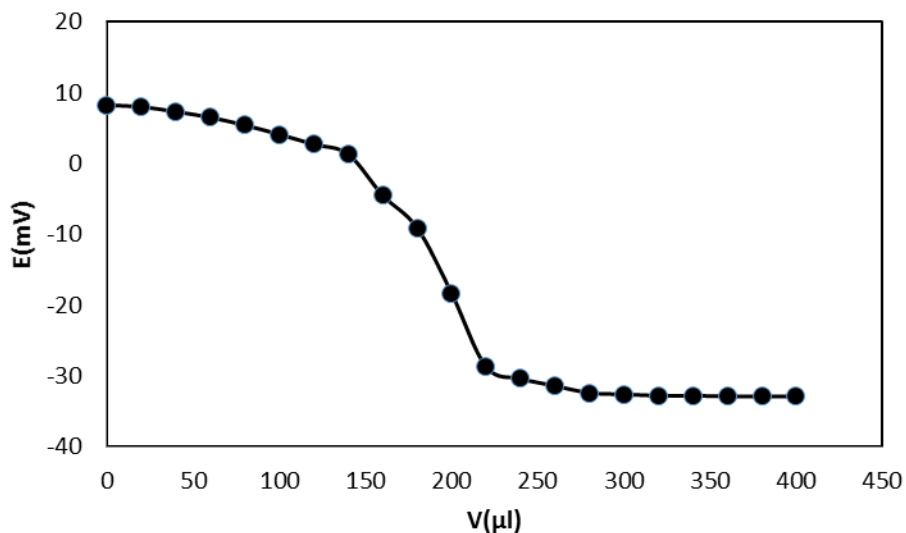


Figure 6. Titration curve 20 ml of 1×10^{-5} M solution of magnesium nitrate with EDTA of 1×10^{-3} M

Real Examples

In order to evaluate the performance of the sensor designed to measure the Mg^{2+} ion in true samples, the amount of this cation was designed in two supplements of magnesium oxide of the Jalin Pharmaceutical Company and 21 centrifugals by an electrode, and the atomic absorption spectrometry was measured with flame atomizer. For this purpose, 40 tablets were first weighed and taken from them. The pills were then powdered and mixed well. In the next step, the weight of one tablet of homogeneous powder was weighed and added to the diluted nitric acid, and the resulting mixture was placed in ultrasonic bath for 15 minutes to release magnesium ions [21-33]. After that, the solvent PH obtained from the previous step was neutralized by concentrated sodium hydroxide and after dilution, the magnesium content was analyzed by both methods, and the results are presented in Table 4. As can be seen from the results, the amount of magnesium obtained from both methods is very close to each other.

Table 4. Evaluation of Membrane Electrode Performance in Magnesium Measurement in Two Pharmaceutical Supplements

Sample name	The amount of magnesium measured in each tablet by the suggested sensor	The amount of magnesium measured in each tablet by the atomic absorption device
	(mgr)	(mgr)
Galenium Magnesium Supplement	248(\pm 1/9)	251 (\pm2/2)^a
21 Century Minium oxide supplement	253(\pm 2/5)	249 (\pm1/8)

Comparison of the electrode designed in this study with previous reports

In Table 5, the main characteristics of an electrodes, such as response time, lifetime, Narnis tilt, detection limit and linear range, have been compared. As can be seen, the electrode designed in this study has a lower detection limit than other magnesium potentiometric sensors, and in fact its ability to measure lower magnesium concentrations, especially in concentrations lower than 1×10^{-5} m, it is from other electrodes. And the same advantage alone demonstrates the superiority of the designed electrode.

Table 5. Electrode comparison proposed with several examples of previous reports

Reference No.	Response time (Second)	life span (Month)	Slope (mV.Decade ⁻¹)	Limit detection (Molar)	Linear range (Molar)
[22]	180	1	30	$6/3 \times 10^{-6}$	$1 \times 10^{-1} - 3/2 \times 10^{-5}$
[23]	15	4	31	Unreported	$1 \times 10^{-1} - 1 \times 10^{-5}$
[24]	15	3	29	Unreported	$1 \times 10^{-1} - 1/9 \times 10^{-6}$
[25]	13	5	29/2	Unreported	$1 \times 10^{-1} - 9/4 \times 10^{-6}$
[26]	15	2	30	8×10^{-6}	$1 \times 10^{-1} - 9/2 \times 10^{-6}$
This work	20	2	30/1	8×10^{-7}	$1 \times 10^{-2} - 1 \times 10^{-6}$

Discussion and conclusion

Measuring magnesium is important because of its key role in the body as a nutrient-rich substance and also a drug. In this study, a graphene-based graphene electrode based on ionophore riiseridone was designed. The sensor made the Narnis slope of 1.30 millivolts per decade in the wide linear range of 1×10^{-2} - 1×10^{-6} moles per liter, and the detection limit was 8×10^{-7} m. The selectivity of the sensor was evaluated against 16 other disturbing ions and, unfortunately, no disturbance was observed. The response time of the electrode is 20 seconds and its lifetime is 2 months. Additionally, the optimum pH range of the sensor was between 4.3 and 7.8. Ultimately, the electrode was made able to accurately measure the amount of manganese in two complementary drugs.

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