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Iron ion sensor based on functionalised ZnO nanorods

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Abstract

In this work, we are presenting an iron ion (Fe^{3+}) potentiometric sensor based on functionalized ZnO nanorods with selective ionophore (18 crown 6). Zinc oxide nanorods with a diameter of about 100 to 150 nm and 1 μm in length were grown on gold coated glass. The selective Fe^{3+} ionophore sensor with highly aligned ZnO nanorods has given high sensitivity, acceptable selectivity, reproducibility and stable signal response for detecting Fe^{3+} . The potentiometric response of the Fe^{3+} sensor with functionalized ZnO nanorods versus a Ag/AgCl reference electrode was observed to be linear over a logarithmic concentration range from 10^{-5}M to 10^{-2}M . The detection limit of the proposed sensor was about $5\mu\text{M}$, which is lower than the normal blood concentration of Fe^{3+} which is about $10\mu\text{M}$ and can be up to $30\mu\text{M}$. The sensitivity of proposed Fe^{3+} sensor was found to be 70.2 ± 2.81 mV/decade with regression coefficient $R^2 = 0.99$ and a response time less than 5 seconds. The functionalized ZnO nanorods with selective iron ionophore has a life time greater than one month and has shown insignificant interference with other ions usually present in the human blood serum. The proposed sensor was used as an indicator electrode for potentiometric titration.

Key words: Immobilization, Ionophore (18 crown 6), Potentiometric chemical sensors, and ZnO nanorods.

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1. INTRODUCTION

In recent past years, one of the most important semiconductors in research is zinc oxide (ZnO). This is due to its many one-dimensional nanostructures such as nanorods, nanotubes, nanowires and nanobelts beside the many interesting properties. These nanostructures have been highly important for nano-phonic applications such as light emitting diodes, optical waveguides and nanolaser [1-7]; and to sensor applications like gas sensors [8, 9] and chemical and biosensors [10, 11]. However, the high surface to volume ratio of the former is the difference between an epilayer and a nanostructure of the same material. This high surface to volume ratio is important for sensor applications [8-11].

In addition, understanding the surface chemical inceptions in ZnO nanostructures are very important for sensor devices, due to the fact that sensor devices depend on the ownerships of surfaces. As an example, the hydroxyl (OH) radical on the tetrahedral geometry of ZnO restricts the surface assimilation of ethanol [12, 13]. The high proportion of OH radical can decrease the surface activity of the ZnO sensor, stops the reaction sites for ethanol molecules and gives less sensing signal.

Iron is a vital element in the human body and is taking effective role in oxygen transport, storage and also in electron transport [14, 15]. The enzymes which are taking part in the synthesis of amino acids, hormones and neurotransmitters need Fe^{3+} . There is around 10-15 mg of iron to be exhibited in the food daily intake, and studies report that the normal subjects assimilate around 10% of the amount of iron from the food [16]. Due to the deficiency of iron, the amount of red blood cells in the body reduces and can become a cause of anaemia. In addition, the surplus amount of iron is stored in the heart, liver and other organs [17, 18] and this extra iron cannot be spontaneously released from the body, but it is stored as mentioned above and can put other organs at risk of impairment [19]. Moreover, excess or less iron compounds in the human body are also cancer causing factors [20]. It is therefore very important for clinical, environmental and industrial purposes to efficiently detect Fe^{3+} .

There are many methods for the detection of iron ions such as atomic absorption spectroscopy (AAS) [21], inductively coupled plasma (ICP) [22], etc. But these methods have many limitations such as high cost and instability if a large number of samples analysis is needed [21, 22]. Moreover, the potentiometric based sensing method is simple, inexpensive, rapid and more reliable for the analysing for ions detection. In the literature, it is also reported that ion selective electrodes (ISEs) was used for the determination of cations

[23-28] as well as anions [29-31] and is also used for pharmaceutical compounds [32-36], with some research also reported on Fe^{3+} detection [37-43]. It is clear that it is necessary to improve the sensitivity of Fe^{3+} sensors and develop selective electrodes having relatively quick response for the determination of the Fe^{3+} concentration; especially when small volumes of the sample is available.

In the present study, a Fe^{3+} sensor based on functionalised ZnO nanorods with 18 crown-6 is developed and the sensor was highly sensitive and can be used for small volumes of analyte solutions.

2. Experiment

2.1. Materials

Chemicals 18-crown-6 (18CE6) [Fluka] was used as the ionophore for iron ion selectivity, while Dioctyl phenylphosphonate (DOPP) [Aldrich] was used as plasticizer. Polyvinylchloride (PVC) [Fluka] was used as the membrane matrix, ferric chloride hexahydrate $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ (sigma Aldrich) and tetrahydrofuran (THF) [Fluka] were used as solvents. All chemicals were of analytical grade.

2.2. The Fabrication and Preparation of the ZnO Nanorods and their Morphological Characterisation

The growth of the ZnO nanorods and fabrication of the substrate is given below. A glass substrate ($70 \times 30 \text{ mm}^2$) was first cleaned with deionized water and it was then dried by air. After that the glass substrate was placed on a flat support inside a vacuum chamber (Evaporator Satis CR725). We evaporated 30 nm thickness of titanium followed by 120 nm of gold. Before growth, the gold coated glass was first washed with isopropanol, followed by deionized water and was again dried in air. ZnO nanorods were grown onto the gold coated glass by using the aqueous chemical growth method [44]. First a uniform layer of a seed solution (zinc acetate dehydrate) was spin coated on the gold coated glass at 3000 rpm, and then the substrate was annealed at 115°C in an oven for 20 minutes. Then the substrates were suspended in a Teflon holder and put into an aqueous solution of 0.075M zinc nitrate hexahydrate [$\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$] and 0.075M hexamethylenetetramine [$\text{C}_6\text{H}_{12}\text{N}_4$]. The beaker was kept into the oven for 5 to 7 hours at 95°C . After that the ZnO grown substrates were taken out from the oven and washed with deionized water, dried by air and the nanostructures were characterised by field emission scanning electron microscopy (FESEM). The FESEM results show that the grown ZnO nanorods were dense with good alignment and controlled

length as shown in figure 1a. The morphological characteristics of the grown nanorods can be controlled by changing the growth parameters like the concentration of the seed solution, growth temperature and the pH of the aqueous solution [45].

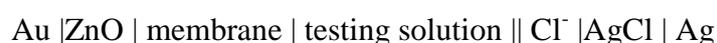
2.3. Functionalization of the ZnO Nanorods with Selective Iron Ionophore

The ionophore membrane was prepared by the following composition, 1% ionophore (18 crown 6), 33% PVC and 66% plasticizer (DOPP) [46]. An amount of 400mg of these components were dissolved into 5 ml of tetrahydrofuran (THF) in 25 ml glass bottle. Then the ZnO nanorods grown on gold coated glass substrate were vertically dipped into the ionophore solution for 5 minutes and left for drying for 1-2 hours at room temperature. SEM image of functionalised ZnO nanorods is shown in figure 1b. All the functionalised ZnO nanorods sensors were kept at 4 °C when not in use. The proposed Fe^{3+} sensors were used as working electrode for the potentiometric measurements in an electrolytic solution of ferric chloride hexahydrate with a concentration range from 10^{-6} M to 10^{-2} M [46]. A Ag/AgCl was acting as a reference electrode. The output voltage of this experiment for each concentration of ferric chloride hexahydrate solution is recorded by using pH meter (model 826 Metrohm). The time response of the developed sensor electrode was measured using Keithley 2400.

3. Result and Discussion

3.1. Response Time of the Functionalised ZnO Nanorods Iron Selective Electrode

The construction of the cell potential of the developed Fe^{3+} sensor presented here can be shown by the diagram below:

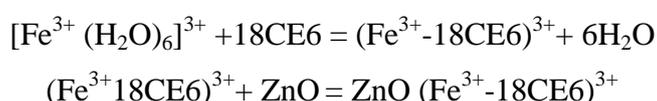


The cell voltage is a function of the concentration of the testing electrolyte solution. This means that the voltage changes because of the change in concentration of iron ion in the testing solution. During the measurement, we tested the selective Fe^{3+} sensor into 10^{-6} M iron electrolytic solution and we observed that the output response of the Fe^{3+} sensor was not stable. Then this Fe^{3+} sensor was tested in 5×10^{-6} M and the proposed Fe^{3+} sensor responded with stable output response. Further we checked the response of the functionalised ZnO Fe^{3+} electrode into 10^{-5} M to 10^{-2} M, and observed that the Fe^{3+} sensor showed very stable output voltage for this concentration range. This is shown in the calibration curve of the logarithm concentration of Fe^{3+} versus the output voltage response. We added 2ml of 10^{-1} M of KNO_3 solution in each testing ferric chloride hexahydrate solution in order to adjust the ionic

concentration inside the solution. In all our measurements the proposed Fe³⁺ sensor obeyed the Nernst's equation. Which states:

$$E = E_0 - 0.05916 \frac{V}{N} \log_{10} \frac{[Reduceed]}{[Oxidized]}$$

Where E is the upper voltage and E_0 is the intercept of the curve with the y-axis of the calibration curve. The response of the iron ion sensor was about 63 mV for 10⁻⁴M solution of **ferric chloride hexahydrate**. We investigated the output voltage and found that the response did not change with the change of the volume of the testing solution. We repeated the measurement in volume ranging from 5 ml to 20 ml with the same selective electrode into the 10⁻⁴M testing solution; the constant output voltage was around 63 to 64 mV for each volume of the testing solution. The sensing mechanism of the electrochemical iron ion sensor followed the equation below:



The proposed iron ion sensor has shown good linearity for a wide concentration range from 10⁻⁵M to 10⁻²M of iron ions. We found that the sensitivity of the sensor is about 70.2± 2.81 mV/decade with a regression coefficient R² = 0.99. The response time of the electrode was less than 5 seconds as shown in figure 2a and 2b, respectively. These two parameters, the response time and the sensitivity fully describe the high efficiency of this proposed iron ion selective electrode.

3.2. Reproducibility, Linearity and Stability of the Sensor

We tested the linearity, stability and reproducibility of proposed sensor by using 4 iron ion selective sensor electrodes prepared separately using the same procedure, the relative standard deviation of the functionalized ZnO nanorods iron sensor in the known concentration of **ferric chloride hexahydrate** solution was varying with less than 3%. This indicated a good reproducibility. The reproducibility from one iron sensor to another iron sensor in a 10⁻⁴M **ferric chloride hexahydrate** solution is shown in figure 3a. We also checked (three times) the reproducibility of the same iron sensor in **ferric chloride hexahydrate** solution and the observed results with long stability and excellent linearity as shown in figure 3b. The proposed sensor was used from time to time and kept at 4 °C for more than four weeks. The sensor maintained its actual working activity up to 90% and almost gave the same response towards the iron ion. We investigated the morphology of the used ZnO membrane

iron sensor using SEM and the result is given in figure 1c. From the SEM images it was observed that the ZnO nanorods were slightly affected during the measurements. This is due to the small change in pH of the testing solution. The solubility of ZnO nanorods can vitiate the performances of the working electrode.

3.3. The Effect of Temperature and Interference on the Response of the Functionalized ZnO Nanorods Selective Iron Ion Sensor

The temperature effect on the functionalized ZnO nanorods selective iron ion sensor was investigated between temperatures of 21 °C to 80 °C using 10⁻⁴M of ferric chloride hexahydrate solution as shown in figure 4a. We observed that the output voltage was increasing gradually from 21 °C to 50 °C. Above 50 °C the output tends to decrease and at 80 °C there was a drastic reduction in voltage due to the decrease in the strength of bonds between the ionophore and the ZnO nanorods and as the temperature increases, the ionic mobility of the iron ions increases which in result make more resistance to the movement of the iron ions towards the respective functionalized ZnO nanorods selective electrode. It was reported that using the present ionophore for constructing Fe³⁺ sensor would give a maximum response in a pH between 1 to 3 [46], but above this pH the signal become unstable due to the possible interference of hydrogen ions [H⁺]. The interference is one of the most important measuring parameter for ion selective electrode systems. For these measurements we prepared a testing solution with concentration ranging from 10⁻⁶ to 10⁻² M of each interfering substance, and then performed the potentiometric measurements. The measured output voltage of Fe³⁺ sensor in presence of the interfering ions with difference concentrations was plotted against the logarithm of concentration using the mixed solution method as shown figure 4b and as well as we calculated the selectivity coefficient ($K_{Fe^{3+},ion}^{pot}$) for each interfering ion using the separation method [47] as given in table 1. The possible interfering substances that we chose for our experiments were Na⁺, Zn²⁺, Cu²⁺, K⁺, Li⁺, Mg²⁺ and Ca²⁺. Figure 4b and Table 1 show that no significant interference was observed.

3.4. Potentiometric Titration

The analytical application of Fe³⁺ sensor based on the functional of ZnO nanorods was tested by the potentiometric titration of Fe³⁺ with EDTA solution. A 20 ml (10⁻⁴M) of testing solution was titrated with 10⁻⁴M EDTA solution. With the addition of EDTA solution into the testing solution, the Fe³⁺ concentration and the output voltage were decreased because of the formation of Fe-EDTA complex as shown in figure 5. In figure 5, which

exhibits that the end point represent the stoichiometric formulation of Fe-EDTA complex and it also suggests that the almost all of the iron ions are used up in the formation of Fe-EDTA complex and so after **the equivalent point**, the potential response was found to almost be constant. It is therefore suggested that the proposed sensor based on functionalized ZnO nanorods can successfully be used as an indicator electrode for the determination of Fe³⁺ by potentiometric titration.

Table 1: The logarithm of selective coefficients for Fe³⁺ sensor with different interfering ions in 10⁻⁴ M

Interference ions	<i>Log K_{Fe³⁺, ion^{pot}}</i>
K¹⁺	0.142
Mg²⁺	0.114
Li¹⁺	0.027
Zn²⁺	0.222
Na¹⁺	0.563
Ca²⁺	0.135
Cu²⁺	0.560

Table 2: Comparison of the result of the proposed Fe³⁺ sensor based on ZnO nanorods with previous published work

No.	Slope (mV/decade)	Times respond (s)	Detection Limit (M)	Linear range (M)	Life times	Reference
1	19.4 ± 0.5	≈ 15	6.8 × 10 ⁻⁷	1.0 × 10 ⁻⁶ - 1.0 × 10 ⁻¹	9 weeks	[36]
2	28.5	≈ 15	-	3.5 × 10 ⁻⁶ - 4.0 × 10 ⁻²	2 months	[37]
3	20.0	15	5.0 × 10 ⁻⁶	6.3 × 10 ⁻⁶ - 1.0 × 10 ⁻¹	2 months	[38]
4	60.0	25 - 30	-	1.0 × 10 ⁻⁶ - 1.0 × 10 ⁻²	3 months	[39]
5	30.5 – 32	8 - 15	1.0 × 10 ⁻⁶ - 4 × 10 ⁻⁸	5.0 × 10 ⁻⁷ - 1.0 × 10 ⁻²	≈ 3 months	[40]
6	19.4 ± 0.5	-	3.6 × 10 ⁻⁷	1.0 × 10 ⁻⁶ - 1.0 × 10 ⁻²	2 months	[41]
7	57.0	30	1.0 × 10 ⁻⁶	1.0 × 10 ⁻⁴ - 1.0 × 10 ⁻¹	2 months	[42]
8	20	20	1.3 × 10 ⁻⁶	1.0 × 10 ⁻⁵ - 1.0 × 10 ⁻¹	2 months	[45]
9	70.2 ± 2.81	≈ 5	5.0 × 10 ⁻⁶	1.0 × 10 ⁻⁵ – 1.0 × 10 ⁻²	4 weeks	this work

4. Conclusions

In this work, we developed a simple, highly sensitive and selective iron ion chemical sensor based on ZnO nanorods coated by a selective ionophore (18 crown 6). The response of the sensor increases with increasing the concentrations of the iron ion from 10^{-5}M to 10^{-2}M with a slope 70.2 ± 2.81 mV/decade. The potential of the presented proposed iron ion sensor is due to its large slope, with a regression coefficient $R^2 = 0.99$ and the relatively small response time obtained (less than 5 seconds). These characteristics of the presented sensor reflect its high sensitivity. Since this sensor is inexpensive, highly selective, and easy to handle for new users and it can be applied for monitoring of iron concentration in human blood serum and in detection of iron from lubrication oils as well as for the environmental analysis it can be of potential in for applications. This iron sensor based on functionalized ZnO nanorods can also be applied as an indicator electrode for the potentiometric titration.

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Figure Captions

Figure 1(a-c). A typical SEM image of ZnO nanorods grown on gold coated glass substrate using the aqueous chemical growth method. The figure exhibit: (a) the ZnO nanorods without membrane, (b) the ZnO nanorods immobilized with ionophore before the use, and in (c) the functionalized ZnO nanorods after use for sensing.

Figure 2(a-b). (a) The calibration curve for iron sensor, and (b) time response of the iron sensor base on ZnO nanorods in 10^{-4} M ferric chloride hexahydrate solution.

Figure 3(a-b). (a) Sensor to sensor reproducibility of ($n = 4$), for electrodes in 10^{-4} M ferric chloride hexahydrate solution, and in (b) the response of three different experiments using the same sensor together a Ag/AgCl reference electrode.

Figure 4(a-b). (a)The output voltage versus with temperature, and (b) response of electrode against Fe^{3+} and other interference cations.

Figure 5. The Potentiometric titration curve of 10^{-4} M Fe^{3+} (20ml) solution versus with EDTA (10^{-4} M).

Figure 1.

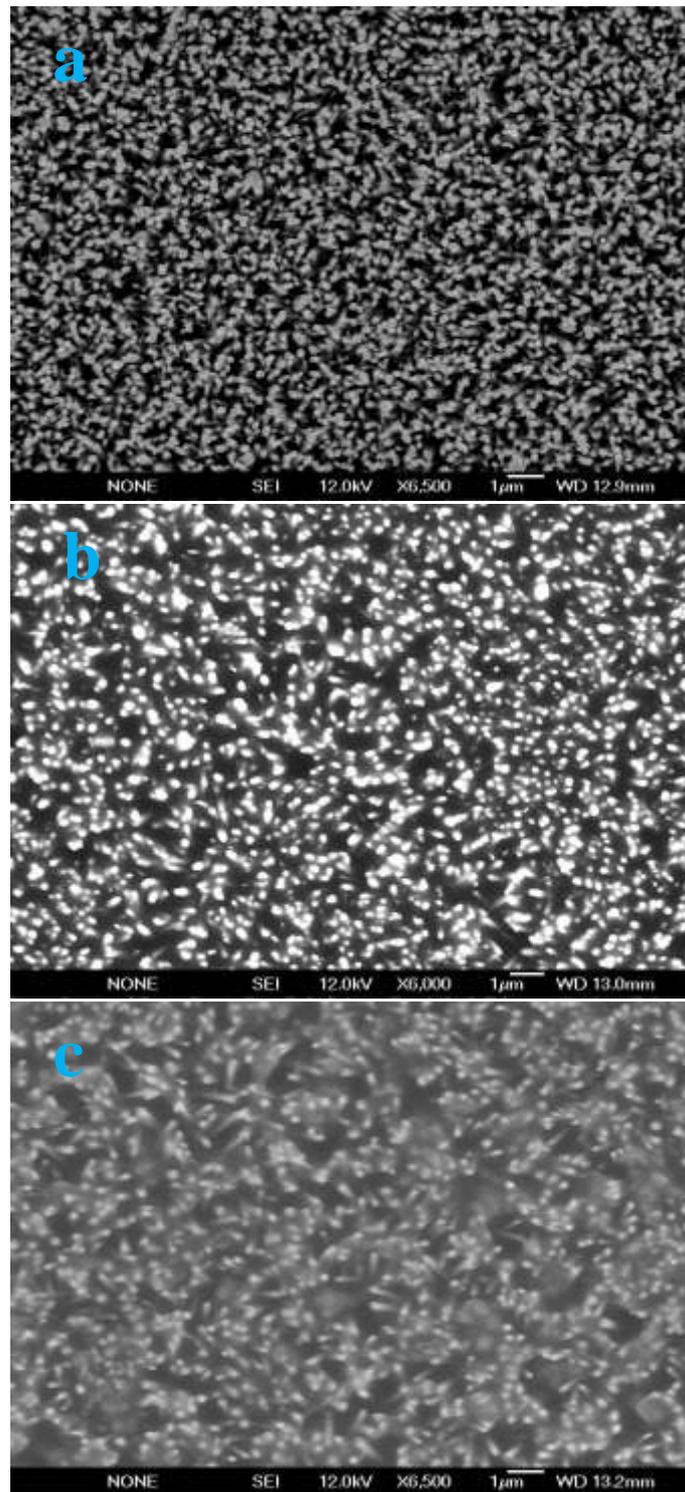
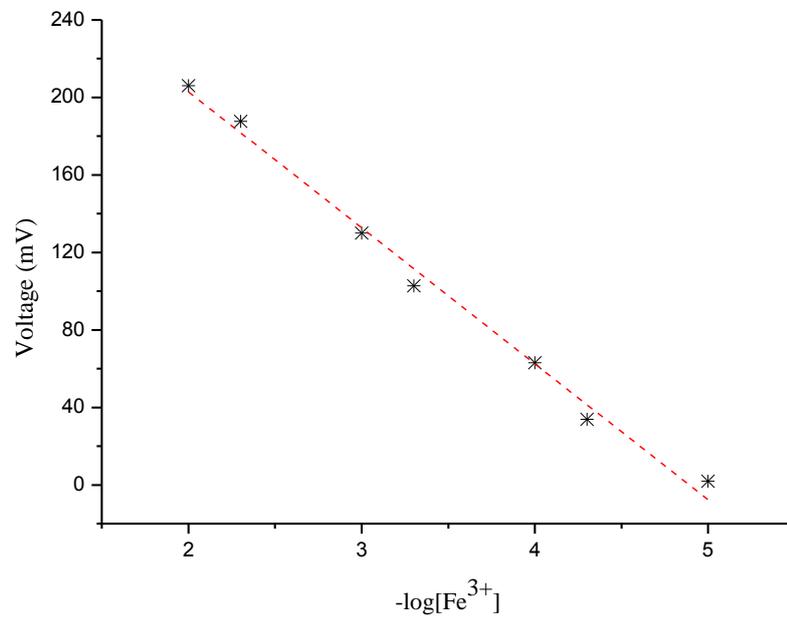


Figure 2.

a)



b)

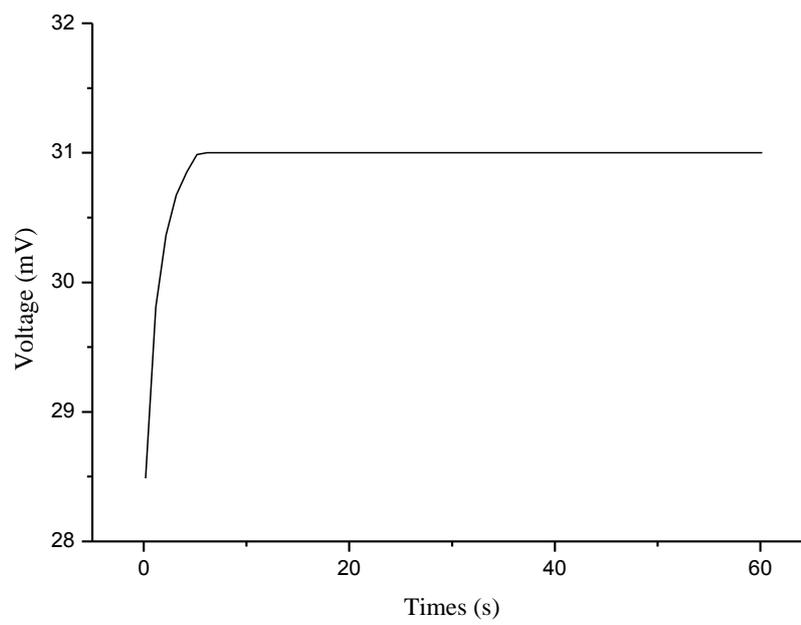
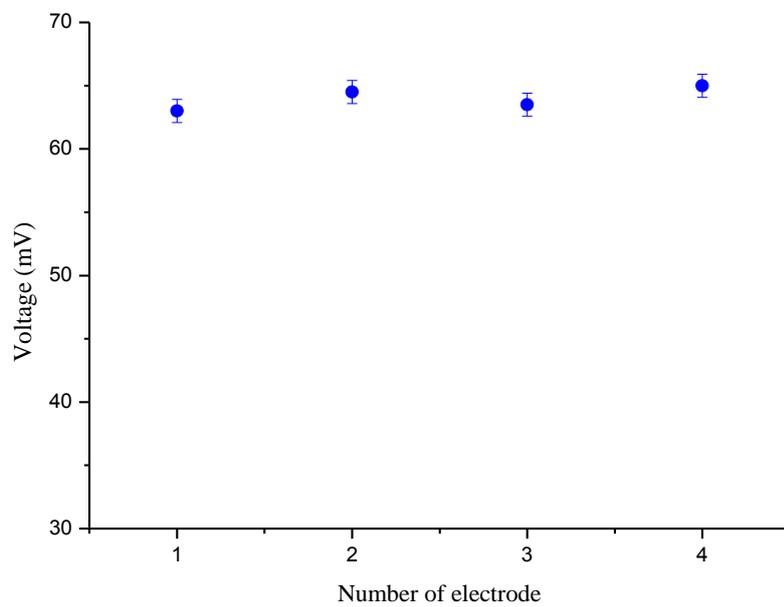


Figure 3.

a)



b)

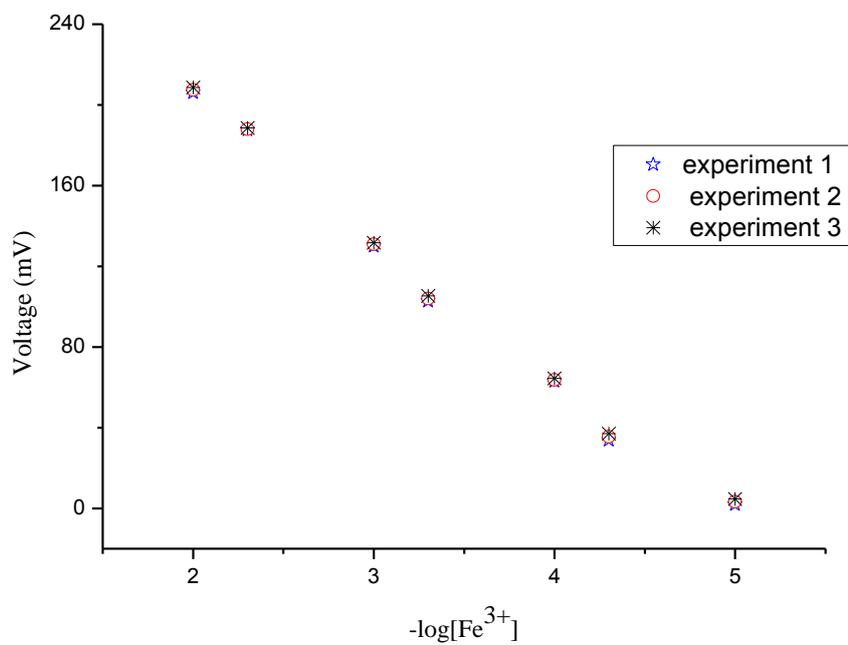
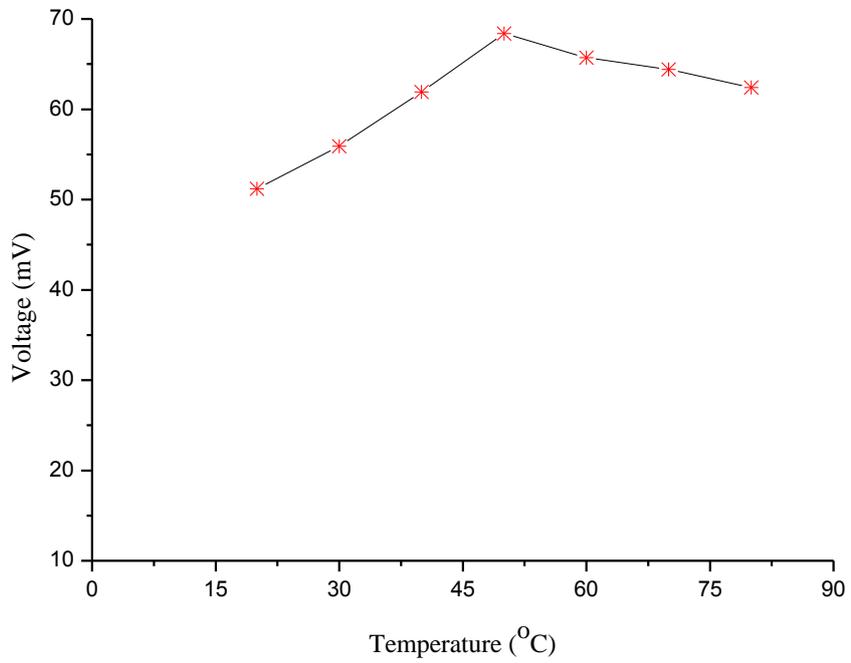


Figure 4.

a)



b)

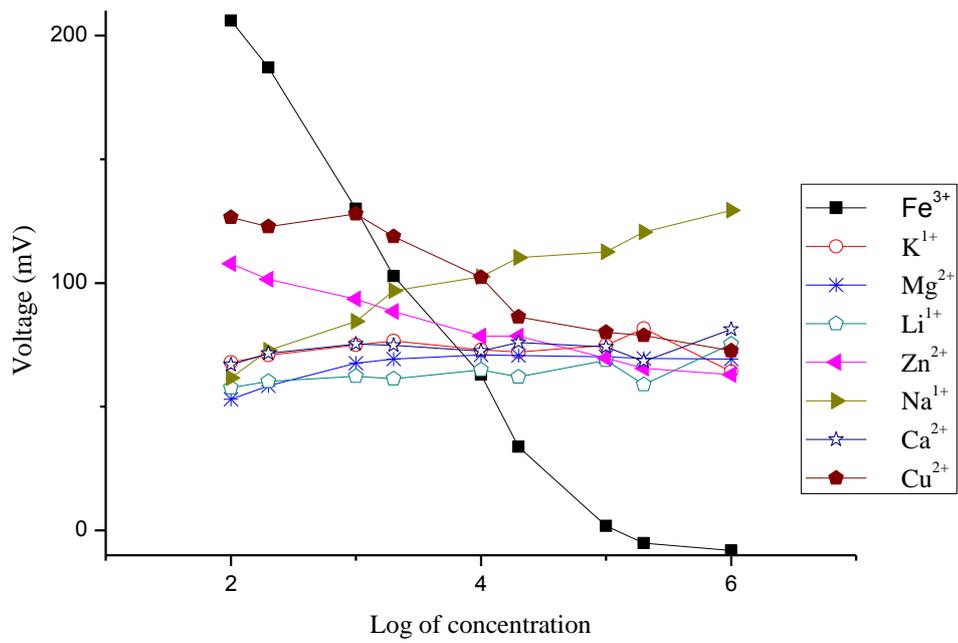


Figure 5.

