A Novel Ion-Selective Electrode of Polythiophene Based on Surfactant-Assisted Dilute Polymerization Method

Tabassum Akhtar* and Masood Alam

Environmental Science Laboratory, Department of Applied Sciences and Humanities, Faculty of Engineering and Technology, Jamia Millia Islamia, New Delhi 110025, India

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Polythiophene (PTh) nanoparticles were synthesized by cationic surfactant assisted dilute polymerization method using FeCl₃ as oxidant in an anhydrous medium. The physical characterizations of the synthesized PTh nanoparticles were studied by FT-IR and UV-Visible spectroscopy, XRD, SEM and EDX. The synthesized PTh nanoparticles were used for the first time to fabricate ion-selective membrane electrode. The experimental results herein concern a novel ion-selective electrode manufactured with an inner solid contact based on PTh nanoparticles. The electrode exhibited a good Nernstian response of 30.2 mV/decade of Mn²⁺ concentration over an ample concentration range (1.0 × 10⁻⁶ to 1.0 × 10⁻¹ mol L⁻¹), with a detection limit of 1.0 × 10⁻⁶ mol L⁻¹. The membrane electrode showed a very fast response time of 45 s and could be operated well in the pH range 3–7. The selectivity coefficients were determined by the mixed-solution method which revealed that the electrode was selective in the presence of interfering cations. The material was used as electroactive component for the construction of an ion-selective membrane electrode. The membrane electrode was mechanically stable, having wide dynamic range, with quick response time and could be operated for at least 5 months without any considerable divergence in the potential response characteristics. The electrode was used satisfactorily act as indicator electrode during the potentiometric titration of Mn²⁺ ions with EDTA.

**Keywords:** Polymer, Polythiophene Particles, Ion-Selective Membrane Electrode, Manganese Ion-Selective Electrode.

1. INTRODUCTION

After 30 years of development, the world of conducting polymers is established as an important branch of material science with many opportunities for applications in electronics and photonics. The term “conducting polymer” covers several types of polymeric materials with electronic and/or ionic conductivity, including (i) doped conjugated polymers, (ii) redox polymers, (iii) polymer composites, and (iv) polymer electrolytes. Conducting polymers are promising materials for a large variety of chemical sensors, as can be seen from several reviews.¹⁻¹¹ However, the present review deals only with conducting polymer-based potentiometric ion sensors (ion-selective electrodes, ISEs).

Ion selective electrode is an important tool allowing the sensitive and selective determination of various ions in the wide range of concentration. Precipitate based ion selective membrane electrodes are well known as they are successfully employed for the determination of several anions and cations. The heterogeneous precipitate ion exchange membranes consist of suitable colloid ion-exchanger particles as electro-active materials embedded in polymer (binder) like poly(vinyl chloride), epoxy resin (Araldite), poly styrene, polyethylene, nylon etc., by physically mixing or chemical reaction and have been extensively studied as potentiometer sensors.¹³⁻¹⁹ The potentiometric sensors offer several advantages such as: low cost, ease of fabrication, low detection limits, wide response range, reasonable selectivity, among others. Ion exchange membrane also finds application in diverse processes such as electrodialysis, diffusion dialysis, electrode-ionization, membrane electrolysis, fuel cells, storage batteries, electro-chemical synthesis etc., which are energy.
sources and environmentally saving. Ion selective electrode is an important tool allowing the sensitive and selective determination of various ions in the wide range of concentrations. However, conducting polymers potentially fall victim to their poor mechanical stability and narrow operating electrochemical windows. These demerits of conducting polymers restrain their practical applications in SCs. A general strategy to overcome the above drawbacks is to introduce conducting polymers into the matrix of other active electrode materials.20 The corresponding composites such as manganese oxide/conducting polymer and carbon nanotube/conducting polymer are expected to convey enhanced electrochemical properties if each component can retain its merits and both individuals can compensate their shortcomings synergistically. Moreover, some previous studies have just focused on controllable formation of nanostructured oxides and conducting agents separately, rather than combining both together.

This research work presents the potentiometric studies for Mn(II) detection on a new ion-selective electrode of polythiophene nanoparticles, prepared by cationic surfactant assisted dilute polymerization method. The synthesized PTh nanoparticles were characterized by XRD, SEM, FTIR, UV-Visible spectroscopy and EDX. In view of the aforementioned, in this work a novel ISE electrode for the selective and quantitative determination of Mn(II) is presented.

2. EXPERIMENTAL DETAILS

2.1. Reagents and Instruments

The main reagents or chemicals used for the synthesis of the material were obtained from CDH, Loba Chemie, E-merck and Qualigens (India Ltd., used as received). All other reagents and chemicals were of analytical grade.

A digital pH-meter (Elico Li-10, India), a digital potentiometer (Equiptronics EQ 609, India; accuracy ± 0.1 mV with a saturated calomel electrode as reference electrode, a digital electronic balance (Sartorius 21OS, Japan) and an agate mortar pestle were used.

The stock solution of $1 \times 10^{-1}$ M Mn(II) was prepared in double distilled water (DMW). The solution was standardized by complexometric titration.22 The working standard solutions ($1 \times 10^{-1}$ M to $1 \times 10^{-10}$ M) were prepared by proper dilution of the stock solution.

2.2. Synthesis of Polythiophene with Surfactant

About 1 ml amount of thiophene was mixed with 50 ml of chloroform in a reaction flask 0.0034 mol of surfactant (CTAB) dissolved in 30 ml of CHCl₃ was added to the monomer solution and stirred on a magnetic stirrer for 15 min. The molar ratio of monomer to surfactant was 7:1. Finally, 0.055 mol of solid anhydrous FeCl₃ was added to the above monomer solution. It is important that FeCl₃ is added in the solid state and not as a solution.23 The molar ratio of FeCl₃ to thiophene was 2:3 and the polymerization was carried out for 24 h at room temperature. The dark-brown precipitate of Polythiophene (PTh) was collected by filtration and washed with CHCl₃ three times. The PTh was further washed with methanol to remove the residual oxidant. During this procedure, the colour of PTh changed from dark-brown to brown. The prepared PTh powder was dried in a vacuum oven at 80 °C for 6 h.

2.3. X-Ray Analysis

Powder X-ray diffraction (XRD) pattern was obtained in an aluminum sample holder for the Polythiophene with surfactant in the original form using a PW, 1148/89 based diffractometer with Cu Ka radiations.

2.4. Scanning Electron Microscopy (SEM) Studies

Scanning Electron Microscopy (SEM) microphotographs of the original form of Polythiophene with surfactant were obtained at various magnifications.

2.5. Fourier Transform Infra Red (FTIR) Studies

The FTIR spectrum of Polythiophene with surfactant in the original form dried at 40 °C were taken by KBr disc method at room temperature.

3. PREPARATION OF ION-EXCHANGE MEMBRANE

The ion-exchange membrane of Polythiophene was prepared by following the procedure of Coetzee and Benson.24 Polythiophene (PTh) powder (100 mg) as electroactive

![Fig. 1. Flow chart of the synthesis of polythiophene nanoparticles.](image-url)
material was ground to fine powder, and was mixed thoroughly with Araldite\textsuperscript{25} (Ciba-Geigy, India Ltd.) (100 mg) in 1:1 (w/w) ratio to make a homogeneous paste which was then spread between the folds of Whatman’s filter paper No. 42. Glass plates were kept below and above the filter paper folds as support. The phase of the exchanger and Araldite was kept under pressure of 2 kg cm\textsuperscript{2} for 24 h and left to dry. Two sheets of different thickness of 0.41 and 0.43 mm of master membranes were prepared. These sheets were dipped in distilled water to remove filter paper. After drying, the membrane sheets were cut in the shapes of discs using a sharp edge blade. The membrane thickness of 0.41 mm exhibited good surface qualities, like porosity, thickness, swelling etc. were selected for further investigations.

3.1. Characterization of Membrane

Physico-chemical characterization is important to understand the performance of membrane. Thus some parameters such as porosity, water content, swelling, thickness, etc. were determined as described elsewhere\textsuperscript{26–28}.

3.1.1. Water Content (%Total Wet Weight)

First, the membranes were soaked into water to elute diffusible salt, blotted quickly with Whatman filter paper to remove surface moisture and immediately weighed. These were further dried to a constant weight in a vacuum over P\textsubscript{2}O\textsubscript{5} for 24 hr. The water content (total wet weight) was calculated as:

\[
\% \text{ Total wet weight} = \frac{W_w - W_d}{W_w} \times 100
\]

Where \(W_w\) = weight of the soaked/wet membrane and \(W_d\) = weight of the dry membrane.

3.1.2. Porosity

The thickness of the membrane was measured by taking the average thickness of the membrane by using screw gauze. Swelling is measured as the difference between the average thicknesses of the membrane equilibrated with 1 M NaCl for 24 hrs and the dry membrane.

\[
\text{Porosity} = \frac{W_w - W_d}{W_w \rho_o L}
\]

Where \(W_w\) and \(W_d\) are weight of wet and soaked (in NaCl) membrane while \(\rho_o\) and \(L\) are the density of water and thickness of the membrane.

3.1.3. Fabrication of Ion-Selective Membrane Electrode

The membrane sheet of 0.41 mm thickness as obtained by the above procedure was cut in the shape of disc and mounted at the lower end of a Pyrex glass tube (o.d. 0.8 cm, i.d. 0.6) with araldite. Finally the assembly was allowed to dry in air for 24 hr. The glass tube was filled with solution of the ion (as reference) towards which the membrane is selective and it was kept dipped in an identical solution of the same ion at room temperature. In case of polythiophene ion-selective membrane electrode, the glass tube was filled with 0.1 M Manganese nitrate solution. A saturated calomel electrode was inserted in the tube for electrical contact and another saturated calomel electrode was used as external reference electrode. The whole arrangement can be shown as:

<table>
<thead>
<tr>
<th>Internal reference electrode (SCE)</th>
<th>Internal electrolyte</th>
<th>Membrane Sample solution</th>
<th>External reference electrode (SCE)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.1 M Mn(^{2+})</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Following parameters were evaluated to study the characteristics of the electrode such as lower detection limit, electrode response curve, response time and working pH range.

3.1.4. Electrode Response or Membrane Potential

To determine the electrode response, a series of standard solutions of varying concentration which were to be studied were prepared. External electrode and ion selective membrane electrode are plugged in digital potentiometer and the potentials were recorded.

For the determination of electrode potentials the membrane of the electrode was conditioned by soaking in 0.1 M Mn(NO\textsubscript{3})\textsubscript{2} solution for 5–7 days and for 1 h before use. When electrode was not in use electrode was in 0.1 M selective ion solution. Potential measurement was plotted against selected concentration of the respective ion in aqueous solution as given in Figure 6.

3.1.5. Effect of pH

pH solutions ranging from 1–13 were prepared at constant ion concentration i.e., (1 \times 10^{-2} M). The value of electrode potential at each pH was recorded and plotted against pH as shown in Figure 7.

3.1.6. Response Time

The method for determining response time in the present work is being outlined as follows: The electrode is first dipped in a 1 \times 10^{-3} M solution of the ion concerned and immediately shifted to another solution of 1 \times 10^{-2} M ion concentration of the same ion (10 fold higher concentration). The potential of the solution was read at zero second i.e., just after dipping the electrode in the second solution and subsequently recorded at the intervals of 5 s. The potentials were then plotted versus time as shown Figure 8.

3.1.7. Selectivity

The most important characteristic of an ISE is its selective response capacity to a primary ion in the presence of other ions. This parameter is directly related to the
thermodynamic equilibrium at the surface/solution interphase. The selectivity of the electrode containing the ligand and PTh/MnO₂ was tested by potentiometry with different divalent cations like: Ca(II), Ni(II), Cd(II), Mg(II), Cu(II), Co(II), Ba(II) and Hg(II). The slope assessment for the majority of cations tested showed values much lower than expected from Nernstian behavior. However, for Mn(II), a linear potentiometric response was observed with a value closer to such behavior, within a wide concentration range and a low detection limit.

The potentiometric selectivity coefficient (as measured in terms of the potentiometric selectivity constant \( K_{pot} \)) that describes the preference of the electrode suggested to an interfering ion in the presence of Mn(II), was determined by means of the fixed interference method, which is based on the Nikolsky-Eisenman semiempirc equation\(^{29,30} \), as follows:

\[
E_{ISE} = E^o + RT/Z_i f \ln (a_i + K_{pot}^{ij} a_j^{Z_i/Z_j}) \quad (1)
\]

where \( E_{ISE} \) is the measured potential, \( E^o \) is the standard potential, \( a_i \) and \( a_j \) are the activities of the primary and interfering ions, respectively, \( z_i \) is the charge of primary ion, \( z_j \) is the charge of the interfering ion, \( F \) is the Faraday constant, \( R \) is the universal gas constant and \( T \) is the absolute temperature. In this method, the primary ion concentration is varied at 1.0 mol L\(^{-1} \) solution where as the secondary ion concentration is kept constant at 1.0 × 10\(^{-3} \) mol L\(^{-1} \). The equations for each ion can be written as

\[
E_i = E^o + RT/Z_i f \ln (a_i) \quad (2)
\]
\[
E_j = E^o + RT/Z_j f \ln (K_{pot}^{ij} a_j) \quad (3)
\]

Equalizing the potentials one gets

\[
\log K_{pot}^{ij} = \frac{a_j}{a_i^{Z_j/Z_i}} \quad (4)
\]

Using the fixed interference method (FIM), the selectivity coefficients were calculated and are shown in Table III.

3.1.8. Storage of Electrodes
The polythiophene electrode was stored in distilled water when not in use for more than one day. It was activated with (0.1 M) Mn(II) solution by keeping immersed in it for 2 h, before use, to compensate for any loss of metal ions in the membrane phase that might have taken place due to a long storage in distilled water. Electrode was then washed thoroughly with DMW before use.

3.1.9. Analytical Application of the Electrode
The analytical utility of this membrane electrode has been established by employing it as an indicator electrode in the potentiometric titration of a 0.01 M Mn(NO₃)₂ solution against 0.005 M EDTA solution. Potential values are plotted against the volume of EDTA.

4. RESULTS AND DISCUSSION
X-ray diffraction study showed the crystalline nature of PTh nanoparticles (Fig. 2). There was a partially crystalline broad peak centered at near 2θ value of 24.2°. The strong diffraction peak associated with the chain-to-chain stacking distance of about 2θ = 24.2° is due to the amorphously packed polythiophene main chain.\(^{31,32} \)
The SEM image of polythiophene nanopowder is shown as Figure 3. The image appears uniformly microporous on the surface and the particles were in nanometer scale.

The EDS spectra of pure polythiophene is shown in Figure 4. It can be seen that there are only two elements C and S and traces of Fe were also found in Polythiophene. It further proves that the polythiophene is synthesized successfully, as FeCl$_3$ is used as an oxidant during polymerization. Table I gives the elemental compositions of Polythiophene.

The FTIR spectrum of polythiophene is shown as Figure 5. In the spectrum of Polythiophene, there are several low-intensity peaks in the range of 2,000–3,000 cm$^{-1}$ which can be attributed to the aromatic C–H stretching vibrations and C=C characteristic band. The absorption in this region is obscured by the bipolaron absorption of the doped PTh. The range of 5,000–1,500 cm$^{-1}$ is the fingerprint region of PTh. The peak at 789 cm$^{-1}$ is usually ascribed to the C–H out-of-plane deformation mode, whereas other peaks in this region are attributed to the ring stretching and C–H in-plane deformation modes. The C–S bending mode has been identified at approximately 690 cm$^{-1}$ which indicates the presence of a thiophene monomer. These results support to the polymerization.

**4.1. Characterization of Composite Membrane**

Sensitivity and selectivity of the ion-selective electrodes depend upon the nature of electro-active material, membrane composition and physico-chemical properties of the membranes employed. A number of samples of the polythiophene membrane were prepared with different amount of composite and Araldite and checked for the mechanical stability, surface uniformity, materials distribution, cracks and thickness, etc. The membranes obtained with 0.41 mm were found to be good.

<table>
<thead>
<tr>
<th>Elements</th>
<th>Wt%</th>
<th>At%</th>
</tr>
</thead>
<tbody>
<tr>
<td>CK</td>
<td>62.03</td>
<td>81.09</td>
</tr>
<tr>
<td>SK</td>
<td>36.19</td>
<td>17.87</td>
</tr>
<tr>
<td>FeK</td>
<td>1.78</td>
<td>1.04</td>
</tr>
</tbody>
</table>

**Table II.** Characterization of ion-exchange membrane.

<table>
<thead>
<tr>
<th>Polythiophene composite</th>
<th>Thickness of the membrane (mm)</th>
<th>Water content as % weight of wet membrane</th>
<th>Swelling of % weight of wet membrane</th>
</tr>
</thead>
<tbody>
<tr>
<td>M-1</td>
<td>0.41</td>
<td>2.012</td>
<td>0.0016</td>
</tr>
<tr>
<td>M-2</td>
<td>0.43</td>
<td>2.233</td>
<td>0.0018</td>
</tr>
</tbody>
</table>

The results of thickness, swelling, porosity and water content capacity of polythiophene membrane are summarized in Table II. The membrane sample M-1 of thickness 0.41 mm was selected for further studies. The low order of water contents, swelling and porosity with less thickness of the membrane suggest that interstices are negligible and diffusion across the membrane occur mainly through the exchanger sites.

**4.2. Potentiometric Studies of Heterogeneous Polythiophene Membrane Electrode**

Polythiophene membrane (M-1) was fabricated into ion-selective electrode. The membrane electrode was placed in 0.1 M Mn(NO$_3$)$_2$ solution for 7 days to get it conditioned. After conditioning the electrode, the potentials for a series of standard solution of the Mn(NO$_3$)$_2$ in the range 10$^{-10}$ M to 10$^{-1}$ M were measured at a fixed concentration of Mn$^{2+}$ ion as internal reference solution.

Potentials of the membrane electrode were plotted against the selected concentration of Mn$^{2+}$ ions (Fig. 6). It gives linear response in the range 1 × 10$^{-1}$ M and 1 × 10$^{-6}$ M. Suitable concentrations were chosen for sloping portion of the linear curve. The limit of detection, is determined from the intersection of the two extrapolated segments of the calibration graph which was found to be 1 × 10$^{-6}$ M. It shows a good Nernstian response of 30.2 mV/decade of Mn$^{2+}$ concentration and the wide linear concentration range from 1.0 × 10$^{-6}$–1.0 × 10$^{-1}$ M. Below 1 × 10$^{-6}$ M non linear response was observed which could
be used for analytical applications. Experiments were conducted a number of times to check the reproducibility of the results. EMFs were plotted against log of activities of manganese ions and calibration curves were drawn for three sets of experiments and a standard deviation of ±0.2 mV was observed.

The influence of the pH on the response of Mn²⁺-ISE based on ionophore was studied. The pH effect is illustrated in Figure 7 by varying the pH of test solutions with 0.1 M HNO₃ and 0.1 M NaOH for 1.0 × 10⁻² M and 1.0 × 10⁻³ M Mn(NO₃)₂, respectively. The results were recorded only for 1 × 10⁻² M ion solutions. It is clear from Figure 7 that the operational pH range was 3.0–7.0 for the electrode based on ionophore. At higher pH, Mn²⁺ will form precipitate with OH⁻, thus causing the potential reduction. When the pH was low, the concentration of H⁺, shows obvious disturbance on the potentiometric response of Mn²⁺-ISE.

Another important factor is the promptness of the response of the ion-selective electrode. The average response time is defined as the time required for the electrode to reach a stable potential after successive immersion of the electrode in different Mn²⁺ ion solutions, each having a 10-fold difference in concentration from 10⁻¹⁰ M to 10⁻¹ M was investigated. In this work, the practical response time was recorded by immediate and successive changing of Mn²⁺-ISE electrode showed reasonably fast and stable potential within 45 s and no change was normally observed up to 5 min after which it started deviating.

It is very important that the performance of any ion-selective electrode should be checked soon every time before using it for any analytical purpose. For the present polythiophene membrane electrode, it was observed that the measured potential of Mn²⁺ ions in a given concentration range of 10⁻¹–10⁻¹⁰ M was reproducible within ±2 mV, and there was no significant change in the slope of the Nernst plot during the experiment over a time period of 5 months. This suggests a longer electrode life and a stable electrode performance.

The selectivity coefficients, K⁺Mn,M of various differing cations for Mn(II) ion selective polythiophene membrane electrode were determined using the mixed solution method. The selectivity coefficient indicates the extent to which a foreign ion (M⁺⁺) interferes with the response of the electrode towards its primary ions (Mn²⁺). By examining the selectivity coefficient data given in Table III it is clear that that the electrode is selective for Mn(II) in the presence of interfering cations.
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Table III. The selectivity coefficients ($K_{Mn}^{+}$) of various interfering cations ($M^{n+}$).

<table>
<thead>
<tr>
<th>Cation</th>
<th>Selectivity Coefficient ($K_{Mn}^{+}$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Na$^{+}$</td>
<td>0.002</td>
</tr>
<tr>
<td>Mg$^{2+}$</td>
<td>0.002</td>
</tr>
<tr>
<td>Sr$^{2+}$</td>
<td>0.005</td>
</tr>
<tr>
<td>Al$^{3+}$</td>
<td>0.008</td>
</tr>
<tr>
<td>Zn$^{2+}$</td>
<td>0.004</td>
</tr>
<tr>
<td>Mn$^{2+}$</td>
<td>0.007</td>
</tr>
<tr>
<td>Pt$^{2+}$</td>
<td>0.006</td>
</tr>
<tr>
<td>Cu$^{2+}$</td>
<td>0.003</td>
</tr>
<tr>
<td>Fe$^{3+}$</td>
<td>0.005</td>
</tr>
<tr>
<td>Ca$^{2+}$</td>
<td>0.003</td>
</tr>
<tr>
<td>K$^{+}$</td>
<td>0.004</td>
</tr>
</tbody>
</table>

5. ANALYTICAL APPLICATION

5.1. Potentiometer Titration

The study suggests that this newly characterized Mn$^{2+}$-ISE could work well under the laboratory conditions. It had been effectively applied as indicator electrode to the potentiometric titration of a spiked $1.0 \times 10^{-2}$ M Mn(NO$_3$)$_2$ solution with 0.01 M EDTA as titration reagent. The addition of EDTA causes a decrease in potential as a result of the decrease in the free Mn(II) ion concentration due to its complexation with EDTA (Fig. 9). The amount of Mn(II) ions in solution can be accurately determined from the resulting neat titration curve providing a sharp rise in the titration curve at the equivalence points. The results demonstrate that the Mn$^{2+}$ amount in this solution can be determined with good accuracy.

Mn$^{2+}$-ion selective membrane electrode was also applied to direct measurements of Mn$^{2+}$ in the drain water collected from Department of Applied Sciences and Humanities, Jamia Millia Islamia, New Delhi. The samples were collected from three different locations of drains and preserved with HNO$_3$, stored in glass bottles and analyzed within 12 h after collection. Since the samples contained particulate matters, they were centrifuged, and the potentials were measured. Three replicate measurements were made to obtain the Mn(II) contents in three samples with this electrode using the membrane sensor’s calibration graph. The concentration of manganese in the drain water samples were in the range of $1.0 \times 10^{-7}$ M to $6.5 \times 10^{-8}$ M, and the reproducibility of the results was checked up to three times.

6. CONCLUSION

Polymer Polythiophene based Araldite membrane electrode was found to be selective and sensitive for Mn(II) ions. The membrane electrode possessed a quick response time of only 45 s. The functional pH range of the electrode was 3–7. A Mn$^{2+}$-ISE based on a newly synthesized ionophore was prepared. The results show that this sensor showed a wide linear range of $1.0 \times 10^{-5}$–$1.0 \times 10^{-1}$ M with a detection limit of $1.0 \times 10^{-5}$ M Mn$^{2+}$. The selectivity of the ion-selective electrode showed good results over a wide range of interfering metal ions. These characteristics and the typical applications presented in this paper make the sensor a suitable for measuring Mn(II) content in real samples without a significant interaction from cationic or anionic species. It could be successfully applied in the potentiometric titration of Mn(II) ions in solution with EDTA in the presence of metal nitrate solutions.

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References and Notes

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