UNIVERSITI PUTRA MALAYSIA

PREPARATION AND CHARACTERIZATION OF POLYPYRROLEPOLYETHYLENE GLYCOL CONDUCTING POLYMER COMPOSITE FILMS

LIM MEI YEE

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PREPARATION AND CHARACTERIZATION OF POLYPYRROLE-POLYETHYLENE GLYCOL CONDUCTING POLYMER COMPOSITE FILMS

By

LIM MEI YEE

Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfilment of the Requirement for the Degree of Master of Science

August 2007
DEDICATION

To my beloved parents Lim Tet Yoong and Lau Mau Ching
For their endless love and concern……

To my beloved Wan Kee Peng
For his romantic love, support, understanding and care……

To my supervisor Prof. Dr. Anuar bin Kassim, PhD
For his guidance, advice, understanding and endless support……

To my co-supervisors Prof Mohd. Zobir Hussein, PhD and Professor Wan Mahmood Mat Yunus, PhD
For their kindly advice and indispensable support……

To my senior H. N. M. Ekramul Mahmud, PhD
For his wonderful encouragement and support……

To my friends
For their wonderful love and generous moral support……
Polypyrrole-polyethylene glycol (PPy-PEG) conducting polymer composite films were electrochemically prepared on Indium Tin Oxide (ITO) glass electrode from an aqueous solution containing pyrrole monomer, \( p \)-toluene sulfonate dopant and poly(ethylene glycol) as an insulating polymer. The PPy-PEG composite films prepared from different process conditions were characterized by Fourier Transform infrared (FT-IR) spectroscopy, electrical conductivity measurement, photoacoustic spectroscopy, X-ray diffraction (XRD) analysis and optical microscopy. The FT-IR study of PPy-PEG composite films shows the evidence of the incorporation of PEG in PPy structure forming PPy-PEG composite films.

The highest electrical conductivity of 61 S/cm and thermal diffusivity of \( 7.88 \times 10^{-7} \) m\(^2\)s\(^{-1}\) were shown by the PPy-PEG composite film prepared from 0.20 M pyrrole, 0.10 M \( p \)-toluene sulfonate and 1 x \( 10^{-3} \) M PEG at 1.20 volt (vs SCE) at room temperature among all the PPy-PEG composite films produced. The conductivity data of PPy-PEG shows that with the increase in PEG concentration in the pyrrole
solution, the electrical conductivity of the prepared PPy-PEG film increased up to a certain level due to the increase in conjugation length and later it decreased with further increase in PEG concentration, which is again linked with the decrease in conjugation length.

The measured values of thermal diffusivity and electrical conductivity for the PPy-PEG composites films showed that there was a correlation between thermal diffusivity and electrical conductivity. Both thermal diffusivity and electrical conductivity showed a similar peak for the same process condition in respective composite films.

The XRD results of PPy-PEG composite films showed that the films were amorphous with very little order. The optical micrographs of PPy-PEG showed the globular surface morphology. The changes in globular surface morphology with the change in process condition of the film preparation indicated that the process parameters used to prepare the composite films had a strong influence over the surface morphology.
Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Master Sains

PENYEDIAAN DAN PENCIRIAN UNTUK POLIPIROL-POLIETILENA GLIKOL FILEM KOMPOSIT POLIMER PENGALIR

Oleh

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Ogos 2007

Pengerusi : Professor Anuar Kassim, PhD

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Filem komposit polimer pengalir bagi pasangan polipirol-polietilena glikol (PPy-PEG) dan telah disediakan melalui kaedah elektrokimia di atas elektrod kaca Indium Stanum oksida (ITO) daripada larutan akueus yang mengandungi monomer pirol, dopan p-toluena sulfonat dan polietilena glikol sebagai polimer selulosa penebat. Filem komposit bagi PPy-PEG telah disediakan melalui keadaan proses yang berlainan dan langkah pencirian telah dilakukan melalui penyerapan infra merah (FT-IR), penentukuran kekonduksian elektrik, spektrokopip fotoakustik, pengimbasan analisis sinar-X, dan mikroskopi optikal. Kajian FT-IR ke atas filem komposit PPy-PEG nyata menunjukkan kemasukan PEG ke dalam struktur PPy berkaitan dengan pembentukan filem komposit PPy-PEG.

Filem komposit PPy-PEG yang memberi nilai konduktiviti elektrik tertinggi dan nilai resapan terma iaitu 61 S/cm dan 7.88 x 10^{-7} m^2s^{-1} berbanding dengan yang lain telah disediakan daripada larutan 0.20 M pirol, 0.10 M p-toluena sulfonat dan 1 x 10^{-3} M PEG pada keupayaan 1.20 v (melawan SCE) pada suhu bilik. Data
kekonduksian elektrik, PPy-PEG menunjukkan dengan penambahan kepekatan PEG dalam larutan pirol, kekonduksian elektrik filem PPy-PEG turut bertambah ke satu paras tertentu disebabkan penambahan panjang konjugatan dan nilai kekonduksian elektrik berkurangan dengan penambahan PEG berlebihan iaitu berkaitan langsung dengan panjang konjugatan yang berkurangan.

Nilai resapan terma dan konduttiviti elektrik untuk filem komposit PPy-PEG menunjukkan satu hubungan pertalian secara bersistem di antara resapan terma dan konduttiviti elektrik. Kedua-dua nilai esapan terma dan konduttiviti elektrik esapan terma dan konduttiviti elektrik menyatakan kesamaan puncak daripada keadaan proses yang sama dalam komposit filem masing-masing.

Keputusan XRD menunjukkan bahawa filem komposit PPy-PEG adalah bersifat amorfus dengan mempunyai sedikit sifat ketertiban. Mikrograf optikal PPy-PEG menunjukkan sifat morfologi permukaannya yang berbentuk sfera. Perubahan keadaan penyediaan turut mengubah morfologi permukaan sfera jelas menunjukkan bahawa parameter dalam proses penyediaan mempunyai kesan untuk mempengaruhi sifat permukaannya.
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The completion of this project is not a one-man work. It is a project, which could only get completed on time with the help of many parties. Therefore I would like to take this opportunity to express my gratitude to all of them to show my appreciation for their support.

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spectroscopy. Special thanks are extended to my lab members Miss Masnizaayu, Mrs. Rozita Yahya, and Mr. Fariz Adzmi who helped me in every possible way and providing a congenial and enthusiastic atmosphere in the laboratory.

Last but not least, special greetings and thanks to my beloved family for their wonderful love and generous moral support.
I certify that an Examination Committee met on 3rd August, 2007 to conduct the final examination of Lim Mei Yee on her Master of Science thesis entitled “Preparation and Characterization Conducting Polymer Composite Films: Polypyrrole-Polyethylene Glycol” in accordance with Universiti Pertanian Malaysia (Higher Degree) Act 1980 and Universiti Pertanian Malaysia (Higher Degree) Regulations 1981. The Committee recommends that the candidate be awarded the relevant degree. Members of the Examination Committee are as follows:

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DECLARATION

I hereby declare that the thesis is based on my original work except for quotations and citations which have been duly acknowledged. I also declare that it has not been previously or concurrently submitted for any other degree at UPM or other institutions.

____________________

LIM MEI YEE

Date: 19 December 2007
# TABLE OF CONTENTS

<table>
<thead>
<tr>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>DEDICATION</td>
</tr>
<tr>
<td>ABSTRACT</td>
</tr>
<tr>
<td>ABSTRAK</td>
</tr>
<tr>
<td>ACKNOWLEDGEMENTS</td>
</tr>
<tr>
<td>APPROVAL</td>
</tr>
<tr>
<td>DECLARATION</td>
</tr>
<tr>
<td>LIST OF TABLES</td>
</tr>
<tr>
<td>LIST OF FIGURES</td>
</tr>
<tr>
<td>LIST OF ABBREVIATIONS</td>
</tr>
</tbody>
</table>

## CHAPTER

### I INTRODUCTION
- Conducting Polymers | 1
- History of Conducting Polymers | 6
- Research Objectives | 14

### II LITERATURE REVIEW
- Monomer | 15
- Counter ion or Dopant | 16
- Insulating Polymer | 18
- Solvent | 19
- Polypyrrole as a Conducting Polymer | 21
- Mechanisms of Pyrrole Electropolymerization- Role of Doping | 23
- Mechanisms of Pyrrole Electropolymerization | 25
- Applications of Polypyrrole Conducting Polymers
  - Batteries | 35
  - Polypyrrole Microactuators | 36
  - Condenser | 36
  - Transparent Loudspeakers | 37
  - Electromagnetic Interference (EMI) | 38
  - Shielding Material | 38
  - Brilliant | 38
  - Microelectronics | 39

### III MATERIALS AND METHODS
- Electrochemical Synthesis | 41
- Electrode | 41
- Monomer, Electrolyte and Insulating Polymer | 42
- Preparation of PPy-PEG Composite Films | 43
Characterization of the PPy-PEG Composite Films

<table>
<thead>
<tr>
<th>Characterization</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>Electrical Conductivity</td>
<td>44</td>
</tr>
<tr>
<td>Fourier Transform Infrared Spectrometry</td>
<td>45</td>
</tr>
<tr>
<td>(FT-IR)</td>
<td></td>
</tr>
<tr>
<td>X-ray Diffraction (XRD)</td>
<td>46</td>
</tr>
<tr>
<td>Optical Microscopy</td>
<td>46</td>
</tr>
<tr>
<td>Open Photoacoustic Technique</td>
<td>47</td>
</tr>
</tbody>
</table>

### IV RESULTS AND DISCUSSION

**Electrochemically Prepared Polypyrrole Composite Films**

<table>
<thead>
<tr>
<th>Molecular Structure of PPy-PEG Composite Films</th>
<th>50</th>
</tr>
</thead>
<tbody>
<tr>
<td>Electrical Conductivity of PPy-PEG Composite Films</td>
<td>54</td>
</tr>
<tr>
<td>Effect of PEG Concentration on the Electrical Conductivity of PPy-PEG Composite Films</td>
<td>55</td>
</tr>
<tr>
<td>Effect of Pyrrole Concentration on the Electrical Conductivity of PPy-PEG Composite Films</td>
<td>57</td>
</tr>
<tr>
<td>Effect of Dopant Concentration on the Electrical Conductivity of PPy-PEG Composite Films</td>
<td>58</td>
</tr>
<tr>
<td>Effect of Applied Voltage on the Electrical Conductivity of PPy-PEG Composite Films</td>
<td>60</td>
</tr>
</tbody>
</table>

**Thermal Diffusivity of PPy-PEG Composite Films**

| Effect of PEG Concentration on the Thermal Diffusivity of PPy-PEG Composite Films | 64   |
| Effect of Pyrrole Concentration on the Thermal Diffusivity of PPy-PEG Composite Films | 66   |
| Effect of Dopant Concentration on the Thermal Diffusivity of PPy-PEG Composite Films | 68   |
| Effect of Applied Voltage on the Thermal Diffusivity of PPy-PEG Composite Films | 70   |

**The Relation Between Electrical Conductivity and Thermal Diffusivity of PPy-PEG Composite Films**

| Molecular Order in PPy-PEG Composite Films | 79   |
| Effect of PEG Concentration on the Molecular Order of PPy-PEG Composite Films | 83   |
| Effect of Pyrrole Concentration on the Molecular Order of PPy-PEG Composite Films | 87   |
| Effect of Dopant Concentration on the Molecular Order of PPy-PEG Composite Films | 89   |
Effect of Applied Voltage on the Molecular Order PPy-PEG Composite Films
Morphological Study of PPy-PEG Composite Films
Effect of PEG Concentration on the Morphology of PPy-PEG Composite Films
Effect of Pyrrole Concentration on the Morphology of PPy-PEG Composite Films
Effect of Dopant Concentration on the Morphology of PPy-PEG Composite Films
Effect of Applied Voltage on the Morphology PPy-PEG Composite Films

VI CONCLUSIONS

REFERENCES/BIBLIOGRAPHY

APPENDICES

BIODATA OF THE AUTHOR

LIST OF PUBLICATIONS
# LIST OF TABLES

<table>
<thead>
<tr>
<th>Table</th>
<th>Description</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.1</td>
<td>Conducting polymers: preparation methods and conductivities</td>
<td>8</td>
</tr>
<tr>
<td>2.1</td>
<td>Examples of copolymer prepared from pyrrole functional polymers</td>
<td>16</td>
</tr>
<tr>
<td>2.2</td>
<td>Polypyrrole films with different anions</td>
<td>18</td>
</tr>
<tr>
<td>2.3</td>
<td>Physical properties of polypyrrole with $\rho$-toulenesulfonate films</td>
<td>20</td>
</tr>
<tr>
<td>3.1</td>
<td>The parameter used to synthesize PPy-PEG composite films prepared from various process conditions</td>
<td>43</td>
</tr>
<tr>
<td>4.1</td>
<td>Physical observation of PPy-PEG Composite films</td>
<td>48</td>
</tr>
<tr>
<td>4.2</td>
<td>Diffraction orders for stretch-aligned polypyrrole (Warren, 2001)</td>
<td>82</td>
</tr>
</tbody>
</table>
**LIST OF FIGURES**

<table>
<thead>
<tr>
<th>Figure</th>
<th>Description</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.1</td>
<td>The conductivity of materials (Przyluski, 1991)</td>
<td>4</td>
</tr>
<tr>
<td>2.1</td>
<td>Pyrrole is synthesized into polypyrrole with the dopant (A⁻)</td>
<td>17</td>
</tr>
<tr>
<td>2.2</td>
<td>Simple band picture explaining the difference between an insulator, a semiconductor and a metal</td>
<td>24</td>
</tr>
<tr>
<td>2.3</td>
<td>First part of the reaction mechanism of polypyrrole film formation</td>
<td>26</td>
</tr>
<tr>
<td>2.4</td>
<td>Second part of the reaction mechanism of polypyrrole film formation</td>
<td>30</td>
</tr>
<tr>
<td>2.5</td>
<td>Third part of the reaction mechanism of polypyrrole film Formation</td>
<td>33</td>
</tr>
<tr>
<td>3.1</td>
<td>The experimental set-up for the electrochemical preparation of PPy-PEG composite films</td>
<td>42</td>
</tr>
<tr>
<td>3.2</td>
<td>Circuit used for conductivity measurements</td>
<td>45</td>
</tr>
<tr>
<td>4.1</td>
<td>FT-IR spectra of PPy-PEG composite film, PPy film, PEG and p-toluene sulfonate</td>
<td>52</td>
</tr>
<tr>
<td>4.2</td>
<td>The formation of PPy-PEG composite film from pyrrole, monomer, p-toluene sulfonate dopant and PEG</td>
<td>53</td>
</tr>
<tr>
<td>4.3</td>
<td>Electrical conductivity of PPy-PEG composite films versus PEG concentration used to prepare the PPy-PEG composite films</td>
<td>56</td>
</tr>
<tr>
<td>4.4</td>
<td>Electrical conductivity of PPy-PEG composite films against pyrrole concentration</td>
<td>58</td>
</tr>
<tr>
<td>4.5</td>
<td>Electrical conductivity of PPy-PEG composite films against different p-toluene sulfonate concentration</td>
<td>60</td>
</tr>
<tr>
<td>4.6</td>
<td>Electrical conductivity of PPy-PEG composite films against different applied voltage (vs SCE).</td>
<td>62</td>
</tr>
<tr>
<td>4.7</td>
<td>Thermal diffusivity of PPy-PEG composite films versus PEG concentration used to prepare the PPy-PEG composite films</td>
<td>65</td>
</tr>
</tbody>
</table>
4.8 Thermal diffusivity of PPy-PEG composite films versus pyrrole concentration used to prepare the PPy-PEG composite films

4.9 Thermal diffusivity of PPy-PEG composite films against different $p$-toluene sulfonate concentration

4.10 Thermal Diffusivity of PPy-PEG composite films against different applied voltage (vs SCE)

4.11 The electrical conductivity and thermal diffusivity of PPy-PEG composite films against different PEG concentration

4.12 The electrical conductivity and thermal diffusivity of PPy-PEG composite films against different pyrrole concentration

4.13 The electrical conductivity and thermal diffusivity of PPy-PEG composite films against different $p$-toluene sulfonate concentration

4.14 The electrical conductivity and thermal diffusivity of PPy-PEG composite films against different applied voltage

4.15 Stacked lamellae

4.16 Fringed mycelles

4.17 XRD diffractograms of PPy-PEG composite films prepared from various concentrations of PEG

4.18 The XRD diffractograms of PEG

4.19 The XRD diffractograms of PPy

4.20 The XRD diffractograms of PPy-PEG composite film

4.21 XRD diffractograms of PPy-PEG composite films prepared from various concentrations of pyrrole

4.22 XRD diffractograms of PPy-PEG composite films prepared from various concentrations of $p$-toluene sulfonate

4.23 XRD diffractograms of PPy-PEG composite films prepared from various applied voltage
4.24 The optical micrographs of the solution side of PPy-PEG composite films produced from using (a) $5 \times 10^{-4}$ M, (b) $1 \times 10^{-3}$ M, (c) $3 \times 10^{-3}$ M, (d) $5 \times 10^{-3}$ M, (e) $7 \times 10^{-3}$ M and (f) $9 \times 10^{-3}$ M PEG in the pyrrole solution (Magnification: 20x)

4.25 The optical micrographs of the electrode side of PPy-PEG composite films produced from using (a) $5 \times 10^{-4}$ M, (b) $1 \times 10^{-3}$ M, (c) $3 \times 10^{-3}$ M, (d) $5 \times 10^{-3}$ M, (e) $7 \times 10^{-3}$ M and (f) $9 \times 10^{-3}$ M PEG in the pyrrole solution (Magnification: 20x)

4.26 The optical micrographs of the solution side of (a) only PPy film and (b) PPy-PEG composite film (magnification: 20x)

4.27 The optical micrographs of the electrode side of (a) only PPy film and (b) PPy-PEG composite film (magnification: 20x)

4.28 The optical micrographs of the solution side of PPy-PEG composite films produced from using (a) 0.10 M, (b) 0.20 M, (c) 0.30 M and (d) 0.40 M pyrrole (magnification: 20x)

4.29 The optical micrographs of the electrode side of PPy-PEG composite films produced from using (a) 0.10 M, (b) 0.20 M, (c) 0.30 M and (d) 0.40 M pyrrole (magnification: 20x)

4.30 The optical micrographs of the solution side of PPy-PEG composite films produced from using (a) 0.05 M, (b) 0.10 M, (c) 0.20 M and (d) 0.30 M $\rho$-toluene sulfonate (Magnification: 20x)

4.31 The optical micrographs of the electrode side of PPy-PEG composite films produced from using (a) 0.05 M, (b) 0.10 M, (c) 0.20 M and (d) 0.30 M $\rho$-toluene sulfonate (Magnification: 20x)

4.32 The optical micrographs of the solution side of PPy-PEG composite films produced from using (a) 0.80 volt (vs SCE), (b) 1.20 volt (vs SCE) and (c) 1.50 volt (vs SCE) (Magnification: 20x)

4.33 The optical micrographs of the electrode side of PPy-PEG composite films produced from using (a) 0.80 volt (vs SCE), (b) 1.20 volt (vs SCE) and (c) 1.50 volt (vs SCE) (Magnification: 20x)
LIST OF ABBREVIATIONS

InSb     Indium Antimonide
AsF₅     Arsenic Pentafluoride.
TTF      Tetrathiafulvalene
TCNQ     Tetracyanoquinodimethane
CH       Methyl
Tc       Glass transition temperature
Ch       Chemical preparation
EP       Electropolymerization
Pt       Platinum
Py       Pyrrole
PPy      Polypyrrole
PEG      Polyethylene Glycol
FT-IR    Fourier Transform Infrared Spectrometry
XRD      X-ray diffraction analysis
vs       Versus
PMMA     Poly (methyl methacrylate)
PTHF     Polytetrahydrofuran
PCL      Polycaprolactone
PDMS     Poly (dimethyl siloxane)
A⁻       Dopant/ counter-ions
BS       Benzenesulfonate
PTS      sodium ρ-toluenesulfonate
EBS  sodium 4-ethylbenzenesulfonate
MXS  sodium m-xylene-4-sulfonate
MSS  sodium mesitylnesulfonate
OBS  sodium 4-n-noctylbenzenesulfonate
DBS  sodium dodecylbenzenesulfonate
ET$_4$NPTS  tetraethylammonium $\rho$-toulenesulfonate
mA  Mega ampere
H$_2$O  Hydrogen oxide
CH$_3$CN  Methyl Cyanide
psi  Per square inch
$\sigma$  Conductivity
R  Monomer
R$^{+*}$  Cation radical
ITO  Indium-tin-oxide
SCE  Saturated calomel electrode
V  Voltage
dc  Direct current
$\mu$m  Micrometre
cm  Centimeter
Hz  Hetze
2$\theta$  2 Theta
$\rho$-TS  $\rho$-toluene sulfonate
DMA  Dynamic mechanical analysis
EMI  Electromagnetic interference
Polymers are long chain giant organic molecules assembled from many smaller molecules called monomers. Polymers consist of many repeating monomer units in long chains. The interlinking of many units has given the polymer its name, poly meaning ‘many’ and mer meaning ‘part’ (in Greek) (Gowariker et al., 1987). A polymer is analogous to a necklace made from many small beads (monomers). These monomers react together chemically to give a variety of molecular architectures ranging from linear structures to a three dimensional network of polymer chains.

Another common name for many synthetic polymers is plastic which comes from the Greek word "plastikos", suitable for molding or shaping. Many objects in daily use from packing, wrapping, and building materials include half of all polymers synthesized. Plastics are polymers, molecules that form long chains, repeating themselves like pearls in a necklace. In becoming electrically conductive, a polymer has to imitate a metal, that is, its electrons need to be free to move and not bound to the atoms. The first condition for this is that the polymer consists of alternating single and double bonds, called conjugated double bonds (Said et al., 2000).

Conducting polymer are polymers which can exhibit significant level of electrical conductivity. The electrical conductivity exhibited by conducting polymers is
attributed to the presence of “free electrons” within the body of the specimen. Conducting polymers are usually polyconjugated structures which are insulators in the pure state but when treated with an oxidizing or a reducing agent can be converted into polymer salts with electrical conductivities comparable to metals. Conducting polymeric materials possess great design flexibility together with a number of characteristics that are desirable for a number of specific applications in the fields of catalysis, conversion and storage of energy, chemical and biochemical sensing, microelectronics and optoelectronics (Skotheim, 1998).

Supercapacitors are attracting great attention because of their high capacitance and potential applications in electronic devices. There has been more interest in two types of supercapacitors, the double layer supercapacitors and the redox supercapacitors with different charge storage modes. The redox supercapacitor involves faradic process due to redox reaction. Due to versatility of structure and low cost compared to noble metal oxides, electronically conducting polymers represents a promising class of active materials for electrodes of the redox supercapacitors (Hughes et al., 2002).

For most of the history of polymer technology, one of the most valued properties of synthetic polymers has been their ability to act as excellent electrical insulators both at high voltages and at high frequencies. In spite of this there has been an interest for many years in the possibility of producing electrically conducting polymers. The obvious attraction is to combine in one material the electrical properties and the high added value applications of a semiconductor or a metal with the advantages of a polymer.
Approximately two decades ago, it has been discovered that polyacetylene, which is a type of polymer containing conjugated single and double bonds in its structure, could become highly conductive after carrying out a structural modification process called “doping” (Shirakawa et al., 1977). During the doping process, an organic polymer, either an insulator or a semiconductor having a small conductivity typically in the range of $10^{-10}$ to $10^{-5}$ S/cm, is converted to a polymer, which is in the metallic conducting regime ($1-10^{-4}$ S/cm). Figure 1.1 shows the conductivity range of metals, semiconductors and insulators.