



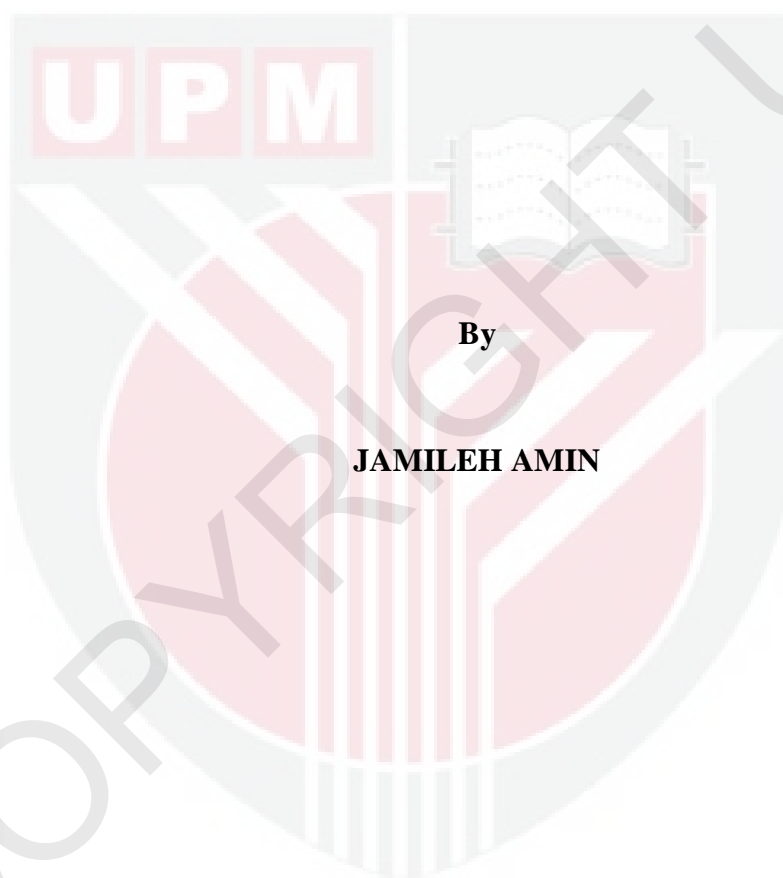
**UNIVERSITI PUTRA MALAYSIA**

**PREPARATION, AND ELECTRICAL, MAGNETIC AND THERMAL  
INVESTIGATION OF POLYPYRROLE-CHITOSAN COMPOSITE AND  
POLYPYRROLE-CHITOSAN-IRON OXIDE POLYMER NANOCOMPOSITE**

**JAMILEH AMIN**

**FS 2013 37**

**PREPARATION, AND ELECTRICAL, MAGNETIC AND THERMAL  
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POLYPYRROLE-CHITOSAN-IRON OXIDE POLYMER  
NANOCOMPOSITE**



**By**

**JAMILEH AMIN**

**Thesis submitted to the School of Graduate Studies, Universiti Putra  
Malaysia, in Fulfillment of the Requirements for the Degree of Doctor of  
Philosophy**

**June 2013**

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*Dedicated to my beloved mother, father, my sister Jelveh and her family*

*Without their understanding and support, I would never have completed this  
project.*



Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfillment of the requirement for the degree of Doctor of Philosophy

**PREPARATION, AND ELECTRICAL, MAGNETIC AND THERMAL INVESTIGATION OF POLYPYRROLE- CHITOSAN COMPOSITE AND POLYPYRROLE-CHITOSAN-IRON OXIDE POLYMER NANOCOMPOSITE**

By

**JAMILEH AMIN**

**June 2012**

**Chairman: Professor Zainal Abidin Talib, PhD**

**Faculty: Science**

Conducting polymer composite based on Polypyrrole-Chitosan (PPy-CHI) and Polypyrrole-Chitosan-Iron oxide nanoparticles (PPy-CHI-Fe<sub>3</sub>O<sub>4</sub>) nanocomposite were prepared by using in-situ chemical polymerization method. (PPy-CHI) composites and (PPy-CHI-Fe<sub>3</sub>O<sub>4</sub>) nanocomposite were prepared with various percentages of CHI and Fe<sub>3</sub>O<sub>4</sub> ranging from 0.0% (w/v) to 0.9% (w/v) and 0 wt% to 15wt% respectively. Results from conductivity experiments revealed that the highest conductivity was obtained from PPy-CHI-Fe<sub>3</sub>O<sub>4</sub> nanocomposite prepared from 0.1 % (w/v) CHI, 3 Molar PPy and 3wt% Fe<sub>3</sub>O<sub>4</sub> in 30 minutes at room temperature

The X-ray diffractogram of PPy and CHI illustrated a broad scattering peak for PPy and two scattering peaks of almost equal intensity for CHI which is due to

their highly amorphous and semi-crystallinity structure respectively. The XRD spectra for PPy-CHI composite was almost similar to those of PPy with a broad scattering at around  $25^{\circ}$ - $26^{\circ}$  indicating an amorphous structure. The XRD spectrum of PPy-CHI- $\text{Fe}_3\text{O}_4$  nanocomposite demonstrated similar to those observed from PPy and PPy-CHI composite matrix especially in the lower weight percentage of  $\text{Fe}_3\text{O}_4$ . However, as the nanoparticle loading increased, the characteristic peaks of  $\text{Fe}_3\text{O}_4$  began to dominate the nanocomposite spectra indicating to some uncoated Iron oxide nanoparticle which was confirmed by Energy-Dispersive X-ray (EDX) and transmission electron microscopy (TEM). The Fourier transform infrared spectroscopy (FT-IR) spectra of PPy-CHI and PPy-CHI- $\text{Fe}_3\text{O}_4$  illustrated almost the same characteristic positions of IR absorption bands similar to those of PPy. The small shift of PPy in the PPy-CHI composites is due to the identical peaks of PPy and CHI while in the PPy-CHI- $\text{Fe}_3\text{O}_4$  nanocomposites, the matrix layer of polypyrrole which covered the surface of iron oxide has absorbed most of the IR radiation. The results of Scanning Electron microscopy (SEM) and transmission electron microscopy (TEM) confirmed that the  $\text{Fe}_3\text{O}_4$  nanoparticles have been coated with the layers of polymeric matrix. A distribution of discrete globular nanoparticles with almost uniform size and dimensions was exhibited in all PPy-CHI- $\text{Fe}_3\text{O}_4$  nanocomposite samples. The reduced size of PPy-CHI and PPy-CHI- $\text{Fe}_3\text{O}_4$  particles was explained by the effect of Chitosan and iron oxide, the steric stabilization effect of CHI and a core-shell structure for PPy-CHI- $\text{Fe}_3\text{O}_4$  nanocomposite which was confirmed by SEM and TEM.

The results of Vibrating Sample Magnetometer (VSM) revealed the magnetic properties for various PPy-CHI- $\text{Fe}_3\text{O}_4$  nanocomposites strongly depended on the

concentration loading of  $\text{Fe}_3\text{O}_4$ . The hysteresis loops of VSM illustrated superparamagnetic behavior for all the nanocomposite of PPy–CHI– $\text{Fe}_3\text{O}_4$  with different loading  $\text{Fe}_3\text{O}_4$  percentage. The  $M_s$  (saturation magnetization) and  $H_c$  (coactivity) were monitored for samples with percentage loaded of  $\text{Fe}_3\text{O}_4$  from 0.1 wt% to 15 wt%.  $M_s$  increased from 0.874 emu/g to 5.97 emu/g while  $H_c$  decreased from 241.4 Oe to 194.48 Oe.

The results of electron spin resonance (ESR) spectroscopy revealed a reduction in the Peak-to-peak line-width ( $\Delta H_{pp}$ ) value between PPy and PPy–CHI. The adding of  $\text{Fe}_3\text{O}_4$  have resulted in ( $\Delta H_{pp}$ ) values increased in the order PPy–CHI < PPy–CHI– $\text{Fe}_3\text{O}_4$  (1wt%) < PPy–CHI– $\text{Fe}_3\text{O}_4$  (3wt%) > PPy–CHI– $\text{Fe}_3\text{O}_4$  (5wt%) < PPy–CHI– $\text{Fe}_3\text{O}_4$  (7wt %) < PPy–CHI– $\text{Fe}_3\text{O}_4$  (10wt %) < PPy–CHI– $\text{Fe}_3\text{O}_4$  (15wt %) at room temperature. The spin concentration ( $N_s$ ) measurement of PPy–CHI– $\text{Fe}_3\text{O}_4$  with the various  $\text{Fe}_3\text{O}_4$  content revealed to be larger than PPy–CHI ( $8 \times 10^6$ ,  $1.59 \times 10^7$ ,  $3 \times 10^7$ ,  $2.29 \times 10^7$ ,  $2.43 \times 10^7$  and  $2.49 \times 10^7$  spin g<sup>-1</sup> for PPy–CHI and 1wt%, 3wt%, 5wt%, 7wt% and 10wt%, of  $\text{Fe}_3\text{O}_4$  respectively). The increase in percentage loading of iron oxide also resulted in better thermal stability of the nanocomposites.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk Ijazah Doktor Falsafah

**PENYIASATAN DAN PENYEDIAAN, ELEKTRIKAL, MAGNETIK DAN  
TERMAL BAGI POLIPYRROLE-KITOSAN KOMPOSIT  
POLIPYRROLE-KITOSAN OKSIDA POLIMER KOMPOSIT NANO**

Oleh

**JAMILEH AMIN**

**Jun 2012**

**Pengerusi : Profesor Zainal Abidin Talib, PhD**

**Fakulti: Sains**

Penyediaan komposit polimer berasaskan ke atas Polipyrrole-Kitosan (PPy-CHI) dan partikel nano Polipyrrole-Kitosan-Besi oksida (PPy-CHI-Fe<sub>3</sub>O<sub>4</sub>) komposit nano telah disediakan menggunakan kaedah pempolimeran kimia setempat. Komposit (PPy-CHI) dan komposit nano (PPy-CHI-Fe<sub>3</sub>O<sub>4</sub>) telah disediakan menggunakan pelbagai peratusan CHI dan Fe<sub>3</sub>O<sub>4</sub> masing-masing dari 0.0% (w/v) sehingga 0.9% (w/v) dan 0 wt% kepada 15wt%. Dapatan daripada eksperimen kekonduksian mendapati bahawa konduksi tertinggi diperolehi daripada komposit nano PPy-CHI-Fe<sub>3</sub>O<sub>4</sub> disediakan daripada 0.1 % (w/v) CHI, 3 Molar PPy dan 3wt% Fe<sub>3</sub>O<sub>4</sub> dalam 30 minit pada suhu bilik.

X-ray difraktogram bagi PPy dan CHI ditunjukkan pada puncak tertinggi serakan terluar bagi PPy dan dua puncak serakan bagi hampir sama keamatannya bagi



CHI yang mana disebabkan masing-masing oleh struktur amorphous yang tinggi dan semi-hablur mereka yang tinggi masing-masing. Spektre XRD bagi komposit PPy-CHI adalah hampir sama dengan PPy yang serakan luasnya adalah pada  $25^{\circ}$ - $26^{\circ}$  menunjukkan struktur amorfus. Spektre XRD spectrum bagi komposit nano PPy-CHI-Fe<sub>3</sub>O<sub>4</sub> menunjukkan hasil sama dengan yang telah diperhatikan daripada matrik komposit PPy dan PPy-CHI khasnya dalam peratusan berat yang lebih rendah bagi Fe<sub>3</sub>O<sub>4</sub>. Walaubagaimanapun, semakin bebanan partikel nano bertambah, karekter puncak bagi Fe<sub>3</sub>O<sub>4</sub> mula mendominasi spektrum komposit nano menunjukkan yang beberapa partikel nano Besi oksida tidak diselaputi yang telah disahkan oleh Energy-Dispersive X-ray (EDX) dan transmisi mikroskopi elektron (TEM). Fourier merubah spektrum infra merah spektroskopi (FTIR) bagi PPy-CHI dan PPy-CHI-Fe<sub>3</sub>O<sub>4</sub> menunjukkan hampir sama karekter kedudukannya bagi penyerapan jalur IR adalah sama dengan PPy. Perubahan kecil PPy dalam komposit PPy-CHI adalah disebabkan kepada puncak yang sama bagi PPy dan CHI sementara dalam komposit nano PPy-CHI-Fe<sub>3</sub>O<sub>4</sub>, lapisan matrik bagi polipyrrole yang telah diselaputi dengan permukaan besi oksida telah menyerap kebanyakan radiasi IR. Hasil daripada mikroskopi Scanning Electron (SEM) dan transmisi mikroskopi elektron (TEM) mengesahkan yang partikel nano Fe<sub>3</sub>O<sub>4</sub> telah dilaputi dengan lapisan matrik polimerik. Taburan bagi partikel nano diskret globular dengan hampir saiz uniform dan dimensi yang sama telah ditunjukkan dalam semua sampel komposit nano PPy-CHI-Fe<sub>3</sub>O<sub>4</sub>. Pengurangan saiz bagi PPy-CHI dan partikel PPy-CHI-Fe<sub>3</sub>O<sub>4</sub> telah diterangkan dengan kesan oksida Kitosan dan besi, kesan penstabilan sterik bagi CHI dan struktur teras-luaran bagi komposit nano PPy-CHI-Fe<sub>3</sub>O<sub>4</sub> telah disahkan dengan SEM dan TEM.

Dapatan bagi Vibrating Sample Magnetometer (VSM) menunjukkan yang sifat magnetik bagi pelbagai komposit nano PPy–CHI–Fe<sub>3</sub>O<sub>4</sub> secara kuatnya bergantung kepada bebanan tumpuan bagi Fe<sub>3</sub>O<sub>4</sub>. Histeresis lilitan bagi VSM berasaskan tingkah laku superparamagnetik bagi kesemua komposit nano PPy–CHI– Fe<sub>3</sub>O<sub>4</sub> dengan peratusan berbeza bebanan Fe<sub>3</sub>O<sub>4</sub>. M<sub>s</sub> (penepuan pemagnetan) dan H<sub>c</sub> (koektiviti) telah diperhatikan kepada sampel dengan peratusan yang mengandungi Fe<sub>3</sub>O<sub>4</sub> daripada 0.1 wt% kepada 15 wt%. M<sub>s</sub> meningkat daripada 0.874 emu/g kepada 5.97 emu/g sementara H<sub>c</sub> berkurangan daripada 241.4 Oe kepada 194.48 Oe.

Dapatan dari putaran resonan elektron (ESR) spektroskopi menunjukkan pengurangan dalam nilai Peak-to-peak line-width ( $\Delta H_{pp}$ ) antara PPy dan PPy–CHI. Penambahan Fe<sub>3</sub>O<sub>4</sub> telah menyebabkan dalam nilai ( $\Delta H_{pp}$ ) meningkat dalam turutan PPy–CHI < PPy–CHI–Fe<sub>3</sub>O<sub>4</sub> (1wt%) < PPy–CHI–Fe<sub>3</sub>O<sub>4</sub> (3wt%) > PPy–CHI–Fe<sub>3</sub>O<sub>4</sub> (5wt %) < PPy–CHI–Fe<sub>3</sub>O<sub>4</sub> (7wt %) < PPy–CHI–Fe<sub>3</sub>O<sub>4</sub> (10wt %) < PPy–CHI–Fe<sub>3</sub>O<sub>4</sub> (15wt %) pada suhu bilik. Pengukuran putaran tumpuan (N<sub>s</sub>) bagi PPy–CHI–Fe<sub>3</sub>O<sub>4</sub> dengan pelbagai kandungan Fe<sub>3</sub>O<sub>4</sub> menunjukkan ianya lebih besar daripada PPy–CHI (8×10<sup>6</sup>, 1.59×10<sup>7</sup>, 3×10<sup>7</sup>, 2.29 ×10<sup>7</sup>, 2.43×10<sup>7</sup> dan 2.49×10<sup>7</sup> spin g<sup>-1</sup> kepada PPy–CHI dan 1wt%, 3wt%, 5wt%, 7wt% dan 10wt%, of Fe<sub>3</sub>O<sub>4</sub> masing-masing). Peningkatan dalam peratusan bebanan oksida besi turut menyebabkan kestabilan terma bagi komposit nano yang lebih baik .

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I certify that an Examination Committee has met on 4 June 2013 to conduct the final examination of Jamileh Amin on her Doctor of Philosophy thesis entitled “Peparation, and Electrical, Magnetic and Thermal Investigation of Polypyrrole–Chitosan Composite and Polypyrrole– Chitosan–Iron Oxide Polymer Nanocomposite” in accordance with the Universities and University Colleges Act 1971 and the Constitution of the Universiti Putra Malaysia [P.U.(A) 106] 15 March 1998. The Committee recommends that the student be awarded the Doctor of Philosophy.

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## DECLARATION

I declare that the thesis is 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 University Putra Malaysia or other institutions.

2013/8/14

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