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SYNTHESIS OF DOPED POLYPYRROLE FILM BY AC PLASMA POLYMERIZATION

Mrs. Kanya Tapaneeyakorn

A Thesis Submitted in Partial Fulfillment of the Requirements
for the Degree of Master of Science Program in Petrochemistry and Polymer Science

Faculty of Science

Chulalongkorn University

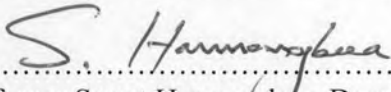
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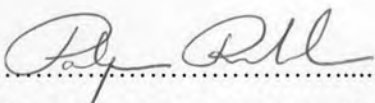
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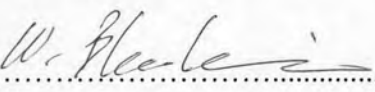
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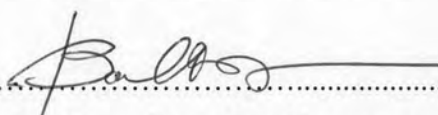
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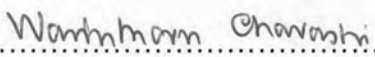
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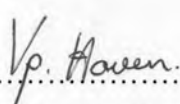
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กัญญา ตปนียากร: การสังเคราะห์โคปโพลีไพโรลฟิล์มโดยเอซีพลาสมาพอลิเมอไรเซชัน (SYNTHESIS OF DOPED POLYPYRROLE FILM BY AC PLASMA POLYMERIZATION) อ.ที่ปริกษาวิทยานิพนธ์หลัก: ผศ.ดร.วรวรรณ พันธุมนาวิน : อ.ที่ปริกษาวิทยานิพนธ์ร่วม: ผศ.ดร.บุญโชติ เผ่าสวัสดิ์ยรรยง; 99 หน้า

งานวิจัยนี้ได้ออกแบบและสร้างระบบเอซีพลาสมาขึ้นเพื่อใช้ในการสังเคราะห์พอลิไพโรลฟิล์มโดยเอซีพลาสมาพอลิเมอไรเซชัน โดยตัวแปรที่ศึกษาในการเกิดพลาสมาพอลิเมอไรเซชันคือความต่างศักย์และเวลาในการสังเคราะห์ในช่วง 800-1500 โวลต์ และ 30-90 นาที ตามลำดับ จากนั้นนำมาวิเคราะห์ด้วยเทคนิคสเปกโทรโฟโตเมทรี การวิเคราะห์หมู่ฟังก์ชันของโครงสร้างฟิล์มพอลิไพโรลด้วยเทคนิคอินฟราเรดสเปกโทรสโกปี พบว่าพอลิไพโรลฟิล์มที่สังเคราะห์ด้วยวิธีนี้มีหมู่ฟังก์ชันสอดคล้องกับสารที่ได้จากการสังเคราะห์ทางเคมี ผลของยูวีวิชันิเบิลสเปกโทรสโกปีแสดงให้เห็นว่าฟิล์มที่ได้มีระบบคอนจูเกตที่สั้นลงเมื่อใช้ความต่างศักย์และเวลาในการสังเคราะห์เพิ่มขึ้น จากการวิเคราะห์พื้นผิวด้วยเทคนิคสแกนนิ่งอิเล็กตรอนไมโครสโคปีพบว่าผิวฟิล์มมีลักษณะเรียบสม่ำเสมอที่ความต่างศักย์ต่ำกว่า 1100 โวลต์ นอกจากนี้เมื่อความต่างศักย์สูงกว่า 1100 โวลต์ จะพบเม็ดเล็กๆ บนพื้นผิวฟิล์ม ผลจากเทคนิคเอนเนอร์จิสเปกโทรสโคปี พบว่าอัตราส่วนอะตอมระหว่างคาร์บอนต่อไนโตรเจนอยู่ในช่วง 4/1 ถึง 5/1 เมื่อเทียบกับพอลิไพโรลในอุดมคติซึ่งมีค่า 4/1 อัตราส่วนอะตอมระหว่างคาร์บอนต่อไนโตรเจนของพลาสมาพอลิไพโรลฟิล์มค่อนข้างสูงกว่าพอลิไพโรลในอุดมคติ สิ่งนี้สอดคล้องกับความน่าจะเป็นไปได้ที่จะเกิดการแตกของพันธะระหว่างคาร์บอนกับไนโตรเจนและคาร์บอนกับคาร์บอนของวงไพโรลในระหว่างเกิดพลาสมาพอลิเมอไรเซชัน การวัดค่าการนำไฟฟ้าแสดงให้เห็นว่าฟิล์มพอลิไพโรลที่ยังไม่ได้โด๊ป (3.4×10^{-8} ถึง 67.2×10^{-8} ซีเมนส์ต่อเซนติเมตร) จะมีค่าสูงกว่าการสังเคราะห์พอลิไพโรลด้วยวิธีทางเคมีและวิธีเคมีไฟฟ้า เมื่อเปรียบเทียบค่าการนำไฟฟ้าของพอลิไพโรลฟิล์มที่ได้ภายใน พบว่าพอลิไพโรลฟิล์มที่ได้ภายนอกจะมีค่าการนำไฟฟ้าที่สูงกว่า อย่างไรก็ตามค่าการนำไฟฟ้าของวัสดุที่ได้ภายนอกจะลดลงอย่างรวดเร็วจนมีค่าใกล้เคียงกับตอนที่ยังไม่ได้โด๊ปภายในเวลาสั้น (5 ชั่วโมง) ส่วนการโด๊ปแบบภายในค่าการนำไฟฟ้าจะมีค่าคงที่เมื่อเวลาผ่านไป (นานกว่า 120 ชั่วโมง)

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KANYA TAPANEEYAKORN: SYNTHESIS OF DOPED POLYPYRROLE FILM
 BY AC PLASMA POLYMERIZATION. THESIS ADVISOR: ASST. PROF.
 WORAWAN BHANTHUMNAVIN, Ph.D., THESIS CO-ADVISOR: ASST. PROF.
 BOONCHOAT PAOSAWATYANYONG, Ph.D., 99 pp


This research designed and assembled an AC plasma system for the synthesis of polypyrrole films by AC plasma polymerization. Plasma polymerization parameters studied were voltage and reaction time in the range of 800-1500 V and 30-90 minutes, respectively. Polypyrrole films were characterized by various spectrophotometric methods. Infrared analyses showed absorption frequencies of important functional groups similar to observed in the case of chemically-synthesized materials. UV-Vis spectra of fabricated polypyrrole films suggested that the conjugative systems were decreased as increased the AC voltage and reaction time. Surface analysis by Scanning Electron Microscopy revealed a smooth surface at the voltage less than 1100 V. In addition, when the voltage is higher than 1100 V, there are grains appearing in it. Results from Energy-dispersive x-ray spectroscopy analysis were suggestive of the atomic ratio of carbon to nitrogen (C/N) of these films in the range of 4/1 to 5/1, comparing to the ideal C/N of polypyrrole at 4/1. The C/N ratio of plasma-polymerized polypyrrole films is somewhat higher than the ideal polypyrrole. This is in good agreement with a possibility of partial cleavage of a C-N and a C-C bond in the pyrrole rings during plasma polymerization. Electrical conductive measurements revealed that the undoped polypyrrole films exhibit higher conductivity (67.2×10^{-8} to 3.4×10^{-8} S/cm) than polypyrrole typically prepared from chemical and electrochemical methods. Compared to *in situ* doped polypyrrole films, the conductivities of polypyrrole films synthesized by *ex situ* doped were higher. However, it drastically decreased and reached undoped values in a short time (5 hours) whereas the conductivity of the one synthesized by *in situ* doped method could sustained for a long period of time (more than 120 hours).

Field of study Petrochemistry and polymer science

Academic year 2008

Student's signature..... 

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LIST OF ABBREVIATIONS

sccm	: Standard Cubic Centimeters per Minute
DC	: Direct Current
AC	: Alternating Current
W	: watt
S	: siemen
μm	: micrometer
min	: minute
mL	: milliliter
cm	: centimeter
nm	: nanometer
mm	: millimeter
m	: meter
V	: voltage
A	: ampere
I	: current
MHz	: Mega Hertz
ATR-FTIR	: Attenuated total reflection-Fourier transform infrared
UV	: Ultraviolet
SEM	: Scanning electron microscope
EDS	: Energy-dispersive x-ray spectroscopy
OES	: Optical emission spectroscopy
E_g	: band gap
VB	: valence band
CB	: conduction band
R	: resistance
eV	: electron volt

LIST OF SYMBOLS

σ	: conductivity
ρ	: resistivity
Ω	: ohms
λ	: wavelength
I	: intensity
A	: transition probability
g	: statistical weight
E	: Energy level
L	: length
w	: width
n	: negative
p	: positive
$^{\circ}\text{C}$: degree celsius