

ไฮโดรจิเนชันของซีล-1,4-พอลิไอโซพรีนและยางธรรมชาติด้วยตัวเร่งปฏิกิริยา  $\text{OsHCl}(\text{CO})(\text{O}_2)(\text{PCy}_3)_2$   
และ  $[\text{Ir}(\text{COD})\text{py}(\text{PCy}_3)]\text{PF}_6$

นาย กิติกร จามรดุสิต

วิทยานิพนธ์เป็นส่วนหนึ่งของการศึกษาตามหลักสูตรปริญญาวิทยาศาสตรดุษฎีบัณฑิต

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คณะวิทยาศาสตร์ จุฬาลงกรณ์มหาวิทยาลัย

ปีการศึกษา 2544

ISBN 947-03-0836-8

จุฬาลงกรณ์มหาวิทยาลัย

HYDROGENATION OF CIS-1,4-POLY(ISOPRENE) AND NATURAL RUBBER  
CATALYZED BY  $\text{OsHCl}(\text{CO})(\text{O}_2)(\text{PCy}_3)_2$  AND  $[\text{Ir}(\text{COD})\text{py}(\text{PCy}_3)]\text{PF}_6$

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A Dissertation Submitted in Partial Fulfillment of the Requirements  
for the Degree of Doctor of Philosophy in Chemical Technology

Department of Chemical Technology

Faculty of Science

Chulalongkorn University

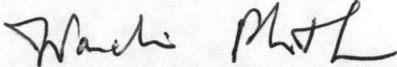
Academic Year 2001

ISBN 947-03-0836-8

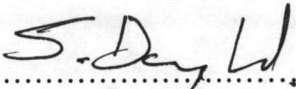
Thesis Title HYDROGENATION OF CIS-1,4-POLY(ISOPRENE) AND  
NATURAL RUBBER CATALYZED BY OsHCl(CO)(O<sub>2</sub>)  
(PCy<sub>3</sub>)<sub>2</sub> AND [Ir(COD)py(PCy<sub>3</sub>)]PF<sub>6</sub>  
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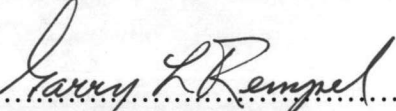
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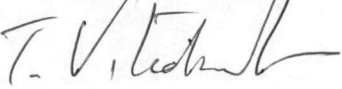
  
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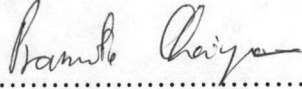
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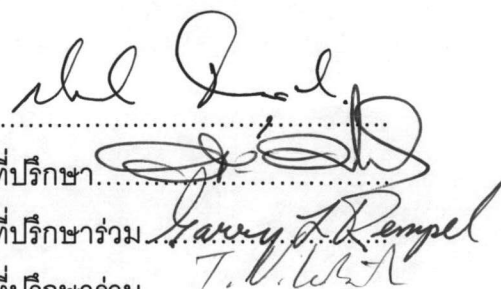
กิติกร จามรดุสิต : ไฮโดรจิเนชันของซีส-1,4-พอลิไอโซพรีนและยางธรรมชาติด้วยตัวเร่งปฏิกิริยา  $\text{OsHCl}(\text{CO})(\text{O}_2)(\text{PCy}_3)_2$  และ  $[\text{Ir}(\text{COD})\text{py}(\text{PCy}_3)]\text{PF}_6$  (HYDROGENATION OF CIS-1,4-POLY(ISOPRENE) AND NATURAL RUBBER CATALYZED BY  $\text{OsHCl}(\text{CO})(\text{O}_2)(\text{PCy}_3)_2$  AND  $[\text{Ir}(\text{COD})\text{py}(\text{PCy}_3)]\text{PF}_6$ ) อ. ที่ปรึกษา : ศ. ดร. ภัทรพรรณ ประศาสน์สาร กิจ, อ. ที่ปรึกษาร่วม : Professor Garry L. Rempel, รศ. ดร. ธราพงษ์ วิทิตสานต์ **115** หน้า ISBN 974-03-0836-8.

งานวิจัยนี้เป็นการศึกษาไฮโดรจิเนชันไฮโดรจิเนชันของซีส-1,4-พอลิไอโซพรีนโดยใช้ตัวเร่งปฏิกิริยา  $\text{OsHCl}(\text{CO})(\text{O}_2)(\text{PCy}_3)_2$  หรือ  $[\text{Ir}(\text{COD})\text{py}(\text{PCy}_3)]\text{PF}_6$  องค์ประกอบทางเคมีของผลิตภัณฑ์ตรวจสอบด้วยเทคนิคคาร์บอนนิวเคลียร์แมกเนติกเรโซแนนซ์สเปกโทรสโคปี ปริมาณไฮโดรจิเนชันสุดท้ายวัดจากเครื่องมือก๊าซออปเทคีนยันด้วยเทคนิคอินฟราเรดสเปกโทรสโคปีและการวิเคราะห์โปรตอนนิวเคลียร์แมกเนติกเรโซแนนซ์ จากการศึกษาจลนพลศาสตร์พบว่าปฏิกิริยาไฮโดรจิเนชันของซีส-1,4-พอลิไอโซพรีนเป็นปฏิกิริยาอันดับหนึ่งกับปริมาณพันธะคู่ ค่าคงที่อัตราเร็วจากการทดลองขึ้นกับความเข้มข้นตัวเร่งปฏิกิริยา ความดันไฮโดรเจนและอุณหภูมิ ค่าพลังงานกระตุ้นของปฏิกิริยาไฮโดรจิเนชันของซีส-1,4-พอลิไอโซพรีนโดยใช้ตัวเร่งปฏิกิริยา  $\text{OsHCl}(\text{CO})(\text{O}_2)(\text{PCy}_3)_2$  และ  $[\text{Ir}(\text{COD})\text{py}(\text{PCy}_3)]\text{PF}_6$  มีค่า 109.3 และ 79.8 กิโลจูล/โมล ตามลำดับ อัตราเร็วไฮโดรจิเนชันด้วย  $\text{OsHCl}(\text{CO})(\text{O}_2)(\text{PCy}_3)_2$  สูงกว่าอัตราเร็วด้วย  $[\text{Ir}(\text{COD})\text{py}(\text{PCy}_3)]\text{PF}_6$  ข้อมูลจลนพลศาสตร์นำไปสู่การศึกษากลไกของไฮโดรจิเนชันด้วยตัวเร่งปฏิกิริยา สมบัติเชิงความร้อนของยางไฮโดรจิเนทตรวจสอบด้วยเทคนิคการวิเคราะห์เทอร์มัลเกรวิเมตริกและดีฟเฟอเรนเชียลสแกนนิ่งแคลอริเมตรี ยางไฮโดรจิเนทมีสมบัติการต้านทานความร้อนดีขึ้นโดยไม่มีการเปลี่ยนแปลงค่าอุณหภูมิคล้ายแก้ว

ยางธรรมชาติมีส่วนประกอบหลักของหน่วยมอโนเมอร์ที่ซ้ำกันคือ ซีส-1,4-พอลิไอโซพรีน งานวิจัยนี้ได้ศึกษาไฮโดรจิเนชันเร่งด้วยกรดของยางธรรมชาติโดยใช้ตัวเร่งปฏิกิริยา  $\text{OsHCl}(\text{CO})(\text{O}_2)(\text{PCy}_3)_2$  ความเข้มข้นของกรดและค่าโคออร์ดิเนตสูงของตัวทำละลายเพิ่มระดับของไฮโดรจิเนชันและอัตราเร็วปฏิกิริยา

ภาควิชา เคมีเทคนิค  
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ปีการศึกษา 2544

ลายมือชื่อนิสิต.....  
ลายมือชื่ออาจารย์ที่ปรึกษา.....  
ลายมือชื่ออาจารย์ที่ปรึกษาร่วม.....  
ลายมือชื่ออาจารย์ที่ปรึกษาร่วม.....



## 4173804523 MAJOR CHEMICAL TECHNOLOGY

KEYWORD: CATALYTIC HYDROGENATION / POLY(ISOPRENE) / NATURAL RUBBER / POLYMER MODIFICATION

KITIKORN CHARMONDUSIT :HYDROGENATION OF CIS-1,4-POLY (ISOPRENE) AND NATURAL RUBBER CATALYZED BY  $\text{OsHCl}(\text{CO})(\text{O}_2)(\text{PCy}_3)_2$  AND  $[\text{Ir}(\text{COD})\text{py}(\text{PCy}_3)]\text{PF}_6$  THESIS ADVISOR : PROF. PATTARAPAN PRASASSARAKICH, THESIS COADVISOR : PROF. GARRY L. REMPEL, ASSOC. PROF. THARAPONG VITIDSANT, **115** pp. ISBN 974-03-0836-8.

The homogeneous hydrogenation of cis-1,4-poly(isoprene), in presence of  $\text{OsHCl}(\text{CO})(\text{O}_2)(\text{PCy}_3)_2$  or  $[\text{Ir}(\text{COD})\text{py}(\text{PCy}_3)]\text{PF}_6$  as a catalyst, has been studied by monitoring the amount of hydrogen consumed during reaction. The composition of hydrogenated product was characterized by  $^{13}\text{C}$  nuclear magnetic resonance spectroscopy. The final degree of olefin conversion measured by computer controlled gas uptake apparatus was confirmed by infrared spectroscopy and  $^1\text{H}$  nuclear magnetic resonance spectroscopy. Kinetic experiments for cis-1,4-poly(isoprene) hydrogenation indicate that the hydrogenation rate is first order with respect to carbon double bond concentration. The experimentally observed rate constants depend on the catalyst concentration, hydrogen pressure, and temperature. The apparent activation energy for the hydrogenation of cis-1,4-poly(isoprene) using  $\text{OsHCl}(\text{CO})(\text{O}_2)(\text{PCy}_3)_2$  and  $[\text{Ir}(\text{COD})\text{py}(\text{PCy}_3)]\text{PF}_6$  were 109.3 and 79.8 kJ/mol, respectively. The rates of hydrogenation in presence of  $\text{OsHCl}(\text{CO})(\text{O}_2)(\text{PCy}_3)_2$  are superior to those obtained with  $[\text{Ir}(\text{COD})\text{py}(\text{PCy}_3)]\text{PF}_6$ . Mechanistic aspects of these catalytic processes are discussed. The thermal properties of hydrogenated rubber samples were determined by thermogravimetric analysis and differential scanning calorimetry. Hydrogenation increases thermal stability of the hydrogenated rubber without affecting its glass transition temperature.

Natural rubber consists of cis-1,4-poly(isoprene) repeating unit in the polymer structure. The hydrogenation of natural rubber in presence of  $\text{OsHCl}(\text{CO})(\text{O}_2)(\text{PCy}_3)_2$  has also been studied. The acid-promote hydrogenation of natural rubber has been observed. The influence of acidity and type of solvent were studied and discussed

Department Chemical Technology  
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## ACKNOWLEDGEMENTS

The author would like to express his gratitude to advisor, Professor Pattarapan Prasassarakich, and co-advisor, Professor Garry L. Rempel and Associate Professor Tharapong Vitidsant for their encouraging guidance, supervision and helpful suggestion throughout this research. In addition, the author is also grateful to Professor Somsak Damronglerd, Dr. Pienpak Tasakorn, and Professor. Pramote Chaivech for serving as chairman and members of thesis committee, respectively, whose comments have been especially valuable.

The author wishes to thank the Golden Jubilee Scholarship (Thailand Research Fund), Chulalongkorn University, and the Natural Science and Engineering Research Council of Canada for financial support of this research. The author also wishes to thank to Dr. Neil T. McManus for his useful discussions and helpful suggestions during this research at University of Waterloo, Canada. Many thanks are going to Technicians of the Department of Chemical Technology, Chulalongkorn University and Department of Chemical Engineering, University of Waterloo for their assistance in building and maintaining the experimental equipment.

Thanks go towards everyone who has contributed suggestions and supports throughout this work. Finally, the author would like to express his deep gratitude to his family for their love, support, and encouragement throughout the tenure of my Ph. D program.

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## ABBREVIATIONS

$\eta_{rel}$	:	Relative viscosity
DSC	:	Differential scanning calorimeter
EPDM	:	Ethylene-propylene copolymers
FT-IR	:	Fourier transform infrared spectroscopy
HNBR	:	Hydrogenated acrylonitrile-butadiene rubber
HNR	:	Hydrogenated natural rubber
HPI	:	Hydrogenated cis-1,4-poly(isoprene)
HPB	:	Hydrogenated poly(butadiene)
HSBR	:	Hydrogenated styrene-butadiene rubber
Ir	:	Iridium
$K_a$	:	Acidity constant
$k_{rds}$	:	Rate-determining step constant
MCB	:	Monochlorobenzene
NBR	:	Acrylonitrile-butadiene rubber
NMR	:	Nuclear magnetic resonance spectroscopy
NR	:	Natural rubber
Os	:	Osmium
PI	:	Cis-1,4-poly(isoprene)
Ru	:	Ruthenium
Rh	:	Rhodium
SBR	:	Styrene-butadiene rubber
SBS	:	Styrene-b-butadiene-b-styrene block copolymers
$T_{id}$	:	Initial decomposition temperature
$T_g$	:	Glass transition temperature
$T_{max}$	:	Maximum decomposition temperature
TA	:	Thermal analysis
TGA	:	Thermogravimetric analysis
THF	:	Tetrahydrofuran