

การสังเคราะห์พอลิยูรีเทน-ยูเรียที่มีสารประกอบเชิงซ้อนซิงค์และคอปเปอร์
4,4'-ไดไฮดรอกซีซาลไซโคลเฮกเซน



นางสาววิภาวี วัฒนอง

ศูนย์วิทยทรัพยากร
จุฬาลงกรณ์มหาวิทยาลัย

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SYNTHESIS OF POLYURETHANE-UREAS CONTAINING
4,4'-DIHYDROXYSALCYCLOHEXANE ZINC AND COPPER COMPLEXES



Miss Wipawee Na Ranong

ศูนย์วิทยทรัพยากร
จุฬาลงกรณ์มหาวิทยาลัย

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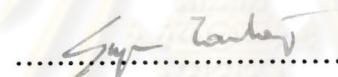
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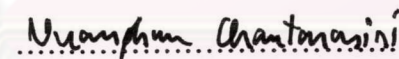


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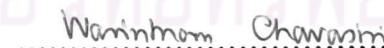
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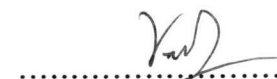
.....Chairman
(Associate Professor Supawan Tantayanon, Ph.D.)



.....Thesis Advisor
(Associate Professor Nuanphun Chantarasiri, Ph.D.)



.....Member
(Assistant Professor Warinthorn Chavasiri, Ph.D.)



.....Member
(Assistant Professor Varawut Tangpasuthadol, Ph.D.)

วิชาวิณ วรรณง : การสังเคราะห์พอลิยูรีเทน-ยูเรียที่มีสารประกอบเชิงซ้อนซิงค์และคอปเปอร์ 4,4'-ไดไฮดรอกซีซาลไซโคลเฮกเซน (SYNTHESIS OF POLYURETHANE-UREAS CONTAINING 4,4'-DIHYDROXYSALCYCLOHEXANE ZINC AND COPPER COMPLEXES) อ. ที่ปรึกษา : รศ.ดร. นवलพรรณ จันทร์ศิริ, 62 หน้า. ISBN 974-17-6484-7.

ได้สังเคราะห์สารประกอบเชิงซ้อนโลหะ 4,4'-ไดไฮดรอกซีซาลไซโคลเฮกเซน (ML₁ เมื่อ M = Mn และ Co) จากปฏิกิริยาระหว่าง 2,4-ไดไฮดรอกซีเบนซาลดีไฮด์, แมงกานีสหรือโคบอลต์แอซีเทตและเททราเอทิลีนเพนทามีน ตรวจสอบโครงสร้างของสารประกอบเชิงซ้อนดังกล่าวโดยใช้เทคนิคไออาร์สเปกโทรสโกปีและการวิเคราะห์ธาตุองค์ประกอบ พบว่าสารที่สังเคราะห์ได้เป็นของผสมและยากต่อการทำให้บริสุทธิ์ จึงได้สังเคราะห์สารประกอบเชิงซ้อนโลหะ 4,4'-ไดไฮดรอกซีซาลไซโคลเฮกเซน (ML₂ เมื่อ M = Zn และ Cu) จากปฏิกิริยาระหว่าง 2,4-ไดไฮดรอกซีเบนซาลดีไฮด์, สังกะสีหรือทองแดงแอซีเทตและ 1,2-ไดเอมีโนไซโคลเฮกเซน ตรวจสอบโครงสร้างของสารประกอบเชิงซ้อนดังกล่าวโดยใช้เทคนิคไออาร์สเปกโทรสโกปี, โพรตอนเอ็นเอ็มอาร์สเปกโทรสโกปี, แมสสเปกโทรเมตรีและการวิเคราะห์ธาตุองค์ประกอบ การสังเคราะห์พอลิยูรีเทนที่มีโลหะเป็นส่วนประกอบทำได้โดยปฏิกิริยาพอลิเมอไรเซชันระหว่างสารประกอบเชิงซ้อนโลหะประเภทเททราเดนเทตชิฟเบส (ML₂) และพรีพอลิเมอร์ชนิดต่างๆ โดยใช้ไดบิวทิลทินไดออกไซด์เป็นตัวเร่งปฏิกิริยา พรีพอลิเมอร์ที่ใช้ได้แก่ โทลิลีน 2,4-ไดไอโซไซยานาต เทอร์มินเตต พอลิ (1,4-บิวเทนไดออล) พรีพอลิเมอร์ที่มีน้ำหนักโมเลกุล 900 (PB900) และ โทลิลีน 2,4-ไดไอโซไซยานาต เทอร์มินเตต พอลิ(โพรพิลีน ไกลคอล) พรีพอลิเมอร์ที่มีน้ำหนักโมเลกุล 1,000 (PP1000) และได้สังเคราะห์พอลิยูรีเทน-ยูเรียทั้งที่มีและไม่มีโลหะในสายโซ่หลักเพื่อเปรียบเทียบสมบัติกับพอลิยูรีเทนที่สังเคราะห์ได้ ติดตามความก้าวหน้าของปฏิกิริยาด้วยไออาร์สเปกโทรสโกปีโดยพีกของหมู่ไอโซไซยานาตที่ 2275 cm⁻¹ จะไม่พบเมื่อปฏิกิริยาพอลิเมอไรเซชันเกิดขึ้นอย่างสมบูรณ์จากนั้นทดสอบสมบัติการละลายและทดสอบสมบัติการทนความร้อนของพอลิเมอร์โดยใช้วิธีเทอร์โมกราวิเมตริกอนาไลซิส

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WIPAWEE NA RANONG : SYNTHESIS OF POLYURETHANE-UREAS CONTAINING 4,4'-DIHYDROXYSALCYCLOHEXANE ZINC AND COPPER COMPLEXES. THESIS ADVISOR : ASSOC.PROF. NUANPHUN CHANTARASIRI, Ph.D., 62 pp. ISBN 974-17-6484-7.

4,4'-Dihydroxysaltetraen metal complexes (ML_1 , $M = Mn$ and Co) have been synthesized by the reaction between 2,4-dihydroxybenzaldehyde, manganese or cobalt acetate and tetraethylenepentamine. These metal complexes were characterized by IR spectroscopy and elemental analysis. It was found that the metal complexes were mixture of many products which could not be purified. Therefore, 4,4'-dihydroxysalicyclohexane metal complexes (ML_2 , $M = Zn$ and Cu) have been synthesized by the reaction between 2,4-dihydroxybenzaldehyde, zinc or copper acetate and 1,2-diaminocyclohexane. These metal complexes were characterized by IR spectroscopy, 1H NMR spectroscopy, mass spectrometry and elemental analysis. Metal-containing polyurethanes have been synthesized by the polymerization reaction between ML_2 and different prepolymers using dibutyltin dilaurate as a catalyst. The employed prepolymers were tolylene 2,4-diisocyanate terminated poly(1,4-butanediol) prepolymer with MW 900 (PB900) and tolylene 2,4-diisocyanate terminated poly(propylene glycol) prepolymer with MW 1,000 (PP1000). Polyurethane-ureas with and without metal in the main chain were also synthesized to compare the properties with synthesized metal-containing polyurethanes. The progress of polymerization reaction was followed using IR spectroscopy. The disappearance of isocyanate peak at 2275 cm^{-1} was observed when the polymerization was completed. The solubility and thermal stability by thermogravimetry of the polymers were also studied.

Department.....

Student's signature.....*Wipawee Na Ranong*.....

Field of study *Petrochemistry and Polymer Science*.....

Advisor's signature.....*Nuanphun Chantarasi*.....

Academic year.....2004.....

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LIST OF SYMBOLS AND ABBREVIATIONS

| | |
|-----------------|---|
| DBTDL | Dibutyltin dilaurate |
| DMF | Dimethylformamide |
| DMSO | Dimethylsulfoxide |
| DSC | Differential scanning calorimetry |
| EA | Elemental analysis |
| IDT | Initial decomposition temperature |
| MALDI-TOF MS | Matrix-assisted laser desorption ionization-time of flight mass spectroscopy |
| ML ₁ | 4,4'-Dihydroxysaltetraen metal complexes |
| ML ₂ | 4,4'-Dihydroxysalicyclohexane metal complexes |
| M-PB900 | Metal-containing polyurethanes based on ML ₂ and PB900 |
| M-PP1000 | Metal-containing polyurethanes based on ML ₂ and PP1000 |
| M-Xy-PB900 | Metal-containing polyurethane-ureas based on ML ₂ , <i>m</i> -xylylenediamine and PB900 |
| M-Xy-PP1000 | Metal-containing polyurethane-ureas based on ML ₂ , <i>m</i> -xylylenediamine and PP1000 |
| PB900 | Tolylene 2,4-diisocyanate terminated poly(1,4-butanediol) prepolymer |
| PP1000 | Tolylene 2,4-diisocyanate terminated poly(propylene glycol) prepolymer |
| TGA | Thermogravimetric analysis |
| Xy-PB900 | Metal-containing polyurethane-urea based on <i>m</i> -xylylenediamine and PB900 |
| Xy-PP1000 | Metal-containing polyurethane-urea based on <i>m</i> -xylylenediamine and PP1000 |