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จุฬาลงกรณ์มหาวิทยาลัย

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**SYNTHESIS OF THERMALLY STABLE POLYUREAS CONTAINING
ZINC AND NICKEL SCHIFF BASE COMPLEXES**




Miss Thussanee Mananunsap

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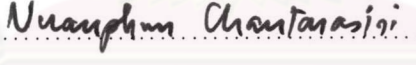
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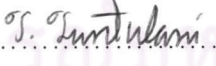

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 สังเคราะห์จากปฏิกิริยาระหว่างซาลิไซลัลดีไฮด์หรืออนุพันธ์ของซาลิไซลัลดีไฮด์, โลหะแอซิเทด
 และไตรเอทิลีนเททระเอมีนในอัตราส่วนโมล 2:1:1 ส่วนสารประกอบเชิงซ้อนของโลหะสังกะสี
 เรียเตรียมจากปฏิกิริยาระหว่างสารประกอบเชิงซ้อนซิงค์เบสสังกะสีและไอโซไซยานาต เพื่อศึกษา
 ความว่องไวของหมู่เอมีนบนสารประกอบเชิงซ้อนซิงค์เบสสังกะสี การทดสอบความเป็นผลึกเหลว
 ของสารประกอบเชิงซ้อนซิงค์เบสของโลหะและสารประกอบเชิงซ้อนของโลหะสังกะสียูเรียทำได้
 โดยใช้เทคนิคดีฟเฟอเรนเชียลสแกนนิ่งแคลอริเมตรี พบว่าสารประกอบเชิงซ้อนเหล่านี้ไม่แสดงสมบัติ
 ผลึกเหลว แต่ทนความร้อนได้ดี จึงนำมาใช้ในการสังเคราะห์พอลิยูเรีย ซึ่งทำได้โดยปฏิกิริยาพอลิ
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 หรือ 4,4-เมทิลีนบิสฟีนิลไอโซไซยานาต (MDI) ที่อัตราส่วนโมล 1:1 และติดตามความก้าวหน้า
 ของปฏิกิริยาพอลิเมอไรเซชันทำได้โดยใช้อินฟราเรดสเปกโทรสโกปี โดยพีคของหมู่ไอโซไซยา
 นาตที่ 2270 cm^{-1} จะหายไป เมื่อปฏิกิริยาพอลิเมอไรเซชันเกิดขึ้นอย่างสมบูรณ์ การพิสูจน์เอกลักษณ์
 พอลิยูเรียที่มีโลหะเป็นส่วนประกอบทำได้โดยใช้เทคนิคอินฟราเรดสเปกโทรสโกปี, เอ็นเอ็มอาร์ส
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 ความร้อนตรวจสอบได้ด้วยเทอร์โมกราวิเมตริกอนาลิซิส (TGA) และลิมิตดิงออกซิเจนอินเดกซ์
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 จุฬาลงกรณ์มหาวิทยาลัย

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Hexadentate Schiff base zinc (II) and nickel (II) complexes were synthesized from the reaction between salicylaldehyde or salicylaldehyde derivatives, metal (II) acetate and triethylenetetramine at the mole ratio of 2:1:1. The reaction between Schiff base zinc complexes and isocyanates to yield zinc-complexes ureas were carried out to studied the reactivity of amine group in Schiff base zinc complexes. The liquid crystalline property of Schiff base metal-complexes and metal-complexes ureas were investigated by differential scanning calorimetry (DSC). It was found that these complexes did not show liquid crystalline property. They exhibited good thermal stability and therefore used in the synthesis of metal-containing polyureas which were done by the polymerization reaction between Schiff base metal-complexes and hexamethylene diisocyanate (HDI) or 4,4'-methylenebis(phenyl isocyanate) (MDI) at a 1:1 mole ratio. The progress of polymerization reaction was followed by using infrared spectroscopy. The disappearance of isocyanate peak at 2270 cm^{-1} was observed when the polymerization was completed. Metal-containing polyureas were characterized by FTIR, NMR, elemental analysis, solubility and viscometry. Heat resistance of polymers was investigated by thermogravimetric analysis (TGA) and measuring limiting oxygen index (LOI) values. It was found that the polymers showed good thermal stability.

Department.....Chemistry..... Student's signature.....*Thussanee Mananunsap*
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LIST OF SYMBOLS AND ABBREVIATION

| | |
|-----------------------------------|--|
| DSC | Differential scanning calorimetry |
| EA | Elemental analysis |
| FAB MS | Fast atom bombardment mass spectroscopy |
| HDI | Hexamethylene diisocyanate |
| IDT | Initial decomposition temperature |
| LOI | Limiting oxygen index |
| MDI | 4,4'-methylenebis(phenyl isocyanate) |
| M(Sal) ₂ trien | Hexadentate Schiff base metal complexes based on salicylaldehyde |
| M(XSal) ₂ trien | Hexadentate Schiff base metal complexes based on salicylaldehyde derivatives |
| M(Sal) ₂ trien-HDI | Metal-containing polyureas based on HDI and M(Sal) ₂ trien |
| M(XSal) ₂ trien-HDI | Metal-containing polyureas based on HDI and M(XSal) ₂ trien |
| M(Sal) ₂ trien-MDI | Metal-containing polyureas based on MDI and M(Sal) ₂ trien |
| M(XSal) ₂ trien-MDI | Metal-containing polyureas based on MDI and M(XSal) ₂ trien |
| POM | Polarizing optical microscope |
| TDI | 2,4-Toluene diisocyanate |
| TGA | Thermogravimetric analysis |
| Zn(Sal) ₂ trien ureas | Hexadentate Schiff base zinc urea derivatives based on Zn(Sal) ₂ trien |
| Zn(XSal) ₂ trien ureas | Hexadentate Schiff base zinc urea derivatives based on Zn(XSal) ₂ trien |