

**SYNTHESIS OF SPIROSILICATE/BENZOXAZINE  
COMONOMERS**

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**Program:** Polymer Science  
**Thesis Advisors:** Prof. Hatsuo Ishida  
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## ABSTRACT

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Benzoxazine, a novel phenolic resin, undergoes polymerization via ring-opening polymerization. Polybenzoxazine shows good properties, such as no production of by-products upon curing, and exhibition of near-zero shrinkage, whereas spirosilicate compounds, obtained by reacting silica with ethylene glycol or ethylene glycol derivatives, also show a near zero shrinkage. The spirosilicate/benzoxazine comonomers were synthesized to obtain the materials which are expected to have a near zero shrinkage property. The new benzoxazines were synthesized from protecting hydroxyl groups of 3-amino-1,2-propanediol or 2-amino-2-methyl-1,3-propanediol by using cyclohexanone to form ketal products. Then ketals were reacted with formaldehyde and phenol to get benzoxazines. The spirosilicate/benzoxazine comonomers were synthesized as crude product. The structures of products were confirmed using FTIR, MS,  $^1\text{H}$ -,  $^{13}\text{C}$ -, and  $^{29}\text{Si}$ -NMR.

## บทคัดย่อ

นางสาวสุภัทรา ประทุมเมฆ: การสังเคราะห์โคมอนอเมอร์จากสไปโรซิลิเกตและเบนซอกซาซีน (Synthesis of Spirosilicate/benzoxazine Comonomers) อ. ที่ปรึกษา: ศ. ดร. ฮัตสึโอะ อิซิดะ และ รศ. ดร. สุจิตรา วงศ์เกษมจิตต์ 43 หน้า ISBN 974-13-0721-7

เบนซอกซาซีนซึ่งเป็นพีนอลลิเกรซิ่นตัวใหม่มีการพอลิเมอร์ไรเซชันแบบเปิดวงของออกซาซีน พอลิเบนซอกซาซีนแสดงสมบัติที่ดีหลายประการ เช่น ไม่ผลิตสารข้างเคียงขณะการขึ้นรูป และแสดงสมบัติในการหดตัวเล็กน้อย ในขณะที่สารประกอบสไปโรซิลิเกตซึ่งได้จากปฏิกิริยาของซิลิกากับเอริสินไกลคอล หรืออนุพันธ์ของเอริสินไกลคอลแสดงการหดตัวใกล้เคียงศูนย์เช่นกัน สารประกอบสไปโรซิลิเกต/เบนซอกซาซีนโคมอนอเมอร์ถูกสังเคราะห์ขึ้นเพื่อให้ได้วัสดุซึ่งคาดว่าจะมีสมบัติการหดตัวใกล้เคียงศูนย์ โดยสังเคราะห์จากการปกป้องหมู่ไฮดรอกซิลของ 3-อะมิโน-1,2-โพรเพนไดออล หรือ 2-อะมิโน-2-เมทิล-1,3-โพรเพนไดออล โดยใช้ไซโคลเฮกซาโนนเพื่อสร้างผลิตภัณฑ์คีทาล จากนั้นคีทาลทำปฏิกิริยากับพอมัลดีไฮด์และพีนอลเพื่อให้ได้เบนซอกซาซีน/สไปโรซิลิเกตโคมอนอเมอร์ โครงสร้างของผลิตภัณฑ์ถูกตรวจสอบโดยใช้เอฟทีไออาร์, แมสสเปกโตรสโคปี, โพรตอน คาร์บอนและซิลิกอนเอ็นเอ็มอาร์

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