

**ONE STEP SYNTHESIS OF A NOVEL COPOLYMER DIRECTLY  
FROM SiO<sub>2</sub>, CATECHOL, HYDROQUINONE, AND  
4-tert-BUTYLCATECHOL**

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A Thesis Submitted in Partial Fulfillment of the Requirements  
for the Degree of Master of Science  
The Petroleum and Petrochemical College, Chulalongkorn University  
in Academic Partnership with  
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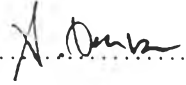
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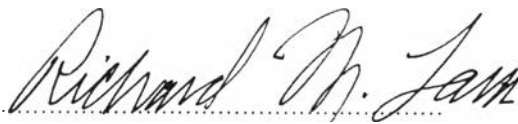
**Thesis Title** : One Step Synthesis of a Novel Copolymer Directly from  
SiO<sub>2</sub>, Catechol, Hydroquinone, and 4-tert-Butylcatechol  
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**Thesis Advisors** : Assoc. Prof. Richard M. Laine  
Asst. Prof. Sujitra Wongkasemjit

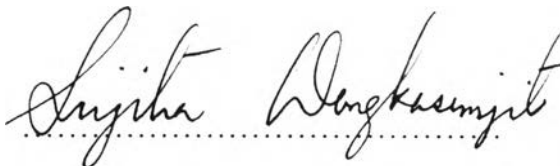
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Accepted by the Petroleum and Petrochemical College, Chulalongkorn University, in partial fulfillment of the requirements for the Degree of Master of Science.

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## ABSTRACT

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KEYWORDS : Copolymer / OOPS process

Wirat Suttavireesan : Thesis title - One Step Synthesis of a Novel Copolymer Directly from SiO<sub>2</sub>, Catechol, Hydroquinone, and 4-tert-Butylcatechol. Thesis Advisors : Assoc. Prof. Richard M. Laine and Asst. Prof. Sujitra Wongkasemjit, 62 pp. ISBN 974-638-518-6

A synthesis method of a new copolymer containing organic ligand and silicon atoms was developed via the one step process called “OOPS process” directly from SiO<sub>2</sub>, catechol, hydroquinone, and 4-tert-butylcatechol using triethylenetetramine (TETA) and ethylene glycol (EG) as catalyst and solvent, respectively. Effect of variation of hydroquinone and 4-tert-butylcatechol ratio, catalyst concentration on the product structure was investigated using fourier transform-infrared spectroscopy (FTIR), nuclear magnetic resonance (NMR), X-ray diffraction (XRD), and electron impact-mass spectroscopy (EI<sup>+</sup>-MS). Thermal stability of the copolymer product was studied using thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC) under O<sub>2</sub>/N<sub>2</sub> atmospheres. Decomposition of the product occurred when the temperature was higher than 250°C. Kinetic study by the initial-rate method showed that the reaction was first order with respect to SiO<sub>2</sub> and its activation energy was 163.5 cal/mol with 100 mole % TETA at an appropriate mole ratio of starting materials.

## บทคัดย่อ

วิรัช สุทธีวิสิทธ์ : การสังเคราะห์โคพอลิเมอร์ตัวใหม่ในขั้นตอนเดียวโดยตรงจาก ซิลิกอนไดออกไซด์ แคททิคอล ไฮโดรควิโนน และ 4-เทอร์เชียรี-บิวทิวแคททิคอล (One Step Synthesis of a Novel Copolymer Directly from SiO<sub>2</sub>, Catechol, Hydroquinone, and 4-tert-Butylcatechol) อ.ที่ปรึกษา: รศ. ดร. ริชาร์ด เลน (Assoc. Prof. Richard M. Laine) และ ผศ. ดร. สุจิตรา วงศ์เกษมจิตต์ 62 หน้า ISBN 974-638-518-6

วิธีการสังเคราะห์สารโคพอลิเมอร์ร่วมที่มีสารอินทรีย์และซิลิกาเป็นองค์ประกอบตัวใหม่ได้มีการพัฒนา โดยกระบวนการสังเคราะห์สารประกอบออกไซด์ในขั้นตอนเดียวที่เรียกว่า OOPS process จาก ซิลิกอนไดออกไซด์ แคททิคอล ไฮโดรควิโนน และ 4-เทอร์เชียรี-บิวทิวแคททิคอล โดยมีไตรเอทิลีนเตตระมีนเป็นสารตัวเร่งปฏิกิริยาในตัวทำละลายเอทิลีนไกลคอล ผลของการเปลี่ยนแปลงสัดส่วนของมอนอเมอร์ร่วมระหว่างไฮโดรควิโนน และ 4-เทอร์เชียรี-บิวทิวแคททิคอล และปริมาณสารตัวเร่งที่ใช้ในปฏิกิริยา ได้ถูกศึกษาและตรวจสอบโครงสร้างโดยใช้เทคนิควิเคราะห์ ได้แก่ FTIR, NMR, XRD และ EI<sup>+</sup>-MS เสถียรภาพในทางด้านความร้อนของสารโคพอลิเมอร์ได้ถูกศึกษาด้วยเทคนิคการวิเคราะห์ TGA และ DSC ภายใต้บรรยากาศของก๊าซไนโตรเจนและออกซิเจน ซึ่งพบว่าสมบัติการสลายตัวของสารจะเกิดขึ้นเมื่อได้รับความร้อนเกินกว่า 250 องศาเซลเซียส การศึกษาจลนศาสตร์ตามวิธีอัตราเริ่มต้นได้แสดงให้เห็นว่าซิลิกามีอันดับปฏิกิริยาเป็นอันดับหนึ่งและให้ค่าพลังงานกระตุ้นเท่ากับ 163.5 แคลอรีต่อโมล สำหรับปฏิกิริยาที่ใช้ปริมาณสารตัวเร่ง 100 โมล เปอร์เซ็นต์ที่สัดส่วน โมล ของสารตั้งต้นที่เหมาะสม

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