

**PURIFICATION OF SINGLE WALLED CARBON NANOTUBES:  
REMOVAL OF SILICA BY TRIETHYLENETETRAMINE  
AND SODIUM HYDROXIDE**

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## ABSTRACT

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The catalytic decomposition of carbon-containing molecules is believed to be the pathway for large-scale production of Single Walled Carbon Nanotubes (SWNTs). This technique utilizes metal deposited on a support such as silica. Therefore, the purification step is very important in the commercial production of SWNTs. In this work, the removal of silica was studied by using either triethylenetetramine (TETA) or sodium hydroxide (NaOH) prior to froth flotation. From the dissolution study of silica, over 99% of silica dissolution was obtained by using 50 mL of 1 M NaOH at 70°C, whereas it required 250 mL of TETA to dissolve the same amount of silica at 70°C. For the purification study of AP-SWNTs, two steps of pre-treatment and froth flotation were investigated. Alcohol ethoxylate (surfonic L 24-7), nonionic surfactant, was used as a foam booster in froth flotation operation. After the froth flotation operation, the maximum purity of carbon in the collapsed foam was 55.67% and 72.74% with TETA and NaOH pre-treatment, respectively. From the Raman spectra, both of the NaOH and TETA pre-treatment with froth flotation do not destroy the structure of SWNTs.

## บทคัดย่อ

เกวลิน เกียรติสูงชาติ : การทำให้คาร์บอนนาโนทิวป์ประเภทผนังชั้นเดียวให้บริสุทธิ์ โดยการกำจัดซิลิกาด้วย ไตรเอทิลีนเตตระเอมีน และโซเดียมไฮดรอกไซด์ (Purification of Single Walled Carbon Nanotubes: Removal of Silica by Triethylenetetramine and Sodium Hydroxide) อ. ที่ปรึกษา : รศ. ดร. สุเมธ ชวเวช และผศ. ดร. บุญยรัชต์ กิตขันธ์ 81 หน้า ISBN 974-9651-94-4

คาร์บอนนาโนทิวป์ประเภทผนังเดี่ยว (Single Walled Carbon Nanotubes) แสดงสมบัติที่โดดเด่นทั้งทางเชิงกล และไฟฟ้า ซึ่งสามารถนำไปใช้งานที่หลากหลาย กระบวนการสังเคราะห์คาร์บอนนาโนทิวป์ประเภทผนังเดี่ยวที่ให้ปริมาณสูงนั้น คือการสลายตัวโดยใช้ตัวเร่งปฏิกิริยาของก๊าซที่มีคาร์บอน ผลผลิตนั้น ไม่ได้มีเพียงคาร์บอนนาโนทิวป์ประเภทผนังเดี่ยวเท่านั้น แต่ยังมีสารอื่นๆ ได้แก่ ตัวเร่งปฏิกิริยา (Catalyst) และวัสดุที่ใช้เป็นฐาน (Support Material) ที่เป็นองค์ประกอบที่มีปริมาณมากที่สุด โดยที่สารอื่นๆเหล่านี้ ทำให้คุณสมบัติของคาร์บอนนาโนทิวป์ประเภทผนังเดี่ยวต่ำลง ด้วยเหตุนี้กระบวนการทำให้คาร์บอนนาโนทิวป์ประเภทผนังเดี่ยวบริสุทธิ์จึงเป็นเรื่องที่น่าสนใจเป็นอย่างยิ่ง ในการวิจัยครั้งนี้ได้มุ่งในการกำจัดซิลิกาที่เป็นฐาน โดยใช้ไตรเอทิลีนเตตระเอมีน และโซเดียมไฮดรอกไซด์ในการกำจัด หลังจากนั้นได้ใช้กระบวนการ Froth Flotation เพื่อเพิ่มความเข้มข้นของคาร์บอนนาโนทิวป์ประเภทผนังเดี่ยวบริสุทธิ์ ความเข้มข้นของโซเดียมไฮดรอกไซด์, ปริมาณไตรเอทิลีนเตตระเอมีน, อุณหภูมิ และระยะเวลาในการเกิดปฏิกิริยาได้ถูกนำมาศึกษา โดยพบว่า 99% ของซิลิกาที่ใช้ ละลายที่ 1 โมลาร์ของโซเดียมไฮดรอกไซด์ ปริมาตร 50 mL ที่ 70°C ขณะที่ไตรเอทิลีนเตตระเอมีนต้องใช้ในปริมาณ 250 มิลลิลิตร ที่ 70°C ในการทำให้คาร์บอนนาโนทิวป์ประเภทผนังเดี่ยวบริสุทธิ์ ขั้นตอนของการกำจัดซิลิกา และกระบวนการทำให้เป็นฟองลอย (Froth flotation) ถูกศึกษา Alcohol ethoxylate (surfonic L24-7) เป็นสารลดแรงตึงผิวประเภทไม่มีขั้ว ถูกนำมาใช้ในฐานะเพิ่มฟองในกระบวนการนี้ ภายหลังจากกระบวนการทำให้เป็นฟองลอย ปริมาณความบริสุทธิ์ของคาร์บอนสูงสุดที่ 55.67เปอร์เซ็นต์ และ 72.74เปอร์เซ็นต์ โดยใช้ ไตรเอทิลีนเตตระเอมีน และโซเดียมไฮดรอกไซด์ ตามลำดับ จากสเปคตรัมรามานของทั้งโซเดียมไฮดรอกไซด์ และไตรเอทิลีนเตตระเอมีนนั้น ไม่ทำลายโครงสร้างของคาร์บอนนาโนทิวป์ประเภทผนังเดี่ยว

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