

**PREPARATION OF NANO METAL OXIDE USING NOVEL METAL  
ALKOXIDES**

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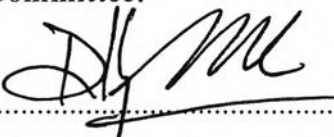
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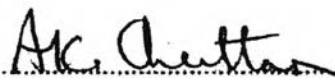
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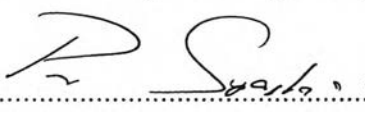
  
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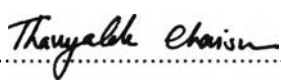
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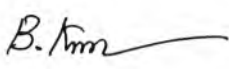
  
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## ABSTRACT

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Ultra-fine composite fibers made from (PVA)/silatrane and (PVA)/tin glycolate were prepared by a combined sol-gel and electrospinning technique. PVA/silatrane composites calcined at 500°C and  $\geq 700^\circ\text{C}$  were converted to amorphous silica and cristobalite fibers, respectively. For (PVA)/tin glycolate composite, the acidity of spinning solution plays an important role to the morphology and the size of the obtained fibers. It was found that the ultrafine tin oxide fiber obtained from calcination at 600°C showed high conductivity value of  $1.59 \times 10^3 \text{ Scm}^{-1}$  and the high surface area of about  $275 \text{ m}^2/\text{g}$ . The effect of calcinations temperature on the phase and the size of both silica fiber and tin oxide fibers were investigated in this study.

Hybridizing carbon nanotubes (CNTs) with complex inorganic nanostructures of titanium-silicate (TS-1) provides a new route to designing next-generation photocatalysts. The hybrids were synthesized via a microwave-assisted solvothermal route, using titanium glycolate and silatrane as Ti and Si sources, with the aid of benzyl alcohol as a linking agent. The photocatalytic performance was tested for the degradation of 4-nitrophenol and rhodamine B under UV light as well as visible light. The hybrids showed up to 5 times higher photocatalytic activities compared with the corresponding nano-composite and the individual components, as well as increased selectivity towards total degradation via ring cleavage. For the CNT itself, the hydrophobicity of the CNT surface disfavours the adsorption of hydrophilic particles, thus limiting the quality and performance of the hybrid material. Herein, we

demonstrated that using benzyl alcohol as a surfactant enables SiO<sub>2</sub> to interact with the hydrophobic surface of pristine CNTs without the need of covalent functionalisation. The quality of the SiO<sub>2</sub> coating is strongly affected by various reaction conditions, including the order of mixing, the presence of benzyl alcohol, and the reaction temperature. The effect of heat treatment on the crystallisation of amorphous SiO<sub>2</sub> to cristobalite is discussed in detail. The key achievement is the well-control of morphology and the structure of the SiO<sub>2</sub> coating and, after removal of the CNTs, of the cristobalite nanotubes, allowing the production of the silica structure with a high surface area-to-mass ratio.

## บทคัดย่อ

เมธีรา กฤษณะเสรณี: การเตรียมโลหะออกไซด์ระดับนาโนจากโลหะแอลกอฮอล์ชนิดใหม่ (Preparation of Nano Oxide via Novel Metal Alkoxides) อ. ที่ปรึกษา: รองศาสตราจารย์ ดร. สุจิตรา วงศ์เกษมจิตต์และศาสตราจารย์แอนโทนี่ เค. ซิทแทม 131 หน้า

สารประกอบเส้นใยขนาดเล็กพิเศษของโพลีไวนิลแอลกอฮอล์/ไซลาเทรน และ โพลีไวนิลแอลกอฮอล์/ไททานเนียมไกลคอลเลท ได้ถูกเตรียมขึ้นโดยผสมผสานกระบวนการโซล-เจลและกระบวนการปั่นเส้นใยจากไฟฟ้า สารประกอบโพลีไวนิลแอลกอฮอล์/ไซลาเทรนที่ได้รับจากการเผาที่อุณหภูมิ 500 องศาเซลเซียสและอุณหภูมิมากกว่าหรือเท่ากับ 700 องศาเซลเซียสได้ถูกแปรสภาพไปเป็นเส้นใยซิลิกาอสัณฐานและเส้นใยคริสโตบาลิทตามลำดับ สำหรับโพลีไวนิลแอลกอฮอล์/ไททานเนียมไกลคอลเลท ความเป็นกรดของสารละลายมีบทบาทสำคัญต่อรูปสัณฐานและขนาดของเส้นใย เส้นใยทินออกไซด์ที่ได้จากการเผาที่อุณหภูมิ 600 องศาเซลเซียส แสดงค่าการนำไฟฟ้าสูงถึง  $1.59 \times 10^3$  ซีเมนต์ต่อเซนติเมตร และมีพื้นที่ผิวสูงถึง 275 ตารางเมตรต่อกรัม งานวิจัยนี้ยังได้ศึกษาผลของอุณหภูมิการเผาต่อเฟสและขนาดเส้นใยของทั้งเส้นใยซิลิกาและเส้นใยทินออกไซด์

สารผสมไฮบริดของท่อนาโนคาร์บอนกับสารอนินทรีย์ไททานเนียมซิลิเกต (ทีเอส-วัน) ที่มีโครงสร้างระดับนาโนให้แนวทางในการออกแบบนวัตกรรมรุ่นใหม่ของการเร่งปฏิกิริยาด้วยแสง ด้วยการสังเคราะห์ภายใต้คลื่นไมโครเวฟ โดยใช้ไททานเนียมไกลคอลเลทและไซลาเทรนเป็นสารตั้งต้นของไททานเนียมและซิลิกา ตามลำดับ และใช้เบนซิลแอลกอฮอล์เป็นสารตัวเชื่อมประสิทธิภาพของความเป็นตัวเร่งปฏิกิริยาโดยใช้แสงได้ถูกทดสอบโดยการติดตามการสลายตัวของ 4-ไนโตรฟีนอลและโรดามีน บี ภายใต้คลื่นแสงยูวีและแสงที่มองเห็นได้ด้วยตาเปล่า จากการทดลองพบว่า สารผสมไฮบริดแสดงประสิทธิภาพที่ดีกว่าถึง 5 เท่า เมื่อเทียบกับสารผสมเชิงกายภาพและสารองค์ประกอบแต่ละตัวที่ใช้เป็นสารตั้งต้นในการสังเคราะห์สารผสมไฮบริด รวมทั้งเพิ่มความสามารถในการเลือกจำเพาะของการสลายตัวจากการแตกวง สำหรับคาร์บอนที่มีลักษณะเป็นท่อระดับนาโนนั้น ความไม่ชอบน้ำบริเวณผิวของท่อคาร์บอน ไม่อำนวยความสะดวกดูดซับอนุภาคของสารที่ชอบน้ำ จึงเป็นข้อจำกัดต่อประสิทธิภาพของสารผสมไฮบริด ในงานวิจัยนี้ จากผลทดลองพบว่าการใช้เบนซิลแอลกอฮอล์เป็นสารลดแรงตึงผิว สามารถช่วยให้ซิลิกอนไดออกไซด์ทำปฏิกิริยากับผิวส่วนที่ไม่ชอบน้ำของท่อคาร์บอนโดยไม่ต้องอาศัยการสร้างพันธะโควาเลนต์ คุณภาพของซิลิกอนไดออกไซด์ที่เคลือบผิวนั้น ขึ้นกับสถานะต่างๆในการทำปฏิกิริยา เช่น ลำดับของการผสม การมีอยู่ของเบนซิลแอลกอฮอล์ และอุณหภูมิที่ใช้ในการทำ

ปฏิกิริยา ผลของการให้ความร้อนต่อการตกผลึกของซิลิกาอสัณฐานไปเป็นสถานะคริสโตบาไลต์ จะมีการอภิปรายในรายละเอียด เป้าหมายสำคัญของงานวิจัยนี้คือความสามารถในการควบคุมรูป สัณฐานและ โครงสร้างของซิลิกอนไดออกไซด์ที่เคลือบบริเวณผิวของท่อนาโนคาร์บอนก่อนเผา และ โครงสร้างคริสโตบาไลต์หลังจากเผาท่อนาโนคาร์บอนออก ซึ่งทำให้สามารถผลิต โครงสร้างซิลิกาที่มีค่าอัตราส่วนของพื้นที่ผิวต่อน้ำหนักที่สูง



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**ABBREVIATIONS**

OOPS	Oxide One Pot Synthesis
TEOS	Tetraethoxysilane
TEA	Triethanolamine
EG	Ethylene Glycol
TETA	Triethylenetetramine
PVA	Poly (vinyl alcohol)
CNTs	Carbon nanotubes
TS-1	Titanium Silicate-1
BA	Benzyl alcohol
4-NP	4-Nitrophenol
Rh-B	Rhodamine B
FT-IR	Fourier Transform Infrared Spectroscopy
TGA	Thermogravimetric Analysis
XRD	X-ray Diffractometer
SEM	Scanning Electron Microscope
BET	Brunaues-Emmett-Teller
DRUV	Diffuse Reflectance Ultraviolet Spectroscopy
EDS	Energy Dispersive Spectroscopy