

**SYNTHESIS AND APPLICATION OF SBA-1 MESOPOROUS SILICA BY
ATRANE ROUTE VIA SOL-GEL PROCESS**

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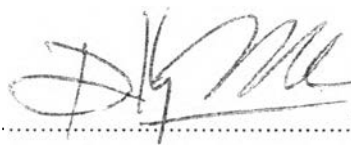
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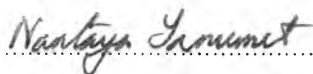
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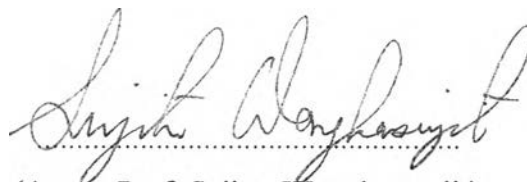


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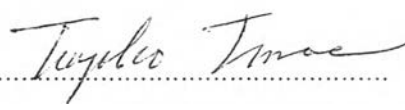
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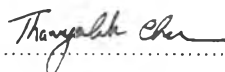
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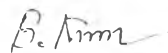
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ABSTRACT

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Silatrane was used as a precursor for SBA-1 synthesis at room temperature using alkyltrimethylammonium bromides, C_n TMAB ($n = 14-18$), as templates in dilute solutions. The influences of acidity, alkyl chain length of the surfactant and synthesis temperature were studied. At high surfactant concentration and elevated reaction temperature, three-dimensionally ordered mesopores were invariably produced. The surface area of the product was as high as 1000–1500 m^2/g with an adsorption volume of 0.6 – 1.0 cm^3/g . When using a poly(amido amine) dendrimer porogen as a template under mild condition, two kinds of crystal arrays were found to be produced in the silica particles. These results indicate that one molecule of silatrane constructed polysiloxane wall in the first array and oligomeric silatranes formed wall in the second array. It was referred that the intrinsic (hydrogen bonding) character of hydroxyl groups in the hydrolyzed silatrane resulted in hydrogen bonded oligomers and reinforced the hydrogen bonding interaction with dendrimer porogen as well as electrostatic interaction, giving rise to two types of template arrays. The mesoporous SBA-1 was also used as a catalyst support with 6wt% Fe and 10wt% Ti without perturbing the ordered mesoporous. The catalytic activity and selectivity of styrene epoxidation using hydrogen peroxide were studied, and showed that the selectivities of benzaldehyde and styrene oxide reached 74 and 11%, respectively, at styrene conversion of 67% over 4%Fe in SBA-1 while those of styrene oxide and benzaldehyde reached 48 and 50%, respectively, at styrene conversion of 69% over 2%Ti in SBA-1.

บทคัดย่อ

พริสา ตั้งล้ำเลิศ : การสังเคราะห์และการประยุกต์สารรองรับ SBA-1 โดยใช้กระบวนการโซล-เจลของสารเอเทรอน (Synthesis and Application of SBA-1 Mesoporous Silica by Atrane Route via Sol-Gel Process) อ. ที่ปรึกษา : รองศาสตราจารย์ ดร. สุจิตรา วงศ์เกษมจิตต์ รองศาสตราจารย์ ดร. ทิโมที จอห์น ไวท์ ศาสตราจารย์ ดร. โทโยโกะ อิมามะ 91 หน้า

ไซลาเทรอนที่สังเคราะห์จากซิลิกาและไตรเอธานอลามีน ถูกใช้เป็นสารตั้งต้นในการสังเคราะห์สารรองรับ SBA-1 ที่อุณหภูมิห้องโดยใช้สารลดแรงตึงผิวชนิดประจุบวก ได้แก่ CnTMAB ($n = 14-18$) เป็นสารต้นแบบ ในสถานะที่เป็นกรด โดยได้ศึกษาปัจจัยต่างๆ ได้แก่ ความเข้มข้นของกรด ความยาวของสายโซ่ ความเข้มข้นของสารลดแรงตึงผิว และอุณหภูมิของระบบ ที่มีผลต่อโครงสร้างของสารรองรับ SBA-1 จากการทดลองพบว่า ที่ความเข้มข้นของสารลดแรงตึงผิว และที่อุณหภูมิสูง สารที่ได้ยังคงมีโครงสร้างลักษณะรูพรุนแบบลูกบาศก์สามมิติ ตัวรองรับที่สังเคราะห์ได้มีพื้นที่ผิวสูงถึง 1000 – 1500 ตารางเมตรต่อกรัม และมีปริมาตรรูพรุน 0.6–1 ตารางเซนติเมตรต่อกรัม เมื่อเปลี่ยนสารต้นแบบเป็นพอลิ(อะมิโดเอมีน)เดนดริเมอร์ในสถานะที่เหมาะสมพบว่า มีโครงสร้างลักษณะรูพรุน 2 แบบภายในอนุภาคของซิลิกานั้น ซึ่งเป็นผลมาจากการสร้างผนังพอลิไซลอคเซนของโมเลกุลไซลาเทรอน และการสร้างผนังโอลิโกเมอริกของโมเลกุลไซลาเทรอน นอกจากนี้ ยังได้นำเอา SBA-1 มาเป็นสารรองรับโลหะทรานซิชันไททานเนียมและเหล็ก ในสถานะที่เหมาะสม SBA-1 สามารถรองรับโลหะเหล็ก ได้มากถึง 6% และรองรับโลหะไททานเนียมได้มากถึง 10% โดยที่ยังคงโครงสร้างของ SBA-1 อยู่ เพื่อนำไปศึกษาประสิทธิภาพของความเป็นตัวเร่งปฏิกิริยา ในปฏิกิริยาอีพอกซิเดชันของสไตรีน ด้วยไฮโดรเจนเปอร์ออกไซด์ จากการทดลองพบว่า ตัวเร่งปฏิกิริยามีประสิทธิภาพดี สามารถเปลี่ยนสไตรีนเป็นสไตรีนออกไซด์ได้ถึง 67 และ 69% โดยความสามารถในการเลือกจำเพาะของ สไตรีน ออกไซด์ เป็น 11 และ 48% ตามลำดับ เมื่อใช้ตัวเร่งปฏิกิริยาที่มีเหล็กอยู่ 4% และไททานเนียมอยู่ 2% ตามลำดับ.

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ABBREVIATIONS

OOPS	Oxide One Pot Synthesis
TEOS	Tetraethoxysilane
TEA	Triethanolamine
EG	Ethylene Glycol
CTMAB	Cetyltrimethylammonium Bromide
TETA	Triethylenetetramine
PAMAM	Poly(amido amine) dendrimer
FT-IR	Fourier Transform Infrared Spectroscopy
TGA	Thermogravimetric Analysis
XRD	X-ray Diffractometer
SEM	Scanning Electron Microscope
TEM	Transmission Electron Microscope
BET	Brunaues-Emmett-Teller
DRUV	Diffuse Reflectance Ultraviolet Spectroscopy
ESR	Electron Spin Resonance