

**SYNTHESIS AND APPLICATIONS OF M-MCM-48 (M = Ti, Ce, AND Cr)  
FROM SILATRANE PRECURSOR**

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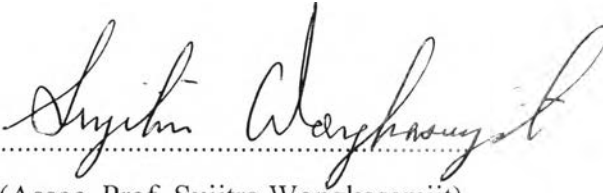
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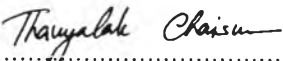
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## ABSTRACT

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Hydrothermal synthesis of cubic MCM-48 was carried out using silatrane as a silica precursor and CTAB as a structure directing agent. The effects of synthesis parameters, viz. crystallization temperature, crystallization time, surfactant concentration, quantity of NaOH, and silica source, on the product structure were investigated. To examine the properties of the synthesized materials, various techniques were introduced, viz. X-ray diffraction (XRD), nitrogen adsorption-desorption measurement, field emission scanning electron microscopy (FE-SEM), transmission electron microscopy (TEM), and surface area analysis. A high specific surface area and narrow pore size distribution were obtained. The materials showed a truncated octahedral shape and cubic  $Im\bar{3}d$  symmetry of pore structure. To improve the catalytic property of mesoporous silica the incorporation of metals (M =Cr, Ce and Ti) was studied, using the optimal synthesis of MCM-48 conditions. Both framework and extraframework of the metals were investigated by diffuse reflectance UV-visible spectroscopy (DR-UV). The catalytic performance of all materials was performed on the oxidation of styrene using hydrogen peroxide as an oxidant. The optimal conditions were studied as a function of reaction time, reaction temperature, ratio of oxidant/styrene, amount of catalyst, and metal content. The % conversion of styrene and the % selectivity of products depended on the nature of the metal loading. Additionally, the kinetic study of Cr-MCM-48 catalysts toward the styrene oxidation was also investigated.

## บทคัดย่อ

รุจิรัตน์ ล่องลอยเลิศ: การสังเคราะห์ และการประยุกต์ M-MCM-48 (M = Ti, Ce, and Cr) จากสารตั้งต้น ซิลาเทรน (Synthesis and Applications of M-MCM-48 (M = Ti, Ce, and Cr) from Silatrane Precursor) อ. ที่ปรึกษา: รองศาสตราจารย์ ดร. สุจิตรา วงศ์เกษมจิตต์ 111 หน้า

มีการศึกษาการสังเคราะห์ MCM-48 ที่มีลักษณะโครงสร้างแบบลูกเต๋าด้วยกระบวนการให้ความร้อนโดยใช้ซิลาเทรนเป็นสารตั้งต้นซิลิกา และใช้ CTAB เป็นสารแม่แบบ ผลกระทบของตัวแปรต่างๆมีต่อการสังเคราะห์และโครงสร้างของผลิตภัณฑ์ ได้แก่ อุณหภูมิ และเวลาที่ใช้ในการสร้างเป็นผลึก, ความเข้มข้นของสารลดแรงตึงผิว, ปริมาณ โซเดียมไฮดรอกไซด์ และชนิดของซิลิกา การศึกษาสมบัติต่างๆของวัสดุที่สังเคราะห์ได้นั้น ใช้เทคนิคการวิเคราะห์ต่างๆ เช่น เอ็กซ์เรย์ดิฟแฟรกชัน, เครื่องมือวิเคราะห์พื้นที่ผิว และกล้องจุลทรรศน์อิเล็กตรอนทั้งแบบส่องผ่านและแบบส่องกราด ซึ่งพบว่าสารมีพื้นที่ผิวสูงและการกระจายตัวของขนาดรูพรุนน้อย อีกทั้งยังพบลักษณะรูปร่างเป็นแบบ truncated octahedral และลักษณะโครงสร้างของรูพรุนสมมาตรแบบคิวบิกชนิด  $1a3d$  การเพิ่มโลหะต่างๆ เข้าไปในโครงสร้างซิลิกาที่มีรูพรุนขนาดกลาง นับว่าเป็นสิ่งจำเป็นสำหรับการปรับปรุงสมบัติของสารในการเป็นตัวเร่งปฏิกิริยา ในการศึกษานี้ได้ใส่โลหะโครเมียม, ซีเรียม, และ ทาแทนียม เพิ่มเข้าไปในวัสดุ MCM-48 โดยใช้สภาวะที่เหมาะสมจากการสังเคราะห์ MCM-48 มาใช้ และศึกษาปริมาณของโลหะที่ใส่เพิ่มเข้าไปอีกด้วย นอกจากนี้มีการพิสูจน์เอกลักษณ์ทางโครงสร้างของโลหะในซิลิกา ทั้งที่อยู่ในโครงสร้างและนอกโครงสร้างโดยใช้เครื่องวิเคราะห์ตัวอย่างที่เป็นของแข็งด้วยรังสียูวีซึ่งมีการศึกษาประสิทธิภาพในการเร่งปฏิกิริยาของวัสดุทั้งหมดที่สังเคราะห์ได้ โดยศึกษาปฏิกิริยาออกซิเดชันของสไตรีน มีไฮโดรเจนเปอร์ออกไซด์เป็นตัวออกซิไดซ์ และศึกษาสภาวะที่เหมาะสมของปฏิกิริยา ผลของเวลาและอุณหภูมิที่ใช้ในการทำปฏิกิริยา, สัดส่วนของตัวออกซิไดซ์ต่อสไตรีน, ปริมาณของตัวเร่งปฏิกิริยา และปริมาณของโลหะ จากการศึกษาพบว่า เปอร์เซ็นต์ความสามารถในการเปลี่ยนสไตรีน และเปอร์เซ็นต์ความเลือกจำเพาะของผลิตภัณฑ์ขึ้นอยู่กับลักษณะพื้นฐานของโลหะแต่ละชนิดที่ใส่เข้าไป กลไกศาสตร์ของปฏิกิริยาออกซิเดชันของสไตรีนจากการใช้โครเมียม-MCM-48 เป็นตัวเร่งปฏิกิริยาถูกศึกษาในงานวิจัยนี้ด้วย

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**ABBREVIATIONS**

BET	Brunauer-Emmett-Teller
CTAB	Cetyltrimethylammonium Bromide
DRUV	Diffuse Reflectance Ultraviolet Spectroscopy
EG	Ethylene Glycol
FE-SEM	Field Emission Scanning Electron Microscope
FTIR	Fourier Transform Infrared Spectroscopy
TEA	Triethanolamine
TEM	Transmission Electron Microscope
TEOS	Tetraethoxysilane
TETA	Triethylenetetramine
TGA	Thermogravimetric Analysis
XRD	X-ray Diffraction