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**SYNTHESIS AND CHARACTERIZATION OF
SCHIFF'S BASE DOPED MESOPOROUS SILICA AND
APPLICATION TO SOLID-PHASE EXTRACTION OF METAL**



Miss Pornmanee Khamloet

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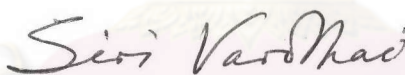
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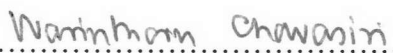
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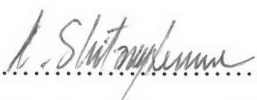
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พรมณี ขำเลิศ : การสังเคราะห์และลักษณะเฉพาะของเมโซพอร์ซิลิกาโดยดัดด้วยซิฟเบส และการประยุกต์ในการสกัดโลหะด้วยเฟสของแข็ง (SYNTHESIS AND CHARACTERIZATION OF SCHIFF'S BASE DOPED MESOPOROUS SILICA AND APPLICATION TO SOLID-PHASE EXTRACTION OF METAL)

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ได้สังเคราะห์เมโซพอร์ซิลิกาโดยดัดด้วยซิฟเบสสี่ชนิด ได้แก่ salen, saltn, salophen และ haen โดยใช้เทระเอทอกซีไซเลนเป็นสารตั้งต้นซิลิกา และใช้ไซเตียมไฮดรอกไซด์เป็นตัวเร่งปฏิกิริยา โดยปัจจัยที่ศึกษา ได้แก่ อัตราส่วนโดยโมลของซิฟเบสต่อ TEOS ที่ใช้ในการสังเคราะห์ การตรวจสอบการมีอยู่ของซิฟเบสโมเลกุลในซิลิกาอาศัยเทคนิค FT-IR การศึกษาสมบัติทางกายภาพของเมโซพอร์ซิลิกาด้วยเทคนิคการเลี้ยวเบนของรังสีเอ็กซ์และการดูดซับไนโตรเจนพบว่า ซิลิกาที่สังเคราะห์ได้ทุกชนิดเป็นแบบเมโซพอร์ซิลิกาที่มีการจัดเรียงโครงสร้างอย่างเป็นระเบียบ มีการกระจายของรูพรุนอยู่ในช่วงที่แคบ มีพื้นที่ผิวสูงและปริมาตรของรูพรุนมีมาก ผลการศึกษาที่ได้จากเทคนิค SEM แสดงให้เห็นว่าซิลิกาที่เตรียมได้มีลักษณะแบบทรงกลม การศึกษาปัจจัยต่างๆ ที่มีผลต่อสมบัติในการสกัด Co(II), Cu(II), Fe(II), Fe(III) และ Mn(II) ของซิลิกา ได้แก่ pH, ผลของเกลือที่มีอยู่ในสารละลายโลหะ, อุณหภูมิและปริมาณของซิลิกา ได้แสดงให้เห็นว่า เมโซพอร์ซิลิกาโดยดัดด้วยซิฟเบสทุกชนิดมีความเลือกจำเพาะต่อการสกัด Cu(II) และ Fe(III) โดยเมโซพอร์ซิลิกาโดยดัดด้วย saltn มีความสามารถในการสกัดโลหะสูงกว่าเมโซพอร์ซิลิกาโดยดัดด้วย salen, salophen และ haen ตามลำดับ การคาย Cu(II) และ Fe(III) ออกจากซิลิกาสามารถกระทำได้อย่างสมบูรณ์เมื่อใช้ 0.1 M HNO₃ เป็นสารคายโลหะ

ศูนย์วิทยทรัพยากร
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PORNMANEE KHAMLOET : SYNTHESIS AND CHARACTERIZATION
OF SCHIFF'S BASE DOPED MESOPOROUS SILICA AND
APPLICATION TO SOLID-PHASE EXTRACTION OF METAL. THESIS
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Four types of Schiff's base doped mesoporous silica were synthesized using one of the following Schiff's base ligand, namely, salen, saltn, salophen and haen as a doping molecule, tetraethoxysilane as a silica precursor and sodium hydroxide as a catalyst. The influence of Schiff's base/TEOS mole ratios was also considered. The existence of Schiff's base molecules in the modified silica was detected by FT-IR analysis. The physical properties of these synthesized materials were characterized using X-ray diffraction technique and N₂ sorption measurement. The results suggested that all synthesized silica had good ordered arrangement of mesopore with narrow pore size distribution. These materials had large surface area and pore volume. The SEM studies demonstrated the spherical morphology of all synthesized silicas. The extraction behavior of the modified silica towards Co(II), Cu(II), Fe(II), Fe(III) and Mn(II) were determined. Various parameters including pH, effect of salts present in metal solution, temperature and amount of silica were investigated. Interestingly, all Schiff's base doped mesoporous silica had high selectivity to Cu(II) and Fe(III) extraction. The metal extraction capacity of Schiff's base doped mesoporous silica was found to be in this order: saltn > salen > salophen > haen doped mesoporous silica. Complete elution of both Cu(II) and Fe(III) ions were achieved using 0.1 M HNO₃ as desorption agent.

Department.....Chemistry.....Student's signature.....*Pornmanee Khamloet*
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ศูนย์วิทยทรัพยากร
จุฬาลงกรณ์มหาวิทยาลัย

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LIST OF ABBREVIATIONS AND SYMBOLS

APD	average pore diameter
BET	Brunauer-Emmet-Teller
BJH	Barrett-Joyner-Halenda
CTAB	cetyltrimethylammonium bromide
DTG	differential thermogravimetry
FT-IR	fourier-transform infrared spectroscopy
haen	2,2'-{ethane-1,2-diylbis[nitrilo(1 <i>E</i>)eth-1-yl-1-ylidene]}diphenol
salen	2,2'-{ethane-1,2-diylbis[nitrilo(<i>E</i>)methylidene]}diphenol
salophen	2,2'-{1,2-phenylenebis[nitrilo(<i>E</i>)methylidene]}diphenol
saltn	2,2'-{propane-1,3-diylbis[nitrilo(<i>E</i>)methylidene]}diphenol
SEM	scanning electron microscopy
SPE	solid-phase extraction
TEOS	tetraethylorthosilicate
TGA	thermogravimetric analysis
TMOS	tetramethylorthosilicate
XRD	x-ray diffraction

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จุฬาลงกรณ์มหาวิทยาลัย