# การสังเคราะห์ผง ซิงค์แกลเลตและ ซิงค์อะลูมิเนตประเภทสไปเนลโดยวิธีโซลโวเทอร์มอล

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## SOLVOTHERMAL SYNTHESIS OF SPINEL-TYPE ZINC GALLATE AND ZINC ALUMINATE POWDERS

Miss Paveena Sangthonganothai

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A Thesis Submitted in Partial Fulfillment of the Requirements for the Degree of Master of Engineering in Chemical Engineering Department of Chemical Engineering Faculty of Engineering Chulalongkorn University Academic Year 2000 ISBN 974-13-0421-8 ถิงสิทธิ์ของจุฬาลงกรณ์มหาวิทยาลัย

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นางสาวปวีณา แสงทองอโณทัย: การสังเคราะห์ผงซิงค์แกลเลตและซิงค์อะลูมิเนตประเภทสไปเนลโดย วิธีโซลโวเทอร์มอล (SOLVOTHEMAL SYNTHESIS OF SPINEL-TYPE ZINC GALLATE AND ZINC ALUMINATE POWDERS) อ. ที่ปรึกษา : คร. สุพจน์ พัฒนะศรี, อ. ที่ปรึกษาร่วม : ศ. คร. ปียะสาร ประเสริฐธรรม, 112หน้า. ISBN 974-13-0421-8

ปฏิกิริยาทางความร้อนของซิงค์อะซิเตตและแกลเลียมอะซิติลอะซิโตเนตตามปริมาณสารสัมพันธ์(อัตรา ้ส่วนโดยอะตอมของสังกะสีต่อแกลเลียมเท่ากับ0.50) ในตัวทำละลายอินทรีย์ประเภทไกลคอล (1,4-บิวเทนได ออล์ )และสารละลายอินทรีย์ประเภทแอลกอฮอล์ (1-บิวทานอล และ 2-โพรพานอล) ที่อุณหภูมิการทำปฏิกิริยา 300°C ภายใต้สภาวะการเพิ่มขึ้นของความคันตามอณหภูมิ จะให้ผลิตภัณฑ์เป็น ซิงค์แกลเลตที่มีโครงสร้างผลึก เป็นแบบสไปเนล ที่มีขนาคเส้นผ่านศูนย์กลาง 8 ถึง 19 นาโนเมตร และพื้นที่ผิว 52 ถึง 113 ตารางเมตรต่อกรับ ้ส่วนในตัวทำละลายอินทรีย์ที่เฉื่อยต่อปฏิกิริยา (โทลูอีน) ไม่เกิดปฏิกิริยาให้ซิงค์แกลเลต แต่ปฏิกิริยาทางความ ร้อนของซิงค์อะซิเตตและอะลูมิเนียมไอโซพรอกพอกไซค์ สามารถเกิดขึ้นได้ในตัวทำละลายอินทรีย์ทุกชนิดดัง กล่าว ให้ผลิตภัณฑ์เป็นซิงค์อะลูมิเนตที่มีโครงสร้างผลึกแบบสไปเนล มีขนาคเส้นผ่านศูนย์กลาง 7 ถึง 11 นาโน เมตร และพื้นที่ผิว 80 ถึง 198 ตารางเมตรต่อกรับ สมบัติทางกายภาพและความเสถียรทางความร้อนของซิงค์แกล-เลตและซิงค์อะลูมิเนตที่ทำการสังเคราะห์ขึ้นสามารถที่จะควบคุมได้โดยปฏิกิริยาของการเกิดผลึกซึ่งขึ้นกับชนิด ของตัวทำละลาย นอกเหนือจากสภาวะการเกิดปฏิกิริยา และโครงสร้างของกลุ่มอัลคิลของโลหะอัลคอกไซด์ . ปฏิกิริยาของการเกิดผลึกที่เกิดขึ้นได้อย่างรวดเร็วจะทำให้การตกผลึกของผลิตภัณฑ์เกิดเป็นซิงค์แกลเลตและซิงค์ อะลูมิเนตที่มีโครงสร้างผลึกแบบ สไปเนลที่สมบูรณ์และมีความเสถียรทางอุณหภูมิสูง ในงานวิจัยนี้ พบว่า ขนาด ผลึกของซิงค์แกล-เลตและซิงค์อะลมิเนตเปลี่ยนแปลงไปตามชนิดของตัวทำละลายอินทรีย์ที่ใช้ แต่ความเสถียร ทางกวามร้อนของซิงก์แกลเลตและซิงก์อะลุมิเนตไม่ขึ้นกับชนิดของตัวทำละลายอินทรีย์ที่ใช้ แต่ขึ้นโดยตรงกับ ้งนาดผลึก นอกจากนี้ยังพบว่า ซิงค์อะลูมิเนตมีความเสถียรทางความร้อนสูงกว่าซิงค์แกลเลตที่ช่วงอุณหภูมิใน แต่เมื่ออุณหภูมิในการคัลไซน์สูงขึ้น ซิงค์แกลเลตกลับมีความเสถียรทางความร้อนที่คีกว่า การคัลไซน์ที่ไม่สูง ้อย่างไรก็ตาม การลคลงของความเสถียรทางความร้อนของซิงค์อะลูมิเนตจะมากกว่า ซิงค์แกลเลตเมื่อมีการเปลี่ยน แปลงขนาดผลึกเพียงเล็กน้อย แต่การมีโลหะตัวที่สองอยู่ในโครงสร้างจะช่วยเพิ่มความเสถียรทางความร้อนให้ กับโลหะออกไซค์ได้

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สาขาวิชาวิศวกรรมเคมี	ลายมือชื่ออาจารย์ที่บรึกษา	S. Photanami
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PAVEENA SANGTHONGANOTHAI: SOLVOTHERMAL SYNTHESIS OF SPINEL-TYPE ZINC GALLATE AND ZINC ALUMINATE POWDERS THESIS ADVISOR: DR.SUPHOT PHATANASRI, THESIS CO-ADVISOR: PROF. PIYASAN PRASERTHDAM,

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Thermal reaction of zinc acetate and gallium acetylacetonate, stoichiometric ratio Zn/Ga = 0.50, in 1,4-butanediol (glycol organic media), 1-butanol and 2propanol (alcohol organic media) at 300 °C under autogeneous pressure yielded nanocrystalline spinel zinc gallate with diameter between 8 and 19 nm and BET surface area between 52 and 113  $m^2 g^{-1}$ . However, the reaction of them did not take place and not yielded zinc gallate in toluene. As thermal reaction of zinc acetate and aluminium isopropoxide took place in all type of organic media (1,4-butanediol, 1butanol, 2-propanol and toluene). That yielded the nanocrystalline zinc aluminate with diameter between 7 and 11 nm and BET surface area between 80 and 198 m<sup>2</sup> g<sup>-1</sup>. When the reaction of crystallite formation occurs rapidly and so does the crystallization of the products then obtain as-synthesized well-crystallized spinel zinc gallate and zinc aluminate having high thermal stability. In this work, found that crystallite sizes of zinc gallate and zinc aluminate depend on type of the organic solvent. As for thermal stability of zinc gallate and zinc aluminate not depend on type of organic solvent but on the crystallite size. Thermal stability of zinc aluminate is better than zinc gallate in early calcination temperature. Conversely, at higher calcination temperature, zinc gallate is more stable than zinc aluminate. And the thermal stability decreasing of zinc aluminate is higher than zinc gallate when crystallite size is decreased. However, the presence of the second metal in the structure could be increased the thermal stability of the single metal oxide.

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Field of studyChemical Engineering	Advisor's signature. S. Phatamasni
Academic year2000	Co-advisor's signature.



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