

**A NOVEL ROUTE TO SYNTHESIS OF LEAD GLYCOLATE AND
PEROVSKITE LEAD TITANATE, LEAD ZIRCONATE, AND LEAD
ZIRCONATE TITANATE (PZT) VIA SOL-GEL PROCESS**

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ABSTRACT

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The reaction of lead acetate trihydrate $\text{Pb}(\text{CH}_3\text{COO})_2 \cdot 3\text{H}_2\text{O}$ and ethylene glycol, using triethylenetetramine (TETA) as a catalyst, provides, in one step, an access to a polymer-like precursor of lead glycolate $[-\text{PbOCH}_2\text{CH}_2\text{O}-]$ via oxide one pot synthesis (OOPS). The lead glycolate precursor has superior electrical properties than lead acetate trihydrate, suggesting that the lead glycolate precursor can possibly be used as a starting material mixed with other precursors such as titanium glycolate and sodium tris (glycozirconate). The lead-titanium glycolates underwent the sol-gel transition through the formation of Pb-O-Ti bonds, for use as electronic-grade PbTiO_3 . Perovskite lead zirconate was synthesized, using lead glycolate and sodium tris (glycozirconate) as the starting precursors. Moreover, the perovskite lead zirconate titanate was synthesized, using lead glycolate, sodium tris (glycozirconate), and titanium glycolate. The obtained molar ratio Pb:Zr:Ti of $\text{Pb}(\text{Zr}_x\text{Ti}_{1-x})\text{O}_3$: $\text{Pb}(\text{Zr}_{0.5}\text{Ti}_{0.5})\text{O}_3$ was 1:0.5:0.5. Our synthesized material appears to be a suitable candidate for producing electrical and semiconducting ceramics, viz. ferroelectric, anti-ferroelectric, and piezoelectric materials.

บทคัดย่อ

นุชนภา ตั้งบริบูรณ์ : นวัตกรรมของกระบวนการสังเคราะห์เลดไกลโคเลตและโครงสร้างแบบเพอโรฟสไกต์ของเลดไททานเต เลดเซอร์โคเนต และ เลดเซอร์โคเนตไททานเต ผ่านกระบวนการโซล-เจล (A Novel Route to Synthesis of Lead Glycolate and Perovskite Lead Titanate, Lead Zirconate, and Lead Zirconate Titanate (PZT) via Sol-Gel Process) อ. ที่ปรึกษา: รองศาสตราจารย์. ดร. สุจิตรา วงศ์เกษมจิตต์, ศาสตราจารย์ ดร. อเล็กซานเดอร์ เอ็ม เจมิสัน และ รองศาสตราจารย์. ดร. อนุวัฒน์ ศิริวัฒน์ 160 หน้า ISBN 974-9990-14-5

งานวิจัยนี้ได้ศึกษาการเกิดปฏิกิริยาระหว่างเลดอะซีเตตไตรไฮเดรต และเอทิลีนไกลคอล โดยใช้สาร ไตรเอทิลีนเตตระมีนเป็นคะตะลิสต์เพื่อใช้ในการผลิตสารตั้งต้น โพลิเมอร์คือเลดไกลโคเลต [-PBOCH₂CH₂O-] ที่สำคัญชนิดหนึ่งคือขบวนการนำไปใช้ในการสังเคราะห์ สารไดอิเล็กตริกชนิดต่างๆ ได้แก่ สารเฟอร์โรไดอิเล็กตริก สารแอนไทเฟอร์โรไดอิเล็กตริก และสารเพียโซไดอิเล็กตริก สารตั้งต้นสามารถสังเคราะห์ได้จากสารประกอบออกไซด์เพียงชั้นตอนเดียวที่เรียกว่า Oxide One Pot Synthesis (OOPS) ทำให้ได้สารตั้งต้นเลดไกลโคเลตมีสมบัติทางไฟฟ้าที่ดีกว่าเลดอะซีเตตไตรไฮเดรต นอกจากนี้ยังสามารถนำไปใช้ผสมกับสารตั้งต้นชนิดอื่นๆ เช่น ไททานเนียมไกลโคเลตและโซเดียมทริสไกลโคเซอร์โคเนตสามารถสังเคราะห์เลดไททานเตได้โดยผ่านกระบวนการโซล-เจลทรานสิชัน ระหว่างเลดไกลโคเลตและไททานเนียมไกลโคเลต ทำให้ได้โครงสร้างพันธะเป็น Pb-O-Ti สำหรับใช้ในการผลิตวัสดุไดอิเล็กตริกเลดไททานเต งานวิจัยนี้สามารถเตรียมเพอโรฟสไกต์เลดเซอร์โคเนตโดยการใช้ เลดไกลโคเลตและโซเดียมทริสไกลโคเลต เซอร์โคเนตเป็นสารตั้งต้นผ่านกระบวนการโซล-เจลเช่นเดียวกันนอกจากนี้เลดเซอร์โคเนตไททานเตสามารถสังเคราะห์ได้จากสารตั้งต้นเลดไกลโคเลต โซเดียมทริสไกลโคเซอร์โคเนตและไททานเนียมไกลโคเลต ด้วยอัตราส่วนโมลระหว่าง Pb:Zr:Ti ของ Pb(Zr_xTi_{1-x})O₃: Pb(Zr_{0.5}Ti_{0.5})O₃ คือ 1:0.5:0.5 สารสังเคราะห์ที่ผลิตได้สามารถนำไปใช้ในการผลิตวัสดุไดอิเล็กตริกและวัสดุเซรามิกส์กึ่งตัวนำ ได้แก่ สารเฟอร์โรไดอิเล็กตริก แอนติเฟอร์โรไดอิเล็กตริก และเพียโซไดอิเล็กตริก

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ABBREVIATIONS

^{13}C NMR	Carbon Nuclear Magnetic Resonance Spectroscopy
EA	Elemental Analysis
EG	Ethylene Glycol
FAB ⁺ -MS	Fast Atomic Bombardment Mass Spectroscopy
FTIR	Fourier Transform Infrared Spectroscopy
OOPS	Oxide One Pot Synthesis
SEM	Scanning Electron Microscope
SOL-GEL	Sol Gel Process
TETA	Triethylenetetramine
TGA	Thermal Gravimetric Analysis
XRD	X-ray Diffraction Spectroscopy