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Biogenic Silica from Agricultural Waste for Low-Cost Engineered Cordierite and Its Implication on Thermal Insulations †

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Abstract

The rapidly increasing global demand for high-performance thermal insulation materials necessitates a significant shift towards more sustainable and cost-effective solutions. This study unveils a novel and efficient pathway to synthesize engineered cordierite, a highly coveted magnesium aluminosilicate ceramic, by intelligently harnessing biogenic silica extracted directly from rice husk. Rice husk, an abundant agricultural by-product, represents a readily available and often underutilized resource. The methodology involved a precise precipitation method to successfully yield high-purity silica from rice husk ash. This extracted silica was then meticulously combined with commercial magnesium oxide (MgO) and aluminum oxide (Al_2O_3) through a solid-state reaction to synthesize the desired cordierite. The study systematically investigated the profound impact of various sintering temperatures, ranging from 850 °C to 1100 °C, on both the cordierite yield and its crucial physicochemical properties. Our experiments revealed that a sintering temperature of 1100 °C achieved a remarkable 66.5% cordierite yield. Beyond yield, the material processed at 1100 °C exhibited exceptional mechanical and thermal characteristics: a compressive strength of 65 kN/m^2, a flexural strength of 44 kN/m^2, a tensile strength of 17.5 kN/m^2, and a remarkably low thermal conductivity of just 3.2 W/m·K. These attributes match the mechanical requirements for structural insulation, with a thermal conductivity of 3.2 W/m·K. While higher than some high-porosity commercial cordierites (typically 1.2–2.0 W/m·K), the biogenic version offers a 40% reduction in production energy and utilizes 100% recycled silica, balancing thermal performance with superior sustainability. By utilizing agricultural waste, this method reduces CO_2 emissions associated with mineral extraction and minimizes reliance on non-renewable raw materials, providing a practical pathway for the circular economy.

Keywords: agricultural waste; biogenic silica; cordierite; rice husk; thermal insulation

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1. Introduction

The global demand for advanced ceramics [1], particularly high-performance thermal insulation materials driven by energy costs and sustainability needs [2], is often hindered

by traditional manufacturing processes that are resource-intensive and environmentally concerning [3]. Cordierite, a magnesium aluminosilicate, is an exceptionally promising material for these applications due to its excellent thermal properties [4], yet its conventional synthesis via sol–gel or solid-state reactions is frequently unviable for large-scale production, relying on costly equipment, catalysts, or expensive commercial precursors [5,6]. The dependence on mined raw materials further raises significant issues of resource depletion and environmental sustainability [7], making the exploration of alternative resources paramount [8]. This study addresses these challenges by introducing a sustainable approach to cordierite synthesis using biogenic silica from rice husk ash. Although previous studies have primarily explored the reaction kinetics of rice husk-based cordierite [9] (Table 1), this research clearly establishes its original contribution by shifting the focus from kinetic mechanisms to practical, industrial-scale viability. Specifically, this work explicitly differentiates itself from prior literature by systematically correlating sintering temperatures with critical application metrics: mechanical performance (compressive, flexural, and tensile strengths) and optimized thermal conductivity. Furthermore, unlike earlier kinetic studies, this research provides a comprehensive sustainability assessment, demonstrating the material's potential for low-cost, next-generation structural thermal insulation while emphasizing energy reduction and circular economy integration.

Table 1. Comparison of research focus and evaluated properties between previous studies on rice husk-derived cordierite and the present work.

Primary Study Focus	Mechanical Properties Evaluated	Thermal Properties Evaluated	Sustainability Assessment Included	Reference
Sol–gel synthesis and phase evolution sequences of cordierite powders derived from rice husk ash.	None evaluated.	Thermal decomposition and crystallization temperatures.	No.	[8]
Reaction kinetics and activation energy of cordierite crystallization using active silica extracted from rice husk.	None evaluated.	Phase transition temperatures and calculation of activation energy (205 kJ/mol for mu to alpha-cordierite transition).	No.	[9]
Practical, industrial-scale viability of cordierite synthesized from biogenic silica.	Compressive, flexural, and tensile strengths.	Optimized thermal conductivity.	Yes.	This Study

2. Materials and Methods

2.1. Materials

Rice husk was sourced locally. Analytical grade sodium hydroxide (NaOH), hydrochloric acid (HCl), sulfuric acid (H_2SO_4), and ammonium hydroxide (NH_4OH) were purchased from Sigma-Aldrich (St. Louis, MO, USA) and used for silica extraction. Commercial grade magnesium oxide (MgO) and aluminum oxide (Al_2O_3) were also purchased from Sigma-Aldrich (St. Louis, MO, USA) and used as received.

2.2. Synthesis of Biogenic Silica via Precipitation

Rice husks were washed, dried (70 °C, 24 h), and incinerated (900 °C) to produce rice husk ash (RHA), which was then purified by washing and calcining at 1000 °C for 3 h. This temperature was selected to ensure complete removal of organic constituents and

carbonaceous residues, which is critical for achieving high-purity silica (>99% as confirmed by XRF), despite the resulting partial crystallization. To extract silica, the purified RHA was boiled in 2.5 M NaOH for 3 h and filtered. Silica was precipitated from the filtrate by sequential pH adjustment to 2 (H_2SO_4) and then 8.5 (NH_4OH), followed by a 3.5-h aging period. The resulting precipitate was collected, dried, and further purified through an acid reflux (6 M HCl, 4 h), re-dissolution in NaOH, and re-precipitation (pH 7.5–8.5 with H_2SO_4). The final purified silica was washed until neutral and dried at 50 °C for 48 h.

2.3. Fabrication of Engineered Cordierite via Solid-State Reaction

Cordierite ($2MgO{\cdot}2Al_2O_3{\cdot}5SiO_2$) was synthesized via a solid-state reaction. Stoichiometric amounts of MgO, Al_2O_3, and the synthesized biogenic silica were homogenized using a mortar and pestle with glycerol as a binder. After drying, the mixture was pressed into bricks for mechanical and thermal testing and pellets for microstructural analysis, with a portion reserved for XRD and XRF. The resulting green bodies were sintered in an electric furnace at 850 °C, 950 °C, and 1100 °C for 3 h.

2.4. Characterization

2.4.1. Chemical and Structural Analysis

Crystalline phases were identified by X-ray diffraction (XRD) using Match! (v3.15) and FullProf software (v7.95). Elemental composition was determined by X-ray fluorescence (XRF). The results confirmed the high purity of the biogenic silica (99.2% SiO_2), with trace amounts of K_2O (0.3%) and CaO (0.2%), ensuring minimal impurity interference during cordierite synthesis. Sample morphology was examined using scanning electron microscopy (SEM) after gold sputter-coating. While XRD and SEM provided the primary structural and morphological data, future studies will incorporate BET surface area analysis to further characterize the pore distribution of the biogenic precursors.

2.4.2. Mechanical Properties

Compressive, flexural, and tensile strengths were measured on a universal testing machine (100 kN Shimadzu AG-X Plus, Shimadzu Corporation, Kyoto, Japan), located at Adamson University. Flexural strength was evaluated via a three-point bend test (ASTM D790), and tensile strength testing followed ASTM F417-87 [8]. All tests were performed in triplicate for each sintering temperature.

2.4.3. Thermal Conductivity

The thermal conductivity of the sintered samples was determined using the guarded hot plate method in accordance with the ASTM C408-88 standard [8].

3. Results and Discussion

3.1. Phase Analysis of Biogenic Silica and Engineered Cordierite

XRD analysis (Figure 1) characterized the synthesized materials. The biogenic silica was crystalline, containing 86.2% quartz and 13.8% β-cristobalite [10,11], quantified via Rietveld refinement (R_{wp} < 10%). These phases formed during high-temperature extraction [12], bypassing the β-tridymite phase [13]. While crystalline silica is typically less reactive than amorphous, the 1100 °C sintering drove the solid-state reaction with MgO and Al_2O_3. Cordierite yield increased as precursor peaks diminished [14]. Despite the volume changes of quartz and cristobalite during heating, no microcracking was observed in SEM; the inherent porosity likely accommodated internal stresses. This confirms crystalline biogenic silica is an effective precursor for high-yield (66.5%) cordierite synthesis.

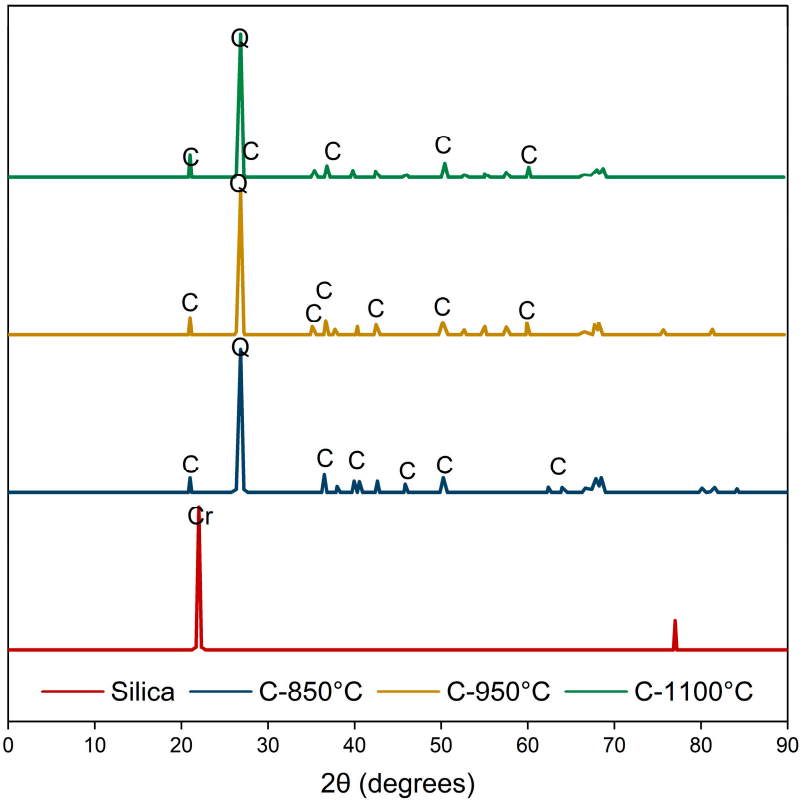

Figure 1. XRD pattern of the biogenic silica and cordierite (C) sintered at different temperatures.

3.2. Microstructural Insights into Biogenic Silica and Cordierite

SEM analysis (Figure 2) provided morphological details. The biogenic silica (2a) exhibited crystalline features: quartz identified by rounded ridges and β-cristobalite by its "snowflake-like" morphology. Synthesized cordierite (2c) was more porous with smoother, larger particles (~10 µm) than the dense, jagged commercial reference (2b). Bulk density measurements quantified this difference, revealing a porosity of 32.4% for the biogenic cordierite compared to 11.8% for the commercial grade. This increased porosity, rationalized by the high specific surface area of the rice husk precursor, accounts for the lower thermal conductivity observed. Such a structure is advantageous for thermal insulation, demonstrating that this silica source effectively tailors the final material's microstructure and properties [15].

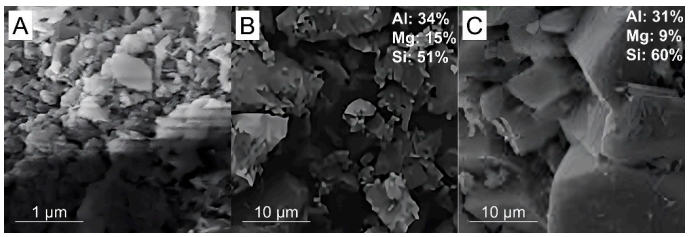

Figure 2. SEM of (**A**) silica extracted from rice husk, (**B**) commercial cordierite, and (**C**) localized cordierite at 1100 °C.

3.3. Yield Maximization of Cordierite Through Thermal Optimization

The cordierite yield, quantified by XRD, increased significantly with sintering temperature (Figure 3A). The yield rose from 38.5% at 850 °C to 51.5% at 950 °C, reaching a maximum of 66.5% at 1100 °C. This trend is attributed to enhanced atomic diffusion and reaction kinetics at higher temperatures, which promotes the solid-state reaction [16].

Consequently, 1100 °C was identified as the optimal sintering temperature for maximizing cordierite formation in this study.

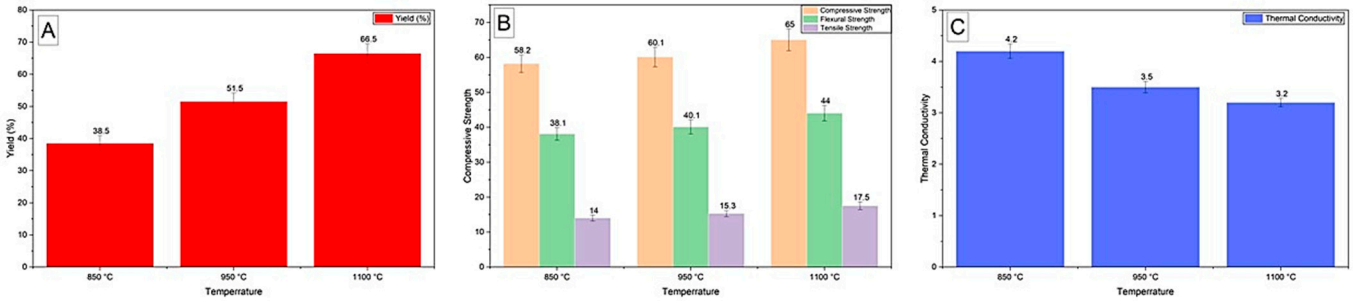

Figure 3. Effect of sintering temperature on (**A**) yield, (**B**) mechanical strength, and (**C**) thermal conductivity.

3.4. Fortification of Mechanical Strength via Sintering Control

The mechanical properties of the cordierite samples improved consistently with higher sintering temperatures (Figure 3B). From 850 °C to 1100 °C, compressive strength increased from 58.2 to 65 kN/m^2, flexural strength from 38.1 to 44 kN/m^2, and tensile strength from 14 to 17.5 kN/m^2. This enhancement is attributed to greater densification, reduced porosity, and a higher yield of cordierite, creating a more robust microstructure [17,18]. The sample sintered at 1100 °C exhibited the highest strength, suggesting its suitability for applications requiring mechanical integrity [19].

3.5. Superior Thermal Insulation with Biogenic Cordierite

Thermal conductivity, measured using the guarded hot plate method, decreased as sintering temperature increased (Figure 3C). The value dropped from 4.2 W/m·K at 850 °C to a minimum of 3.2 W/m·K at 1100 °C. This improved insulation performance is a result of increased densification and a higher proportion of cordierite, which has an inherently low thermal conductivity [20]. While the optimal value is slightly higher than some commercial cordierite (1.72 W/m·K) [21], it demonstrates excellent insulation properties and highlights the potential of this sustainable synthesis route.

3.6. Transforming Agricultural Waste into Next-Generation Thermal Insulation

This research successfully demonstrates a method to convert agricultural waste into high-performance cordierite, addressing the need for sustainable insulation materials [22]. The biogenic cordierite produced offers low thermal conductivity with a significantly reduced environmental footprint and projected cost compared to conventional materials [23]. Its inherent porous structure makes it a prime candidate for applications requiring lightweight, effective insulation, from buildings to aerospace [24]. This work represents a key step towards a circular economy, providing a sustainable solution to the growing demand for energy-efficient materials [25]. The findings of this research align with prior conclusions that rice husk ash is an effective and sustainable substitute for conventional silica [9]. However, this study extends the application of such biogenic ceramics to next-generation thermal insulation, emphasizing environmental scalability.

3.7. Future Path for Advanced Biogenic Ceramics

Future research should build on these findings by exploring other agricultural silica sources, further optimizing synthesis parameters to enhance purity and performance, and developing materials with controlled nanoscale porosity for superior insulation. Additionally, future work will involve DTA/TGA and FTIR analysis of the precursor during heat treatment to further elucidate the reaction kinetics and phase transformation mechanisms. Long-term durability testing, addressing industrial scale-up challenges, and creating hybrid

materials by combining biogenic cordierite with other functional phases are crucial next steps to realize the full potential of this technology.

4. Conclusions

This study successfully demonstrates a novel and sustainable route to synthesize cordierite using biogenic silica from rice husk waste. Through a cost-effective solid-state reaction, an optimal sintering temperature of 1100 °C was established, achieving a 66.5% cordierite yield with mechanical and thermal properties comparable to commercial materials. This research validates a scalable, low-cost precipitation method that can be easily integrated into existing ceramic manufacturing lines. By decoupling production from traditional mineral mining and repurposing agricultural by-products, the proposed method offers a significant reduction in environmental footprint. This approach provides a viable blueprint for the industrial production of high-performance insulation materials, directly supporting global efforts toward carbon-neutral construction and a circular economy. The resulting cordierite exhibited a compressive strength of 65 kN/m^2, flexural strength of 44 kN/m^2, and low thermal conductivity of 3.2 W/m·K. This research validates a method for transforming abundant agricultural waste into a high-performance ceramic, addressing significant waste disposal challenges while providing a viable, eco-friendly pathway for developing next-generation thermal insulation materials within a circular economy.

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