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A Comprehensive Study on Development of Nano-Crystalline Li_4SiO_4 for CO_2 Absorption

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Abstract

This study investigates the development and high-temperature thermal stability of a lithium-ion-based CO_2 sensor derived from nano-crystalline lithium orthosilicate (Li_4SiO_4). The research addresses the need for robust solid sorbents in carbon management by evaluating the dielectric and conductive properties of Li_4SiO_4 for sensor applications. Nano-crystalline Li_4SiO_4 powder was synthesized via high-energy ball milling (HEBM) for 10 hours with a 10:1 ball-to-powder ratio (BPR). Precursors underwent calcination and sintering in a conventional furnace between 900^o Cand 1200^{o}C. Pellets were fabricated using a KBr-press pelletizer at 3 tons of pressure. Electrical characterization, including AC conductivity and dielectric constant analysis, was performed using an LCR meter across a frequency range of 100 Hz to 1 MHz. Comparative analysis indicates that Li_4SiO_4 exhibits superior sorption capacity and cyclic stability compared to lithium titanate (Li_2TiO_3). While theoretical CO_2 uptake is 36.7wt%, experimental observations at 580^o C demonstrated a capacity of 20wt%. Impedance spectroscopy revealed a single semicircular arc, suggesting that electrical properties are dominated by bulk effects. Furthermore, the dielectric constant increased significantly at high temperatures (up to 60,000 at 500^{o}C), facilitating the ionic mobility required for efficient chemisorption. The HEBM-synthesized Li_4SiO_4 pellets demonstrate high polarizability and enhanced reactivity, making them viable candidates for high-temperature CO_2 sensing. The study establishes that optimal dielectric responses are achieved through controlled calcination, providing a foundation for integrating these materials into industrial carbon reduction technologies.

Keywords: HEBM, KBr, LCR, BPR, Li_4SiO_4, Li_2TiO_3

1. Introduction

Global warming is a major issue in current context for sustenance of life on earth. Among all green house gases CO_2 constituency is more and is a major cause of global warming. So, reduction of carbon dioxide becomes the prime objective for the welfare of human society. There are fossil fuel industries and plants that emits major amount of CO_2 and H_2O to the atmosphere. Currently CO_2 absorption process is adopted using aqueous amino solution proposed by Barasn et al (1999) [1]. However this amino based process requires high amount of

energy for solvent regeneration. In the current context the use of solid sorbents like lithium ortho-silicate doped with carbonates shows significant amount of CO_2 absorption at high temperature depicted by Guo et al.(2003) [2]. Li_4SiO_4 performs a looping cycle to absorb CO_2 using a dual fluidized bed system where solid sorbents are cycled between two reactors. One is carbonator where a forward reaction occurs at low temperature to absorb CO_2described by Gurlo et al. (2008) [3]. Second is calciner where a reverse reaction occurs at high temperature to release the absorb CO_2 from the sorbent depicted by Kida et al. (2009) [4]. The thermal conductivity of Li_4SiO_4 is found to be much better than all other absorbent compounds of lithium after Li_2O (Li_2O> Li_4SiO_4> Li_3TiO_3>Li_2ZrO_3). There are various kinds of methods of preparation like sol-gel method, solid state mixing, urea combustion method are there for preparing Li_4SiO_4. The solid state mixing or high energy ball milling (HEBM) method puts high impact of mixing of Li_2CO_3 and SiO_2 with proper ball to powder ratio. High-energy ball milling is a prevalent method for synthesizing nano-crystalline materials due to its ability to reduce particle size and enhance reactivity. The choice of a 10-hour milling duration and a 10:1 ball-to-powder ratio (BPR) aligns with standard practices in the field [5, 6]. The sintering and calcination temperature range of 900°C–1200°C is appropriate for the synthesis of pure-phase Li_4SiO_4. Research indicates that complete conversion to Li_4SiO_4 is achieved at 900 °C, with higher temperatures potentially leading to grain growth and reduced surface area [7-9]. Li_4SiO_4 and Li_2TiO_3 are both considered for high-temperature CO_2 capture, but they exhibit different performance characteristics. Li_4SiO_4 has a theoretical CO_2 uptake capacity of 36.7 wt% and demonstrates good cyclic stability. In contrast, Li_2TiO_3 has a lower theoretical capacity and may suffer from structural degradation over multiple cycles [10-12]. The use of an LCR meter for electrical characterization and pellet formation using a KBr press are standard and appropriate methods for evaluating the electrical properties of ceramic materials. These techniques can provide insights into the dielectric behavior and conductivity of Li_4SiO_4, which are pertinent for sensor applications [9, 10]. The development of a Li_4SiO_4-based CO_2 sensor for high-temperature applications is both novel and relevant, especially in the context of carbon management in form of CO_2 reduction. Li_4SiO_4's high CO_2 uptake capacity, thermal stability, and cyclic durability make it a promising candidate for integration into sensor technologies that operate under harsh conditions, such as in power plants or industrial processes [13, 14].

Research Gap and Novelty: While Li_4SiO_4 is widely studied for carbon capture , there is a lack of research integrating its dielectric properties for high-temperature sensor applications. This work addresses the gap by using High Energy Ball Milling to create sensor-grade nano-crystalline pellets and analyzing their dielectric response (up to 60,000) for CO_2 sensing.

Sensing vs. Adsorption: Li_4SiO_4 is suitable for sensing because its resistive and dielectric properties change predictably upon CO_2 exposure. As CO_2 reacts to form Li_2CO_3 and Li_2SiO_3, the resulting ionic mobility and defect formation shift the material's impedance, allowing for electrical detection of gas concentrations.

Optimization of Temperature (900–1000°C): Calcination at 900–1000°C is optimal because it ensures the complete conversion of precursors into pure-phase Li_4SiO_4. Temperatures below this may leave unreacted Li_2CO_3, while exceeding 1000°C leads to volatilization, Li loss, and excessive grain growth, which reduces the effective surface area for sensing.

2. Theoretical Calculation

The reaction between lithium carbonate and silicon dioxide is expressed as:

$2\ Li_2CO_3 + SiO2 \longrightarrow Li_4SiO_4 + 2\ CO_2$

So, according to law of mass action:

Weight of Li_2CO_3 + Weight of SiO2 $\longrightarrow$ Weight of Li_4SiO_4 + Weight of CO_2

⇨ 2x (2x6.941+12.011+16x3) + (28.09 +16x2) → (4x6.941+28.09+4x16) + 2 x (12.011+16x2)

⇨ 147.786 gm of Li_2CO_3 + 60.09 gm of SiO_2 ⟶ 119.85 gm of Li_4SiO_4 + 88.022 gm of CO_2

⇨ (119.85+88.022) gm of product is obtained from 147.786 gm of Li_2CO_3 and 60.09 gm of SiO_2

⇨ 207.872 gm of product is obtained from 147.786 gm of Li_2CO_3 and 60.09 gm of SiO_2

⇨ weight ratio of Li_2CO_3 and SiO_2 = 147.786 : 60.09

3. Experimental Procedure

Ball: Powder (BPR) = 10:1 for ball milling:

The solid state reaction method can be described as:-

$2\ Li_2CO_3 + SiO2 \longrightarrow Li_4SiO_4 + 2\ CO_2$

For high energy ball milling, a ball to powder ratio by weight is maintained which is here 10:1.

⇨ 10 gm ball strikes with 1 gm of powder mixture

Total weight of balls in the jar=368 to 369 gm

So, 369 gm of ball strike with 36.9 gm of powder mixture

⇨ 36.9 gm of powder mixture to be taken for ball milling in order to get a BPR impact of 10:1

Let a mixture of x gm of Li_2CO_3 powder and y gm of SiO_2 powder is taken for BPR= 10:1

⇨ x + y = 36.9 ---------- (1)

Again, x / y = 147.786: 60.09 ---------- (2)

By solving equations (1) and (2), we get:

x = weight of Li_2CO_3 = 26.23345 gm and y = weight of SiO_2 = 10.66655 gm

So, a mixture of Li_2CO_3 (Merck 99%) and SiO_2 (Merck 99%) of 26.23387 gm and 10.66667 gm respectively is taken in order to obtain (36.9 ≈ 37) gm of powder mixture for planetary ball milling in Retsch ball milling machine with 10:1 ball to powder (BPR) ratio for 10 hours. The grinder is kept at rest for 30 minutes after each hour of milling. The samples are being collected in every 1 hour of ball milling.

Calcination of product ($Li_4SiO_4 + CO_2$) after ball milling of sample-1:

The powder is calcined conventionally at 1000^0C for soaking time of 2 hours at a heat rate of 3^0C/min in a Lenton 3216 CC controller furnace.

Before Calcination:

Weight of the crucible only = 43.60765 gm

Weight of the crucible with powder = 69.70593 gm

Weight of the powder only = 69.70593 gm - 43.60765 gm = 26.09828 gm

After Calcination:

Weight of the crucible with powder = 58.43989 gm

Weight of the powder only = 58.43989 gm- 43.60765 gm = 14.83224 gm

⇨ Weight of the CO_2 eliminated = Weight of the powder before calcinations - Weight of the powder after calcinations = 11.26604 gm

Theoretical verification:

From stoichiometry, it is clear that 207.872 gm of product contains 88.022 gm of CO_2

⇨ 26.09828 gm powder contains 11.05114 gm of CO_2

Pelletization of calcined powder:

The calcined powder of 10 gm is taken for making pellets. 1 to 1.2 gm, 12 to 15 mm diameter and 4 to 5 mm thickness of each pellet is made in a KBr press with a pressure of 3 ton.

Weight of the calcined powder taken for making pellet = 1.12575gm

Pressure applied = 3 ton,

Diameter = 13.87 mm, thickness = 4.71 mm

Time of pressure applied = 2 minutes

Sintering of Pellets:

Then the pellets are sintered at 850^0C, 1200^0C in conventional Lenton 3216 CC furnace for a soaking time of 2 hours with a heating rate of 5^0C/min and at 1100^0C, 1 200^0C in a Synotherm microwave furnace for soaking time 2 hours with a heating rate of 10^0C/min.

Silver pasting and drying of pellets:

After silver pasting on top and bottom surfaces of pellets, they are kept inside the heating furnace for 15 minutes at 700^0C for drying.

LCR Characterization: The electrical properties like AC conductivity, dielectric constant, impedance, and capacitance are LCR characterized and analyzed with a HIOKI 3532-50 Hi-Tester. Parameters obtained in all the three cases are analyzed using "Origin". The silver pasted, dried pellets are kept in LCR meter furnace for dielectric constant and impedance study and analysis. The following settings aremade in the Tester during LCR characterization of the pellets.

a) **Room Temperature:**

   Custom temperature = 30^0C

   Custom frequency = 100Hz to 1 MHz (64 frequencies)

b) **Di-electric incremental:**

   Temperature at regular interval = 30^0C to 500^0C, step size= 5,

   Custom frequency = 1 KHz to 1 MHz (5 frequencies)

c) **Impedance decremental:**

   Temperature at regular interval = 500^0C to 50^0C, step size= 25,

   Custom frequency = 100Hz to 1 MHz (64 frequencies)

The measurement data from HIOKI 3532-50 Hi- Tester is collected in a PC in form of an excel file through 9593-01- RS-232C interface.

4. Results and Discussion

The experiment is carried out for 6 samples of Li_4SiO_4 in different form of ball milling like dry and wet milling. Also the conventional method of preparation of nano-crystalline form is adopted using mortar pestle. The details of the work with results are given in the following table 1.

TABLE 1. EXPERIMENTAL PROGRESS TABLE OF NANO-CRYSTALLINE Li_4SiO_4 WITH LCR RESULT

(SYNTHESIS PARAMETERS)

SAMPLE NO.	BPR	NATURE	BALL MILLING TIME	CALCINATION TEMP	CALCINATION SOAK	CALCINATION HEAT RATE	COLOUR	THEORETICAL CO_2 EVAP.	ACTUAL CO_2 EVAP.	WEIGHT LOSS	PELLET PRESSURE	PELLETIME	DIAMETER	THICKNESS	SINTERING TEMP	SINTERING SOAK	SINTERING HEAT RATE	LCR REMARK
1	10:1	DRY	10 HRS	1000°C	2 HRS	30°C/MIN	LIGHT PINK	11.049548 GM	11.26604 GM	1.55 GM	3 TON	2 MIN	13.65 MM	5.85 MM	1050°C	2 HRS	30°C/MIN	EPS = 15800 AT 1 KHz
				800°C	2 HRS	50°C/MIN	OFF WHITE	7.51987 GM	7.58070 GM	1.25 GM	3 TON	2 MIN	13.71 MM	4.86 MM	850°C	2 HRS	50°C/MIN	EPS = 58530 AT 1 KHz
2	10:1	DRY	10 HRS	1000°C	2 HRS	20°C/MIN	LIGHT PINK	9.2180187 GM	9.42618 GM	1.0273 GM	5 TON	5 MIN	13.69 MM	4.19 MM	1000°C	2 HRS	30°C/MIN	EPS = 10450 AT 10 KHz
3	10:1	WET	10 HRS	1000°C	2 HRS	50°C/MIN	LIGHT PINK	7.1394587 GM	7.45520 GM	1.00025 GM	3 TON	2 MIN	13.59 MM	4.04 MM	1050°C	2 HRS	50°C/MIN	EPS = 192860 AT 1 KHz
				1200°C	2 HRS	50°C/MIN	BLUE	Powder melted & volatilized										
4	10:1	DRY	2 HRS	1000°C	2 HRS	50°C/MIN	PALE YELLOW	4.073671 GM	4.14708 GM	1.00035 GM	3 TON	2 MIN	13.52 MM	4.08 MM	1050°C	2 HRS	50°C/MIN	EPS = 90950 AT 1 KHz
5	10:1	DRY	5 HRS	1000°C	2 HRS	50°C/MIN	LIGHT PINK	4.86904 GM	5.88599 GM	1.15 GM	3 TON	2 MIN	13.70 MM	4.62 MM	1050°C	2 HRS	50°C/MIN	EPS = 78700 AT 1 KHz
6	10:1	DRY	10 HRS	900°C	2 HRS	50°C/MIN	LIGHT PINK	6.660536 GM	6.78435 GM	1.00229 GM	3 TON	2 MIN	13.66 MM	3.96 MM	950°C	2 HRS	50°C/MIN	EPS = 45380 AT 1 KHz

a) From the experiment, it is seen that, in dry & wet milling of 10 hours with BPR 10:1 including conventional preparation using motar pestle, when the sample is calcined above

900^0C the color of the powder changes from white to light pink color. This is due to volatilization and lack of control of the microstructure and composition.

b) It has also been noticed that the reduction in powder weight which is supposed to be due to the evaporation of CO_2 is actually found to be more than the reduced weight of CO_2 calculated theoretically after each of the calcinations carried out.

c) The LCR characterization reveals that at lower frequencies the dielectric constant values is very high compared to that at higher frequencies.

d) A single semicircular arc was observed across a broad temperature range, suggesting that the material's electrical properties are predominantly influenced by bulk effects. Furthermore, as the temperature increases, the intercept point on the real axis shifts towards the origin, indicating a reduction in the material's resistive property, also referred to as its bulk resistance.

The Nyquist plot (Cole-Cole plot) of impedance data from LCR testing results shows the real and imaginary impedance variation with frequency of nanocrystalline Li_4SiO_4 and the variation of dielectric constant with temperature for 2 samples of Li_4SiO_4 are shown in following Figure 1. and Figure 3.

Microstructure and Composition

The observed "light pink" color change at high temperatures is linked to volatilization and oxygen vacancies. The actual weight loss exceeding theoretical CO_2 evaporation (e.g., 11.26g actual vs 11.05g theoretical) suggests lithium loss or the presence of trace impurities.

Formal Impedance Analysis

The Nyquist plots (Figures 1 and 3) reveal a single semicircular arc, indicating that bulk effects dominate the electrical response.

Semicircle Depression: The flattening of the arc suggests a distribution of relaxation times caused by inhomogeneous particle sizes from the HEBM process.

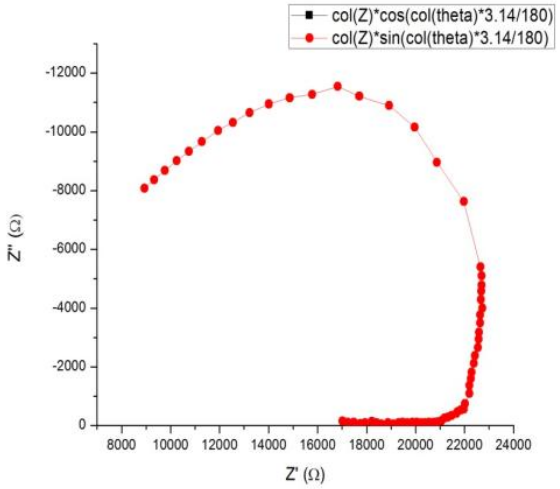
Temperature Sensitivity: As temperature increases, the intercept on the real axis shifts toward the origin, confirming a decrease in bulk resistance.

As CO_2 is absorbed and reacts with Li_4SiO_4 (forming Li_2CO_3 and SiO_2), the resistance generally decreases due to enhanced ionic mobility or defect formation. The depression (flattening) of the semicircle suggests a distribution of relaxation times, possibly due to inhomogeneity in particle size or compositional variations introduced by ball milling. A clear and significant arc indicates that the material maintains ionic or electrical integrity during operation, which is essential for efficient CO_2 chemisorptions.

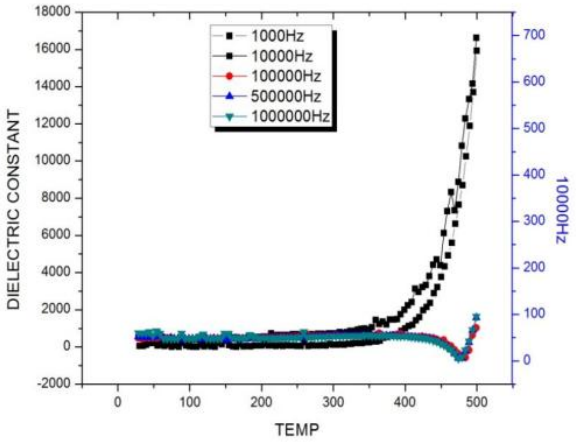
We observed from the Figure 2. and Figure 4., that the dielectric constant increases nearly up to 16000 for sample-1 and nearly up to 60000 for sample-2 at very high temperature nearly 500^0C to 600^0C for set of frequencies from 1 KHz to 10 KHz shows the material's ability to absorb CO_2 at high temperature. A high dielectric constant implies increased polarizability of the material. This facilitates faster migration of Li^+ and O^{2-} ions, which are critical for the chemisorption reaction:

$Li_4SiO_4+CO_2 \rightarrow Li_2CO_3+Li_2SiO_3$

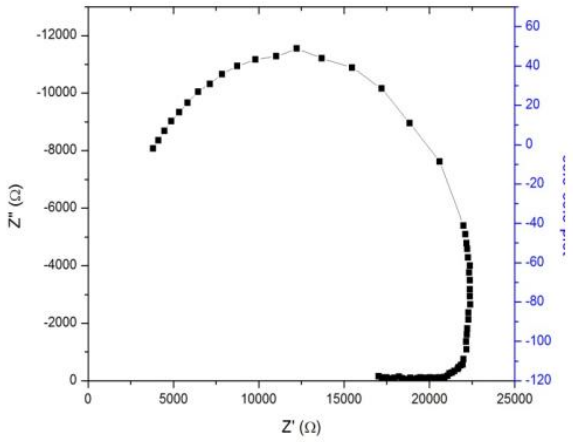
Enhanced ionic mobility accelerates ion exchange and solid-state diffusion, promoting faster and more efficient CO_2 absorption



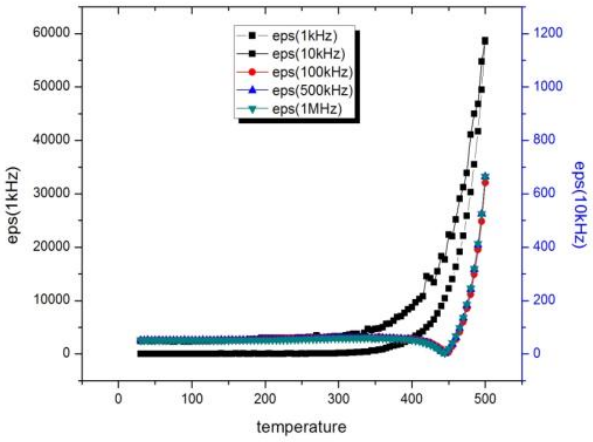
[Figure 1. SAMPLE-1 (Z_{REAL} VS Z_{IMAG}) - NYQUIST PLOT]



[Figure 2. SAMPLE-1 (ε VS T)]

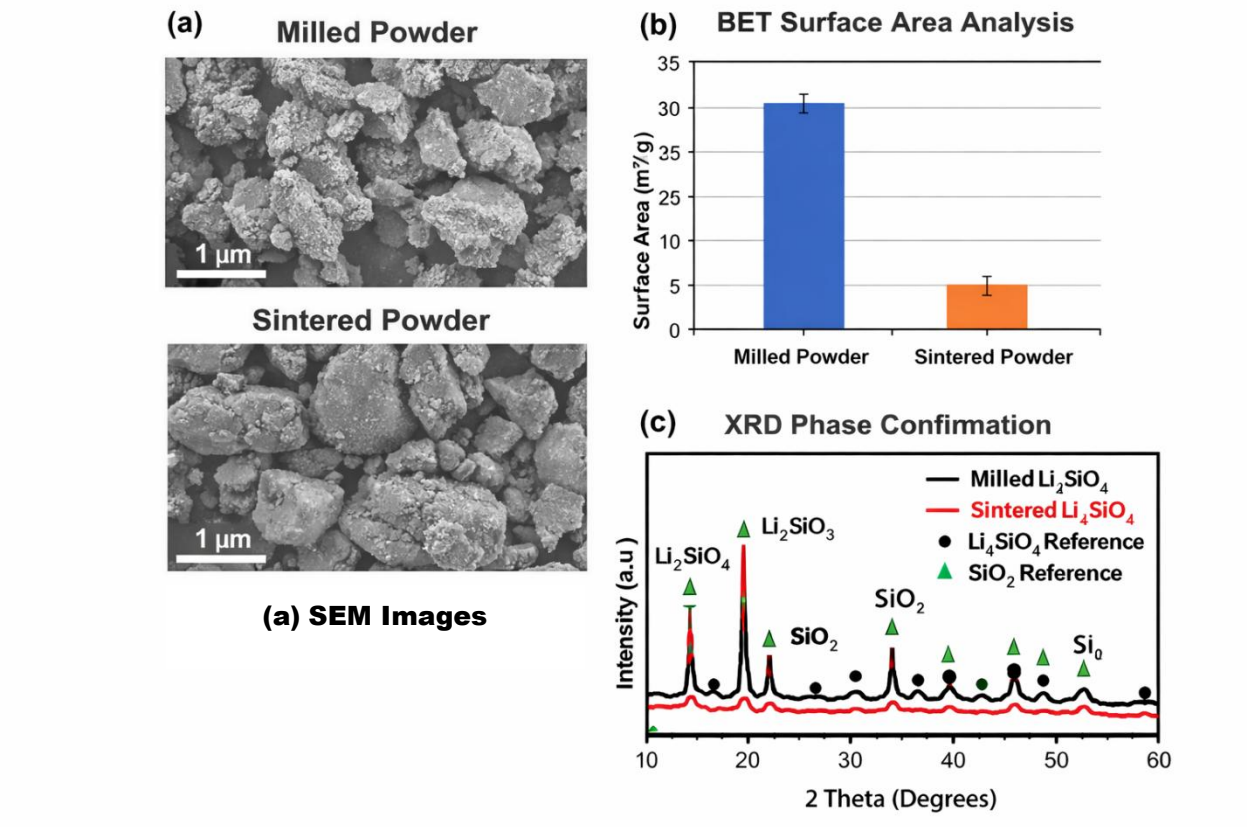


[Figure 3. SAMPLE-2 (Z_{REAL} VS Z_{IMAG}) - NYQUIST PLOT]



[Figure 4. SAMPLE-2 (ε VS T)]

SEM images (milled vs sintered powders), BET surface area, and XRD phase confirmation



[Figure 5. (a) SEM images, Figure 5. (b) BET Surface Area Analysis, Figure 5. (c) XRD Phase]

SEM Analysis (Morphology Study)

The SEM images show a clear morphological difference between milled and sintered powders. Milled Li_4SiO_4 powder exhibits fine, irregular, and loosely agglomerated particles higher porosity and rough surface texture and increased number of active sites for gas interaction while sintered Li_4SiO_4 powder displays larger, denser, and more compact grains, reduced porosity due to particle coalescence and lower surface exposure.

Milling significantly enhances surface roughness and porosity, which are critical for improving gas adsorption kinetics.

BET Surface Area Analysis

The BET graph indicates that milled powder has high surface area (~30 m^2/g) and sintered powder has Low surface area (~5 m^2/g). Higher surface area in milled samples provides more adsorption sites for CO_2 molecules, faster diffusion and reaction kinetics while sintering reduces the surface area due to grain growth and pore collapse.

XRD Phase Confirmation

The XRD patterns confirms the dominant presence of Li_4SiO_4 phase. Minor traces of secondary phases (e.g., SiO_2 or Li_2SiO_3) may appear. Sharp peaks indicates high crystallinity in sintered samples.Slight peak broadening in milled samples (indicating smaller crystallite size). The material retains its desired phase stability while milling introduces defects and smaller crystallites, which enhance reactivity of the material.

The combined analysis of SEM, BET, and XRD results clearly demonstrates that the particle size reduction (via milling) significantly improves the CO_2 capture efficiency of Li_4SiO_4. Surface area and morphology play a more critical role than crystallinity alone. An optimal balance between high surface area (milled) and structural stability (sintered) is essential for practical applications. Therefore, nano-crystalline or milled lithium orthosilicate is highly effective for carbon capture technologies, especially in high-temperature industrial environments.

CO_2 Uptake and Kinetics

Theoretical Calculation:

Molar Mass of Li_4SiO_4 = 119.85 g/mol

Molar Mass of CO_2 = 44.01 g/mol

$$CO_2 \text{ uptake (wt\%)} = \frac{\text{Molar mass of } CO_2}{\text{Molar mass of absorbent}} \times 100$$

CO_2 wt% = (44.01 / 119.85) × 100 ≈ 36.7%

Thermal Gravimetric Method (400-580 °C Temperature Variation in 2 Hours):

$$Li_4SiO_4 + CO_2 \rightarrow Li_2CO_3 + Li_2SiO_3$$

$$CO_2 \text{ uptake (wt\%)} = \frac{\text{Weight after } CO_2 \text{ absorption} - \text{Weight before } CO_2 \text{ absorption}}{\text{Weight before } CO_2 \text{ absorption}} \times 100$$

Initial weight of Li_4SiO_4 = 1.000 g

Final weight after absorption (Li_2CO_3+Li_2SiO_3) at 580 °C ≈ 1.200 g

CO_2 wt% = [(1.200 - 1.000) / 1.000] × 100 = 20%

Uptake Performance

Theoretical vs. Actual: The theoretical capacity is 36.7 wt%. Experimental results showed 20 wt% uptake (200 mg per 1.0g sample) at 580°C within 2 hours.

Kinetic Interpretation: The uptake is diffusion-limited. The initial reaction is rapid on the surface, but further absorption is hindered by the formation of a product layer (Li_2CO_3/Li_2SiO_3), requiring slower solid-state diffusion.

Porosity: Incorporating silicates acts as a pore-forming agent. Degradation of these phases prior to the first run increases the surface-to-volume ratio, enhancing reactivity.

TABLE 2. A COMPARATIVE ANALYSIS OF PRESENT WORK WITH STATE OF THE ART (LCR & DIELECTRIC RESULTS)

Authors (Year)	Synthesis Method	Modifications / Additives	CO_2 Absorption Temp (°C)	Max CO_2 Uptake	Kinetics / Mechanism Insight	Key Findings
Nair & Raghavan (2021)	Ball milling	Li_4SiO_4 only	~650	~32 wt%	Avrami-Erofeev kinetics	Faster sorption/desorption; kinetics improved by ball milling
Kanki et al. (2016)	Ball milling	K_2CO_3 surface modifier (Potassium carbonate)	600	~35 wt%	Enhanced surface diffusion	Surface coating improves kinetics & thermal stability
Zhang et al. (2023)	Solid waste-derived	Solid waste (e.g., coal fly ash, slag-derived precursors)	600–750	30–34 wt%	Stable cycling	Eco-friendly, cost-effective, stable sorbents
Puccini et al. (2023)	Ball milling + pelletization	mechanical activation only	650–700	~33 wt%	Enhanced kinetics	Mechanical activation improves reactivity
Present Work	Ball milling (10h, BPR 10:1), pelletization	Li_2CO_3 + SiO_2 used; compared with Li_2TiO_3	400–580	200 mg/2h (~20 wt%)	Bulk-controlled impedance, dielectric studies	Sensor-grade pellets, high dielectric constant (up to 60,000), improved ionic mobility at high T

5. Conclusion

This study focused on developing novel Li_4SiO_4-based sorbents in the form of pellets for CO_2 capture at high temperatures (400–580 °C) and low CO_2 concentrations. The pellets were created

through a mechanical pelletization method using lithium silicate (Li_4SiO_4) powder. To enhance the porosity of the structure, silicates were incorporated, which was confirmed by the pellet morphology. The enhanced results are attributed to the increased porosity of the pellets, which was a result of silicate degradation in air prior to the first sorption run. This increased surface area led to a higher sorption capacity. Optimal conditions for the highest dielectric response are established as 10 h HEBM followed by 1000°C calcination and 1050°C sintering (yielding epsilon (ε) = approx 192,860 at 1 kHz).Additionally, no significant difference in CO_2 adsorption capacity was found when comparing pellets of varying sizes. The inclusion of silicates improves the CO_2 sorption capacity of the Li_4SiO_4-based pellets. Further study shown that the enhancement in absorption ability of silicates specifically; at 580 °C the nano-crystalline Li_4SiO_4 sorbents of 6 mm in diameter and 3.5 mm in thickness made with 20 wt% silicates can uptake approximately 200 mg CO_2 in 2 hours from 400-580 °C temperature variation.

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Ethics, Consent to Participate, and Consent to Publish declarations: Not Applicable.

Declaration: The datasets generated during and/or analysed during the current study are available from the corresponding author on reasonable request.

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