

INFLUENCE OF PRECURSOR CHEMISTRY ON PHASE FORMATION AND MORPHOLOGY OF SOL-GEL DERIVED BAGHDADITE

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Received date: Jan. 26th 2026; Accepted date: March 16th 2026; Published date: Sept. 30th 2026

Ca₃ZrSi₂O₉, commonly known as baghdadite, is a bioactive ceramic material with great potential in bone tissue engineering applications. In this study, baghdadite powders were synthesized using a sol-gel method with different combinations of calcium (nitrate, chloride) and zirconium (nitrate, chloride) precursors, followed by thermal treatment at 900°C. Phase formation, morphology, and surface characteristics were evaluated using BET surface area analysis, FT-IR spectroscopy, SEM-EDS, and X-ray diffraction. The combination of calcium nitrate and zirconium chloride demonstrated the most favorable baghdadite phase development. These results show that the choice of precursors is important for achieving phase uniformity and the desired texture, which are necessary for future processing and scaffold use.

Keywords: baghdadite, sol-gel, precursor chemistry, phase development.

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1. Introduction

The engineering of bone tissue holds great promise for the future treatment of critical-size bone defects and for improving healing in orthopedic applications. The choice of scaffold material is one of the most critical factors in this field because it must promote bioactivity and possess sufficient mechanical strength to support adherence, proliferation, and differentiation of bone cells [1]. Bioactive ceramics such as hydroxyapatite (HA) have long been seen as the gold standard because of their chemical similarity to natural bone minerals. However, HA possesses intrinsic weaknesses, including low fracture toughness, brittleness, and

slow in vivo resorption rates, which may impede the long-term integration and mechanical stability in load-bearing environments [2].

Calcium-silicate bioceramics (which include, but are not limited to, wollastonite, larnite, and diopside) have demonstrated potential to be viable alternatives because they can form apatite layers in the body, release biologically active ions like Ca^{2+} and SiO_4^{4-} and also promote the development of bone-forming cells [3]. However, these materials often have issues with phase consistency, mechanical strength, and controllable degradation rates, so they are usually not used for load-bearing or major bone repairs.

In this context, the ternary calcium-zirconium silicate phase, baghdadite ($\text{Ca}_3\text{ZrSi}_2\text{O}_9$), has attracted notable attention. According to the literature, compared with simple Ca-Si ceramics, silicate materials containing Zr^{4+} exhibit better stability, more controlled dissolution, improved mechanical properties, and enhanced cell-material interactions [4]. Studies reported in recent years demonstrate that baghdadite is a highly bioactive material capable of supporting osteoblast-related functions and has been successfully applied in multiple bone tissue engineering strategies, including scaffolds, cements and coatings [5-7].

Many methods have been explored and reported in the literature for baghdadite synthesis, but the sol-gel process stands out because it offers several advantages, such as the ability to produce fine particle sizes, good chemical uniformity, low synthesis temperature, and good control over porosity and shape [8]. In sol-gel systems, the choice of precursors (nitrates, chlorides, acetates) has a significant impact on the chemistry, reactivity, impurities and the final structure of the product [9]. Previous studies show that even small variations in precursor type and solution chemistry can result in secondary silicate or zirconate phases, insufficient phase transformation, and even residual contaminants such as chlorine or nitrates [10].

Although recent studies on baghdadite ceramics [11-16] have been performed, significant knowledge gaps continue to exist about the systematic effects of calcium and zirconium precursor chemistry on phase development, morphology, chemical homogeneity, and microstructure of sol-gel-derived powders. Learning more about how these precursors interact and break down is important for making pure, accurate powders with the right texture and shape for scaffold manufacturing.

This work evaluated the influence of various calcium and zirconium precursors on the phase formation and morphology of sol-gel-derived baghdadite powders. Nanostructured baghdadite powders were synthesized using different calcium (nitrate, chloride) and zirconium (nitrate, chloride) precursors, followed by thermal treatment at 900°C. Phase formation, morphology, and chemical composition were evaluated using BET surface area analysis, FT-IR spectroscopy, SEM-EDS, and X-ray diffraction.

2. Materials and Methods

2.1 Materials

Analytical-grade reagents were used in synthesizing baghdadite powders. Hydrated zirconyl nitrate ($\text{ZrO}(\text{NO}_3)_2 \cdot x\text{H}_2\text{O}$, Sigma-Aldrich, made in USA) and zirconium tetrachloride (ZrCl_4 , Merck, made in France) were employed as zirconium sources, while calcium nitrate tetrahydrate ($\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$, Sigma-Aldrich, made in India) and calcium chloride dihydrate ($\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$, Sigma-Aldrich, made in Germany) provided the calcium component. Tetraethyl orthosilicate (TEOS, Sigma-Aldrich, made in China) served as the silicon precursor, with ethanol as the solvent and distilled water as the hydrolysis medium. Nitric acid (HNO_3) was used as a catalyst, and all reagents were used as received without further purification.

2.2 Sol-gel synthesis

The sol-gel route for baghdadite synthesis and the corresponding steps are presented in Fig.1.

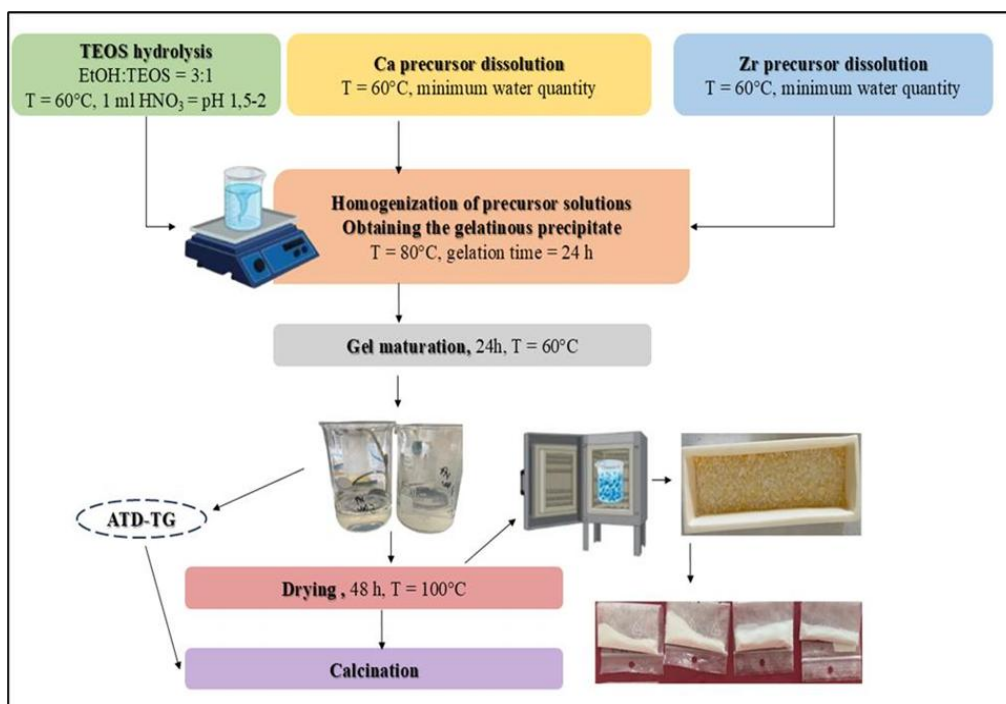


Figure 1. Schematic representation of the sol-gel method for baghdadite synthesis.

Following this route, starting from different precursor combinations (presented in Table 1), four synthesis experiments were performed to obtain baghdadite powder (noted as C₃ZS₂-1, C₃ZS₂-2, C₃ZS₂-3 and C₃ZS₂-4).

Table 1. Precursor combination used for the sol-gel synthesis of baghdadite powders.

Sample code	Precursors			Thermal treatment
C ₃ ZS ₂ -1	Ca(NO ₃) ₂ *4H ₂ O	ZrO(NO ₃) ₂ *XH ₂ O	TEOS	900°C/2h
C ₃ ZS ₂ -2	Ca(NO ₃) ₂ *4H ₂ O	ZrCl ₄	TEOS	900°C/2h
C ₃ ZS ₂ -3	CaCl ₂ *2H ₂ O	ZrO(NO ₃) ₂ *XH ₂ O	TEOS	900°C/2h
C ₃ ZS ₂ -4	CaCl ₂ *2H ₂ O	ZrCl ₄	TEOS	900°C/2h

2.3 Characterization methods

Chemical composition: The silicon content of all samples was assessed by gravimetric chemical analysis according to the SR ISO 2598-1:1998 standard. The calcium (Ca) content was determined by flame atomic absorption spectroscopy (FAAS) following the STAS 1269/17-82 standard, with the AAS ZEEnit 700, Analytik Jena AG equipment (Germany), while the zirconium (Zr) was analyzed by inductively coupled optical emission spectrometry (ICP-OES) with an Agilent 725 ICP-OES (USA) spectrometer based on the ASTM E1479-24 standard.

Fourier Transform Infrared Spectroscopy (FT-IR) measurements were made on an ABB MB 3000 FT-IR spectrometer (ABB Inc., Quebec, QC, Canada). The powder analysis was performed with an EasiDiff accessory (PIKE Technologies, Inc., Madison, WI, USA). The powder samples were prepared by mixing 1 wt.% of the solid with KBr. Spectra were collected in transmittance mode over the range of 550-4000 cm⁻¹ with 64 scans recorded at a resolution of 4 cm⁻¹. Experimental data were examined using Horizon MBTM FTIR software version 3.4.0.3.

X-ray diffraction measurements were made using a Bruker AXS D8 ADVANCE equipment (Bruker AXS GmbH, Karlsruhe, Germany) in Bragg-Brentano geometry. The instrument was equipped with a Cu radiation source and a SOL X vertical geometry detector. All measurements were performed by continuous scanning between 4 and 74 ° using a step size of 0.02°. Phase identification was conducted through analyzing the obtained diffraction patterns with DIFFRAC.EVA software version 5.2019 with reference to the ICDD PDF-5+2025 database.

The morphology of the powder samples was evaluated using a high-resolution scanning electron microscope (SEM), Quanta FEI 250 (FEI Company, Eindhoven, Netherlands). The instrument is equipped with an Energy Dispersive X-ray Spectrometer (EDS) from EDAX (Mahwah, NJ, USA), featuring an

ELEMENT Silicon Drift Detector and the Element EDS Analysis Software Suite APEX version 1.0.

BET specific surface measurement: The specific surface area was evaluated by N₂ gas physisorption at 77K (-196°C) using adsorption-desorption isotherms. The measurements were performed in a Micromeritics TriStar II Plus analyzer (Micromeritics Instrument Corporation, Norcross, GA, USA). The specific surface area was established by the Brunauer-Emmett-Teller (BET) method, and before analysis, the powder samples were thermally treated at 300°C for several hours to remove remaining water and impurities, using VacPrep 061 degassing stations.

3. Results and Discussion

3.1 Chemical composition

The measured amounts of Ca, Zr, and Si presented in Table 2 show small differences from the theoretical stoichiometry of baghdadite (Ca₃ZrSi₂O₉), indicating the importance of the precursor choice in obtaining the desired composition.

Table 2. Chemical composition analysis results.

Sample code	Ca, %	Zr, %	Si, %	Calculated formula
Theoretical baghdadite	29.2	22.2	13.6	Ca ₃ ZrSi ₂ O ₉
C ₃ ZS ₂ -1	25.86	18.63	9.75	Ca _{2.66} Zr _{0.84} Si _{1.43} O ₉
C ₃ ZS ₂ -2	28.25	23.03	11.44	Ca _{2.9} Zr _{1.04} Si _{1.68} O ₉
C ₃ ZS ₂ -3	26.60	15.40	13.15	Ca _{2.73} Zr _{0.69} Si _{1.93} O ₉
C ₃ ZS ₂ -4	20.50	18.90	16.50	Ca _{2.11} Zr _{0.85} Si _{2.43} O ₉

Among all samples, C₃ZS₂-2 exhibits the best correspondence with the theoretical baghdadite formula, as reflected by its calculated composition Ca_{2.9}Zr_{1.04}Si_{1.68}O₉, which indicates a nearly stoichiometric material.

In contrast, C₃ZS₂-1 has a small shortage of Ca and Zr, which could lead to more vacancies or defects in the crystal structure. C₃ZS₂-3 and especially C₃ZS₂-4 show bigger differences, with C₃ZS₂-4 having much more silicon than expected. This extra silicon content might help with surface chemical activity and hydroxyapatite formation in lab tests, but it could also change the mechanical properties and how the material densifies compared to ideal baghdadite.

This evidence suggests that small changes in Ca, Zr and Si content can help adjust material properties for specific uses, such as bone tissue engineering in which controlled ion release, porosity and bioactivity are essential factors.

3.2 Fourier transform infrared spectroscopy (FT-IR) results

FT-IR spectra of the samples thermally treated at 900°C for 2 hours are shown in Fig.2. All samples have a clear band near 3400 cm⁻¹ attributed to the stretching vibrations of hydroxyl groups (-OH) and a band around 1640 cm⁻¹, which corresponds to the bending vibration of water molecules (H-O-H), indicating the presence of residual or weakly bound water within the structure. C₃ZS₂-1, C₃ZS₂-2 and C₃ZS₂-3 each display a band between 1380-1450 cm⁻¹, presumably from nitrate (NO₃⁻) groups, meaning that some nitrogen-containing precursors are still present and unreacted.

This implies that the calcination process is incomplete and may require optimization to achieve full removal of these residual species.

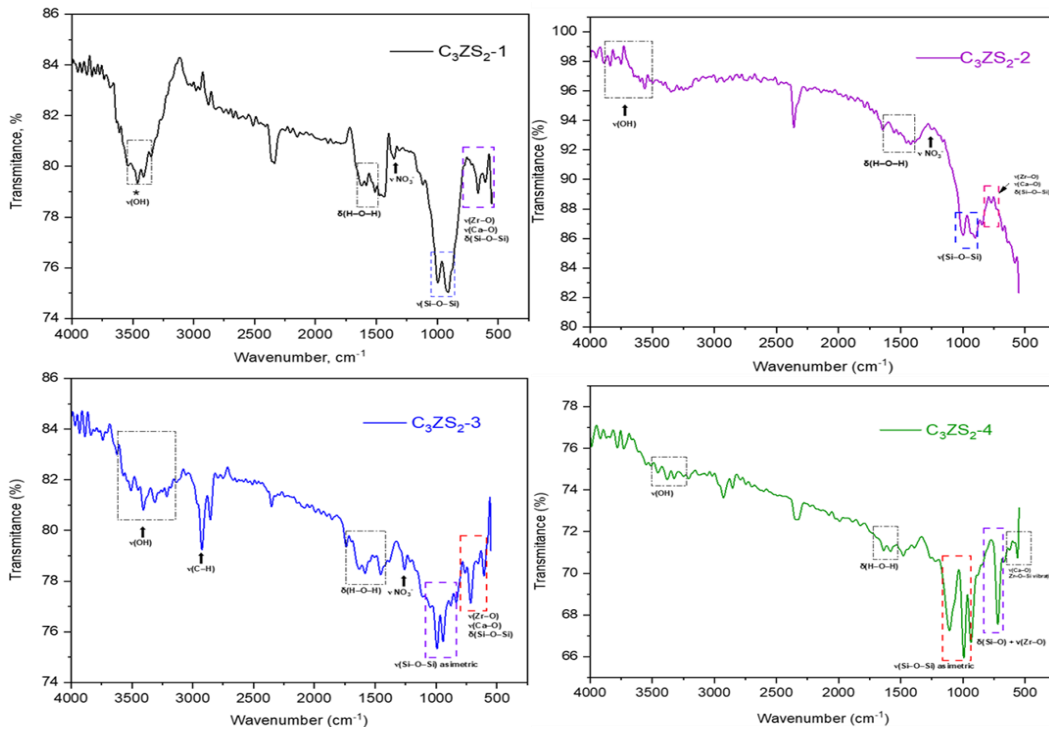


Figure 2. FT-IR spectra for C₃ZS₂ samples calcined at 900°C.

In sample C₃ZS₂-3, a weak absorption between 2920-2850 cm⁻¹ was detected, presumably from C-H groups. This suggests some leftover organic compounds. The fingerprint region (1200-400 cm⁻¹) is dominated by strong and clearly defined bands typical for silicate networks: bands in the 1100-900 cm⁻¹ range correspond to symmetric and asymmetric Si-O-Si stretching vibrations, while bands in the 750-500 cm⁻¹ range are attributed to metal-oxygen vibrations (Zr-O and Ca-O) and Si-O-Si network bending modes.

3.3 X-ray diffraction analysis results

Qualitative phase analysis by X-ray diffraction (XRD) of the C_3ZS_2 samples, as shown in Fig.3, revealed multiple crystalline phases within the CaO - ZrO_2 - SiO_2 ternary system, with particular emphasis on identifying the target phase, baghdadite ($Ca_3ZrSi_2O_9$ or $Ca_3Zr(Si_2O_7)_2$). Phase analysis was carried out on all samples using the DIFFRAC.EVA 2019 software and the ICDD PDF-5+2025 database. For clarity, some diffractograms are presented in a reduced 2θ range focusing on the most relevant diffraction peaks.

In order to better assess the relative proportion of the crystalline phases, a semi-quantitative phase estimation was performed based on the relative intensities of the characteristic diffraction peaks of the identified phases. For sample C_3ZS_2 -1, baghdadite $Ca_3Zr(Si_2O_7)_2$ (PDF 04-009-2584) reflections exhibit low relative intensities compared to those of secondary phases such as lakargite $CaZrO_3$ (PDF 01-080-6212) and the tetragonal $Ca_xZr_{1-x}O_2$ solid solution (PDF 04-016-3766), indicating that baghdadite is present only as a minor phase.

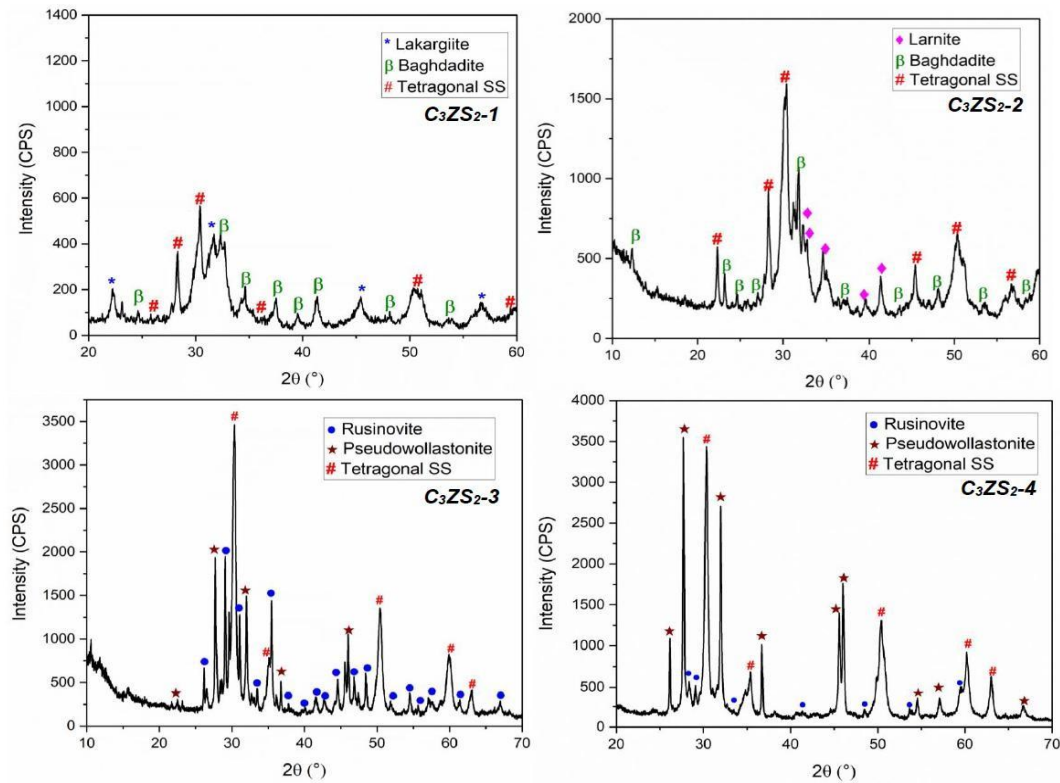


Figure 3. X-ray diffraction patterns for C_3ZS_2 samples.

This suggests incomplete phase development, likely due to limited precursor reactivity and local compositional imbalance.

In contrast, sample C₃ZS₂-2 shows a noticeable increase in the relative intensity of baghdadite diffraction peaks, indicating an improved formation of the target Ca₃ZrSi₂O₉ phase. However, secondary phases are still present (tetragonal solid solution Ca_xZr_{1-x}O₂ and larnite Ca₂SiO₄ (PDF 00-033-0302), suggesting partial rather than complete phase stabilization.

The diffraction patterns for C₃ZS₂-3 and C₃ZS₂-4 samples show no detectable baghdadite peaks. Instead, the most significant phases such as rusinovite Ca₁₀(Si₂O₇)₂Cl₂ (PDF 01-088-7389), pseudowollastonite CaSiO₃ (PDF 01-080-9543), and a tetragonal solid solution (Ca_xZr_{1-x}O₂) indicate a compositional mismatch and a possible oversaturation in Ca-Si species.

The formation of rusinovite is most likely associated with residual chlorine and silicon excess from incompletely reacted precursors.

This evidence further demonstrates that the chemistry of calcium and zirconium precursors is critical in phase development, governing the competition between baghdadite formation and the stabilization of secondary silicate or zirconate phases.

3.4 Powder morphology

The SEM images and corresponding EDS results presented in Fig.4 demonstrate that the morphology and microstructure of the C₃ZS₂ samples are strongly influenced by the type of precursors used. Sample C₃ZS₂-1 exhibits a well-defined microstructure characterized by polygonal grains.

However, EDS analysis shows a considerable excess of calcium and lower levels of silicon and zirconium, suggesting the formation of Ca-rich secondary phases, such as CaZrO₃. This composition suggests that the reactivity of the silicate precursors is low and phase transformation for the target Ca₃ZrSi₂O₉ structure is missing.

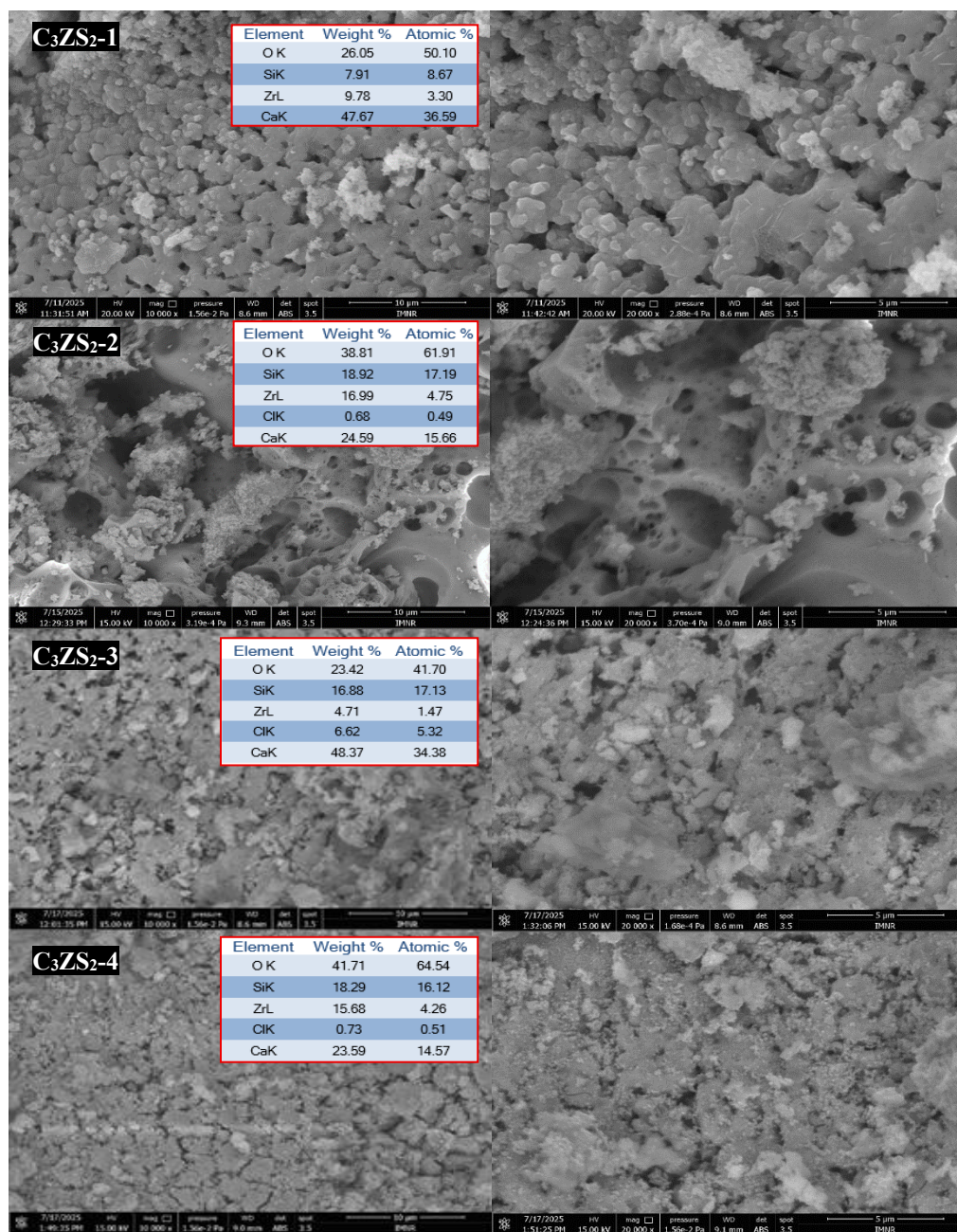


Figure 4. SEM-EDS results for C₃ZS₂ calcined samples: left – mag. x10 000, right – mag. x20 000.

C₃ZS₂-2, on the other hand, has a porous network and a chemical composition significantly corresponding to the theoretical stoichiometry of Ca₃ZrSi₂O₉, suggesting partial crystallization of the desired phase. Although C₃ZS₂-3 shows a more homogeneous fine-particle morphology, its composition

indicates zirconium deficiency and excess calcium and chlorine, suggesting residual precursor impurities. These results indicate that the selected precursors did not effectively interact to form a pure baghdadite phase.

In the case of the C_3ZS_2 -4 sample, a homogeneous texture and an apparently balanced elemental composition are observed. However, the presence of residual chlorine identified by EDS suggests that chloride-containing precursors were not entirely eliminated, thereby preventing full formation of the desired baghdadite phase.

Overall, these results show that choosing the right precursors is necessary to control both the phase and the structure of sol-gel-derived baghdadite ceramics.

3.5 BET specific surface

Table 3 summarizes the specific surface area, pore parameters, and density values of the investigated C_3ZS_2 powders. Among the analyzed samples, C_3ZS_2 -2 achieves a well-balanced combination of moderate surface area ($5.43 \text{ m}^2/\text{g}$) and reduced particle size (325 nm), which is useful for ensuring sufficient surface interaction with cells while maintaining mechanical stability.

Table 3. BET specific surface analysis results for $Ca_3ZrSi_2O_9$ powders.

Sample code	BET specific surface area, m^2/g	Particle size, nm	Pore volume, cm^3/g	Pore size, nm	Density, g/cm^3
C_3ZS_2 -1	4.37	395	0.0018	1.11	3.47
C_3ZS_2 -2	5.43	325	0.0021	1.09	3.39
C_3ZS_2 -3	4.08	427	0.0015	1.12	3.44
C_3ZS_2 -4	7.25	231	0.0028	1.07	3.57

The C_3ZS_2 sample densities, measured using a pycnometer attached to the Micromeritics Tristar II Plus surface area analyzer, showed no significant difference, with a small increase from 3.39 to $3.57 \text{ g}/\text{cm}^3$.

The pore volumes measured range from 0.0015 to $0.0028 \text{ cm}^3/\text{g}$, and the average pore diameter varies between 1.09 and 1.12 nm, indicating the existence of micropores. These results match the nitrogen adsorption-desorption data (Fig.5), which show type III behavior and confirm the presence of microporous networks.

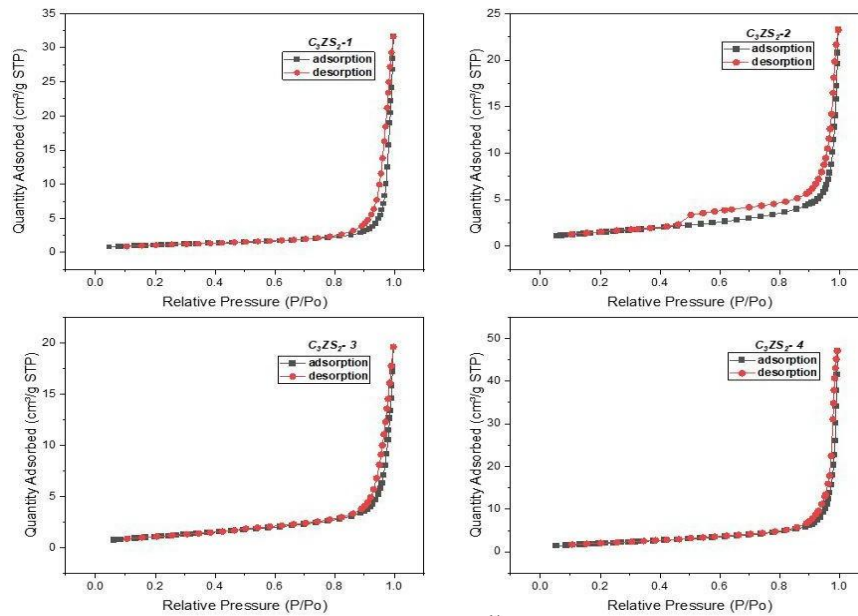


Figure 5. Adsorption-desorption isotherms for the baghdadite powder samples.

Such textural features are typical for sol-gel-derived ceramic powders subjected to thermal treatment and are favorable for subsequent densification and scaffold processing. Overall, these results show that the choice of precursors has a big effect on the texture and structure of baghdadite powders. The C_3ZS_2 -2 sample demonstrates the most favorable morphological and textural properties, combining microporosity, suitable for bone tissue engineering applications, with mechanical stability appropriate for scaffold fabrication.

4. Conclusions

Baghdadite ($Ca_3ZrSi_2O_9$) ceramic powders were synthesized using a sol-gel approach with different combinations of calcium and zirconium precursors. The results show that the chemical nature of these precursors is critical in determining phase formation, morphology, and even distribution of elements. Of all the tested combinations, the one prepared from calcium nitrate and zirconium chloride (C_3ZS_2 -2) reached the best results, with a near-stoichiometric $Ca_3ZrSi_2O_9$ formula, a higher relative proportion of the baghdadite phase compared to the other samples, and a porous morphology suitable for bone scaffold applications. However, it was found that using only chloride-based precursors led to unwanted silicate and chlorine-containing phases (such as rusinovite) and residual chlorine.

FT-IR and XRD characterization results show that not all nitrate and chloride are removed during heating, thereby preventing full development of the baghdadite phase. This happens because different precursors break down in

different ways, producing by-products that alter the local composition and lead to the crystallization of other phases. To fix this, it may be necessary to adjust the heating temperature, dwell time, or atmosphere. Despite the success of the sol-gel synthesis, multiple difficulties remain, including controlling phase quality, preventing residual chlorine incorporation, and achieving gel homogeneity at larger scales.

To summarize, this study demonstrated that choosing suitable precursors and making sure they work well together are essential for obtaining single-phase baghdadite ceramics. Subsequent work will emphasize improving synthesis conditions, trying new precursor combinations, and adjusting heating steps. Further studies will also test the strength and bioactivity of scaffolds made from these powders to see if they are suitable for bone tissue engineering use.

Author Contributions

Conceptualization, S.C. and L.M.C; methodology, S.C.; validation, L.M.C. and G.V.; formal analysis, G.F.I; investigation, S.C, G.F.I, M.A.I, D.V.D, E.B; writing—original draft preparation, S.C.; writing—review and editing, L.M.C, G.V., S.P.S.; supervision, L.M.C and G.V.; funding acquisition, L.M.C. All authors have read and agreed to the published version of the manuscript.

Data Availability Statement

Data supporting the findings of this study are available upon reasonable request from the corresponding author.

Funding

This research was funded by the Ministry of Research, Innovation and Digitization (MCID), Core Program 5N/01.01.02023-ENERCLEAN, grant number PN 23250201/2023.

Conflicts of Interest

The authors declare no conflict of interest.

Artificial Intelligence Disclosure

No artificial intelligence-based or generative tools were used in the preparation of this manuscript.

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