

Influence of block copolymer (P123) concentration on the morphology and pore structure of calcium silicate materials

Phi Hung Dao¹, Thuy Chinh Nguyen^{1,2}, Thi Huong Nguyen³,
Thao Linh Bui² and Hoang Thai^{1,2†}

¹*Institute of Materials Science, Vietnam Academy of Science and Technology, 18 Hoang Quoc Viet, Nghia Do, Ha Noi, 10000, Vietnam*

²*Graduate University of Science and Technology, Vietnam Academy of Science and Technology, 18 Hoang Quoc Viet, Nghia Do, Ha Noi, 10000, Vietnam.*

³*Hanoi University of Industry, 298 Cau Dien, Tay Tuu, Ha Noi, 10000, Vietnam*

E-mail: [†]hoangth@ims.vast.vn/hoangvast@gmail.com

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Abstract. *In this study, porous calcium silicate (CaSiO_3) powders were synthesized using a sol-gel approach combined with hydrothermal treatment and subsequent calcination. Different concentrations of the nonionic surfactant P123 (10 wt.%, 20 wt.%, and 30 wt.%) were employed to evaluate their influence on the structural and textural properties of the materials. X-ray diffraction (XRD) and Fourier-transform infrared spectroscopy (FTIR) were used to confirm the formation of $\beta\text{-CaSiO}_3$ and to compare the structural features between the control sample (Ca0) and the P123-assisted sample (Ca10). Scanning electron microscopy (SEM) and nitrogen adsorption-desorption (BET/BJH) analyses were performed for all samples to assess morphology, porosity, and surface area. The obtained results show that Ca10 exhibits the most desirable textural characteristics, with a high surface area ($117.3 \text{ m}^2/\text{g}$) and well-developed mesoporosity. Increasing P123 content to 20 wt.% further enhances pore volume but results in partial structural densification, whereas excessive surfactant loading (30 wt.%) leads to pore collapse and a significant decrease in surface area. These findings demonstrate that appropriate control of P123 content is crucial for optimizing the mesoporous architecture of CaSiO_3 , offering potential benefits for applications such as adsorption, catalysis, and functional coatings.*

Keywords: calcium silicate (CaSiO_3); mesoporous materials; sol-gel synthesis; P123 surfactant; BET surface area; pore size distribution.

Classification numbers: 81.05.Rm; 81.20.Rw; 81.40.Pq.

1. Introduction

Porous materials are a class of substances distinguished by the presence of pores with varying sizes and distributions, which enable them to adsorb, store, and release a broad range of molecules [1]. Based on pore size, they are typically classified into three categories: microporous (< 2 nm), mesoporous (2–50 nm), and macroporous (> 50 nm). The incorporation of a porous architecture significantly increases the specific surface area, enhances diffusion properties, and improves interfacial interactions, thereby making these materials highly attractive for applications in catalysis, adsorption, ion exchange, energy storage, sensing, biomedicine, and other advanced functional technologies [2].

Representative examples include porous carbons [2], zeolites [3], mesoporous silica [4], metal-organic frameworks (MOFs) [5] and so on. Owing to their tunable pore structures and high surface areas, these materials are extensively employed as carriers in controlled-release systems, such as drug delivery [6] and corrosion inhibition [7]. Their ability to modulate release rates in response to environmental stimuli-including pH, temperature, and ion concentration-makes them especially valuable for smart and responsive applications [6, 7].

Calcium silicate (CaSiO_3) is an inorganic material with promising potential in both industrial and biomedical fields due to its chemical stability [8], biocompatibility [8–10], and structural versatility [8]. However, conventional CaSiO_3 typically exhibits dense structures, low specific surface areas, and poor diffusion capacity, which limit its efficiency in adsorbing and releasing active agents [11]. In recent years, porous CaSiO_3 has attracted growing interest because of its high surface area, enhanced adsorption capacity, and tunable porosity-characteristics that allow it to fulfill various functional roles [12]. The development of porous CaSiO_3 aims to expand its applications in areas such as adsorption, controlled release, corrosion protection, catalysis, and biomedical engineering [13, 14].

Porous materials can be synthesized using a variety of techniques, including wet precipitation [15], sol-gel processes [16], hydrothermal methods [17], and templating strategies involving surfactants or structure-directing agents [18]. Among them, the use of structure-directing agents such as P123-a triblock copolymer of polyethylene oxide (PEO) and polypropylene oxide (PPO)-has proven particularly effective for generating well-ordered, uniformly distributed mesopores. The P123-assisted synthesis approach greatly enhances the specific surface area and porosity of the resulting material. Consequently, the porous materials fabricated using P123 typically exhibit a homogeneous pore structure, rendering them ideal for the encapsulation and environmentally responsive release of active molecules triggered by changes in pH, temperature, or ionic strength [19, 20]. However, although P123 has been widely used for mesoporous silica and metal oxides, its application in tailoring the microstructure of CaSiO_3 remains relatively underexplored. In particular, the influence of varying P123 content on the pore architecture, surface characteristics, and functional performance of CaSiO_3 has not been systematically studied. In this context, our work provides new insights into the role of P123 as a structure-directing agent in the synthesis of porous CaSiO_3 . By adjusting the P123 content, we aim to tailor the pore morphology and optimize the material for potential use in encapsulation and controlled release systems. These findings are expected to support the design of smart CaSiO_3 -based carriers for advanced applications such as corrosion protection, drug delivery, and responsive functional coatings.

This study presents the synthesis and characterization of porous CaSiO_3 materials with varying P123 content, and evaluates their structural properties and potential for responsive molecule release under different environmental stimuli.

2. Experiment

2.1. Materials

This work used calcium nitrate tetrahydrate ($\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$, $\geq 99\%$ purity), tetraethyl orthosilicate (TEOS, $\geq 98\%$), Pluronic® P123, NaBH_4 and AgNO_3 , all purchased from Sigma-Aldrich. Some other chemicals including distilled water, acetic acid (CH_3COOH), nitric acid (HNO_3), ethanol ($\text{C}_2\text{H}_5\text{OH}$), and sodium hydroxide (NaOH), were obtained from Duc Giang Chemicals Company (Vietnam).

2.2. CaSiO_3 preparation

CaSiO_3 powder was prepared using a sol - gel process followed by hydrothermal treatment and high-temperature calcination. To begin, 118.1 g of calcium nitrate tetrahydrate ($\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$) was dissolved in 180 mL of distilled water to supply the Ca^{2+} ions. Concurrently, 114.3 g of tetraethyl orthosilicate (TEOS), serving as the silica precursor, was dissolved in 270 mL of ethanol. A mixture of nitric acid (HNO_3), acetic acid (CH_3COOH), and distilled water was added to the calcium solution to adjust its pH and facilitate TEOS hydrolysis. Separately, the triblock copolymer Pluronic P123 was dissolved in ethanol with ratio 1.5 mL ethanol/g P123, acting as a structure-directing agent to aid in pore formation (The precursor composition of CaSiO_3 was presented on Table 1). The TEOS-P123 mixture was slowly introduced into the calcium-containing solution under continuous stirring for 2 hours to form a homogeneous precursor gel. This gel was transferred into a Teflon-lined autoclave and subjected to hydrothermal treatment at 90°C for 24 hours to induce crystallization and develop the porous network. The formed solid was collected via filtration, rinsed thoroughly with distilled water and ethanol to eliminate residual reactants, and dried. Finally, the dried powder underwent calcination at 1150°C for 7 hours, with a gradual temperature ramp-up from room temperature, followed by natural cooling. This thermal treatment effectively removed the organic template (P123) and enhanced the crystallinity of the final CaSiO_3 material. The obtained CaSiO_3 materials were ground using an agate mortar and pestle before being used for subsequent characterization.

Table 1. The precursor composition of CaSiO_3 .

No	$\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$	TEOS	P123	Code
1	118.1g	114.3 g	0 g	Ca0
2	118.1 g	114.3 g	25.82 g	Ca10
3	118.1 g	114.3 g	38.88 g	Ca20
4	118.1 g	114.3 g	99.6 g	Ca30

2.3. Analysis

2.3.1. Fourier-transform infrared spectroscopy

Fourier-transform infrared (FTIR) spectroscopy was employed to identify the chemical functional groups of the CaSiO_3 samples. The powder samples were thoroughly mixed with spectroscopic-grade potassium bromide (KBr) at a weight ratio of 1:50, then pressed into transparent pellets using a hydraulic press under high pressure. Their FTIR spectra were recorded in the range of $4000 - 500 \text{ cm}^{-1}$ using a Nicolet iS10 spectrometer (Thermo Scientific) with a resolution of 2 cm^{-1} and 32 scans per sample.

2.3.2. X-ray diffraction patterns

The crystalline structure of the samples was analyzed using X-ray diffraction (XRD, Siemens D5000) equipped with a CuK_α radiation source ($\lambda = 1.5406 \text{ \AA}$), operated at 40 kV and 40 mA. The diffraction patterns were recorded over a 2θ range of $20^\circ - 80^\circ$, with a scanning rate of $2^\circ/\text{min}$ and a step size of 0.02° . The obtained patterns were compared with standard Powder Diffraction File (PDF) reference data for phase identification.

2.3.3. Morphology

The surface morphology of the CaSiO_3 particles was examined using a scanning electron microscope (SEM, JSM - IT800, JEOL, Japan). Prior to imaging, the samples were fixed onto aluminum stubs using conductive carbon tape and sputter-coated with a thin layer of gold to enhance conductivity and prevent charging during analysis. Their SEM images were obtained at an accelerating voltage of 5 - 15 kV under high-vacuum mode.

2.3.4. Surface area and pore volume

Nitrogen adsorption - desorption measurements were carried out to determine the specific surface area and porosity of the samples using the Brunauer-Emmett-Teller (BET) method. Prior to analysis, the samples were degassed under vacuum at 150°C for 12 hours to remove physically adsorbed species. Nitrogen adsorption-desorption isotherms were recorded at 77 K using a surface area and porosity analyzer (TriStar II 3020). The BET surface area was calculated from the adsorption data in the relative pressure (P/P_o) range of 0.05 to 0.30. Pore size distribution and total pore volume were evaluated using the Barrett-Joyner-Halenda (BJH) method based on the desorption branch of the isotherm.

3. Results and discussion

3.1. Fourier-transform infrared spectroscopy analysis

In order to evaluate the fundamental effect of P123 on the formation of the Ca-Si-O network FTIR analysis was conducted for two samples calcium silicate synthesized with and without P123, namely Ca0 and Ca10. The FTIR spectra of Ca0 and Ca10 samples are shown in Fig. 1, and the main FTIR absorption peaks of CaSiO_3 samples are summarized in Table 2.

As shown in Fig. 1 and Table 2, both Ca0 and Ca10 samples exhibit characteristic absorption bands associated with the vibrational modes of silicate and calcium-oxygen groups. A strong band at 1063 cm^{-1} corresponds to the asymmetric stretching vibration of Si-O-Si, which is a typical feature of silicate frameworks. Additional bands at 942 cm^{-1} and 796 cm^{-1} are attributed to Si-O stretching and bending vibrations, whereas the band at 779 cm^{-1} is assigned to the Si-O-Si bending

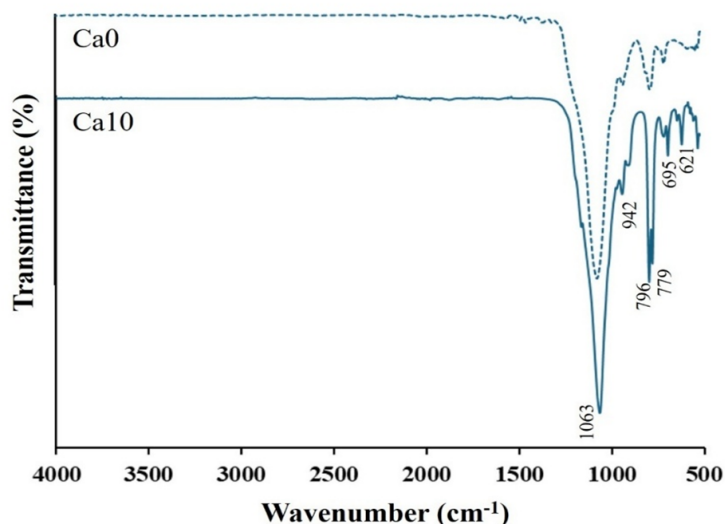


Fig. 1. FTIR spectroscopy of Ca0 and Ca10 samples.

Table 2. Main FTIR absorption peaks of Ca0 and Ca10 samples [13].

No	Absorption band (cm ⁻¹)	Corresponding vibration	Interpretation
1	1063	$\nu_{\text{Si-O-Si}}$ (asymmetric stretching)	Characteristic of silicate groups
2	942	$\nu_{\text{Si-O}}$ or $\nu_{\text{Si-O-Ca}}$	Bonding between SiO_4^{2-} and Ca^{2+} cations
3	796	$\delta_{\text{Si-O}}$ (bending)	Si-O groups in the network structure
4	779	$\delta_{\text{Si-O-Si}}$ (bending)	Bending vibration of silicate network
5	695	$\delta_{\text{O-Si-O}}$	Bending vibration of silicate groups
6	621	$\nu_{\text{Ca-O}}$ or $\delta_{\text{O-Si-O}}$	Ca-O bond vibration or ring vibration of silicate

mode, indicating the presence of a polymerized silicate network. Weaker absorptions at 695 cm⁻¹ and 621 cm⁻¹ correspond to O-Si-O bending and Ca-O stretching vibrations, respectively [13]. The absence of broad absorption in the 3200–3600 cm⁻¹ region indicates minimal hydroxyl (-OH) content, confirming that the materials are well-dried and contain negligible structural water. These features are consistent with the formation of crystalline calcium silicate with a wollastonite-type silicate network, consistent with previous reports on wollastonite-type materials [13,21].

For the Ca0 sample (synthesized without P123), the characteristic Ca-Si-O bands appear weaker and less defined, suggesting a lower degree of silicate condensation and polymerization. This implies that the resulting material possesses a more disordered structure in the absence of a

structure-directing agent. In contrast, the Ca10 sample (synthesized with P123) displays sharper and more intense FTIR peaks, particularly the strong band at 1063 cm^{-1} associated with asymmetric stretching of Si-O-Si/Si-O-Ca. The bands at 942 cm^{-1} , 796 cm^{-1} , 779 cm^{-1} , and the lower-wavenumber features at 695 cm^{-1} and 621 cm^{-1} are significantly more pronounced compared with the Ca0 sample, indicating enhanced silicate network formation. These results confirm that P123 effectively promotes the condensation and ordering of the silicate framework during synthesis.

3.2. X-ray diffraction patterns analysis

Figure 2 indicates the X-ray diffraction (XRD) pattern of the Ca0 and Ca10 samples as well as the detailed list of the main diffraction peaks, corresponding crystal planes, assigned phases, and their reference PDF numbers is summarized in Table 3.

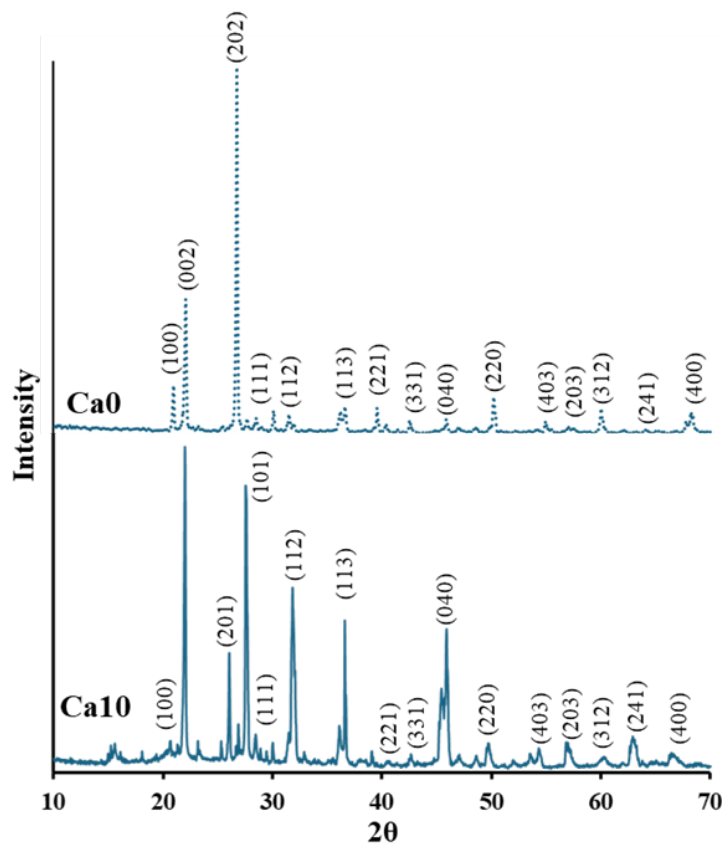


Fig. 2. X-ray diffraction patterns of Ca0 and Ca10 samples.

As shown in Fig. 2 and Table 3, both samples display the characteristic diffraction peaks of β -wollastonite (CaSiO_3 , monoclinic; PDF 84-0655). The main reflections, including (002), (101), (111), (112), (113), (221), (040), (220), (403), (203), (312), (241), and (400), confirm the successful formation of the wollastonite phase after calcination at 1150°C .

Table 3. Summary of experimental 2θ positions and corresponding crystal phases.

No	2θ	Crystal plane (hkl)	Assigned phase
1	20.9 °	(100)	Quartz (SiO ₂)
2	22.9 °	(002)	Wollastonite (CaSiO ₃)
3	26.0 °	(201)	Wollastonite (CaSiO ₃)
4	26.7 °	(202)	Wollastonite (CaSiO ₃)
5	27.6 °	(101)	Wollastonite (CaSiO ₃)
6	29.4 °	(111)	Wollastonite (CaSiO ₃)
7	32.1 °	(112)	Wollastonite (CaSiO ₃)
8	36.0 °	(113)	Wollastonite (CaSiO ₃)
9	40.6 °	(211)	Wollastonite (CaSiO ₃)
10	43.3 °	(221)	Wollastonite (CaSiO ₃)
11	45.9 °	(040)	Wollastonite (CaSiO ₃)
12	49.7 °	(220)	Wollastonite (CaSiO ₃)
13	54.6 °	(403)	Wollastonite (CaSiO ₃)
14	57.2 °	(203)	Wollastonite (CaSiO ₃)
15	59.9 °	(312)	Wollastonite (CaSiO ₃)
16	63.3 °	(241)	Wollastonite (CaSiO ₃)
17	68.9 °	(400)	Wollastonite (CaSiO ₃)

Marked differences, however, occur between the Ca0 and Ca10 samples. The Ca0 sample (without P123) exhibits broad, low-intensity peaks, indicating poor crystallinity, and shows an additional reflection at $\sim 20.9^\circ$, corresponding to the (100) plane of α -quartz SiO₂ (PDF# 46-1045), suggesting incomplete reaction of the silica precursor. In contrast, the Ca10 (with P123) presents sharp and intense CaSiO₃ peaks, demonstrating significantly enhanced crystallinity. The quartz-related peak at 20.9° is greatly reduced, evidencing more complete conversion of SiO₂ into CaSiO₃. This trend is consistent with FTIR results, where the Ca10 sample exhibits stronger Si-O-Si and Si-O-Ca bands, indicating improved silicate network development.

Overall, the XRD analysis results show that P123 plays a key role in promoting homogeneous precursor mixing and facilitating Ca-Si-O framework formation, leading to a purer and more ordered β -CaSiO₃ phase.

3.3. SEM image analysis

SEM images of synthesized powder CaSiO₃ samples with varying P123 content (Ca0, Ca10, Ca20, and Ca30 as shown in Table 1) with different magnification are displayed in Fig. 3. The microstructures exhibit clear morphological variations, confirming the pronounced influence of P123 on particle formation and surface development.

The Ca0 sample presents bulky particles with severe agglomeration, heterogeneous grain sizes, and a dense, low-porosity structure. In contrast, the incorporation of P123 leads to substantial changes in morphology. The Ca10 displays loosely aggregated particles with a highly porous network, rough surfaces, and irregular grain shapes. This structure indicates effective templating

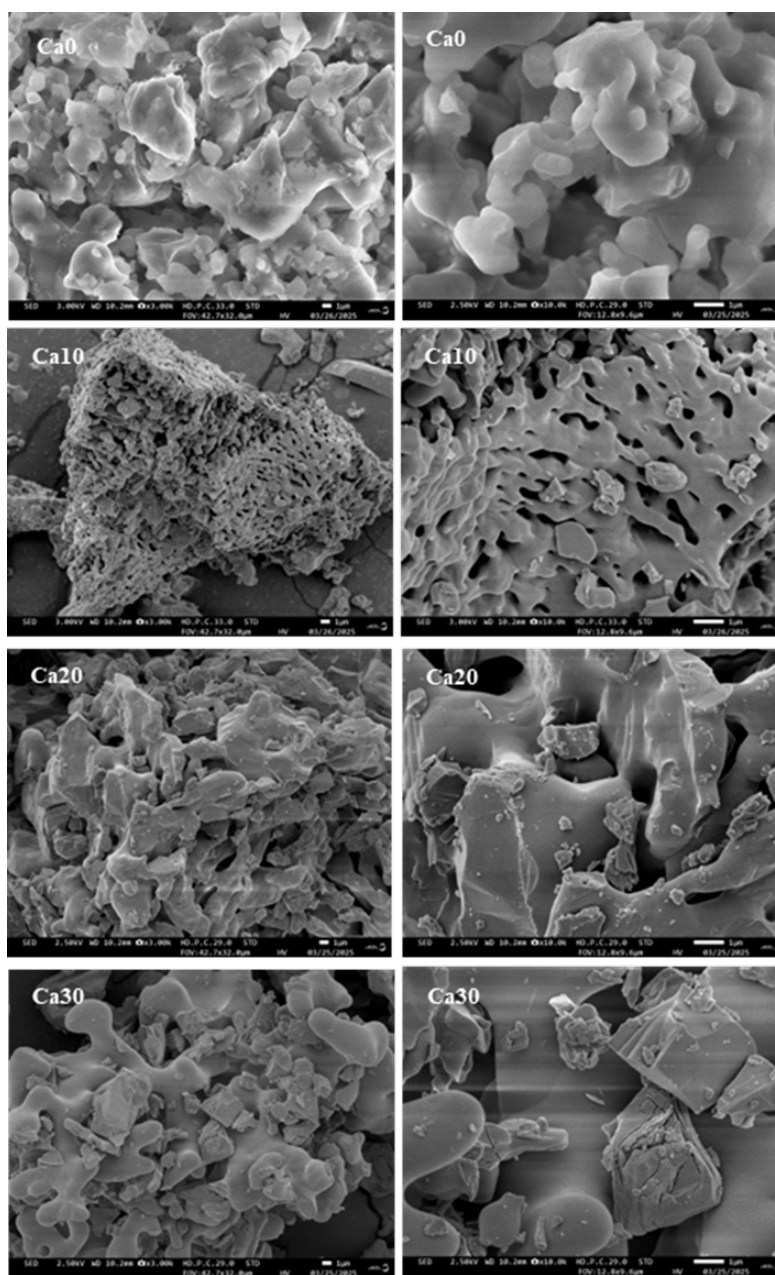


Fig. 3. SEM images of synthesized CaSiO_3 .

by P123 at this concentration, facilitating the formation of a high-surface-area material. As the P123 content increases, the Ca20 particles become denser and more compact, with smoother surfaces and partially fused grains. The reduction in porosity implies partial collapse or overgrowth

of the templated structure, likely due to increased micelle aggregation or insufficient space for pore development.

At the highest P123 content (the Ca30), the particles reveal a more crystalline-like and blocky morphology. The pores appear to diminish significantly, and the structure becomes more rigid and aggregated. This could be attributed to excessive surfactant interfering with controlled growth, leading to phase separation or crystal overgrowth during the synthesis process.

Overall, the SEM analysis demonstrates that the content of P123 plays a critical role in tuning the morphology and surface structure of CaSiO_3 . An optimal P123 level is essential to balance porosity and structural integrity for potential applications in adsorption, drug delivery, or coating materials.

3.4. Surface area and pore volume of materials analysis

The nitrogen adsorption-desorption isotherms and pore size distribution curves of CaSiO_3 samples synthesized with different P123 contents are demonstrated in Figures 4 and 5, respectively. The corresponding BET surface areas and pore structure parameters including cumulative pore volume and cumulative pore area are summarized in Table 4.

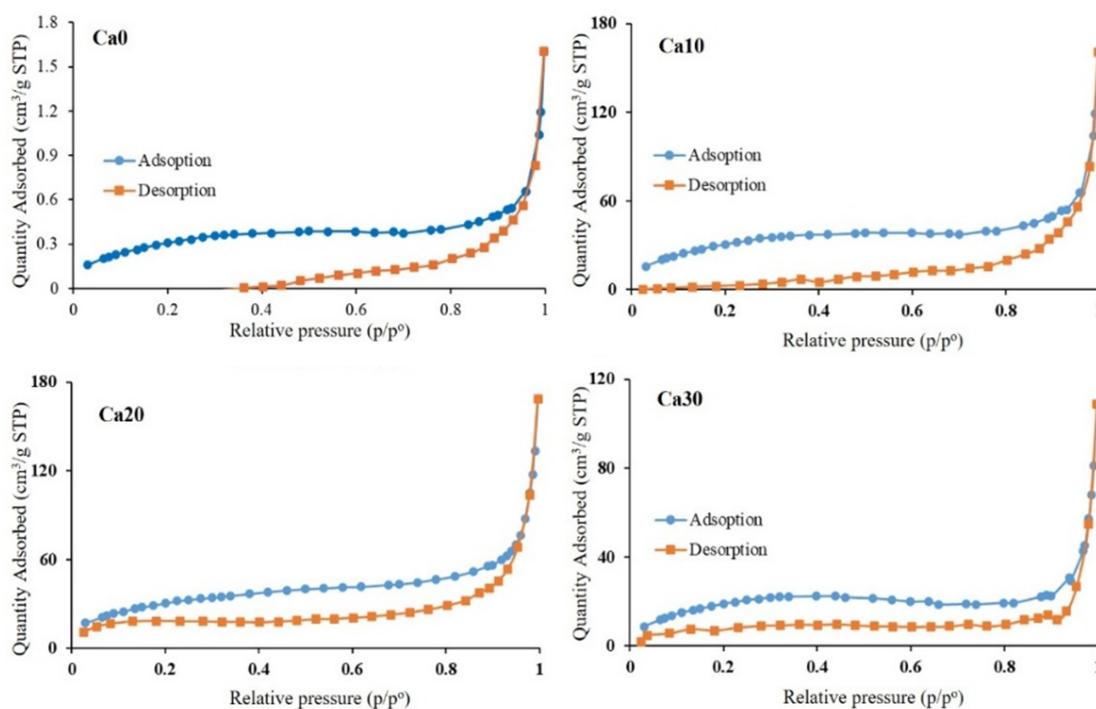


Fig. 4. Nitrogen adsorption-desorption isotherms of CaSiO_3 samples synthesized with different P123 contents.

As can be seen from Fig. 4, the Ca10 and Ca20 display a steep increase in nitrogen uptake at relative pressures (p/p^0) between 0.4 and 0.9, suggesting the presence of well-developed

mesopores. In contrast, the Ca0 sample (synthesized without P123) displays a very low total adsorption volume, reflecting its dense and low-porosity nature, where the limited porosity primarily originates from inter-granular macropores rather than controlled mesopores. Meanwhile, the Ca30 sample shows a significantly lower nitrogen adsorption capacity and a less pronounced hysteresis loop, indicating a reduced mesoporous structure likely due to excessive P123 interfering with proper pore formation.

The Ca10 sample exhibited the highest BET surface area ($117.27 \text{ m}^2/\text{g}$) and a relatively large cumulative pore area ($110.2 \text{ m}^2/\text{g}$), indicating a well-developed porous structure. The pore size distribution of Ca10 sample was predominantly within the mesoporous range (2-50 nm), with a strong concentration of pores between 2.3 - 3.5 nm, suggesting effective templating by a moderate amount of P123.

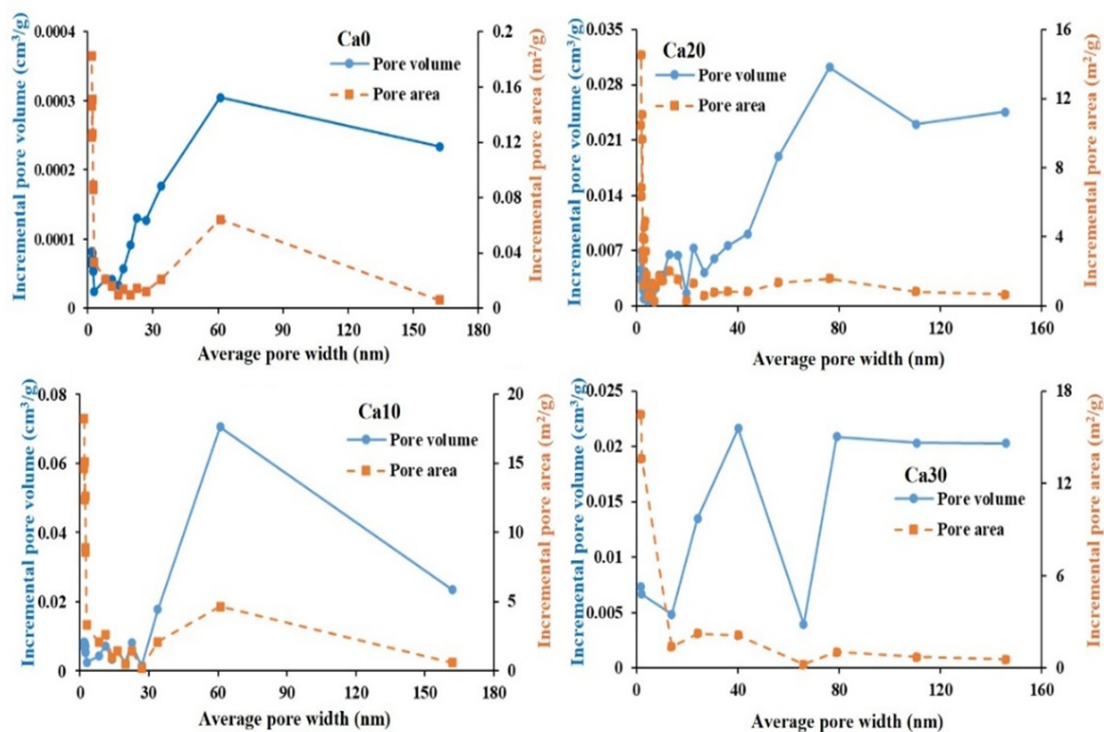


Fig. 5. Pore size distribution of CaSiO₃ synthesized with different P123 contents.

The Ca20 sample synthesized with a higher amount of P123, maintained a comparable cumulative pore area ($108.3 \text{ m}^2/\text{g}$) and slightly higher cumulative pore volume ($0.2136 \text{ cm}^3/\text{g}$) than the Ca10 sample, indicating enhanced pore interconnectivity and pore accessibility. Its pore size distribution was broader, extending from mesopores into larger meso/macropores, which may be beneficial for applications requiring high mass transfer.

In contrast, the Ca30 sample prepared with the highest P123 content, showed a significant drop in surface area ($73.2 \text{ m}^2/\text{g}$) and cumulative pore area ($38.5 \text{ m}^2/\text{g}$), despite still having a moderate cumulative pore volume ($0.1196 \text{ cm}^3/\text{g}$). The pore size distribution of the Ca30 sample

Table 4. Comparison of surface area and pore parameters for CaSiO₃ samples with varying P123 content.

No	Content	Ca0	Ca10	Ca20	Ca30
1	BET surface area (m ² /g)	1.19	117.27	111.88	73.23
2	Cumulative pore volume (cm ³ /g)	1.8x10 ⁻³	0.19	0.21	0.12
3	Cumulative pore area (m ² /g)	1.12	110.20	108.32	38.49

shifted noticeably toward macropores, and the fraction of small mesopores decreased. This trend suggests that excessive P123 may lead to pore collapse, coalescence, or a less uniform porous network during the calcination process.

Overall, the Ca10 and Ca20 samples demonstrated superior textural properties in terms of specific surface area and mesopore development, while the Ca30 revealed structural degradation at high surfactant content. These observations emphasize the critical role of most suitable investigated P123 content in tailoring the porosity of CaSiO₃ materials for specific applications such as drug delivery, adsorption, or catalysis.

4. Conclusion

Porous CaSiO₃ materials were successfully synthesized using varying contents of P123 surfactant, and their pore structure was effectively tailored through this approach. The study demonstrated that a moderate amount of P123 (10–20 wt.%) promoted the formation of well-developed mesoporous networks with high surface areas and pore volumes. However, excessive P123 (30 wt.%) caused structural degradation and a shift toward macroporous characteristics, reducing the material's performance potential. Among three investigated samples, the Ca10 sample exhibited the most favorable porous structure, suggesting it as the most suitable composition for applications requiring high surface area and mesoporosity. These results underscore the critical role of surfactant content in designing porous CaSiO₃ materials with tunable textural properties.

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Authors contributions

Phi Hung Dao: Writing - Original Draft, Formal analysis, Visualization; Thuy Chinh Nguyen: Methodology, Writing - Review & Editing, Resources; Thi Huong Nguyen: Investigation; Thao Linh Bui: Validation, Investigation; Hoang Thai: Conceptualization, Writing - Review & Editing, Supervision, Funding acquisition, Project administration.

Conflict of interest

The authors declare no competing interests.

Data Availability

The data used to support the findings of this study are included within the article.

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