





# Synthesis methods in the production of porous metakaolin-based geopolymers

Alexandre Alves Belga Esteves<sup>1\*</sup>   
Afonso Rangel Garcez de Azevedo<sup>2</sup>   
Madeleing Tabora Barraza<sup>1</sup>   
Markssuel Teixeira Marvila<sup>3</sup> 

## Abstract

Porous geopolymers are sustainable alternative binders with applications in lightweight structures and thermal or acoustic insulation. Their performance depends on pore distribution and stability, which directly affect mechanical properties. This review examines synthesis methods for producing metakaolin-based porous geopolymers, focusing on porosity formation mechanisms and their impact on microstructure and functional behavior. The methodology involves a critical analysis of recent studies, including raw material characterization and porosity incorporation techniques such as foaming agents, surfactants, and sacrificial templates. Literature results confirm that the synthesis method significantly influences pore structure and connectivity, directly impacting the functional efficiency of porous geopolymers in insulation and environmental applications.

**Keywords:** Geopolymers; Metakaolin; Porosity; Microstructure.

## 1 Introduction

Portland cement, widely used in civil construction, faces significant environmental challenges due to its high energy consumption and substantial CO<sub>2</sub> emissions during production. The production of one ton of Portland cement can result in almost one ton of CO<sub>2</sub> emission. In response to this significant impact, geopolymers as binder alternative, can be manufactured from industrial by-products such as fly ash and slag have emerged as more sustainable alternatives. These conditions promote dependence on Portland cement consumption as well as offering superior properties, including higher mechanical strength and improved durability [1,2].

Studies indicate that geopolymers can reduce energy consumption by up to 0.110 kWh per gram and CO<sub>2</sub> emissions by approximately 800 kg per ton compared to Portland cement [3], aligning with the Sustainable Development Goals (SDGs) by minimizing greenhouse gas emissions [4]. Additionally, they are gaining popularity due to their environmental benefits and versatility in various applications, particularly in construction and wastewater treatment [5].

The alkali activation process, responsible for geopolymer synthesis, is based on aluminosilicate-rich precursors reaction with alkaline activators, such as sodium and potassium hydroxides or silicates, which are the most used.

This process can be carried out through two-part systems with liquid activators or one-part systems using solid activators, both resulting in cementitious materials characterized by an amorphous alkali-aluminosilicate-hydrate structure [5,6]. The efficiency of this transformation is influenced by factors such as activator concentration and precursor reactivity, which directly affect the final properties of the material [7]. However, even when alkali activation presents significant advantages, challenges such as handling high-pH activators and heterogeneous raw material need to be addressed for large-scale application [5].

In this context, geopolymers are amorphous inorganic materials cured below 100 °C. The commonly used precursors are fly ash, red mud, granulated blast furnace slag and metakaolin. This last one, a calcined clay, is widely preferred for its high reactivity, large specific surface area, and consistent chemical composition [8].

Porous geopolymers, in turn, are a variant designed to exhibit high porosity, obtained through pore-forming agents such as surfactants and foaming agents. This porous structure enhances the material's ability to adsorb contaminants, enabling the capture and retention of large amounts of pollutants [9]. Due to these characteristics, geopolymers have been studied

<sup>1</sup>Laboratório de Materiais Avançados – LAMAV, Programa de Pós-graduação em Engenharia e Ciência dos Materiais, Universidade Estadual do Norte Fluminense Darcy Ribeiro – UENF, Campos dos Goytacazes, RJ, Brasil.

<sup>2</sup>Laboratório de Engenharia Civil – LECIV, Programa de Pós-graduação em Engenharia e Ciência dos Materiais, Universidade Estadual do Norte Fluminense Darcy Ribeiro – UENF, Campos dos Goytacazes, RJ, Brasil.

<sup>3</sup>Universidade Federal de Viçosa, Rio Paranaíba, MG, Brasil.

\*Corresponding author: 202411120050@pq.uenf.br

E-mails: afonso@uenf.br; madelatb@gmail.com; markssuel.marvila@ufv.br



for applications in wastewater treatment and environmental remediation processes, such as the removal of heavy metals, dyes, and pharmaceuticals [10]. The porous structure reduces thermal conductivity and contributes to thermal and structural stability, expanding their applicability in fields such as construction materials and environmental remediation [8].

Specifically, due to their high porosity, the thermal conductivity of these systems decreases, while maintaining resistance to high temperatures without degradation—benefiting the construction sector where energy efficiency is essential, as they can be used in coatings and adhesives for outdoor environments [11]. With a porous structure similar to zeolite—a mineral known for its high adsorption capacity—these materials display unique adsorption behavior [12]. Their high surface area also enhances efficiency in processes such as carbon dioxide adsorption and environmental remediation due to synergistic effects [13].

The use of foaming agents plays a key role in the creation of porous geopolymers, with hydrogen peroxide ( $H_2O_2$ ) standing out as one of the most effective in this context. In the preparation of metakaolin-based porous geopolymers, the choice of foaming agents is crucial for improving the mechanical properties of the final product.

$H_2O_2$  is one of the most widely used foaming agents in the direct foaming method, helping to create a stable foam structure that ensures a more uniform pore distribution within the geopolymer matrix [14,15].

Therefore, this review aims to present and critically discuss the main synthesis techniques employed in the production of metakaolin-based porous geopolymers, correlating pore-forming strategies, foaming agents, and resulting pore structures reported in the literature. The scope of this work is restricted to metakaolin-based systems, emphasizing qualitative and comparative analysis of previously published studies. No new experimental data are presented, and the discussion is limited to information available in the existing scientific literature, which constitutes the main limitation of this review.

## 2 Development

### 2.1 Materials used in the production of porous geopolymers

Geopolymer formation relies on two main components: alkaline activators and aluminosilicate precursors (Figure 1). These activators dissolve the silica ( $SiO_2$ ) and alumina ( $Al_2O_3$ ) present in the precursors [16]. Typical examples of such precursors are in an amorphous state as a consequence of a previously applied heat treatment [6]. This dissolution occurs in a highly alkaline environment ( $pH > 11.5$ ), which is essential for the subsequent polymerization reaction. After dissolution, the Si and Al ions become solubilized, forming aluminate and silicate groups available to interact with each other and with the alkaline ions supplied by the activator solution, resulting in a three-dimensional network known as a geopolymer [16,17].

This network is mainly composed of two types of gels: N-A-S-H (sodium aluminosilicate hydrate) and C-A-S-H (calcium aluminosilicate hydrate) [18,19], depending on the calcium oxide content of the precursor. This reaction products are detected by other microstructural techniques as ray-x diffraction and spectroscopy curves, as a microstructural shift on raw-materials.

The production of porous geopolymers from metakaolin-based alkali-activated pastes requires a systematic and well-structured approach to evaluate their technical and environmental feasibility [20], ensuring control over the porosity created [21].

The development of this material involves key steps that ensure proper characterization of the precursors, optimized paste formulation, as shown in Figure 2, and the execution of comparative tests to understand the properties and performance of the compositions [11]. The optimal time to add such additives corresponds to the moment immediately after the activator solution has cooled, avoiding excessive delay in precursor incorporation, which could reduce its foaming potential, ensuring uniform pore structure and, consequently an improved mechanical property [22].



**Figure 1.** Schematic representation of the preparation process of two-part alkali-activated materials using a liquid activator solution. Adapted from [16].

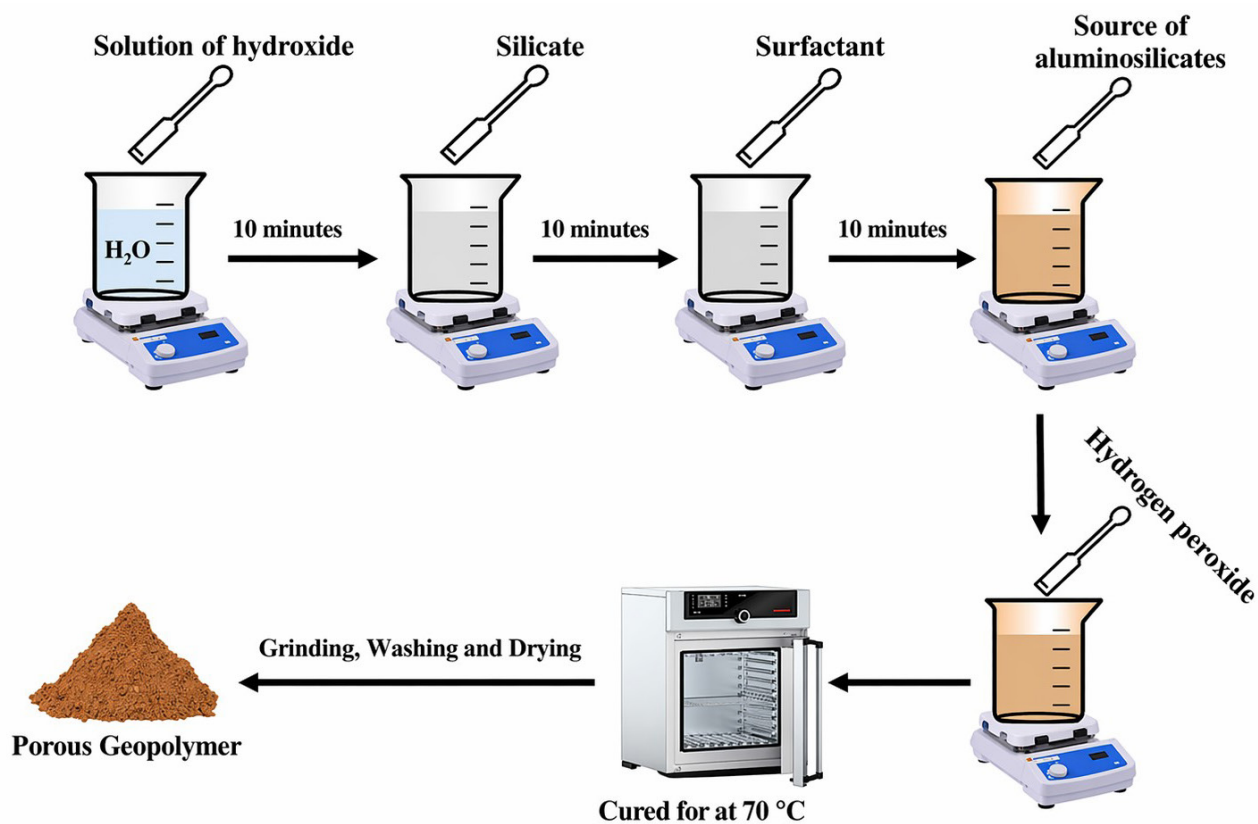


Figure 2. Schematic diagram of the preparation process for producing a porous geopolymer. Adapted from [11].

The best way to monitor the development of the pore network is through destructive or non-destructive porosimetry techniques, such as nitrogen absorption isotherms, mercury intrusion porosimetry and X-ray microtomography. Considering that each technique has operating and depth limitations. Precursor morphology, percentual additions and solid-liquid ratio concentrations will also influence this property [23,24].

## 2.2 Synthesis techniques

Among the main methods, direct foam formation (DF) stands out [25], which uses foaming agents such as hydrogen peroxide and metallic powders (silicon or aluminum) to generate gas bubbles in the geopolymeric matrix, creating porous structures [26]. Another method is the sacrificial template method (SFM), in which materials such as polylactic acid (PLA), ice, or sodium chloride are used as temporary molds that, once solidified and removed, leave pores with controllable geometry [27].

Additive manufacturing (AM), including techniques such as 3D printing, direct ink writing (DIW), and binder jetting, enables precise control over macroporous structures and the creation of complex geometries [28].

In addition, innovative methods such as microwave foaming (MF) have gained attention for providing rapid

preparation; however, they may produce non-uniform pore structures, which can affect overall performance [29].

The reactive emulsion templating (RET) method can produce hierarchical meso- and macroporous open-cell structures, improving both the mechanical and thermal insulation properties of the material [30].

Combining different approaches offers a pathway to enhance both porosity and mechanical strength. For example, routes that integrate direct foaming with reactive emulsion templating have achieved promising results [31]. Another notable technique is the suspension solidification method, in which the use of polyethylene glycol or silicone oil as a suspension medium allows the formation of granules or spheres through surface tension control [21].

The variations in these techniques, summarized in Table 1 and illustrated in Figure 3, offer a wide range of possibilities for customizing porous geopolymers and expanding their industrial applications by optimizing structure and final properties.

Curing conditions are also critical, as temperature and duration—such as curing at 60 °C or 70 °C for 4 to 24 hours—are decisive for pore formation and stability [21]. Studies have also explored the combined use of surfactants and chemical foaming agents to enhance pore stability and mesoporosity. Common surfactants reported in metakaolin-based geopolymer systems include sodium dodecyl sulfate (SDS), Triton

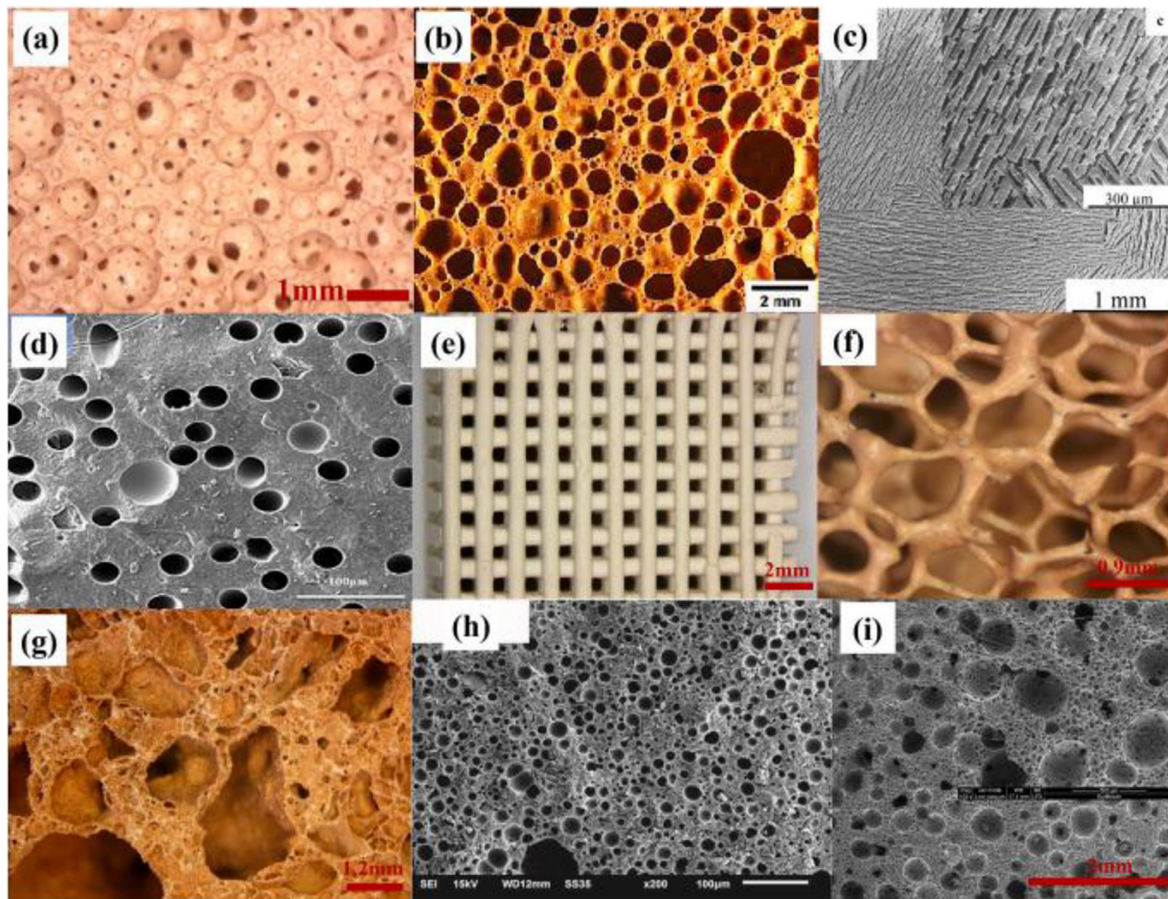
X-100, and Tween 80. These compounds act by reducing surface tension and stabilizing gas bubbles generated during the foaming process, particularly in systems incorporating hydrogen peroxide. Their use has been associated with improved pore uniformity and reduced bubble coalescence,

although their effectiveness depends strongly on dosage and curing conditions [11,22,23].

The diversity of synthesis techniques reflects their importance in optimizing geopolymers for advanced applications, showing how proper material and method

**Table 1.** Advantages and Disadvantages of Foam Formation Methods. Adapted from [21]

Method	Characteristics	Porosity Formation Time	Advantages	Disadvantages
Direct Foaming (DF)	Dynamic liquid–gas system	Short	Controllable porosity; Easy large-scale production	Ostwald ripening and coarsening; Broad pore size distribution; possible stratification
Sacrificial Template (SFM)	Pore geometry defined by the mold	Long	Controlled pore geometry	Additional mold removal step Limited by mold material
Additive Manufacturing (AM)	3D printer-based designability	Short	Precise macroporous structure control	High rheological requirements (DIW) limited printing time (DIW)
Reactive Emulsion (RET)	Oil–water interface with saponification	Long	Micro- to mesoporous structure	Possible water collapse Time-consuming process
Replica Method (RM)	Porous characteristics of the model	Long	Open-cell porous structure High porosity	Limited by foam model selection
Microwave Foaming (MF)	Unstable liquid–gas system under heating	Short	Rapid foaming and curing	Heterogeneous pore distribution Low performance stability



**Figure 3.** Typical pore structures of metakaolin-based porous geopolymers produced by various methods: (a) DF-H<sub>2</sub>O<sub>2</sub>; (b) DF-Al; (c) SFM with ice mold; (d) SFM with PLA mold; (e) AM; (f) RM; (g) MF; (h) RET; (i) DF/RET. Adapted from [21].

selection can lead to superior performance [11]. On the other hand, incorporating lightweight fillers during foam formation can reduce thermal conductivity, though at the expense of mechanical strength [14]. Balancing these properties is fundamental for applications requiring both insulation and structural integrity.

The incorporation of  $H_2O_2$  as a chemical foaming agent in metakaolin-based systems is commonly reported in proportions typically below 10 wt.% relative to the precursor mass. Its decomposition releases oxygen, directly controlling the overall volume of generated pores and significantly increasing total porosity, often to values above 50%, depending on formulation and curing conditions [32].

In contrast, surfactants are generally used in smaller proportions, typically below 5 wt.% relative to the solid content, primarily to stabilize the gas–liquid interface rather than to increase pore volume. Their incorporation improves bubble dispersion, reduces coalescence, and promotes narrower pore size distributions, contributing to more homogeneous microstructures without necessarily increasing total porosity [11].

Table 2 summarizes consolidated ranges of physical and mechanical properties of metakaolin-based porous geopolymers reported in the literature [8,14,22,33], highlighting the strong dependence of performance on pore architecture and processing conditions.

### 2.3 Applications

In civil construction, porous cementitious structures are widely used to produce lightweight and durable components, noted for their thermal stability and low thermal conductivity, making them ideal for thermoacoustic insulation [14,34].

They also play an essential role in environmental applications, being effective in drinking water purification as adsorptive substrates and in removing emerging pollutants from wastewater due to their porous structure and high adsorption capacity [11].

Porous geopolymers are also explored in energy storage technologies, such as supercapacitors and electrode materials, contributing to energy sustainability [31]. In the agricultural sector, they have been used as fertilizers and slow-release materials, promoting greater efficiency and sustainability in crop production [35]. In the medical field, they show potential for oral drug delivery systems [36] and as pH-buffering materials, revealing promising applications in the pharmaceutical area [34].

They are also suitable for high-temperature environments, resisting extreme conditions, making them useful in coatings and adhesives [37]. Their adaptability enables use in 3D printing, allowing the creation of complex, application-specific structures [38].

Some authors have investigated specific applications of porous geopolymers. For example, studies analyzed the synthesis of porous geopolymers produced from volcanic pozzolans for nickel and cobalt removal [39]. The authors activated the geopolymers with sodium hydroxide and used hydrogen peroxide as a pore-forming agent. The results demonstrated the efficiency of porous geopolymers in adsorbing nickel and cobalt, especially at higher temperatures.

Lightweight fillers, such as expanded clay or perlite, can also be added to the geopolymer matrix [40]. These fillers not only reduce the composite's overall density but also enhance its mechanical properties by creating additional voids within the structure, which contribute to its porosity. This method is particularly advantageous for applications requiring both strength and reduced weight.

### 2.4 Challenges

Controlled pore formation is a fundamental objective in structural design since increasing porosity typically causes an exponential reduction in compressive strength. This relationship presents a challenge in balancing the porosity required for lightweight or insulating applications with the need for adequate mechanical strength. The direct foaming method is considered ideal due to its simplicity and wide applicability.

However, one of the main challenges of direct foaming is controlling pore structure according to the foaming agent content. The dynamic nature of the gas–liquid system can lead to inconsistencies in pore size and distribution, making it difficult to achieve desired characteristics in the final product. If the paste is unstable, a dense surface film may form, hindering the foaming process and affecting overall porosity.

Even when direct foaming is the most widely studied method, it can present challenges in controlling pore size distribution due to phenomena such as Ostwald ripening, in which molecules on the surface of smaller particles dissolve and recrystallize on larger ones. These processes can produce larger pores at the expense of smaller ones, resulting in a less uniform pore structure that may not meet the specific requirements of certain applications.

Therefore, controlling the amount and type of additives, as well as their reaction time, are critical factors in determining

**Table 2.** Ranges of physical and mechanical properties of metakaolin-based porous geopolymers

Property	Typical range in literature	References
Total porosity (%)	50–90	[8,22]
Bulk density ( $kg/m^3$ )	300–1100	[8,14,22]
Compressive strength (MPa)	0.1–15	[8,22,33]
Thermal conductivity ( $W/m \cdot K$ )	0.08–0.40	[8,22,33]

pore distribution. This control enables the production of porous materials with reproducible and application-tailored functionality.

### 3 Conclusions

Porous geopolymers demonstrate remarkable versatility, with applications spanning civil construction, environmental remediation, energy storage, agriculture, medicine, acoustic insulation, 3D printing, and even space exploration. Their adaptability and multifunctionality make them innovative and sustainable materials capable of addressing numerous technological and environmental challenges.

Another noteworthy aspect is their potential use in supercapacitors, controlled drug-release systems, and even extraterrestrial construction, such as on the lunar surface. Moreover, metakaolin-based porous geopolymers have shown promise as catalytic supports in chemical reactions,

particularly in the photocatalytic degradation of organic pollutants, reinforcing their relevance for both structural and catalytic applications.

Additionally, porous geopolymers can be applied in the capture of greenhouse gases, including carbon dioxide (CO<sub>2</sub>), nitrogen oxides (NO<sub>x</sub>), and sulfur dioxide (SO<sub>2</sub>). This gas-capture capacity is crucial in mitigating atmospheric emissions and combating climate change, contributing to air pollution control strategies.

### Acknowledgements

The author expresses sincere gratitude to the Universidade Estadual do Norte Fluminense Darcy Ribeiro (UNEF) for institutional support and for granting the master's scholarship, which was essential for the completion of this research.

### References

- 1 Amar M, Ladduri B, Alloul A, Benzerzour M, Abriak NE. Geopolymer synthesis and performance paving the way for greener building material: a comprehensive study. *Case Stud Constr Mater*. 2024;20:e03280.
- 2 Kovalchuk G, Zozulynets O. Corrosion resistance of alkali-activated binders. *Construction & Building Materials*. 2022;279:122412.
- 3 Fetouh M. Energy consumption and CO<sub>2</sub> emission reduction in geopolymer production. *Journal of Cleaner Production*. 2024;382:136786.
- 4 Ikotun J O, Aderinto G E, Madirisha M M, Katte V Y. Sustainable geopolymer production in line with SDGs. *Mater Today Sustain*. 2024;15:100228.
- 5 Luukkonen T. Advances in alkali-activated materials: a review. *Construction & Building Materials*. 2022;305:124711.
- 6 Luukkonen T, Heponiemi A, Runtti H, Pesonen J, Yliniemi J, Lassi U. Alkali-activated materials: a review of uses and performance. *Journal of Materials Science*. 2019;54:12402-12443.
- 7 Echeverri-Aguirre M Y, Rudas-Flórez J S, Molina-Mosquera J E, Hoyos-Montil A A. Influence of alkaline activators on the geopolymerization process. *Cement and Concrete Research*. 2023;157:106834.
- 8 Bai C, Zheng K, Sun F, Wang X, Zhang L, Zheng T, et al. Structure and applications of metakaolin-based porous geopolymers. *Journal of the European Ceramic Society*. 2024;44(3):320-332.
- 9 Yuan L, Wang H, He T, Gao S. Adsorption capacity of porous geopolymer for contaminants. *Journal of Hazardous Materials*. 2024;447:130732.
- 10 Sreenivasan R, Kandasubramanian B. Geopolymer-based adsorbents for wastewater remediation. *Journal of Environmental Chemical Engineering*. 2023;11(1):109123.
- 11 Ettahiri Y, Bouargane B, Fritah K, Akhsassi B, Pérez-Villarejo L P, Aziz A, et al. A review on the potential of porous geopolymers synthesized using hydrogen peroxide and surfactants. *Journal of Building Engineering*. 2023;67:105891.
- 12 Wu J, Chen H, Lv S, Zhou Y. Adsorption behavior of geopolymer-based materials. *Chemosphere*. 2024;320:138038.
- 13 Boscherini M, Miccio F, Papa E, Medri V, Landi E, Doghieri F, et al. Surface area and adsorption properties of porous geopolymer. *Microporous and Mesoporous Materials*. 2021;319:111026.
- 14 Yan S, Ren X, Wang W, He C, Xing P. Effect of H<sub>2</sub>O<sub>2</sub> content on properties of geopolymer foams. *Construction & Building Materials*. 2024;328:127053.
- 15 Alves CR, Muller AA, Dal-Bó AG, Bernardin AM. Effect of sodium dodecyl sulfate and hydrogen peroxide on the microstructure evolution of fiber-reinforced lightweight geopolymer composite. SSRN, 2024 [cited 2025 Oct 13]. Available at: <https://www.ssrn.com/abstract=4833115>.

- 16 Qin Y, Qu C, Ma C, Zhou L. Mechanism and performance of geopolymer materials. *Materials Chemistry and Physics*. 2022;285:126118.
- 17 Yi C, Boluk Y, Bindiganavile V. Experimental characterization and multi-factor modelling to achieve desired flow, set and strength of N-A-S-H geopolymers. *Materials*. 2022;15(16):5634.
- 18 Myers RJ, Bernal SA, Provis JL. Phase diagrams for alkali-activated slag binders. *Cement and Concrete Research*. 2014;66:27-44.
- 19 Garcia-Lodeiro I, Fernandez-Jimenez A, Palomo A. Crystalline phases in alkali-activated fly ashes: N-A-S-H vs C-A-S-H gels. *Cement and Concrete Research*. 2011;41(7):703-709.
- 20 Ouafu O J G, Belibi B P D, Essomba J S, Ankoro N O, Ndi J N, Elimbi A. Sustainable development of metakaolin-based geopolymer pastes using industrial waste. *Environ Technol Innov*. 2024;31:103238.
- 21 Bai C, Zheng K, Sun F, Wang X, Zhang L, Zheng T, et al. Structure and applications of metakaolin-based porous geopolymers. *Journal of the European Ceramic Society*. 2024;44(3):320-332.
- 22 Kim KW, Lim HM, Yoon S-Y, Ko H. Fast-curing geopolymer foams with an enhanced pore homogeneity derived by hydrogen peroxide and sodium dodecyl sulfate surfactant. *Minerals*. 2022;12(7):821.
- 23 Papa E, Landi E, Miccio F, Medri V. K<sub>2</sub>O-metakaolin-based geopolymer foams: production, porosity characterization and permeability test. *Materials*. 2022;15(3):1008.
- 24 Vaz André D Jr, Barraza MT, Gleize PJP, Marvila MT, Vieira CMF, Azevedo ARGD. Ecological alkaline activator based on coffee waste ash applied to geopolymers. *Journal of Materials in Civil Engineering*. 2025;37(9):04025305..
- 25 Xu M X, He Y, Liu Z H, Tong Z F, Cui X M. Foam forming techniques for geopolymer synthesis. *Materials Letters*. 2019;253:152-155.
- 26 Papa E, Landi E, Miccio F, Medri V. Properties of geopolymer foams using sacrificial templates. *Journal of Cleaner Production*. 2022;342:130939.
- 27 Rasouli H R, Golestani-fard F, Mirhabibi A R, Nasab G M, Mackenzie K J D, Shahraki M H. Structured porosity in geopolymers using PLA. *Journal of Materials Science*. 2015;50:4468-4475.
- 28 Ma S, Jiang Y, Fu S, He P, Sun C, Duan X, et al. Additive manufacturing of porous geopolymers: a review. *Additive Manufacturing*. 2023;59:103161.
- 29 Zheng J, Li X, Bai C, Zheng K, Wang X, Sun G, et al. Microwave foaming method for geopolymer synthesis. *Materials Today. Communications*. 2024;38:106126.
- 30 Wang X, Lu J. Reactive emulsion templating for porous geopolymer fabrication. *Chemical Engineering Journal*. 2020;381:122666.
- 31 Bai C, Colombo P, Homaeigohar S. Porous geopolymer components with thermal and acoustic insulation properties. *Journal of the European Ceramic Society*. 2016;36(12):2833-2839.
- 32 Petlitckaia S, Vincente J, Poulesquen A. Characterization of a geopolymer foam by X-ray tomography. *Frontiers in Chemistry*. 2021;9:754355.
- 33 Bai C, Franchin G, Elsayed H, Zaggia A, Conte L, Li H, Colombo P. Thermal conductivity of geopolymer foams. *Journal of Non-Crystalline Solids*. 2017;462:45-52.
- 34 Sekkal W, Zaoui A. Acoustic performance of porous geopolymers. *Applied Acoustics*. 2023;205:109261.
- 35 Yan H, Zhu X, Dai F, He Y, Jing X, Song P, et al. Controlled-release fertilizer application of porous geopolymer. *Journal of Environmental Management*. 2021;297:113361.
- 36 Jämstorp E, Strømme M, Frenning G. Modeling structure–function relationships for diffusive drug transport in inert porous geopolymer matrices. *Journal of Pharmaceutical Sciences*. 2011;100(10):4338-4348.
- 37 Jiang C, Wang A, Bao X, Ni T, Ling J. Geopolymers as coating materials. *Journal of Building Engineering*. 2020;32:101734.
- 38 Zhong S, Zhang X. 3D-printed geopolymers for structural applications. *Construction & Building Materials*. 2022;316:125780.
- 39 Matakah F, Bani Hani M, Al-Momani I. Synthesis of porous volcanic tuff-based geopolymer for nickel and cobalt removal. *Results Chem*. 2025;15:102179.
- 40 Zhang X, Bai C, Qiao Y, Wang X, Jia D, Li H, et al. Porous geopolymer composites: a review. *Composites. Part A, Applied Science and Manufacturing*. 2021;150:106629.

Esteves et al.

Received: 13 Out. 2025

Accepted: 1 Mar. 2026

Editor-in-charge:

André Luiz Vasconcellos da Costa e Silva 