

A Review on the Optimization of Porous Glass-Ceramics Through a Mixed-Method Approach

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Abstract: This review investigates the optimization of the controlled crystallization process for porous glass-ceramic materials, with a focus on basalt-based compositions, to enhance their mechanical and thermal properties for functional applications such as thermal insulation and lightweight structural components. A critical gap identified in the current literature is the lack of comprehensive studies that integrate process parameters, pore structure, and material performance. Additionally, the potential of local materials such as basalt, particularly in the context of Indonesia's abundant basalt reserves, remains underexplored. This review synthesizes findings from studies employing a mixed-method approach, combining quantitative experimental designs that evaluate the influence of key variables—temperature, heating time, and composition—on the crystallization process and pore formation in glass-ceramics. The findings aim to provide a more nuanced understanding of process-property relationships and contribute to the development of sustainable, energy-efficient basalt-based ceramic materials. The insights presented are expected to inform the formulation of practical and efficient production strategies, driving the application of porous glass-ceramics in industrial sectors as advanced materials.

Keywords: porous materials, basalt, glass-ceramic, ceramics, mixed-method

1. Introduction

Ceramic and glass porous materials have attracted significant attention in various technological applications, including biomedicine, catalysis, insulation, and gas and liquid filtration. In this context, porous materials can be classified based on pore size according to standards set by the International Union of Pure and Applied Chemistry (IUPAC): micropores (less than 2 nm), mesopores (2–50 nm), and macropores (greater than 50 nm). Additionally, macroporous polymer materials with pore sizes ranging from 50 nm to 1 μm fall under this classification. The concept of hierarchical porosity also plays a crucial role, where structures can be bimodal (combining two categories) or trimodal (combining three categories) [1],[2].

Pore topology also plays a significant role in the effectiveness of porous materials. Examples of pore topologies include open porosity (connected and permeable), blind porosity (blind ends), and closed porosity (isolated or only connected to surrounding cells but lacking permeability), and mixtures. The shape and arrangement of pores can sometimes be uniformly structured (graded porosity) or aligned. The shape and arrangement of pores determine the mechanical properties of the material [3].

Basalt rock from East Lampung, Indonesia, shows great potential for the development of high-quality glass-ceramic materials. The Lampung region contains volcanic basalt rock, but it has not been fully utilized, only as aggregate and building ornaments. Basalt can be applied as a raw material for the production of glass-ceramics [4]. The process of preparing basalt rock for glass-ceramics involves heating it in a muffle furnace until it melts at temperatures between 1,200°C and 1,500°C. In Anwar's 2022 [5] study, basalt rock was heated to 1,200°C and held for 2 hours, then quenched with water. Following this, a heat treatment at 600°C was conducted and held for 2 hours, during which nucleation occurred at 600°C. After being held for 2 hours, the temperature was raised to 1050°C and held for 3 hours, followed by cooling inside the furnace. At this temperature, crystallization occurred, and the glass-ceramics could then be used. In this study, the base material is heated to 1200°C and held for 2 hours, then quenched with water. After that, it was subjected to heat treatment at 600°C and held for 2 hours. At 600°C, nucleation occurs. After being held for 2 hours, the temperature is raised to 950°C and held for 3 hours, followed by cooling inside the furnace. At this temperature, crystallization occurs, and the glass-ceramic can then be used. Glass ceramics have advantages such as high thermal stability and corrosion resistance [6], [7].

Additionally, basalt-based glass ceramics exhibit good flexibility in applications. Porous glass ceramics can be used in the refractory industry, including thermal insulation and radiation protection. However, further research is needed to develop basalt processing technology and optimize the physical and chemical properties of the resulting glass ceramics [8], [9]. A thorough understanding of the characteristics of porous materials and the development of local basalt materials from East Lampung are important for creating porous materials that can be used in biomaterials and industry.

2. Production Process

The production of porous ceramic glass uses four methods, namely phase separation, sol-gel method, sintering, and foam formation. Based on the synthesis approach, ceramic glass is divided into three categories: classical ceramic glass, open-porous ceramic glass, and silica ceramic glass. Classical porous ceramic glass is produced through a dissolution process. Open-porous ceramic glass is obtained through glass sintering. Silica porous ceramic glass is produced using the sol-gel method.

2.1. Sol-Gel Method

Homogenization of the solution is achieved at room temperature for the sol-gel method. At room temperature, a uniform composition is ensured before the subsequent process. Gel formation is carried out to develop cross-links and stabilize the liquid structure. The gel is then strengthened through heat treatment. This heat treatment process aims to remove organic species, hydroxyl groups, and reduce material porosity, which can affect the physical and chemical properties of the final product.

The sol-gel method is a dynamic, reliable, and environmentally friendly approach [10], [11]. Additionally, this method is practical and versatile due to its simple procedures and low cost. The sol-gel method offers significant flexibility in the application of high-purity materials. The use of this method allows the production of various forms of high-purity materials such as monoliths, nanoparticles, thin films, foams, and fibers, among others [10][12].

2.2. Phase Separation

Phase separation in borosilicate glass is a process involving the formation of two distinct phases after heat treatment. The two phases used are the alkali-rich borate phase and the nearly pure silica phase. The first phase is the borate phase, which is soluble and insoluble in acids, soluble in dilute acid

solutions such as hydrochloric acid (HCl) and sulfuric acid (H₂SO₄). The second phase is the silica phase, which is soluble and insoluble. This process forms a porous structure within the material [13]. Figure 1 illustrates a schematic diagram of the phase separation process for producing porous glass ceramics. By using acid corrosion treatments such as hydrochloric acid (HCl), nitric acid (HNO₃), and sulfuric acid (H₂SO₄) on the silicate phase, phase separation can be achieved [14]. The weak borate phase dissolves and creates continuous porosity. This phase separation is achieved through a combination of heat treatment and acid corrosion. Pore size can be adjusted by controlling composition and heat treatment. Adjusting composition and heat treatment allows the material to be processed at lower sintering temperatures, below 1000°C, compared to traditional methods at 2000°C [15][16].

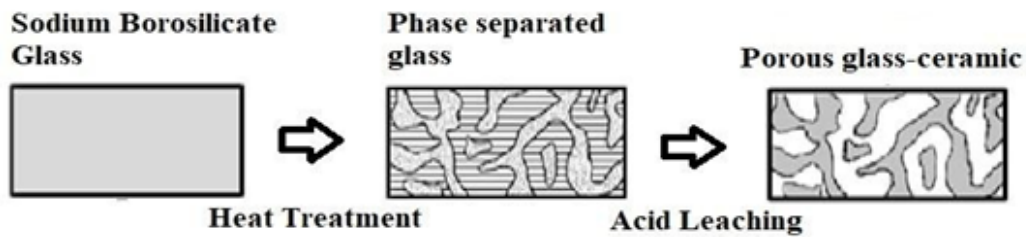


Figure 1. Schematic diagram of the phase separation process [16]

Basalt rock from East Lampung has the potential to be made into porous glass-ceramics. Basalt rock contains 50% silica dioxide (SiO₂). Basalt rock can be used in the formation of sodium borosilicate glass ceramics. The phase separation process aims to improve the mechanical and thermal properties of glass-ceramics. Previous studies have shown that the borate and silica phases can be separated. The borate and silica phases are separated through thermal and chemical treatments [16][17]. Therefore, basalt has potential for the development of glass ceramics. Research by Khater et al. (2012) [16] showed that basalt-based glass ceramics have optimal density and mechanical properties. The optimal density and mechanical properties are due to variations in composition and appropriate heat treatment.

In the study by Huo et al. (2022) [17], it was shown that the use of basalt olivine as a raw material can accelerate crystallization kinetics and improve the physical properties of glass-ceramics. Research results indicate that basalt olivine contains magnesium (Mg), iron (Fe), silicon (Si), and oxygen (O) [7]. The elemental composition of basalt olivine produces glass-ceramics with a homogeneous surface. The homogeneous surface is due to a stable crystallization process.

2.3. Sintering Process

Sintering is a densification process in the production of composite materials. The densification process occurs when solid powders bond at a certain temperature (during cooling inside the furnace). The bonding that occurs during cooling results in recrystallization and solidification of the material. This process produces materials with increased mechanical strength [18][19][20]. In the production of ceramic glass, sintering heat treatment is used. Sintering-crystallization heat treatment is an economical method suitable for producing glass ceramics with complex shapes [21]. The production of ceramic glass through the sintering process begins with crushing the raw materials (soda-lime silicate glass, basalt, and clay balls). The materials are then ball milled to produce powder. The powder is sieved with a 325 mesh to obtain fine powder. The powder from the 325 mesh is then pressed into pallet shapes using hydraulic pressure at room temperature. The molded material is subjected to sintering heat treatment at temperatures between 750°C and 950°C for one hour, with a heating rate of 2°C per minute and natural cooling [22].

The production process of glass-ceramics using basalt rock as the raw material involves a devitrification heat treatment. Devitrification is a heat treatment process consisting of nucleation and crystallization. The nucleation heat treatment is conducted at 600°C for one hour. After being held at this temperature for one hour, the temperature is raised to 1050°C and held for three hours. At this temperature, crystallization occurs, and only then can the glass-ceramics be used [23]. Crystallization typically begins at the glass surface and then spreads inward over time [19][24]. The devitrification process is performed to facilitate the transition from the powder phase to the amorphous glass phase and then to the crystalline phase, which provides good stability and mechanical strength [22].

During devitrification, control of heating time and heating rate must be carefully monitored. Excessively long heating treatment durations can lead to larger crystal growth, which may reduce the material's mechanical strength. In this case, additives (TiO_2 , $\text{SiO}_2 \cdot 6\text{H}_2\text{O}$, MgO , CaO , and ZrO_2) act as nucleating agents. Nucleating agents accelerate the crystallization process by providing starting points for crystal growth. These nucleating agents are crucial for accelerating the transition from the amorphous phase to the crystalline phase during devitrification [18].

Glass-ceramics produced from basalt exhibit resistance to abrasion, corrosion, and high mechanical strength. Glass-ceramics are promising materials for biomaterial applications and industry [25]. Adjusting devitrification process parameters, such as temperature and time, plays a key role in controlling crystallization and ensuring optimal material outcomes [26]

2.3. Foaming Process

Porous glass ceramics are produced by forming gas bubbles within the glass melt, which are then compacted. The formation of these bubbles is typically caused by the decomposition of carbonates during the melting process [6]. Nam et al. developed a method for creating porous frameworks using effervescent salts (ammonium bicarbonate) as a foaming agent. Effervescent salt particles are sieved from a polymer gel paste and then sprayed into a mold to obtain a specific shape. After that, the particles are immersed in boiling water. Ammonia gas (NH_3) and carbon dioxide (CO_2) are produced from the effervescent particles dissolved from the polymer matrix during solidification. This process causes the effervescent salt particles to dissolve, leaving the polymer matrix, and forming pores with a high pore structure [27]. Figure 2 shows a schematic diagram of the gas foaming process for the production of porous glass-ceramics.

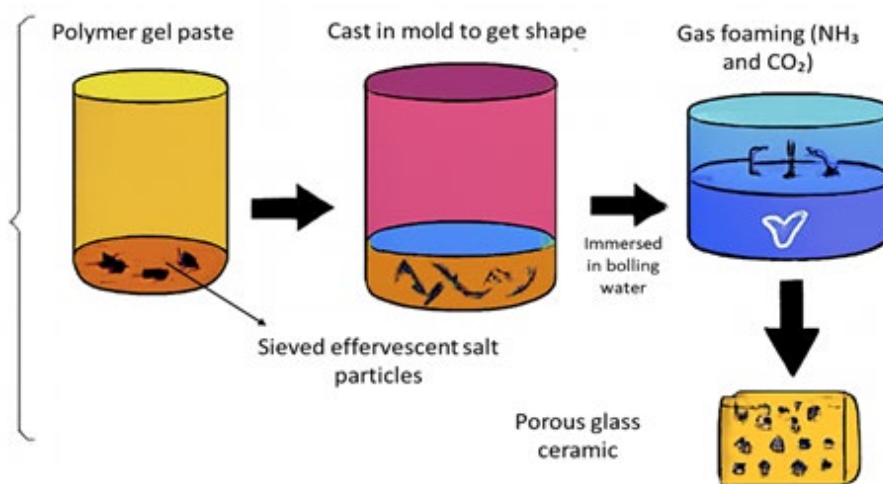


Figure 2. Schematic diagram of the gas bubble formation process.

3. Properties and Applications

Porous glass-ceramics offer numerous technical advantages, making them highly sought after for a wide range of applications, both in biomedicine and other industries. These advantages include high mechanical strength, excellent thermal stability, a large specific surface area, and greater pore volume compared to other materials. As a result, porous glass-ceramics are often chosen for applications such as biomaterials, lightweight concrete, thermal and sound insulation, and as filters in various systems.

3.1. Biomaterials

Currently, porous glass-ceramics are widely used in biomedical applications due to their high biocompatibility and bioactive properties. For example, foam glass has been used to make bone prostheses. Sindut et al. developed bioactive porous glass-ceramics for use as implants in plastic surgery and other medical applications. Microstructure observations show that sintering at 1200°C with a heating rate of 1.5°C/min produces a highly favorable microstructure for implant applications, where closed pore structures are formed using slip casting deposition on polymer sponges.

Iatsenko et al. [28] reported that the strength of the glass-ceramic samples increased significantly from 0.8 MPa to 10.5 MPa with increasing sintering temperature, leading to the formation of closed porosity and vitrification of the sample surface, see Table 1. The volume shrinkage, as well as the total and open porosity, showed a sharp increase in volume shrinkage from 8% to 76% as sintering temperature increased, resulting in a reduction in total and open porosity. The transformation of pore structures from open to closed, along with optimal mechanical properties, makes this material suitable for use as a replacement for damaged cancellous bone.

Table 1. Compressive strength and microhardness of glass-ceramics

| Temperature (°C) | Raw Materials | Microhardness (HV0.05) [29] | Material Result | Compressive Strength (MPa) [30] | Hardness (HV) [6] |
|------------------|---------------|-----------------------------|-----------------|---------------------------------|-------------------|
| 1450 (melting) | Basalt | Not specified | Melted Glass | Not Available | Not Available |
| 800 | Basalt | 835 - 946 | Glass-Ceramic | 0.8 | 725 |
| 900 | Basalt | 751 - 856 | Glass-Ceramic | 1.0 | 700 |
| 1000 | Basalt | 659 - 896 | Glass-Ceramic | 3.3 | 675 |
| 1100 | Basalt | Not specified | Glass-Ceramic | 10.5 | 710 |

3.2. Thermal Insulation and Lightweight Construction

Porous glass-ceramics are also used as environmentally friendly thermal insulation materials with high strength, fire resistance, and low density. These materials are highly suitable for use in lightweight construction or as thermal and sound insulation. Hartung et al. demonstrated that the use of capillary suspension in the processing of porous glass-ceramics allows for better control over the pore network, porosity, mechanical properties, and chemical resistance of the material. The resulting lightweight porous glass-ceramics have low density (200 kg/m³) and good compressive strength (0.6 MPa), making them ideal for lightweight construction or insulation applications.

In another study, Bernardo et al. developed crystalline foam glass from glass waste using a treatment at temperatures ranging from 900°C to 1050°C. The use of MnO₂ as a foaming agent

effectively promotes the oxidation of SiC, accelerating the formation of foam glass at lower temperatures (<1000°C) during a 60-minute soaking period. It was found that the density of the foam glass decreased as the soaking time increased at temperatures between 950°C and 1050°C, due to gas released during the foaming process. This result suggests that foam glass is suitable as an aggregate in lightweight concrete because it is light, simple, and cost-effective.

3.2. Filtration: Controlling Porosity for Filter Applications

Controlling porosity and pore size is a key aspect in the production of filter products made from porous glass-ceramics. Thermal characteristics, particle size, binder usage, and compressive strength affect the type of porosity and pore size in the produced filter samples. Generally, porous materials used in filter development exhibit open pore types, with average pore sizes ranging from 0.01 to 100 µm, depending on the desired filter application.

Sadighzadeh et al. produced porous glass bodies by partial sintering of glass waste. They found that the porosity of the resulting samples ranged from 15.5% to 32%. This study showed that sintering time and temperature had a significant impact on porosity values. Samples sintered at 750°C and 800°C showed higher shrinkage and lower porosity (8%), while sintering at 700°C for two hours increased porosity to 16-20% without structural cracking. Therefore, to obtain samples with higher porosity, sintering time was reduced to 75 minutes using glass particles in the 40-63 µm size range. These findings are relevant for filtration applications in the pharmaceutical industry.

4. Conclusions

Porous glass ceramics can be produced using various methods related to their properties and different applications. Through various production processes, the structure of porous glass ceramics can be tailored for various applications based on their properties. Classification of pore structure can also determine the application of porous glass ceramics, particularly for biomaterials, which require transformation of pore structure from open to closed types and optimal structural mechanics. For insulation materials and lightweight construction, high strength, low density, and high-quality macro-structural properties are required. Filter applications require open pore structures. Sintering time and temperature influence porosity values. Longer soaking times increase density. To produce lightweight materials and filters, density and porosity must be controlled. Sintering time should be shorter (less than 2 hours) and crystallization temperature should be higher (above 700°C). This review provides a clearer map of the relationship between process parameters, pore structure, and material performance. The review is expected to make a significant contribution to the development of porous glass ceramics based on local resources, specifically basalt rock.

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