

Article

The Effect of Flux on a Waste-Derived Foamed Ceramic: Analysis of Microstructure and Properties

Zhiwu Zuo ¹, Minghao Mu ¹, Xue Liu ¹ and Congcong Jiang ^{2,*}

¹ Shandong High-Speed Group Innovation Research Institute, Jinan 250102, China; Zzw9000@163.com (Z.Z.); sdgsmmh@126.com (M.M.); sdhseeg@163.com (X.L.)

² Shandong Provincial Key Laboratory of Preparation and Measurement of Building Materials, University of Jinan, Jinan 250022, China

* Correspondence: mse_jiangcc@163.com

Abstract: Foamed ceramics with high closed porosity were prepared using granite scrap as the raw material and silicon carbide as a foaming agent, and the effects of Na₂O, K₂O, and MgO on the pore structure and properties of the foamed ceramics were investigated. The results show that both Na₂O and K₂O could reduce the viscosity of the melt and promote the formation of the liquid phase, and the increase in content could enhance the foaming ability of the blank. When the dosage of Na₂O was 4–6 wt% and the dosage of K₂O was 6–8 wt%, the homogeneity of the pore structure of the foamed ceramics could be effectively improved, and the samples exhibited an optimal performance, including a bulk density of 510.36–593.33 kg/m³, a closed porosity of 68.24–78.04%, a compressive strength of 1.33–2.66 MPa, and a water absorption capacity of 0.57–1.31%. A further increase in the Na₂O and K₂O dosages destroyed the uniformity of the pore structure, resulting in a large number of irregular macropores. MgO had a slight effect on regulating the pore structure of the foamed ceramics, and the increase in dosage promoted the precipitation of forsterite crystals, creating conditions suitable for the preparation of foamed ceramics with small pores.

Keywords: foamed ceramic; granite scrap; flux; pore structure



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1. Introduction

Foamed ceramics have been widely used in thermal and acoustic insulation, light aggregates, catalyst carriers, and automobile exhaust purification due to their good chemical stability, high porosity, low density, thermal and sound insulation, and fire resistance [1,2]. When used as insulation boards for building envelopes, foamed ceramics can overcome the drawbacks of traditional organic insulation materials like extruded polystyrene (XPS) and expandable polystyrene (EPS), which are flammable and prone to aging [3,4]. Furthermore, foamed ceramic is produced by using solid waste as a raw material and adding high-temperature foaming agents, followed by high-temperature sintering. Various solid wastes, such as red mud, fly ash, tailings, waste glass, coal bottom ash, etc., have been successfully used to prepare foamed ceramics, leading to the consumption of a large amount of solid waste, reductions in the consumption of natural resources, and the alleviation of environmental pollution issues [5–9].

Granite scrap (GS) is solid waste generated during the mining and processing of granite stone. According to statistics, approximately 50% of granite stone becomes solid waste after it is processed, meaning that there are tens of millions of metric tons of GS produced annually [10]. Usually, GS can be utilized for cement production [11], the preparation of ceramic glazes [12], and as a substitute for natural aggregates in concrete for reuse [10]. It is noted that the abundant SiO₂ and Al₂O₃ components in GS are the main constituents of the frameworks of ceramic material and contain a certain amount of alkaline oxides that can melt and thus enter a liquid phase at high temperatures, indicating the potential for the large-scale utilization of GS in the preparation of foamed ceramics, facilitating its bulk

utilization. In our previous works, we prepared foamed ceramics from granite scrap and clay tailings [13] and granite scrap and red mud [14], and the utilization rate of granite scrap reached more than 85%. However, due to GS's high initial melting temperature and low liquid phase formation in its initial melting state, adjustments to the content of fluxing agents are required to facilitate pore structure [15]. Thus, flux additives are required to regulate the foaming process of solid waste-based foamed ceramics to obtain products with a well-developed pore structure. And adjustments to the content of fluxing agents are also necessary to improve the foaming effect.

In this paper, we used GS as a basic raw material, doped with 1 wt% SiC as a foaming agent, by adding the analytically pure chemical reagents Na_2CO_3 , K_2CO_3 , and MgO to change the content of the flux components in the billet. The influences of the flux components on the pore structure and properties of foamed ceramics were explored in detail to provide a scientific theoretical basis for the design optimization of the billet system for preparing foamed ceramics from GS, which are of great practical importance.

2. Experimental Procedures

The GS used in the study was obtained from waste generated during the cutting and processing of granite minerals from Shandong Province, China, and the chemical composition of these minerals was examined using an X-ray fluorescence spectrometer (XRF, PANalytical Axios, Bruker, Germany), as detailed in Table 1. Sodium carbonate (Na_2CO_3), potassium carbonate (K_2CO_3), and magnesium oxide (MgO) (Sinopharm Chemical Reagent Co., Ltd., Shanghai, China) were added as fluxing agents to adjust the Na_2O , K_2O , and MgO content in the batches. Silicon carbide (SiC, with an average particle size of 74 μm , Shanghai McLean Biochemical Technology Co., Ltd., Shanghai, China) was added as a foaming agent.

Table 1. Chemical composition of granite scrap (wt%).

SiO_2	Al_2O_3	Na_2O	K_2O	Fe_2O_3	CaO	MgO	TiO_2	P_2O_5	Others
67.89	14.57	4.72	4.40	3.23	2.78	1.26	0.43	0.34	0.38

The high-temperature foaming method was selected to prepare foamed ceramics in this experiment. First, the GS was dried at 105 °C for 24 h in a resistance furnace, and then it was sieved using a 200-mesh sieve, and 1 wt% SiC was added as a foaming agent. By externally mixing different mass fractions of Na_2CO_3 and K_2CO_3 into the GS, 0–8 wt% of Na_2O and 0–10% of K_2O were introduced, and the resulting samples were labeled as N0–N8 and K0–K10, respectively. Similarly, 0–8 wt% of magnesium oxide was introduced into the GS, and the resulting samples were labeled as M0–M8. All raw materials were proportionally weighed and mixed well and placed in a mold with a height of 4 cm and a diameter of 3 cm and pressed into shape; then, the pressed billet was placed on corundum heat-resistant bricks lined with heat-resistant paper and put it into a resistance furnace. These materials were heated, at a heating rate of 5 °C/min, to 1130 °C and sintered for 30 min. Then, after naturally cooling down to room temperature in the furnace, the materials were removed to obtain foamed ceramics, which were cut into regular rectangles and tested for their performance.

The cut samples were dried in an oven at 105 °C until they reached a constant weight, cooled to room temperature in a desiccator, and weighed in terms of mass m_1 in air. Then, the samples were submerged in deionized water and boiled for 4 h to make them fully absorb water. The saturated samples were quickly moved to a deionized water container with an overflow tube. When a sample had been completely removed from the deionized water, it was hung on the hook of a balance and weighed to obtain the apparent mass m_2 of the saturated sample. Each sample was then removed from the deionized water, and the water adhering to the sample surface was wiped away with a fully absorbent towel. Then, we quickly weighed the saturated sample's mass m_3 in air. The bulk density (ρ_1),

open porosity (P_1), and water absorption (W) of foamed ceramics can be calculated using Formulas (1)–(3):

$$\rho_1 = \frac{m_1}{m_3 - m_2} \quad (1)$$

$$P_1 = \frac{m_3 - m_1}{m_3 - m_2} \times 100\% \quad (2)$$

$$W = \frac{m_2 - m_1}{m_1} \times 100\% \quad (3)$$

The samples were ground into powder and sieved through a 200-mesh sieve, and powder density was measured using the pycnometer method (ρ_2). Total porosity (P) was calculated based on the volume density and powder density using Formula (4). The closed porosity of a sample was calculated by subtracting the open porosity from the total porosity.

$$P = \frac{\rho_2 - \rho_1}{\rho_2} \times 100\% \quad (4)$$

The apparent morphology of the foamed ceramic samples was characterized using a digital camera. The pore size distribution and average pore size of the samples were determined using Image Pro Plus 6.0 analysis software. The fired samples were ground into a powder with a particle size of less than 75 μm , dried, and analyzed for their phase composition using an X-ray diffractometer (Bruker D8 Advance, Cu ($K\alpha$); target scanning speed: $10^\circ/\text{min}$). The compressive strength of all the samples was measured at a loading speed of 2 mm/min using an electronic universal tester (CMT5504, Beijing, China).

3. Results and Discussion

3.1. The Effect of Na_2O Content on Foamed Ceramics

Figure 1 shows the pore structure morphology and pore size distribution of the foamed ceramics prepared with different Na_2O dosages referred to as N0, N2, N4, N6, and N8. When no additional Na_2O was added, sample N0 exhibited insufficient foaming, with small and irregularly shaped pores, which were mostly flattened. Upon increasing the Na_2O content, the range of pore size distribution expanded, and the average pore size increased from 0.23 mm for N0 to 2.09 mm for N8. The porosity also increased from 50.21% to 83.36%, as shown in Figure 2a, indicating that the addition of Na_2O significantly enhanced the foaming ability of the blanks. However, when the Na_2O content exceeded 6 wt%, numerous irregular large pores appeared, disrupting the uniformity of the pore structure.

Na_2O was an effective fluxing agent because the bridging oxygen bonds were broken, leading to the dissociation of $[\text{SiO}_4]$ units. As a result, the original silicon–oxygen anionic groups disintegrated into simpler structural units, increasing the concentration of oligomers and reducing the flow activation energy [16,17], thus lowering the viscosity of the melt and reducing the expansion resistance for pore growth. Additionally, the increase in Na_2O content promoted the formation of low-melting-point sodium feldspar. This sodium feldspar gradually melted, generating a significant amount of liquid phase, creating a high-temperature liquid phase environment suitable for pore formation. As shown in Figure 3, the phase composition of the samples changed with the increase in Na_2O content. The peak intensity of minerals such as quartz, pyroxene, and feldspar decreased gradually with increasing Na_2O doping, while the intensity of the amorphous phase scattering peak increased. This change became particularly evident when the Na_2O dosage reached 8 wt%, indicating that the increase in Na_2O content reduced the melting point, facilitated phase melting, and generated a greater amount of the liquid phase, providing a suitable environment for pore growth. Consequently, the pores were more likely to expand, resulting in an increase in pore size and porosity. However, the gas generated was prone to breaking through the pore walls, leading to interconnected or open pores because of the low melt viscosity, which, in turn, increased the open porosity and water absorption, as shown in Figure 2b.

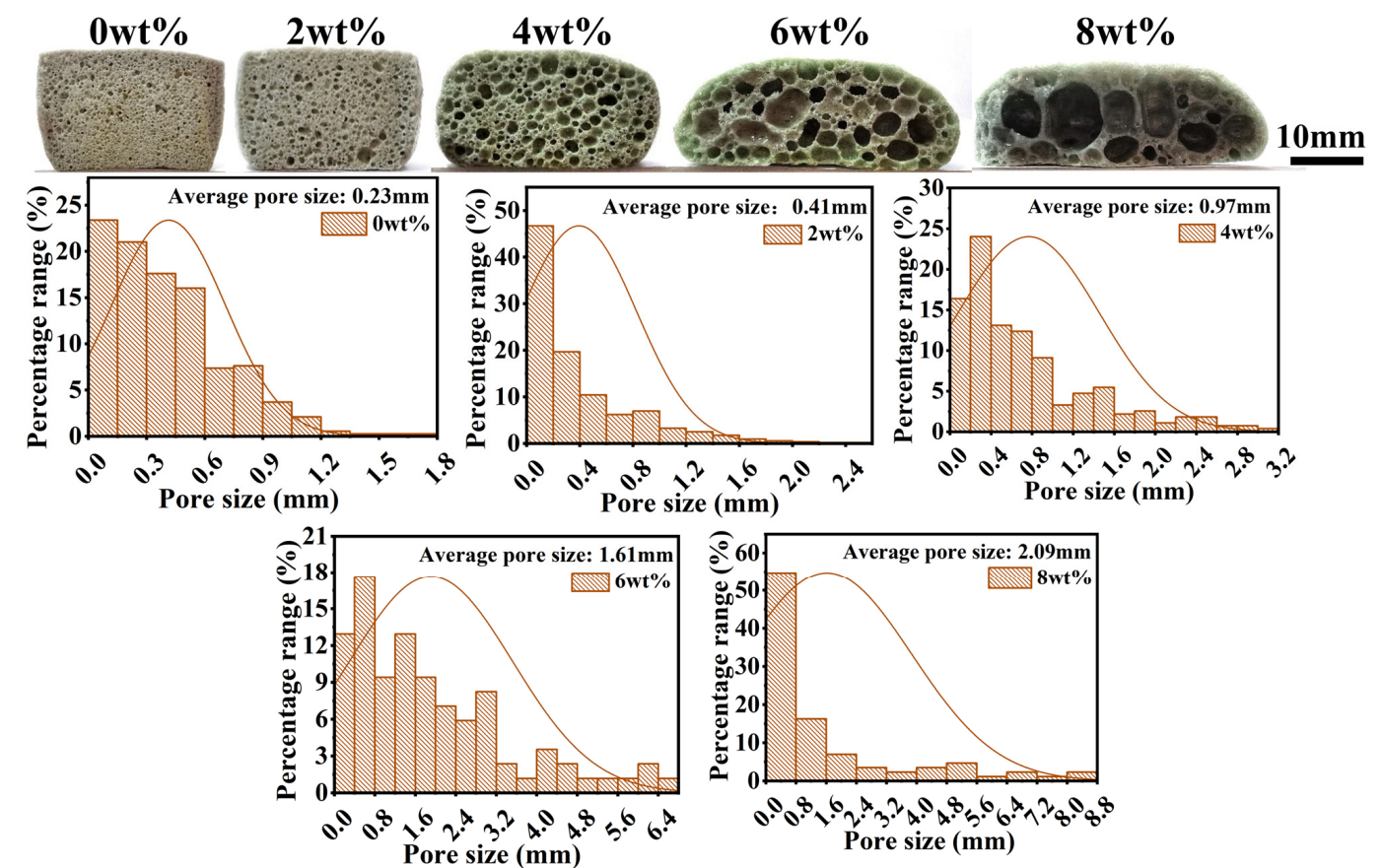


Figure 1. Pore structure morphology and pore size distribution of foamed ceramics with different Na_2O dosages.

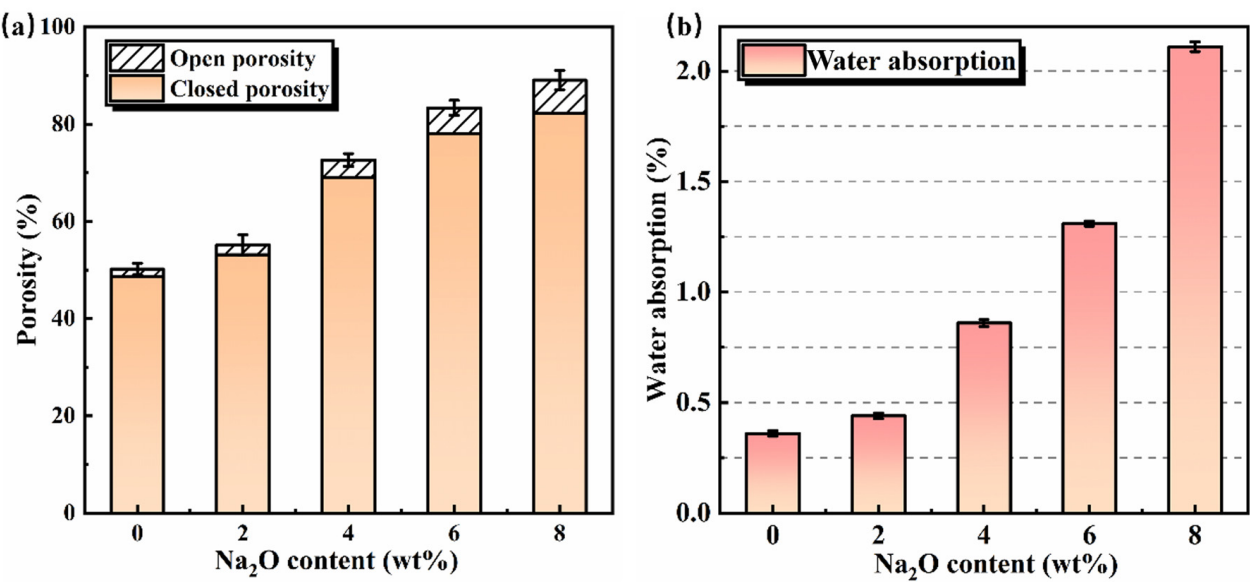


Figure 2. The porosity and water absorption of foamed ceramics with different Na_2O dosages. (a) Open porosity and closed porosity, (b) Water absorption.

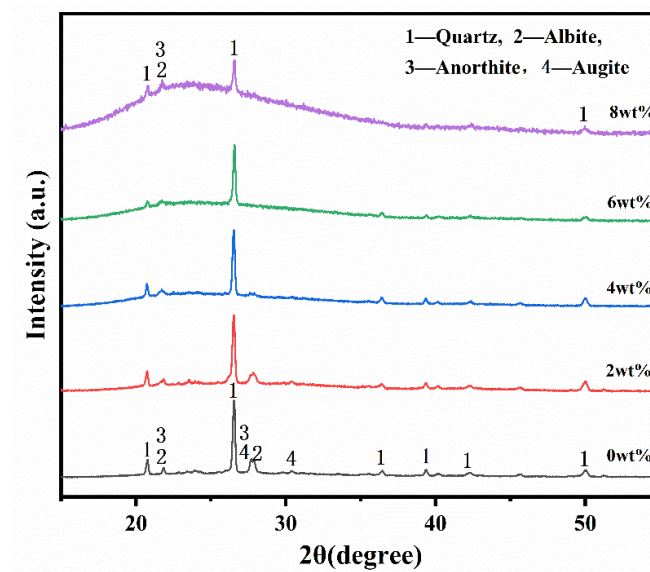


Figure 3. XRD pattern of samples with different Na_2O dosages.

There was a negative correlation between bulk density and porosity: the bulk density decreased while porosity increased. As shown in Figure 4, with the increase in the Na_2O dosage from 0 wt% to 8 wt%, the bulk density of the samples gradually decreased from 826 kg/m^3 to 461 kg/m^3 . Foamed ceramics bear pressure from the pore walls, and an increase in porosity led to a decrease in the load-bearing area, resulting in a decrease in compressive strength. The uniformity of the pore structure also affected the compressive strength of the foamed ceramics, as a uniform pore structure reduced the stress concentration, while poor pore structure uniformity led to significant stress concentrations during load bearing, leading to a decrease in compressive strength [18]. The reduction in crystalline phase content was another factor contributing to the decline in the mechanical properties of the foamed ceramics [19]. Therefore, with the increase in Na_2O dosage, the compressive strength of the samples decreased from 6.85 MPa to 0.91 MPa. Sample N8 exhibited a greater number of large pores and an uneven pore structure distribution, which led to a significant decrease in compressive strength.

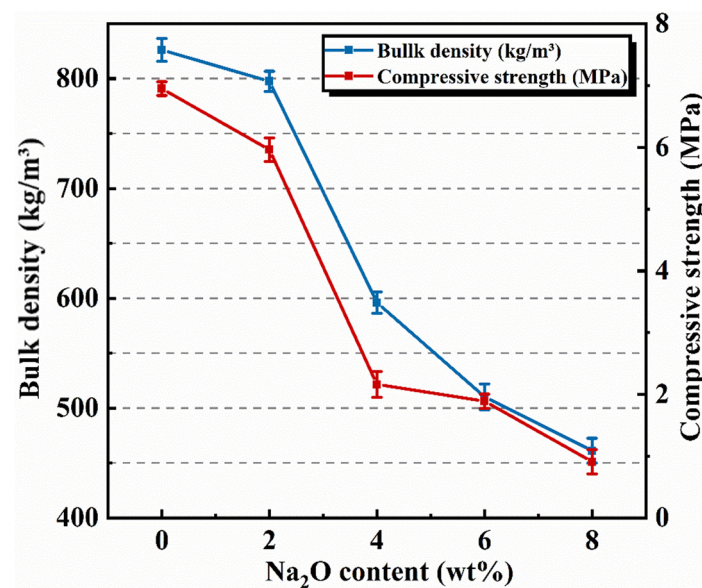


Figure 4. The bulk density and compressive strength of foamed ceramics with different Na_2O admixtures.

The above analysis indicates that Na_2O is an effective fluxing agent that can significantly reduce the viscosity of the liquid phase, facilitate the generation of large quantities of the liquid-phase material, and enhance the ability of the green body to form pores at high temperatures. When the Na_2O dosage was 4–6 wt%, the pore structure and properties of the sample improved, with a bulk density of 510.36–596.11 kg/m^3 , a closed porosity of 69.02–78.04%, a compressive strength of 1.89–2.16 MPa, and a water absorption capacity of 0.86–1.31%. But exceeding the appropriate amount of Na_2O had adverse effects on the pore structure and properties of the foamed ceramics.

3.2. The Effect of K_2O Content on Foamed Ceramics

Figure 5 shows the pore structure morphologies of the foamed ceramics with different K_2O dosages referred to as K0, K6, K8, and K10. As the K_2O content increased, the pore size of the samples gradually increased. In some regions, the gas broke through the liquid phase and penetrated large pores with fully formed shapes. The pore size distribution expanded from 0 to 2.0 mm for sample K0 to 0 to 4.5 mm for sample K10, as shown in Figure 6. The role of K_2O was similar to that of Na_2O discussed in the previous section. It acted as a typical modifier of the silicate network, reducing the viscosity of the liquid phase and decreasing the expansion resistance of the pores [20], making it easier for the pores to expand and grow. But when the K_2O content exceeded the appropriate range, the lower viscosity of the liquid phase caused the pores to aggregate and merge with each other, resulting in the appearance of larger pores with irregular shapes, as evidenced, for example, by the uniformity of the pore structure in sample K10 being disrupted.

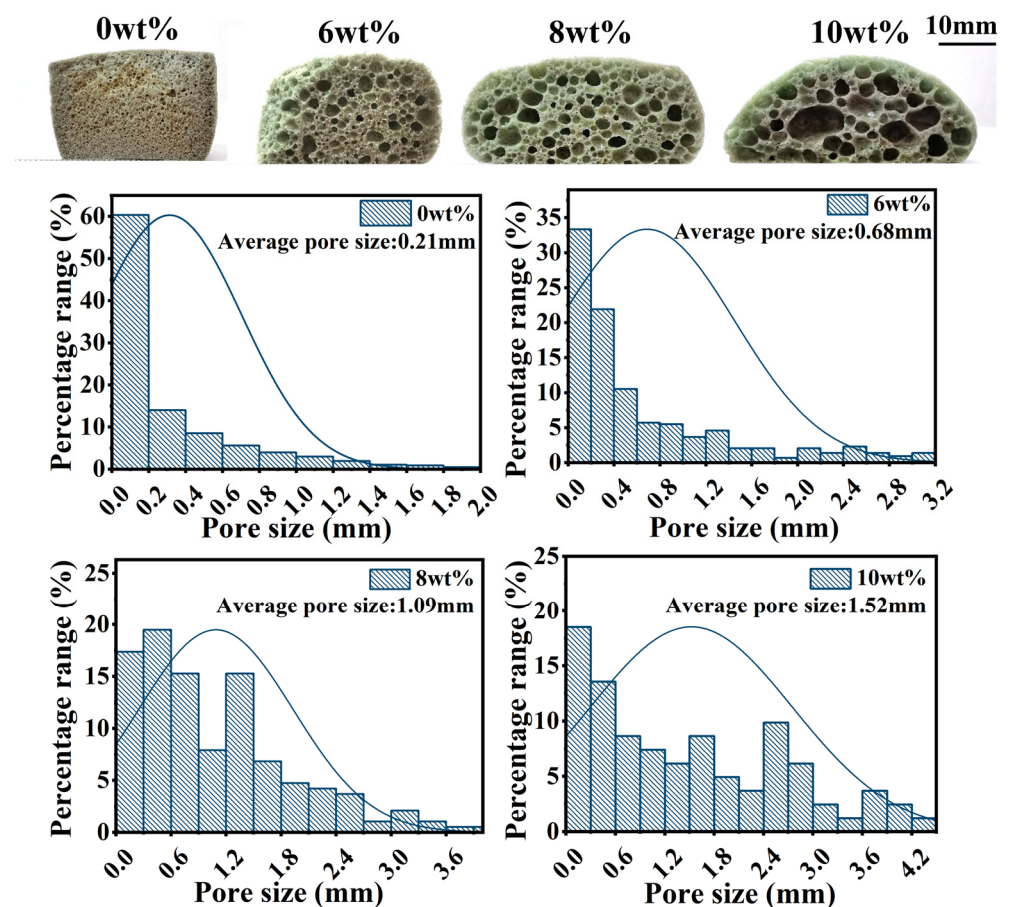


Figure 5. Pore structure morphology and pore size distribution of foamed ceramics with different K_2O dosages.

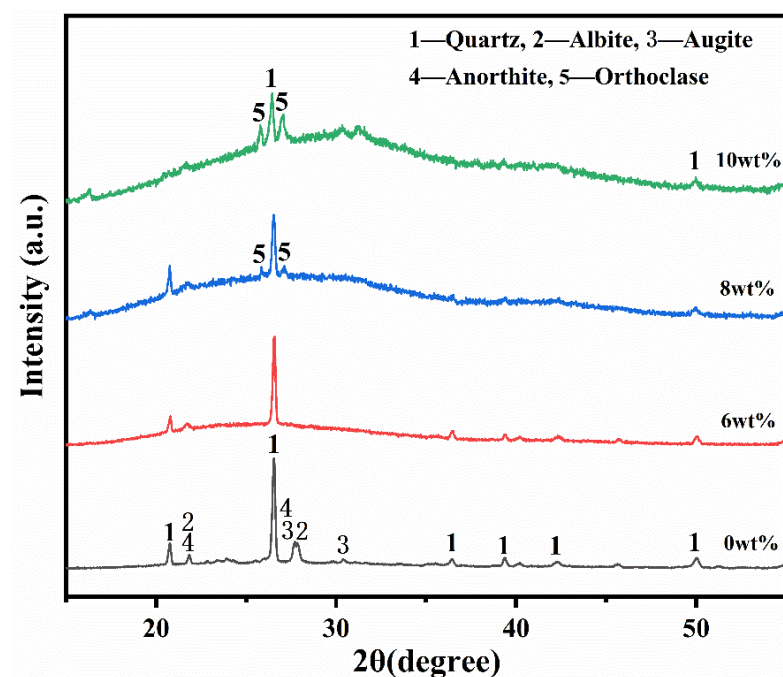


Figure 6. XRD patterns of samples with different K₂O dosages.

Figure 6 shows that the change in K₂O content had an impact on the phase composition of the foamed ceramics. The XRD spectra of samples K8 and K10 exhibit a prominent amorphous hump, while the peaks corresponding to quartz and feldspar are significantly weakened. This indicates that the increase in K₂O content promoted the melting of quartz and feldspar, resulting in the formation of more glassy phases, which are more conducive to the generation and growth of more pores, leading to changes in the pore structure. At the same time, the increase in K₂O content promoted the formation of potassium feldspar, and the crystalline phases that precipitated on the pore walls play a role in “particle stabilization” [21]. Therefore, the magnitude of the changes in pore structure for samples K8 to K10 was reduced.

The pore structures and phase compositions of the foamed ceramics directly determined their physical properties. As shown in Figure 7, as the K₂O dosage increased from 0 wt% to 8 wt%, the number of pores and pore size increased, resulting in a gradual increase in porosity from 50.16% to 81.69%. The trend in bulk density was opposite to that of porosity, gradually decreasing from 822 kg/m³ to 489 kg/m³. The reduced area of the pore wall that bears the load led to a gradual reduction in compressive strength from 6.21 MPa to 1.12 MPa. The increase in K₂O content lowered the viscosity of the liquid phase, making it easier for gas to escape and form interconnected, large voids and open pores. The foamed ceramics absorbed water through these open pores, resulting in a rise in water absorption.

In summary, an increase in K₂O dosage increased the generation of the liquid phase and reduced its viscosity and altered the phase composition of the foamed ceramics, resulting in an increase in pore size and porosity, a decrease in bulk density (as well as a decrease in compressive strength), and an increase in water absorption. The addition of an appropriate amount of K₂O can improve the pore structure and enhance the overall performance of foamed ceramics. When the K₂O dosage was 6–8 wt%, the pore structure and properties of the sample were relatively better, with a bulk density of 523.69–596.33 kg/m³, a closed porosity of 68.24–72.40%, a compressive strength of 1.33–2.66 MPa, and a water absorption capacity of 0.57–0.69%.

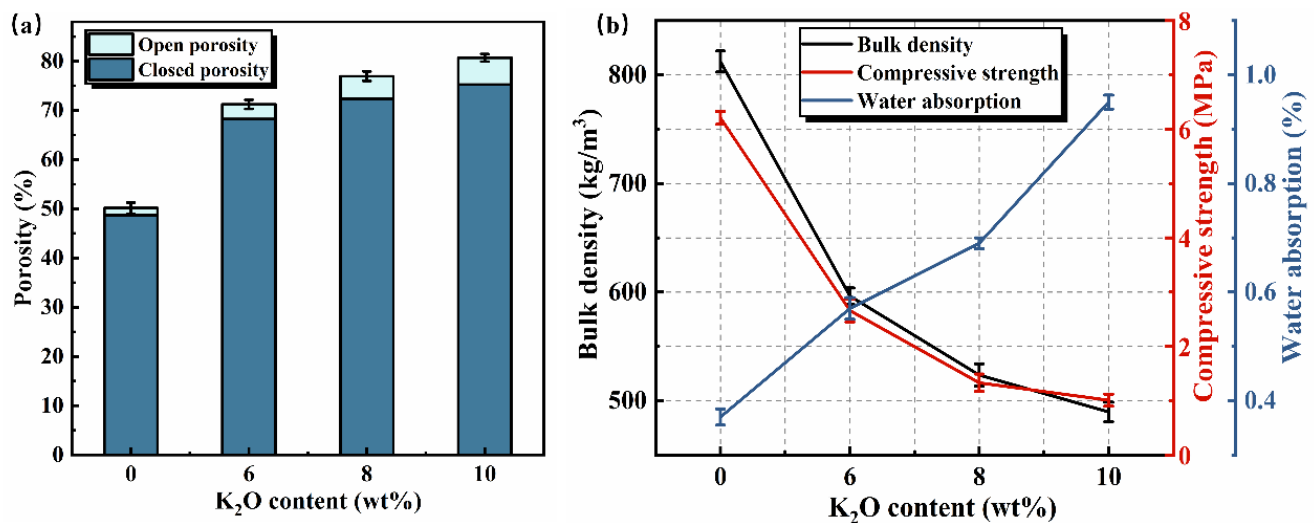


Figure 7. The physical properties of samples with different K₂O dosages. (a) Open porosity and closed porosity, (b) Bulk density, compressive strength and water absorption.

3.3. The Effect of MgO Content on Foamed Ceramics

Studies have indicated that alkaline-earth oxides will strongly reinforce the fluxing effect of alkali oxides during ceramic sintering [22,23]. As observed from the pore structure morphologies of the samples prepared with different MgO dosages in Figure 8, samples with different MgO dosages were referred to as M0, M2, M4, M6, and M8. The change in MgO content had a slight influence on this morphology. The pore size of the samples initially increased and then decreased with the increase in MgO content, and no significant large pores were observed in the prepared samples. MgO acted as a network modifier, disrupting the continuity of the silicate structure by breaking Si-O bonds and forming non-bridging oxygen, thereby reducing the viscosity of the liquid phase. Additionally, MgO can increase the surface tension of the liquid phase [24]. When the MgO dosage was below 4 wt%, the reduction in the viscosity of the liquid phase led to a decline in the expansion resistance of the pores. Therefore, the pore size gradually increased from sample M0 to M4. The gas pressure inside the pores was proportional to the surface tension of the liquid phase. This increased surface tension made it difficult for the pores to break through the pore walls and interconnect with each other, limiting the range of pore enlargement and preventing the formation of large pores. When the MgO dosage was in the range of 6–8 wt%, the effect of increased surface tension outweighed the decrease in the viscosity of the liquid phase, resulting in an enhancement in the expansion resistance of the pores. As a result, the pore size gradually rose. The ability to form pores was mainly influenced by the generation of the liquid phase at high temperatures, while the sintering conditions remained unchanged. The changes in phase composition due to variations in MgO dosage are depicted in Figure 9. The differences in the generation of the liquid phase between different samples were not significant. The increase in MgO dosage did not greatly promote the generation of the liquid phase, but it did promote the formation of forsterite, and the diffraction peaks corresponding to forsterite gradually intensified. The precipitation of a large number of solid-phase grains impeded the growth of pores [25], and the presence of a small amount of liquid-phase material was also not conducive to the expansion and enlargement of the pores, so the generated pores were relatively small.

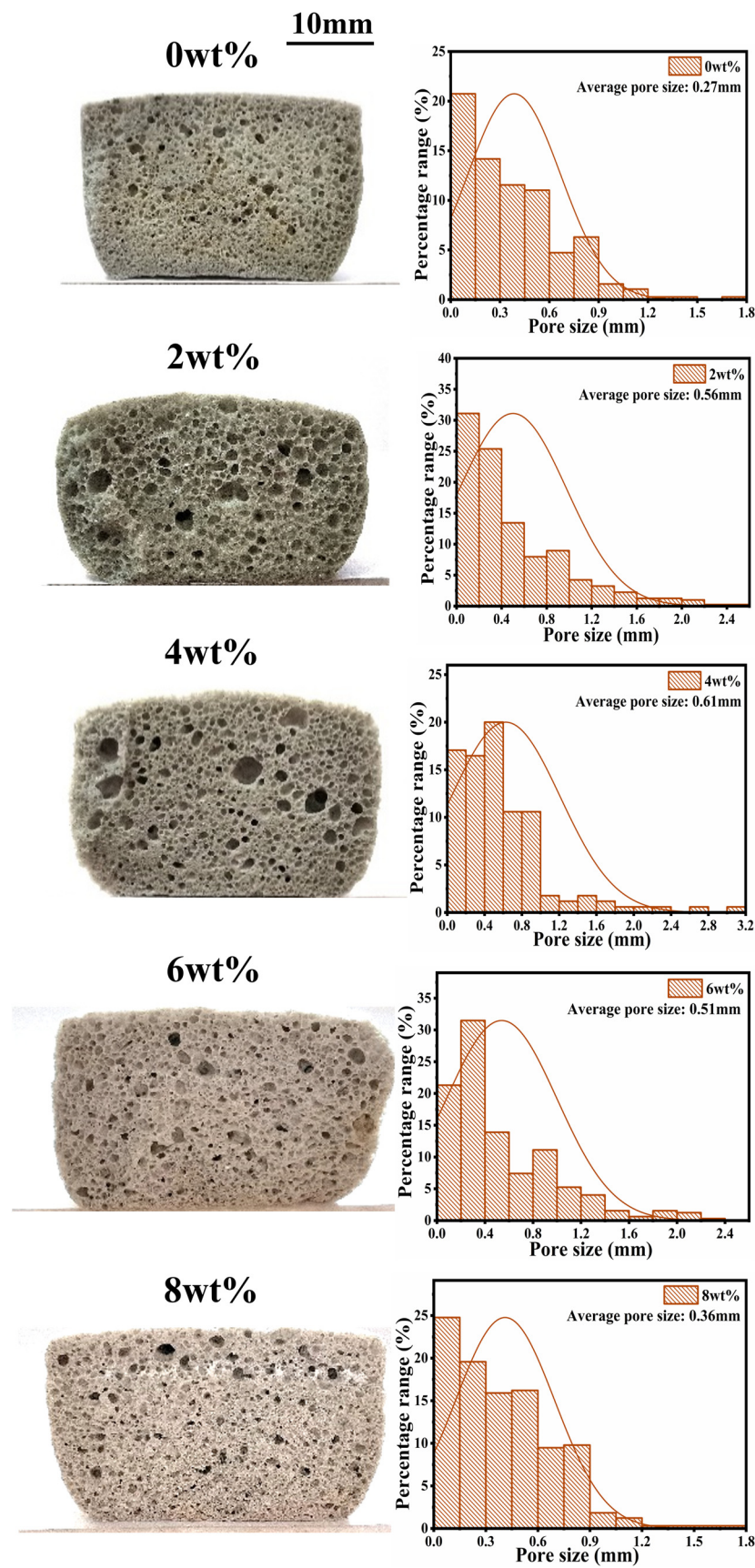


Figure 8. Pore structure morphology and pore size distribution of foamed ceramics with different MgO dosages.

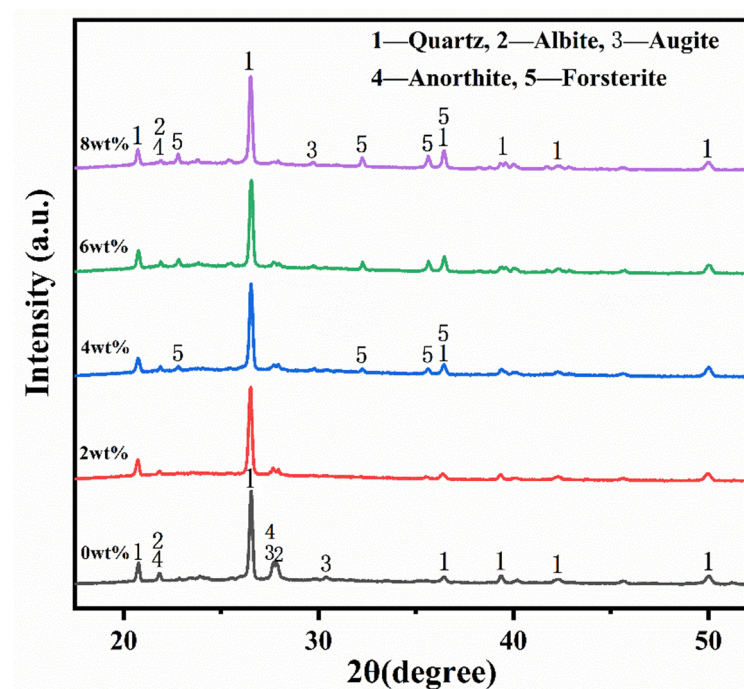


Figure 9. XRD pattern of samples with different MgO dosages.

The changes in the physical properties of the foamed ceramics were consistent with the changes in pore structure. As shown in Figure 10, with an increase in MgO content, the porosity of the samples initially increased and then gradually decreased, while the bulk density decreased first and then gradually increased. The compressive strength was primarily determined by the area of the pore walls under compression, resulting in a decrease followed by an increase. The compressive strength was also influenced by the phase composition, and the formation of a forsterite crystal phase contributed to an improvement in compressive strength [26]. Therefore, while the compressive strength of sample M-4 was affected by changes in the pore structure, the presence of forsterite crystals mitigated the decrease in compressive strength. Samples M-6 and M-8 exhibited a significant increase in compressive strength. When the MgO dosage increased slightly (0–4 wt%), the viscosity of the liquid phase decreased due to its fluxing effect, and part of the gas escaped, increasing the opening of the pores, so the water absorption rate initially rose. When the MgO dosage (6–8 wt%) continued to increase, the surface tension of the liquid phase increased, resistance to the expansion of the gas pores increased, and less gas broke through the barrier of the liquid phase, so the water absorption rate decreased.

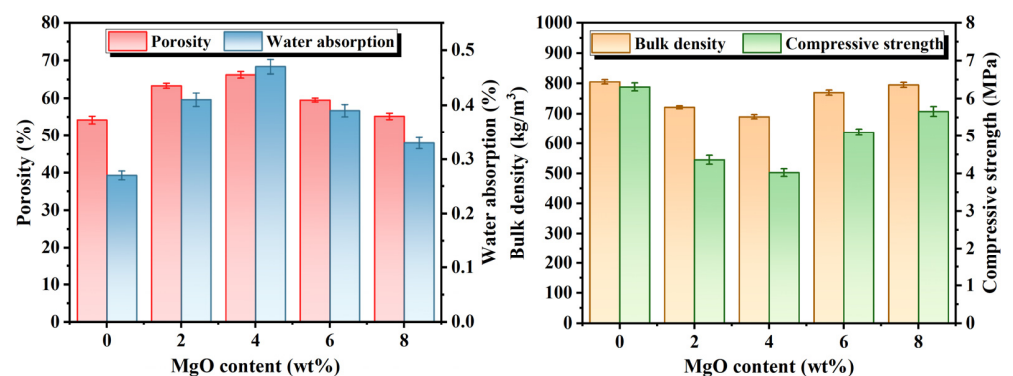


Figure 10. The physical properties of samples with different MgO dosages.

In summary, MgO reduced the viscosity of the liquid phase while increasing its surface tension, changing the pore structure of the foamed ceramics by a slight margin, and it was suitable for the preparation of foamed ceramic materials with small pores.

4. Conclusions

In this study, by adding the chemically pure reagents Na_2CO_3 , K_2CO_3 , and MgO to change the content of Na_2O , K_2O and MgO in GS, the effect of flux components on the pore structure and properties of foamed ceramics was investigated. The results showed that increasing the Na_2O and K_2O content had a significant effect on improving the foaming ability of the green body, reducing the viscosity of the melt and increasing the amount of liquid phase generated, thereby promoting the growth of pores and widening the pore size distribution, resulting in an increase in porosity and a decrease in bulk density. With an Na_2O doping amount of 4–6 wt%, the shapes of the pores were relatively regular, and the pore size was moderate; with a K_2O doping amount of 6–8 wt%, the pore structure was relatively homogeneous, and the prepared foamed ceramics had the most optimal physical properties, with a bulk density of 510.36–593.33 kg/m^3 , a closed porosity of 68.24–78.04%, a compressive strength of 1.33–2.66 MPa, and a water absorption capacity of 0.57–1.31%. MgO could reduce the viscosity of the melt and increase the surface tension of the liquid phase, but it had a slight effect on the pore structure of the foamed ceramics. Increasing the amount of doping promoted the precipitation of magnesium olivine crystals, which played a role in stabilizing the pores, and this aspect makes this a suitable method for preparing foamed ceramics with small pores. This study not only provides a theoretical basis for the optimization of the blank system design of granite scrap-based foamed ceramics, but is of great practical importance. The obtained foamed ceramics can be used in thermal insulation, light aggregates, catalyst carriers, and automobile exhaust purification in the future.

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