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PREPARATION OF POROUS THERMAL INSULATION CERAMICS BASED ON LOW GRADE POTASSIUM FELDSPAR

XINCHI LI

School of Architecture, University of Sheffield, Sheffield S102TN, United Kingdom

#E-mail: lhanlandclee@163.com

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To address the comprehensive utilisation of low-grade potassium feldspar and further enhance the thermal insulation performance of green building facades, this study proposes the use of low-grade potassium feldspar as the primary raw material, with silicon carbide (SiC) serving as the foaming agent. Porous heat-insulating ceramics for green building exterior walls were prepared by the high-temperature foaming method. The test results demonstrate that the porous ceramic material prepared at a sintering temperature of 1250 °C and a holding time of 30 min exhibits the optimal performance, with a thermal conductivity of 0.072 W/(m-K), a compressive strength of 3.429 MPa, a water absorption rate of 13.5 %, and a bulk density of 0.542 g·cm^{-3}. With the rise in sintering temperature and the progression of the foaming reaction, the pores within the ceramic material become increasingly concentrated, leading to a generally denser pore structure. With the increase in the sintering temperature, the foaming reaction continued, as the pores in the ceramic material became increasingly dense, the overall pore structure developed greater regularity and uniformity in size, while the pore dimensions gradually increased.

INTRODUCTION

With an annual addition of nearly 2 billion square metres in new construction, China experiences extremely high building energy consumption [1, 2]. As a result, the development of high-performance building insulation materials has attracted significant attention from researchers and engineers [3]. Air possesses significantly lower thermal conductivity compared to most solid materials, and the abundance of pores within porous insulation materials can effectively impede the heat transfer, thereby contributing to a reduction in energy consumption. In recent years, there have been more studies related to porous thermal insulation ceramic materials [4]. In comparison to organic thermal insulation materials, porous thermal insulation ceramics have excellent fire resistance and durability, are environmentally friendly, non-polluting and safe. Compared to traditional inorganic thermal insulation materials, porous thermal insulation ceramics possess a lower water absorption rate, enhanced chemical stability, and more stable thermal insulation performance. Foaming methods are currently one of the most commonly adopted methods for the preparation of porous ceramics, have the advantages of simple process, outstanding foaming effect, etc. The gas generated by the foaming agent at high temperatures creates a large number of closed pores within the material, promoting the formation of an effective thermal insulation structure. As a result, the foaming method offers a significant advantage in the preparation of porous heat-insulating ceramics for green building facades.

Using low-grade non-metallic minerals or solid waste as the raw materials for the production of porous thermal insulation ceramics offers advantages such as low raw material costs and high resource utilisation rates [5, 6], which have become a hot research topic in recent years. Potassium feldspar is a silicate mineral with a layered structure, characterised by a high melt viscosity and extended melt intervals. When melted to form a viscous melt phase, it can lower the melting temperature of the billet during the ceramic firing process [7], conducive to the formation of ceramics and lower firing temperatures. Accordingly, high-quality potassium feldspars with a potassium content of more than 11 % are widely utilised in the production of ceramics as frits

and fluxes. Nevertheless, a large amount of low-grade potassium feldspar is discarded in the process of utilising high-quality potassium feldspar, resulting in a serious waste of resources and environmental pollution. Silicon carbide is a commonly used inorganic foaming agent that, at high temperatures, can react with alkali metal oxides to form a silicate liquid phase. This reaction helps achieve better foaming results in the building material at lower temperatures [8-10]. Consequently, in this study, low-grade potassium feldspar tailings were adopted as the main raw material, silicon carbide (SiC) was added as a foaming agent, and the high-temperature foaming method was employed to prepare porous heat-insulating ceramic materials for green building facades. This paper presents a comprehensive study on the preparation of porous thermal insulation ceramics for green building facades and their thermal insulation performance. It explores a new approach for the utilisation and treatment of potash feldspar tailings, which holds significant importance for the effective utilisation of low-grade potash feldspar mineral resources, the industrialisation of low-cost thermal insulation ceramics, and for energy saving, consumption reduction, and environmental protection.

LITERATURE REVIEW

Makrygiannis.et al. [11] employed SiC and carbonaceous materials as raw materials, which were mixed in a specific ratio and thoroughly blended through ball milling. Alumina-SiC powders with excellent properties were prepared by using processes including a carbothermal reduction reaction and the removal of residual carbon. Among them, α-Al_2O_3 is 23 % - 65 % (by mass), and the β-Si C crystal phase content is 32 % - 70 % (by mass). The powder exhibits a high degree of physical phase dispersion and a fine particle size. Due to its low production cost, it can be utilised in industrial production to enhance the utilisation rate of SiC. However, the thermal insulation performance of this material is limited, making it unsuitable for application in cold northern regions. Furthermore, Khan et al. [12] synthesised porous mullite ceramics by using decontaminated low-grade gangue as the predominant raw material and crosslinked propionic acid resin and alumina as the pore-making agent. Moreover, the effect of pore-making agent content and size on porous mullite materials was further investigated. It was observed that the apparent porosity of the porous mullite ceramic material increased with the rise in the content of cross-linked acrylic resin and alumina. As the content of the foaming agent increased, the bulk density and heat resistance decreased. However, the material exhibited poor mechanical properties and had a high cost. Consequently, there is a need to develop cost-effective materials with improved thermal insulation properties.

EXPERIMENTAL

Raw materials

In this study, low-grade potassium feldspar tailings with a particle size of less than 150 mesh were adopted as the main raw material, provided by a mining company in Luonan, Shaanxi Province [13]. The specific chemical composition of the used potassium feldspar is provided in Table 1. The particle size distribution of potassium feldspar powders is irregular, with individual particles having relatively large sizes, approximately 10 μm. The majority of the particles have a particle size of only 1-2 μm, even in the sub-micron range. Moreover, the size of the powder particle size affects its activity, and a smaller particle size of the raw material is favourable to reduce the melt temperature of the billet and enhance the viscosity homogeneity of the molten liquid state [14].

Table 1. Chemical composition of the raw materials.

Chemical Components	SiO_2	Al_2O_3	K_2O	Fe_2O_3	Na_2O	CaO	MgO
potassium feldspar	46.76	10.78	9.22	1.30	1.24	0.302	0.183

Major instruments and equipment

The following instruments and equipment were used: An X-ray fluorescence spectrometer (XRF), ARL-PERFORM-X, Thermo Fisher, USA. Laser Particle Size Analyser, Winner 2308, Jinan Microparticle Instrument Co. An X-ray Powder Diffractometer, Mini Flex 600, Rigaku, Japan. A Thermal Conductivity Tester, TCi, C-THERM, Canada. An Electronic Universal Testing Machine, WDW-100, Jinan Sida Testing Technology Co. A Super Depth of Field 3D Microscope, VHX-5000, Keenshine. A Planetary Ball Mill, YXQM-4L, Changsha Miqi Instrument Co.

Sample preparation

Potassium feldspar was mixed with 1 wt. % SiC by mass and dry ball milled for 30 minutes using a planetary ball mill. After ball milling, 13 wt. % water was added to the mixture and thoroughly stirred [15]. The wetted powder mixture was loaded into a mould (50 mm in length, width, and height) and pressed under a pressure of 10 MPa for 60 seconds. The resulting

green bodies were dried at 105 °C for 10 hours and then sintered. This sintering temperature allows the material's microstructure to gradually compact. If the sintering temperature is too high, fusion between ceramic particles may occur, potentially causing deformation, expansion, and cracking of the ceramic sheet, which would degrade its overall performance [16]. The sintering was conducted as follows: firstly, the temperature was increased to 300 °C at a rate of 15 °C·min^{-1} and held for 50 min, and then the temperature was increased to the final sintering temperature at a rate of 15 °C·min^{-1} and held for a period of time [17]. This article tests and characterises the volume density, water absorption rate, thermal conductivity, compressive strength, morphology, and other physical properties. The experimental data are divided into three groups, and the average value of each group is used for the calculations (Figure 1).

Figure 1. Photographs of the composite samples.

RESULTS AND DISCUSSION

Particle size and physical phase analysis of insulation materials

The particle size distribution of the raw material is shown in Figure 2, and the morphology of the powder raw material is shown in Figure 3. Figure 2 indicates that the particle size of the potassium feldspar raw material powder is primarily distributed between 1-13 μm, with an average particle size of 3 μm. Particles with a size of 2 μm or less account for 50 % of the total. It can also be observed from Figure 3 that the distribution of the particle size of potassium feldspar powder is not uniform, and the individual powders have larger particle sizes, around 10 μm. Most of the particles have a size ranging from 1-2 μm, with some even reaching sub-micron sizes, which is consistent with the particle size distribution results shown in Figure 2. The particle size of the powder affects its activity, as well as a smaller particle size of the raw material is conducive to lowering the melt temperature of the billet and enhancing the uniformity of the liquid phase distribution in the melt state [18], which, in turn, improves the uniformity of the distribution of the closed pores in the billet.

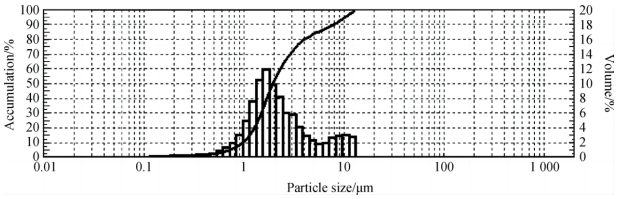

Figure 2. Particle size distribution of the potash feldspar powder.

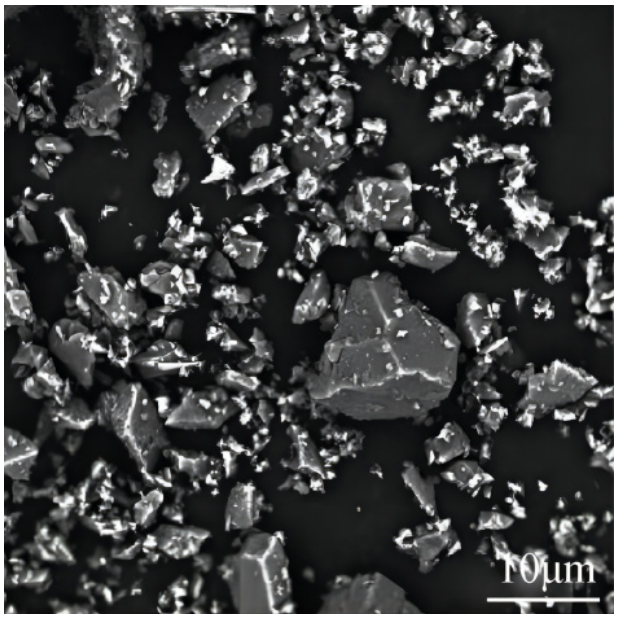

Figure 3. SEM image of the potash feldspar powder.

Effect of the sintering temperature on the properties of porous thermal insulation ceramics

The cross-sectional morphology of the porous ceramic samples at different sintering temperatures is indicated in Figure 4. It can be observed that the porous ceramic samples at lower sintering temperatures exhibit fewer and smaller pore diameters, irregular shapes, and thicker pore walls, as shown in (a) and (b) in Figure 4. This is because, at low temperatures, the foaming reaction is not fully executed, resulting in low gas generation and a low internal pore pressure, which hinders the formation of larger pores [19]. As the sintering temperature increases, the foaming reaction continues and the chemical reaction rate rises, the pores in the ceramics become denser and denser, the pore

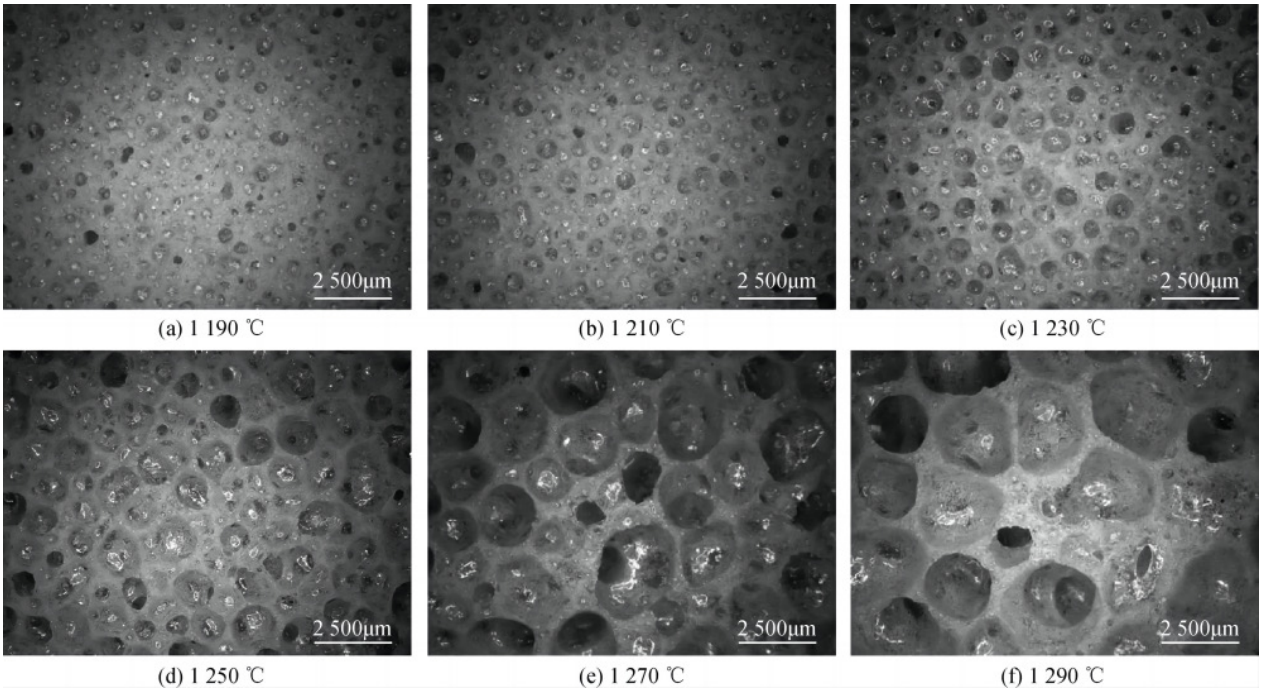

Figure 4. Ultra depth of the field morphology of cross sections of ceramic samples at different sintering temperatures.

structure, in general, becomes regular and uniform in size, and the size of the pores gradually increases, as indicated in Figure 4 (c) and (d). Nonetheless, at too high sintering temperatures, the shape of the pores in the samples changed from spherical to irregular, the size of the pores was not uniform, and even some through pores appeared, as well as the walls of the pores became very thin, as illustrated in (e) and (f) in Figure 4.

The variation in bulk density and water absorption with the sintering temperature for the porous thermal insulation ceramic samples for green building facades is shown in Figure 5, while the variation in the thermal conductivity with the sintering temperature is presented in Figure 6.

As can be seen in Figure 5, the bulk density of the samples decreases and the water absorption increases as the sintering temperature of the building envelope ceramics increases. Moreover, the changes in bulk density and water absorption are both associated with changes in the pore structure in porous ceramic materials. During the foaming process of porous ceramics for green building facades, when the foaming agent SiC begins to

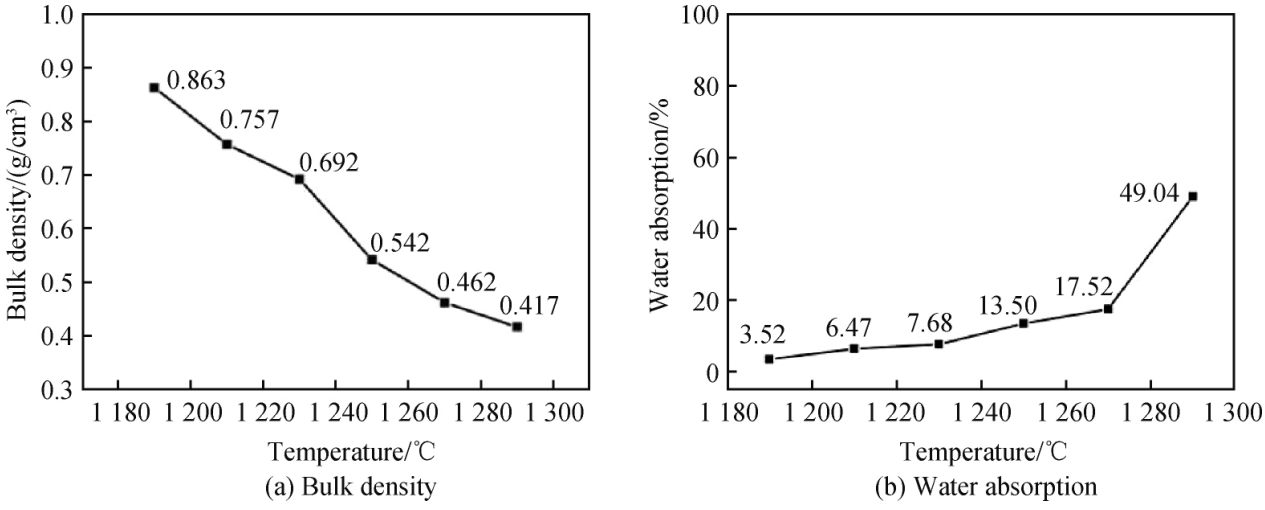

Figure 5. Changes in the bulk density and water absorption of the samples with different sintering temperatures.

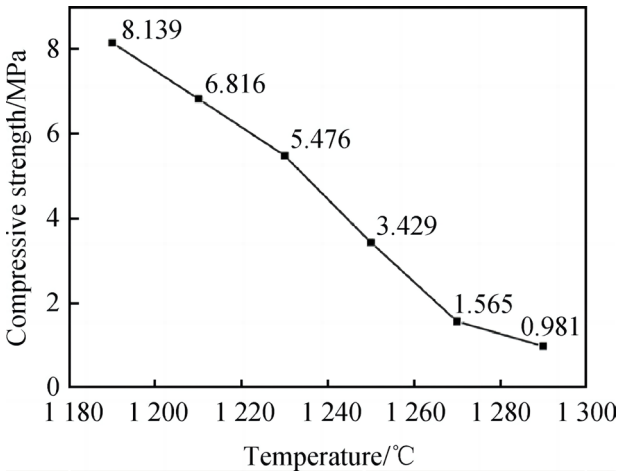

Figure 6. Changes in the thermal conductivity of the samples with different sintering temperatures.

react, the gas generated is initially dissolved in the high-temperature melt. As the temperature rises, the amount of gas generated by the blowing agent increases, the solubility of the gas phase in the melt decreases, and, on the condition that the critical point is reached, the gas begins to appear in the form of bubbles [20]. As a result, only a small number of small pores can be formed in the ceramic billet during the low-temperature stage, and the ceramic samples remain relatively dense, with the majority of the material consisting of a solid content. At this point, the sample has a higher bulk density and lower water absorption rate. When the temperature continues to rise, the melt surface tension as well as viscosity is reduced, the gas expansion resistance is reduced, conducive to the expansion of gas growth, as the foaming agent SiC reacts more intensely, this stage marks the peak of the ceramic foaming process, where it is easier to form a dense pore structure with uniform pore sizes [21]. Nonetheless, as the temperature continues to increase and the viscosity of the melt continues to decrease, small pores in the sample tend to merge to form large pores and even break out of the melt. Additionally, when the pressure inside the pore is greater than the surface tension of the melt, some bubbles will rupture, resulting in an increase in the open pore rate [22]. Therefore, as the number and size of pores in the sample increase, along with the increase in the number of open pores, the bulk density of the sample decreases, and the water absorption rate increases. The thermal conductivity of green building exterior wall insulation materials is generally required to be less than or equal to 0.12 W/(m·K), while the thermal conductivity of water at room temperature is approximately 0.5 W/(m·K), and the thermal conductivity of ice at 0 °C is 2.23 W/(m·K), which is higher than that of commonly used green building exterior wall insulation materials. If the material's water absorption rate is excessively high, in the process of employing the building thermal insulation material, it will reduce its green building exterior wall thermal insulation performance due to the water absorption of the material [23], and consequently it is necessary to control the water absorption rate of the sample.

Heat transfer in porous materials primarily depends on the thermal vibration of atoms and molecules in the solid phase and the collision of gas molecules within the pores. The heat transfer capacity of the gas phase is significantly lower than that of the solid phase. When heat is transferred along the solid, the encountered pore structure will change the direction of its transfer, prompting its transfer line to become longer, which can effectively slow down the heat loss [24]. As shown in Figure 6, the thermal conductivity of the porous ceramic samples for green building facades decreases with an increase in the sintering temperature. The reason for this is that as the sintering temperature increases, the internal pore structure of the ceramic body becomes more developed, with the majority of pores being closed. This independent closed pore structure effectively reduces the convection effect of air, resulting in a decrease in thermal conductivity.

The variation in the compressive strength with the sintering temperature of porous thermal insulation ceramic samples for green building facades is indicated in Figure 7. As illustrated in Figure 7, the compressive strength of the samples declined from 8.139 MPa to 0.981 MPa with the increase in the sintering temperature from 1190 °C to 1290 °C. Moreover, the compressive strength of a material is closely related to its bulk density and pore structure. As observed in the ultra-depth-of-field images of the samples in Figure 5, the degree of foaming in the ceramics increases progressively with the rise in the sintering temperature, resulting in thinner pore walls. This structural change leads to a reduction in the

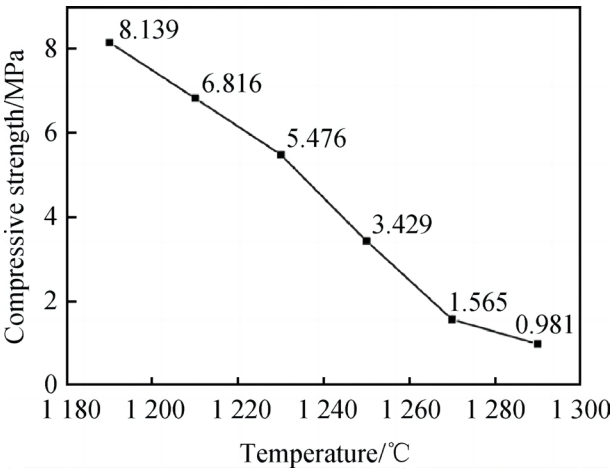

Figure 7. Changes in the compressive strength of the samples with different sintering temperatures.

compressive strength of the porous ceramic materials used for green building facades. From the results of the water absorption and sintering density of porous ceramics, it can be determined that as the sintering temperature increases, the internal pore structure of the ceramics increases, and high sintering temperatures are prone to the formation of large pores. Under the influence of external forces, large pores are more susceptible to cracking, and their formation tends to cause stress concentration, thereby reducing the material's ability to withstand external loads [25], the compressive strength is reduced. In general, the compressive strength of thermal insulation materials used in buildings should exceed 0.3 MPa. In this study, although the lowest thermal conductivity was achieved at 1290 °C, the compressive strength of the material under this condition was relatively low, and the water absorption rate was high. Therefore, considering multiple factors, the optimal sintering temperature for the porous thermal insulation ceramics intended for green building facades was determined to be 1250 °C.

Effect of the soaking time on the performance of porous thermal insulation ceramics

On the basis of determining the optimal sintering temperature of porous ceramics for green building facades as 1250 ℃, the effect of the heat preservation time on the performance of porous ceramics was further investigated. That is, upon reaching the final sintering temperature, the ceramic samples were subjected to the heat preservation process for 20 min, 30 min, 40 min, 50 min and 60 min. The fixed SiC content was 1 wt. %, the ball milling duration was 30 minutes, the added water content was 13 wt. %, and the moulding pressure was 10 MPa.

Figure 8 indicates the variation in the water absorption and compressive strength of the samples with the holding time. As shown in Figure 8, porous ceramic materials intended for green building facades exhibit higher compressive strength and lower water absorption with shorter insulation times. With an increased holding time, the water absorption rises while the compressive strength declines. When the insulation time is shorter, the added SiC foaming agent foaming time is short. Besides, due to insufficient reactions, fewer pores are formed in the ceramic material, resulting in a higher ceramic density, lower water absorption rate, and higher compressive strength. With the prolongation of the insulation time, it promotes the full reaction of the blowing agent, the number of pores increases, and the pore size becomes larger, and it can also effectively reduce the internal temperature gradient of the material during sintering, weakening the tensile thermal stress caused by the temperature gradient. Additionally, the prolongation of the insulation time is likewise conducive to making the vapour pressure distribution inside the material uniform, the vapour pressure effect can uniformly penetrate from the interior to the surface, facilitating the formation of regularly shaped and uniformly sized pores. At this time, in the ceramic samples within the pore content increases dramatically, the density decreases, and the compressive strength is subsequently reduced. On the condition that the holding time is too long, the pores will be further expanded due to the continuous reaction of SiC to generate gas. Especially under prolonged high temperatures, the viscosity of the melt leads to insufficient support of the formed pore walls for the bubbles, causing some small bubbles to rupture and merge into larger pores. This may even result in the formation of interconnected pores, leading to greater pore size variation and increased water absorption.

The variation in the thermal conductivity of porous ceramic samples for green building facades under distinct insulation times is shown in Figure 9. As shown

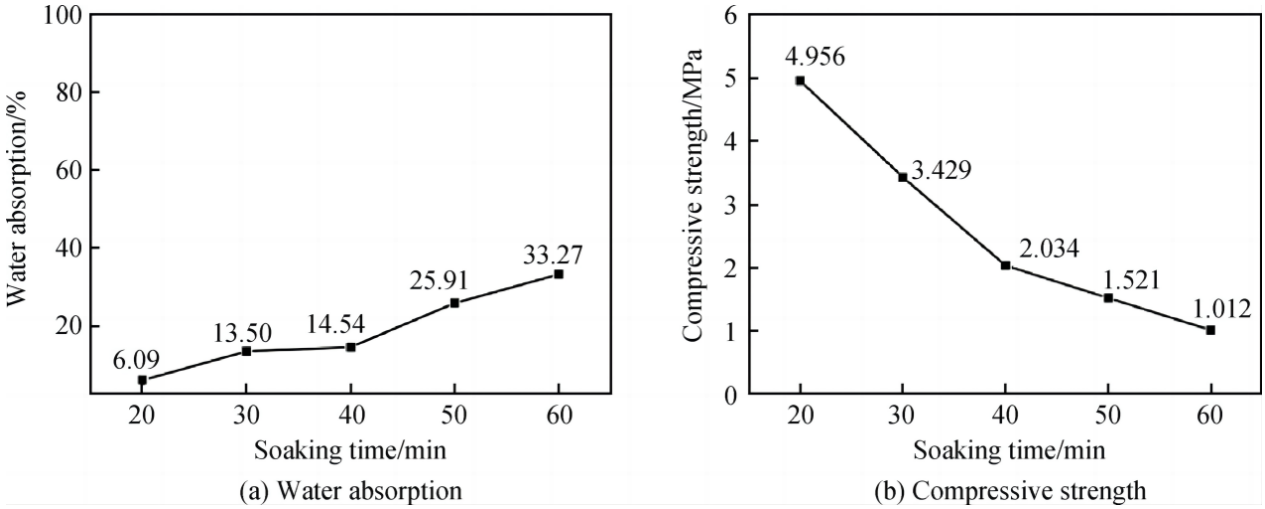

Figure 8. Changes in the water absorption and compressive strength of the samples with different soaking time.

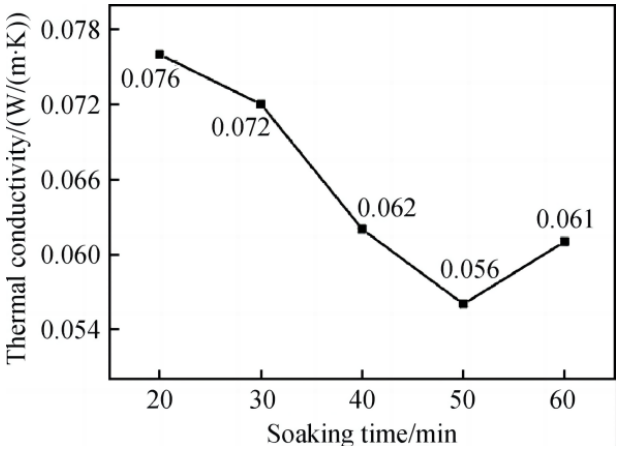

Figure 9. Changes in the thermal conductivity of the samples with different soaking time.

in Figure 9, with the extension of the holding time, the thermal conductivity of the samples first decreases and then increases, reaching its minimum value at a holding time of 50 minutes. With a short holding time, the number of air pores is small, the pore size is limited, the pore distribution is uneven, and as a result, the thermal conductivity of the sample is relatively high. With the prolongation of heat preservation time, porous ceramics within the pore size increases, and the distribution is uniform [26], greatly reducing the thermal conductivity of the material. Nevertheless, on the condition that the thermal insulation time is too long, the air holes formed within the porous ceramic of the green building facade gradually form through holes, uneven distribution of air holes, structural collapse. At this point, although the material's density is very low, the presence of interconnected pores enhances the convection effect between the gases, thereby reducing the thermal insulation properties of the porous ceramics and causing an increase in the material's thermal conductivity. At a holding time of 50 min, the compressive strength was merely 1.521 MPa, although the thermal conductivity was the lowest (Figure 9(b)). Consequently, the water absorption rate, compressive strength and thermal conductivity of porous ceramics in green building facades under distinct heat preservation times, a heat preservation time of 30 minutes is more appropriate. At this duration, the thermal conductivity of the material is 0.072 W/(m·K), the compressive strength is 3.429 MPa, the water absorption rate is 13.5 %, and the bulk density is 0.542 g·cm^{-3}.

Crystal phase composition and structural morphology of the porous thermal insulation ceramic samples

Figure 10 indicates the XRD spectrum of the porous thermal insulation ceramic samples for green building facades. The XRD test results reveal that the sintered specimens contain crystalline SiO_2 and SiC phases, along with a significant amount of glassy phases. The presence of SiC crystalline phases suggests that a small amount of SiC has not fully reacted, which can be attributed to the fact that the oxidation of SiC at high temperatures occurs in two stages: active and inert oxidation. In the event of inert oxidation, a protective SiO_2 film is generated, preventing further reaction of SiC. Nevertheless, as the oxidation temperature increases, on the contrary, the amount of gas produced by the oxidation of SiC increases, and on the other hand, the molten SiO_2 film undergoes crystallisation and the phase transition produces a change in volume, which results in the structure of the SiO_2 film becoming loose, its protective effect is weakened, and SiC is able to undergo further reactions. It can be inferred that the small amount of SiC present in the porous ceramic samples may be residual from the inert oxidation process.

SiC is a high-temperature resistant powder, and an appropriate amount of its addition can increase the firing temperature of the blank. The oxidation of SiC generates a large amount of SiO_2, which also increases the viscosity of the melt. As a foaming agent for the preparation of high-temperature foamed ceramics, the pore formation process involves several stages: glass phase melting, bubble nucleation, bubble growth, and bubble fusion. SiC powder primarily consists of β-SiC and quartz phases. Due to its excellent resistance to high-temperature oxidation, SiC continues to promote foaming even after pre-firing, resulting in slightly more homogeneous foaming.

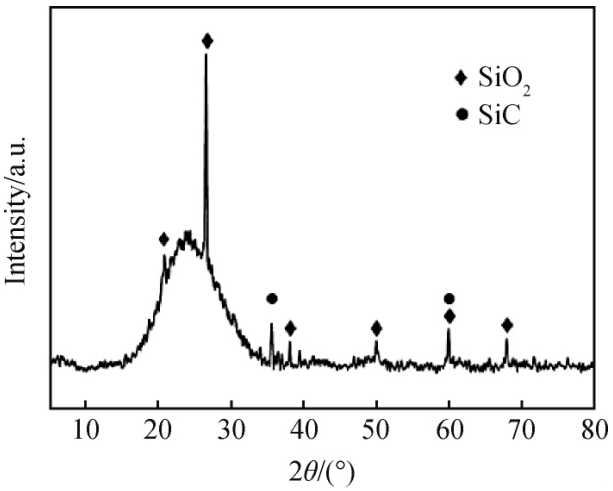

Figure 10. XRD pattern of the potash feldspar porous ceramics.

CONCLUSION

In this study, low-grade potassium feldspar tailings were employed as the main raw material, 1 wt. % SiC was added as the foaming agent, and porous ceramic materials with outstanding thermal insulation properties were prepared by high-temperature foaming method

and controlled sintering system. It was observed that as the sintering temperature and holding time increased, a porous thermal insulation ceramic material with a uniform structure and improved performance could be successfully prepared. Nevertheless, too high sintering temperature and too long holding time will result in the penetration and collapse of the pore structure of foam ceramics, which will affect the performance of the ceramic samples. Furthermore, the optimal sintering temperature is 1250 °C and the optimal holding time is 30 min. Under the optimal experimental conditions, the thermal conductivity, compressive strength, water absorption and bulk density of the green building exterior wall porous insulation ceramic material were 0.072 W/(m·K), 3.429 MPa, 13.5 % and 0.542 g·cm^{-3}, respectively. Additionally, the manufacturing method of porous ceramic materials in this study is simple, green and suitable for large-scale production.

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