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A study on ceramic sintering preparation process and properties with the addition of silicon carbide foaming agent

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Abstract

Foamed ceramics are widely used in construction as a building material. This article used potassium feldspar tailings as the primary raw material and added a silicon carbide (SiC) foaming agent to prepare samples through a sintering process. The impact of three factors − SiC content, sintering temperature and heat preservation time − on sample properties (such as bulk density and water absorption rate) was analyzed using a single-factor variable method. It was found that the increase in SiC content, sintering temperature, and heat preservation time increased pores inside the sample, thereby leading to a higher water absorption rate, lower compressive strength and higher mass loss rate, i.e., decreased sample performance. Overall, the optimal sintering process parameters were as follows: a SiC content of 1.0 wt%, a sintering temperature of 1,250°C and a heat preservation time of 30 minutes. Under these conditions, the obtained sample had a bulk density of 0.54 g/cm^3, a water absorption rate of 13.45%, a compressive strength of 4.75 MPa, a thermal conductivity of 0.06 W/(m·K) and an acid resistance mass loss of 1.21%, exhibiting the optimal performance. The experimental results provide appropriate SiC content and sintering parameters that can be applied in practice to obtain higher-performance foam ceramics.

Keywords: Silicon carbide; Foaming agent; Ceramics; Sintering preparation; Water absorption rate

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1. Introduction

In the face of the escalating contradiction between energy supply and demand, the development of energy-saving products has become a current focus, and building energy efficiency is also an essential content [1]. With building energy consumption intensifying, waste reuse and energy-saving materials have been increasingly widely studied [2]. Foamed ceramics are environmentally friendly materials. Compared with ordinary ceramics, foamed ceramics perform better in heat preservation, insulation, sound insulation, and other aspects due to the formation of a large number of bubbles in the firing process [3]. Moreover, foamed ceramics have the characteristics of lightweight and high-strength. It can be used not only as an excellent filtration or carrier material [4] but also for artificial bone repair or drug-loaded materials [5]. In construction engineering, foamed ceramics, as a new building material, have also been extensively used for thermal insulation of external walls [6], reducing construction loads while achieving good effects. Due to the excellent performance of foamed ceramics, the preparation process and properties have also received more and more attention from researchers [7]. Sandoval et al. [8] prepared porous ceramics by using mullite as raw material and the direct foaming method (1,600 °C, two hours). They conducted an analysis of the microstructure in order to comprehend the impact of bovine serum albumin as a foaming and binding agent. Alias et al. [9] utilized treated desulfurization sludge to generate porous ceramics through foaming. They analyzed the porosity and mechanical

Nomenclature

L – mass loss rate
m – mass, kg
m_1 – mass of the sample, kg
m_2 – mass of the sample after immersing in water and drying, kg
m_3 – mass of the dry sample, kg
m_4 – mass of the sample after cooling, kg
P – failure load, MN
S – area of pressure surface, m^2
V – volume, m^3
w_a – mass water absorption rate

Greek symbols

ρ – density, kg/m^3
σ – compressive strength, MPa

strength of the samples and found that the increased porosity significantly reduced the flexural strength of the samples after sintering at 1,200°C for three hours. Hu et al. [10] prepared porous ceramics using Al_2O_3-SiO_2, TiO_2 and silicon carbide (SiC) with low density and high porosity. Jia et al. [11] studied the properties of porous ceramics prepared by the freeze-gelation method. They discovered that the samples had relatively high mechanical strength and good adiabatic properties. According to the current practical requirements of foamed ceramics in building material applications, there is a higher demand for its various properties. Moreover, deeper research on energy-saving properties of building materials is also needed to face further development of green buildings. Currently, many materials have been applied to prepare foamed ceramics. This article conducted an in-depth study on the sintering preparation process of foamed ceramics using potassium feldspar tailings as raw material. The ceramic properties under different sintering preparation processes were analysed using the single-factor variable method. This paper contributes to further optimizing the preparation of foamed ceramics, improving their performance and achieving better application in the construction materials market.

2. Materials and methods

2.1. Experimental subjects

Produced by firing natural ores, ceramics are characterized by high hardness, low density, and corrosion resistance, and have extensive applications in daily life, art, culture and the construction industry [12]. With the progress of technology and evolving demands, there has been an increasing focus on researching various new types of ceramics [13].

Adding SiC foaming agent to cores can produce foamed (porous) ceramics, a novel functional material. Compared to regular ceramics, foamed ceramics exhibit significantly enhanced properties and further broaden their range of applications.

Foamed ceramics are light as abundant pores are generated during sintering. Moreover, their stable skeleton structure provides excellent strength, making them highly suitable for applications in the construction industry. The presence of pores allows for adequate heat and sound insulation, making them excellent thermal and acoustic insulating materials. Moreover, the high porosity enables reliable filtration and adsorption capabilities [14]. Additionally, foamed ceramics exhibit exceptional resistance to corrosion from acids and alkalis, providing a distinct advantage in scenarios like wastewater filtration and high-temperature dust removal [15].

The study of the preparation process for foamed ceramics holds significant practical value due to their excellent performance and diverse application scenarios. It enables better control over product performance and ensures alignment with market demand.

2.2. Experimental materials

The following materials are considered:

(1) Potassium feldspar tailings: They are produced by a mining company in Henan Province. Their chemical composition contains a large amount of SiO_2 and Al_2O_3, making them suitable for ceramic preparation. Their main components are shown in Table 1.

(2) Silicon carbide (SiC) foaming agent: It is prepared by Sinopharm Group Chemical Reagent Co., Ltd., and is chemically pure. In a high-temperature environment, SiC can react with oxygen [16] to produce bubbles, and the reaction equations of this process are:

$$SiC + O_2 \rightarrow SiO_2 + CO \uparrow,$$

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Table 1. Main chemical composition of potassium feldspar.

	Chemical composition, wt%
SiO_2	46.87
Al_2O_3	10.93
K_2O	9.46
Fe_2O_3	1.42
Na_2O	1.36

2.3. Experimental equipment

The main equipment used in the experiment is listed in Table 2.

2.4. Ceramic preparation process

The preparation process of ceramics is shown in Fig. 1. Based on Fig. 1, the preparation steps are outlined as follows:

(1) Potassium feldspar was ball-milled with the SiC foaming agent;
(2) A suitable amount of water was added to moisten the mixture, and then it was screened using a 100-mesh sieve;
(3) The mixture was pressed into a mould and pressed to form the desired shape;
(4) The moulded sample was dried in an oven for 12 hours;
(5) The ceramic embryo was sintered and foamed in a chamber furnace.

The sample was heated at a rate of 15°C/min to 300°C, kept at 300°C for 50 min, and heated again at a rate of 15°C/min until reaching the sintering temperature. After being held at this tem-

Table 2. Experimental equipment.

Equipment	Model number	Factory
Electronic balance	HZF-A200	Shanghai Shuangxu Electronics Co., Ltd.
Planetary ball mill	YXQM	Guangzhou Gurui Technology Co., Ltd.
Standard test sieve	100 mesh	Xinxiang Tongxin Machinery Co., Ltd.
Microcomputer control pressure tester	YAW-300	Xian County Rushi Technology Co., Ltd.
Vacuum drying oven	DZ-1BCIV	Tianjin Taisite Instrument Co., Ltd.
Chamber furnace	SX-G	Tianjin Zhonghuan Electric Furnace Co., Ltd.
Static water mechanics balance	JY5001	Hebei Ningke Instrument Co., Ltd.
Crucible	TC5645-30	Jinan Zhongbote Special Ceramics Co., Ltd.
Constant water bath pan	HH-2S	Shanghai Jingxin Industrial Development Co., Ltd.
TCi thermal conductivity analyzer	C-THERM	Shanghai RaocheLab Technology Development Co., Ltd.

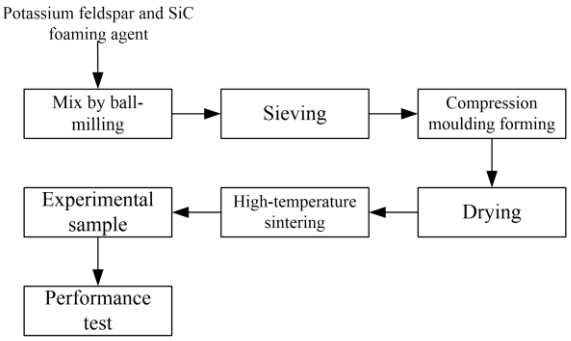

Fig. 1. Flow chart of the ceramic preparation process.

perature for a certain period of time, it was cooled down to room temperature to obtain the experimental sample.

This study investigated three parameters, namely SiC content, sintering temperature and holding time for sample preparation using a single-factor variable method [17]. The interpretation of these factors is shown below.

(1) SiC content: SiC content impacts the gas generation within the ceramic embryo, subsequently influencing the formation of porosity.

(2) Sintering temperature [18]: The sintering temperature affects the reaction of the foaming agent during the sintering process, thereby affecting the internal structure and properties of the sample.

(3) Heat preservation time: The sample may only foam adequately if the heat preservation time is short, resulting in small pores and high bulk density. Conversely, an excessively long heat preservation time can lead to continued growth of pores, resulting in thinner pore walls and reduced compressive strength. Therefore, selecting an appropriate heat preservation time is essential.

2.5. Performance testing

The following parameters were investigated:

(1) Bulk density: The sample was dried in an oven until no further change in mass; its mass was called m, and its bulk density was calculated:

$$\rho = \frac{m}{V}, \tag{1}$$

where V is volume.

(2) Water absorption rate: The sample was weighed using a static water mechanics balance, and its mass of the sample was denoted as m_1. The sample was immersed in water until saturated and then dried, and its mass was denoted as m_2. The water absorption rate was calculated:

$$w_a = \frac{m_2 - m_1}{m_1} \times 100\%. \tag{2}$$

(3) Compressive strength: Using a microcomputer-controlled pressure testing machine, the sample was cut into standard blocks. The area of pressure surface (S) is calculated. A pressure test was conducted. If the failure load was P, the compressive strength was

$$\sigma = \frac{P}{S}. \tag{3}$$

(4) Thermal conductivity coefficient: it was determined using a TCi thermal conductivity meter.

(5) Acid resistance: The dry sample with a mass of m_5 was added with 20% sulfuric acid, boiled for one hour, washed and burned in a crucible at 700 °C until constant weight. After cooling at room temperature, its mass was weighed and denoted as m_4. The mass loss rate is calculated:

$$L = \frac{m_3 - m_4}{m_3} \times 100\%. \tag{4}$$

3. Results and analysis

The properties of the sample mixed with different SiC contents are presented in Table 3.

It can be observed that an increase in SiC content resulted in a higher number of internal pores within the samples, leading to a decrease in bulk density and a significant increase in water absorption rate. Specifically, the water absorption rate of the 1.5 wt% SiC sample showed a 22.92% increase compared to the 0.5 wt% SiC sample. The increased SiC content contributed to

Table 3. Effect of SiC content on properties.

	0.5 wt%	1.0 wt%	1.5 wt%
Bulk density, g/cm³	0.51	0.43	0.35
Water absorption rate, %	7.11	18.02	30.03
Compressive strength, MPa	7.23	5.35	2.12
Thermal conductivity coefficient, W/(m·K)	0.07	0.06	0.05
Acid-resistance mass loss rate, %	0.92	1.12	3.16

a rise in the number of internal pores, which subsequently reduced the compressive strength and thermal conductivity of the samples. The compressive strength decreased from 7.23 MPa to 2.12 MPa, while the thermal conductivity decreased from 0.07 W/(m·K) to 0.05 W/(m·K). With the increase of pores, the contact area between the sample and acid increased, leading to decreased acid resistance. It can be concluded that the changes in water absorption rate, compressive strength, and mass loss were significant when increasing SiC content from 1.0 wt% to 1.5 wt%. Therefore, opting for a SiC content of 1.0 wt% was more suitable.

The properties of the samples mixed with 1.0 wt% SiC under a heat preservation time of 30 minutes at different sintering temperatures are presented in Table 4.

Table 4. Effect of sintering temperature on properties.

	1,200°C	1,250°C	1,300°C
Bulk density, g/cm³	0.68	0.54	0.42
Water absorption rate, %	3.84	13.45	48.94
Compressive strength, MPa	8.07	4.75	0.98
Thermal conductivity coefficient, W(m·K)	0.08	0.06	0.05
Acid-resistant mass loss rate, %	0.87	1.21	4.37

It can be observed that as the sintering temperature increased, the reaction of SiC became more intense, resulting in the formation of a more significant number of expansion pores in the embryo. However, as the sintering temperature continued to rise, there was a possibility of pore rupture and penetration, leading to an increase in the water absorption rate. Specifically, the water absorption rate increased by 9.61% from 1,200°C to 1,250°C and by 35.49% from 1,250°C to 1,300°C. These findings indicated that the sample contained more pores at higher sintering temperatures. Additionally, in the presence of multiple pores, the sample became more susceptible to cracking under pressure, decreasing compressive strength. The sample's compressive strength at 1,300°C was only 20.63% of that at 1,250°C. Similarly, the mass loss rate exhibited a significant increase, from 1.21% at 1,250°C to 4.37% at 1,300°C. While the sample demonstrated the lowest thermal conductivity coefficient at 1,300°C, their compressive strength and water absorption rate were poor. Therefore, a sintering temperature of 1,250°C was preferred.

Table 5 shows the properties of the samples under different heat preservation durations when the SiC content and sintering temperature were fixed at 1.0 wt% and 1,250°C.

According to Table 5, it can be observed that as the heat preservation time was extended, the bulk density of the sample decreased, and the water absorption rate increased. This was because a longer heat preservation time allowed for more complete foaming of the sample, resulting in a decrease in density, an increase in water absorption rate, and a decrease in compressive strength. For instance, at a heat preservation time of 60 minutes, the compressive strength was only 1.03 MPa, indicating a significant decrease of 78.32% compared to the 30-minute heat preservation time. The thermal conductivity coefficient increased when extending the heat preservation time from 50 minutes to 60 minutes, reaching 0.06 W/(m·K). This could be attributed to the appearance of more penetrating holes within the sample, enhancing gas convection and subsequently increasing the thermal conductivity coefficient. In addition, the mass loss rate in the acid resistance test also increased due to the increase of pores, rising from 1.21% at 30 minutes to 3.547% at 60 minutes. Thus, a heat preservation time of 30 minutes was more suitable.

Table 5. Effect of heat preservation time on performance.

	30 min	40 min	50 min
Bulk density, g/cm³	0.54	0.47	0.42
Water absorption rate, %	13.45	15.56	25.87
Compressive strength, MPa	4.75	2.02	1.46
Thermal conductivity coefficient, W/(m·K)	0.06	0.06	0.05
Acid-resistant mass loss rate, %	1.21	1.46	2.12

4. Conclusions

This study investigated the foamed ceramics prepared by sintering with SiC foaming agent to achieve better application of ceramic materials in practical construction projects. The results showed that increasing the amount of SiC led to more pores, higher water absorption rate, and lower compressive strength. An appropriate SiC content was found to be 1.0 wt%. Improving the sintering temperature resulted in a more complete reaction of the foaming agent, more pores, lower compressive strength and higher mass loss. An appropriate sintering temperature was determined to be 1,250°C. Extending the heat preservation time generated more internal pores in the samples, and the optimal time was 30 minutes.

A sample sintered using 1.0 wt% SiC at 1,250°C and insulated for 30 minutes exhibits good performance with a compressive strength of 4.75 MPa and a thermal conductivity coefficient of 0.06 W/(m·K), making it suitable for practical applications. However, this study also has some limitations. For instance, it only investigated the influence of sintering preparation processes on performance and lacked analysis of sample phase transitions and pore structures. In future work, a comparison will be made among more preparation methods, and the performance of the samples will be further analyzed.

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