

Research on preparation and properties of porous ceramsites sintered with high-ash coal slime

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Abstract: In order to realize the resource and harmless utilization of high-ash coal slime in coal preparation plants, porous ceramsites were prepared by the high-temperature sintering method with coal slime as raw material. The influences of sintering temperature, sintering time and ash content on the properties of porous ceramsites were studied by experiments, and the phase composition, micro-morphology and pore structure characteristics of ceramsites were analyzed by XRD, SEM and BET. The experimental results showed that with the increase of sintering temperature and sintering time, the amount of molten liquid in ceramsite green bodies increased, the densification degree of ceramsites increased gradually, the bulk density and the apparent density increased gradually, and the water absorption and the apparent porosity decreased gradually. However, with the increase of coal slime ash content, the quantity of pores within ceramsites increased first and then decreased. When the coal slime ash content was 55%, the bulk density of porous ceramsite sample was 0.549g/cm³, the water absorption rate was 64.63%, the specific surface area was 19.40m²/g, the crushing rate and wear rate were 0.14%, with rough surface, porous structure and excellent water absorption performance, which met the optimum performance requirements of porous ceramsites. At the same time, this research also provides a new idea and method for the reuse of high-ash coal slime resource, a by-product of coal washing and dressing.

Keywords: high-ash coal slime, porous ceramsites, sintering temperature, ash content

1. Introduction

Coal slime mainly refers to solid matter composed of fine coal particles and clay minerals, which is a by-product of coal washing and dressing (Wang, 2021). It has the disadvantages of high ash content, high viscosity, fine particles and low calorific value, so it cannot be directly used as fuel (Wang, 2003; Li et al., 2011; Xia et al., 2019). In recent years, due to the increase of coal mining mechanization, the deterioration of raw coal quality and the clean and efficient classified utilization of coal, the output of coal slime has been increasing, and its use has been limited (Min et al., 2021; Yang et al., 2023). According to statistics, China's coal output in 2022 was 4.56 billion tons, and the washing proportion was 69.7%, of which about 90% was wet separation, and the amount of high-ash coal slime generated by wet separation was relatively large (Chen et al., 2014; Zhu, 2023). Meanwhile, the coal slime dewatering has disadvantages such as low efficiency, high treatment cost and low calorific value, and the coal preparation plants cannot directly sell coal slime and has no good treatment measures; as a result, they can only choose open storage (Wu et al., 2017; Lv et al., 2021). Therefore, the treatment, consumption and comprehensive utilization of high-ash coal slime has become an urgent problem to be solved.

The resource utilization of coal slime depends on its characteristics, and its main uses includes fuel utilization, building admixture, modified chemical materials, filter materials for waste water treatment, other auxiliary chemical raw materials, etc. (Yang et al., 2005; Zhang et al., 2010; Ma et al., 2019; Chen et al., 2020). Among them, Tsai et al. (Tsai et al., 2006) prepared ceramic particles using sludge incineration ash sintering and experimentally studied the effect of SiO₂-Al₂O₃ flux ratio in raw

materials on the expansion performance of ceramic particles. Cheeseman et al. (Cheeseman et al., 2004) used sludge incineration ash to prepare ceramsite, analyzed the phase change of ceramsite during sintering, studied the properties, microstructure and leaching characteristics of heavy metals, and found that the preparation of ceramsite at high temperature had a good control effect on the leaching of heavy metals. Laursen et al. (Laursen et al., 2006) used industrial sludge and marine clay as the main raw materials, and the ceramic particles prepared by sintering had good swelling performance. In addition, it was found that the addition of sludge would cause uneven pore size distribution of the ceramics, thus reducing the strength of the ceramics. Chindaprasirt et al. (Chindaprasirt et al., 2009) used rice husk ash as the main raw material to sinter and prepare low-density ceramic particles. The research results showed that rice husk ash ceramic particles had good scalability, solubility, and application value. Malaikien et al. (Malaikien et al., 2011) successfully prepared ceramic particles using burned and unburned mullite waste as raw materials. The study found that when the composition of mullite in the raw material increased, the number of predicted freeze-resistant mixtures was reduced and water absorption was increased. Lu An'min (Lu, 2008) made the fine tailings of Tianzhuang Coal Preparation Plant into coal water slurry with 70% concentration, extremely low viscosity and stable performance, which not only solved the technical problems of preparation and combustion of coal slime-based coal water slurry, but also provided a new technical way for the development and utilization of coal slime. Through pretreatment such as calcined activation and iron removal by acid leaching, Wu Tao et al. (Wu et al., 2015) used coal slime as raw material to prepare Molecular Sieve 13X featuring with high purity, uniform particle size, complete crystal form and size of about 5 μ m based on the hydrothermal synthesis method. Yang Ren et al. (Yang et al., 2011) used coal slime as raw material to prepare ceramsite filter material through sintering, researched the influences of preheating temperature, preheating time, roasting temperature and roasting time on the properties of ceramsites, and discussed the expansion mechanism of coal slime ceramsites. The ceramsite filter material sintered under the optimum technological conditions had a specific surface area of 35.337 m²/g, a bulk density of 0.586 g/cm³, a water absorption of 48.89%, a porosity of 56%, as well as a solubility in hydrochloric acid of 0.54%. Xu Xiaohong et al. (Xu et al., 2014) used coal slime as the main raw material and the non-pressure sintering process to prepare thermal storage ceramics for solar heat utilization, and discussed the influences of sintering temperature on water absorption, porosity, bulk density, folding strength and thermal shock resistance of samples. However, the existing researches on coal slime utilization mainly focuses on medium-ash and low-ash coal slime, and there is little research on high-ash coal slime; there are few reports on the research on preparation of porous ceramsites with high-ash coal slime and the research on its properties.

The main components of high-ash coal slime are a small amount of combustible carbon and a large amount of non-combustible clay minerals, which not only meet the chemical composition requirements of raw materials for ceramsite preparation, but also play the role of pore-forming agent and binder in sintering process. Therefore, based on the special chemical component characteristics of high-ash coal slime, ceramsites with certain strength and porosity were sintered through melting in high temperature and recrystallization, and the influences of sintering temperature, sintering time and ash content of coal slime on the properties of ceramsites were discussed in the research; porous ceramsites featuring with certain properties were prepared by optimizing and regulating key process parameters.

2. Materials and methods

2.1. Raw material

The coal slime used in the test was filter-pressed coal slime made from tailings of a coal preparation plant in Huainan mining area, Anhui Province. According to GB/212-2008, the particle size composition and industrial analysis of coal slime raw material were carried out. The analysis showed that the ash content of coal slime was 55.13%, and the moisture content was 15~20%. According to the definition of coal slime classification by Si Yucheng et al. (Si et al., 2017), the coal slime belonged to high-ash content coal slime, because its ash content was greater than 55%. X-ray fluorescence spectrometer (XRF) was used to detect the chemical composition of coal slime, as shown in Table 1.

The main components of high-ash coal slime were SiO_2 , Al_2O_3 , Fe_2O_3 , CaO and C . Among them, the content of C was 46.4%, which could not only provide energy to achieve self-sintering, but also release gas during the sintering process of ceramsites, resulting in a large number of pore structures of ceramsites (Liao et al., 2021). Therefore, high-ash coal slime met the raw material requirements for preparing porous ceramsites, and there was no need to adjust the components by adding other raw materials.

Table 1. Main chemical components of coal slime raw material

Components	SiO_2	Al_2O_3	Fe_2O_3	CaO	K_2O	TiO_2	Na_2O	MgO	CO_2
Mass fraction/%	52.4	35	3.5	3.2	1.5	1.1	0.7	0.7	46.4

The particle size distribution of high-ash coal slime raw materials is shown in Fig. 1. After grinding, the particle size was mainly below $50\mu\text{m}$, of which the particle size less than $10\mu\text{m}$ accounted for about 65%; from the perspective of particle size, the coal slime sample belonged to fine particle. Research has shown that (Wei et al., 2011; Wang et al., 2013), the smaller the particle size of raw materials used to prepare ceramsites is, the more favorable it is for the preparation of ceramsites; the main reason is that the finer the particle size of raw materials is, the larger the surface energy is, the larger the action area that can be provided during chemical reaction is, and thus the stronger the reaction capacity is.

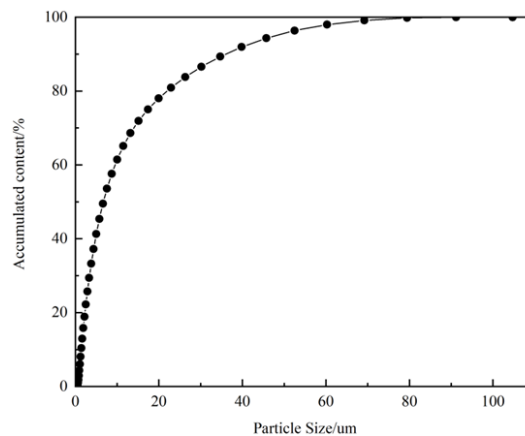


Fig. 1. Particle size distribution curve of coal slime raw material

2.2. Ceramsite preparation method

The high-ash coal slime raw materials were mixed and stirred by a mixer (Model NJ-160A, manufactured by Wuxi Xiyi Building Materials Instrument Factory) for 5 min, during which water with a mass ratio of 30% of the raw materials was evenly added by using a measuring cylinder to make the raw materials stirred fully and evenly, and then the mixture was granulated into pellets by a granulator (Model ZL18, manufactured by Mingda Machinery Factory), and the particle size of raw material pellets was controlled within the range of 8~12mm. Then raw material pellets aged for a few hours in a natural state were placed in an air blast drying oven (Model DHG-9960A, manufactured by Shanghai Sanfa Scientific Instrument Co., Ltd.) to dry them completely at 105°C ; after that, they were put into a chamber electric furnace and heated to the set temperature (950°C , $1,000^\circ\text{C}$, $1,050^\circ\text{C}$, $1,100^\circ\text{C}$, $1,150^\circ\text{C}$) at a heating rate of $5^\circ\text{C}/\text{min}$; and then kept them warm (for 30min, 45min, 60min, 75min, 90min), and finally cooled them naturally with the decreasing furnace temperature to obtain high-ash coal slime porous ceramsites.

2.3. Performance testing and characterization

Ceramsites are synthetic lightweight aggregates. Their physical and mechanical properties should be tested according to the national standard Lightweight Aggregates and Its Test Methods –Part 2: Test

Methods for Lightweight Aggregates, which tests bulk density, apparent density, water absorption, apparent porosity, and the sum of crushing rate and wear rate accordingly. The X-ray diffractometer (XRD), the scanning electron microscope (SEM), the specific surface area and porosity analyzer (BET) and the thermogravimetry and differential scanning calorimetry (TG-DSC) were used to research the phase composition, microstructure, pore diameter, pore volume, specific surface area and thermal analysis characteristics of the sintered samples.

Firstly, the weight of the empty standard funnel was measured, and then the ceramic particles were evenly put into the standard funnel and the total weight of the ceramic particles and the standard funnel was measured. The weight of the ceramic particles was calculated and divided by the volume of the standard funnel to determine the stacking density. After weighing 300-500g of dry ceramic particles, the ceramic particles were soaked in water for 1 hour, removed and filtered, and then placed in a measuring cylinder to measure its volume. The apparent density was determined by dividing the weight of the dry ceramic particles by the volume after soaking in water. The water absorption rate was determined by subtracting the weight of the dry ceramic particles from the weight of the ceramic particles immersed in water for 24 hours, and then dividing the difference between the two by the weight of the dry ceramic particles. The apparent porosity of ceramsite refers to the proportion of gas pores in the ceramsite sample, and the proportion of the volume of water reduced in the ceramsite sample after being placed in the measuring cylinder to the volume of ceramsite is called the apparent porosity. The sum of crushing rate and wear rate was the percentage of broken and worn ceramsites in the total number of ceramsites.

The ceramsite samples were crushed and ground into powder, and the phase composition of ceramsite samples was analyzed by XRD (Type XRD-6000, manufactured by Shimadzu Corporation). The micro-morphology of sintered ceramsite was tested and studied by SEM (Type S-3000N, manufactured by Hitachi Limited). Firstly, the ceramsite sphere was broken to form a cross section, which was fixed to the sample table with conductive adhesive and subjected to vacuum gold spraying treatment. After gold spraying treatment, the sample was taken to the SEM sample room for testing and analysis. In N_2 atmosphere, the initial temperature was room temperature, the end temperature was 1,200 °C, and the heating rate was 10 °C/min. The thermal analysis characteristics of sintered ceramsite were tested by TG-DSC (Type Q600, manufactured by TA Instruments).

3. Results and discussion

3.1. Analysis of raw material properties

3.1.1. Mineral composition

The samples were tested by XRD, and the results were shown in Fig. 2. The main crystalline phases of coal slime were kaolinite, quartz and other clay minerals. Among them, kaolinite is a kind of hydrated

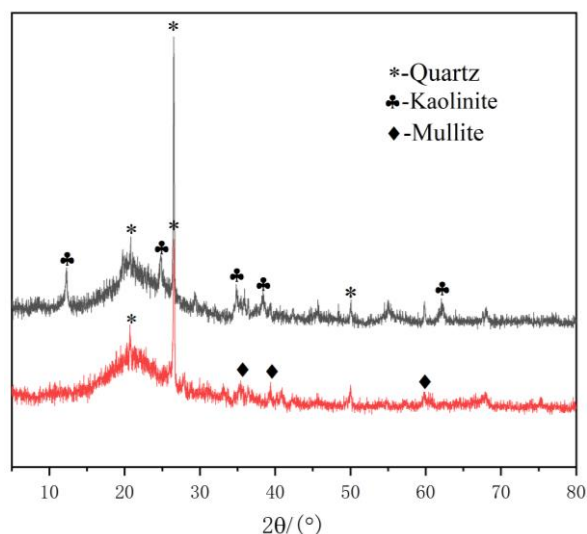


Fig. 2. XRD pattern of coal slime

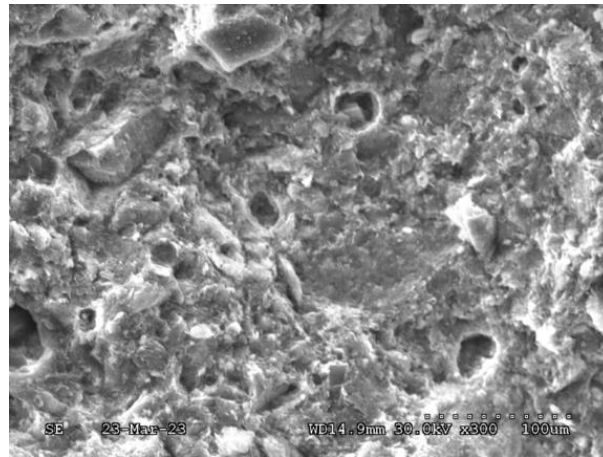


Fig. 3. SEM image of the cross-section of ceramsite raw material pellets

kaolinite clays. As shown in Fig. 2, the upper curve represents "before sintering", and the lower curve represents "after sintering". Compared with the XRD pattern of coal slime raw material before sintering, the XRD pattern after sintering showed that the diffraction peak of kaolinite disappeared, the intensity of the diffraction peak of quartz was weakened, the mullite crystal phase was generated, and the overall peak shape was more chaotic, indicating that the amorphous phase in the sample increased (Malaikien et al., 2011), i.e., crystal phase transition occurred at high temperature.

3.1.2. Analysis of appearance

SEM was used to characterize the microstructure of cross section of ceramsite raw material pellets. As shown in Fig. 3, the cross section of ceramsite raw material pellets had a small number of pores, which were distributed independently, without forming a pore network, and had a relatively obvious lamella structure with dense structure and compact arrangement.

3.1.3. Analysis of thermal stability characteristics

The change of thermal stability characteristics of high-ash slime raw materials was researched by TG-DSC. In the process of heating to 1,200°C at a heating rate of 10°C/min in air atmosphere, the test results of high-ash coal slime were reflected by the TG-DSC curve in Fig. 4. There were three distinct exothermic peaks on the DSC curve: The first one was between 380°C and 580°C, which was wide in peak shape and significant in weight loss, mainly due to the exothermic reaction caused by the oxidation of high ash slime raw material middling coal, volatile matter in coal and combustion decomposition of fixed carbon (Du et al., 2006). The second one was between 580°C and 920°C, which was relatively flat in peak shape, the dehydroxylation of kaolinite was completed gradually, and the crystal phase transition occurred from crystal to amorphous metakaolinite. The third one was between 920°C and 1,080°C, during which kaolinite was gradually recrystallized and began to form new phase mullite, thus it belonged to the high-temperature calcination stage (Yang et al., 2006; Yan, 2010).

3.2. Properties and characterization of ceramsite samples

3.2.1. The influence rule of sintering temperature on the properties of ceramsites

Under the conditions of a sintering time of 60 min, a preheating temperature of 500°C, a preheating time of 30 min and a heating rate of 5°C/min, the influences of sintering temperature on bulk density, apparent density, water absorption and apparent porosity of ceramsites were researched, and the test results were shown in Fig. 5 and Fig. 6. When the sintering temperature was too low, the melting phenomenon of the ceramsites was not obvious, most of the raw materials still maintained their original appearance, and loose ceramsites resulted in the lowest bulk density and apparent density as well as relatively high water absorption and apparent porosity (Yang et al., 2018). When the sintering temperature was too high, a large amount of molten liquid phase was generated, thus filling and

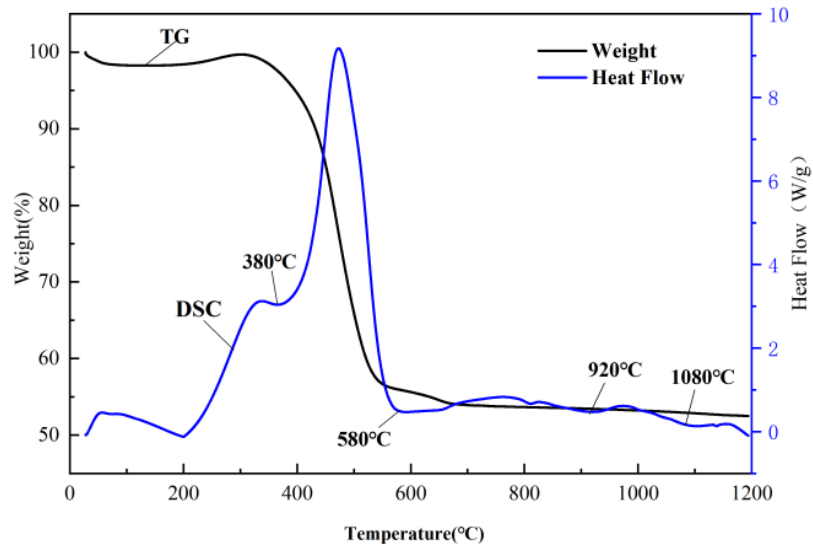


Fig. 4. TG-DSC diagram of raw materials

destroying the pores generated inside the ceramsites; and the spheres collapsed, the densification degree of ceramsites increased, the bulk density and the apparent density increased, and the water absorption and the apparent porosity decreased significantly (He et al., 2019). However, when the sintering temperature was about 1,000°C, the liquid phase viscosity generated in ceramsites was suitable, and the surface tension generated by the liquid phase inhibited the gas from escaping, thus forming a sound pore structure on the surface and inside of ceramsites; at this time, ceramsites had low bulk density and apparent density as well as the highest water absorption and apparent porosity (Zhou et al., 2021; Wang et al., 2022). Therefore, from the aspects of saving energy and meeting the performance requirements, 1,000°C was selected as the optimum sintering temperature in this research.

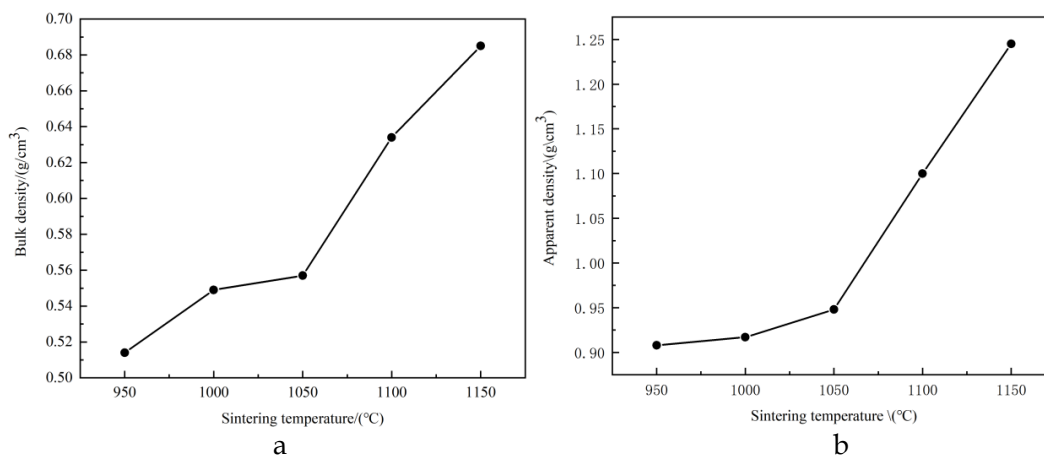


Fig. 5. Influences of different sintering temperatures on the bulk density and apparent density of ceramsites, a: bulk density; b: apparent density

3.2.2. Influences of Sintering Time on the Properties of Ceramsites

Under the conditions of a sintering temperature of 1,000°C, a preheating temperature of 500°C, a preheating time of 30 min and a heating rate of 5°C/min, the influences of holding time on bulk density, apparent density, water absorption and apparent porosity of ceramsites were researched, and the test results were shown in Fig. 7. When the sintering time was short, the insufficient reaction of raw materials was a "underfiring" phenomenon, resulting in less molten liquid phase; no dense enamel layer was formed on the surface of ceramsites, and there were many pores, so the bulk density and the apparent density were low, and the water absorption and the apparent porosity were high

(Liu et al., 2010). When the sintering time was too long, ceramsites showed an "over-burning" phenomenon, and too much molten liquid filled the pores, resulting in increased densification degree; so ceramsites had increased bulk density and apparent density as well as decreased water absorption and apparent porosity (Lei et al., 2013). However, when the sintering time was about 60 min, ceramsites generated appropriate liquid phase amount, and the expansion effect was good, thus forming well-developed pore structure; at this time, the bulk density and the apparent density were relatively low, and the water absorption and the apparent porosity were relatively high (Mi et al., 2021). Therefore, under the premise of meeting the optimum performance requirements and low energy consumption, 60 min was selected as the optimum sintering time for this research.

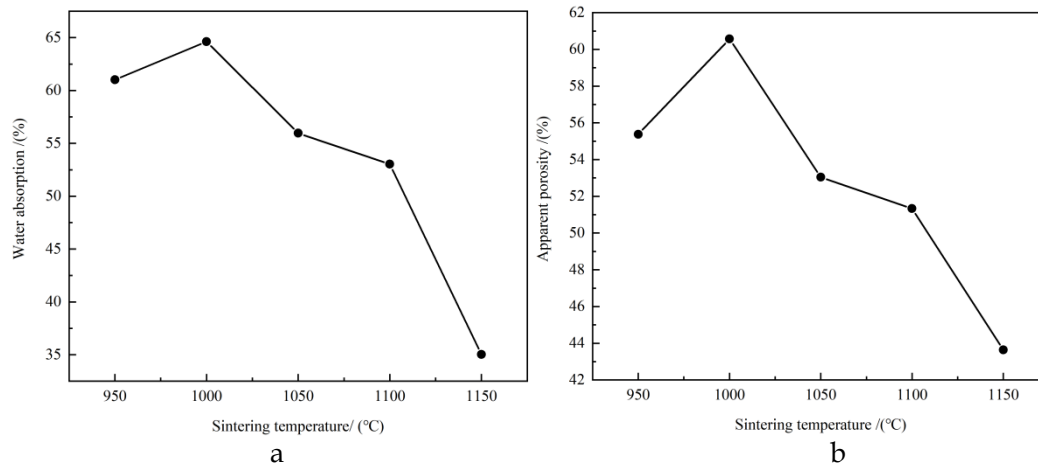


Fig. 6. Influences of different sintering temperatures on water absorption and apparent porosity of ceramsites
a: water absorption; b: apparent porosity

3.2.3. Influences of Sintering time on the properties of ceramsites

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3.2.4. Influences of Ash Content on the Properties of Ceramsites

The coal slime with different ash contents was prepared into raw materials of ceramsites, and the influences of ash content on the properties of ceramsite were analyzed. The ash contents of coal slime raw materials set in the test were 45%, 50%, 55%, 60% and 65%, and the optimum sintering system was determined as follows: a sintering temperature of 1,000°C, a holding time of 60 min, a preheating temperature of 500°C, a preheating time of 30 min, and a heating rate of 5°C/min; and the test of coal slime ash content on the performance of ceramsites was shown in Fig. 8. When the ash content was 45%, the carbon content in raw materials was high, and a large volume of gas was released after the

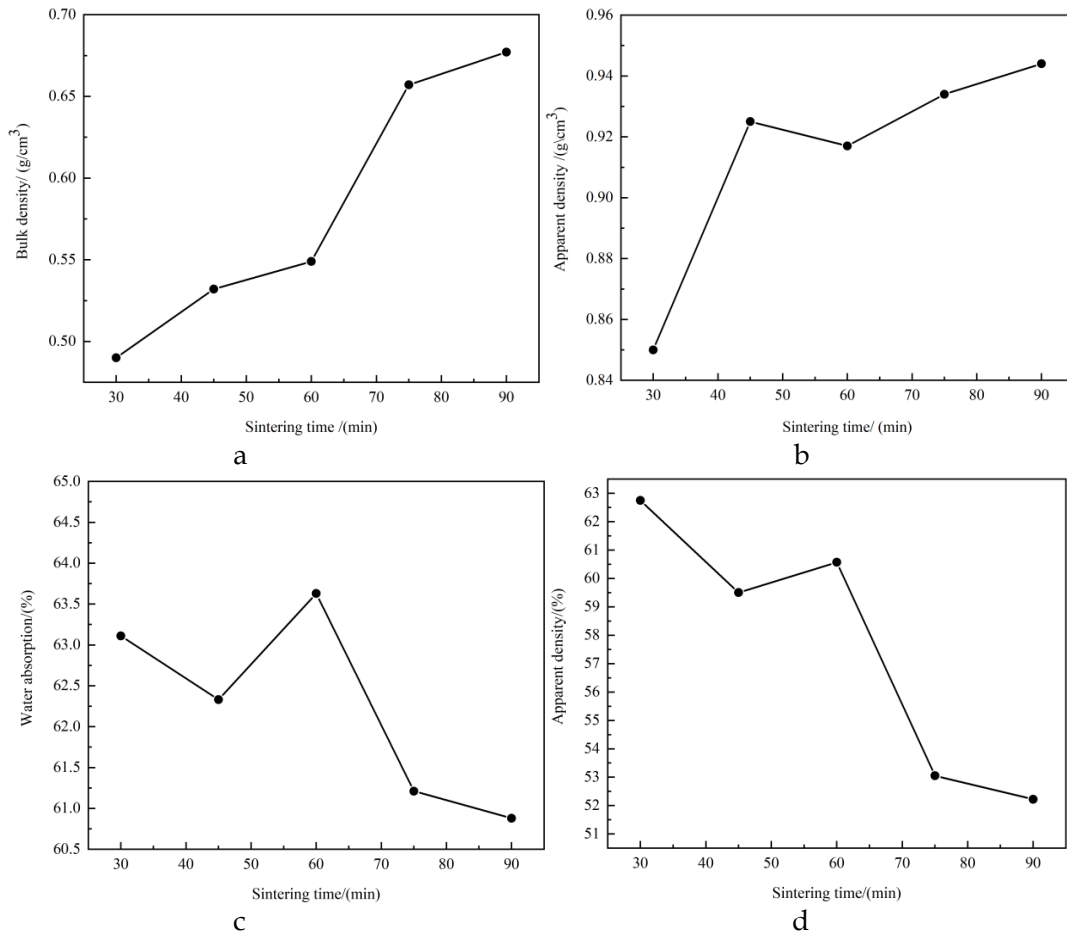


Fig. 7. Influences of different sintering time on the properties of ceramsites, a: bulk density; b: apparent density; c: water absorption; d: apparent porosity

carbon was combusted and lost, thus leaving a large number of pores on the surface and inside of ceramsites; at this time, the liquid phase amount was relatively less, and loose ceramsites could not form a skeleton structure with a certain strength, so the bulk density and the apparent density were minimum, and the water absorption, the apparent porosity, the crushing rate and wear rate were maximum. With the increase of ash content, the carbon content in raw materials decreased, and the volume of gas generated after sintering decreased; excessive liquid phase filled the pores in ceramsites, and the densification degree of ceramsites increased; at this time, the bulk density and the apparent density reached the maximum, and the water absorption, the apparent porosity, the crushing rate and wear rate decreased significantly (Qu et al., 2016). However, when the ash content was about 55%, the contents of combustible and non-combustible materials in the raw material were nearly equal, and appropriate liquid phase content and viscosity were formed during the sintering process. At the same time, ceramic particles generated and slowly released gas in the process of sintering. Most of the gas escaped from the surface of the ceramic particles to form pores, while a small portion of the gas was suppressed by the surface tension generated by the liquid phase, forming a connected pore structure inside the ceramic particles, so that the bulk density and apparent density of ceramsite were moderate. During the sintering process, the pores and voids formed inside the ceramic particles made the water absorption and apparent porosity higher. Due to the presence of liquid phase, it could also promote the wetting and fusion of particles, thus promoting material transfer and product densification in the sintering process, resulting in the lowest crushing and wear rates of ceramic particles.

3.2.5. Influences of ash content on the internal pore structure of ceramsites

According to the influence rule of ash content on the performance of ceramsites, it can be seen from that the ash content of coal slime had a close relationship with the internal gas generation and liquid

phase generation of ceramsite at high temperature. Based on the XRD, SEM and BET characterization tests of ceramsites with different ash contents, the characterization test results were shown in Figs. 9, 10 and Table 2 respectively. Combined with the XRD patterns of ceramsite samples with different ash contents in Fig. 9, it can be seen that with the increase of ash content, the crystal phase in ceramsites did not change, but the diffraction peak was gradually enhanced, indicating that the degree of crystallization became better. It can also be seen from Fig. 10 and Table 2 that with the increase of coal slime ash content, the pore structure of ceramsites gradually decreased, the specific surface area and average pore diameter gradually decreased, and even closed pores were formed, so the structure became denser and the crystalline phase gradually increased.

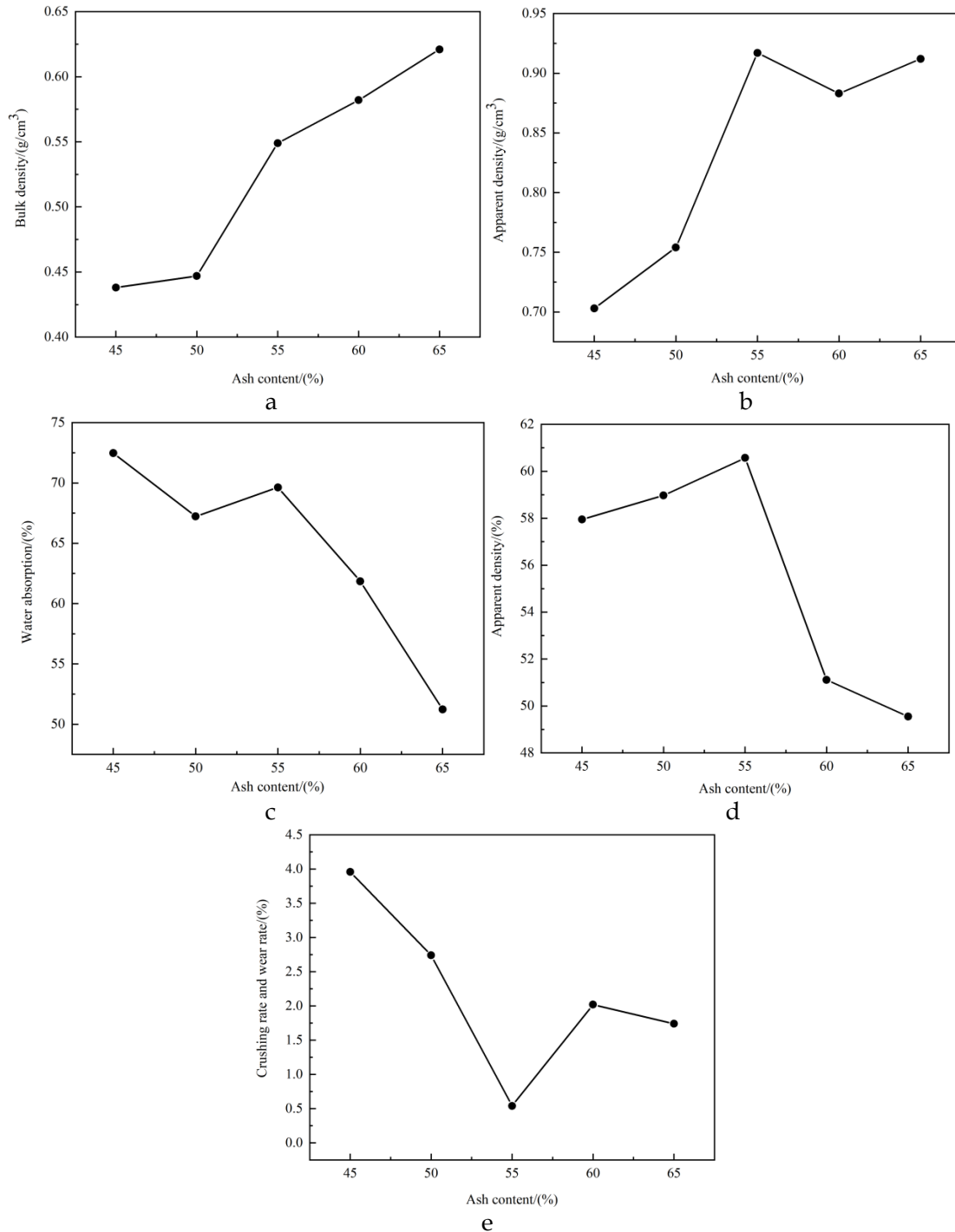


Fig. 8. Influences of different ash contents on the properties of ceramsites, a: bulk density; b: apparent density; c: water absorption; d: apparent porosity; e: crushing rate and wear rate

As shown in Figs. 10a and 10b, when the ash content was low, the pore size of ceramsite samples was large and irregular, and these pores were mainly left by the carbon burnt and lost in coal slime; at this time, the liquid phase generated was relatively less, the crystallization around the pores was not obvious, and the surface and interior of ceramsites were loose. As shown in Figs. 10c, 10d, and 10e, with the increase of coal slime ash content, the pore size inside the ceramic particles shrunk, the number of pores decreased obviously, and the ceramic particles gradually became denser. This was mainly because high ash ceramic particles generated an increase in liquid phase during high-temperature sintering, and excessive liquid phase filled the pores, resulting in an increase in sample densification and obvious crystallization (Cai et al., 2005). As shown in Fig. 10c, when the coal slime ash content was 55%, the pore structure of ceramsite sample was obvious, the quantity of small pores increased with a uniform distribution, and the crystalline phase around the pores was obvious, so ceramsite sample had a high porosity (Gennaro et al., 2005).

As can be seen from the BET data in Table 2, the average pore diameter, pore volume and specific surface area of the samples gradually decreased with the increase of coal slime ash content. Combined with the influence rule of ash content on the properties and internal pores of ceramsite samples, the optimum ash content for preparing porous ceramsites can be determined to be 55%.

Table 2. BET parameters of ceramsite samples with different ash contents

Ash content parameter	45%	50%	55%	60%	65%
BET specific surface area / (m ² /g)	20.26	17.32	19.40	17.03	16.08
Langmuir specific surface area / (m ² /g)	19.21	18.43	17.87	16.44	15.95
Average pore diameter / (nm)	32.86	27.68	20.65	15.72	14.65
Average mesoporous pore diameter/(nm)	17.69	24.43	17.03	16.28	15.00
Total pore volume by single point method / (10 ⁻³ cm ³ /g)	0.126	0.112	0.103	0.092	0.073
Micropore volume by t-plot method / (10 ⁻³ cm ³ /g)	1.358	1.142	1.171	0.753	0.646

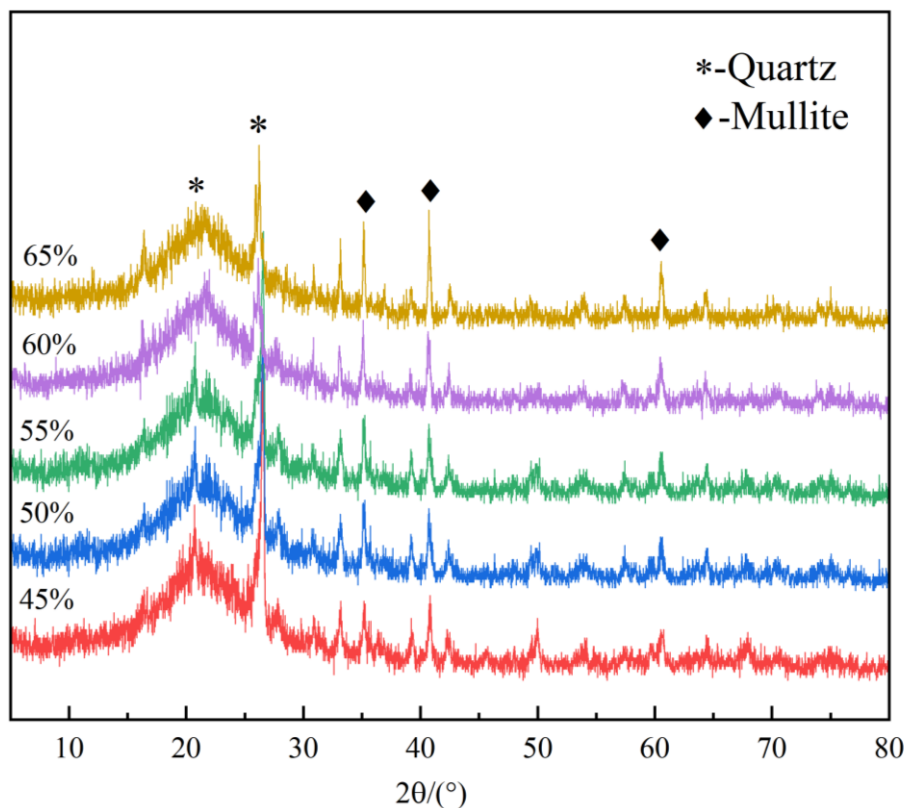


Fig. 9. XRD patterns of ceramsite samples with different ash contents

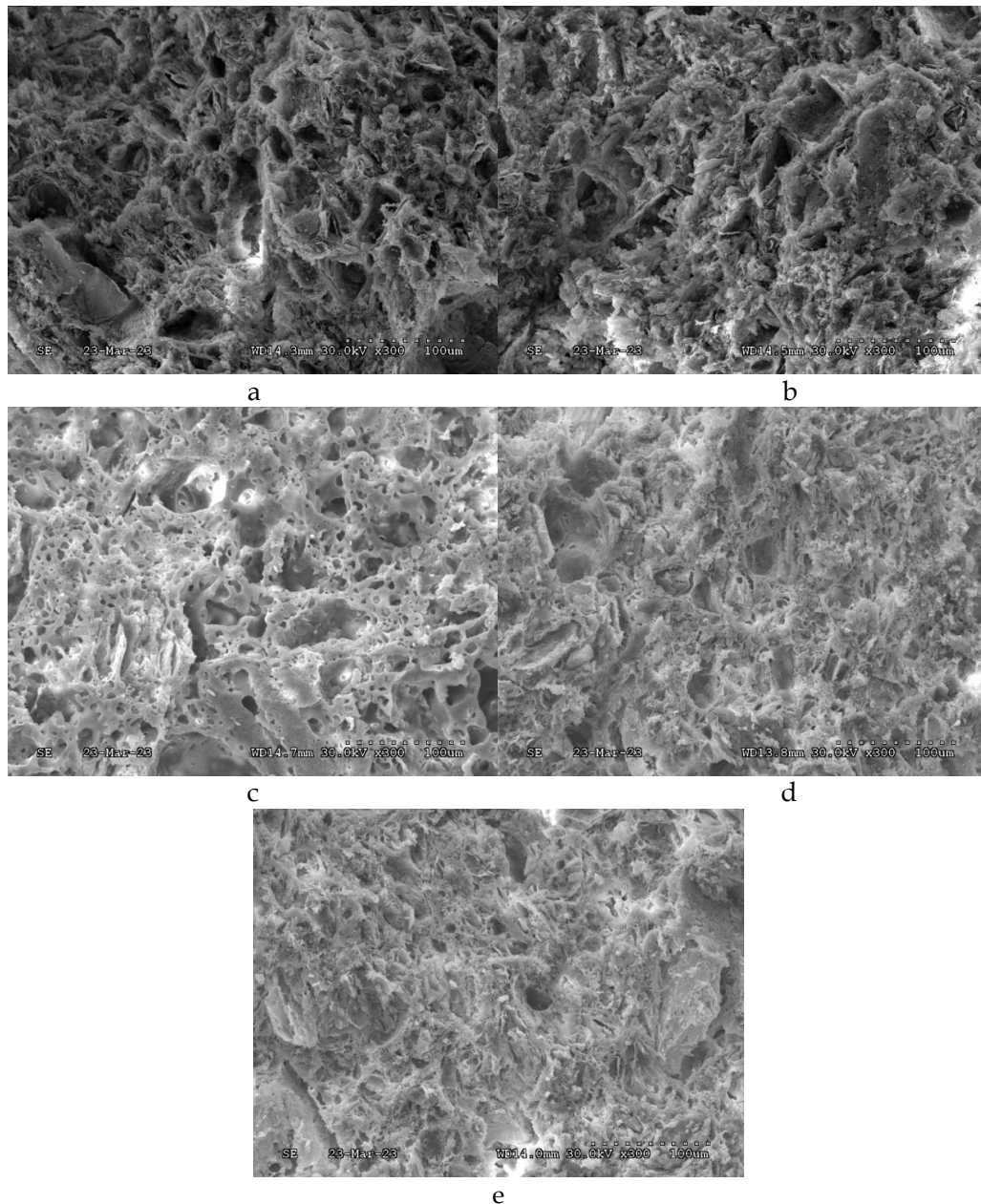


Fig. 10. SEM images of coal slime samples with different ash contents, a: 45% ash; b: 50% ash; c: 55% ash; d: 60% ash; e: 65% ash

4. Conclusions

- (1) The main components of high-ash coal slime were kaolinite, quartz and carbon. Porous ceramsites were prepared from high-ash coal slime. The optimum sintering temperature was 1,000°C, the optimum sintering time was 60 min, and the optimum ash content was 55%. The prepared porous ceramsites had a bulk density of 0.549 g/cm³, an apparent density of 0.917 g/cm³, a water absorption rate of 64.63%, an apparent density of 60.57%, the crushing rate and wear rate of 0.14%, and a specific surface area of 19.40 m²/g.
- (2) Through XRD, SEM and other characterization and test analysis, it is concluded that porous ceramsites were mainly composed of quartz, mullite and other amorphous oxides. The internal pores of ceramsites were well-developed, and a large number of pores were dominant mesopores, and some pores were connected to form through holes. Ceramsites had rough surface and dense shell, and a large number of pores were distributed on the surface and connected with the inside to form through holes, so ceramsites had excellent water absorption performance and high porosity.

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