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Fabrication of Porous Ceramics using Tailings Mud from Huzhou City as Raw Material

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Abstract. The comprehensive utilization of tailings mud has become a common concern of the society. The properties of tailings mud produced from Huzhou city were analyzed in this work. Moreover, porous ceramics with high strength were prepared by using the tailings mud as main raw material and adding a small amount of foaming agent. The results show that the foaming agent contents have a certain effect on the porosity and compressive strength of the porous ceramic samples. These results provided a certain theoretical basis for the comprehensive utilization of tailings mud and solve the pollution of tailings mud to the environment.

1. Introduction

Porous ceramic, which combines high specific surface area of porous materials with stability of ceramic materials, has become an important environmental material [1, 2]. The ability to control its pore size, porosity and bulk density has been improved with the development of technology [3, 4]. Compared with the traditional ceramic particle filter, there are many advantages for porous ceramic, such as energy saving, low cost, and good filtering effect. So, porous ceramics are widely used in the fields of water treatment, filter materials and automobile exhaust treatment [5].

There are many preparation methods of porous ceramics: adding pore forming agent, foaming method, organic precursor impregnation and sol-gel method [6, 7]. Besides, the preparation of raw materials is also diversified, such as chemical reagents, diatomite and kaolinite [8-10]. However, many researchers devote to develop different systems porous ceramics with solid wastes as raw materials in order to reduce the production cost and achieve sustainable utilization of resources [11-14]. Although the development of porous ceramics has made some progress, the research was mainly carried out in the laboratory due to the complexity of preparation technology of porous ceramics, and still far away from industrialization [15]. Moreover, the research on porous ceramics prepared by tailings mud is still in its infancy.

Tailings mud, as a common industrial solid waste, not only occupies a large amount of land resources, but also pollutes the environment seriously. The utilization of tailings mud has become a common concern of the society. In this work, we present the preparation of porous ceramics that could be used for water treatment and building materials. The porous ceramics of low volume density and high strength are prepared by foaming method with tail mud as raw material and SiC as foaming agent. The physical properties, crystal phase, and morphology of the porous ceramics were analysed. These results are meaningful to solve the problem of environmental pollution and the comprehensive utilization of the tailings mud.

2. Materials and methods

2.1. Preparation of porous ceramic samples

Porous ceramic samples were prepared via foaming process. Tailings mud, which was collected from Donglin Town, Huzhou City, was ground into a powder and mixed with a foaming agent and water. The samples were then pressed into strips with a tablet press, followed by further sintering in air by heating at a rate of 5 ℃/min. The preparation conditions of different samples were listed in Table 1.

Table 1. Preparation conditions of different samples

Sample	Sintering temperature (℃)	Soaking time (min)	SiC (wt%)	Tailings mud (wt%)
T3	1130	30	3	97
T5	1170	20	1	99
T7	1200	15	2	98

2.2. Characterization

X-ray diffraction (XRD) analysis was performed with an XD-6 X-ray diffractometer at room temperature, with CuKα radiation, 36 kV voltage and 20 mA current. X-ray data were collected in the $10<2\theta<80$ range.

Differential thermal analysis (DTA) test was carried with a Thermogravimetric analyzer (CRY-2P, China). The sample mass was about 10.00 mg, and alumina was used as the reference. The data were collected in temperature range of 20~1100 °C in the air.

The particle size of the tailing mud was determined by Mastersizer 2000 laser particle size analyzer. Water was used as a dispersant in the process of experiment.

The porosity and volume density of the samples were determined using the vacuum method and the true density method [16].

Compressive strength was tested using an electronic universal testing machine (WDW-H10) following the standard test method for ceramic material compressive resistance (China, GB/T 4740-1999).

3. Results and discussions

3.1. Physical properties of tailings mud

XRD patterns obtained for the tailing mud samples are shown in Figure 1. Two major crystalline phases, i.e., quartz and albite, were observed in the samples. There is also some albite, Ca-rich in the sample. The individual contents of the different crystalline phases of the samples were also determined from the XRD patterns. The relative contents of quartz, albite and albite, Ca-rich were 45%, 40% and 15% (wt%), respectively.

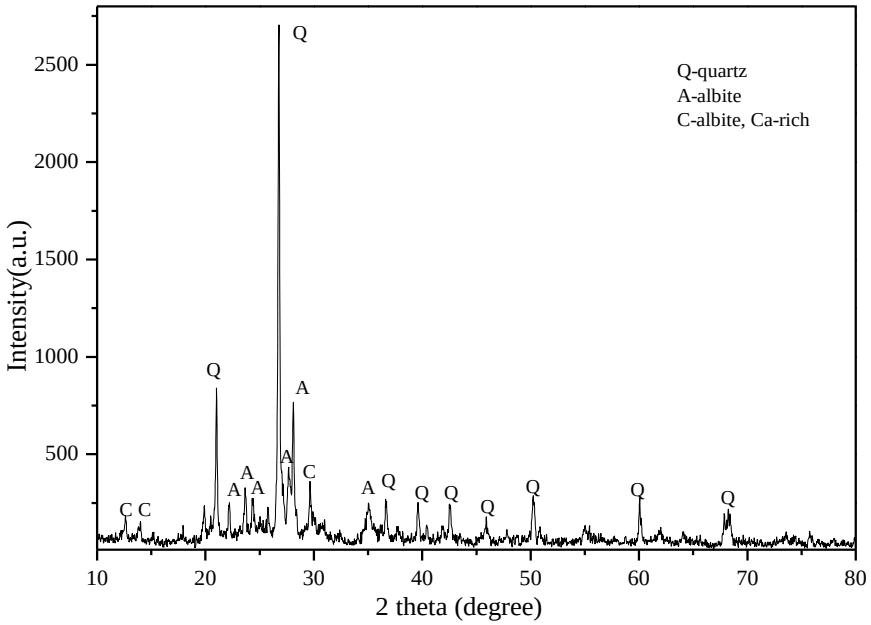

Figure 1. Room temperature XRD patterns of tailing mud

The size distribution of tailings mud was shown in Figure 2. It can be concluded that its D_{50}=14.00μm, D_{90}=51.66μm, the average volume particle size is 23.11μm while the specific surface area is 0.651 m^2/g. The size distribution width of particles is 3.408μm. The particle size is fine, the particle size distribution is uniform. and the reaction activity is good when used as the raw material.

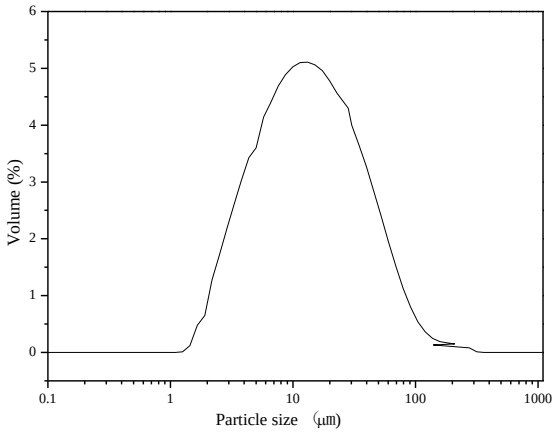

Figure 2. Particle size distribution of tailing mud

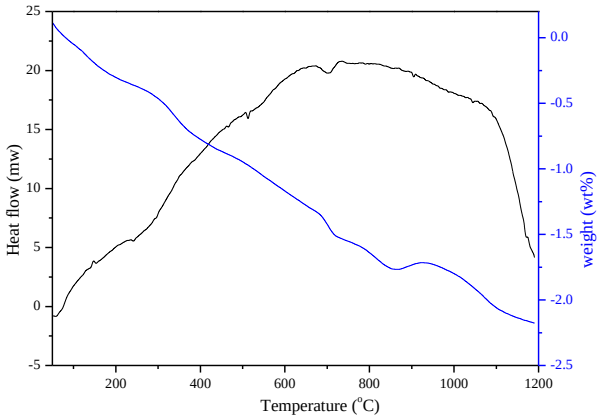

Figure 3. TG-DTA scans of tailing mud recorded at a heating rate of 20 ℃ /min

To illustrate the thermal behaviour of the tailing mud samples, a typical TG-DTA scan of tailing mud at a heating rate of 20 ℃ /min were shown in Fig. 3. Three weight loss stages were observed in the temperature ranges of 25-250 ℃, 250-600 ℃, and 600-1000 ℃. The weight losses of about 0.4% at 25-250 ℃ could be ascribed to the evaporation of absorbed waters. The weight loss of 0.9% at 250-600 °C accompanied by a small endothermic peak might belong to the transformation of β-quartz to α-quartz. Finally, the endothermic peaks centred at 703 ℃ accompanied by the weight losses of 12.6% were likely due to the transformation of α-quartz to α-tridymite. It is generally believed that the transformation temperature of α-quartz to α-tridymite is 870 ℃, which is a slow transformation. Because of the large difference in crystal structure between α-quartz and α-tridymite, the transformation energy is large, the transformation time is very long, and the transformation speed is very slow. However, because tailing mud samples contains Fe, Ca and other mineralizer, and the particle size of the tailing mud is small. All these make the transformation temperature of α-quartz to α-tridymite to a lower temperature. Furthermore, in combination with XRD results, the exothermic

peak at 903 ℃ and 1050 ℃ could be ascribed to the oxidation reaction of SiC [17].
Based on the above analysis, the tailings mud can be used as the raw material for the preparation of porous ceramics because of the primary conditions of the composition, phase and grain size of tailings mud.

3.2. Surface morphology and crystal phase of porous ceramic samples
The photos of surface morphology of porous ceramic samples were shown in Fig 4. The samples have smooth surfaces and compact structures. Both macroporous and mesoporous exist in all samples. However, the sizes of the pore are different and the distributions are uneven. The reason could be that the raw material and the foaming agent were not evenly stirred in the preparation process, which made the size distribution of the pore uneven after the sintering.

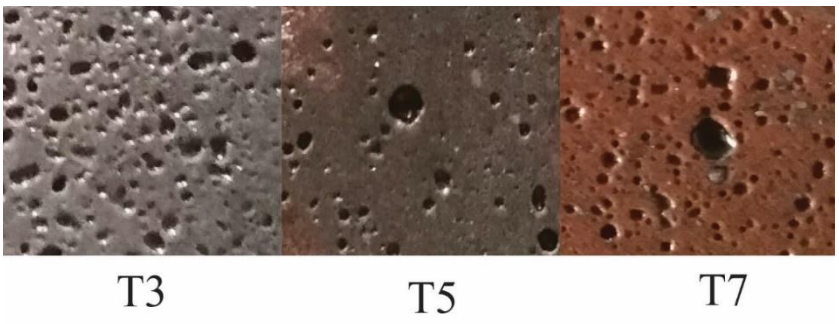

Figure 4. Photos of surface morphology of porous ceramic samples

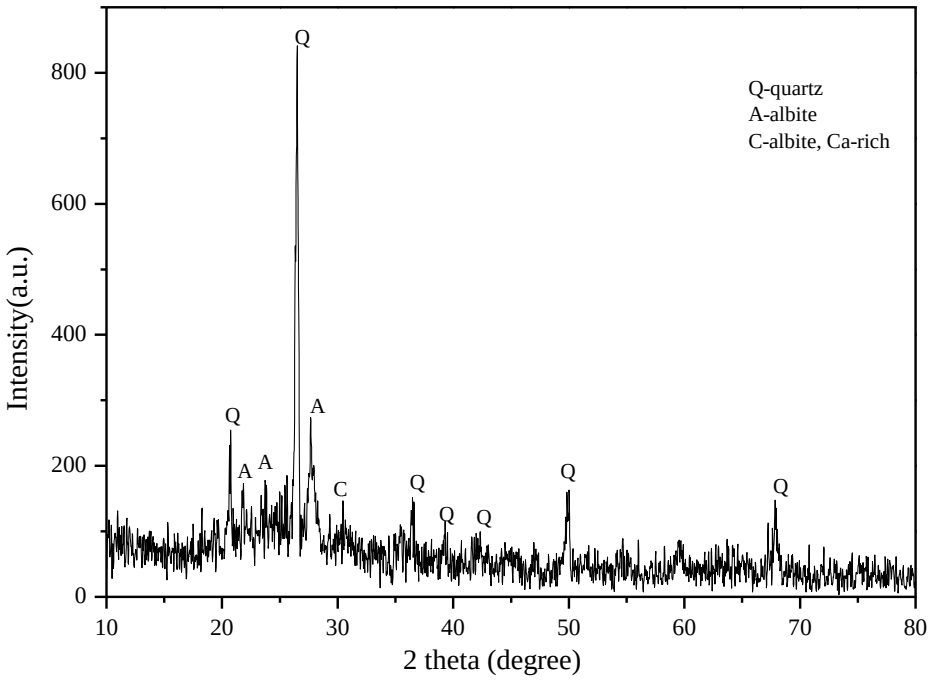

Figure 5. Room temperature XRD patterns of porous ceramic sample T3

XRD patterns of the porous ceramic sample T3 was shown in Fig. 5, demonstrating the presence of a mixture of crystalline phases. Two major crystalline phases, quartz and albite were observed in sample. In addition to those phases, albite, Ca-rich phase was also presented in the XRD pattern of sample T3. The phase of the porous ceramic sample is basically unchanged before and after the sintering compared to the raw materials. SiC was not observed in the XRD pattern of porous ceramic sample. During the sintering process, the SiO2 protective film formed on the surface of SiC react with feldspar melt to form silicate liquid phase, resulting in the destruction of the protective layer, so that

SiC was continuously reacted with oxygen to generate a large amount of CO or CO_2 gas, resulting in the disappearance of silicon carbide [18].

3.3. Physical properties of porous ceramic samples

Table 2. Physical properties of porous ceramic samples

Sample	Porosity (%)	Apparent porosity (%)	Volume density	Compressive strength (MPa)
T3	28.37	0.04	1.51	12
T5	29.24	0.33	1.74	22
T7	22.81	0.02	2.10	20

The physical properties of the porous ceramic samples, as determined by the vacuum method and an electronic universal testing machine, are listed in Table 2. Porosities of all the samples are over 20%, indicating that the samples are of moderate porosity. The foaming agent content has a certain influence on the porosity of the sample. The CO or CO_2 gas formed by foaming agent cannot be discharged rapidly in silicate liquid phase due to the liquid bond resistance. Therefore, a large number of pore structures are formed in the liquid phase, and the closed pores account for the vast majority of the pores. The compressive strength of the sample is up to 20MPA, which is related to the crystalline phases of the sample. The results of XRD show that the major phase in the sample are quartz, albite and other minerals. The formation of feldspar, quartz and other amorphous silicate materials and glass phases because of the melting and sintering of raw powder particles at high temperatures, which made the porous ceramic samples with higher strength and greater hardness.

4. Conclusions

In this work, porous ceramic samples were prepared via foaming method with tail mud as raw material. The results show that the tailings mud can be used as the raw material for the preparation of porous ceramics and foaming agent content impacts strength and pore size distribution of the resulting porous glass ceramics, which could provide a theoretical basis for the establishment of the preparation process of porous ceramic.

5. Acknowledgments

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6. References

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