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Synthesis and characterization of calcium aluminate compounds from gehlenite by high-temperature solid-state reaction

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ABSTRACT

The mineral transition mechanism and self-pulverization property of the sintered products in the $\text{Ca}_2\text{Al}_2\text{SiO}_7$ -CaO system were systematically studied using pre-synthesized gehlenite determined by XRD, SEM, FTIR and particle size analyses. The minerals of $\text{Ca}_{12}\text{Al}_{14}\text{O}_{33}$, CaAl_2O_4 , Ca_3SiO_5 and Ca_2SiO_4 are formed by the direct reactions of $\text{Ca}_2\text{Al}_2\text{SiO}_7$ with CaO. CaAl_2O_4 reacts with CaO to form $\text{Ca}_{12}\text{Al}_{14}\text{O}_{33}$ or $\text{Ca}_3\text{Al}_2\text{O}_6$, while Ca_3SiO_5 reacts with $\text{Ca}_2\text{Al}_2\text{SiO}_7$ to form Ca_2SiO_4 and calcium aluminate compounds. The sintered products mainly contain CaAl_2O_4 , $\text{Ca}_{12}\text{Al}_{14}\text{O}_{33}$ and Ca_2SiO_4 at 1350 °C or above 1500 °C when the molar ratio of CaO to Al_2O_3 is 1.0. Increasing the sintering duration or the CaO consumption promotes the transition of $\text{Ca}_2\text{Al}_2\text{SiO}_7$ to Ca_2SiO_4 and calcium aluminate compounds when sintered at 1350 °C, which accordingly improves the self-pulverization property of the sintered products. The formed minerals of $\text{Ca}_{12}\text{Al}_{14}\text{O}_{33}$, CaAl_2O_4 and Ca_2SiO_4 transform into $\text{Ca}_2\text{Al}_2\text{SiO}_7$ again when the sintering temperature is between 1400 °C and 1450 °C, and the corresponding self-pulverization property of the sintered products deteriorates sharply.

1. Introduction

As a regular constituent of ceramic body, the molten rheology and the crystallization of gehlenite ($\text{Ca}_2\text{Al}_2\text{SiO}_7$, C_2AS) from glasses play great role in the preparation of bio-soluble glass fibers and glass ceramics [1–3]. Gehlenite has also been increasingly considered as reference phases for luminescent materials, based on the introduction of rare-earth ions in the relatively large octahedral sites normally occupied by Ca^{2+} ions [4–6]. Meanwhile, the gehlenite-type structure has good applications in magnetic materials [7] and inorganic pigments [8] when doped with Fe^{3+} and Eu^{2+} ions respectively.

On the other hand, gehlenite is of important target product and intermediate phase based on the CaO- Al_2O_3 - SiO_2 system [9–11]. According to the phase diagram of CaO- Al_2O_3 - SiO_2 system, increasing sintering temperature or decreasing lime consumption can promote the formation of lime-rich calcium aluminates (e.g. $\text{Ca}_3\text{Al}_2\text{O}_6$ (C_3A), $\text{Ca}_{12}\text{Al}_{14}\text{O}_{33}$ (C_{12}A_7) and C_2AS) at the initial stage of solid-state reactions [12,13]. The authors have found that all stable calcium aluminate compounds can form at the initial reaction stage during high-temperature sintering, and they play roles as the intermediate phases to the final equilibrium phases [14,15]. It was reported that the formation of gehlenite deteriorates the self-pulverization (because of the transformation from β - Ca_2SiO_4 (C_2S) to γ - Ca_2SiO_4) and alumina leaching property of calcium aluminate clinkers [16].

As shown in Fig. 1, the crystal structure of gehlenite is a sorosilicate type (silicate with isolated group of tetrahedra) from the family of melilites, which belongs to the space group $P\bar{4}2_1m$ [17]. The cations of gehlenite are localized at three types of sites [9,18]: a voluminous eightfold coordinated site called the Thomson cube occupied by Ca^{2+} ($r = 1.12 \text{ \AA}$); a regular tetrahedral site fully occupied by Al^{3+} ($r = 0.39 \text{ \AA}$); a distorted tetrahedral site where Si^{4+} ($r = 0.26 \text{ \AA}$) and Al^{3+} ions are statistically distributed. In the layered structure, Ca^{2+} is located in the interlayers of the double tetrahedral layers consisting of corner-shared SiO_4 and AlO_4 tetrahedral. The formation kinetics and transformation mechanism of calcium aluminate compounds from CaO- Al_2O_3 system were widely studied in the past decades [19–22], but the research on the formation process from gehlenite has rarely been reported. The aim of this paper is to study the mineral transition mechanism and self-pulverization property of calcium aluminate compounds from pre-synthesized gehlenite by high-temperature solid-state reaction based on the $\text{Ca}_2\text{Al}_2\text{SiO}_7$ -CaO system.

2. Experimental

The starting materials used in this study are analytical reagents of Al_2O_3 , CaCO_3 and SiO_2 . Table 1 shows the chemical analyses of raw materials. The average particle size of Al_2O_3 , CaCO_3 and SiO_2 are 13.25 μm , 43.37 μm and 17.59 μm , respectively. Gehlenite was

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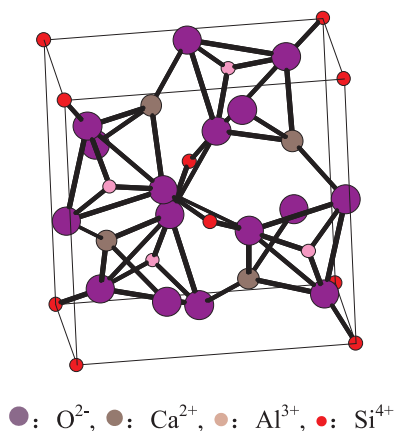


Fig. 1. Crystal structure of gehlenite.

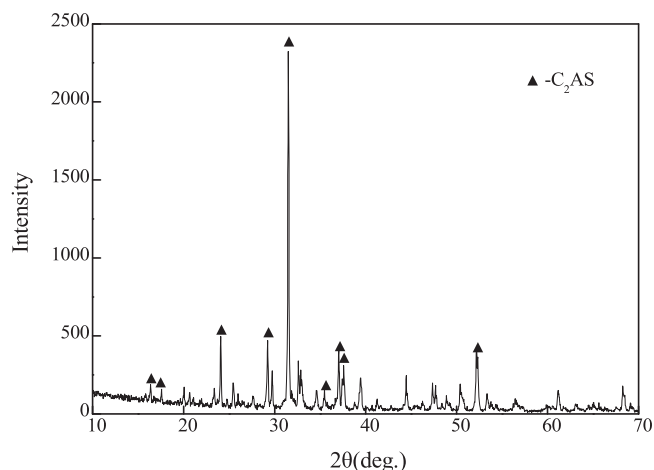


Fig. 2. XRD pattern of gehlenite synthesized at 1500 °C for 1.5 h.

Table 1

Chemical analyses of raw materials (wt%).

Raw material	CaO	Al ₂ O ₃	SiO ₂	Fe ₂ O ₃	MgO	Na ₂ O	LOI
CaCO ₃	55.91	0.009	0.007	0.001	0.08	0.1	43.88
Al ₂ O ₃	0.02	94.20	0.04	0.01	0.05	0.1	5.0
SiO ₂	0.03	0.05	96.46	0.007	0.04	0.2	3.0

synthesized according to its stoichiometric ratio at 1500 °C for 1.5 h using analytical reagents of Al₂O₃, CaCO₃ and SiO₂. The milling operation of gehlenite and CaCO₃ was carried out in a ball milling system for 2.0 h when the C/A ratios (the molar ratio of CaO in CaCO₃ to Al₂O₃ in gehlenite) are between 1.0 and 1.6. After the milling process, the samples were cold isostatically pressed at 30 MPa to produce cylindrical samples. Then the samples were sintered in a MoSi₂ resistance furnace followed by cooling in the furnace.

In order to strip CA and C₁₂A₇ in the sintered products, the leaching experiments were carried out at 85 °C for 0.5 h in sodium carbonate solution with caustic soda. The concentration of sodium carbonate (in form of Na₂O) and caustic soda (in form of Na₂O) are 80 g L⁻¹ and 10 g L⁻¹ respectively. The liquid to solid ratio for the leaching is 10.

The minerals of the sintered products and leached residues were determined by X-ray diffraction (XRD, SHIMADZU XRD-7000). The microstructure was carried out by a scanning electron microscopy (SEM, SHIMADZU SSX-550) fitted with an energy dispersive spectrometer (EDS, DX-4). The Fourier transform infrared spectroscopy (FTIR, SHIMADZU IRPrestige-21) was used to study the absorption spectra of the sintered products using KBr as matrix material. The particle size distribution of the sintered products was determined by a Malvern Mastersizer instrument laser scattering equipment (Hydro 2000MU), and the specific surface area was determined by a surface area and pore porosimetry analyzer (V-Sorb 4800 P). Full adsorption-desorption isotherms of nitrogen at 77 K and the nitrogen uptake in the partial pressure region below 0.30 provided a linear fit to the Brunauer-Emmett-Teller (BET) equation.

3. Results and discussion

3.1. Characterization of synthesized gehlenite

Gehlenite was firstly pre-synthesized at 1500 °C for 1.5 h characterized by XRD, FTIR and particle size distribution. As shown in Fig. 2, the synthesized product is mainly C₂AS. The structure of C₂AS consists of frameworks of center-shared AlO₄ tetrahedral with Si atoms in their interstices. As shown in Fig. 3, the absorption band situated at 855 cm⁻¹ attributes to the Al-O bonds stretching vibration of AlO₄ tetrahedral. The absorption band situated at 493 cm⁻¹ attributes to the

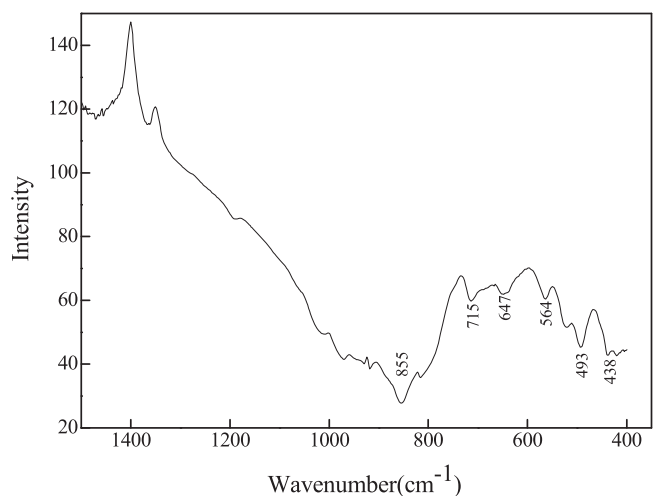


Fig. 3. FTIR spectrum of gehlenite synthesized at 1500 °C for 1.5 h.

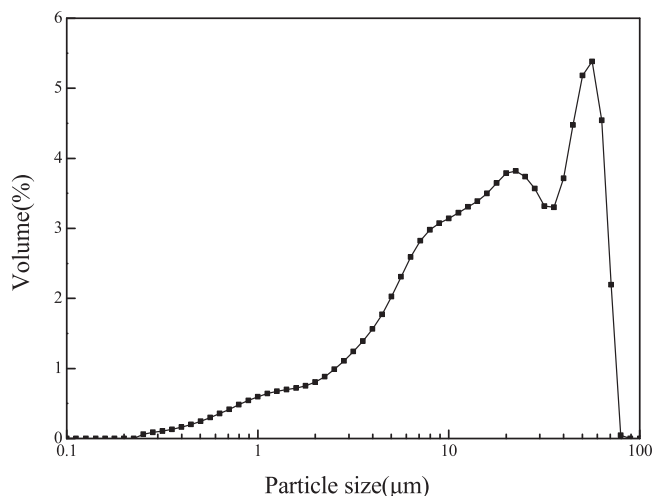


Fig. 4. Particle size distribution of gehlenite synthesized at 1500 °C for 1.5 h.

Al-O-Si bonds stretching vibration. As shown in Fig. 4, the particle size distribution of gehlenite ranges from 0.25 μm to 79.62 μm. The average size and the specific surface area are 23.83 μm and 1.08 m² g⁻¹, respectively.

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