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Preparation and characterization of coal gangue geopolymers

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HIGHLIGHTS

- Coal gangue is silicon-aluminium materials, which can be used to prepare geopolymer after high-temperature calcination.
- Coal gangue geopolymer has a high early strength, higher than that of OPC.45 cement paste specimens.
- The generation of the zeolite facies crystallization plays an obvious role on the compressive strength of coal geopolymer.
- This paper provides experimental and theoretical basis for the development and wide application of coal gangue cementitious materials.

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ABSTRACT

This paper explores the impacts of sodium hydroxide modulus, alkali lye amount and liquid–solid ratio on the strength and microstructure of coal gangue geopolymer materials, involving a total of five sodium hydroxide moduli, four NH/NS mass ratios and seven liquid–solid ratios. To characterize the morphology and structure of coal gangue geopolymer materials, we carried out XRD, TG-DTG, FT-IR and SEM analysis on the specimens respectively. The results show that NH concentration has no impact on paste fluidity and has significant impact on compressive strength of geopolymers. Both paste fluidity and compressive strength of geopolymers increase with the increase in liquid–solid ratio. However, it should be noted that the compressive strength decreases with the increase in liquid–solid ratio when the liquid–solid ratio is greater than 0.32. The optimal NH/NS mass ratio is 1:1.5 ~ 1:2. In comparison with P.O42.5 cement specimens, coal gangue geopolymers have higher initial strength and lower 28 d compressive strength. The analytical results of microstructure are consistent with those of compressive strength, which demonstrates that the polymerization products of coal gangue geopolymers are N-A-S-H gels and some other aluminosilicate zeolite crystals.

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1. Introduction

Traditional cement industries consume lots of resources and emit enormous amounts of greenhouse gases such as CO₂ [1]. It has become the research focus on environmental protection to seek alternative inorganic industrial byproducts to OPC, and reduce pollution and environment burden by virtue of the reuse of industrial byproducts [2]. Geopolymer, which is a kind of inorganic polymers, is a mixture of aluminosilicate powders and alkali metal hydroxides or alkali metal silicate solutions [3]. Currently, the most widely used aluminosilicate materials in geopolymers are fly ash and metakaolin. Studies have shown that geopolymer concrete has excellent strength, corrosion resistance, fire resistance and durability [4–7], thereby being able to replace OPC and act as concrete adhesives [8]. Therefore, geopolymer techniques are

expected to be an important part of sustainable byproduct materials [9].

Coal is the world's richest and most used fossil resources and the total reserves of 10 trillion tons or more, the United States, Russia, China and India and other countries rich in coal reserves. Coal gangue, which refers to the solid waste discharged during coal mining and coal washing processes, is one of the largest industrial solid wastes in China. It accounts for 10%–25% of the total amount of coal mining [10–12]. So far, China has accumulated more than 7 billion tons of coal gangue. Due to the high output and low utilization, the accumulation of coal gangue not only occupies lots of land, but also seriously damages the environment and ecology [13,14]. Main mineral components of coal gangue (quartz, kaolinite and muscovite) are decomposed into SiO₂ and Al₂O₃ at high temperatures [15,16]. In comparison with OPC, calcined coal gangue has lower carbon footprint, thereby becoming the most promising industrial byproducts to substitute fly ash, slag and metakaolin

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[15]. This provides new research directions on raw materials of coal gangue geopolymers.

Currently, coal gangue is mainly used to pave roads, produce cements and prepare sintered bricks, indicating the low comprehensive utilization ratio [17]. There are few researches on coal gangue geopolymers. Huang et al. [14] mixed calcined coal gangue with granulated blast-furnace slag and hydrated lime to prepare geopolymers, and found that the low geopolymer strength was mainly attributed to the low content of active calcium in coal gangue. Geng et al. [18] prepared high-strength red mud-coal gangue geopolymers with 20% coal gangue and 80% red mud, without further determining the optimal application parameters. Xu et al. [19] polymerized K_2O and Na_2O in sludge with aluminosilicates in coal gangue to prepare sludge-coal gangue polymers with excellent compressive strength. Zhang et al. [20] used NaOH, KOH and sodium silicate as the activators to prepare coal gangue geopolymers and observed amorphous alkali metal aluminosilicate gels similar to zeolite precursors. Han et al. [21] used sodium silicate as the activator to prepare alkali-activated coal gangue-slag cementitious materials and discovered that compressive strength of the composite polymers was greater than 40Mpa when the coal gangue content was less than 30%. Duan et al. [22] studied early hydration of alkali-activated coal gangue materials, using $Ca(OH)_2$ + sodium silicate and $Ca(OH)_2$ + NaOH as the activators respectively. The results show that coal gangue + $Ca(OH)_2$ + sodium silicate has induction period and compact internal structure.

To sum up, coal gangue has great space for comprehensive utilization and can be used as the raw materials of geopolymers. This paper selects sodium hydroxide and sodium silicate as the alkali activators to explore impacts of sodium hydroxide concentration, alkali lye amount and liquid–solid ratio on workability, strength, non-evaporable water content and microstructure of coal gangue geopolymers, which provides experimental and theoretical basis for the massive and efficient use of coal gangue.

2. Materials and methods

2.1. Materials

High aluminum coal gangue from Shanxi as the research object, the coal gangue was crushed by a jaw crusher, grinded by a small ball mill and calcined in a muffle furnace at 700 °C for 2 h. The particle size distribution of coal gangue shown in Fig. 1. 96% sodium hydroxide and sodium silicate with a modulus of 3.22 (26.5% SiO_2 , 8.5% Na_2O , 65% H_2O) as the alkali activators were selected.

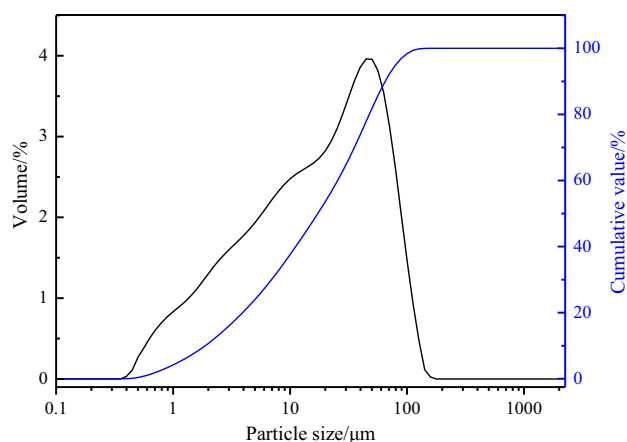


Fig. 1. Particle size distribution of coal gangue.

Main chemical components of calcined coal gangue was analyzed by X-ray fluorescence spectroscopy (XRF), as shown in Table 1. The mineral components of original coal gangue and coal gangue calcined at 700 °C by X-ray diffraction (XRD) and demonstrated the results in Fig. 2. The main mineral components of coal gangue include quartz, kaolinite and muscovite. In addition, high-temperature calcination weakens the diffraction peak of kaolinite and enhances the diffraction peak of quartz. Based on smoothing and fitting the XRD spectra of coal gangue and the analysis of crystallinity of the coal gangue samples by Jade software, it was found that the crystallinity of samples before calcination is 53.60 (0.56)% and that after calcination is 15.07 (0.41)%, and the number in parentheses above means variance. Besides, the crystallinity of Silica in calcined coal gangue is 11.18 (0.14)%. The amorphous phase includes silicon and aluminum mineral composition.

2.2. Mixes design and specimens preparation

In this research, to test the impacts of sodium hydroxide concentration, alkali lye amount and liquid–solid ratio, a total of 15 experiments were carried out, involving five sodium hydroxide moduli (8 M, 10 M, 12 M, 14 M and 16 M), seven liquid–solid ratios (0.28, 0.30, 0.32, 0.34, 0.36, 0.38 and 0.40) and 4 NaOH/ Na_2SiO_3 mass ratios (1:1, 1:1.5, 1:2 and 1:2.5), P.042.5 pure cement paste specimen as control specimen. The specific mixing ratio was shown in Table 2. The concentration of alkali-activated solutions in the mixture was obtained by adjusting the amounts of solid sodium hydroxide, sodium silicate solution and water, and the liquid–solid ratio in Table 2 was achieved by regulating additional water. The alkali-activated solutions were prepared 24 h before use and cooled to room temperature to ensure uniform mixing of activator components and improve polymerization. Added alkali-activated solutions to coal gangue, mixed them for 5 min with a cement paste mixer, quickly poured the fresh paste into steel dies (40 mm × 40 mm × 40 mm) and vibrated them for 60 s on an electric vibration table to remove residual air. The dies were covered with thin polyethylene films and cured for 1 day at $RH = 95 \pm 1\%$ and $T = 20 \pm 2$ °C. They were then demoulded, transferred to standard curing rooms and cured for 1 d, 3 d, 7 d and 28 day respectively.

2.3. Methods

2.3.1. Paste flow test

Standard truncated conical dies (upper diameter $\Phi = 36$ mm; lower diameter $\Phi = 60$ mm; $H = 60$ mm) were selected for the paste fluidity test. The truncated conical dies were placed on the center of a smooth glass plate, filled with fresh paste, scraped by a scraper and lifted up vertically (the stopwatch was started at the same time). After the paste flew on the glass plate for 30 s, we measured the maximum diameters in two mutually perpendicular directions with the ruler and took the average as the fluidity of coal gangue geopolymers. Each group of specimens was measured three times and average of the three fluidity results was used as the final paste fluidity.

2.3.2. Compressive strength test

A YAW-300 pressure testing machine was employed to test 1 d, 3 d, 7 d and 28 d compressive strength of the cube specimens of coal gangue geopolymers cementitious materials. The loading speed ranged from 0.5 to 0.8 MPa/s. Three samples of each mixture proportion were tested, with the experimental values averaged to generate the test value for each mixture proportion.

2.3.3. Non-evaporable water content test

Non-evaporable water is also referred to as bound water. Specimens cured for 1 d, 3 d, 7 d and 28 d were demoulded, cored,

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