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## Improving strength of calcinated coal gangue geopolymer mortars via increasing calcium content

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### HIGHLIGHTS

- Two methods were applied to improve the active calcium content in coal gangue geopolymer.
- The compressive strength of coal gangue geopolymer was considerably improved by increasing calcium content.
- The activity of coal gangue can be further enhanced by calcined at 700 °C.

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### ABSTRACT

In this study, the calcinated coal gangue was mixed with granulated blast furnace slag (GBFS) and slaked lime in making geopolymer based on increasing calcium content to improve the strength. Besides, the coal gangue geopolymer were characterized for performance, phase transformation, molecular structure and microstructure by means of Thermogravimetric Analysis (TGA), X-ray diffraction (XRD), Fourier Transform infrared spectroscopy (FT-IR), and Scanning Electron Microscope (SEM) analysis. Results showed that low content of active calcium in coal gangue was the main reason for low strength of geopolymer. The strength of geopolymer improved gradually with the increase of GBFS and slaked lime due to the addition of activated calcium content. However, the increase of the compressive strength of geopolymer was not obvious when the content of GBFS was more than 40%, and the compressive strength of geopolymer dropped sharply when the slaked lime content exceed over 5%.

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## 1. Introduction

Coal gangue is a type of solid waste, which is produced in the process of coal mining, washing, and processing, accounting for 10%–25% of the total amount of coal. It is estimated that the annual production of coal gangue wastes is about 0.75 billion tons for coal mining since 2010, which accounts for a quarter of the total industrial solid wastes in China. What's more, there are total 4.5 billion tons of coal gangue wastes stockpiled at 1700 waste dumps which occupied 15,000 ha lands [1]. The output of coal gangue is high and its utilization rate is low, resulting in massive accumulation, which causes severe environmental pollution [2,3]. However, coal gangue is a type of available resources, which can replace fuel for electricity generation and produce chemical products like aluminum chlo-

ride, water glass [4]. But the utilization of coal gangue in this way has not been promoted due to the high production cost and low profit. Therefore, economical and rational utilization of coal gangue relieves the environment, saves resources, and reduces land occupation as well as promotes sustainable development [5]. Consequently, finding a suitable method to utilize coal gangue has become a focus for research.

The use of coal gangue as raw material for concrete is a suitable way to achieve utilization of this resource [6,7]. In recent years, scholars have carried out extensive research on prospective applications of coal gangue as instead of concrete aggregates [8,9], concrete mineral admixtures [10,11] and so on [12]. Current research shows that coal gangue is less compatible with cement-based materials and it is a sedimentary rock with laminar structure, when the coal gangue is broken into aggregate, it is characterized by loose and soft particles with a needle or flake-like shape. For use as concrete aggregate, the water demand of mixing is high and the coal gangue aggregate cannot provide adequate support

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for the concrete skeleton. Furthermore, it is less active [13–16], causing a substantial decline in concrete strength when it is used as mineral admixture of concrete to replace cement.

Geopolymer is a kind of binding materials produced in the reaction in which strong alkali activates aluminosilicate at room temperature or autoclaved condition. Generally, industrial solid waste mainly composed of aluminosilicate can be used as its raw material [17]. The main components of calcinated coal gangue are aluminosilicate, so the geopolymer materials provide an effective way for the utilization of coal gangue. However, the uncalcinated coal gangue has a low reactivity and cannot be made of geopolymer materials. The activity of coal gangue could be enhanced by calcined between 700 °C and 900 °C and a certain strength of the geopolymer material can be prepared at room temperature. Unfortunately, the compressive strength of the geopolymer prepared from calcined coal gangue is too low compared with the alkali activated GBFS. The difference of calcium content is the main reason for the difference in performance of the two materials [18,19]. It is hopeful to increase the compressive strength of coal gangue geopolymer by increasing the calcium content. Therefore, researching the way and mechanism of enhancement by increasing the calcium content in coal gangue geopolymer to enhance the compressive strength of it is a significant step for the development and application of coal gangue.

In this study, two methods were proposed to increase the amount of calcium content in coal gangue geopolymer, i.e. GBFS and slaked lime, and calcined coal gangue to enhance its polymerization reaction activity. In order to study the impact of the calcium content on the geopolymer and the enhancing mechanism, the compressive strength of coal gangue geopolymer were measured, meanwhile conduct analysis of Thermal gravity analysis (TGA), X-ray diffraction (XRD), Scanning electron microscope (SEM) and Fourier transform infrared spectroscopy (FT-IR) of coal gangue geopolymer.

## 2. 2. Experimental materials and methods

### 2.1. Materials

The coal gangue was ground into fine powder (CG) [20,21] and its surface area is 400 m<sup>2</sup>/kg; the industrial analysis is shown in Table 1 and the chemical composition is shown in Table 2. The GBFS used in the experiment is the S95 granulated blast furnace slag powder which conforms to the Chinese standard GB/T 1806-2008 and the chemical composition is also shown in Table 2. The slaked lime adopts industrial grade. The purity of it is 95% and it had a surface area of 400 m<sup>2</sup>/kg. For alkali activated solutions, sodium hydroxide (NaOH) and sodium silicate (Na<sub>2</sub>SiO<sub>3</sub>) with 9.65% Na<sub>2</sub>O, 25.22% SiO<sub>2</sub>, and 65.13% H<sub>2</sub>O were used. The sodium silicate solution has a modulus of 2.7 and a Baume degree of 37°. Refer to the dosage of NaOH and water in Table 3, 125 ml 4.8 M NaOH solution was prepared to make each set of mortars. The solu-

tion was then left for 24 h before use. The utilized test sand was ISO standard sand with specific gravity of 2.58 and fineness modulus of 2.9.

### 2.2. Methods

#### 2.2.1. Determination of active calcium content in CG

With reference to the “method for chemical analysis of cement” GB/T176-2008 [22], the contents of CG active components were detected via dissolution test. (1) Acid dissolution: CG was mixed with water to form slurry. A specific amount of hydrochloric acid was added into the slurry and completely mixed. The mixture was heated in boiling water for 15 min and then filtered; the filtered residue was obtained via alkali dissolution. (2) Alkali dissolution: the NaOH solution was added into the beaker that contained the residue. The beaker was placed in boiling water for 15 min of heating and finally the insoluble residue was filtered out. (3) Calcination: The insoluble residue was dried and calcined in a muffle furnace at 1000 °C for half an hour. The weight of the residue after the calcination was measured and the contents of chemical components of the residue was analyzed via X-ray fluorescence spectrometer (XRF). The mass loss after chemical dissolution was taken as the reactive content and it was calculated according to Eq. (1).

$$P_r = (M_t \times P_\omega - M_r \times P_\omega) / M_t \quad (1)$$

where,  $P_r$  is the proportion of active chemical composition;  $M_t$  denotes the total mass before reaction.

$M_r$  denotes the mass of the residue after the reaction; and  $P_\omega$  is the proportion of the chemical composition.

#### 2.2.2. Preparation and mechanical properties of CG geopolymer

2.2.2.1. *Mixing proportion of specimens.* With reference to the “Method of testing cements–Determination of strength–ISO” GB/T17671-1999 [23], the mortar specimens of the CG geopolymer were prepared. Since the uncalcinated CG is less active, the strength of the CG geopolymer prepared in this way was low. To improve the mechanical properties, CG was calcinated at 350 °C, 700 °C and 1050 °C respectively, to examine the enhancement effect of different calcination temperatures. Then, GBFS and slaked lime was mixed into the CG geopolymer to achieve enhancement via increased calcium content.

Table 3 shows the mixing proportion of mortar specimens of the CG geopolymer. The groups from L-1 to L-4 represent CG geopolymer prepared by calcined CG at different temperatures. The groups from L-5 to L-8 represent a binary composite geopolymer, which was blended with different amount of GBFS. The groups from L-9 to L-12 represent a ternary composite geopolymer, which was blended with different amount of GBFS and slaked lime.

2.2.2.2. *Casting of specimens.* The CG geopolymer specimens were mixed in a 20 ± 2 °C controlled room. The procedure started by mixing the CG, GBFS and slaked lime for 1 min, then NaOH solution

**Table 1**  
Industrial analysis of coal gangue w/%

	Moisture	Ash content	Volatile	Fixed carbon fcd	Caloric value	Bomb test
Coal gangue	0.8%	86.59%	9.07%	3.54%	1620 kJ/kg	810 kJ/kg

**Table 2**  
Chemical compositions of raw materials w/%

Raw material	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	CaO	MgO	Na <sub>2</sub> O	K <sub>2</sub> O	Others	Ignition loss
CG	59.73	20.16	5.5	1.96	1.01	0.23	2.52	0.34	8.24
GBFS	31.35	18.65	0.57	34.65	9.31	–	–	0.41	0.7

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