



Effect of elevated temperature on the properties of geopolymer synthesized from calcined ore-dressing tailing of bauxite and ground-granulated blast furnace slag



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HIGHLIGHTS

- Geopolymer mortars severely deteriorate in strength after heating up to 1200 °C.
- Dehydration, dehydroxylation, decomposition of gels and crystallization lead mortars to deteriorate.
- Crystalline phases like calcium aluminates and anorthite are formed at 1000 °C and 1200 °C.
- Thermal mismatch between contracting gels and expanding quartz is observed.

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ABSTRACT

A geopolymer was prepared using calcined ore-dressing tailing of bauxite and ground-granulated blast furnace slag. To explore the effect on its properties of heating the geopolymer to 1200 °C, the physical evolutions, including the appearance, length, compressive strength and porosity, and the chemical processes involving the decomposition and crystallization of specimens after heating were investigated. After exposure to elevated temperatures up to 1000 °C, the studied geopolymer mortars exhibited the following changes. The linear length initially showed shrinkage, responding to free water loss and gel dehydration, and then, it exhibited expansion, resulting from a volume increase due to the displacive and reconstructive phase transition of quartz in the mortars. The compressive strength of the mortars decreased due to the increasing cracks and porosity arising from the dehydration, dehydroxylation and thermal mismatch between the contracting gels and expanding quartz. Additional conversion of the X-ray amorphous gels, which exhibit excellent thermal stability below 800 °C, into such crystalline phases as calcium aluminum oxide and anorthite ($\text{CaAl}_2(\text{SiO}_4)_2$) was found at 1000 °C, resulting in a severe deterioration of strength and a reduction of approximately 40.0 MPa. After exposure to 1200 °C, a strength gain was observed as a result of the densification and self-healing caused by the viscous sintering and the formation of ceramic phases, such as anorthite (sodian, ordered, $(\text{Ca},\text{Na})(\text{Al},\text{Si})_2\text{Si}_2\text{O}_8$).

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1. Introduction

Geopolymer is considered a green building material and is a new type of eco-friendly cementitious material due to its low energy requirements compared with traditional OPC and associated low CO_2 emissions from using common aluminosilicate wastes. Geopolymer research has attracted many investigators who are active in materials science and engineering to this field over the past several decades [1]. Accordingly, the scope of the research that exists in the scientific literature is significant. The

geopolymerization model, geopolymerization products, structural characterization and the preparation technology regarding the effect of the processing conditions, raw materials, aluminum incorporation and contaminants on the chemical and physical properties have been summarized by Duxson et al. [2]. The classification of geopolymer and its properties and chemical and structural characteristics were reviewed by Shi et al. [3]. Based on the excellent mechanical performance, durability and inflammability, Davidovits, a pioneer of geopolymer, has presented 13 types of geopolymer applications since 1972 [4]. These potential applications include low-tech building materials, insulated panels and walls and decorative stone artifacts. Geopolymer technology is also being considered for sustainable development by the minerals

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sector due to its utilization of mining and metallurgical wastes and by-products, such as coal gangue, fly ash, steel slag and red mud. Furthermore, potential applications such as green concrete for building sustainable cities with a reduced carbon footprint have been proposed by researchers [5–7]. Among the above applications, fire-resistant components have been closely examined by investigators. It was inferred from previous studies that the inflammability resulting from the highly cross-linked geopolymerization gels would make geopolymer fire resistant, and therefore, it is being used in fireproof building materials and sound and heat insulators [8,9].

When geopolymer is used in applications in the building industry in which elevated temperature may be expected, such as in walls and floors adjacent to various heat machines, its volumetric stability, phase stability and resistance to strength deterioration at high temperature play a significant role [1]. Therefore, the physical evolution of geopolymer made with fly ash, metakaolin and/or ground-granulated blast furnace slag (hereafter referred to as slag) after heating has been intensively studied over the past several years. The literature shows that fly ash-based Na-geopolymer, which exhibits a small expansion in the temperature range from 700 to 800 °C, displays the best strength performance [9]. When jointly activated by sodium silicate and potassium hydroxide, fly ash-based geopolymer paste exhibits a strength increase of approximately 53% after exposure to 800 °C [10], while metakaolin-based geopolymer activated by the same solutions shows a strength reduction after a similar exposure [11]. Slag-based geopolymer activated by sodium silicate and hydrated lime displays strength loss behavior similar to that of Portland cement [12]. It has also been found that metakaolin-based K-geopolymer with a low Si/Al ratio has outstanding thermal stability, with melting points in the vicinity of 1400 °C [8], while metakaolin-based Na-geopolymer does not have above excellent thermal stability, and its initial amorphous structure is replaced by the crystalline Na-feldspars at 650 °C [13]. Although the above geopolymers exhibit different thermal characteristics due to the different starting materials and preparation parameters, geopolymer has a high potential for use in refractories or heat insulators via the optimization of the mixture design and curing conditions.

The ore-dressing tailing of bauxite (hereafter referred to as tailing), an aluminosilicate-rich byproduct of the ore-dressing-Bayer process or ore-dressing-sintering process, which are fairly suitable for the low-grade bauxite in China, is a new type of solid waste of the Chinese alumina industry in addition to red mud. In general, approximately 20% of the tailing of bauxite is emitted in the ore-dressing flow as low-grade bauxite. The total annual emission of tailing in China is now approximately 700,000 tons from three lines in the Zhongzhou factory of the Aluminum Corporation of China Limited. It should be noted that the ore-dressing-Bayer process and ore-dressing-sintering process will become more popular in China due to a decrease in the import of overseas bauxite and an increase in the use of local bauxite with high Si and Fe, increasing the generation of this tailing. Moreover, this tailing is more difficult to dispose of than red mud due to its fine particles, high water content and slow depositing and non-cementitious properties. Therefore, it is important to develop methods for the reuse of this tailing.

This tailing is rich in aluminosilicate minerals, such as kaolinite, and thus conforms to the requirements of a starting material for geopolymer in some aspects. In fact, previous studies show that geopolymer prepared using calcined tailing and slag has the same excellent mechanical properties and resistance to the chemical erosion of sulfate as other geopolymers synthesized from fly ash and slag [14]. However, its volumetric stability, strength degradation and gel structure changes at elevated temperature are unknown. Furthermore, many impurities in the tailing, such as corundum, muscovite and anatase, may affect its thermal behavior.

Hence, it is of great necessity to illustrate the thermal properties of this geopolymer.

This investigation will elucidate the physical evolution of tailing-slag geopolymer, including the appearance, length, strength and pore structure, after heating to 1200 °C. The chemical changes after heating, such as the gel decomposition and crystallization, will also be studied. From these results, the thermal behavior of tailing-slag geopolymer will be clarified.

2. Materials and methods

2.1. Materials

The tailing was sourced from the Zibo factory of the Aluminum Corporation of China Limited in Shandong province, China. The slag powder with a specific surface area of 405 m²/kg used to aid setting at ambient temperatures was supplied by the Shougang Group, Beijing, China. The chemical compositions of the tailing and slag are shown in Table 1.

The tailing was heated in a muffle furnace at 800 °C for 1 h. Diaspore and kaolinite were converted into corundum and metakaolinite, respectively, due to dehydration, as shown in Fig. 1, while the other minerals in the tailing, such as anatase and muscovite, did not change. Then, the calcined tailing was ground in a ball mill to a fineness of 10% residue on a 45 μm sieve by weight.

Sodium silicate solution with 13.64% Na₂O (in weight, same as below) and 32.13% SiO₂ was used as the activator. Solid sodium hydroxide was used to modify the Ms (molar ratio of SiO₂ to Na₂O) of the sodium silicate solution from 2.43 to 1.80 based on the stoichiometry. After efficient mixing, the modified solution was sealed using glass plates and was heated on an electric resistance heater until boiling and then cooled to room temperature; it was stored for a minimum of 24 h prior to use to allow equilibration.

ISO quartz sand, which was used as fine aggregates in the mortars, was provided by the China Building Materials Academy (CBMA), and its chemical and physical parameters, such as the particle size, are consistent with ISO 679.

2.2. Experimental procedures

It was found that the geopolymer synthesized solely from the calcined tailing did not harden at room temperature. However, the presence of slag in this system could aid its setting and hardening. Hence, slag was selected to be used as a setting accelerator at room temperature in this work. The results showed that the strength of tailing-slag geopolymer increased with the increasing gradual incorporation of slag at the fixed ratio of powder to activator. For example, the compressive strength at 28 days was 20.5 MPa in mortars with 20% slag, while the strength increased to 50.0 MPa with 30% slag. Although the further increase of slag may cause a further strength gain, the content of slag was fixed at 30% to control the total cost of geopolymer, as slag powder is an expensive commercial product that is used as a supplementary cementitious material in cement and mineral admixtures in concrete in China. Currently, the price of this powder is approximately 200 yuan per ton, which is equivalent to approximately 32 US dollars.

A 20% sodium silicate solution (by weight, excluding the water in the sodium silicate solution) was added to blended powder consisting of 70% calcined tailing and 30% slag. After mechanical mixing for 4 min, mortars with a water/powder ratio of 0.5 (including the water in the sodium silicate solution) and a powder/ISO sand ratio of 1/3 were molded into prismatic molds measuring 40 × 40 × 160 mm and were vibrated for a further 2 min to remove entrained air. Then, the mortars were cured at RH 95% ± 5% and 20 °C ± 2 °C for 24 h. After demolding, the mortars were continually cured under the above conditions for an additional 20 days. At the end of this curing regimen, the mortars were stored for 7 days at 20 °C ± 2 °C in open air to obtain weathered specimens.

The 20 × 20 × 20 mm geopolymer paste samples used for FTIR analysis had the same mixture ratio, molding parameters and curing procedure as that of mortars.

Weathered mortars and pastes were exposed to elevated temperature at 200 °C, 400 °C, 600 °C, 800 °C, 1000 °C and 1200 °C for 2 h at a gradual incremental rate of approximately 10 °C per minute from room temperature. Then, the calcined specimens were quickly cooled to room temperature in open air using an electric fan. Geopolymer mortars were selected to investigate the effect of heating on physical and chemical changes because the geopolymer-based composition used in construction often comprises aluminosilicate binders and fine aggregates, such as quartz sand.

The appearance, dimension, strength and porosity of the calcined and reference mortars were investigated. Three specimens were used for each length measurement, and six were used in the compressive strength testing; however, only three specimens were used in strength measurement due to the thermal damage after exposure to 1200 °C. The dimensions were measured using Vernier calipers (Botou, China). Strength testing was performed using a TYE-300D flexural and compressive strength testing machine, Wuxi Jianyi Instrument, China. A load rate of 2400 N/s ± 200 N/s was used in the compressive strength testing. The pore parameters were measured on a Micromeritics Autopore III 9420, and a maximum pressure of 414 MPa (0.003 μm) was recommended.

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