

Synthesis and strength optimization of one-part geopolymer based on red mud



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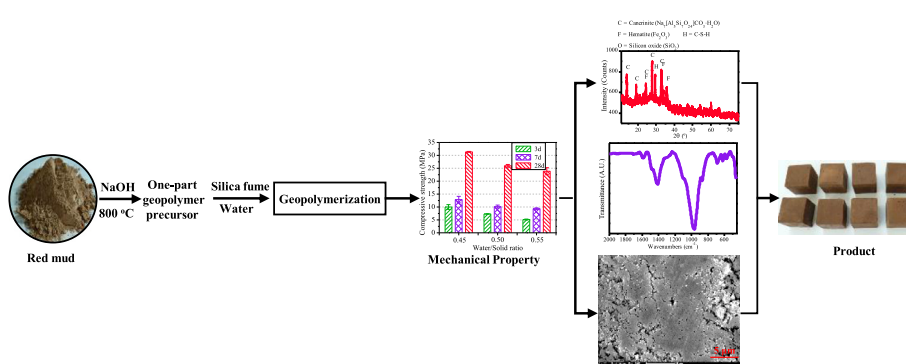
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HIGHLIGHTS

- One-part geopolymer was synthesized by using Bayer red mud as main raw material.
- Long-term strength of binder was significantly improved with addition of 20–30 wt% SF.
- Lower water/solid ratio contributed to increasing the strength.
- The compressive strength of geopolymer cured for 28 d reached 31.5 MPa.
- Geopolymerization of dissolved aluminosilicate and silica formed dense matrices.

GRAPHICAL ABSTRACT



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ABSTRACT

One-part geopolymer was synthesized from alkali-thermal activated Bayer red mud (RM) with addition of silica to optimize its composition. The RM was pretreated through alkali-thermal activation and turned to geopolymer precursor, which could be used by only adding water in blending process. However the long-term strength of the binder with only RM was poor because of the unstable polymerization due to the low SiO₂/Al₂O₃ molar ratio (1.41). Silica fume (SF) was chosen to increase the SiO₂/Al₂O₃ molar ratio of the geopolymer formulation. By adding 25 wt% of SF, the 28 d compressive strength of the geopolymer with a SiO₂/Al₂O₃ molar ratio of 3.45 could reach 31.5 MPa at a water/solid ratio of 0.45. Sodium aluminosilicate in the activated RM dissolved in water and formed an alkaline environment to dissolve SF. The dissolved silica participated in geopolymerization, leading to a satisfactory geopolymer composition. Typical amorphous geopolymer matrices were formed in the binder completely cured.

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1. Introduction

Bayer red mud (RM) or bauxite residue is the residue of bauxite ores after digestion by caustic soda through the Bayer process to produce alumina. It is a high alkaline waste with an average pH of 11.3 ± 1.0 [1] and is classified as a toxic industrial waste [2].

The high alkalinity and superfine particle size make proper disposal of RM difficult. Most of RM is still disposed through storage on land, including lagooning, dry stacking, and dry cake disposal [3]. But land disposal may cause serious environmental pollution, if RM was leaked into the surrounding environment. Ecological disasters caused by RM dam-break have occurred for many times, such as the event in Hungary in 2010 [4].

The research of economical alternatives to utilize red mud have been carried out for more than 50 years. Numerous application

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possibilities have been researched and developed. The main research areas could be summarized as: metallurgical applications [5], filler or substrate for composite materials [6], catalysts [7], adsorbents [8], construction and building materials [9]. Despite thousands of publications and patents on the subject have been published, large-scale utilization of RM is still absent. Klauber et al. summarized the barriers that need to be overcome for RM utilization as: volume, performance, cost and risk [10]. Research to refine the utilization technology still needs to be conducted. Among the utilization options, construction and building materials pose lower risk for implementation. Manufacture of geopolymers based on RM including controlled low strength materials are one of the suggested research project [10].

Geopolymer poses as a viable alternative for utilizing RM in building materials to avoid the alkali-aggregate reaction since alkali is a necessary component for geopolymer. In recent decades, geopolymer has been attracting worldwide attentions for their low CO₂ emissions and high properties. Geopolymers are synthesized by activating solid aluminosilicate sources with alkali metal hydroxide or silicate solutions through a series of dissolution–reorientation–solidification reactions [11]. The binding property of the geopolymer results from the amorphous alkali aluminosilicate gels, which have a general formula as $M_n[-(Si-O)_2-z-Al-O]_n \cdot wH_2O$, wherein M represents one or more alkali metals and z is 1, 2 or 3 [12]. Some geopolymers also contain alkaline earth cations, particularly Ca²⁺ based on industrial wastes such as granulated blast furnace slag or fly ash, but it's not sure whether the alkaline earth cations are actually incorporated into the geopolymer structure [13]. The satisfactory geopolymer compositions are suggested to be in the range of M₂O/SiO₂, 0.2–0.48; SiO₂/Al₂O₃, 3.3–4.5; and H₂O/M₂O, 10–25 [14].

RM is not a quite ideal material for preparing geopolymer directly due to its poor activity and low SiO₂/Al₂O₃ molar ratio (lower than 2), thus it is usually pretreated and mixed with other materials to prepare geopolymer. Some researches have been done by combining RM with other excellent geopolymer precursors such as metakaolin [15], fly ash [16–18], and rice husk ash [19] and using sodium hydroxide or sodium silicate solutions as an activator to synthesize geopolymer. In our previous study [20], a type of geopolymer was synthesized from thermal-pretreated RM and granulated blast furnace slag by using sodium silicate as the activator. These geopolymers synthesized by mixing solid aluminosilicate sources with an alkaline activator solution were called as two-part geopolymer for their two-part mix process, which was the conventional design of geopolymer. If the alkali came from the solid phase, and the blending process was just one-part mix (i.e. only need to add water), geopolymer would present the convenience of ordinary Portland cement (OPC). Koloušek et al. proposed the new procedure for synthesizing geopolymer based on direct calcinations of low-quality kaolin with Na/K hydroxides to get one-part geopolymer precursor [21]. Feng et al. synthesized a one-part geopolymer from albite by calcinating it with addition of NaOH and Na₂CO₃ [22]. One-part geopolymers would present opportunities beyond the traditional two-part geopolymers because one-part geopolymers were ideal for large-scale deployment, as most of the quality control can be dealt with centrally [23].

In the previous work, a one-part geopolymer had been synthesized from Bayer red mud through an alkali–thermal activation process [24]. But the binder collapsed in long-term curing after 7 d since the polymerization of Al–O and Si–O was unstable due to its low SiO₂/Al₂O₃ molar ratio of only 1.41 [24], much lower than the appropriate range of 3.3–4.5. This article presents a research on solving the strength deterioration problem of the one-part geopolymer synthesized from Bayer red mud, by adding another silica-rich material with high activity – silica fume (SF) – to improve the SiO₂/Al₂O₃ molar ratio of the binder, thus to improve the stability of the product.

2. Material and methods

2.1. Raw materials

A local Bayer red mud, provided by an alumina plant of Chalco Co. in Zhengzhou, China, was dried to constant weight at 105 °C and grinded to pass a 0.30 mm mesh sieve. It was a typical residue from the Bayer process to produce alumina using Chinese low-Fe diasporite bauxite ores [25]. The particle size of the RM was in the range of 0.1–70 μm with a median diameter (*d*₅₀) of 3.5 μm as determined by laser granulometry. A condensed silica fume, provided by China Construction Ready Mixed Concrete Co. Ltd. in Wuhan, China, was used to improve the SiO₂/Al₂O₃ molar ratio of the formulations. SF is a by-product of the manufacture of silicon or of various silicon alloys. The particle size of the SF was in the range of 0.25–150 μm with a *d*₅₀ of 23.66 μm as determined by laser granulometry. The chemical compositions of the raw materials are presented in Table 1, which were detected by an Axios Advanced X-ray Fluorescence Spectrometer (XRF). The RM is an alkaline aluminosilicate source with a SiO₂/Al₂O₃ molar ratio of 1.41 and the SF is a silica-rich source with 94.43 wt% of SiO₂.

The mineral phases of raw RM and SF were investigated by the powder X-ray diffractometry (XRD), using an Empyrean (PANalytical B.V., Holland) with Cu Kα radiation and λ = 1.5418 Å, operated at 40 mA, 40 kV with a scanning rate of 0.2785°/s for 2θ in the range from 10° to 75°. The XRD patterns of the mineral phases of RM and SF are shown in Fig. 1. The mineral phases in the raw RM include gibbsite, hematite, calcite, cancrinite, muscovite-2, and katoite. Cancrinite is a group of normal zeolitic mineral in alkaline massifs as a rock-forming mineral, and has the common generalized formula (Na, Ca, K)_{7–8}[(Si, Al)₁₂O₂₄](CO₃, OH)₂·2–3H₂O [26]. The aluminosilicates were mainly in the crystal form with low activity, such as cancrinite, muscovite-2, and katoite. The SF mainly consist of amorphous active silica, with a little of unburnt carbon in the form of silicon carbide.

2.2. Alkali–thermal activation of red mud

To get one-part geopolymer precursors from Bayer RM, the RM was pretreated through an alkali–thermal activation process. RM samples were mixed with sodium hydroxide pellets of analytical grade (99.9%), and calcined at 800 °C for 1 h in a muffle furnace, and then cooled naturally in the furnace to room temperature. Two specified amounts of sodium hydroxide were chosen, 10 and 15 wt% (based on Na₂O with respect to the mass of RM), and the corresponding alkali–thermal activated RM were called RM-10N and RM-15N, respectively. The alkali–thermal activated RM was grinded in a sample preparation comminuter for 3 min to pass a 0.3 mm mesh sieve.

2.3. Synthesis of one-part geopolymer

To design one-part geopolymer with different formulations, the alkali–thermal activated RM were mixed with 0, 5, 10, 15, 20, 25, 30 wt% of SF with respect to the mass of total solid to produce different geopolymer precursors. The geopolymer precursors were then mechanically blended with water at a water/solid ratio of 0.65 for 5 min to obtain homogeneous pastes. The binder made from RM has strong absorbing capacity of water due to the superfine particle-size distribution and large surface area of RM. To get a geopolymer with higher strength, the water/solid ratio was decreased. The water/solid ratios of 0.60 and 0.55 were adopted for formulations at the SF addition of 25 wt%. It was known that sodium lignosulphonate was usually used as dispersant to improve the flowability and workability of concrete [27]. By adding 0.5 wt% of sodium lignosulphonate as dispersant, the water/solid ratios were further reduced from 0.55 to 0.45.

The pastes were then moulded in steel molds (40 × 40 × 40 mm) and covered by plastic films and then cured at 20 ± 1 °C for 24 h. The binders were then demoulded and sealed in polyethylene zip-lock bags and cured again under the same condition. Afterwards, the compressive strengths of the binders cured for 3, 7 and 28 d were measured using a YAW-300 automatic compression testing machine (Kent mechanical & electrical Co., China). Each set of binders had triplicates. The formulation designs of one-part geopolymers are shown in Table 2, and the experimental designs of the effect of water/solid ratio on the compressive strength are presented in Table 3.

In addition, control tests without alkali–thermal activation were also carried out. The same dosages of NaOH pellet were added after the RM sample was directly calcined at 800 °C, and then mixed with 0, 10, 20 wt% of SF. But the binders prepared from the mixtures at a water/solid ratio of 0.65 were not able to harden, and showed no enough strength for demould.

2.4. Leaching test

Leaching tests for the one-part geopolymer binders prepared from RM-10N with different SF addition and water/solid ratio were performed to study the transformation of pH. The binders were crushed into particles with a size lower than 2.38 mm, and then leached in deionized water at a liquid/solid (L/S) ratio of 20:1 for 18 h. The pHs of the leaching solutions were then measured.

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