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Investigation on the microstructure and mechanism of geopolymer with different proportion of quartz and K-feldspar

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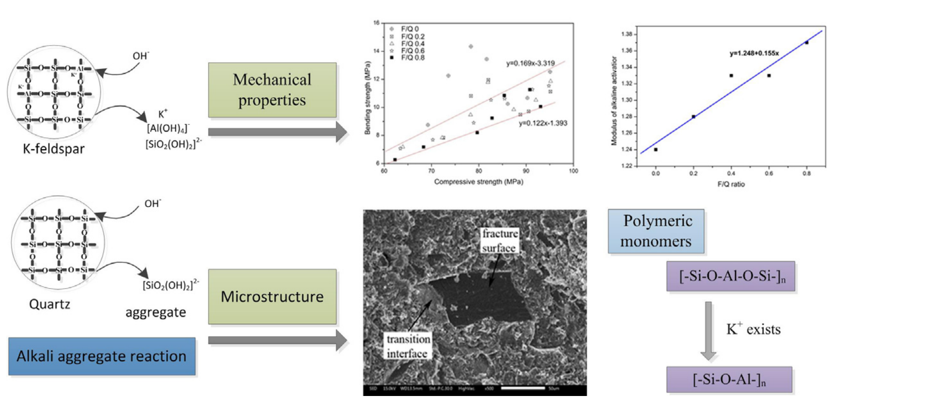
HIGHLIGHTS

- Effect of the aggregate contents of quartz and K-feldspar on the compressive and bending strength is studied.
- Determine the optimum modulus of alkaline activator at different proportions of quartz and K-feldspar.
- Microstructure and microanalysis of the region between the aggregate and matrix are studied.

GRAPHICAL ABSTRACT



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ABSTRACT

Geopolymer appears to be an alternative to ordinary Portland cement as a new sustainable material. The effect of the aggregate contents of quartz and K-feldspar on the microstructure and mechanical properties of Geopolymer samples was investigated in the present study. It was found that the aggregate contents influence the compressive strength and bending strength of geopolymeric bricks. Also, The K-feldspar/quartz mass ratio (F/Q) has a linear relation with the modulus of alkaline activator at the peak value of bending strength. This is due to the fact that the quartz reacts with the sodium hydroxide and forms $[H_2SiO_4]^{2-}$ which is the main component of sodium silicate, and that the K-feldspar has the ability to provide additional alkaline. The results of electron-microprobe analysis indicated that the matrix phase is composed of more amount of $[—Si—O—Al—O—Si—]_n$ polymerization monomer with increasing alkaline activator modulus ratio when K^+ is not involved in the reaction. However, the polymerization monomer of $[—Si—O—Al—]_n$ was only favored despite of variation of alkaline activator modulus when the K^+ exists.

1. Introduction

Ordinary Portland cement (OPC) is widely used in construction materials in the world, which, however, has high emissions of carbon dioxide (CO_2) ranging from 0.66 to 0.82 kg per kilogram of OPC manufactured [1–4]. The main causes of CO_2 emissions can be attributed to: (i) the calcination of the key ingredients, limestone;

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and (ii) the high energy consumption by heating raw materials in a rotating kiln at above 1400 °C [2]. Besides, significant expansion and cracking in concrete structures caused by alkali aggregate reaction (AAR) is one of the main durability problems when OPC is used in concrete [5].

Disposal of piles of tailings has been a worldwide problem. In recent years, with the further continuing exploitation of mineral resources, especially for the mining of low-grade mineral resources, the amount of tailings rose sharply, which became a big threat on the ecological environment. Using feldspar and quartz rich tailings as aggregate to produce building materials can reduce the production cost of building materials and meanwhile also consume piles of tailings, which realize the comprehensive utilization of resources. The contents of feldspar and quartz in tailings vary in a large range, and the effect of different contents of aggregate of feldspar and quartz on the properties of building material is still unknown.

As a new sustainable material, geopolymer can provide an alternative to OPC, which is formed by alkali-activated aluminosilicate materials like metakaolin, coal fly ash, and metallurgical slag after curing at ambient temperature or temperature between 60 and 120 °C [6]. Due to the avoidance of calcining limestone and burning fossil fuels, using geopolymer instead of cements reduce the CO_2 emissions greatly [1,2]. Although the effects of several parameters, such as aluminosilicate raw material [8–12], curing condition [13–16], ratio of reactant[17–19], reaction kinetics and mechanisms [20–24], and the composition of polymer-geopolymer [25–28], on the properties of geopolymer have been studied over the last several years, there's almost no researches reported about the role of aggregate species and contents in geopolymer. It's still not clear how aggregate behave in the concentrate alkaline paste and influence the macroscopical properties of the geopolymer. In this study the effect of aggregate content of quartz and K-feldspar on the mechanical and microstructural properties of the alkali-activated paste is investigated. In addition, this study is of great significance for the utilization of feldspar quartz rich tailings as the aggregate of geopolymer.

2. Experimental

2.1. Materials

The aggregate used in geopolymer was usually natural rocks like granite and quartzite which mainly consist of silicate and/or aluminosilicate minerals with stable chemical properties, corrosion resistance and high hardness [29]. In order to make certain the aggregate has enough large superficial area to react with the matrix completely, quartz sand and K-feldspar sand were selected and ground finely to a particle size less than 100 μm (Fig. 1). The surface area of the fines of quartz and K-feldspar sands were 15.066 m^2/g and 19.585 m^2/g, respectively. The aggregate used in this study are comprised of quartz and K-feldspar sands, with the mass fraction content of quartz ranging from 20% to 100%. Metakaolin, an aluminosilicate raw material, was obtained by the calcination of kaolinite at 750 °C for 4 hours in a muffle. There is no distinct diffraction peak in the X-ray diffraction pattern of metakaolin except the hump between 20 and 37 degree (Fig. 2), which indicates an amorphous state of metakaolin. In order to demonstrate an apparent effect of aggregate on mechanical properties, the solid mixture was obtained by the mixing of aggregates and metakaolin with a mass ratio of 3 according to our previous experience. The chemical compositions of metakaolin, quartz and K-feldspar determined by X-ray Fluorescence (XRF) are listed in Table 1.

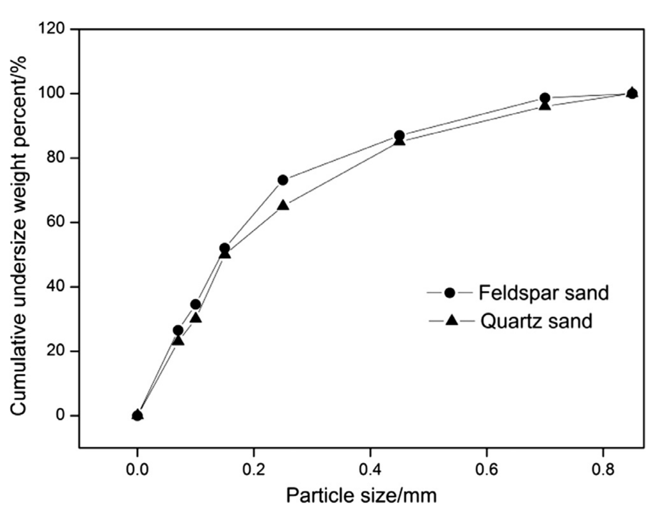

Fig. 1. Particle size distribution of the K-feldspar and quartz sands.

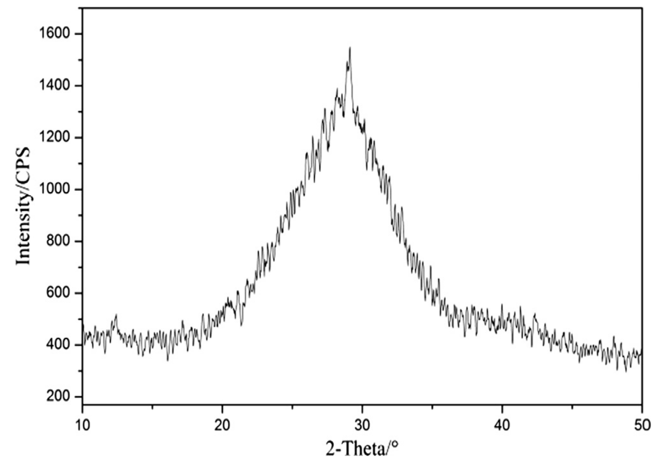

Fig. 2. X-ray diffraction pattern of the metakaolin.

Alkaline activators used in geopolymers were consisted of sodium hydroxide (95% of purity, Beijing Chemical Reagent Ltd. China) and water glass (Modulus = 2.46, $w(Na_2O)$ = 15.17 and $w(SiO_2)$ = 36.22 by mass, Baume degree 50°, Beijing Chemical Reagent Ltd. China). Distilled water was used to dissolve the sodium hydroxide solid to avoid the unknown contaminants in the mixing water [29]. The mixed sodium hydroxide solution was prepared 24 h before addition to solid aluminosilicates. Alkaline activators were added to solid mixture with a L/S (liquid to solid mass ratio) of 0.36. Previous study showed that the modulus ratio of alkaline activators solution between 1.2 and 1.4 lead to high bending and compressive strength of geopolymer [7]. Therefore, the modulus of alkaline activator solution in this study was set to 1.13–1.42.

2.2. Preparation

Metakaolin and aggregate were mixed in a cement mixer for 3 min, followed by the addition of mixed alkaline activators. The ratio of metakaolin (g)/solid powder (g) and alkaline activator (g)/solid powder (g) were 0.25 and 0.36, respectively. The resulting paste was transferred to a stainless steel mould, and then was pressed under an isostatic pressure of 10 MPa for 5 min. After being removed from the mould, the sample was dried at 80 °C in an oven for setting for 24 h, and then maintained in atmosphere for 27 days.

2.3. Methods

The compression test was performed on the cylinder samples with diameter of 40 mm and height of 100 mm, and the bending strength was determined on cuboid samples with size of 40 mm × 40 mm × 160 mm. The failure load of bending test was determined by the load-distortion curve obtained from three-point bending test using an electronic universal testing machine. The standars for measuring the compressive strength and bending strength is based on the Chinese standar *GB28635-2012/ Concrete paving bricks*. The microstructure of sample was studied by the scanning electron microscope (SEM, Hitachi, S2300), and polarizing microscope analysis (POM, caikon, XP400), respectively. Chemical composition was measured by electron probe microanalysis (EPMA, JXR-8230) with beam spot size of 5 μm at 50 nA and 20 kV.

3. Results

3.1. Compressive strength

Compressive strength of the as-prepared geopolymeric bricks is shown in Fig. 3. Note that samples using alkaline activators solution with a modulus ratio of 1.18 and aggregate with K-feldspar/