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Research paper

Novel method for preparation of calcined kaolin intercalation compound-based geopolymer

Lu Zeng*, Dan-yang Cao, Yan Xu, Chun-wei Fan, Xiao-qin Peng

College of Materials Science and Engineering, Chongqing University, Chongqing 400044, China

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ABSTRACT

This paper introduces a novel method for the preparation of a geopolymer using kaolinite intercalation compound which is calcined at a low temperature and forms a geopolymer by alkaline activation. The samples were characterized by X-ray diffraction (XRD), Thermogravimetric/Differential scanning calorimetry (TG/DSC) and Fourier transformed infrared spectroscopy (FTIR). Coal-bearing strata kaolin (CSK) mainly consisting of kaolinite and quartz was used to prepare the intercalation compound (CSK-K) with potassium acetate (KAc) and calcined below 400 °C. The calcination temperature and the dosage of KAc were analyzed. The results showed that CSK calcined below 400 °C cannot be alkali-activated to harden and when the dosage of KAc was 30% by mass of CSK, CSK-K calcined between 350 °C and 400 °C can be alkali-activated to form the geopolymer with a high compressive strength around 31 MPa. This was attributed to the formation of metakaolin and geopolymerization which were related to the effect of intercalation reaction and the content of K⁺. CSK-K calcined at low temperature has great potential as a novel material for the manufacture of geopolymers.

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1. Introduction

The term geopolymer, which is a class of binder materials formed by the activation of aluminosilicate materials with alkaline or alkaline-silicate solutions, was first introduced and named by Davidovits (1976). Accordingly, a geopolymer consists of a polymeric silicon–oxygen–aluminum framework with alternating silicon and aluminum tetrahedra joined together in three directions by sharing the oxygen atoms. Alkali cations (typically Na⁺ or K⁺) are associated with the AlO₄⁻ groups as charge balancers (Davidovits, 1976; Alvarez-Ayuso et al., 2008). According to the electron diffraction analysis conducted by Van Jaarsveld et al. (1999), the structure of geopolymers is amorphous to semi-amorphous. A study of the alkali cation effect (Na⁺ or K⁺) on the mechanical properties of geopolymer concluded that K⁺ promotes a greater degree of geopolymerization and a higher degree of Al incorporation when compared with sodium systems, and geopolymers with high Si/Al ratio and mixed-alkali cations exhibit significant increases in strength (Duxson et al., 2005). Researches on geopolymers have been carried out for several decades. In recent years, geopolymer materials are of scientific interest owing to their low environmental impact, high early strength, thermal stability, and excellent durability (Davidovits, 1989; Lyon et al., 1997; Nowak, 2008; Shi et al., 2011). At present, the usual aluminosilicate source for the preparation of a geopolymer is metakaolin transformed from the

calcined kaolin (Zhang et al., 2007; Zuhua et al., 2009; Burciaga-Diaz et al., 2012). Coal-bearing strata kaolin (CSK) is a kaolin rock associated with coal, and its main mineral component is kaolinite. Kaolinite is a dioctahedral phyllosilicate formed by the superposition of silicon tetrahedral sheets and aluminum octahedral sheets (Brindley, 1951; Brigatti et al., 2006). Adjacent layers are linked by hydrogen bonds or van der Waals forces and can be intercalated by some organic and inorganic molecules to enhance the usability (Hui, 2004). Through intercalation, the properties of kaolinite can be improved such as lipophilicity, dispersibility, specific surface area, and thermal stability (Sidheswaran et al., 1987; Tsunematsu and Tateyama, 1999; Orzechowski et al., 2006; Zeng et al., 2012). An investigation by White et al. (2010) studied the effect of temperature on the structure of kaolinite intercalated with potassium acetate (KAc), which showed that the intercalated compound transformed into an amorphous state at a lower temperature than the nonintercalated kaolinite counterpart.

Calcined CSK is generally used as the raw material to manufacture the geopolymers (Li et al., 2010; Lyu et al., 2013; Liu et al., 2014), and the calcination temperature for kaolinite is usually between 600 °C and 900 °C (Wang et al., 2010; Elimbi et al., 2011; Rashad, 2013). However, there are few reports regarding the CSK intercalation compound calcined at lower temperatures for the manufacture of the geopolymer, and the characterization of the geopolymer has not been reported correspondingly. In this paper, the CSK intercalation compound with KAc was prepared and calcined below 400 °C to prepare the geopolymer. The lowest calcination temperature and the dosage of KAc were investigated to prepare the geopolymer

* Corresponding author. Tel./fax: +86 23 65127940.
E-mail address: zool@foxmail.com (L. Zeng).

with the best strength. Several methods were used to characterize samples and explain the mechanism of geopolymer preparation in a low calcination temperature.

2. Experimental

2.1. Preparation of CSK intercalation compound

CSK supplied by Hengyu Company was used as a precursor material for intercalation compound with KAc (CSK-K), which consists of kaolinite and a small amount of quartz, according to powder X-ray diffraction (XRD) analysis (Fig. 1a) and its chemical composition (Table 1). For preparation of CSK-K, 1 kg of CSK was mixed with 0.2, 0.3, and 0.4 kg of KAc (AR, Aladdin Co. Ltd), respectively, and then 10% deionized water was added to each solid mixture by mass. The mixture was stirred in a mortar mixer (Xiwei, JJ-5, China) for 2 h, aged for 12 h, and then dried to constant mass in an oven at 105 °C, denoting each corresponding sample as CSK-K2, CSK-K3, and CSK-K4.

2.2. Preparation of the geopolymer

To prepare the geopolymer, CSK-K was calcined between 300 and 400 °C for 4 h and then milled to less than 45 μm after natural

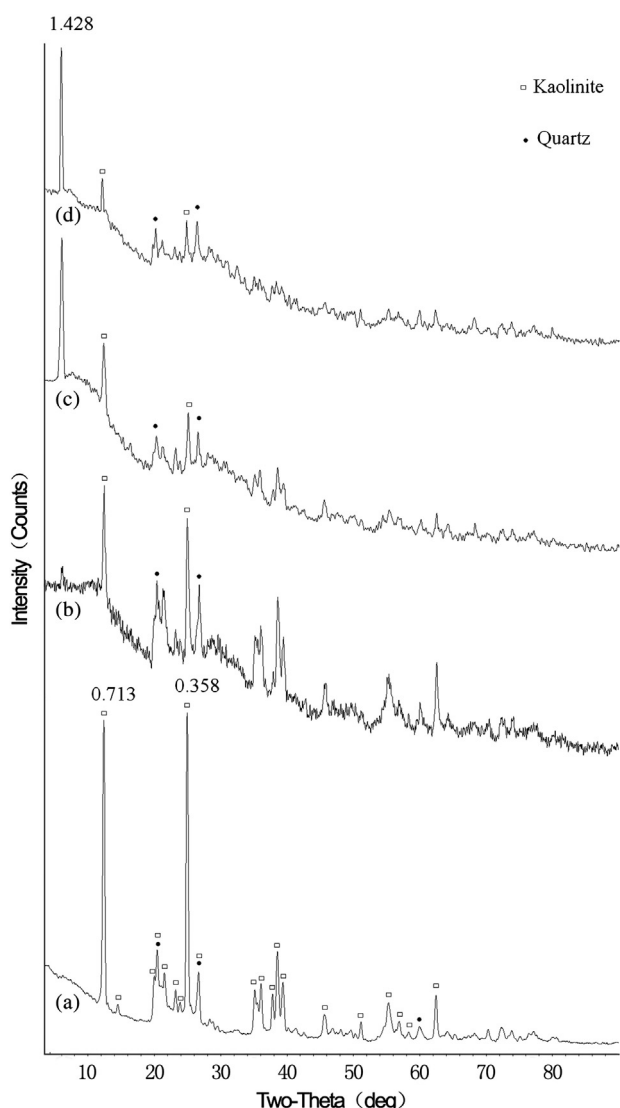


Fig. 1. XRD patterns of CSK (a), CSK-K2 (b), CSK-K3 (c) and CSK-K4 (d).

Table 1
Chemical composition of CSK (wt.%).

Al ₂ O ₃	SiO ₂	Fe ₂ O ₃	TiO ₂	CaO	MgO	Na ₂ O	P ₂ O ₅	Loss
37.34	46.96	0.31	0.43	0.14	0.09	0.07	0.04	14.62

cooling. Sodium silicate solution (chemical composition in wt.% is SiO₂ 31.55, Na₂O 13.45, H₂O 56.0, Hongjun Co. Ltd), NaOH (AR, Aladdin Co. Ltd), and water were mixed in a beaker and then cooled down to room temperature. The prepared solution was added to the calcined CSK-K, mixed for 5 min, and then poured into cubic steel molds of 40 mm × 40 mm × 40 mm, which were vibrated on a vibration table for 2 min to remove any air bubbles. The specimens were covered with polyethylene film to prevent evaporation. The molds were removed after 24 h and the specimens were cured under conditions of 20 °C and 95% relative humidity for 28 days. The geopolymer prepared in this study had a designed composition with molar ratios of SiO₂/Al₂O₃ = 4.0, Na₂O/Al₂O₃ = 1.0, and H₂O/Na₂O = 6.0, as described previously (Davidovits, 1988; Zhang et al., 2010; Burciaga-Diaz et al., 2012).

2.3. Methods of analysis

2.3.1. Compressive strength of the geopolymer

CSK, CSK-K2, CSK-K3, and CSK-K4 calcined at 300 °C, 350 °C, and 400 °C were used to prepare the geopolymer, and the corresponding samples were denoted as GT-CSK, GT-CSK-K2, GT-CSK-K3, and GT-CSK-K4 (T denotes 300, 350, and 400). Compressive strength of the samples was tested (four cubes were tested and the average was obtained) at given ages of 28 days on an auto-compressive resistant tester (Hualong, WHY-200, China).

2.3.2. Characterization

Some chosen samples were dried, milled, and then characterized by several methods, including XRD analysis, Thermogravimetric/Differential scanning calorimetry (TG/DSC) analysis and Fourier transformed infrared spectroscopy (FTIR) analysis. XRD was performed on a Rigaku D/max-3Bx diffractometer with Cu Kα radiation, in the range of 3–90° 2θ and at a step size of 0.02°. TG/DSC analysis was performed on a thermal analyzer (STA449C), from 25 °C (room temperature) to 1200 °C with a heating rate of 5 °C/min. Infrared spectra were recorded by means of a Thermo-Electron FTIR spectrometer (Avatar370) in the range of 4000–400 cm⁻¹.

3. Results and discussion

3.1. Compressive strength of the geopolymer

The compressive strength of the geopolymer as a function of calcination temperature and dosage of KAc are presented in Fig. 2. It denotes

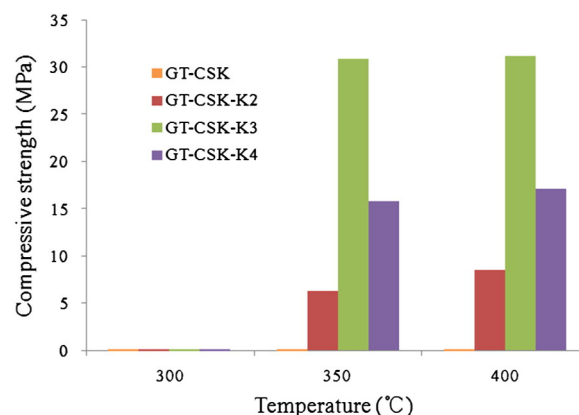


Fig. 2. Compressive strength of geopolymer.

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