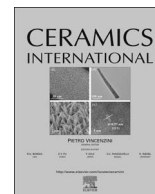




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Role of alkali cation in compressive strength of metakaolin based geopolymers

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ABSTRACT

In this work, we carried out comprehensive studies on the role of alkali cations in the geopolymerization reaction and the subsequent network formation which affected the compressive strength of metakaolin (MK) based geopolymers. Rheological studies were used to provide real-time information for the geopolymerization from the initial stage. It was found that the difference in dissolution tendency between Na⁺ and K⁺ resulted in different morphologies, which affected the compressive strength. The compressive strength of the systems using Na⁺ was found to be higher than those using K⁺ under the same Si/Al ratio and alkali cation concentration. The highest compressive strength (~40.1 MPa) was obtained from a system using 12 M of Na⁺ with Si/Al ratio of 1.9:1 where a continuous monolithic microstructure was observed. Pycnometer was employed for the first time to gain an insight into the factors responsible for the compressive strength when similar morphologies were observed. The observation obtained from pycnometer compensates for the lack of visual evidence seen from similar morphologies and can serve as additional evidence for the different compressive strengths of MK based geopolymers.

1. Introduction

Geopolymer, firstly named by Joseph Davidovits [1], is a synthetic alkali aluminosilicate material formed by alkali activation of aluminosilicates. It is generally prepared by activation of source materials, such as metakaolin (MK), fly ash or slag, using solutions of hydroxides or silicates of sodium or potassium [2,3]. After it first developed as an alternative to thermosetting polymer for fire resistant applications, the development of geopolymer was mainly concentrated on the field of civil engineering [4] as a potential replacement material for traditional cementitious binder owing to its strong mechanical properties with the additional advantage of significantly reduced greenhouse gas emissions during production [5,6].

The compressive strength of geopolymers, a common indicator of the success of a geopolymerization process [7] have been attracted attention from civil engineers and researchers. It was highly dependent on Si/Al ratio [8,9], hydroxide concentration [8], alkali cation used [10], and curing condition [11,12]. For example, the compressive strength of MK based geopolymers was improved by using sodium or potassium silicate solution instead of hydroxide [13,14]. This improvement is attributed to the presence of increased number of Si–O–Si

bonds, which possess higher bond energy compared to Si–O–Al bond. Sodium cation (Na⁺) and potassium cation (K⁺) are the two most widely used cations in alkaline activators. It was reported that geopolymers made with K⁺ possessed a higher compressive strength [10,15,16]. For example, Xu et al. [15] reported that the presence of K⁺ increased the extent of disorder in the formed gel phase and increased the compressive strength of geopolymers. However, the mechanisms proposed were based on geopolymeric gels and might not be a proper representation of the solidified sample. P. Duxson et al. [13] studied the compressive strength of MK based geopolymers containing M⁺ (M: K and Na) with various Si/Al ratios. Inconsistency in the compressive strength was observed throughout various Si/Al ratios and the underlying mechanisms were inadequately discussed. The study on MK-slag-clinker blends by Fernández-Jiménez et al. [17] suggested that Na⁺ favored coagulation/precipitation more effectively than K⁺. The sample made with Na⁺ exhibited higher compressive strength than that made with K⁺ when moderate alkaline activators, such as carbonates were introduced. Recently, Narayanan et al. [18] reported that systems made with Na⁺ demonstrated higher compressive strength than the ones with K⁺. The large amount of pores observed in geopolymers made with K⁺ was considered to be the detrimental factor leading to inferior

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compressive strength.

Based on the literature review, it appears that there exists some confusion regarding the effects of alkali cations on the compressive strength of geopolymers. The controversy of previous studies indicated that these research outcomes might be case specific, which prompted us to explore the roles played by different M^+ responsible for the underlying mechanisms of geopolymerization as well as compressive strength. In this work, MK was selected as a source material owing to its high purity and easier control of Si/Al ratio [19]. The contributions of the Si/Al ratio and the M^+ concentration were considered simultaneously, even though they were always discussed separately in previous works [20,21]. Rheological study is a common approach applied in polymers and was performed to address the geopolymerization resulting from different M^+ , and then correlated with compressive strength. Scanning electron microscope (SEM) was used to examine the morphologies obtained. Quantified gas pycnometer was employed for the first time to characterize the different networks formed under different M^+ concentrations, which is a critical factor affecting the compressive strength of the geopolymers.

2. Experimental

2.1. Raw materials

MK of particle size approximately 1 μm was obtained from BASF (Metamax[®]) with molar ratio Si:Al \approx 1. The chemical composition of MK is provided in Table 1. The specific density of MK is 2.6 g/cm³. Sodium hydroxide, potassium hydroxide and sodium silicate solution were purchased from Sigma-Aldrich. The composition of sodium silicate solution is 10.6 wt% Na₂O, 26.5 wt% SiO₂, and 62.9 wt% H₂O. Potassium silicate was purchased from International Laboratory USA. The content of K₂O in potassium silicate is 47–51 wt%. All the materials and chemicals were used as received.

2.2. Sample preparation for compression tests

Molar ratio of Si:Al: M^+ was used to effectively study the role of alkali cations. In this work, the molar ratio of Al: M^+ was kept constant as 1:1 throughout the entire study in order to maintain the charge balance, i.e., M^+ balances the negative framework charge carried by tetrahedral Al [2,22]. Two groups of samples were studied. In group 1, the M^+ concentration was fixed at 8 M, the molar ratio of Si:Al: M^+ was changed from 1:1:1 to 1.9:1:1 by adding certain amounts of alkaline silicate solution (calculated based on the chemical composition of source materials) to alkaline hydroxide solution. In group 2, the M^+ concentration was increased to 12 M, the molar ratio of Si:Al: M^+ was changed from 1:1:1 to 1.9:1:1. In the case of Si:Al: M^+ =1:1:1, in order to keep the molar ratio constant, the concentration of M^+ was increased from 8 to 12 M through reducing the water content in the system. In the case of Si:Al: M^+ = 1.9:1:1, in addition to the reduction in water content, alkaline silicate solution was added proportionally based on the chemical composition of source materials.

MK was mixed with the different alkaline activators (pH=14) in a Vortex Mixer for 2 min, followed by mechanical stirring in ice bath for 5 min and finally mixed in a Vortex Mixer for 2 min. The slurries were poured into a cylindrical Teflon mold with the dimension of $\Phi 25 \times 50$ mm (ASTM C39-96) and subjected to degassing, followed by progressive curing at 40 °C for 2 h, 60 °C for 24 h and room temperature for 6 days. The chemical compositions of different samples were

Table 1
Chemical composition of MK.

Component	SiO ₂	Al ₂ O ₃	Na ₂ O	K ₂ O	TiO ₂	Fe ₂ O ₃	CaO	MgO	P ₂ O ₅	SO ₃
wt%	53.0	43.8	0.23	0.19	1.70	0.43	0.02	0.03	0.03	0.03

Table 2

Comparison of the compressive strength (MPa) of different geopolymers.

Concentration of M^+	Molar ratio of Si/Al	Compressive strength (MPa)	
		GeoNa	GeoK
8 M	1:1	3.9 \pm 0.5	1.0 \pm 0.3
	1.9:1	34.2 \pm 4.3	7.3 \pm 2.1
12 M	1:1	^a	4.2 \pm 0.9
	1.9:1	40.1 \pm 3.5	15.4 \pm 2.4

^a The samples cannot be prepared due to the high viscosity.

summarized in Table 2. The geopolymer systems made with Na⁺ and K⁺ were labelled as GeoNa and GeoK, respectively.

2.3. Characterizations

Compression tests were carried out using an Instron 8516 with a loading rate of 1.3 mm/min according to ASTM C39-96. The load cell was 100 kN. The ultimate yield strength was reported as the compressive strength. All the results presented were based on the average of five tests.

The rheological behaviors were measured at room temperature using a Discovery Hybrid Rheometer (DHR) from TA Instruments, equipped with cone and plate (stainless steel, 25 mm diameter). Oscillatory experiments were performed with 2 ml of geopolymer slurry, at a constant frequency ω of 1 Hz and a constant strain of 1%. According to the Winter-Chambon [23] criterion, the gel point is defined as the point at which the modulus and frequency obeys a scaling law of $G'(\omega)=G''(\omega)$, namely the crossover point of G' (dynamic elastic modulus or storage modulus) and G'' (dynamic viscous modulus or loss modulus). The time taken to reach the gel point is defined as the gel time. The linear viscoelastic region was determined prior to the rheological measurements to ensure accurate evaluation.

The fracture surface of geopolymers was examined by Field Emission Scanning Electron Microscope (FESEM) JEOL 7600 without further polishing. The specimens were coated with platinum prior to the observation.

The void volume formed within the specimen networks was measured using a Quantachrome Ultra Foam Gas Pycnometer (Model: 1200e). The helium (He) gas purge pressure was kept at 145 KPa (1.43 atm). All the specimens were dried at 40 °C for 24 h prior to the measurements.

3. Results and discussion

A systematic study on the compressive strength of the geopolymers was conducted. Table 2 lists the compressive strength of GeoNa and GeoK with various Si/Al ratios and M^+ concentrations. The results showed that under the same Si/Al ratio, the compressive strength of the geopolymers increased when the M^+ concentration increased from 8 to 12 M, except for the sample containing Na⁺ which could not be prepared due to the poor workability. More interestingly, at 8 M M^+ concentration, the compressive strength of GeoNa was found to be higher than that of GeoK under the same Si/Al ratio. Furthermore, it can also be seen that the compressive strength increased with the increase of Si/Al ratio from 1:1 to 1.9:1 under the same M^+ concentration.

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