Cement and Concrete Research 100 (2017) 14–22

Contents lists available at ScienceDirect

Cement and Concrete Research

journal homepage: www.elsevier.com/locate/cemconres

Hot-pressed geopolymer

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ARTICLE INFO

Keywords:
Hot-pressing
D. Alkali activated cement
D. Fly ash
A. Acceleration
B. Characterization

ABSTRACT

This research explores the use of simultaneous heating and pressing techniques in order to enhance the mechanical properties of fly ash (FA) based geopolymer under relatively low temperature conditions to ensure minimum-porosity. Four effective parameters of pressing force, alkali activator/FA, duration of hot-pressing and sodium concentration are studied. Together with detailed experimental studies, our results reveal that the most dominant factor is the induced pressure. The main results indicated that the highest compressive strength of the geopolymer (134 MPa) could be obtained by employing the hot pressing, temperature and duration of 41.4 MPa, 350 °C and 20 min, respectively. The microstructure of the hot-pressed specimens showed more developed geopolymer matrix compared with conventional ones leading to higher compressive strength in much shortest time. The improved mechanical properties are generally attributed to the dense structure of the material and higher geopolymer gel production during the hot pressing process. However, further reaction of partially reacted particles improves the mechanical properties over time.

1. Introduction

Precast concrete construction has several advantages compared to the in situ concrete regarding quality, fast assembling, safety, and reduction in manpower demand [1]. However, the importance of the space availability and material supply according to work schedule is highlighted more in precast construction since the concept of just-in-time construction is a key consideration [2]. In view of the fact that numerous precast members need to be fabricated in a short time, satisfying the design code specifications and being able to withstand the lifting loads, a fast curing method for concrete is required [3]. Among different curing methods of concrete, steam curing, electrical curing, and heat supply curing are classified as accelerated curing methods which are based on the increase of the curing temperature of the materials [4–6]. Steam curing is one of the most common methods in the fast development of the Portland cement-based composites, increasing the strength to about 70% of the ultimate strength in one day [3,7]. This strength is almost enough for lifting, safe transportation, and fast installation, however, this method of curing consumes a considerable amount of energy.

Geopolymers are considered as alternatives to Portland cement due to their superior properties of low carbon dioxide release, high early strength (at the present of heat) and elevated temperature resistance [8,9]. Several parameters have been studied to enhance the kinetics of geopolymerization and their consequent setting and mechanical properties such as the role of silicate, aluminate, sodium, water, and curing temperatures [4,10–14]. Curing of the geopolymers is possible in a wide range of temperatures. However, most of the previous studies have focused on the lower temperatures which are more applicable to conventional construction. These conventional methods of curing are limited to the ambient and oven curing. It has been known that the increase in curing temperature has a significant effect on kinetics of FA-based geopolymers by increasing both dissolution and polycondensation rates at high temperature and the consequent fast setting of the material [15,16]. However, rapid setting results in an increase in the pore volume, preventing the mixture from forming a compact structure; this mechanism leads to a decrease in the ultimate compressive strength of the geopolymer. Whereas, if the geopolymer is cured at ambient temperatures, the material gradually fills some of the pore fractions and thus forming a denser matrix [5,17]. Although the ultimate strength reduction is not preferable, the high early strength at elevated temperatures (mostly 45–85 °C) makes the geopolymers suitable for precast construction [18]. To speed up the rate of hardening, microwave curing has been used to provide a uniform and fast heating. This phenomenon is owed to the interaction between the polar molecules and microwave electric fields. Such a superfast efficient microwave curing leads to the formation of a high strength geopolymer (100 MPa), which may be heated up to 1000 °C without cracking, failure or

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http://dx.doi.org/10.1016/j.cemconres.2017.05.010
Received 28 October 2016; Received in revised form 22 April 2017; Accepted 9 May 2017

vitrification [19,20]. Although this method of curing has promising applications to geopolymers, this technology may have some drawbacks in practice. A preferred method would be the use of already available equipment for precast construction.

Herein, we demonstrate the construction of an integrated high strength and ultra-fast hardening FA-based geopolymer through hot-pressing method. The geopolymer specimens are subjected to heat treatment at a fixed temperature of 350 °C together with pressure. The effects of changes in curing pressure, curing duration, alkali activator/FA and sodium concentration on the performance of hot pressed FA-based geopolymer are studied. The initial and long term compressive strength, hardness and fracture toughness of this FA-based geopolymer are evaluated. To characterize the microstructure and kinetic of the reaction, thermogravimetric analysis (TGA), differential scanning calorimetry (DSC) analysis, X-ray diffraction analysis (XRD), Fourier transform infrared spectroscopy (FTIR), scanning electron microscopy (SEM) and density measurement are conducted. The strategy of using hot-pressing offers a new path to development of high performance and integrated geopolymer composites.

2. Materials and methods

2.1. Row materials characterization

In this study, a low calcium (class F) FA collected from Lafarge Malayan Cement Bhd (Malaysia) was used to prepare the geopolymer paste. This FA was a waste product of coal power stations and consists of typical spherical particles. The chemical composition of the FA was analyzed by X-ray florescence (XRF) using a PANalytical Axios mAX instrument as it is shown in Table 1. The median particle size and specific gravity of the FA were indicated to be 12.19 μm and 2.18 g/cm3, respectively.

A mixture of sodium silicate and sodium hydroxide has been prepared with mass ratio of 2.5:1.0 to activate the aluminosilicate particles. The sodium silicate was used in liquid form with about 1.5 g water per milliliter at 20 °C with a SiO_2/Na_2O mass ratio of 2.5, obtained from PC Laboratory Reagent (West Malaysia). The NaOH pellets were provided by Merck (Germany).

2.2. Geopolymer specimen preparation using a hot press

The geopolymer specimens were fabricated by mixing as-received FA with alkali activator for 5 min, poured into a steel pressing mold and hot-pressed using a Mount Press and a mold heater (Buehler Ltd., USA). The heating temperature was fixed at 350 °C for all the specimens. This temperature was chosen due to the reported positive effects on mechanical properties of FA-based geopolymers [16,21]. Following the hot-pressing treatment, the heater was removed and the mold cooled down by a cooler in 5 min. Finally, the specimens were removed and kept in an ambient environment until the testing time. Four sets of specimens are prepared to evaluate the influence of induced pressure (13.8 to 41.4 MPa), hot pressing duration (15, 20, 25 and 30 min), alkali activator/FA mass ratio (0.25, 0.30, 0.35 and 0.40) and sodium concentration (8 to 16 M) on the compressive strength and microstructure performance of the hot-pressed FA-based geopolymer. To evaluate the effects of hot pressing, the characteristics of hot-pressed geopolymers are compared with those of the corresponding geopolymers prepared by the same materials employing a typical curing method (after mixing and vibration, the specimens were placed at 65 °C for 24 h and further cured at room temperature up to the time of testing) as described in our previous study [22]. Noteworthy, to have a workable mix some amount of water is needed to be used in the conventional method to have a final alkali activator mass ratio of sodium silicate:NaOH:H_2O of 2.5:1.0:0.7. A summary of the specimen preparation is shown in Table 2.

2.3. Analysis methods

X-ray fluorescence (XRF- PANalytical Axios mAX, Netherlands) was used to determine the oxide composition of the FA. The XRD patterns were measured on an Empyrean PANALYTICAL diffractometer (Netherlands) with monochromated Cu Kα radiation (λ = 1.54056 Å), operated at 45 kV and 40 mA with a step size of 0.026° and a scanning rate of $0.1° s^{-1}$ in the 2θ range of 5 to 80°. FTIR spectroscopy was carried out using a Perkin Elmer System series 2000 (USA) spectrophotometer in a frequency range of 4000–400 cm^{-1} to identify the functional group of the geopolymer specimens. DSC (Mettler DSC 820, Switzerland) was applied to evaluate the kinetics of the geopolymer gel formation and hardening during the process of hot pressing over a temperature range of 30 to 350 °C with a rate of $10 °C{\cdot}min^{-1}$. TGA was conducted using a STA 449 F3 Jupiter thermogravimetric analyzer (NETZSCH, Germany) device with a heating rate of $10 °C{\cdot}min^{-1}$ from 30 °C to 350 °C. SEM-BSE images were taken using a Phenom ProX scanning electron microscope (Phenom-World Inc., Netherlands). The images were processed using multispectral analysis to do a supervised classification of pores and geopolymer mixture made on MultiSpec© (Purdue Research Foundation). The multispectral images were analyzed by imageJ software to quantify the pore, geopolymer gel, and unreacted particle fractions. Nanoindentation experiments were also performed using a nanomechanical test system (Micro Materials Ltd. Wrexham, U.K.) employing Berkovich diamond tip with a radius of 20 nm. The test was conducted using 100 mN in a load-controlled mode with a dwell time of 10 s and indentation velocity of $3 nms^{-1}$. To prepare the samples for nanoindentation test, the geopolymer specimens were ground using a fine silicon carbide paper (up to 2000 grit size) and polished to a mirror finish using diamond powders of different grades from 15 to 0.25 μm in an auto polisher (laboforce-3, Struers, Denmark). The minimum of ten indentations were applied to determine an average value for each sample. Based on the obtained load–displacement unloading curves, the Elastic modulus of each specimen was calculated using the Oliver–Pharr method [25]. The reduced modulus (E_r) is taken from the nanoindentation data and is related to the sample's elastic modulus (E_s). The following equation was used to calculate the sample's elastic modulus (E_s) [26]:

$$\frac{1-\nu_s^2}{E_s} = \frac{1}{E_r} - \frac{1-\nu_i^2}{E_i} \quad (1)$$

where the elastic modulus (E_i) and Poisson's ratio (ν_i) of the indenter are 1140 GPa and 0.07, respectively. The Poisson ratio of the sample (ν_s) is taken to be 0.2 since the effect of variation in Poisson's ratio in the range 0.18–0.22 has been reported to be insignificant for similar systems [27–30].

The fracture toughness was determined by the following equation [31].

$$K_{IC} = 0.016\left(,\frac{E}{H}\right)^{1/2}\left(\frac{P}{C^{2/3}}\right) \quad (2)$$

where, K_{IC} is the indentation toughness (MPa $m^{1/2}$) signifying fracture

Table 1
Chemical composition of FA.

Composition	SiO_2	Al_2O_3	Fe_2O_3	K_2O	TiO_2	CaO	SO_3	MgO	P_2O_5	Na_2O	ZrO_2	MnO	LOI
FA (%)	75.76	15.86	3.90	1.14	0.97	0.95	0.35	0.26	0.21	0.16	0.13	0.06	6.8