Construction and Building Materials 107 (2016) 95–102

Contents lists available at ScienceDirect

Construction and Building Materials

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

Mechanical and micromechanical properties of alkali activated fly-ash cement based on nano-indentation

Hyuk Lee [a,*], Vanissorn Vimonsatit [a], Prinya Chindaprasirt [b]

[a] Department of Civil Engineering, Curtin University, WA, Australia
[b] Sustainability Infrastructure Development and Research Centre, Department of Civil Engineering, Faculty of Engineering, Khon Kaen University, Thailand

HIGHLIGHTS

- Mix design using Taguchi's method was presented to determine critical factors.
- Silica fume has the most adverse impact on the compressive strength.
- Using deconvolution technique confirms four phases of reaction products.
- High strength is achieved N-A-S-H phase greater than 40% of total volume fraction.
- Activation degree is recommended to be 60% to achieve high strength mixture.

ARTICLE INFO

Article history:
Received 26 March 2015
Received in revised form 19 October 2015
Accepted 5 December 2015
Available online 5 January 2016

Keywords:
Alkali activated cement
Fly-ash based cement
Micromechanics
Mechanical properties
Nanoindentation

ABSTRACT

This paper presents mechanical and micromechanical properties of alkali activated fly-ash cement (AAFA) based on statistical analysis with nano-indentation test. Mix proportions of AAFA are designed using Taguchi's approach. Four variables viz, silica fume (SF), sand to cementitious material ratio (s/c), liquid to solid ratio (l/s) and superplasticiser (SP) content were the parameters tested. Indentation elastic modulus, hardness and packing density are studied. The results show that the increase in sand has the greatest contribution to the increase in density. For compressive strength, normal paste without SF, sand and SP with l/s of 0.6 gives the highest strength and the increase in SF significantly contributes to the adverse effect on compressive strength. For the indentation data, the analysis using deconvolution technique confirms the four phases of reaction products of AAFA. The main phase is sodium aluminosilicate hydrate (N-A-S-H), which is over 40% of the volume fraction. The microporomechanics of AAFA paste and mortar also demonstrate the relationships between the N-A-S-H volume fraction and strength; and activation degree and strength.

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1. Introduction

Alkali activated cement (AAC) is a potential cementitious system for sustainable development [1]. Its main constituent is pozzolan, which can react with alkali activator to form binder [2]. A number of researches [2–4] reported the difference between the composition of traditional Portland cement and fundamental rock forming minerals of the earth crust. The common chemical compositions of Portland cement and fundamental rock are silica and alumina which can also be found in a number of industrial wastes. Ground industrial wastes or by-products containing aluminosilicate when mixed with rich alkalis could form a hydraulic binder which is a type of inorganic polymer called 'geopolymer' or alkali activated cement (AAC). Davidovits [5] classified different types of geopolymer according to Si to Al ratio in the mixtures for various industry applications. Lloyd et al. [6] reported that calcium contents in geoploymer is important for alkali mobility that may be significant to limit the durability of embedded steel reinforcement. A precaution should be taken as some of ground industrial wastes or by-products such as those containing calcium aluminate and metakaolin geopolymer have loss of strength during long-term ageing [7]. Despite extensive research in this area, the entire polymerization process of AAC is not totally understood. It is classified as a polymer because of its huge molecule formed by a number of smaller groups of molecules. AAC has superior mechanical, chemical and thermal properties compared to ordinary Portland cement (OPC) [8]. The main benefit of AAC is that the source material is not a carbonate bearing material; therefore, it does not release vast

* Corresponding author.
E-mail addresses: leehyuk1982@gmail.com (H. Lee), V.Vimonsatit@curtin.edu.au (V. Vimonsatit), prinya@kku.ac.th (P. Chindaprasirt).

http://dx.doi.org/10.1016/j.conbuildmat.2015.12.013
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quantities of CO_2 as in the case of Portland cement. Turner et al. [9] reported that the carbon emission of AAC concrete is around 9% less than comparable OPC concrete as alkali activators are high carbon footprint materials. Also, high early age strength, high chemical durability and resistance to high temperature are beneficial properties of AAC.

Normally, cementitious materials have several phases that contribute to the mechanical properties. A number of researchers [10–13] studied the characterization of alkali-activated materials by a variety of experimental methods including XRD, SEM, DTA and TGA. Jennings et al. [14] and Tennis et al. [15] proposed a model for the determination of two types of calcium silicate hydrate (C-S-H) viz., high density (HD) and low density (LD) C-S-H, at different points of the specimens geometry. The content of C-S-H is determined in term of its volume fraction of the indentation grid. Constantinides et al. [16] determined the two C-S-H types, Portlandite (CH), and clinker using this nano-indentation method. The result shows that decalcification of C-S-H phases is the primary source of nanometer-scale elastic modulus degradation.

Recently, Němeček et al. [17] studied the reaction products of AAFA paste using NaOH solution with the liquid to solid ratio of 0.531 cured at 80 °C for 12 h. Nano-indentation and environmental scanning electron microscope (ESEM) were used, and four phases of reaction products were found. The four phases were identified as N-A-S-H (sodium aluminosilicate hydrate), partly-activated slag (N-A-S-H gel intermixed with slag-like particles), non-activated slag (porous non-activated slag-like particles), and non-activated compact glass (solid non-activated glass spheres or their relicts). N-A-S-H is the main reaction product which is linked to the atomic scale and nano-structure and is independent of precursor material or the temperature curing regime. N-A-S-H phase is pure and is related to the mechanical strength of AAFA matrix. The contents of Si ions in N-A-S-H can be increased by the presence of Si ions in the raw materials. It has been found that the increasing condensation degree of Si ions in N-A-S-H relates directly to the mechanical strength gain [11,18]. The partly-activated slag phase is intermixed with the slag-like particles. The non-activated slag phase is porous and contains non-activated slag-like particles. The non-activated compact glass phase is solid, non-activated glass sphere.

The aim of this research is to investigate the mechanical and micromechanical properties of AAFA paste and mortar. The experimental program is designed according to Taguchi's method [19], which is efficient for investigating optimum design parameters for the required performances. The investigated parameters are density and compressive strength of AAFA. Nano-indentation and statistical analysis of the test data are used for the evaluation of microporomechanics of AAFA samples. A background on nano-indentation and how to determine the indentation modulus, hardness, Poissons ratio, cohesion, friction coefficient and packing density of materials are presented in brief.

2. Nano-indentation

2.1. Principal nano-indentation

Nano-indentation test method is now well established that the response of material upon the reversal of touch loading provides access to the elastic properties of indentation materials [20]. The measurement of the hardness and elastic modulus of material can be obtained from the relationship between indentation loads (P) and depth (h) during loading and unloading [21–27]. Fig. 1 shows a typical indentation load-depth ($P-h$) curve. The important quantities that must be measured from the $P-h$ curve are the maximum indentation load (P_{max}), the maximum indentation

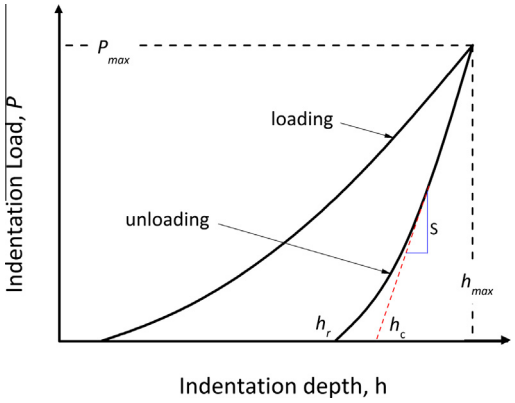

Fig. 1. Typical indentation load-depth ($P-h$) curve.

depth (h_{max}), and the elastic unloading stiffness ($S = dP/dh$), which is defined as the slope of the upper portion of unloading curve. The modulus of material can be estimated from the elastic unloading stiffness (S), and is generally called 'indentation modulus (M)', which is affected by the testing method. Nevertheless, an indentation test provides the rate of piling-up and sinking-in of the test surface around the indentation points, thus, ideally the indentation modulus (M) has the same meaning as the elastic modulus, or Youngs modulus, measured by other conventional test methods.

The contact mechanics between a rigid indenter and a non-rigid flat surface of a specimen can be written as [26]:

$$\frac{1}{M} = \frac{1-\nu^2}{E} + \frac{(1-\nu'^2)}{E'} \tag{1}$$

where E is the elastic modulus of the specimens, ν is the Poissons ratio, E' is the indenter elastic modulus and ν' is the indenter Poissons ratio. The analysis of indentation hardness and modulus of any axis-symmetric indenter can be estimated from Eq. (2).

$$H = \frac{P_{max}}{A_c} \quad M = \frac{\sqrt{\pi}}{2}\frac{S}{\sqrt{A_c}} \tag{2}$$

The indentation depth h_r and h_c correspond to the applied load; h_r is the depth of the residual impression and h_c is the contact depth. The contact depth h_c can be determined as:

$$h_c = h_{max} - \xi \cdot \frac{P_{max}}{S} \tag{3}$$

According to Oliver and Pharrs methodology [26], h_c can be determined by setting $\xi = 0.75$ for Berkovich indenter. The project contact area A_c in Eq. (2) can be expressed by the indentation depth h_c in the form of the following relationship:

$$A_c = 3\sqrt{3} \cdot h_c^2 \cdot tan^2\theta \tag{4}$$

where θ is the face angle, which is 65.35° for Berkovich indenter, which is used in the present case. It should be noted that composite materials exhibit several types of heterogeneity which can be observed from the perspective of different lengths scale. One of the most important elements of continuum approach is the use of Representative Volume Element (RVE) to describe heterogeneous material statistically [28–31]. A proper indentation grid [28] is necessary to ensure that the statistical analysis of each material phase will present the surface fraction of the total phases.

2.2. Microporomechanics

Based on the nano-indentation tests, the indentation modulus and hardness can be used to determine the particle properties such as particle stiffness (λ), Poisson's ratio (ν_s), cohesion (κ), friction

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