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Creep properties of cement and alkali activated fly ash materials using nanoindentation technique

Hyuk Lee [a,*], Vanissorn Vimonsatit [a], Prinya Chindaprasirt [b], Tuan Ngo [c], Priyan Mendis [c]

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

HIGHLIGHTS

- The capillary porosity phase is the main phase that increases the creep compliance rate of the OPC matrix.
- The creep behaviour reveals that partly-activated and non-activated phases are main factor of creep in AAFA mixtures.
- The upscaling approach of nanoindentation is able to link the creep properties of AAFA paste to the of AAFA concrete with the long-term creep of AAFA concrete.

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ABSTRACT

This paper presents creep properties of cement and alkali activated fly ash (AAFA) paste and mortar determined from statistical analysis of nanoindentation data. Cement paste having 95 MPa compressive strength at 28 days was tested for comparison and validation with a conventional test. Using nanoindentation, the specific creep of the cement paste after one year was predicted as 18.32 microstrain/MPa. For AAFA samples, an experimental program was set up using Taguchi's Design of Experiment method to consider four parameters, silica fume, sand to binder ratio, liquid to solid ratio, and superplasticiser, each with three variations.Using ANOVA, the percentage contributions of these parameters on the creep modulus of AAFA samples are: silica fume 26%, sand to binder ratio 21%, liquid to solid ratio 22%, and superplasticiser 31%. The results using deconvolution technique to identify the creep modulus of different phases of AAFA matrices show that partly-activated, non-activated slag and non-activated compact glass phases are leading the creep behaviour of AAFA samples due to their high creep modulus. Compare to other parameters, the liquid to solid ratio contributes the most to the creep property of partly-activated slag, non-activated slag and non-activated compact glass phases, that is, 51%, 89%, 68%, respectively. Sand to binder ratio and superplasticiser have minor effect on the creep behaviour. The results of the creep properties of AAFA paste were then compared with those of AAFA concrete using an upscaling process. The creep rate of AAFA concrete was defined by the creep properties of the matrix and the interface between aggregates and matrix assuming perfect bonding and slip bonding conditions. The results from the upscaling process show that the creep properties of AAFA paste from nanoindentation are representative of the long-term creep properties of AAFA concrete determined from a conventional test method.

1. Introduction

Durability of structural materials is an important aspect in the designed life of a structure. Concrete, being the most used structural material in construction, is known to have viscoelastic properties. After a long sustained load, strain in concrete increases with time which exhibits creep [1]. Determining creep properties of concrete and factors influencing creep are necessary when aiming for a more sustainable design. Concrete is conventionally made of ordinary Portland cement (OPC), but due to concerns over the environmental impact caused by cement production, alternative low-carbon cementitious materials have been introduced as a full or partial replacement of OPC in producing concrete. These materials

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

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are such as blended cement, alkali-activated pozzolan cement, and Geopolymer. This paper presents the creep properties of some of these materials and how different compositions in the mixtures are affecting their creep properties. The main aim is to obtain the optimum mix design, within the range of the parameters considered, for the minimum creep behaviour.

The measurement of viscoelastic properties of a material can be obtained by considering the nature and the rate of configurational rearrangements and the interaction of the properties. Creep testing involves applying a constant instantaneous stress to the specimen and measuring strain as a function of time during stressing [2]. The creep properties of cementitious materials are still an enigma because of the difficulty in linking to the timescale involved. The complex creep behaviour of cementitious material is largely related to the viscoelastic response of the primary hydration or reaction products and the binding phase of hardening [3]. However, Fischer-Cripps et al. [4] and Vandamme et al. [3,5,6] presented that time-dependent viscoelastic properties such as creep behaviour could be measured by nanoindentation technique.

The nanostructure of concrete made with OPC is controlled by the structure of calcium silicate hydrated (CSH) which governs its fundamental properties, such as strength, relaxation, creep, and fracture behaviour. Determining these properties demands a detailed knowledge of the nanostructure and how it relates to local mechanical properties. Thus, it is important to understand the nanostructure of OPC pastes. Several techniques have been used to understand CSH structures. Thomas et al. [7] determined two CSH morphologies using nitrogen, and the content of the two CSH phases depended on varying parameters such as internal water content. Similarly, Tennis et al. [8] provided a microstructure model of OPC paste that could quantitatively predict the volume of various phases. This model provides a mean for quantifying a volumetric proportion of major hydration products such as capillary porosity (MP), low-density CSH (LD CSH), and high-density CSH (HD CSH). Constantinides et al. [9,10] determined two types of CSH, Portlandite (CH) and clinker with nanoindentation. The results show that decalcification of CSH phases are the primary source of nanometre-scale elastic modulus degradation. Generally, the complex viscous characteristic of OPC pastes is according to the viscous behaviour of CSH, which is the primary hydration products of OPC paste. Advances in cement technology have recently enabled the understanding of the properties of CSH. However, CSH exhibits significant local variations and is still difficult to probe into its overall characteristics directly. Thus, a study on viscoelastic properties of hydration products by nanoindentation is well-suited, making it possible to probe sub-micrometric volume of material [11].

Alkali activated cement (AAC) has pozzolan as the main constituent, which can react with alkali to form binder [12]. The composites of cementitious components and alkali activated pozzolan cement are classified according to the type of pozzolan such as fly ash, metakaolin, soda lime glass and natural pozzolan [13]. Several researchers reported that common chemical compositions of traditional Portland cement and alkali activated pozzolan cement are silica and alumina which can also be found in industrial by products [1,14]. Though, several macro characteristics of AAC prepared from different aluminosilicate sources may appear similar, their physico-chemical and thermal properties vary to a large extent depending predominantly on the raw materials used [15]. Generally, AAC is a source material that does not contain carbonate, therefore, it does not release vast quantities of CO_2 as compared to traditional Portland cement. Recently, some research articles presented that AAC has several phases that contribute to the physical properties of the material. Němeček et al. [16] identified the reaction products of alkali activated fly ash cement (AAFA) pastes 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 slag (solid non-activated glass sphere or their relicts) using nanoindentation. Similarly, Lee et al. [14] investigated the effect of varying factors on the physical properties of AAFA and also found that N-A-S-H phase was a major reaction product of AAFA.

Viscoelastic properties of a material can be under the influence of time-dependent stress-strain factors. Previously, the method of analysis of nanoindentation assumed that behaviours of material are in an elastic-plastic manner. However, time-dependent viscoelastic properties, such as creep, can occur under indentation impress [17,18]. Literature shows that indentation properties of OPC and AAFA are well known from several research studies, but microporomechanics and creep behaviour of hydration products of OPC are still not well determined. This research is to fill this gap by investigating the creep properties of OPC and AAFA matrices using micromechanics, Creep properties are studied and compared with the results from conventional test methods. This study also reveals how different chemical phases in the matrices affect the creep properties, and suggests an optimum mix design to minimise creep.

2. Methodology

2.1. Materials and experimental procedure

Taguchi's Design of Experiment (DoE) is an efficient method to determine the contribution of parameters on the tested properties within the ranges considered [19]. In this investigation, two series of samples were tested, OPC and AAFA mixtures. OPC properties were tested for comparison and validation with the properties obtained from conventional tests. General purpose (Type I) cement available locally in Australia with the water to cement ratio of 0.3 was used to prepare the OPC paste samples. The samples were cured in lime water at ambient temperature, 23 ± 3 °C. The average compressive strength of the samples was obtained as 94.6 MPa at 28 days curing age. Nanoindentation test was carried out on the three sets of samples with minimum 300 indentation points per sample.

Nine different AAFA mixtures were prepared according to Taguchi's DoE approach. In the authors' previous research [14], four variables viz., silica fume (SF), sand to binder ratio (s/c), liquid to solid ratio (l/s) and superplasticiser (SP) contents were the parameters considered in the experimental program. The fly ash used was from Collie Power station in Australia, the chemical composition of fly ash is given in Table 1.

The medium particle size and Loss on Ignition (LOI) of fly ash were 45 μm and 1.5%, respectively. The experiment work in this research is to determine creep characteristics of AAFA matrices by considering three levels of the parameters. The standard L_9 (3^4) orthogonal array defining the contents of the mixtures is as shown in Tables 2 and 3. The data obtained from the nanoindentaion test can be used to determine meaningful mechanical properties of the material [18]. The following section will provide an overview of nanoindentation technique and equations necessary for statistical analysis in this research.

2.2. Principle and microporomechanics

Measurement of elastic modulus and hardness of material can be obtained from indentation load-displacement data [20–22]. Hertz contact equation determines the indentation modulus M as:

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