



Effect of natural carbonation on the pore structure and elastic modulus of the alkali-activated fly ash and slag pastes

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HIGHLIGHTS

- The effect of natural carbonation on the pore structure and E_m of the alkali-activated FA and GBFS pastes was investigated.
- GBFS content and pore structure are the decisive parameters for carbonation resistance.
- Better carbonation resistance of the GBFS-rich pastes is related to their finer pore structure.
- E_m might be needed in future predictive models for complete assessment of service life in alkali-activated materials.

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ABSTRACT

The aim of this paper was to investigate the effect of natural carbonation on the pore structure, and elastic modulus (E_m) of alkali-activated fly ash (FA) and ground granulated blast furnace slag (GBFS) pastes after one year of exposure in the natural laboratory conditions. The chemical changes due to carbonation were examined by X-ray diffraction (XRD), scanning electron microscope/energy-dispersive X-ray (SEM–EDX) and attenuated total reflectance Fourier transformed infrared spectroscopy (ATR–FTIR). Subsequently, the pore structure and E_m of the degraded material were tested by mercury intrusion porosimetry (MIP), nitrogen (N_2) adsorption, and nanoindentation.

The chemical degradation of alkali-activated pastes due to natural carbonation is showed to be dependent on the GBFS content and their pore structure development. It was found that the pure alkali-activated GBFS paste was not carbonated at all within the tested period due to fine gel pore structure. On the other hand, carbonation of the gel in the pastes consisting FA and GBFS generated significant mineralogical and microstructural changes. The extensive decalcification of the gel was reflected in the increase of nanoporosity. Consequently, the E_m of the carbonated pastes decreased.

This study suggests that the degradation of alkali-activated FA and GBFS pastes due to carbonation may be accurately evaluated through micromechanical properties measurements rather than only by testing alkalinity of the pore solution and corrosion of reinforcement such as commonly studied carbonation effect in the ordinary Portland cement (OPC)-based materials.

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1. Introduction

Alkali-activated materials (AAM), as environmentally friendly alternatives to OPC-based materials due to their comparatively low CO_2 emission and low natural resources consumption, have received much attention in the last twenty years [1,2]. AAM can

be produced from mixing different industrial by-products, such as FA, GBFS, bottom ash, waste glass, agricultural waste etc. with appropriate alkaline solutions [3]. In comparison with OPC-based materials, AAM might have better mechanical properties, stronger interfacial transition zone (ITZ), and better fire and chemical resistance [4]. However, the long-term properties of AAM, specifically carbonation resistance, are still unknown, which limits their application in engineering practice [5,6].

In general, the CO_2 gas reaction-diffusion process in porous media is controlled by both the concentration gradient of CO_2

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and the rate of the chemical reaction. Carbonation rate and degree in OPC-based materials and AAM depend on their physical and chemical properties and exposure conditions (i.e. relative humidity (RH), CO₂ concentration, temperature, duration of exposure). However, OPC-based materials and AAM exhibit different mechanisms during carbonation [7–10], due to formation and interaction of different phases in these two systems. While portlandite (Ca(OH)₂) and calcium silicate hydrate gel (C-S-H) are the primary sources of Ca²⁺ ions in the carbonation process of OPC-based materials [11], the alkali calcium aluminate silicate hydrate gel, denoted further in the text as a C-(N)-A-S-H gel, similar to [12], is the main source of Ca²⁺ ions for carbonation reactions in GBFS-based or different blended AAM (GBFS, FA, metakaolin). The removal of the calcium ions from the gel interlayers and gel sheet layers during carbonation induces shrinkage due to the molecular structural reorganization of the gels. This reaction is followed by a decrease in the volume of the paste, which is called carbonation shrinkage [13].

Depending on the binder composition, the effect of carbonation on the mechanical properties can vary. Mechanical properties of OPC pastes can be considerably improved by carbonation [14,15], because carbonation of the Ca(OH)₂ leads to a reduction in porosity due to the positive difference of molar volumes between Ca(OH)₂ and CaCO₃. Measured by nanoindentation, hardness and elastic modulus tend to shift to higher values in the OPC paste. On the other hand, micro-mechanical properties of blended OPC and GBFS (such as CEM III-B) pastes decrease due to carbonation, since they contain less Ca(OH)₂ and have a lower Ca/Si ratio in the C-S-H gel [16,17]. It has been found that at extensive levels of decalcification (when Ca/Si is below ~0.66), decomposition of the Ca-O sheets of the C-S-H leads to significant shrinkage due to precipitation of calcium carbonates and subsequent polymerization of the silica gel [18], resulting in decrease of the mechanical strength.

Only a few studies so far have discussed mechanical behavior of carbonated AAM. For instance, Bakharev et al. [7] found that the carbonation resistance of alkali-activated slag (AAS) concrete was lower than that of OPC concrete. The AAS concrete had higher strength loss and carbonation depth than OPC concrete in both investigated exposure conditions, i.e. immersion in 0.352 M (M) sodium bicarbonate solution, and exposure to atmosphere with 10–20% of CO₂ at 70% RH. Nevertheless, both sets of the conditions are highly aggressive and the results cannot be directly used to predict in-service behavior of AAS concrete subjected to natural carbonation.

Bernal et al. investigated accelerated carbonation of alkali silicate-activated GBFS mortars, under the condition of CO₂ concentration of 3.0 ± 0.2%, temperature of 20 ± 2 °C, and 65 ± 5% RH [19]. Noncarbonated specimens had compressive strengths of up to 63 MPa after 28 days of curing when granulated GBFS was used as the sole binder. The strength decreased by 40–50% after carbonation.

Puertas et al. [9] studied the effect of alkaline activator on carbonation degree in AAS mortars in terms of mechanical properties. When sodium waterglass was used as an activator, decalcification of the C-S-H gel prompted by carbonation led to a loss of cohesion in the matrix, increase in porosity, and the reduction of the compressive strength. On the other hand, when NaOH was used as activator, carbonation enhanced the compressive strength of the mortar. The authors assumed that this was due to the precipitation of calcium carbonates in the pores, causing a decline in the total porosity and average pore size.

Since effect of carbonation in the previous studies was mainly investigated on bulk macro-mechanical properties of alkali-activated mortars or concretes, it is difficult to understand its

effects on the local micro-mechanical properties of the binder. Several parameters can affect the interpretation of the mechanical behavior of the material due to the carbonation if they are studied at concrete (macro) level and not at the paste (micro) level. The most important are:

- Effect of the material scale: Pastes are more suitable for the analysis due to their homogeneity, compared to concrete. The presence of aggregates in the concrete encompasses paste aggregate interface, which can carbonate faster due to its higher porosity compared to the paste itself. Therefore, the carbonation study on the paste samples is needed first for understanding of more complex carbonation mechanism at the concrete scale.
- Effect of the sample size: Carbonation reaction is dominant at the concrete surface and in the first several millimeter depth from the surface. Therefore, effects of measuring the bulk mechanical properties (compressive, tensile or flexural strength) will be largely dependent on the size of the sample. For instance, the standard 150 × 150 × 150 mm³ concrete cubes NEN [5988:1999] will be useless in evaluation of the natural carbonation effect on the compressive strength if the carbonated depth is only a few millimeter. On the other hand, the coupled effects of different mechanisms at the concrete surface (e.g. drying shrinkage that can lead to cracking) and not only carbonation can overestimate the effect of the carbonation on the material properties.
- Effect of the testing scale (local/bulk): Testing of the local micromechanical properties provides a more reliable evaluation on chemical degradation of the paste, compared to a bulk test such as the compressive strength test of a standard concrete cube. With a local analysis the chemical degradation of the different phases present in the system can be distinguished and correlated with the chemical analysis. Furthermore, on the same sample, testing of both, carbonated and noncarbonated areas is possible.

Considering these effects, it is clear that understanding the influence of carbonation on the micro-mechanical properties at the paste level is the first step towards understanding the properties of carbonated concrete. Therefore, assessment of the micro-mechanical properties is needed, such as E_m, which might be of great importance for predicting the service life and degradation degree of AAM.

The aim of this study was to evaluate the effect of chemical changes in the alkali-activated FA and GBFS pastes when exposed in the natural laboratory conditions (~55% RH, ~0.038% CO₂ v/v) on the pore structure and E_m. The gel structural changes were investigated by XRD, SEM-EDX and ATR-FTIR, while MIP, N₂ adsorption, and nanoindentation were used to examine pore structure and E_m after carbonation.

2. Materials and methods

2.1. Materials and sample preparation

The precursors used in this study were FA from VLIEGASUNIE BV and GBFS from ORCEM (the Netherlands), with chemical compositions as shown in Table 1. The main crystalline phases in FA were quartz, mullite, and hematite, while GBFS was mainly amorphous, as showed by XRD scans (Fig. 1). The alkaline activator was synthesized by mixing anhydrous pellets of sodium hydroxide with deionized water and commercial sodium silicate solution (27.5 wt% SiO₂, 8.25 wt% Na₂O). The activator concentration was

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