



Mechanisms of autogenous shrinkage of alkali-activated fly ash-slag pastes cured at ambient temperature within 24 h

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HIGHLIGHTS

- Autogenous shrinkage of AAFS pastes with various slag replacement ratios of fly ash measured.
- Autogenous shrinkage and chemical shrinkage of AAFS pastes increase with increasing slag content.
- Autogenous shrinkage of AAFS pastes in liquid stage results from chemical shrinkage.
- Autogenous shrinkage of AAFS pastes in hardening stage is not solely due to self-desiccation.
- Volume fraction of small pores of AAFS pastes decreases with increasing slag content.

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ABSTRACT

This paper aims to provide a better understanding of autogenous shrinkage of alkali-activated fly ash-slag (AAFS) pastes at very early age (<24 h). The autogenous shrinkage of AAFS pastes with 10%, 20% and 30% slag replacement ratios of fly ash is measured. The results show that increasing the slag content in AAFS pastes would accelerate the chemical reaction process and cause a higher autogenous shrinkage and chemical shrinkage. The autogenous shrinkage of AAFS pastes in the liquid stage (from 0 h to initial setting time) is caused by chemical shrinkage, which arises from the absolute volume reduction of reaction products comparing with the original volume of unreacted binder. This phenomenon is similar to that found in Portland cement (PC) systems. Nevertheless, in hardening stage (from final setting time to 24 h) the autogenous shrinkage may be not fully attributed to the self-desiccation process that occurs in PC systems. Some other reactions including the continuous reorganization and rearrangement of aluminosilicate gel structure may also contribute to the development of autogenous shrinkage. In addition, the autogenous shrinkage in early 24 h occurs mainly due to volume contraction by chemical shrinkage in fresh state, which occupies approximately 70% of total autogenous shrinkage.

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1. Introduction

Alkali-activated materials (AAM) have been studied over the past decades because of its environmental benefits and superior engineering properties, e.g., mechanical properties and chemical durability [1–3]. Based on the chemical compositions of raw materials, AAM can be divided into two sole systems, including high calcium system (e.g., alkali-activated slag (AAS)) and low calcium system (e.g., alkali-activated fly ash (AAF)) [4]. However, these two sole systems have some critical drawbacks. For instance, AAS has issues related to fresh properties such as quick setting problems [5], while AAF needs to be cured under an elevated temperature of 60–85 °C [6–8]. Therefore, it is essential to find a new AAM

system, e.g., alkali-activated fly ash-slag (AAFS) blended system, which can achieve superior engineering properties under ambient curing condition [9–17]. Nevertheless, the practical applications of AAFS remain low mainly due to the uncertain long-term durability and insufficient ability against shrinkage and micro-cracking [18,19].

Shrinkage is an important engineering property of Portland cement (PC) concrete that can affect the cracking probability and thus durability of concrete by providing easy access of water and aggressive species into the interior of concrete. Shrinkage is a broad term given to the volume reduction of concrete, which can be classified as different types including plastic shrinkage, chemical shrinkage, autogenous shrinkage, drying shrinkage and carbonation shrinkage [20]. Plastic shrinkage occurs if the water is allowed to escape from the surface of concrete during the plastic state. Chemical shrinkage results from the reduction in absolute

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volume of hydration products compared to the original volume of unhydrated cement. When concrete is sealed, cement hydration leads to autogenous shrinkage via reducing the internal moisture. When concrete is exposed to normal drying environmental conditions, moisture diffusion of the hardened concrete results in drying shrinkage along with carbonation shrinkage due to the reaction with carbon dioxide [20].

Similarly, shrinkage is also one of the important properties for AAM based concrete. An increasing number of studies have been conducted to investigate the shrinkage of AAM. It was found that the sole AAM systems have significantly higher autogenous shrinkage and drying shrinkage than PC systems [21–23]. It was also reported that the shrinkage of the sole systems is influenced by different factors, such as the chemical and physical properties of binder, the type and dosage of alkaline activator, and curing conditions [24–27]. Up to now, however, there are only a few available studies on shrinkage of AAFS. Deb and Nath [18] and Hojati and Radlinska [28] explored the effect of fly ash and slag proportions and the type of alkaline activator on shrinkage of AAFS mixtures (paste, mortar, and concrete) and found that increasing slag content and decreasing ratio of sodium silicate to sodium hydroxide in AAFS mixtures would result in high autogenous shrinkage and drying shrinkage. Nevertheless, the related mechanism of the shrinkage, particularly the autogenous shrinkage of AAFS is still not clear. Lee et al. [19] studied the shrinkage characteristics of AAFS pastes and mortar during the first 28 days and concluded that autogenous shrinkage of AAFS occurred due to the self-desiccation in hardened state rather than the volume contraction by chemical shrinkage in fresh state. However, this research mainly focused on the autogenous shrinkage at a relatively later age from days to weeks. There is still a lack of theoretical explanation of the autogenous shrinkage of AAFS during the first day after casting.

For PC systems, it is known that autogenous shrinkage is a consequence of chemical shrinkage at liquid stage. After hardening, autogenous shrinkage becomes less than chemical shrinkage since the volume reduction is restrained by a rigid skeleton of hydrating cement paste [20]. With the formation of skeleton microstructure, the contraction of chemical shrinkage is restrained, leading to the formation of voids. In addition, the continuing hydration causes the variation of water molecules and the empty of pores, which finally leads to the decrease in relative humidity as well as the increase in curvature of water-air menisci [29–31]. Such high curvature of menisci would increase the surface tension and impose compression on the walls of capillary pores and solid hydrates and thus induces contraction in hardened cement paste [22,32]. This phenomenon is called self-desiccation [20]. According to this theory, the small diameter of pores would increase the curvature of menisci and capillary, and thus cause higher autogenous shrinkage [33]. For AAFS systems, however, the autogenous shrinkage mechanism occurring at first 24 h after casting may be different when compared with the well-known self-desiccation process due to the total different chemical reaction process. The alkaline reaction of AAFS is an extremely complex process, which consists of two basic reaction processes of sole AAM systems (i.e., AAF and AAS). During this process, two main reaction products (i.e., N-A-S-H gel from AAF system and C-A-S-H gel from AAS system) do not develop separately, but they undergo structural and compositional change [34,35]. Therefore, it is necessary and vital to explore autogenous shrinkage immediately after casting in order to fully understand the mechanisms associated with autogenous shrinkage of AAFS pastes.

The main purpose of this study is to provide a better understanding of early-age autogenous shrinkage of AAFS pastes. The autogenous shrinkage and chemical shrinkage of AAFS pastes with slag content as 10%, 20% and 30% of the total binder during first 24 h after casting were measured. A series of tests were conducted to

investigate the mechanisms of early-age shrinkage of AAFS pastes. The workability was measured by flow table test. The early-age reaction and setting process was monitored through isothermal conduction calorimetry (ICC) and Vicat setting time test, respectively. In addition, the internal relative humidity (RH) was measured by humidity data logger. Furthermore, the phases composition and pore structure were characterized by X-ray diffraction (XRD) and mercury intrusion porosimetry (MIP), respectively.

2. Experimental program

2.1. Raw materials

Class F fly ash (FA) and ground granulated blast-furnace slag (GGBS) were used in this study. The chemical compositions of FA and GGBS are shown in Table 1. The particle size distribution curves are plotted in Fig. 1, with a volume-based average particle size of 14.77 μm for GGBS (Fig. 1a) and 26.81 μm for FA (Fig. 1b). The alkaline activator was prepared by mixing sodium hydroxide (>98% purity) with distilled water and sodium silicate solution. The concentration of sodium hydroxide (SH) was 10 M for all mixtures. The $\text{SiO}_2/\text{Na}_2\text{O}$ molar ratio of the sodium silicate (SS) was 2.0 with chemical composition of 26.42 wt% SiO_2 , 13.65 wt% Na_2O and 53.93 wt% H_2O . The alkali modulus (i.e., the mass ratio of SiO_2 to Na_2O) in the alkali solution was 1.12. Since the polycarboxylate-based superplasticizers (SPs) not only have a negligible effect on the reaction kinetics of AAFS but also have a significant effect on the workability [36], it was used to improve the workability of AAFS paste in this work. The properties of SPs are given in Table 2.

2.2. Mixture proportions

The AAFS pastes with three different GGBS replacement ratios for FA were designed and tested in this work. The mixture proportions of AAFS paste are given in Table 3 and denoted with specific codes. The label 'FS' represents AAFS paste, while the number, '10', '20', or '30', stands for the percentage of GGBS replacement for FA by weight. The ratio of alkaline activator to binder by weight (AL/B) was 0.4. The mass ratio of sodium silicate to sodium hydroxide (SS/SH) was 2.0. In addition, the mass ratio of SPs to binder (B) was 0.01.

2.3. Testing methods

Herein, a non-contact testing method was applied to measure the autogenous shrinkage occurring immediately after mixing the AAFS paste and continuing for the first 24 h. Moreover, a series of tests were conducted to investigate the chemical and physical phenomena causing the early-age autogenous shrinkage.

2.3.1. Autogenous shrinkage test

Early-age autogenous shrinkage can be determined by volumetric or linear measurements. Volumetric measurements monitor the volume changes of paste or mortar specimens at a constant temperature, while linear measurements can be used to evaluate the linear dimension change of paste, mortar, or concrete specimens cast in a rigid slab mould and recorded by sensors at one end or both ends of the specimens. Linear measurements can reflect the actual material behaviour as compared with volumetric measurements because the effects of other influencing factors such as thermal dilation and bleeding can be conducted in the slab test [37]. According to the sensors used in the test the linear measurements can be classified into contact and non-contact methods. Linear Variable Differential Transformer sensors (LVDTs) are commonly

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