



EFFECT OF SHRINKAGE REDUCING ADMIXTURES ON SHRINKAGE BEHAVIOUR OF GEOPOLYMER MORTAR

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Abstract: Alkali-activated binder (so-called geopolymer) is an eco-friendly binder alternative for cement. It is characterized by a very low carbon footprint. However, shrinkage of geopolymer materials is still one of the main challenges that impede its in-situ applications. The purpose of this study is to investigate the shrinkage behaviour of geopolymer mortars activated with different activator concentrations and incorporating different dosages of shrinkage reducing admixture (SRA). Moreover, it explores the influence of SRA on the correlation between shrinkage behaviour and different factors including the concentration of activator and curing temperature. Geopolymer mortars were prepared using the conventional two-part alkali activation mechanism. Specimens were exposed to different curing conditions. A series of tests including setting time, flowability, drying shrinkage, and compressive strength of geopolymer mortar were conducted on mixtures with and without SRA. Regardless of the activator dosage, results showed that similar to cement-based materials, SRA was effective in reducing the shrinkage strains experienced by geopolymer mortars. However, the compressive strength was decreased. Furthermore, SRA effectiveness increased with higher curing temperatures and activator concentrations.

1 INTRODUCTION

Global concrete production has grown widely over the recent decades parallel with the population growth and the need for more infrastructures. The high concrete demand in the construction industry increased ordinary Portland cement (OPC) production. The significant environmental pollution associated with carbon dioxide (CO₂) emissions generated during cement manufacturing process are of major concerns. Alkali-activated binders represent a promising alternative to OPC in many constructional applications (Yang et al. 2013).

The acceptance of alkali activated materials in the industry has been hindered by concerns related to their shrinkage. Shrinkage is a critical parameter affecting the durability of the alkali activated material and its resistance to cracking. Shrinkage can be defined as a reduction in the volume of the material due to either drying or chemical reactions taking place in the system. These reductions in volume induce stresses in the material matrix causing deformations. As deformations are restrained in the hardened binder, cracking occurs. The formation of cracks allows the ingress of aggressive chemicals into the concrete, therefore, significantly reducing its service life.

The world's first shrinkage-reducing admixture (SRA) was developed in Japan in 1982 in a partnership between Nihon Cement Co., Ltd. and Sanyo Chemical Industries, Ltd. (Sato et al. 1983). Several different SRAs are commercially available, and although they do not exactly have the same chemical composition, they are similar in nature and all serve to reduce the surface tension of concrete's pore fluid (Nmai et al. 1998). It is commonly understood that by reducing the surface tension of concrete's pore fluid, SRA significantly reduces the magnitude of capillary stresses and shrinkage strains that occur when concrete is losing moisture (i.e., through drying or self-desiccation) (Shh et al. 1992, Tazawa and Miyazawa 1995). It has been shown that by lowering the surface tension of pore fluid, SRA can reduce drying shrinkage of concrete by up to 50% (Shh et al. 1992). In addition, SRA was shown to reduce the capillary stresses that are generated by autogenous shrinkage in high-performance low w/c concretes (Weiss et al. 1999, Bentz et al. 2001). These attributes are extremely beneficial in reducing the risk of shrinkage cracking in concrete elements (Radlinska et al. 2007).

The addition of SRA was also shown to improve the resistance of concrete against plastic shrinkage cracking (Lura et al. 2007). In contrast to the significant benefits of SRA in improving the shrinkage performance of concrete, some negative side effects have also been reported. Namely, it has been observed that SRA reduces the rate of cement hydration and strength development in concrete (He et al. 2006, Bentz 2006). Concrete containing SRA exhibits a delay in setting (Brooks et al. 2000) and a reduction in the compressive and tensile strength for well-cured specimens (Weiss et al. 1999), compared to control specimens with similar mixture proportions. Moreover, it has been reported that the addition of SRA may cause loss of entrained air, which may negatively affect the resistance of concrete to freezing damage (Berke et al. 2003).

Although extensive research has been performed on measuring the shrinkage and mechanical properties of concrete containing SRA, few studies addressed the effect of SRA in alkali-activated concrete. Alkali concentration, binder type, curing regime, and SRA dose, are all important parameters that would give an image of the effect of SRA on fresh, and hardened properties of alkali-activated concrete.

2 EXPERIMENTAL PROGRAM

2.1 Materials

In the present study, FA class-F was used as the precursor. The chemical compositions and physical properties of the FA class-F are listed in **Table 1**. Sodium hydroxide with a purity level of 98% and sodium silicate (SiO₂ (30%), Na₂O (20%), H₂O (50%), specific gravity 1.38 g/mL) were used to prepare the alkali activators. The alkali activator was prepared as the following: Distilled water was used to dissolve the NaOH solid at 3M, 6M, and 12M, and left overnight to cool down to the room temperature while covered to prevent carbonation. Sodium silicate solution was then mixed with the NaOH solution to form the alkali activator solution (mass of sodium silicate solution/ NaOH solution = 2). Commercial shrinkage reducing admixture conforming to the requirements of ASTM Standard C494 / C494M - 17, Type S was used in this study. ASTM Standard C109 / C109M-16a river sand was used as the fine aggregate material. The oven-dry, saturated surface dry specific gravity, and absorption of the standard sand used were 2.54, and 2.55, 0.08 %, respectively.

Table 1: Properties of the precursor used.

Chemical composition (%)	Fly ash
CaO	2.60
SiO ₂	46.00
Al ₂ O ₃	33.00
Fe ₂ O ₃	10.50
SO ₃	-
Physical properties	
S.S.D Specific gravity (g/cm ³)	2.38
Surface area (m ² /kg)	290 (Blaine)

2.2 Mix proportion and specimen preparation

The mixture proportions of the alkali-activated fly ash (AAF) mortars are shown in **Table 2**. Nine AAF mortar mixtures were designed using a solution made of three different NaOH concentrations to cover a wide range of concentrations (3, 6, and 12M) each mixed with a constant sodium silicate concentration. The mass ratio of sand to precursor was fixed at 2:1 for all mixtures. The mass ratio of activator solution to precursor was 0.40 for all mixtures. The mass ratio of the water glass to NaOH solution was 2.0 in all activator mixtures. Two curing regimes were applied: 23°C with relative humidity of 60 ± 5% (ambient) and at 60°C (oven). Specimens were cured under oven conditions to enhance the alkali activation and increase the degree of the polymerization reaction in AAF mortars.

2.3 Specimen preparation

Initially, fly ash was dry mixed with sand in an electric mixer at a slow speed of (140 ± 5 revs/min) and medium speed (285 ± 5 revs/min) for 2 minutes to ensure homogeneity of the mixture. The prepared alkali activator solution is then added to the mixture and mixed for an additional 2 minutes. Immediately after mixing, 50 mm cubic molds for compressive strength evaluation according to ASTM Standard C109 / C109M-16a, and 25 mm × 25 mm × 285 mm prismatic molds for shrinkage measurements according to ASTM Standard C490 / C490M – 17, were cast. Specimens were compacted using a vibrating table and a tamping rod. All specimens were cured at room temperature of 23 °C and at a relative humidity of 60% ± 5% inside molds for 24 hours. After 24 hours, specimens were demolded and stored in ambient room conditions or in the oven at 60°C until testing age. All reported results represent the average of three replicates.

Table 2: Mix proportion of AAF mortar specimens

Mixture	NaOH solution molarity (M)	Ratio NaOH: water glass	Sand/binder ratio	SRA dosage (% of binder mass)
F3	3	1:2	2:1	0
F6	6	1:2	2:1	0
F12	12	1:2	2:1	0
F3R1	3	1:2	2:1	1
F6R1	6	1:2	2:1	1
F12R1	12	1:2	2:1	1
F3R2	3	1:2	2:1	2
F6R2	6	1:2	2:1	2
F12R2	12	1:2	2:1	2

2.4 Testing program

The workability and flow of mortar mixtures were evaluated using the procedure listed in ASTM Standard C1437-15. Initial and final setting times were measured using a Vicat needle as described in ASTM Standard C191-13. Compressive strength development was evaluated at ages of 1, 7, 14, and 28 days using a 300 kN universal testing machine (UTM) in accordance with ASTM Standard C109 / C109M-16a. Cubes were loaded until failure at a load rate of 345 kPa/s. For each mortar and at each age, 3 cubes were tested, and the results were averaged. Drying shrinkage was measured using prism samples (25.4 × 25.4 × 254 mm) in accordance with ASTM Standard C490 / C490M - 17 using a length comparator with dial gauge accuracy of 0.0001-inch.

3 EXPERIMENTAL RESULTS

3.1 Flowability

Flow table results for the mortar mixtures with and without SRA are presented in **Table 3**. Generally, the flow values of AAF mortar mixtures without admixtures are high, this was attributed to the spherical shape and large specific surface area of FA particles (Fernández-Jimenez et al. 2006). Compared to mortars without admixtures, the flow values were slightly increased by the addition of SRA admixture, hence showing a positive effect on mortar workability. For example, the flowability values for AAF mortars activated with 3M, 6M, and 12M NaOH concentrations, and containing 1% SRA were increased in the range of 1-4%, compared to AAF mortars without SRA. Increasing the dose of SRA to 2% by mass of fly ash, induced even further increase in the flowability. The addition of liquid SRA does increase the specific liquid to solid ratio hence, directly influencing the flowability of mortar specimens (Palacios and Puertas 2005). The flowability values for AAF mortar specimens containing 2% SRA and activated with 3M, 6M, and 12M NaOH concentrations were increased by 1-2%, compared to specimens containing 1% SRA.

3.2 Setting time

The initial and final setting times of AAF mortar specimens are given in **Table 3**. It is well known that a binder should have an acceptable setting time to allow its transportation and casting. If setting time is too long, it is not economical. Contrary to that, irreversible mistakes might happen if the setting time is very short. Thus, the effect of SRA admixtures on the initial and final setting time of AAF mortars was noted in this study. Generally, the setting times of AAF mortars specimens decreased with increasing sodium hydroxide concentrations in the used activator as shown in **Table 3**. The addition of SRA admixture retarded both the initial and final setting times of AAF mortars. The retardation effect is even more pronounced when the percentage of SRA is increased from 1% to 2% (by mass of binder). For instance, the addition of 1% SRA admixture retarded the initial setting time of AAF mortars activated with 3M, 6M, and 12M NaOH by 6, 12, and 13 min, respectively compared to specimens without SRA. The corresponding retardation in final setting time were 8, 10, and 10 min, respectively. Moreover, increasing the dose of SRA admixture to 2% caused an initial setting time retardation of 11, 15, 19 min, respectively and a final setting time retardation of 16, 17, and 18 min, respectively compared to specimens without SRA.

Table 3: Setting time and flowability index of AAF mortar specimens

Mixture	Setting time (min)		Flowability (cm)
	Initial	Final	
F3	144	286	108
F6	126	254	102
F12	89	186	92
F3R1	150	294	112
F6R1	138	262	104
F12R1	102	196	93
F3R2	155	302	114
F6R2	141	271	105
F12R2	108	204	94

3.3 Compressive strength

3.3.1 Effect of SRA on compressive strength of AAF mortars under ambient curing

The development of compressive strength for all AAF mortars, with and without SRA, was studied according to activator concentration, curing condition, SRA content, and age. **Figure 1** shows the compressive

strength values over time of 3M, 6M, and 12M NaOH activated fly ash mortar specimens ambient cured. Generally, the addition of SRA reduced the compressive strength of AAF mortars. The addition of 1% SRA admixture caused AAF mortars activated with 3M, 6M, and 12M NaOH to encounter a reduction of 5.88, 11.29, and 9.8% in the 28-day compressive strength values respectively, compared mortars without SRA. The corresponding reduction percentages for the 2% SRA containing AAF mortars were 11.99, 18.02, and 17.2% compared to mortars without SRA.

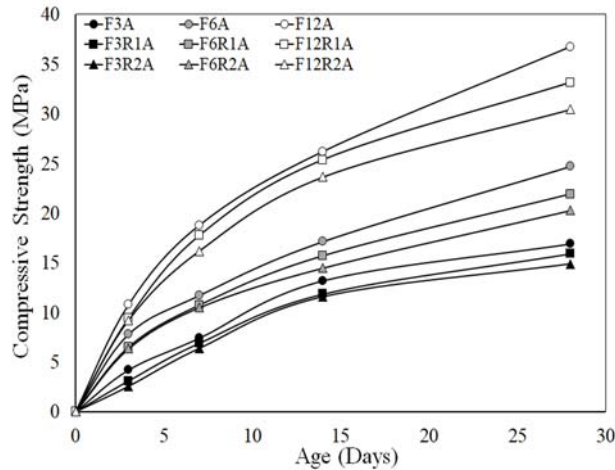


Figure 1: Compressive strength of AAF mortars containing 0, 1, and 2% SRA ambient cured.

Figure 2 shows the compressive strength values over time for 3M, 6M, and 12M NaOH activated fly ash mortars oven cured. The addition of SRA reduced the compressive strength of oven cured mortar specimens. Reductions in compressive strength varied in the range of 6.25-17.23% depending on activator concentration and SRA dosage used. For example, the addition of 1% SRA admixture caused AAF mortars activated with 3M, 6M, and 12M NaOH to encounter a reduction of 9.34, 10.47, and 8.05% in the 28-day compressive strength values respectively, compared to mortars without SRA. The corresponding reduction percentages for the 2% SRA containing AAF mortars were 16.55, 17.23, and 13.97% compared to specimens without SRA.

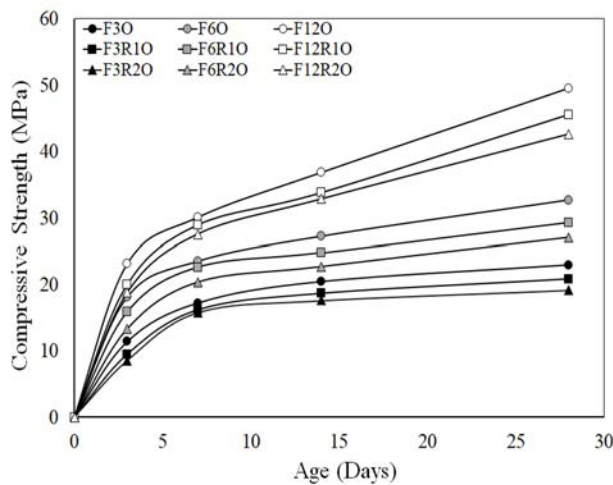


Figure 2: Compressive Strength of AAF mortars containing 0, 1, and 2% SRA oven cured.

3.4 Drying Shrinkage

3.4.1 Effect of SRA on drying shrinkage of AAF mortars under ambient curing

The effect of SRA addition on the drying shrinkage strains of AAF mortar specimens was studied. Drying shrinkage strains of ambient cured fly ash mortars containing 0, 1, and 2% SRA and activated with 3M, 6M, and 12M (NaOH) concentrations are plotted in **Figs. 3-4**, respectively. Results indicated reductions in the drying shrinkage of AAF mortars with the addition of 1% SRA compared to samples without SRA. Reductions in drying shrinkage were more pronounced when SRA dose was increased to 2%. Reduction percentages in the total drying shrinkage of AAF mortars activated with 3M, 6M, and 12M NaOH, caused by the addition of 1% SRA were 26.79, 16.50, and 25.94% respectively, compared to mortars without SRA. The corresponding reduction percentages in total drying shrinkage, when 2% SRA was used were 36.71, 26.58, and 35.96% respectively.

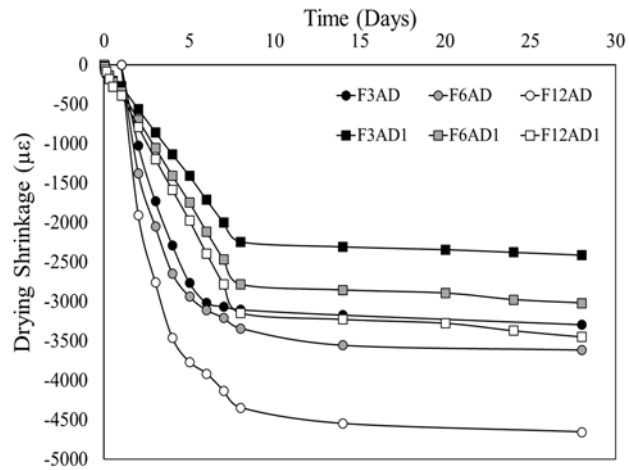


Figure 3: Drying shrinkage of AAF mortars containing 1% SRA (ambient cured).

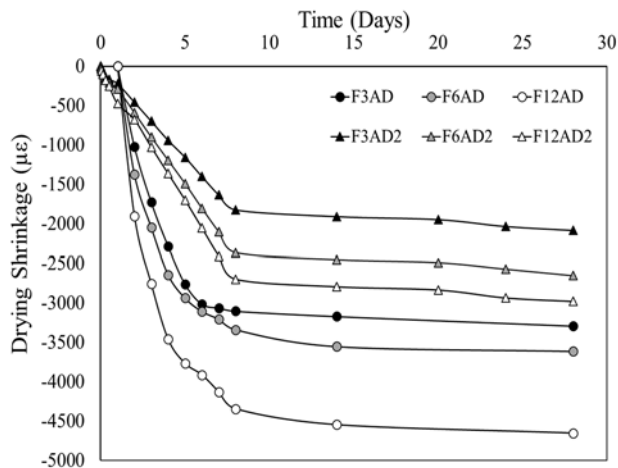


Figure 4: Drying shrinkage of AAF mortars containing 2% SRA (ambient cured).

3.4.2 Effect of SRA on drying shrinkage of AAF mortars under oven curing

Drying shrinkage strains of oven cured Fly ash mortars containing 0, 1, and 2% SRA and activated with 3M, 6M, and 12M (NaOH) concentrations are plotted in **Figs. 5-6**, respectively. Results indicated reductions

in the drying shrinkage of AAF mortars with the addition of 1% SRA compared to samples without SRA. Reductions in drying shrinkage were higher when SRA dose was increased to 2%. Reduction percentages in the total drying shrinkage of AAF mortars activated with 3M, 6M, and 12M NaOH, caused by the addition of 1% SRA were 42.42, 45.31, and 46.04% respectively, compared to mortars without SRA. The corresponding reduction percentages in total drying shrinkage, when 2% SRA was used were 52.45, 51.95, and 54.52% respectively.

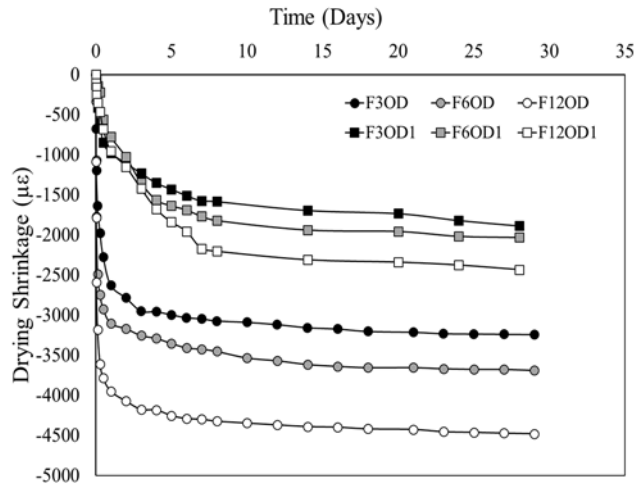


Figure 5: Drying shrinkage of AAF mortars containing 1% SRA (oven cured).

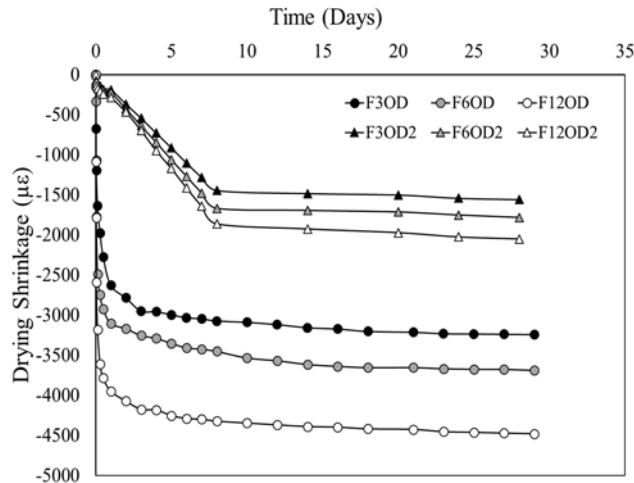


Figure 6: Drying shrinkage of AAF mortars containing 2% SRA (oven cured).

4 CONCLUSION

- Shrinkage reducing admixtures increased the flowability in all alkali activated mortar specimens tested.
- Addition of SRA retarded both the initial and final settings of AAF mortars.
- The addition of SRA reduced the overall compressive strength of alkali activated mortar specimens. Reductions were higher when SRA dose was increased from 1 to 2%.
- The addition of SRA significantly reduced the drying shrinkage of mortar specimens. Reductions were more pronounced when SRA dose was increased from 1 to 2%.

- Reductions in drying shrinkage reached up to 67.08%. The main mechanism of SRA that is responsible for the beneficial shrinkage reductions in AAM, is found to be the reduction in surface tension of pore water prompted by the admixture.

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