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Factors affecting the drying shrinkage of alkali-activated slag/fly ash mortars

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Abstract
Climate change associated with carbon emissions is driving a demand for low-carbon cements. One of the most promising low-carbon alternatives is alkali-activated binder systems consisting of fly ash and ground granulated blast furnace slag. However, there are some issues related to their drying shrinkage which can be problematic when upscaling these binders to produce full size construction products. This study found that modifying the curing regime can reduce the drying shrinkage of alkali-activated mortars by up to 60%. The proportion of slag and fly ash employed also influences drying shrinkage behaviour but is less significant than the curing temperature and curing duration before drying is initiated. The drying shrinkage performance appears to be linked to the maturity and strength of the different mixes when drying begins.

Introduction
In recent decades significant efforts have been made to develop alternative cementitious materials with less embodied carbon than traditional Portland cement based materials. One of the most promising binders investigated are alkali-activated binder systems which are also often described as geopolymers [1]. Alkali-activated binders typically consist of aluminosilicate precursor materials such as fly ash, ground granulated blast furnace slag (slag) or metakaolin which are chemically activated by an alkaline source [2, 3]. The precursor materials have a lower carbon footprint compared with that of Portland cement. Among the most commonly used activators are solutions of sodium silicate and sodium hydroxide. The alkaline activators can be the largest source of embodied carbon within alkali-activated binders but despite this, alkali-activated systems are still reported to have lower embodied carbon than Portland cement systems. In recent years, progress has also been made with regards to the production of alkaline activators from waste sources such as waste glass and bamboo leaf ash [4–6].

Numerous studies have reported acceptable or even advantageous properties of alkali-activated binders when compared with traditional Portland cement based binders. These include rheological properties, setting behaviour, mechanical properties and resistance to aggressive media [1, 7–9].

Several valuable studies have been conducted to consider the shrinkage behaviour of alkali-activated systems [10, 11]. Humad et al. [12] studied the drying shrinkage of high alkali-activated MgO rich slag concrete. The effect of high temperature curing (65 °C) for 24 h and sealing the samples for one month was studied. Findings showed that a combination of heat curing and sealing the samples had the potential to reduce drying shrinkage by 30–50%.

This study explored lower curing temperatures and shorter sealing/curing durations. The purpose was to find the conditions which are most practical and cost effective for improving drying shrinkage behaviour during the production of full scale alkali-activated construction products in an industrial setting. Drying shrinkage was the main focus of this study, with the effect of various curing conditions and slag/fly ash proportions investigated. Additional characteristics were also investigated including workability, setting time, volume of permeable voids (VPV) and compressive strength. These properties were not specifically targeted, as they were not the aim of this research, but help to provide context for the drying shrinkage performance observed.
Materials and methods

Materials

The fly ash was obtained from Kilroot Power Station, Northern Ireland. The slag was obtained from Ecocem, Ireland. The main chemical composition obtained by X-ray fluorescence of the fly ash includes 56% SiO$_2$, 23% Al$_2$O$_3$, 6% Fe$_2$O$_3$, 5% CaO, 2% MgO, 2% K$_2$O. The slag includes 43% CaO, 36% SiO$_2$, 11% Al$_2$O$_3$, 8% MgO, 1% SO$_3$. Mineralogical analysis and the particle size distribution of the fly ash and slag are similar to that given in a previous paper [13]. The alkaline activators and the sand used were described in another previous paper [14].

Mix proportions and sample preparation

S75/F25 contained 75% slag (455 kg/m$^3$) and 25% fly ash (152 kg/m$^3$) within the powdered component of the binder. A similar nomenclature was used for S50/F50 (297 kg/m$^3$ slag and 297 kg/m$^3$ fly ash) and S25/F75 (145 kg/m$^3$ slag and 436 kg/m$^3$ fly ash). Each mix also includes 140 kg/m$^3$ sodium silicate, 115 kg/m$^3$ sodium hydroxide, 102 kg/m$^3$ water, and 1348 kg/m$^3$ sand. The water includes the water needed to bring the sand into a saturated surface dry state (i.e. absorption water, 12 kg/m$^3$). The alkali dosage refers to the percentage of Na$_2$O in the binder and was 7.5%. The alkali modulus is defined as the ratio between Na$_2$O and SiO$_2$ and was 1.25. These mix parameters were largely chosen based on studies conducted in [15–18]. The volume of paste was fixed at 50% for each mix, which results in the use of specific proportions of materials in this study. Mixing, casting, compaction, demoulding and curing procedures were described in a previous paper [19].

Testing procedures

Workability of the fresh mortars was assessed with a flow table test according to the procedure given in BS EN 13395-1:2002. The test started immediately after mixing was completed. A truncated conical metal mould (60 mm in height and with internal diameter of 70 mm at the top and 100 mm at the bottom) was placed centrally on the surface of the flow table disk. The mould was filled with mortar in two layers. Each layer was compacted by 10 short strokes of a metal tamper (40 mm in diameter). The excess mortar was skimmed off with a palette knife. Then the mould was gently lifted (approximately 15 s after the finishing of mortar placing), and once the free flow stopped, the mortar sample was subjected to 15 table jolts at a rate of one jolt per second. This was followed by the measurement of the diameter of the resulting spread (measurements in two perpendicular directions were taken and the average was reported as the spread in mm). The initial and final setting times testing procedure was described in detail in a previous paper [19].

The volume of permeable voids (VPV) was assessed according to the standard procedure given in ASTM C642, using a procedure similar to that described in a previous paper [13]. The compressive strength development of the mortar mixes was assessed [20]. Three curing temperatures (20, 30 and 40 °C) for the first 24 h of curing were investigated. Afterwards samples were stored at 20 °C in the curing containers until testing.

Drying shrinkage was measured using 285 × 25 × 25 mm prisms using a length comparator and reference bar. At each length measurement, the mass of the specimens was also taken, and reported values are the average of readings obtained for 3 or 4 specimens. An initial reading of length was taken 24 h after casting the specimens which was immediately after demoulding. Some samples were immediately returned to the curing containers and others were moved immediately to the drying room, conditioned at 20 ± 1 °C and 50 ± 1% RH.

To study the effect of curing temperature, samples were cured at 20, 30 and 40 °C for 24 h until demoulding. After demoulding, initial readings were taken and samples were placed into the drying room. Subsequent readings were taken after 1 to 56 days of drying.

To study the effect of curing duration, samples were moved to the drying room at different intervals after casting (1, 7, 14 and 21 days). Before being moved to the drying room, they were cured at 20 ± 1 °C. Prior to drying, readings were taken every 7 days until the samples were moved to the drying room. Subsequent readings were taken.

Results and discussion

Fundamental properties of alkali-activated mortars

This measurement of the spread of the mortars was used to assess the workability of the different mixes. For S75/F25 the spread was 189 mm which increased to 226 mm for S50/F50 and eventually to 231 mm for S25/F75 as the fly ash content increased from 25 to 75%. Given that the water/solid ratio was 0.38, the increased workability as the fly ash content increased indicated that slag had a higher water demand than fly ash, which agrees with previous work [17, 18]. This may be related to the slag’s finer particle size. The individual particle morphology of slag and fly ash may also be a factor. Slag is composed of irregular angular particles whereas fly ash consists of spherical particles. Another factor is the
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Effect of slag/fly ash content

Figure 2 shows the mass change and drying shrinkage of the mortars. Figure 2a shows that as the fly ash content was increased the mass loss due to the evaporation of moisture increased from around 2.7% (S75/F25) to 6.0% (S25/F75). The drying shrinkage also increased with the fly ash content from around 7500 µε (S75/F25) to around 8100 µε (S25/F75) after 56 days of drying. It should be noted that the drying shrinkage of S25/F75 appears to have become stable after 56 days, whereas minor drying shrinkage appears to continue for S75/F25 up to 56 days. It was observed that in mixes with an increased fly ash content, drying shrinkage occurred more rapidly initially and stabilised more quickly. This could be due to the lower water demand of fly ash and more water remaining free in the system as the w/s ratio was 0.38 for all mixes. Another factor could be the lower strength of the mixes with increased fly ash content (Fig. 1) which may have provided lower resistance to tensile forces in the matrix due to the release and evaporation of moisture. The increased VPV observed for mixes with increased fly ash content may also be a factor in allowing the moisture to be released more quickly when drying commenced, compared with mixes with increased slag content.
Effect of curing temperature

Figure 3 shows the mass change and drying shrinkage of the mortars after being cured at 20, 30 and 40 °C for 24 h. For each slag and fly ash blend as the curing temperature was increased the mass change due to the evaporation of moisture decreased. The drying shrinkage also decreased. This can be linked to the more mature and stronger microstructure developed when the samples were cured at 30 and 40 °C. This is also illustrated by the increased compressive strength observed after curing at slightly elevated temperatures for 24 h (Fig. 1). These findings demonstrate the major impact that the initial curing temperature can have on drying shrinkage. S75/F25 underwent drying shrinkage of around 7500 µε after 56 days of drying. This decreased to around 5800 and 4600 µε when cured at 30 and 40 °C, respectively, for the first 24 h. This is a reduction in drying shrinkage of up to 40%. S25/F75 underwent drying shrinkage of around 8100 µε after 56 days of drying. This decreased to around 6200 and 5800 µε when cured at 30 and 40 °C, respectively, for the first 24 h, a reduction in drying shrinkage of around 28%. Therefore, the use of an increased initial curing temperature is effective at decreasing drying shrinkage within alkali-activated mortars regardless of the slag/fly ash proportions employed.

Effect of curing duration

Figure 4 shows the mass change and drying shrinkage of alkali-activated mortars after curing for different durations before commencement of drying. The samples that were cured at high RH beyond 1 day, continued to gain mass. This was likely due to ongoing hydration, geopolymerisation and the binding of water molecules within reaction products. As shown in Fig. 4a, c and e, when drying commenced the samples lost mass due to the evaporation of moisture, immediately and continued slowly thereafter. As the curing duration was increased the mass loss due to drying decreased. For each blend of slag and fly ash, the difference was most pronounced when the curing duration was increased from 1 to 7 days with less difference typically observed as the curing duration was increased to 14 and 21 days.

Similarly, when the curing duration was increased the drying shrinkage was also significantly reduced. Therefore, increasing the curing time can significantly improve the drying shrinkage behaviour. This is likely due, at least in part, to the more mature and higher strength matrix developed as the curing duration is increased.

These findings demonstrate the potential impact curing temperature and duration can have on the drying shrinkage of alkali-activated mortars. Drying shrinkage can be reduced by up to 40% when employing a particular curing temperature and up to 48% when employed a longer curing duration.

Conclusions

Alkali-activated binders are increasingly being seen as an alternative to conventional Portland cement based binders. It has been established that there remains some technical issues which appear to limit the widespread uptake of alkali-activated binders. One of these is their relative susceptibility to drying shrinkage. This paper examines some of the factors which influence the drying shrinkage of alkali-activated mortars containing slag and fly ash such as slag/fly ash proportion, curing temperature and curing duration. This information could be useful for researchers and industry practitioners wishing to use alkali-activated binders in full scale construction products. The following conclusions have been reached:

- The slag/fly ash ratio can be used to control setting time and mechanical strength of alkali-activated binders. In terms of drying shrinkage, mixes with increased slag content appear to undergo less drying shrinkage, at least during the early stages of drying.
eral weeks, the overall drying shrinkage remains less for mixes with increased slag content. A difference of around 600 µε was observed after 56 days of drying between mixes with 25% and 75% slag. The water demand of slag and fly ash is a key parameter that should be considered.

- The curing temperature can have a significant influence on the drying shrinkage of alkali-activated mortars. For mixes containing 75% slag, a curing temperature of 40 °C instead of 20 °C can reduce drying shrinkage by up to 40%. In the case of mixes containing 75% fly ash a 28% reduction in drying shrinkage was observed.
- The curing duration also had significant influence on drying shrinkage behaviour, to an even greater extent than the curing temperature. For mixes containing 75% slag, extending the curing duration from 1 to 7 days resulted in a 40% reduction in drying shrinkage. When the curing duration was extended further, to 21 days, a 60% reduction in drying shrinkage was observed. Similarly for mixes containing an increased fly ash content, the curing duration had a major impact on drying shrinkage behaviour. A 48% reduction in drying shrinkage was observed when the curing duration was increased from 1 to 21 days.

Fig. 3 a, c, e Mass change and b, d, f drying shrinkage of alkali-activated mortars cured at 20, 30 and 40 °C. a, b S75/F25; c, d S50/F50; e, f S25/F75
It was also observed that curing duration and curing temperature had more influence on the drying shrinkage of alkali-activated mortars than the proportion of slag and fly ash employed.

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**Data availability** The datasets generated during and/or analysed during the current study are available from the corresponding author on reasonable request.

**Declarations**

**Conflict of interest** The authors have no relevant financial or non-financial interests to disclose.

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References

15. A. Rafeet, Mix design, fresh and hardened properties and micro-structural characterization of alkali-activated concrete based on PFA/GGBS blends, PhD thesis (Queen’s University Belfast, 2016)

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