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Effect of curing environment on length changes of alkali-activated slag/cement kiln by-pass dust mixtures

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Abstract. This paper investigates the combination of commonly used blast furnace slag (BFS) and quite non-traditional cement kiln by-pass dust (BD) for the preparation of alkali-activated binder. More specifically, the possibilities of alkali-activated slag (AAS) shrinkage compensation and cracking mitigation using BD were studied. For this purpose, mixtures with ratios of BFS/BD ranging from 100/0 to 60/40 were prepared and cured in different environment, namely autogenous conditions, water curing, air curing and their combinations. The results showed that curing regime is a determinative parameter for performance of the mixtures: BD containing specimens showed a certain expansion during the first days of water curing leading to AAS shrinkage compensation, while in absence of water curing increasing dose of BD increased drying shrinkage significantly.

1. Introduction

Nowadays, it is a great challenge to develop and use some binders with lower ecological impact compared to traditional Portland cement (PC). The most common way is partial PC replacement by various industrial by-products like granulated blast furnace slag and fly ash. Nevertheless, these aluminosilicate precursors can also be used to produce non-cement binders thanks to the development of hydration products after mixing with alkaline solutions. Such alkali-activated binders can reach excellent mechanical properties, durability, resistance to elevated temperatures and relatively low heat of hydration [1, 2]. On the other hand, particularly the binders based on alkali-activated slag (AAS) suffer from severe shrinkage and cracking.

Several possibilities of AAS shrinkage reduction have been studied. Very common is, for example, alkaline activation of slag/fly ash blends, which is advantageous not only from the viewpoint of shrinkage, but also setting time. However, this is usually connected with the decrease of strengths, particularly when cured at room temperatures [3]. Moreover, introducing of nitrogen oxides reductions technologies to power plants result in the presence of ammonium sulfate and bisulfate salts [4] which are converted to ammonia under alkaline pH or can be leaching issues and thus such fly ashes are not usable even in common PC-based concretes.

Other industrial by-product with potential to reduce or rather compensate AAS shrinkage is cement kiln by-pass dust (BD). The role of rotary kiln by-pass is to take away part of the kiln gases rich in alkali chlorides and sulfates and thus prevent formation of coatings on kiln walls that can even lead to its blockage as well as to reduce chloride content in clinker. Therefore, BD is rich in chlorides and sulfates,



but also in particles of partially burned raw meal, particularly free CaO. This means that BD would act as an expansive agent (EA).

For PC-based concretes, the role of EAs is well understood [5, 6], while only a few papers focused on the use of EAs in AAS have been published. Authors in the paper [7] used two forms MgO differing in their reactivity and observed that their effect depended on their nature. The more reactive (faster) MgO reduced drying shrinkage up to 25% during the first days but its effectiveness decreases with increasing time, while the less reactive MgO started having effect on shrinkage after 15 days. Nevertheless, maximum shrinkage reduction was only 13%. Shrinkage reduction was attributed to the formation of hydrotalcite, while in another study [8] beneficial effect of MgO related to the brucite formation. Also EA containing quick lime, periclase and anhydrite was investigated [9]. Shrinkage was reduced by up to 75% for high doses of EA, but increasing dose of EA simultaneously led to significant acceleration of setting time and workability decrease.

It can be seen that some more or less successful attempts to reduce AAS shrinkage using EAs have been done. This paper pioneers the possibilities of the use of BD as EA which would be beneficial from the viewpoint of both AAS shrinkage reduction and wider utilization of BD.

2. Experimental part

2.1. Materials and mixtures composition

Ground granulated blast furnace slag from Czechia (Kotouč, spol. s r. o.) with Blaine fineness of 400 m²/kg was used as a main part of a binder. As determined using XRD with Rietveld evaluation method, it contained about 90% of amorphous phase and some merwinite and akermanite. Slag was partially replaced with BD produced in Slovakia (cement plant Horné Srnie, Cemmac, a.s.) which also consisted mainly of amorphous phase (~44%), free CaO (~16%), KCl (~20%), larnite (~10%) and arcanite (~9%) and traces of quartz and alunite. Chemical composition of both slag and BD as determined using X-ray fluorescence (XRF) is given in table 1.

Table 1. Chemical composition of slag and BD as determined using XRF.

	CaO	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	MgO	Na ₂ O	K ₂ O	SO ₃	Mn ₂ O ₃	TiO ₂	P ₂ O ₅	Cl
slag	39.6	38.9	8.9	0.27	8.8	0.39	0.40	1.4	0.83	0.32	0.01	–
BD	25.9	6.5	2.5	1.6	0.48	0.44	17.1	5.2	–	0.14	0.08	10.3

Waterglass with silicate modulus of 2.0 was used to alkali-activate slag/BD blends with ratios ranging from 100/0 to 60/40 (or even 0/100 in the case of setting time). The dose of waterglass was adjusted to 8% with respect to binder (slag + BD) weight. Siliceous sand with maximum grain size of 2 mm was used as a fine aggregate. Its weight was three times higher than the weight of the binder. Water to binder ratio including water from waterglass was 0.35 and 0.46 for pastes and mortars, respectively.

2.2. Specimens preparation and testing

2.2.1. Mixing. Mixing procedure was based on that standardized one for testing of PC in EN 196-1. First of all, waterglass and water were homogenized, then the binder was added and after 30 s addition of sand in the case of mortars followed. Total mixing time was four minutes.

2.2.2. Workability and setting time. Just after the mixing of mortar, workability was tested using a jolting table and expressed as a spread diameter after lifting the truncated cone with lower base of 100 mm as well as after 15 jolts of the table. Unlike workability, initial and final setting time was determined on pastes using Vicat needle apparatus.

2.2.3. Strength development. After the mixing, the mixtures were cast into the steel molds to prepare prisms with dimensions of 40 × 40 × 160 mm further used for flexural and compressive strength testing

after 24 hours, 7 days and 28 days. After 24 hours, specimens were demolded and sealed with polyethylene foil to ensure curing in autogenous conditions, i.e. to prevent the mass exchange between specimens and ambient laboratory climate (23°C, 60% relative humidity). Two specimens of each mixture at each age were tested, i.e. two values of flexural and four values of compressive strength were obtained and averaged.

2.2.4. Length changes. The same procedure was used to prepare prisms with dimensions of $25 \times 25 \times 285$ mm for length changes determination, but specimens were demolded earlier, after 5 hours, to start the length changes measurement as soon as possible. At the age of 24 hours, some of the specimens were unsealed and immersed in water or left to dry at ambient laboratory conditions (23°C, 60% relative humidity). Also combination of water and air curing was tested starting with water curing for 1, 3 and 7 days followed by air curing. Length changes were determined using ASTM C490 apparatus.

3. Results and discussion

Effect of partial slag replacement by BD on setting time and workability is given in figure 1. It can be seen that while workability is not significantly affected by various slag/BD ratios up to 60/40, both initial and final setting time slightly decrease at low replacement levels (up to 10% BD), then they somewhat increase with increasing replacement levels up to 30% of BD and after this threshold value, they rapidly decrease. The use of EAs in AAS is often accompanied with significant acceleration of the setting time and early loss of workability [9] and therefore no or slight setting time acceleration with BD doses below 30 or 40% is desirable. Although the described behavior appears strange, the same trends were observed during the repeating of this experiment so it is not a random error. It is related to the chemistry of BD since, according to our experience, also rapid setting even for low doses of BD can be observed. It depends on the given BD batch composition which can be highly variable even for the same source of BD. Acceleration of setting time in presence of BD was expected due to presence of high contents of free CaO in BD which suggest consequent formation of portlandite. Accelerated setting time related to portlandite and ettringite formation has already been reported for slag/BD systems [10].

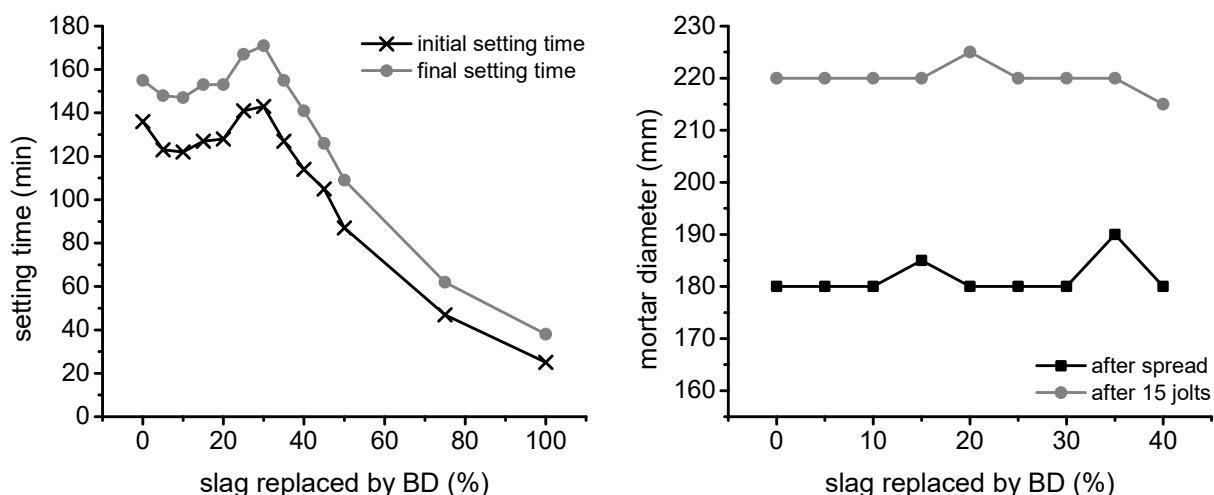


Figure 1. Effect of slag replacement by BD on and workability of AAS-based mixtures.

Gradual decrease in compressive strength with increasing amount of incorporated BD is obvious from figure 2. Quite similar trend can also be seen for flexural strength, but lack of statistics and naturally scattered values limits further discussion. The main reason for decrease in compressive strengths with increase of BD is simultaneously decreasing slag amount and thus amount of amorphous aluminosilicate phase needed for C-S-H formation. Furthermore, formation of other hydration products without strong binding properties is possible, e.g. chloro-aluminate-hydrates as was reported for PC/BD mixtures [11].

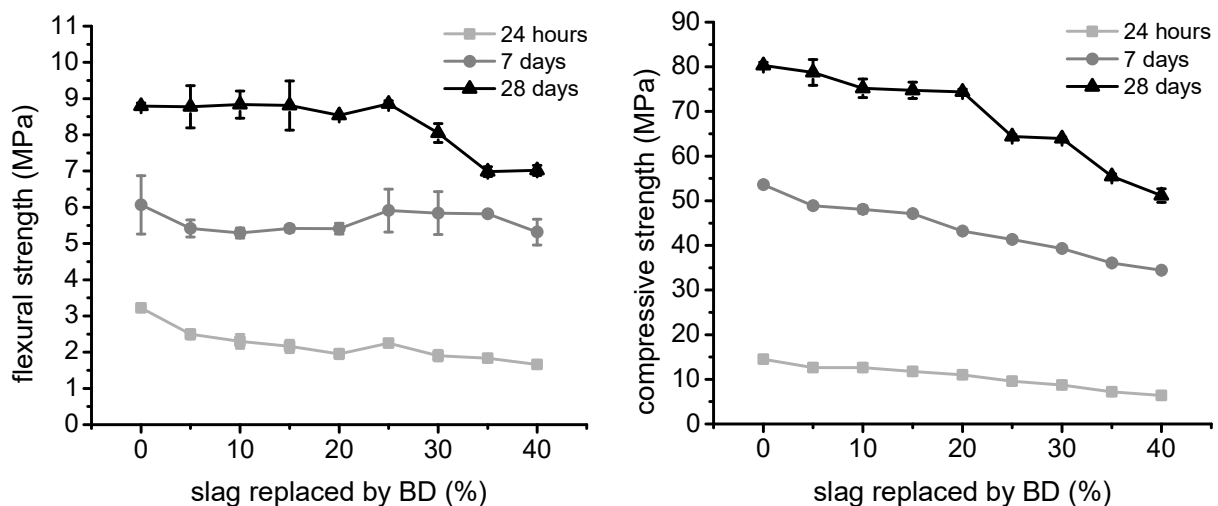


Figure 2. Effect of slag replacement by BD on strength development of AAS-based mixtures.

Length changes of AAS/BD mixtures depending on the curing environment are given in figure 3. It should be noted that due to high number of mixtures and curing regimes only one specimen of each mixture at each curing environment was used. Also, in some cases, some specimens were broken during the demolding which results in missing data.

Nevertheless, the obtained results show that curing environment plays a crucial role in dimensional stability of the prepared mixtures. In autogenous conditions only, some shrinkage reduction for BD incorporating mixtures was recorded, but no expansion. If air curing is applied after 24 hours of autogenous conditions, presence of BD resulted in increased drying shrinkage of AAS mortars and it seems that the higher the BD dose, the higher the drying shrinkage in this case of curing.

Furthermore, different length of water curing before the start of drying was applied. It can be seen that one day of water curing led to results similar to those without water curing, i.e. increasing drying shrinkage with increasing dose of BD. However, if water curing period was prolonged to three or six days, noticeable expansion of mortars, particularly those with 30% and higher amounts of BD, was achieved. This led to a decrease in total shrinkage during the subsequent drying stage compared to the mixtures with lower or no BD addition which tend to shrink even immersed in water. It seems that three days of water curing are enough to finish the expansion but prolonged water curing (six days) is beneficial during following drying stage due to reduced drying shrinkage.

From the observed trends, it is clear that the amount of mixing water is not sufficient for the formation hydration products responsible for volume expansion of the specimens and therefore the expansion was observed only if the specimens are surrounded by additional water. This is in line with general knowledge of EA in PC-based materials [5, 6]. Increased drying shrinkage of AAS by BD observed in the cases of insufficient curing before the start of drying would be related to the changes in the hydration process leading to a decrease of C-S-H phase content and thus reduced mechanical properties, particularly elastic modulus, as well as possible changes in porosity that play key role in shrinkage processes. These aspects will be the object of further study. Now, it can be noted that it was reported that the application of EAs in AAS can lead to portlandite or ettringite formation [10, 12], but additionally to these phases also monosulfoaluminate and Friedel's salt were found in slag/BD systems [13].

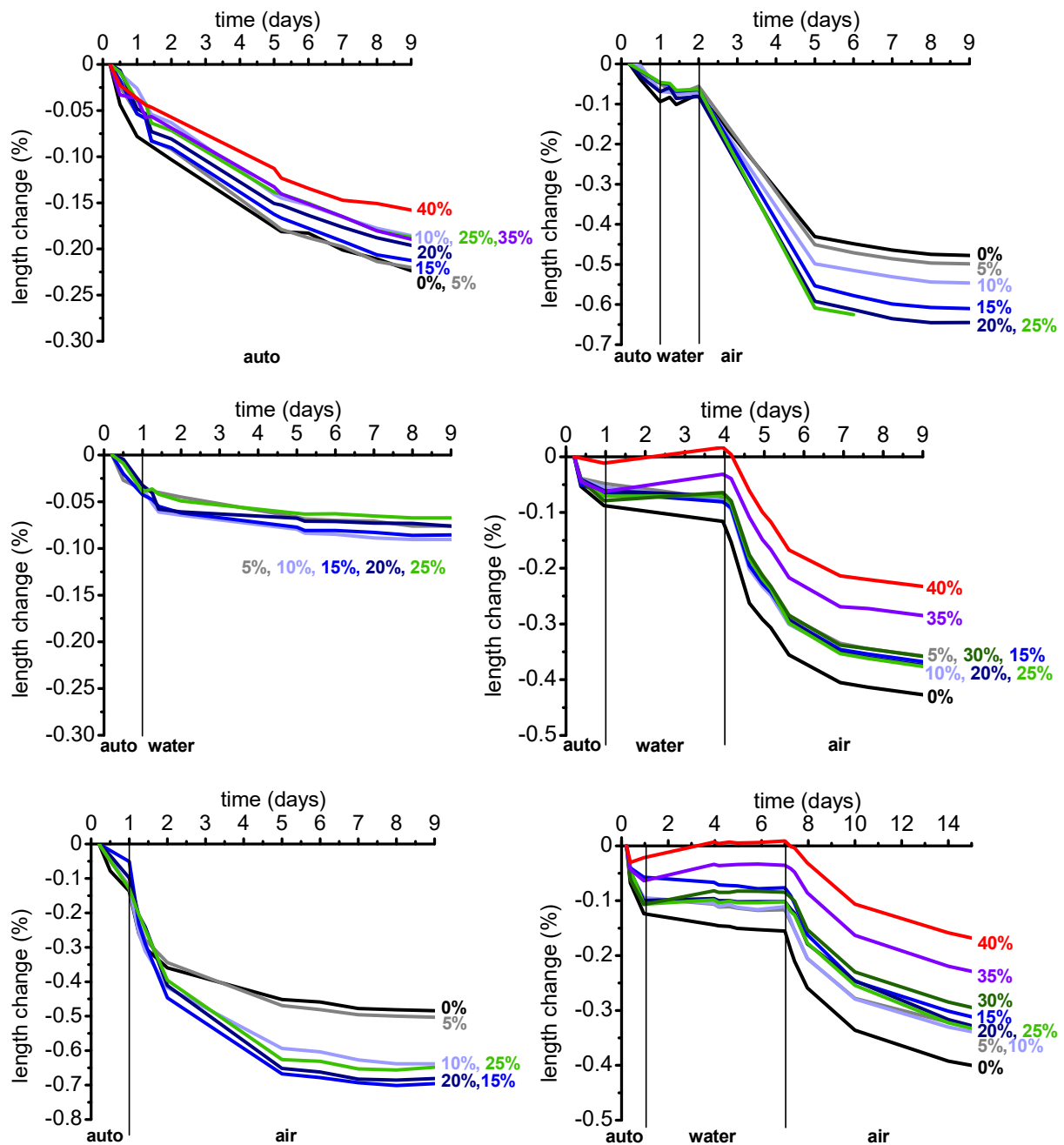


Figure 3. Effect of slag replacement by BD (in wt. %) on length changes development of AAS-based mixtures at various curing regimes.

4. Conclusions

This paper investigated the expansion potential of mixtures with various slag/BD contents in various curing conditions. It was observed that curing regime strongly affected the resulting trends in length changes development. It was found that doses of BD exceeding 25% and sufficiently long period of water curing were needed to develop some expansion which then caused the reduction in total shrinkage during the following air drying. If too short (24 hours) or no water curing was used before air curing, increasing dose of BD resulted in increasing drying shrinkage. Based on these results, BD can be used as an expansive agent in AAS. Increasing dose of BD also resulted in decreasing strengths in autogenous conditions and not very significant changes in setting time until the threshold value (30%) after which strongly accelerating effect of BD was observed.

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