Shrinkage of the alkali-activated slag mortars containing alternative activator

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Abstract. The paper deals with the experimental determination of the shrinkage process of the alkali-activated slag mortars (AASM). Two mortars which differed especially in the type of used aggregate were mixed for the experiment. The slag-aggregate ratio was 1:3 for both mortars. The waste sludge, obtained from the water glass production, was used as an activator. The desired workability of the fresh mixtures was reached by addition of water. Because of the high water demand of waste sludge, the water-slag coefficient (w/s) was relatively high – 0.98 for the mortar with the natural sand and 0.73 for the mortar with the standard sand. All test specimens were removed from the moulds at the age of 24 hours and left to dry freely in the laboratory conditions with a temperature of 21 ± 2°C and relative humidity of 50 ± 10% during the whole time of measurement. The results show that AASMs which contain waste sludge exhibit rapid shrinkage with a high magnitude independent on the type of aggregate. During the first 40 hours of ageing, both mortars reached approx. 50% of the final value of shrinkage. The high shrinkage was strengthened by a high w/s coefficient needed to maintain the workability of the fresh mixtures.

1. Introduction
The research of utilization of industrial byproducts in building industry is a very actual topic around the world due to severe ecological and economical demands of the production of Portland clinker (PC). One traditional and widely used possibility is blending of PC with secondary raw materials such as slag and fly ash to produce blended cements. Another option is the production of wide range of non-cement alternative binders [1].

Alkali-activated materials (AAMs) have potential to replace PC-based materials in some applications because of their mechanical properties or durability [2, 3]. However, they suffer from shrinkage and related high cracking tendency, particularly those based on alkali-activated slag (AAS). It was reported that the drying shrinkage of AAS is several times higher compared to PC-based materials [4–7]. Moreover, the cost of alkaline activators can also be an issue as well as their usually non-ecological production.

The solution can be using of some alternative activators. In this study, the waste sludge from water glass production was applied. Because of significant amounts of dissolved alkalis and silicates, it is dangerous waste and its disposal is very expensive. On the other hand, it has noticeable potential for alkaline activation. Moreover, it contains significant amounts of solid particles and rest of siliceous sand.
used for water glass production, which would save natural fine aggregate. Therefore, this study is focused on physical-mechanical characterization of mortars where only this alternative activator was used for slag activation and simultaneously as a partially replacing material for natural and standard siliceous sand.

2. Experiment details

The pilot study presented herein is a part of the comprehensive pilot study which main objective is to obtain a start-up information about physical, mechanical and fracture characteristics of slag mortars activated by a waste sludge produced in the Czech Republic. This article focuses especially on the shrinkage process which is a general and still actual problem in all cases of the alkali-activated slag materials.

2.1. Materials

Ground granulated blast furnace slag with predominant content of amorphous phase and Blaine fineness of 400 m²/kg was used as a precursor for AAS-based mortars. Chemical composition of slag is given in Table 1. It was activated by the waste sludge from water glass production which contained 4.1% Na₂O, 7.3% soluble SiO₂, 16.5% water and 72.1% of non-reacted siliceous sand. The dose of activator was adjusted to 8% Na₂O with respect to the slag weight. Two kinds of mortars with a sand to binder ratio of 3:1 were prepared. The mortars differed in the type of the used sand. For one mortar, natural sand with grain size up to 4 mm was used, and the second one was formulated using standardized CEN siliceous sand with maximum grains of 2 mm consisting of three fractions according to EN 196-1 [8]. The type of sand was used to distinguish between the two mortar series: N (natural) and S (standard).

The water to slag (w/s) ratio including water from activator was 0.98 for N mixture and 0.73 for S mixture. Such high values of the water to slag ratio are caused by high water demand of the solid particles in an activator since they are relatively fine and porous. These solid particles introduced into the mixtures within the activator were also calculated to the dose of the above mentioned sand to binder ratio, i.e. they partially replaced the used sands. For S series, they replaced the whole finest fraction and a part of the middle one of the CEN sand, while for N series its total amount was reduced accordingly. Replacement of fine fraction of CEN sand was preferred because its particle size distribution is the closest to relatively fine particles from activator (see Figure 1). It was possible because, contrary to natural sand, individual fractions of CEN sand were weighed separately.

![Figure 1](image-url). Particle size distribution of the used sands as determined using laser diffraction (combined with sieve analysis for particles higher than 1 000 µm).
### Table 1. Chemical composition of slag as determined using X-ray fluorescence.

|       | CaO  | SiO₂ | Al₂O₃ | Fe₂O₃ | MgO  | Na₂O | K₂O  | SO₃  | Mn₂O₃ | TiO₂ | P₂O₅ |
|-------|------|------|-------|-------|------|------|------|------|-------|------|------|
| Value | 39.6 | 38.9 | 8.9   | 0.27  | 8.8  | 0.39 | 0.40 | 1.4  | 0.83  | 0.32 | 0.01 |

#### 2.1.1. Specimens preparation and curing.
Mixing was performed using Hobart mixer and took approximately 8 minutes. First, the activator and water were mixed together to prepare homogenous activating solution. Then, the slag and finally the sand were added. The fresh mortar was then tested in terms of consistency using a flow table test with a base diameter of 100 mm, density and air content using the device according to EN 1015-7 [9]. The mortar was cast into the polyethylene moulds (Hakorite) for preparation of prisms with dimensions of 40 × 40 × 160 mm and sealed for 24 hours at a temperature of 21 ± 2 °C. Then the specimens were demoulded and stored in an air conditioned room where the temperature of 21 ± 2 °C and relative humidity of 50 ± 10% were maintained. The informative compressive strength was determined for both mortars at the age of 28 days (see Figure 2b). The test procedure follows the standard EN 196-1 [8].

#### Table 2. Basic properties of fresh and hardened mortars: Mean value, (SSD).

| mixture | fresh state | hardened |
|---------|-------------|----------|
|         | spread (mm) after lifting the cone | 15 jolts | bulk density (kg/m³) | air content (%) | bulk density (kg/m³) | f₈₂₈days (MPa) |
| N       | 120         | 170      | 2070 | 4.2    | 1900 (10.7) | 14.9 (0.53) |
| S       | 160         | 235      | 2130 | 3.2    | 1990 (16.5) | 17.9 (0.30) |

*The air content was measured for both mortars at the start and at the end of their pouring into the moulds. Both mortars exhibited a decrease in the air content from 4.8 to 3.6 % for mixture N and from 4.0 to 2.4 % for mixture S.*

#### 2.2. Measurement of shrinkage and mass losses
The shrinkage process of the AAS mortars was determined based on the measurement of the length changes of the prismatic specimens with dimensions of 40 × 40 × 160 mm equipped with the special markers embedded into the ends of the test specimens (in the longitudinal axis of the specimens). The measurement was started right after demoulding of the test specimens at the age of 24 hours.

The changes in length were measured in regular intervals using the measurement gauge equipped with the digital probe with resolution of 0.001 mm (see Figure 2c).
Simultaneously, the actual mass of each test specimen was registered using the laboratory balance with resolution of 0.5 g. The changes in the mass were used for determination of the process of mass losses which is one of the indicators used for prediction of the drying shrinkage process.

All measurements were performed in an air-conditioned laboratory with a temperature (T) of 21 ± 2 °C and relative humidity (RH) of 50 ± 10%. Three test specimens made from mortar N and S were taken for determination of the shrinkage and mass losses process.

3. Results and discussion

The results of measurement are shown in the following figures. The steady-state values are related to the conditions maintained in the laboratory during measurement (T = 21 ± 2 °C; RH = 50 ± 10%).

Figure 3 shows the process of shrinkage determined for mortar N and S. Both figures display results obtained from three independent measurements. As shown, the absolute value of shrinkage is very high for both mortars with the maximum of about 16.100 mm/m and 16.700 mm/m for mortar N and S, respectively. The high value of shrinkage is related to a high w/s ratio which is needed to maintain the workability of the mortars. In spite of the fact that the w/s for mortar S was about 25% lower in comparison to the mortar N, the final steady-state values of shrinkage are almost identical.

The differences in the shrinkage process of both mortars can be seen during the first 7 days of the measurement, when the mortar S started to shrink rapidly approx. 1 day after demoulding of the test specimens (see Figure 4a).

The reason for the differences in short-term shrinkage behavior would be different porosity and pore size distribution of the prepared mortars, which is supported by the different w/s ratios, air content (Table 2) as well as the mass loss (Figure 4b). The combination of these parameters suggests that mortar N had higher content of larger pores and more permeable structure which resulted in more intense drying during the first several days and simultaneously in lower shrinkage extent. After this stage, drying of finer pores inside the mortar N accompanied with higher shrinkage took place and consequently the differences in the shrinkage extent of both tested mortars partially diminished after seven days.

The cracks formation is another factor which influences the shrinkage process. The formation of the cracks is reflected in the shape of shrinkage curve as retardation of the shrinkage. The wider the cracks are, the more visible effect on the curvature of shrinkage is recorded. To verify the existence of the cracks in the internal structure of both mortars, a visual inspection of the test specimens subjected to the quasi-static fracture test of the specimens with an initial notch was made. The visual inspection confirmed the existence of the macro-cracks in specimens made from mortar N (see Figure 5a). No visible cracks were observed in the case of specimens made from mortar S (see Figure 5b).
Figure 4. Initial process of shrinkage (a) and mass losses (b) for mortar N and S.

Figure 5. Cracks in the internal structure of the specimens made from mortar N (a).

The process of mass losses displayed in Figure 6 shows that the steady-state value is about 10.5% and 8.9% for mortar N (with higher w/s) and S, respectively. The difference in mass losses of the mortars is about 16%.

Differences in the process of shrinkage ($\varepsilon$) and mass losses ($\Delta m$) is also visible from the ($\Delta m/\varepsilon$)-curve displayed for both mortars in Figure 7. There is an observable phenomenon, in the time interval from 25 to 53 hours after specimens demoulding, when the ($\Delta m/\varepsilon$)-curve changes the shape due to the retardation of shrinkage in the case of mortar N.
4. Conclusions
The results of the pilot study show particular problems which can occur during the production and maturation of the mortars made from slag activated by the waste sludge from water-glass production.

The results show that the high water demand of the waste sludge due to the relatively high content of fine and porous particles is one of the main disadvantages of this materials. The high w/s ratio affect negatively the pore size distribution, process of desiccation and shrinkage. The process of desiccation is accompanied also by the cracks’ formation, which influences the strength parameters. The shrinkage is for at least 10 times higher than in the case of ordinary cementitious mortars. The results of this study serve as the basis of the ongoing research which focuses especially on the ways for decreasing the w/s and shrinkage value at the preservation of required workability during the manufacturing process.

Acknowledgments
This work was supported by the Czech Science Foundation [project No. GA17-14302S and No. 18-12289Y].
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