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Polyethylene glycol molecular weight as an important parameter affecting drying shrinkage and hydration of alkali-activated slag mortars and pastes

Vlastimil Bílek Jr. ^{*}, Lukáš Kalina, Radoslav Novotný

Brno University of Technology, Faculty of Chemistry, Materials Research Centre, Purkyňova 464/118, Brno CZ-612 00, Czech Republic

H I G H L I G H T S

- Increasing glycol dose had minor effect on strength while significant on shrinkage.
- Longer molecules reduced drying shrinkage of AAS more than short molecules.
- Shrinkage was reduced thanks to surface tension decrease and porosity changes.
- Ethylene glycol reduced both 2nd and 3rd peaks of AAS calorimetric curve.
- 3rd AAS calorimetric peak reduction increased with increasing molecular weight.

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The aim of this study was to explore the influence of ethylene glycol in a wide range of polymerization degree, i.e. from monomer (EG) up to polyethylene glycol (PEG) with molecular weight of about 35,000, on properties of alkali-activated slag mortars. Changes in molecular weight and dose of all tested glycols had only minor effect on compressive strength, while drying shrinkage was significantly affected by both these factors. EG had negligible impact on drying shrinkage, but other tested glycols reduced it significantly with PEG2000 and PEG10000 being the most effective. Such different shrinkage behavior was attributed to the changes in surface tension and pore structure. All tested glycols reduced the total heat released during the hydration and interestingly modified the heat flow depending on their molecular weight.

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1. Introduction

Materials based on alkali activated slag (AAS) can achieve excellent mechanical properties and durability, but their wider utilization is limited by significant drying and autogenous shrinkage, the values of which are usually considerably higher compared to those of OPC-based materials [1]. Therefore, many different approaches focused on the shrinkage reduction have been published. From possible solutions, partial replacement of slag by other materials such as fly ash or silica fume [2], prolonged the

curing period [3], heat curing [4], and the use of expanding [5] or shrinkage reducing admixtures [6–9] can be depicted.

Shrinkage reducing admixtures (SRAs) were introduced in 1980s to mitigate the drying shrinkage of OPC concretes [10]. Although several products are available on market, only rare studies dealing with their application in AAS systems have been published. Palacios and Puertas [6] studied the effect of SRA based on polypropylene glycol (PPG) on the properties of slag activated by waterglass with the silicate modulus of 1.0–1.2 and the dose of 4% Na₂O by mass of slag. SRA had rather beneficial effect on the flexural strength and little or no effect on the compressive strength, it also slightly retarded the hydration of slag, but later the precipitation of CSH was more intense in pastes containing SRA. SRA based on PPG also reduced the drying shrinkage of AAS after 180 days at 50% relative humidity (RH) by 7 and 35% for 1

^{*} Corresponding author.

E-mail addresses: bilek@fch.vut.cz (V. Bílek Jr.), kalina@fch.vut.cz (L. Kalina), xcnovotny2@fch.vut.cz (R. Novotný).

and 2% of SRA, respectively. The same doses of SRA were markedly more efficient at 99% RH, where the shrinkage reduction was 50 and 80%, respectively. Eberhardt [10], who studied OPC-based mortars, explained different efficiency of SRA depending on RH as the consequence of an increase of interfacial area during the drying, on which SRA molecules have to act.

The action of SRA based on PPG in AAS was studied also by Bilim et al. [7]. The silicate modulus of waterglass and sodium hydroxide mixture was 0.75 and 1.0, while its dose was 5.4 and 8.1% Na₂O by mass of slag and the dose of SRA was 1.0%. Regardless the type of curing and the dose and modulus of activator, the long-term drying shrinkage was reduced generally by 15–30%, although during first few days of drying, the shrinkage reduction was significantly higher, particularly for the specimens which were heat cured in water before exposing to the atmospheric conditions. Similar shrinkage performance of AAS, at least from the long term point of view, was also reported for the activation by solid sodium silicate [8]. Generally, SRA did not have significant effect on the strength and setting time of AAS, but some exceptions like prolonged setting time in the case of mixtures with lower activator dose could be found.

Besides SRAs, other organic admixtures are also able to modify the shrinkage behavior of AAS. Slight shrinkage reduction was reported for the set retarding and water reducing admixture by Bilim et al. [7,8]. Bakharev et al. [9] reduced the long-term drying shrinkage of AAS concrete by 30% with lignosulphonate plasticizer and even by 75% with alkyl aryl sulphonate-based air entraining agent, while an unspecified SRA reduced the drying shrinkage by 67%. On the other hand, the application of superplasticizer based on modified naphthalene formaldehyde polymers resulted in the shrinkage increase by more than 100% after seven days and by almost 40% after 224 days.

Based on this brief review, we can conclude that SRAs and other chemical admixtures can lead to significant decrease in shrinkage of AAS-based materials. However, its values are still higher compared to those of OPC without SRAs in most cases. Moreover, some studies [11,12] revealed that common commercially available SRAs can retard the hydration of AAS by several days even at relatively low doses (1%) and, in this case, it is likely that observed shrinkage reduction was caused by coarser pore structure, which resulted from the retardation of slag hydration. SRAs are generally based on lower monoalcohols and diols, while promising results were obtained for SRA based on polymers (polypropylene glycol), as was discussed above. Therefore, it was our interest to investigate the effect of molecular weight of such organic compounds, e.g. polyethylene glycol which was used in this study.

2. Materials and experimental procedures

2.1. Materials and sample preparation

Ground granulated blast furnace slag with specific surface area of about 400 m²/kg was used as an aluminosilicate precursor for alkaline activation by liquid sodium silicate with SiO₂-to-Na₂O molar ratio of 2.1 at the dose expressed as 8% Na₂O by slag weight. Ethylene glycol (EG) and polyethylene glycol with molecular weight of about 400 (PEG400), 2000 (PEG2000), 10,000 (PEG10000) and 35,000 (PEG35000) were used in amounts corresponding to 1.0 and 2.0% of slag weight. The type and amount of these chemicals was also used for the designation of prepared mortars and pastes, and those, which contained no additive, were marked as Ref. The water-to-slag ratio (w/s) was adjusted to 0.35 for pastes and to 0.42 for mortars. The siliceous sand with the maximum grain size of 2 mm was used as a fine aggregate in the

amount three times higher than the weight of slag for the mortars preparation.

At the beginning of the mixing procedure, all the liquid components were mixed together. EG and PEG400 are liquids at laboratory conditions, while longer PEGs are solids; therefore they were dissolved in water before combining it with activator. The longer the PEG was the more time was needed for its dissolution with PEG10000 and particularly PEG35000 being time consuming. After combining aqueous solution of PEG with waterglass, the immiscibility of all PEGs and activating solution was observed. The mixing procedure was performed in accordance with the EN 196-1 standard, fresh mortar properties were determined (see Section 2.2) and the mortar was cast into the molds with dimensions of 25 mm × 25 mm × 285 mm for the drying shrinkage and strength testing. After 24 h of moist curing, the specimens were demolded and cured according to specific testing procedure.

2.2. Fresh mortar properties

Mortar consistency was determined using flow table test. After the lifting the steel truncated cone with a lower diameter of 100 mm mortar diameter after consequent spread as well as after 15 table strokes was measured. Mortar density and air content were determined using FORM + TEST Seidner & Co., Ltd. air content tester with a vessel volume of 1 dm³.

2.3. Drying shrinkage tests

The drying shrinkage was evaluated as the average of length changes of three mortar bars for each mixture measured with the ASTM C490 apparatus. Unlike the specimens for the strength testing, the drying shrinkage specimens were removed from water after three days and stored at the laboratory conditions (approximately 50% relative humidity and 23 °C). Their length changes during drying were measured until the age of 28 days.

2.4. Compressive strength tests

Compressive strengths were tested on the same type of specimens as were used for drying shrinkage tests, i.e. with dimensions of 25 mm × 25 mm × 285 mm. Unlike the shrinkage tests, specimens for compressive strength testing were cured immersed in water until their testing. Compressive strength values were obtained after 1, 7 and 28 days on the broken parts of mortar bars on which the flexural strength tests were done.

2.5. Isothermal conduction calorimetry

The effect of EG and PEGs on the hydration of AAS at 25 °C was studied by means of isothermal conduction calorimetry. For each measurement, 4.0 g of slag were used and the dose of activating solution was adjusted to w/s ratio of 0.35 and 8% Na₂O. The admixtures were added together with the activating solution. Slag and the mixture of liquid components were tempered separately inside the TAM Air (TA Instruments) calorimeter. The measurement started immediately after the addition of the mixture of liquids to slag. The paste was homogenized by mechanical stirring during the first three minutes of measurement.

2.6. Mercury intrusion porosimetry

Mercury intrusion porosimetry (MIP) tests were carried out using paste bars with dimensions of approximately 5 mm × 5 mm × 12 mm. These small bars were prepared from larger ones by sand paper abrading at the age of four days after the same curing regime as was used for mortar bars on which shrinkage was

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