



Effect of elevated temperature curing on properties of alkali-activated slag concrete

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Abstract

This investigation is focused on the effect of curing temperature on microstructure, shrinkage, and compressive strength of alkali-activated slag (AAS) concrete. Concrete prepared using sodium silicate and sodium hydroxide as the activator had greater early and flexural strength than ordinary Portland cement concrete of the same water/binder ratio, but it also had high autogenous and drying shrinkage. Heat treatment was found to be very effective in reducing drying shrinkage of AAS concrete and promoting high early strength. However, strength of AAS concrete at later ages was reduced. Microstructural study revealed an inhomogeneity in distribution of hydration product in AAS concrete that can be a cause of strength reduction. Pretreatment at room temperature before elevated temperature curing further improved early strength and considerably decreased shrinkage in AAS concrete. © 1999 Elsevier Science Ltd. All rights reserved.

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1. Introduction

Use of alkali activated slag (AAS) in concrete manufacturing has environmental benefits because its production requires less energy than ordinary Portland cement (OPC) and utilises industrial by-products. AAS concrete has superior durability in aggressive environments compared to OPC [1–4]. However, it was found that AAS has considerably higher shrinkage than that of OPC concrete [5]. Previous investigation showed the effect of admixtures on workability, strength, and shrinkage of AAS concrete [6]. It was found that admixtures developed for OPC are not effective in the case of AAS and have some side effects such as reduced strength. The focus of the current investigation is on study of the effect of heat treatment. The aim is to determine how curing temperature influences shrinkage and compressive strength of AAS concrete. Compared with OPC, AAS has a benefit of early strength development. This property is important for precast concrete manufacturing because it permits a short cycle. Curing at elevated temperature may further accelerate strength development in AAS concrete. The

study on OPC concrete showed that heat treatment provided significant improvement in volume stability [7]. Thus it can be expected that AAS will have a benefit of low shrinkage.

2. Methods

2.1. Slag

The chemical composition and properties of the slag used are summarised in Table 1. The blast furnace slag (SteelCement Ltd., Port Melbourne, Australia) is a granulated product ground to fineness of about 460 m²/kg, with the particle size range of 1–10 µm, and is neutral with the basicity coefficient $K_b = (\text{CaO} + \text{MgO})/(\text{SiO}_2 + \text{Al}_2\text{O}_3)$ equal to 0.93. The slag is supplied with 2% blended gypsum.

2.2. Activators

The activators investigated were liquid sodium silicate (PQ Australia Ltd., Dandenong, Victoria, Australia; sodium silicate solution grade D; wt. ratio: $\text{SiO}_2/\text{Na}_2\text{O} = 2$, % $\text{Na}_2\text{O} = 14.7$, % $\text{SiO}_2 = 29.4$; technical grade) and sodium hydroxide solution (Ajax Chemicals, Auburn, NSW, Australia; 60% w/v water solution; technical grade). Liquid sodium silicate and sodium hydroxide were blended, providing the modulus in

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Table 1
Composition of slag

Oxide	Slag (% oxide)
SiO ₂	35.04
Al ₂ O ₃	13.91
Fe ₂ O ₃	0.29
CaO	39.43
MgO	6.13
K ₂ O	0.39
Na ₂ O	0.34
TiO ₂	0.42
P ₂ O ₅	<0.1
MnO	0.43
SO ₃	2.43
Sulphide sulphur as S ²⁻	0.44
Cl	80 ppm
Loss on ignition	1.45

solution (mass ratio of SiO₂ to Na₂O), M_s , equal to 0.75, and 4% Na (mass of slag). Previous investigations of Collins and Sanjayan of slag activated by powdered sodium silicate utilised activator in the dry powder state, together with hydrated lime [8]. The current study does not include lime for slag activation.

2.3. Concrete mixes

Table 2 shows mix designs of AAS concrete specimens. The water to binder (w/b) ratio of AAS was fixed to 0.5 to enable comparison and reasonable concrete workability. OPC samples of Gr 60 with w/b = 0.45 were used for comparison purposes since OPC Gr 40 does not provide high early strength. Mixing of concrete was performed in a 70-L mixer. The sequence of mixing was as follows: mix for 2 min, rest for 2 min, followed by remixing for 2 min. Activators were added in water and the chemical admixture was added into the concrete mix.

2.4. Testing procedures

Table 3 shows the experimental programme including a summary of specimens and tests. Elevated temperature curing was utilised in experiments with OPC and AAS concrete. Two methods of temperature exposures were considered. One utilised a water bath in which the temperature was stabilised at 70°C before the experiment. The cast cylinders (100 × 200 mm) were placed in plastic bags and immersed in the bath for 6 h, during which time the water temperature was maintained at 70°C. After that the heating element was turned off and concrete continuously cooled down to room temperature. In the other method, the concrete samples had a precuring period of 2 h at room temperature when they were placed under a tarpaulin cover after casting; after that, the cylinder samples were placed in plastic bags and immersed in a water bath at 23°C. The temperature in the bath was ramped to 65°C for 3.5 h and after that it was stabilised for 5 h at that level. Then the heating element was turned off and the concretes continuously cooled down to room temperature.

Compressive strength testing was conducted on cylinders (100 × 200 mm), and a total of three cylinders were tested for each data point. Shrinkage measurement was performed using concrete shrinkage prisms 75 × 75 × 285 mm. A minimum of two shrinkage prisms were used in each test. The first reading was taken immediately after demolding (24 h after casting). The prisms for drying shrinkage determination were stored in drying room where temperature was maintained at 21 ± 2°C and relative humidity was maintained at 50 ± 5%. Shrinkage prisms used for measurements of autogenous shrinkage were stored in the 20-L plastic containers (four prisms in each container) with a sealed lid that prevented exchange of moisture with atmosphere.

Microstructural studies utilised a scanning electron microscopy (JEOL JSM-840 A, JEOL, Japan). The polished

Table 2
Summary of experimental programme AAS concrete: elevated and room temperature curing

Mix no.	Type of specimen	Type of activator	Concentration	Admixture	Curing temperature	Compressive strength	Shrinkage
1	AAS concrete, w/b = 0.5	Liquid sodium silicate + sodium hydroxide solution	4% Na ^a $M_s = 0.75$	–	Room temperature	1, 3, 7, 28, 200, 360 days	X
2	AAS concrete, w/b = 0.5	–	–	–	6 h at 70° C	1, 3, 7, 28 days	X
3	AAS concrete, w/b = 0.5	–	–	–	2 h room temperature, ramp 3.5 h to 65° C, 5 h at 65° C	1, 3, 7, 28, 100, 200, 360 days	X
4	OPC concrete, grade 60, w/b = 0.45	–	–	Superplasticiser ^b	Room temperature	1, 3, 7, 28, 200, 360 days	X
5	OPC concrete, grade 60, w/b = 0.45	–	–	Superplasticiser ^b	6 h at 70° C	1, 3, 7, 28, 200, 360 days	X
6	OPC concrete, grade 40 w/b = 0.5	–	–	–	Room temperature	1, 3, 7, 28, 200, 360 days	X

^aPercentage of Na refers to percent by mass of slag.

^bSodium naphthalene sulphonate polymerised with formaldehyde (45%) (ASTM C-494 Type A & F).

Table 3
Mix proportions of concrete

Mix description	Mix no.	Admixture (kg/m ³)	w/b	Added water (kg/m ³)	Cementitious, (kg/m ³)	Na-hydroxide solution (kg/m ³)	Total water ^a (kg/m ³)	Coarse aggregates ^b (kg/m ³)	Fine aggregates ^c (kg/m ³)
4% Na, $M_s = 0.75$	1, 2, 3	–	0.5	147.4	Slag, 360	30.14	180	1130	830
OPC grade 60	4, 5	Superplasticiser ^d , 1.5	0.45	180	Cement, 400	–	180	1310	630
OPC grade 40	6	–	0.5	180	Cement, 360	–	180	1130	830

^aTotal water includes added water and water in sodium silicate and sodium hydroxide solutions.

^bOld basalt 14/10 mm (Pakenham, Victoria).

^cConcrete sand, FM = 2.18 (Lyndhurst, Victoria).

^dSodium naphthalene sulphonate polymerised with formaldehyde (45%) (ASTM C-494 Type A & F).

Table 4
Properties of AAS concrete

Specimen type	Compressive strength at 1 day, (MPa)	Compressive strength at 7 days (MPa)	Compressive strength at 1 month (MPa)	Flexural strength at 1 month (MPa)	Drying shrinkage at 56 days (microstrain)
Sodium silicate-activated concrete, 4% Na, $M_s = 0.75$, w/b = 0.5	16	36	46	6.5	1300
OPC grade 40	6.6	35	45	5.6	600

sections of OPC and AAS concrete were examined in the mode of backscattered electron image (BEI). In the BEI mode contrast is derived from differences in the atomic number of each constituent, because the intensity with which electrons are backscattered increases monotonically with average atomic number.

Anhydrous material, slag, (white bright phase), hydration products, calcium silicate hydrate (light grey phase), and pores (dark grey) are easily distinguished on the basis

of their grey levels. The accelerating voltage of 20 kV was utilised in the BEI studies.

3. Results

Table 4 shows the results of strength measurements of AAS and OPC concretes cured at room temperature. AAS exceeds OPC in flexural and compressive strength at early

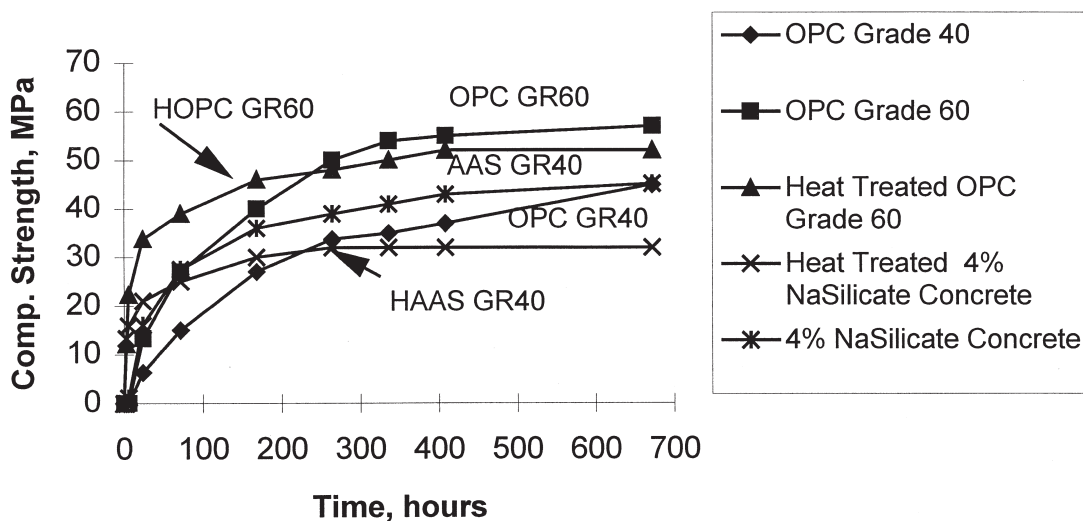


Fig. 1. Compressive strength development of heat-cured concrete at 65°C.

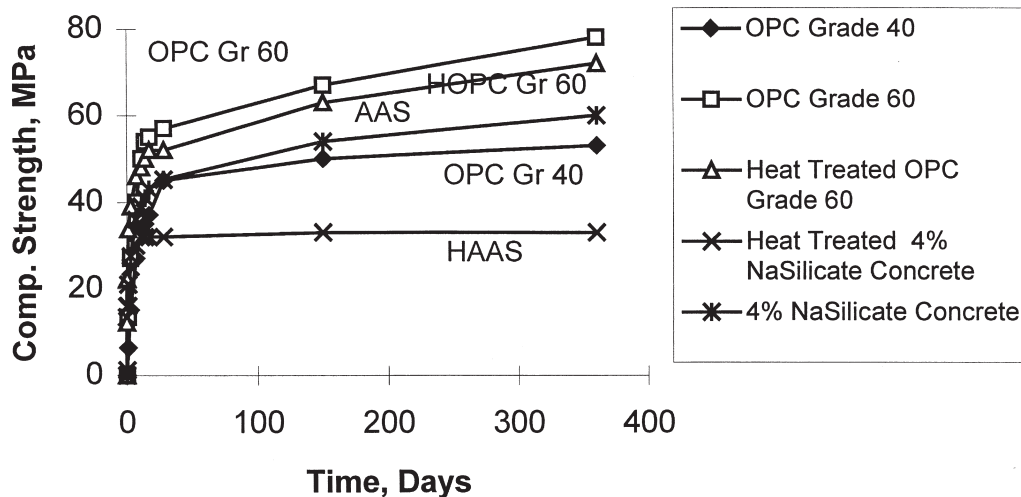


Fig. 2. Compressive strength development for AAS concrete cured at elevated temperature.

age; however, shrinkage of AAS exceeds by 2 to 2.2 times that in OPC concrete.

3.1. Strength

It was found that when cured at elevated temperatures, AAS concrete developed strength quite rapidly (Fig. 1). After 3 h of curing at 70°C AAS concrete attained a strength of 14 MPa; after 6 h of curing the strength was 17 MPa, and after 24 h the strength was 21 MPa, exceeding 1-day strength of the samples cured at normal temperature. Strength development of AAS concrete during the first 3 h was more rapid than that of OPC concrete, even of higher grade (grade 60) [9].

For the elevated temperature curing with the pretreatment period, the strength obtained for AAS at 1 day was 24 MPa, exceeding the strength of the same material heat-cured without a pretreatment period. However, at 1 month and later ages the strength of all AAS concrete samples cured at elevated temperature was 35 to 45% lower than that of samples cured at room temperature (Fig. 2).

3.2. Shrinkage

Figs. 3 and 4 and Table 5 present the shrinking results of the AAS concrete activated by sodium silicate and sodium hydroxide solutions and cured at elevated and room temper-

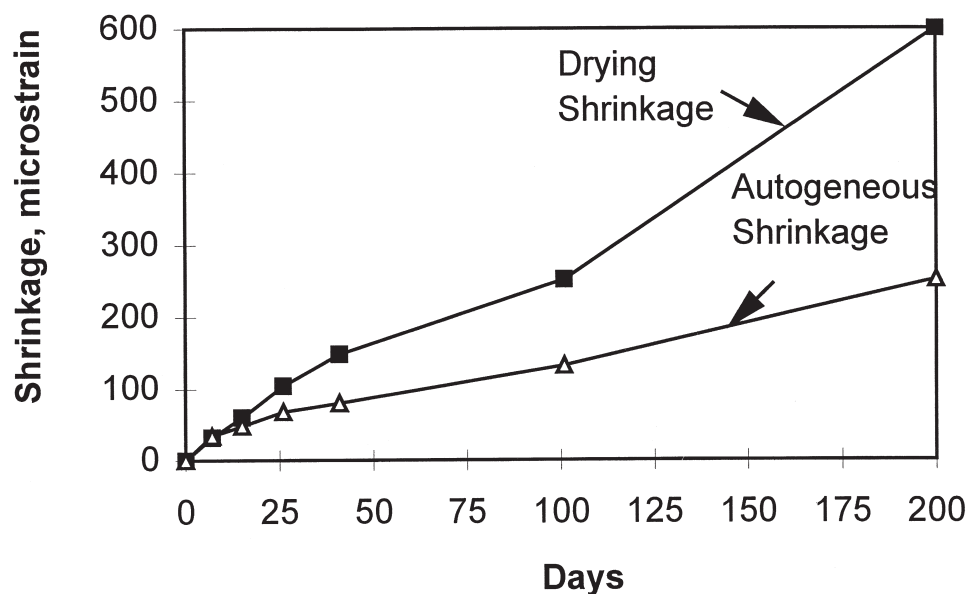


Fig. 3. Shrinkage of heat-treated AAS concrete with pretreatment at room temperature (mix 10).

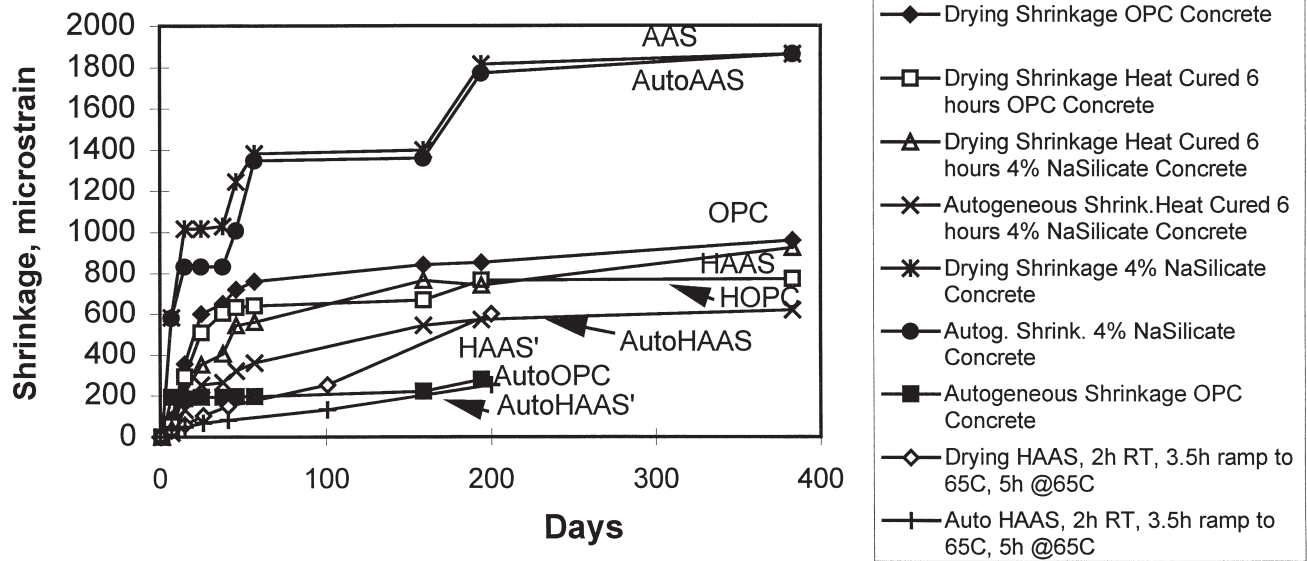


Fig. 4. Autogenous and drying shrinkage of AAS concrete with sodium silicate, 4% Na in the mix, heat treated and cured at room temperature.

ature [9,10]. For concrete cured at elevated temperatures, shrinkage was considerably lower than of concrete cured at room temperature. At early ages it was even lower than of heat-treated OPC concrete. Pretreatment caused further reduction in shrinkage.

3.3. Microstructure of heat-treated OPC and AAS concretes

Figs. 5 and 6a,b show microstructure of OPC and AAS concretes heat-treated for 6 h at 70°C and stored in a bath with lime water for 1 and 12 months, respectively. Development of microstructure of heat-treated AAS and OPC concretes can be compared and followed in time. Hydrated OPC and slag grains are readily distinguishable on the basis of their grey level. Hydration product is localised in the area around cement and slag grains so that interstitial pores remained open. Microstructure in OPC is more homogeneous than in AAS. Slag grains tend to hydrate in place without distribution of hydration product in the interstitial spaces. This tendency persisted at 12 months, and is more developed in slag than OPC. During heat treatment a bright rim of hydration product was deposited on the surface of AAS and OPC grains that was not

dissolved after complete hydration of slag and OPC particles. Hydration product deposited at later ages (presumably at room temperature in both AAS and OPC) was darker in colour.

4. Discussion

4.1. Strength

The observed initial rapid strength development of heat-cured AAS concrete and its slow later growth are similar to results obtained for OPC (Figs. 1 and 2) and are reported earlier [11]. For AAS the later strength was significantly reduced. It is correlated with an inhomogeneity of microstructure observed in heat-treated AAS. Similar to OPC, slow strength development in AAS at elevated temperatures was attributed to inhomogeneity of the microstructure, localisation of hydration product near slag grains, and resulting coarse pore structure. The rate of reaction was so much faster than the rate of diffusion that most of the hydration products remain near the slag grains, leaving interstitial space relatively open. More dense precipitates deposited at elevated temperature may form a barrier for ion diffusion, thus causing an inhomogeneity in the microstructure.

4.2. Shrinkage

It is known that curing at elevated temperatures makes OPC concrete more dimensionally stable and less susceptible to shrinkage [7,12–14]. This was clearly seen from Fig. 4. Heat treatment for 6 h considerably reduced drying shrinkage in the sodium silicate and sodium hydroxide-activated concrete; it became close to drying shrinkage of heat-treated OPC concrete and OPC concrete cured at room temperature. Scrivener reported the effect of heat treatment on

Table 5
Summary of drying shrinkage test

Mix no.	Drying shrinkage (microstrain)					
	7 days	14 days	28 days	112 days	224 days	400 days
1	580	980	1015	1400	1814	1865
2	40	216	352	640	740	924
3	32	72	100	252	600	–
5	52	292	508	650	760	772
6	86	356	500	800	850	910

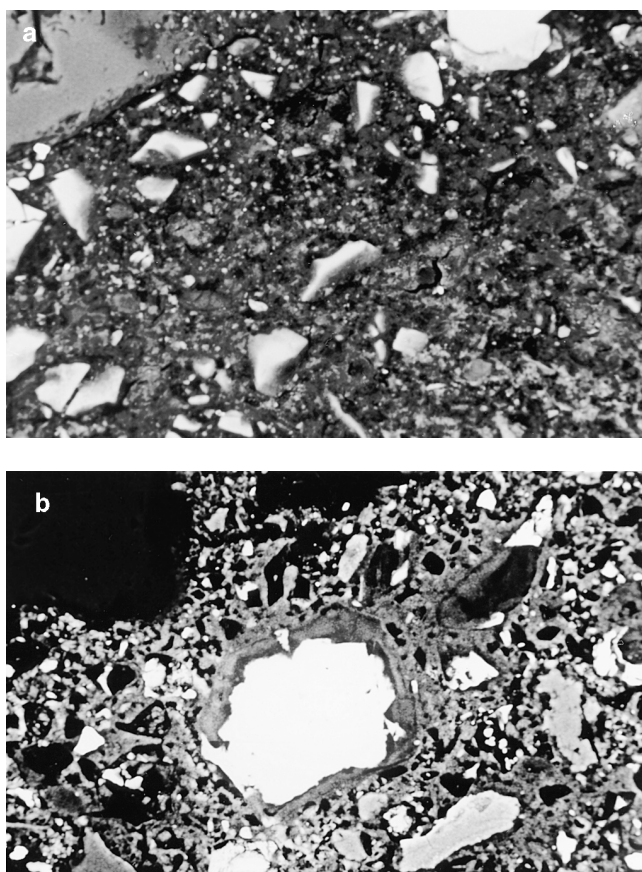


Fig. 5. BEI of concrete cured for 6 h at 70°C and stored for 1 month in water bath at room temperature: (a) AAS; (b) OPC.

inner product calcium-silicate-hydrate (C-S-H) [15]. In this investigation the outer part of the C-S-H rim that was formed at 80°C, was noticeably brighter than the inner part, which was formed at 20°C. The typical two-tone appearance of the inner product was attributed to a greater degree of fine porosity in the material, which could be due partly to more water originally present in the C-S-H structure. Thus, from the present investigation it follows that C-S-H formed during heat treatment has less water content, which is beneficial for reducing the shrinkage. This is possibly the reason why low shrinkage of heat-treated OPC and AAS concrete was achieved.

5. Conclusions

1. Heat treatment considerably accelerates the strength development of AAS concrete, but at later ages compressive strength of the materials is reduced compared with concrete cured at room temperature.
2. Heat curing considerably reduces shrinkage of AAS concrete, making it comparable with shrinkage of OPC concrete.
3. Compared to OPC heat-treated AAS has a more open microstructure, with hydration products localised near slag grains and relatively open interstitial spaces.

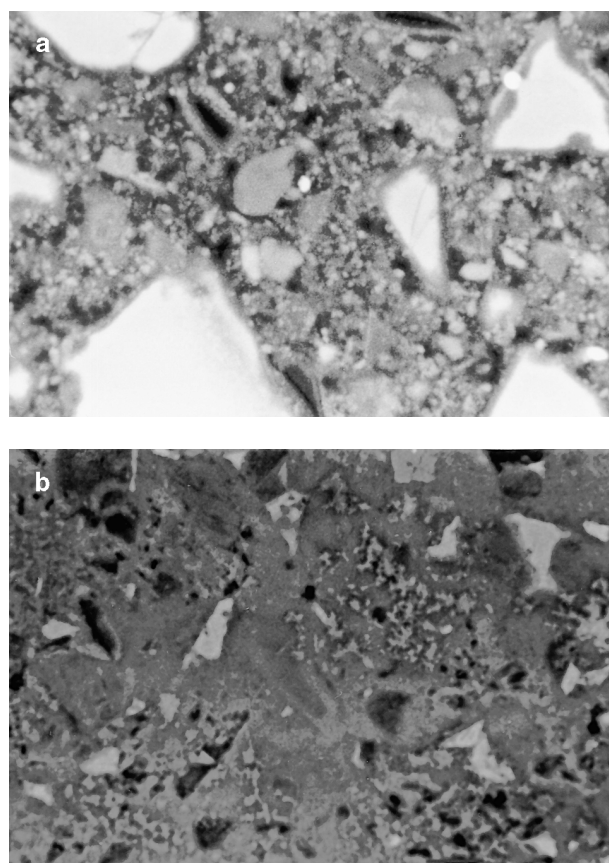


Fig. 6. BEI of concrete cured for 6 h at 70°C and stored for 12 months in water bath at room temperature: (a) AAS; (b) OPC.

4. The heat treatment history has an effect on drying shrinkage since 2 h pretreatment at room temperature before elevated temperature curing considerably decreased shrinkage in AAS concrete.

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