Pergamon

Cement and Concrete Research 30 (2000) 1367–1374

CEMENT AND CONCRETE RESEARCH

Effect of admixtures on properties of alkali-activated slag concrete

T. Bakharev[a,]*, J.G. Sanjayan[a], Y.-B. Cheng[b]

[a]Department of Civil Engineering, Monash University, Wellington Road, Clayton, Victoria 3168, Australia
[b]Department of Materials Engineering, Monash University, Clayton, Victoria 3168, Australia

Received 28 May 1999; accepted 21 June 2000

Abstract

This paper reports the results of an investigation on concrete that incorporated alkali-activated slag (AAS) as the only binder. The activators were liquid sodium silicates (4–7% Na, mass of slag) and a multi-compound activator ($NaOH + Na_2CO_3$) (8% Na, mass of slag). AAS utilizes industrial by-products and develops high early strength. However, some of its properties such as high shrinkage and poor workability impede its practical application. Admixtures used for ordinary portland cement (OPC) were tested to improve these properties of AAS concrete. Superplasticiser based on modified naphthalene formaldehyde polymers (S), air-entraining agent (AEA), water-reducing (WRRe), shrinkage-reducing (SHR) admixtures at dosages of 6–10 ml/kg, and gypsum (G) (6% of slag weight) were used. The paper presents the study of workability in the fresh state, shrinkage and compressive strength of AAS concrete, and the effect of admixtures and type of activator on these properties. Concrete activated by liquid sodium silicate had the best mechanical properties. AEA, SHR, and G significantly reduced its shrinkage. AEA also improved workability and had no negative effect on compressive strength. On the basis of this investigation, AEA was recommended for use in AAS concrete. © 2000 Elsevier Science Ltd. All rights reserved.

Keywords: Ground-granulated blast furnace slag; Alkali-activated cement; Admixtures; Workability; Shrinkage; Strength properties

1. Introduction

Alkali-activated slag (AAS) is a new type of binder used in concrete manufacturing. Its production utilises industrial by-products, requires less energy than ordinary portland cement (OPC) and is associated with low CO_2 emission. AAS concrete has been found to have some superior properties as compared to OPC concrete, namely, low heat of hydration, high early strength, and excellent durability in aggressive environment [1–4]. Utilisation of a new material in the cement industry requires practical experience in solving processing problems such as rapid set and high shrinkage, without impeding strength development. Previous investigations of AAS have shown that the strength of AAS pastes is dependent on the concentration and type of activator [5,6]. It has also been revealed that the material has a workability problem, and that the shrinkage in most cases exceeds that of OPC concrete [5,7,8]. Set-regulating admixtures developed for OPC concrete can be used to overcome these problems. A previous investigation by Collins and Sanjayan [9] of slag activated by sodium carbonate and sodium hydroxide utilized such admixtures. The study included mini-slump testing and was conducted on pastes, but it did not consider the effect of admixtures on shrinkage and compressive strength; also these tests were conducted on pastes, whereas for concrete results could be different. Our earlier study on the early hydration of alkali-activated slag has shown that considerable differences exist in the mechanism of hydration when compared with OPC [10]. Thus, it can be expected that admixtures could have different effects on AAS than OPC. The focus of the current investigation is on the study of the effects of admixtures on workability, shrinkage, and compressive strength of AAS concrete activated by sodium silicate glass and multi-compound activator in order to improve these properties of AAS concrete.

2. Materials

2.1. Slag

The chemical composition of the slag supplied by SteelCement (Port Melbourne, Australia) is summarised

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* Corresponding author. Tel.: +61-3-9905-4976; fax: +61-3-9905-4944.

E-mail address: tanya.bakharev@eng.monash.edu.au (T. Bakharev).

PII: S0008-8846(00)00349-5

Table 1
Composition of slag

Oxides	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	K_2O	Na_2O	TiO_2	P_2O_5	MnO	SO_3	Sulfide (sulphur as S^{2-})	Cl	Loss on ignition
Percentage	35.04	13.91	0.29	39.43	6.13	0.39	0.34	0.42	< 0.1	0.43	2.43	0.44	80 ppm	1.45

Table 2
Mix proportions of slag concrete

Mix no.	Activator, % Na, and M_s	Admixture, ml/kg slag	Na hydroxide solution, kg/m^3	Activator, kg/m^3	Added water, kg/m^3
1	Na silicate, 4% Na, $M_s = 0.75$	–	30.14	Na silicate, D, 36.72	147.4
2		WRRe, 10			
3		S, 10			
4		AAE, 6			
5		G, 6%			
6		SHR, 6			
7	Na silicate, 7% Na, $M_s = 1.25$	WRRe, 10	32.47	Na silicate, D, 128.43	95.22
8	Na_2CO_3, 1.7% Na; NaOH, 6.3% Na	WRRe, 10	65.99	Na_2CO_3, 14.4	154.86

in Table 1. The blast furnace slag is a granulated product ground to fineness of about 460 m^2/kg, with the particle size range of 1–10 μm and is neutral with the basicity coefficient $K_b = (CaO + MgO)/(SiO_2 + Al_2O_3)$, which is equal to 0.93. The slag is supplied with 2% blended gypsum (G).

2.2. Activators

The activators investigated were sodium silicate glass,[1] sodium hydroxide solution,[2] and sodium carbonate.[3] Liquid sodium silicate and sodium hydroxide were blended providing the modulus in solution (mass ratio of SiO_2 to Na_2O), M_s, equal to 0.75 and 1.25. A multi-compound activator, composed of sodium hydroxide (6.3% Na)[4] and sodium carbonate (1.7% Na) with a total Na content of 8% in the mixture was also used in the investigation. Previous investigations of Collins and Sanjayan [7,8] of slag activated by powdered sodium silicate utilized activator in dry powder state, together with hydrated lime. The mechanism of activation and, consequently, properties were rather different.

2.3. Chemical admixtures

The chemical admixtures[5] investigated in this work were as follows: S = short-life superplasticiser based on modified naphthalene formaldehyde polymers, which complies with ASTM C-494 Type A and F; WRRe = water-reducing and set-retarding admixture based on lignosulphonates, which complies with ASTM C 494 Types B and D; AEA = air-entraining admixture with a soluble salt of an alkyl aryl sulphonate, complying with ASTM C260, AS 1478-Type AEA; SHR = shrinkage-reducing admixture, nonstandard in ASTM. In addition, G supplied by SteelCement was also used. G = gypsum, 80% $CaSO_4 \cdot 2H_2O$, 15% $CaSO_4 \cdot 0.5H_2O$, and 5% $CaSO_4$ ground to a fineness of 550 m^2/kg.

3. Procedures

3.1. Concrete mixes

The main concrete mix design comprised slag (360), coarse aggregates (1130), fine aggregates (830), and total water (180) kg/m^3. Table 2 lists the various mixes that were made using different admixtures. The water to binder (w/b) ratio was fixed at 0.5 to enable direct comparison and reasonable concrete workability. 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, the chemical admixtures were added to concrete during mixing.

3.2. Test procedures

Table 3 shows the experimental programme including the summary of specimens and tests. For AAS concrete, the effect of type of activator on compressive and flexural strength was elucidated first. Then the effect of admixtures on workability, compressive strength, and shrinkage was

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[1] PQ Australia, sodium silicate solution grade D, weight ratio $SiO_2/Na_2O = 2$, $\%Na_2O = 14.7$, $\%SiO_2 = 29.4$, technical grade.

[2] Ajax Chemicals, 60% w/v water solution, technical grade.

[3] Ajax Chemicals, technical grade.

[4] Everywhere in the paper, percentage of Na refers to percent by mass of slag.

[5] W.R. Grace, Melbourne, Australia.

Table 3
Summary of experimental programme

Mix no.	Type of specimen	Type of activator	Na content,% weight slag	Admixture	Compressive strength	Slump	Shrinkage
1	AAS concrete, w/b = 0.5	Liquid sodium silicate	4% Na, $M_s = 0.75$	–	1, 3, 7, 28, 100, 200, 350 days	X	X
2				WRRe	1, 3, 7, 28 days	X	X
3				S		X	X
4				AEA		X	X
5				G	–	X	X
6				SHR	–		
7	AAS concrete, w/b = 0.5	Liquid sodium silicate	7% Na, $M_s = 1.25$	WRRe	1, 3, 7, 28, 100, 200, 350 days	X	X
8	AAS concrete, w/b = 0.5	Na_2CO_3; NaOH	1.7% Na; 6.3% Na	WRRe	1, 3, 7, 28, 100, 200, 350 days	X	X
9	OPC Gr40	–	–	–	1, 3, 7, 28, 100, 200, 350 days	X	X

Table 4
Properties of fresh concretes

Mix no.	Admixture, ml/kg slag	Na/slag	Slump, mm	Unit weight, kg/m^3	Air content, %	Concrete temperature, °C
1	–	4	55	2462	2	22
2	WRRe, 10	4	200	2460	1.5	20
3	S, 10	4	200	2480	2	21
4	AEA, 6	4	200	2367	6	22
5	G, 6%	4	60	2445	2	22
6	SHR, 6	4	95	2523	1	23
7	WRRe, 10	7	200	2512	2	21
8	WRRe, 10	9.4	250	2435	2.5	23
9	–	–	100	2470	0.5	22

investigated mainly for concrete with sodium silicate activator. OPC concrete samples were used for comparison.

Compressive strength testing was conducted on cylinders (100×200 mm), and a total of three cylinders were tested for each data point. Workability of concrete was assessed by slump loss test. Shrinkage measurement was performed using concrete shrinkage prisms 75×75×285 mm. A minimum of two shrinkage prisms was used in each test. The first reading was taken immediately after demoulding (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 at 50 ± 5%. Shrinkage prisms used for measurements of autogenous shrinkage were stored in 20-l plastic containers (four prisms in each container) with sealed lids that prevents exchange of moisture with atmosphere.

4. Results

4.1. Slump

The results of measurements of properties of the freshly mixed concrete, i.e., temperature, slump, unit weight, and air content, are presented in detail Table 4. Fig. 1(a–b) shows the effect of admixtures on workability of AAS concrete prepared with multi-compound activator and sodium silicate glass. Initially, slump was measured for AAS concrete without any admixture. Sodium-silicate-activated slag concrete, with 4% Na, had a slump of 55 mm immediately after mixing, and remained workable during half an hour, although slump continued to decrease. The same sodium-silicate-activated slag concrete with WRRe admixture of 6 ml/kg had a slump of 80 mm

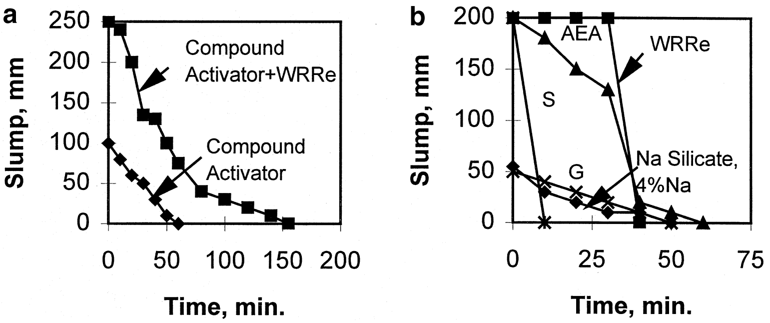

Fig. 1. Effect of admixtures on slump loss for AAS concrete samples with various activators: (a) multi-compound activator; (b) sodium silicate activator.

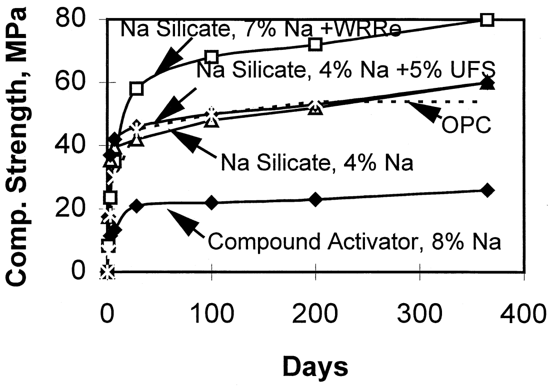

Fig. 2. Compressive strength of AAS concrete.

and had better workability than concrete without admixture, but it also had some retardation in the initial set. WRRe admixture addition of 10 ml/kg in the same concrete mix produced a slump of 200 mm, which was reduced to zero after 40 min. AEA at a dosage of 6 ml/kg produced a considerable improvement in workability. The mixes had a good workability during 30–40 min.

The sodium-silicate-activated slag concrete mix (4% Na), with the addition of superplasticiser was initially very fluid, having a slump above 200 mm. However, after 10 min, the concrete lost its fluidity completely, and it appeared that the superplasticiser was acting as set accelerating. Addition of 6% G did not improve workability.

The AAS concrete activated by sodium carbonate and sodium hydroxide and containing the WRRe (10 ml/kg) had a slump exceeding 200 mm after mixing. Later, slump remained high for a long period of time. Thus, interaction of AAS concrete with admixture depended on the activator.

WRRe and AEA were the most effective admixtures for improving workability. In all cases, no adverse effects on drying shrinkage have been observed, although the early strength of AAS concrete for WRRe was relatively impaired (see Fig. 3).

4.2. Strength

It was found that the type of activator has a significant effect on strength of AAS concrete. Fig. 2 shows the results of strength measurements for concretes activated by different activators. Table 5 summarises the strength measurements at 1 month. As reported earlier [5], the best results were obtained for sodium silicate glass. Two concretes were prepared using sodium silicates at 4% Na and 7% Na by mass of slag, and they exhibited compressive strength of 40 and 58 MPa at 28 days, respectively. The higher percentage of sodium in the activator produced a higher strength. Workability in the fresh state was decreasing rapidly with concentration of activator. Thus, WRRe had to be introduced in the second mix. The modulus of rupture determined for these concretes by the four point bending method (AS 1012-Part 11) was 6.5 and 6.4 MPa, respectively. Introduction of 5% ultrafine slag (UFS) with fineness of 1200 m^2/kg produced improvement in strength and workability. At 28 days, strength for the sodium-silicate-activated concrete with UFS was increased to 46 MPa (comparing to 40 MPa), and continued to rise at later stages. The concrete with the multi-compound activator did not produce high strength; it was 20 MPa at 28 days with modulus of rupture of 3.2 MPa.

Fig. 3 shows the effect of admixtures at dosages of 6–10 ml/kg slag (mix proportions in Table 2) on compressive strength of AAS concrete for up to 28 days. Superplasticiser caused 25% loss of 28 day strength, and use of water reducing admixture resulted in reduction of early strength, up to 14 days. AEA had some effect on early strength up to 7 days, after that strength development was similar to AAS concrete without admixture.

4.3. Shrinkage

Table 6 shows the summary of drying shrinkage test results. The highest drying shrinkage was observed for the AAS concrete with superplasticiser based on modified naphthalene polymers (see Fig. 4). The drying shrinkage in this case considerably exceeded the autogenous shrinkage. It was concluded that it is not desirable to use this type of admixture in AAS concrete, although it may give good results for OPC concrete.

Fig. 5 shows shrinkage of the sodium-silicate-activated slag concrete prepared with lignosulphonate based WRRe, at a dosage of 10 ml/kg slag, with 4% Na in the mix. This concrete mix had shrinkage values considerably lower than those for the concrete with superplasticiser, but the shrinkage was still higher than that of OPC concrete.

Table 5
Compressive and flexural strength of AAS concretes

Specimen type	Compressive strength at 1 month, MPa	Flexural strength at 1 month, MPa
Sodium-silicate-activated concrete, 4% Na, $M_s = 0.75$, w/b = 0.5	40	6.5
Sodium-silicate-activated concrete, 4% Na, $M_s = 0.75$ with 5% ultrafine slag, w/b = 0.5	46	7.0
Sodium-silicate-activated concrete, 7% Na, $M_s = 1.25$, w/b = 0.5	58	6.4
AAS concrete with compound activator, 8% Na, w/b = 0.5	20	3.2
OPC Gr40, w/b = 0.5	45	5.6

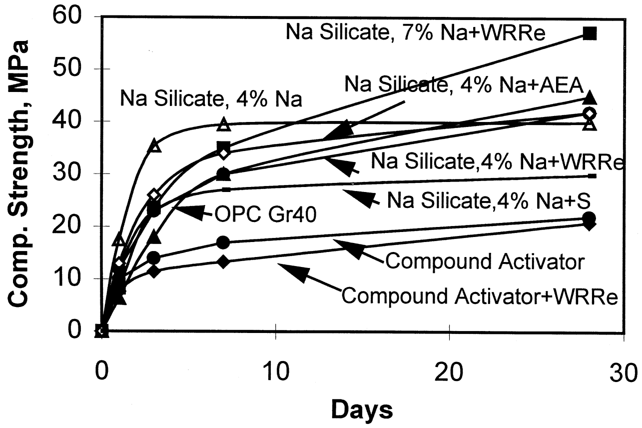

Fig. 3. Compressive strength of AAS concrete prepared using different admixtures (dosage of admixtures in Table 2).

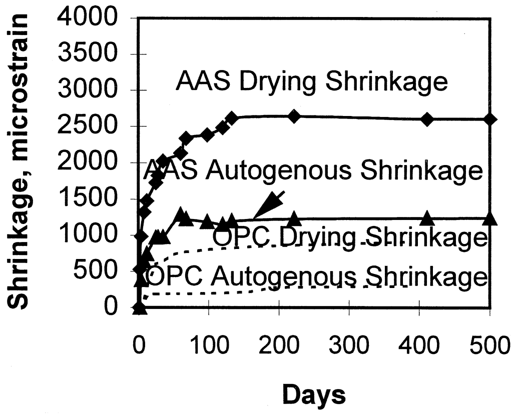

Fig. 4. Autogenous and drying shrinkage of sodium-silicate-activated slag concrete, 4% Na with superplasticiser S at a dosage of 10 ml/kg slag.

Fig. 6 shows shrinkage characteristics for the concrete with multi-compound activator and WRRe at a dosage of 10 ml/kg slag. The autogenous and drying shrinkage exceed the shrinkage of OPC concrete.

Shrinkage curves for the AAS concretes activated by sodium silicate (4% Na) and containing the air entraining and shrinkage reducing admixtures, 6 ml/kg slag, are shown in Figs. 7 and 8, respectively. The drying shrinkage exceeded the autogenous one, but in both cases, it was not more than 400 microstrain at 56 days, and 450 microstrain at 130 days, lower than for OPC concrete, though shrinkage continued to grow. Shrinkage reducing admixture was found to be insoluble in alkaline solution, so its interaction with the concrete can give variable results.

Shrinkage curves for the AAS concretes activated by sodium silicate (4% Na) and containing 6% G are given in Fig. 9. The autogenous shrinkage exceeded the drying shrinkage during the first 125 days. The drying shrinkage was below 150 microstrain at 56 days. The autogenous shrinkage was about 500 microstrain at 56 days. XRD traces of AAS pastes with G (4–10%) at 3 days after start of hydration are presented in Fig. 10. The traces show that formation of Aft and AFm phases is high at 6% G, while at 8% and 10% G was reacted and no traces of G and AFt or AFm type phases are evident.

5. Discussion

5.1. Workability

An increase in workability was achieved with the lignosulphonate water-reducing and -retarding admixture for the activators used, namely, sodium silicate and combination of sodium hydroxide and sodium carbonate, whereas the time of initial set was retarded in both cases. With superplasticiser, the increase in workability was only at the initial stage, and then a quick set occurred, which appeared to have been accelerated by the admixture. Therefore, the behaviours of the pastes with these two admixtures were considerably different.

The lignosulphonate admixture had a prolonged positive effect on workability as compared with the superplasticiser, possibly due to its essentially nonpolar molecule and low coulomb attraction of the slag particles with adsorbed nonpolar molecules. The superplasticiser has a polar molecule,

Table 6
Results of drying shrinkage test

		Drying shrinkage, microstrain					
Mix no.	Admixture	7 Days	14 Days	28 Days	112 Days	224 Days	400 Days
1	–	580	980	1015	1400	1814	1865
2	WRRe	200	350	420	1000	1250	1300
3	S	1300	1500	1800	2300	2500	2600
4	AEA	100	140	230	390	440	–
5	G	22	30	56	350	550	–
6	SHR	82	100	116	260	600	–
7	WRRe	350	550	780	1235	1350	1450
8	WRRe	170	240	470	1612	1970	1990
9	–	86	356	600	800	850	910

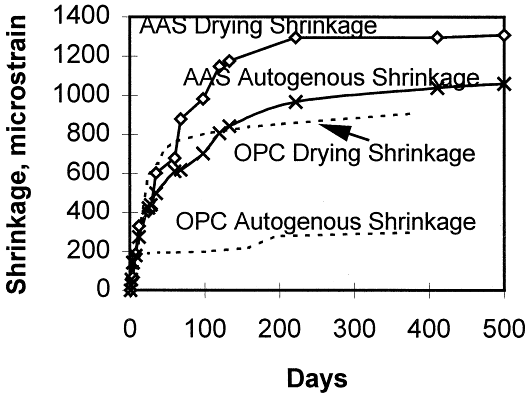

Fig. 5. Autogenous and drying shrinkage of sodium-silicate-activated slag concrete, 4% Na with lignosulphonate WRRe admixture at a dosage of 10 ml/kg slag.

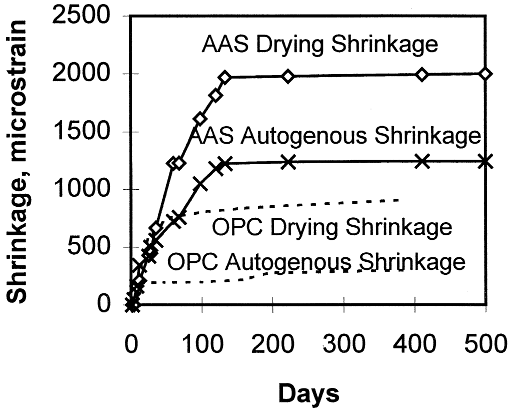

Fig. 6. Autogenous and drying shrinkage of alkali-activated slag concrete with compound activator with WRRe at a dosage of 10 ml/kg slag.

which can be absorbed rapidly on charged particles. This can increase the zeta-potential of the hydrating particles and promote quick set due to coulomb attraction between positively and negatively charged particles. A hypothesis is that admixtures with nonpolar molecules are better suited to work in a media of strongly charged particles of AAS and activator.

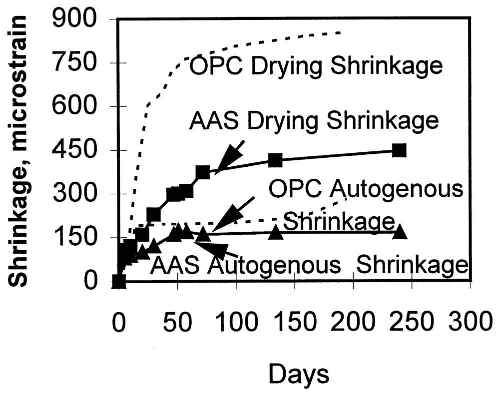

Fig. 7. Shrinkage of slag concrete activated by sodium silicate, 4% Na, with AEA at a dosage of 6 ml/kg slag.

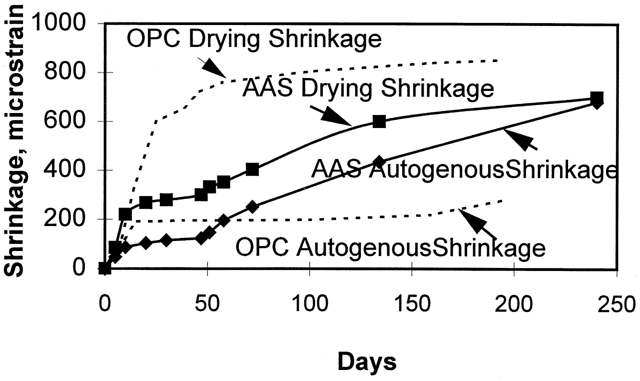

Fig. 8. Shrinkage in AAS concrete with SHR admixture at a dosage of 6 ml/kg slag.

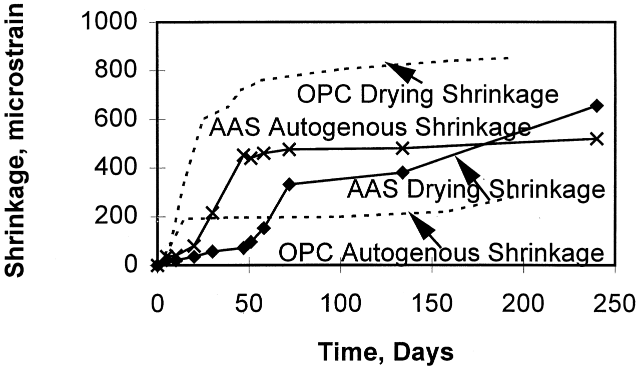

Fig. 9. Shrinkage in AAS concrete with 6% G.

Collins and Sanjayan [9] indicated that slag activated by NaOH + Na_2CO_3 had a reasonable response to retarders, but the loss of workability beyond 30 min was significant, and the 1-day strength was reduced. These results are close to our findings. The effect of lignosulphonates is similar to the action on portland cement concrete, except the time of improved workability is much shorter, and dosage to obtain

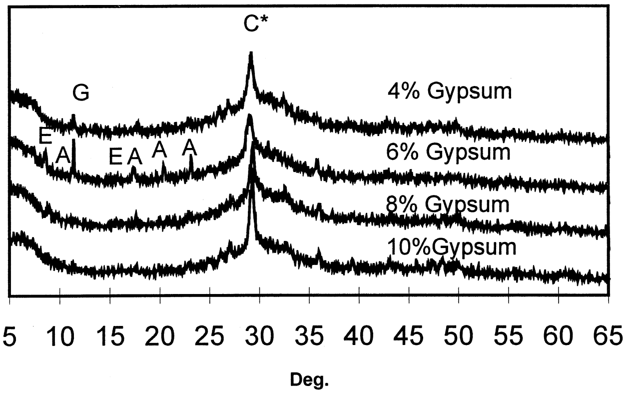

Fig. 10. XRD of AAS paste with different content of G, 3 days after start of hydration; C* = overlapping C–S–H and calcite, G = gypsum, $CaSO_4 \cdot 2H_2O$; E = AFt phase, $C_3A \cdot 3CaSO_4 \cdot 32H_2O$; A = AFm phase, type $C_3A \cdot CaSO_4 \cdot H_{11-13}$.

desirable effect is higher. AEA considerably improved workability of AAS concrete possibly due to the physical effects of entrained air.

5.2. Strength

Fig. 2 shows that AAS has the benefit of early strength development as compared to OPC concrete. The strength properties of AAS concrete measured are close to those reported earlier by Collins and Sanjayan [7]. The effect of the state of activator: powdered sodium silicates (Collins and Sanjayan) and liquid sodium silicate glass (present study) disappeared after the first 7 days. Liquid sodium silicates provide a higher early strength, but concrete has a lower workability due to immediate reaction and rapid hardening of AAS [5,10]. However, powdered sodium silicates are reliable for use only in a dry climate due to the hygroscopic properties of dry sodium silicates.

The admixtures had different effects on the strength of AAS concrete. Lignosulphonates retarded strength development. Similar to the effect on OPC, they increased the workability and delayed the set of AAS concrete. Although the superplasticiser caused an accelerated set, the compressive strength at later ages was lower than that of AAS concrete without any admixture. Together with its high shrinkage effect, this makes the superplasticiser unfavourable for use in AAS concrete. AEA had the least effect on strength, but reduction in drying shrinkage and improvement in workability was significant.

5.3. Shrinkage

The reported values for shrinkage of AAS concrete vary significantly [5,7,8,11,12], possibly due to different testing procedures used. Similar to our work, Collins and Sanjayan [7] and Kutti et al. [11] found that drying shrinkage in AAS concrete activated by sodium silicates for RH>50% is 2.3 times drying shrinkage in OPC concrete, but Kutti et al. [11] also show that for RH>70%, the drying shrinkage is similar to OPC concrete. Collins and Sanjayan [8] reported that shrinkage of AAS concrete with fully saturated blast furnace slag coarse aggregate was 19% higher than that of OPC concrete. Possibly, the saturated aggregates gradually release the moisture, thus reducing the shrinkage of AAS concrete. According to Australian Standard AS 1012, Part 13, standard shrinkage prisms after demoulding have 7 days bath curing followed by curing at drying conditions 23°C and $RH = 50 \pm 5\%$. It is well known that shrinkage rate is at maximum during first 7 days, thus, the measured shrinkage would be less for bath cured prisms than for those exposed to drying conditions from the first day after demoulding. Collins and Sanjayan [8,12] observed some expansion during first 3 days for bath cured AAS concrete. It was not observed for prisms exposed to drying conditions immediately after demoulding. However, the reported drying shrinkage for AAS concrete is, in most cases, much higher than for OPC concrete and reflects the high sensitivity of AAS concrete to curing conditions.

Admixtures have a significant influence on volume stability of AAS concrete. The superplasticiser increased shrinkage considerably, while the lignosulphonate admixture did not increase it, but caused a slight decrease in shrinkage. A low-drying shrinkage, comparable to that of OPC concrete was observed for the AAS concrete prepared with AEA, shrinkage reducing admixtures and G (6%). According to the XRD results (Fig. 10), the observed low shrinkage was due to the compensating effect of AFt and AFm phases which formed in significant amounts at 6% G addition and had a volume expansion when they were formed. Possibly, at 8% and 10%, G was incorporated in a low-Ca C–S–H that formed in this system [13]. The binding properties of C–S–H increase with a decrease in the C/S ratio.

AAS concrete with AEA, G, and SHR had also small differences between the autogenous and drying shrinkage. AAS concrete develops large differences in strain inside the member and on the surface due to large differences between autogenous and drying shrinkage (see Figs. 4–6). Inside the member the conditions are effectively autogenous, while exterior surface is exposed to drying conditions. The measurements of in situ strain at the center and at the faces of a column with the cross-section 800×800 mm^2 made by Collins and Sanjayan [12] showed that the differential strain was 149 microstrain at 91 days. It is not high, but it may cause cracks in OPC concrete, and, possibly, was a reason for surface crazing in AAS concrete as it was reported in Ref. [12]. For AAS concretes with AEA, SHR, and G, the difference between autogenous and dying shrinkage was less than 300 microstrain after 250 days, compared to 600 microstrain for OPC concrete. The drying shrinkage was close to or less than for OPC, although it continued to grow after 250 days. Thus, AEA, SHR, and G were effective in minimising shrinkage cracking.

On the basis of our investigation, we can conclude that AEA admixture is the most suitable for use in AAS concrete activated by sodium silicate glass, as it improves workability, reduces shrinkage, and does not deteriorate later strength. However, admixtures for OPC often have to be used in large dosage, and may have significant side effects (increased shrinkage, strength loss) when applied for AAS. Effect of admixtures on AAS depends on the type of activator, and can be opposite to the effect on OPC.

6. Conclusions

(a) An increase in workability was achieved with the lignosulphonate admixture for all types of activators tested.
(b) With the naphthalene formaldehyde superplasticiser, the increase in workability was only at the initial stage and then a quick set occurred, which was considered to have been accelerated by the admixture.

(c) Lignosulphonates may cause retarded strength development and reduced flexural strength of AAS concrete.
(d) Lignosulphonate-based admixture caused a slight reduction in shrinkage, while naphthalene-based superplasticiser significantly increased the shrinkage and reduced the strength of AAS concrete.
(e) Air-entraining and SHR admixtures, as well as G (6%) were effective in reducing shrinkage in AAS concrete; AEA also greatly improved workability. Thus, it was considered that AEA is the most suitable for use in AAS concrete.
(f) Replacement of the ordinary slag by UFS (at the 5% replacement level) improved workability and strength.

Acknowledgments

Financial support for this project was provided by Independent Cement and Lime, Blue Circle Southern Cement and Australian Steel Mill Services. Assistance with the laboratory work provided by Jeff Doddrell, Roger Doulis, and Peter Dunbar is also gratefully acknowledged.

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