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Alkali activation of Australian slag cements

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Abstract

Investigation of alkali activation of Australian slag (AAS) was carried out using sodium silicate, sodium hydroxide, sodium carbonate, sodium phosphate, and combinations of these activators. Compressive strengths in the range from 20 to 40 MPa were achieved for the pastes. The most effective activator was liquid sodium silicate. With this activator, the effect of curing at 60°C, modulus (M_s) of sodium silicate solution and concentration of alkalis on the compressive strength and setting times have been studied. On the basis of this investigation, a sodium silicate solution with a low Na content and $M_s = 0.75$ is recommended for formulation of AAS concrete.

Keywords: Alkali activated cement; Workability; Shrinkage; Granulated blast-furnace slag

Alkali-activated slag (AAS) and Portland-granulated blast furnace slag cements have a great potential for using industrial by-products and saving energy in cement production [1]. Due to significant interest in new types of binder, research on alkali activation of slags has been conducted in many countries including the United States [1–3], England [4–6], Canada [7–9], China [10–13], Australia [14], and Russia [15–18]. Alkali activation of local slags, hydration products, and properties of AAS pastes and concretes were studied. Concrete manufactured using AAS and slag cements have been reported to have high chemical resistance and superior durability in aggressive environments as compared with ordinary Portland cement (OPC) concrete [10,15,16]. AAS concrete is based on another type of binder that, in many respects, behaves differently than OPC. However, there is insufficient knowledge about the relationship between AAS properties and processing parameters of alkali activation.

Slags are by-products of metallurgical industry and consist mainly of calcium-magnesium aluminosilicate glass. The most commonly produced slags are from the iron and steel industry, called blast furnace slag. As by-products they have a variable composition depending on the raw materials and the industrial process; hence, each slag differs in response to activation [5,6]. In each case it is necessary to probe a number of activators to find the most suitable one. Previously reported results from Russia and China have suggested that the best results of compressive strength were obtained in AAS concretes activated with waterglass solution (liquid sodium silicate) [10,16].

In China, compressive strength of 60 to 150 MPa for AAS concrete activated by waterglass was achieved without heat treatment or special additives [10,11]. In Scandinavian countries, F-activator, composed of NaOH, Na_2CO_3, and lignosulfonate as the main ingredients together with sodium gluconate and tributyl phosphate as additives, was used [6]. The F-activator yielded good results for a number of local slags. Recently, the results on activation of Australian slag by combination of NaOH and Na_2CO_3 were published by Collins and Sanjayan [14]. The AAS concrete had compressive strength about 20 MPa at 30 days, which was lower than for OPC concrete having the same water-to-binder (w/b) ratio = 0.5. AAS showed high early strength, exceeding that of OPC concrete.

Because the investigations revealed that activation depends on the chemical and phase compositions of slags, the type and concentration of activators must be studied for different slags. Furthermore, it is critical to know the compressive strength, workability, setting, and shrinkage characteristics for cements used in construction. The study of these properties for an Australian AAS is reported in this paper.

1. Experimental programme

1.1. Materials

1.1.1. Slag

Granulated blast furnace slag supplied by SteelCement Ltd., Australia, was used in the investigation. The composi-

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Table 1
Composition of cementitious materials

Oxide (%)	Cement*	Fly ash†	Slag‡
SiO_2	19.9	46.13	35.04
Al_2O_3	4.62	25.03	13.91
Fe_2O_3	3.97	7.25	0.29
CaO	64.27	8.02	39.43
MgO	1.73	1.81	6.13
K_2O	0.57	0.63	0.39
Na_2O	0.15	4.74	0.34
TiO_2	0.23	4.70	0.42
P_2O_5	0.12	0.42	<0.1
MnO	0.06	0.03	0.43
SO_3	2.56	0.12	2.43
Sulfide sulphur as S^{-2}			0.44
Cl			80 ppm
Loss on ignition	2.9	0.53	1.45

* Type I/II, Geelong, Victoria, Australia.

† Pozzolanic fly ash, Pozzolanic Enterprises Pty. Ltd., Milton, Brisbane, Australia.

‡ SteelCement Ltd., PortMelbourne, Australia.

tions of the cement, fly ash, and slag are shown in Table 1. The blast furnace slag is a granulated product ground to fineness of about 460 m^2/kg and is neutral with the basicity coefficient $Kb = (CaO + MgO)/(SiO_2 + Al_2O_3)$ equal to 0.93. The slag is supplied with 2% gypsum, which is blended with the slag during grinding. The blast furnace slag consists of glass (~90%) and some crystalline components such as gehlenite, merwinite etc.

1.1.2. Activators

Different alkali-containing activators were used for activation of slag, e.g., liquid sodium silicate,[1] sodium hydroxide solution,[2] compound activator, sodium carbonate,[3] and sodium orthophosphate.[4]

Table 2 shows the summary of specimens and tests. Liquid sodium silicate and sodium hydroxide were blended in different proportions, providing the modulus in solution (mass ratio of SiO_2 to Na_2O) ranging from 0.75 to 1.5. Three levels of Na concentration in mixtures, 4%, 6%, and 8%, were investigated. Solutions of sodium hydroxide, sodium carbonate, and sodium orthophosphate were used for slag activation at a concentration of 7% Na in mixtures with slag. A multicompound activator, composed of sodium hydroxide (6.9% Na), sodium carbonate (2.5% Na), and lignosulphonate (0.5 mass %), with a total Na content of ~9.4% in the mixture was used in the investigation.

A w/b ratio of 0.5 was used to prepare paste and mortar specimens. In the case of mortars, the sand-to-cementitious binder ratio was 2:1. The sand was a locally produced river sand from Lynhurst.

Corresponding to the concentration of the alkaline activator added in a solution, the amount of water was varied to maintain a constant w/b ratio. In some samples, a mixture of 30% cement and 70% slag was used. The activation of Portland cement/slag mixtures was probed with liquid sodium silicates $M_s = 0.75$, and 4% Na in the mix. Both sodium hydroxide and liquid sodium silicate were used for mortars with moduli (M_s) in solutions ranging from 0.75 to 1.5 at two levels: 4% and 8% Na.

In an attempt to improve workability at $M_s = 1.25$ and 8% Na, 30% fly ash was introduced to replace the slag in mortars.

The size of the paste and mortar cylinders for compressive strength testing was 35 mm in diameter and 70 mm in length. The cylindrical specimens were cured in a fog room until tested. To evaluate the effect of curing temperature on strength development in the sodium silicate-activated paste, the cylinders were sealed and cured at 60°C during the first 7 days, and up to 1 month.

For shrinkage measurements, concrete with the corresponding binder was used. The shrinkage prisms had 75-mm square cross-section and approximately 285 mm in length. Three to six specimens were prepared for each test condition. The prisms were demolded 24 hours after casting when the first reading of length was taken. The shrinkage prisms were stored afterwards in the drying room at 20°C and relative humidity = 55%.

Portland cement pastes, mortars, and concrete were used as standard specimens for comparison with AAS pastes, mortars, and concrete.

2. Testing procedures

Compressive strength measurements of pastes and mortars were performed on a Baldwin machine in a load control regime with a loading rate of 2 MPa/min. Three to six cylinders were tested for each data point. The specimens were tested at 1, 3, 7, and 28 days after casting.

The activity of AAS is shown by transition from a paste to a hard mass when slag is mixed with water and activating admixtures. It indicates whether a material is of a quick or slow-setting nature. The setting times of the AAS pastes were determined by means of the Vicat apparatus. A needle of a known weight and area was used in this method [19]. Two distinct stages of setting were recorded in the laboratory for pastes: the initial set (time of commencement of the setting) and the end, or final, set.

3. Results

3.1. Strength

3.1.1. Pastes

Fig. 1 shows the results of the compressive strength test for the pastes after 28 days of hydration. The strengths obtained were in the range from 20 to 40 MPa. Liquid sodium

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[1] PQ Australia Ltd., 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] Ajax Chemicals, technical grade.

Table 2
Summary of experimental programme

Type of specimen	Type of activator	Concentration	Curing temperature	Compressive strength	Workability	Shrinkage
Paste, w/b = 0.5	Liquid sodium silicate	4%, 6%, 8% Na, $M_s = 0.75, 1, 1.25, 1.5$	Room	1, 3, 7, 28 days	X	X
Mortar, w/b = 0.5	Liquid sodium silicate	4%, 8% Na, $M_s = 0.75, 1, 1.25, 1.5$	Room	1, 3, 7, 28 days	—	X
Paste, w/b = 0.5	Liquid sodium silicate	6%, 8% Na, $M_s = 0.75$, 1, 1.25, 1.5	60°C	3, 7, 28 days	—	—
Paste and mortar, OPC/slag 30/70, w/b = 0.5	Liquid sodium silicate	4% Na, $M_s = 0.75$	Room	1, 3, 7, 28 days	X	—
Mortar, fly ash/slag 30/70, w/b = 0.5	Liquid sodium silicate	8% Na, $M_s = 1.25$	Room	1, 3, 7, 28 days	X	—
Paste, w/b = 0.5	Na_2CO_3	7% Na	Room	1, 3, 7, 28 days	X	X
Paste, w/b = 0.5	Na_3PO_4	7% Na	Room	1, 3, 7, 28 days	X	X
Paste, w/b = 0.5	NaOH	4%, 7%, 10% Na	Room	1, 3, 7, 28 days	X	X
Paste, w/b = 0.5	Na_2CO_3 NaOH	2.5% Na 6.9% Na	Room	1, 3, 7, 28 days	X	X
OPC paste, mortar, w/b = 0.5	—	—	Room	1, 3, 7, 28 days	X	X

silicate was the most effective activator and produced paste strengths greater than 30 MPa (Fig. 2a).

The effects of other activators on the strength development were not as significant as the sodium silicate. Fig. 2b shows the results of the compressive strength test for slag pastes activated by sodium hydroxide and compound activator. The strength of the slag pastes was comparable to the strength of OPC until 7 days, after which it was lower

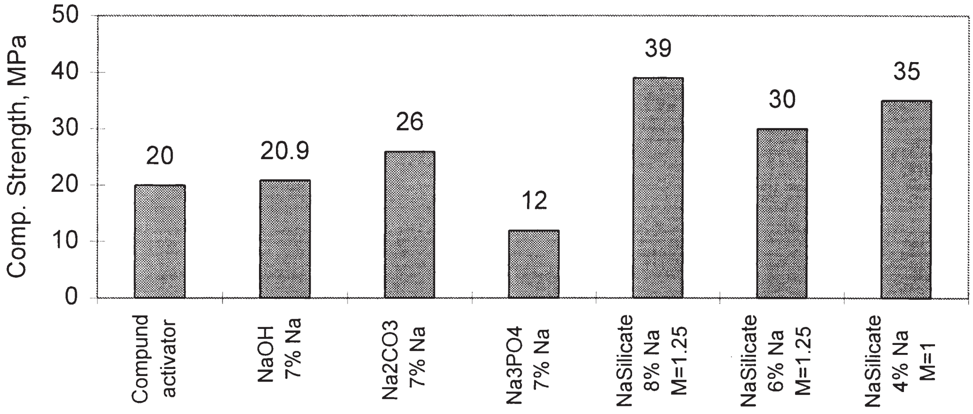

Fig. 1. The 28-day compressive strength of alkali-activated slag pastes.

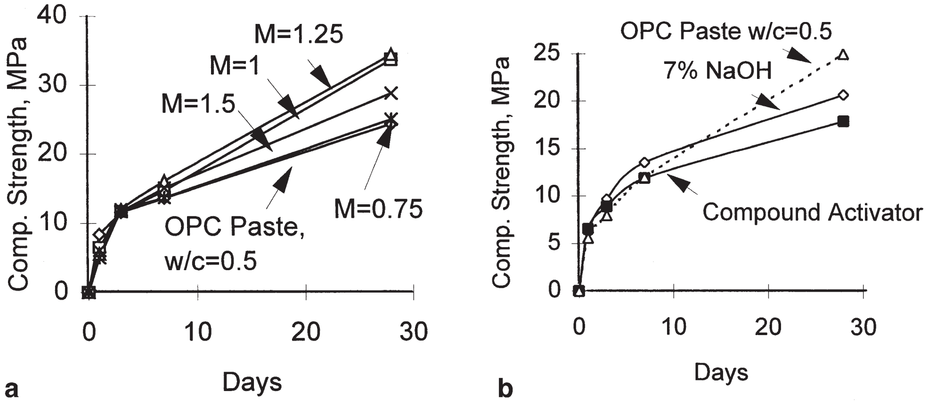

Fig. 2. Compressive strength of pastes activated with (a) Na Silicate with 4% of Na in the mix and (b) sodium hydroxide and compound activator.

than the strength of OPC paste with the same w/b ratio. The strength and workability for the sodium orthophosphate and sodium carbonate-activated pastes were low.

The effect of sodium silicate on paste activation was investigated further. The mix with a concentration below 4% Na in slag could not provide sufficient activation for paste hardening during the first 24 h of hydration. Therefore, 4% was chosen as the lowest level necessary for activation. Fig. 2a shows the evolution of the compressive strength for pastes activated by the sodium silicate solution with 4% Na in the mix and modulus ranging from 0.75 to 1.5. Fig. 3 shows the relationship of compressive strength vs. sodium silicate solution modulus. In comparison with Fig. 2a, at all the three levels of Na concentration (4%, 6%, and 8%), slag activated by sodium silicate solution achieved a strength exceeding that of OPC pastes of the same w/b ratio.

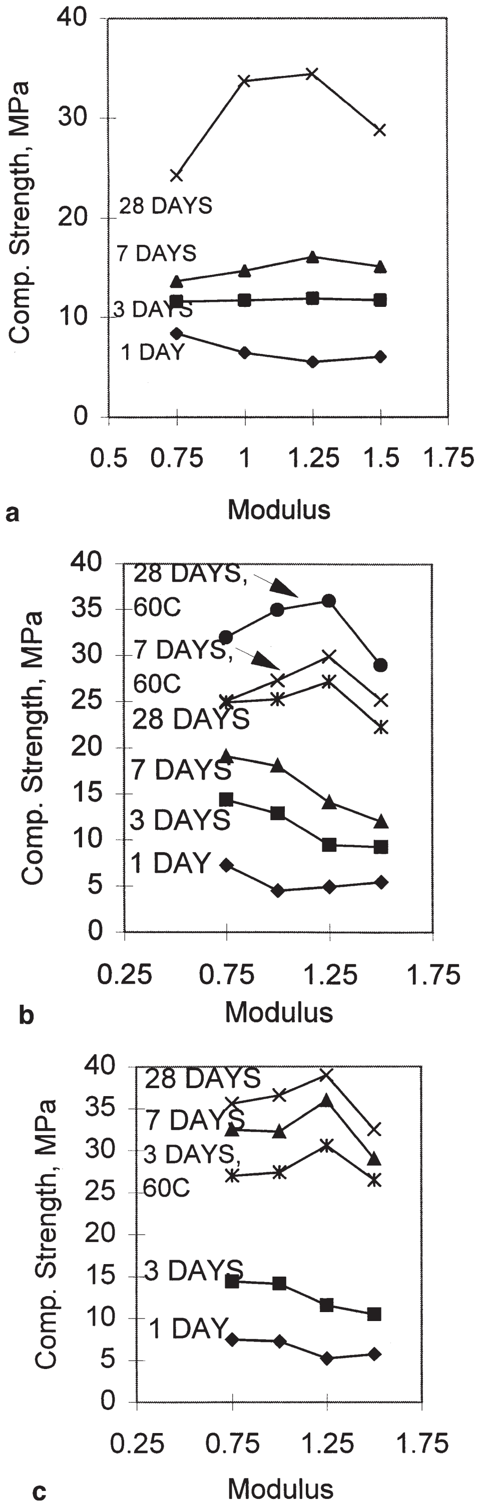

Fig. 3. Compressive strength of pastes activated by sodium silicate with (a) 4% Na, (b) 6% Na, and (c) 8% Na.

Compressive strength is slowly growing with an increase in the Na content in the mix. Heat curing at 60°C produced considerable acceleration in strength development. After 7 days of heat treatment, the strength of the material exceeded values for material cured for 1 month at normal temperature (Fig. 3b).

The compressive strength of alkali-activated 30% OPC/70% slag paste and mortar was quite low, (Fig. 4). At the age of 28 days, only 18 MPa was achieved. Such a low strength was not encouraging for further use of this type of binder.

3.1.2. Mortar

Sodium silicate-activated pastes with a high percentage of sodium exhibited a high shrinkage rate, which resulted in microcracking, so that strength data were low. Mortar specimens were prepared, having more similarity with concrete than paste and less susceptible to shrinkage. Fig. 5 shows the evolution of compressive strength for mortar specimens activated by sodium silicate solution with 8% of Na. The compressive strength obtained at 28 days was about 30 MPa.

Fly ash was not well activated at this level of Na in solution, and the strength of fly ash mortar specimens was low (Fig. 5).

3.2. Shrinkage

Shrinkage is an important technical parameter because it influences structural properties and durability of the material. Fig. 6 presents shrinkage observed for AAS concrete with different activators. Fig. 7 shows specimens of AAS pastes stored at 20°C and relative humidity = 55%. Slag activated with sodium carbonate, sodium phosphate, sodium hydroxide, and by compound activator had shrinkage higher than OPC (w/b = 0.5).

A significantly higher shrinkage was observed for specimens with the sodium silicate solution, all of which had visible shrinkage cracks. For all activators, shrinkage increased with concentration of alkalis. For sodium silicates it also depended on the modulus of solution, with a maximum observed at $M_s = 1$.

3.3. Workability studies

Table 3 shows the initial and final setting times determined for AAS pastes. Fig. 8 presents the results of workability studies for sodium silicate-activated pastes with different moduli of the solution. Workability of pastes was

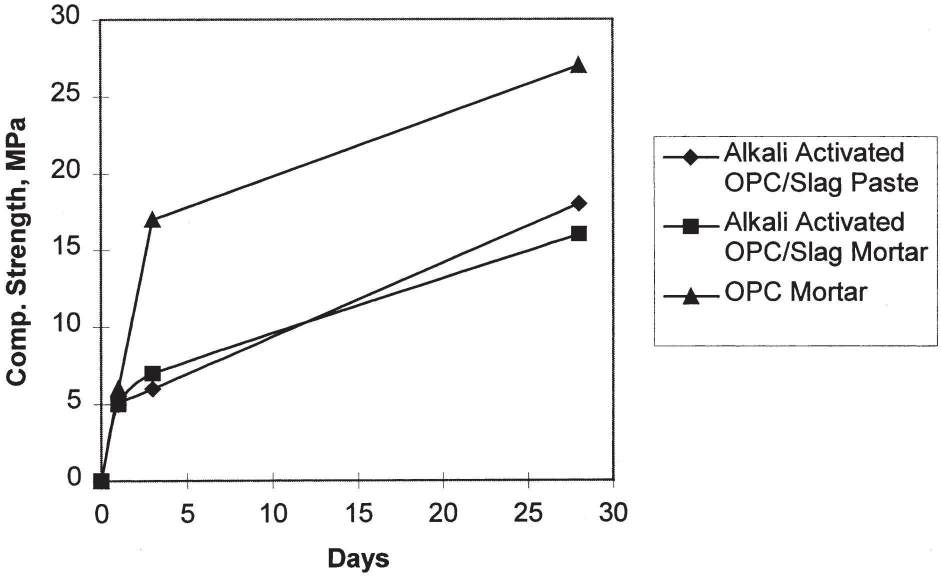

Fig. 4. Compressive strength of alkali-activated OPC/slag paste and mortar.

good at low modulus of solution as $M_s = 0.75$ and dropped rapidly with additional increases in modulus.

Setting times for some other activators also were investigated, namely sodium hydroxide, with 4% and 10% Na in the mix; sodium carbonate and sodium orthophosphate, both with 7% Na in the mix; and OPC/slag activated by sodium silicate with 4% of Na in the mix. It was found that NaOH-activated slag with 4% Na in the mix had normal setting characteristics, but at percentages above 7% significant false set (i.e., the stiffened paste that could be made workable by remixing) is demonstrated with this activator. Slag activated by sodium carbonate and sodium phosphate with 7% Na in the mix was found to be quick setting. OPC/slag blend activated by sodium silicate had characteristics close to quick-setting cements.

4. Discussion

It is known that AAS cements have different rates of strength development and their ultimate strength depends on the degree of activation [1,7,10,15]. Fig. 1 showed that the compressive strength obtained for different activators varied significantly. Previous researchers used calorimetry data and showed that slags exhibited selectivity towards the anion or anion groups in activators that had the same amount of Na [9]. A similar conclusion was reached from our observation of variation in compressive strength of slags that were activated by sodium-containing salts with various anions (Fig. 1).

The best activation in this study was obtained with the sodium silicate solution. It was found that the strength of all

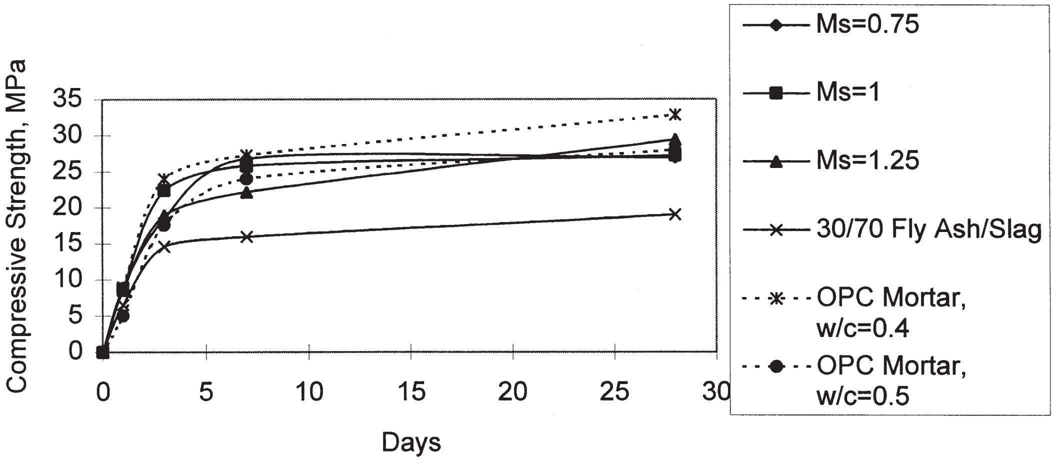

Fig. 5. Compressive strength of sodium silicate-activated mortar specimens with high content of Na in the mix.

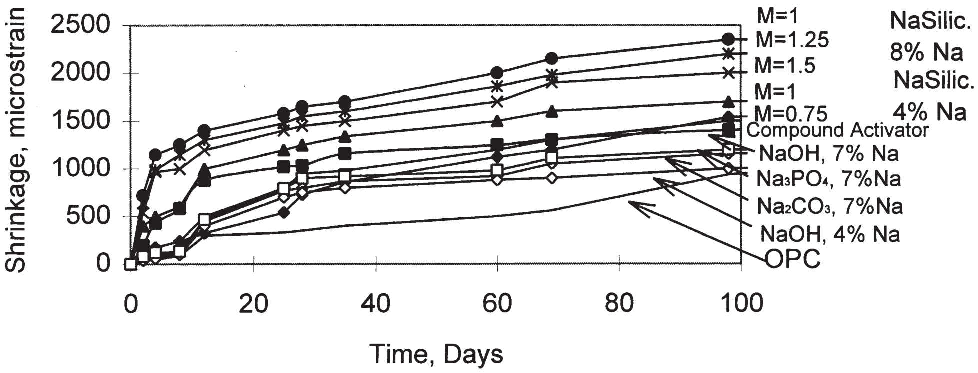

Fig. 6. Shrinkage of AAS concrete.

the sodium silicate-activated pastes exceeded that of Portland cement paste of similar w/b ratio (w/b = 0.5) at early age. The general observation is that high early strength can be obtained with the solution of low modulus, such as $M_s = 0.75$. However, at later ages the maximum shifts towards higher modulus, so that at 28 days maximum strength for all three levels of concentration occurred at $M_s = 1.0$ to 1.25, in agreement with observations of Wang et al. [5] and Wang [6] for neutral slags.

For other activators the strength development was not so fast. According to Ramachadran [20], three types of activators can be used for slags, caustic alkalis (Na, K, or Li hydroxide), nonsilicate salts of weak acids (R_2CO_3, R_2SO_3, R_2S, or RF, R = Na, K, or Li), and silicate salts of type $(R_2O)^{\bullet}(n)SiO_2$. All three types were recommended for use with basic slags, but for neutral or acid slags nonsilicate salts of weak acids are not desirable as they yield a low strength [20]. The slag we studied is neutral, with a basicity index of 0.93. The low compressive strength results for this material with sodium carbonate, sodium othophosphate, and compound activators have supported the assumption that nonsilicate salts of weak acids do not provide good activation for neutral slags.

The compressive strength of the slag/cement blend activated by sodium silicates with 4% Na in the mix was low. It can be due to the effect of alkaline activator on Portland cement hydration, which results in formation of a significant amount of calcium hydroxide and Na-substituted C-S-H (N-C-S-H), which was reported to be less dense and has lower strength than hydration product of cement and water [21,22].

Other researchers have indicated that slag was more sensitive to heat than Portland cement due to its high apparent activation energy [1,9]. Roy and Idorn [1] even suggested that the combined effect of alkalis and heat may be synergetic during early hydration. The results of these experiments support this hypothesis.

It is considered that normal setting, ordinary, and rapid-

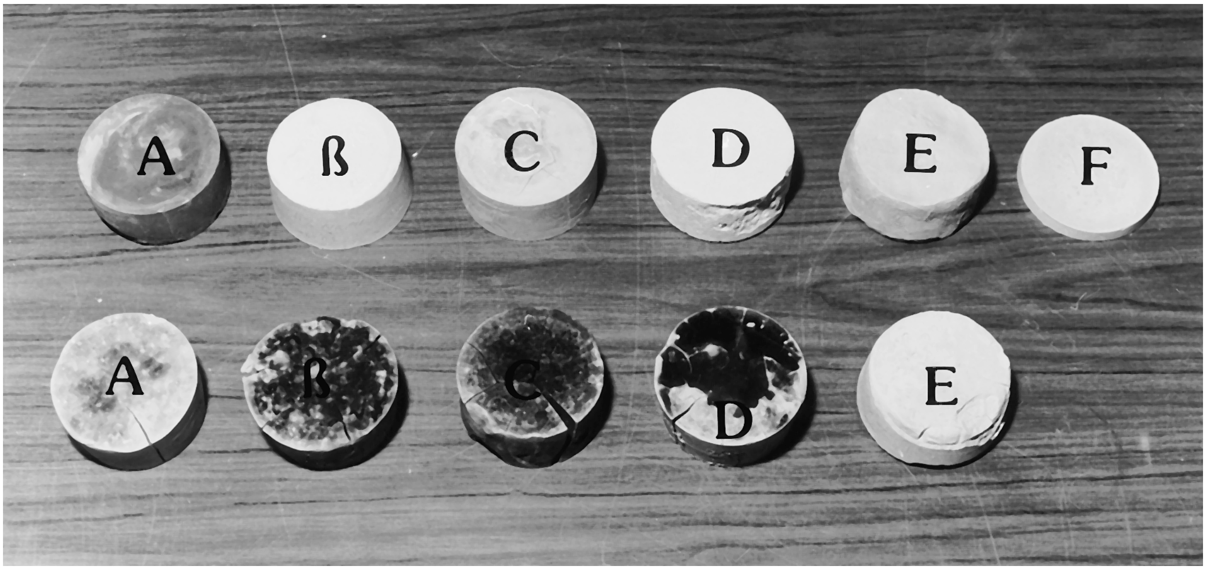

Fig. 7. Shrinkage of AAS pastes. Top row: (A) OPC, w/c = 0.5; (B) NaOH, 4% Na; (C) NaOH, 10% Na; (D) Na_2CO_3, 7% Na; (E) compound activator, 9.4% Na; (F) Na_3PO_4, 7% Na. Bottom row: activator for all samples sodium silicate solution (A) 8% Na, $M_s = 1.5$; (B) 8% Na, $M_s = 1.25$; (C) 8% Na, $M_s = 1$; (D) 8% Na, $M_s = 0.75$; (E) 4% Na, $M_s = 0.75$.

Table 3
Initial and final setting times of AAS pastes

Activator	Initial setting time (min)	Final setting time (min)
Na_2CO_3, 7% Na	30	46
Na_4PO_4, 7% Na	12	30
NaOH, 4% Na, w/b = 0.5	66, bleeding	255
10% NaOH, w/b = 0.5	45	130
Sodium silicate, low % Na, $M_s = 0.75$	40	70
30/70 OPC/slag OPC paste, w/b = 0.5	272	600
OPC paste w/b = 0.4	156	247

hardening Portland cements shall have an initial setting times of not less than 30 min and final setting times of not more than 10 h [23,24]. A quick-setting cement must have an initial setting time of not less than 5 min and a final setting time of not more than 30 min [23,24]. According to these criteria, the sodium silicate-activated slag with $M_s = 0.75$ and 1% and 4% Na complies with the setting time limits for normal setting cements, whereas the sodium silicate-activated slag with 4% Na and $M_s = 1.25$ to 1.5, and 8% Na in the mix exhibit setting times similar to quick-setting cements. Generally, alkali-activated slags are fast-setting materials, whereas blended Portland/slag cements have slow-setting characteristics.

The setting behaviour had strong dependence upon the modulus of sodium silicate solution for both 4% and 8% Na concentrations, although the type of dependence was different. For 8% concentration of Na in the mix the best workability was obtained at $M_s = 0.75$ and $M_s = 1.5$. The relationship between final setting time and modulus is close to quadratic, as shown in Fig. 8a. At 4% concentration of Na in the mix the best workability was obtained at $M_s = 0.75$. The workability of the mix decreased with a high modulus, and the lowest workability was found at $M_s = 1.5$ (Fig. 8b).

Because the increase in strength with Na concentration was quite small, and because of low workability of sodium silicate paste with a high Na content, a formulation with 4% Na in sodium silicate/slag mixtures is recommended for use in the alkali-activated concrete.

Slags activated by liquid sodium silicates had a shrinkage considerably higher than OPC paste. Other researchers also have found that the shrinkage of sodium silicate-activated cements was higher than OPC paste [6]. For all concentrations of liquid sodium silicates the highest shrinkage was observed for modulus $M_s = 1$. The properties of the calcium silicate hydrate (C-S-H) that forms at this modulus is probably responsible for the higher drying shrinkage. Way and Shayan [21] showed that C-S-H formed in the presence of high NaOH concentrations had 0.17 moles Na replacing Ca in C-S-H structure, and the Na-substituted form was less dense than normal C-S-H. The silica gel intermixed with N-C-S-H that was observed at early stages of hydration [6] in slags activated by sodium silicates also may contribute to this phenomenon.

Depending on activators, slag materials can offer great flexibility in performance for different applications. AAS can provide high early strength and is more temperature sensitive than OPC, so its use in precast prestressed concrete production needs further investigation.

5. Conclusions

The results of this initial investigation indicate:

1. Slags exhibit selectivity towards the anion or anion groups of activators that have the same content of Na. Sodium silicates provided the best activation, with compressive strength of pastes and mortars exceeding that of OPC pastes of the same w/b ratio.

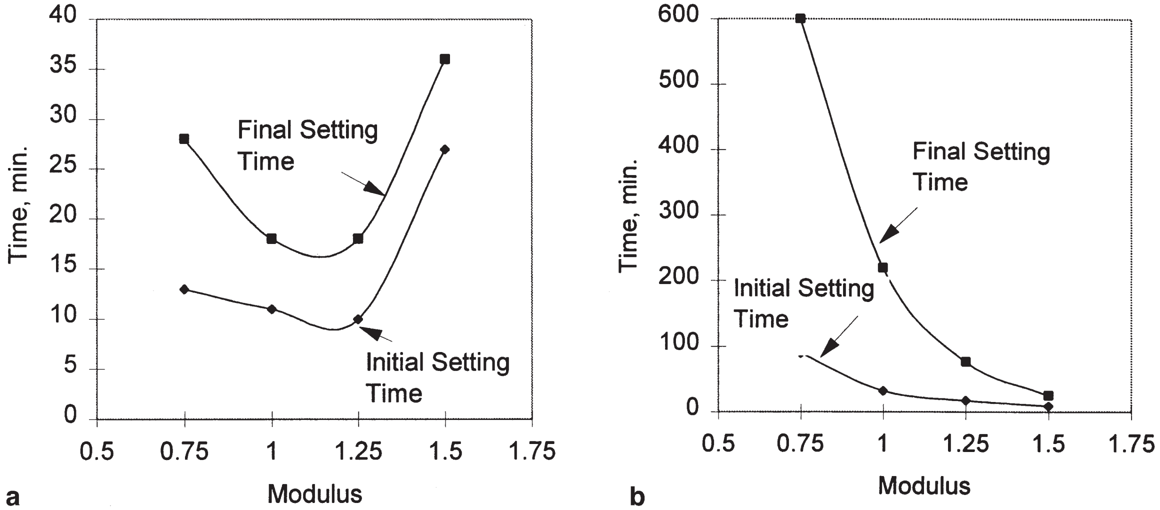

Fig. 8. Setting times for sodium silicate pastes with (a) 8% Na in the mix and (b) 4% Na in the mix.

2. The compressive strength obtained for sodium silicate-activated slag cements was in the range from 20 to 40 MPa and depended on the modulus of the solution and concentration of alkalis. At a high modulus early strength decreased, and the setting time was significantly shortened. At high concentrations of alkalis the slag activated with the sodium silicate solution was found to have a high shrinkage and behaved like a fast-setting cement.
3. Heat treatment had a significant accelerating effect on strength development of slag pastes.
4. Fly ash introduced in AAS at a high percentage (30%) reduced the compressive strength of mortar.
5. OPC/slag mixtures activated by alkalis showed lower strength than the slag alone activated by an alkaline solution.
6. Sodium silicate solution of $M_s = 0.75$, and 4% Na was recommended for use in AAS concrete based on study of workability and compressive strength.

Acknowledgments

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