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Influence of admixtures on the properties of alkali-activated slag mortars subjected to different curing conditions

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ABSTRACT

This paper presents the influence of shrinkage-reducing (SHR) and superplasticizing and set-retarding admixtures (SSRe) on the properties of slag pastes and mortars activated by liquid sodium silicate with different dosage and modulus ratio. Properties in the fresh and hardened state for these binders were investigated by means of measuring some properties including setting time, flowability, flexural strength, compressive strength, carbonation and shrinkage. In this study, fifteen pastes and mortars were prepared. Liquid sodium silicate was used to activate the slag at two sodium concentrations, 4% and 6% by mass of slag. Liquid sodium silicate and sodium hydroxide were blended to obtain 0.75 and 1 modulus ratio of SiO_2/Na_2O. Results showed that although the higher percentage of sodium in the activator produced a higher strength, workability and setting times rapidly decreased with the higher sodium concentration due to instantaneous reaction and quick hardening of slag activated by liquid sodium silicate. None of the admixtures generally had an impact on the setting times of alkali-activated slag (AAS) pastes. SSRe admixture increased the flow rate of AAS mortars while SHR admixture partially affected the flow values of AAS mortars. SHR admixture exhibited a slight decrease in the carbonation depths of AAS mortars. SSRe and particularly SHR chemical admixtures reduced the shrinkage of AAS mortars. However, the shrinkage values of AAS mortars still were higher than those of ordinary Portland cement (NPC) mortars. Curing conditions had a significant effect on the mechanical behavior in the hardened state of AAS mortars compared to NPC mortars.

1. Introduction

Cement manufacturers blends or intergrinds mineral admixture with the cement to reduce energy consumption, CO_2 emission and increase production [1]. Slag is one of the mineral additives often used as a supplementary cementitious material and partial replacement to Portland cement and, it is obtained during steel production as a by-product. Even though the cement industry utilizes large quantities of slag, there is a still great volume available for use as an alternative binder. The main benefits of using slag in concrete are the better durability and lower heat of hydration as compared to NPC binder. However, the low early strength of these concretes constitutes a restriction in practical applications. This problem can be surpassed by using AAS which is a new type of binder used in concrete technology. These binders, which are a blend of blast furnace slag and activators, have received much attention from the academic field owing to significant advantages such as the lower energy demands and CO_2 emissions in comparison with the manufacturing of Portland cement. Therefore, AAS cement should be considered as an effective binder to produce more qualified concrete than NPC concrete [2].

Results of many researches on this material have been published recently. In comparison with NPC, AAS binders have been found to have higher strength and good performance in chemical attack, frost–thaw cycles and high temperatures [3–6]. Sodium silicate-based activator (either blend of sodium silicate and sodium hydroxide or only sodium silicate) was revealed to have the best strength development performance compared to only sodium hydroxide and sodium carbonate activators [7,8]. However, it has been reported that these binders have a workability problem, and that the shrinkage in most cases exceeds that of NPC concrete [9–11]. Additionally, some researchers [12] indicated that the carbonation rate of AAS concrete was higher than Portland cement concrete for the equivalent compressive strength grade. The disadvantages in question constitute an obstacle for the definitive use of AAS as an alternative to normal Portland cement binders. Thus, these quick setting and high shrinkage problems should be solved to make the use of AAS binders widespread as construction materials.

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Even though the effects of the various chemical admixtures, developed to be used with NPC, have been investigated comprehensively in Portland cement concretes, mortars and pastes so far, their effects on other binders like AAS binders remain to be investigated. In the literature, some researches on AAS binders containing various chemical admixtures have been published [13–15]. However, there are some differences in the results due to some factors such as test conditions, nature and concentration of activator, slag composition, type and dosage of admixture used. Since the utilization of a new material in the cement industry requires a lot of surveys, the additional studies should be performed to contribute to the knowledge at this level. Therefore, the aim of this study is to investigate the influence of SHR and SSRe admixtures on the properties of slag pastes and mortars activated by liquid sodium silicate with the different dosage and modulus.

2. Experimental study

2.1. Materials

The cement used was CEM I 42.5 R. Chemical composition and physical properties of cement and ground granulated blast furnace slag obtained from OYAK Adana cement factory are given in Table 1. The particle size distributions of these materials, which were obtained using a laser scattering technique, are presented in Fig. 1. Sand used in the experimental study was standard Rilem Cembureau type according to TS EN 196-1 [16]. Liquid sodium silicate was used in the alkali activation of slag. Liquid sodium silicate had a SiO_2/Na_2O ratio (modulus, M_s) of 2. SHR based on polypropylenglycol and SSRe based on modified polymer liquid were used as chemical additives. One percent of each admixture by mass of binder was added to the activator solution.

2.2. Experimental program

Water/binder (w/b) ratio of 0.5 was used to prepare paste and mortar specimens throughout the experimental program. In the case of mortars, the sand to cementitious binder ratio was 3:1. For liquid sodium silicate activator, SiO_2/Na_2O ratios of 0.75 and 1 were chosen. These different ratios were obtained by adding sodium hydroxide to liquid sodium silicate. Sodium concentrations in the mixture proportions were also chosen as 4% and 6% by mass of slag. The amount of water in the liquid sodium silicate activator was taken into consideration while adjusting the amount of water to obtain 0.5 w/b ratio in all mixtures. A summary of the experimental program is presented in Table 2.

Table 1
Physical, chemical and mechanical properties of cement and slag.

Chemical composition (%)	Cement	Slag	Physical properties of Portland cement	
SiO_2	18.69	33.78	Specific gravity	3.12
Al_2O_3	5.61	9.55	Initial setting time (min)	190
Fe_2O_3	2.52	0.88	Final setting time (min)	225
CaO	62.68	39.80	Volume expansion (mm)	1.0
MgO	2.63	6.80	Specific surface (Blaine) (cm^2/g)	3200
Na_2O	0.13	0.32	Compressive strength (MPa) of cement	
K_2O	0.77	0.88	2 days	27.2
SO_3	2.73	1.66	7 days	41.0
Cl^-	0.01	0.03	28 days	51.2
LOI	2.88	2.89	Physical properties of slag	
Insoluble residue	0.96	–	Specific gravity	2.78
Free CaO	0.93	–	Specific surface (Blaine) (cm^2/g)	5200
			Pozzolanic activity index (%) of slag	
			7 days	62
			28 days	94

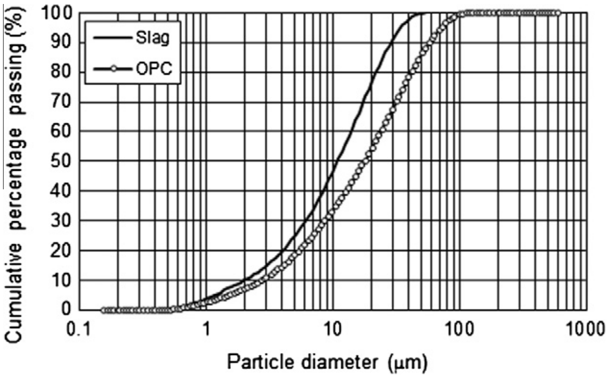

Fig. 1. Particle size distributions of cement and slag.

Table 2
Summary of experimental program.

Mix no.	Binder	Activator	Concentration	Admixture
1	OPC	–	–	–
2	AAS	Liquid sodium silicate	4% Na, M_s = 0.75	–
3	AAS	Liquid sodium silicate	6% Na, M_s = 0.75	–
4	AAS	Liquid sodium silicate	4% Na, M_s = 1.00	–
5	AAS	Liquid sodium silicate	6% Na, M_s = 1.00	–
6	OPC	–	–	SSRe
7	AAS	Liquid sodium silicate	4% Na, M_s = 0.75	SSRe
8	AAS	Liquid sodium silicate	6% Na, M_s = 0.75	SSRe
9	AAS	Liquid sodium silicate	4% Na, M_s = 1.00	SSRe
10	AAS	Liquid sodium silicate	6% Na, M_s = 1.00	SSRe
11	OPC	–	–	SHR
12	AAS	Liquid sodium silicate	4% Na, M_s = 0.75	SHR
13	AAS	Liquid sodium silicate	6% Na, M_s = 0.75	SHR
14	AAS	Liquid sodium silicate	4% Na, M_s = 1.00	SHR
15	AAS	Liquid sodium silicate	6% Na, M_s = 1.00	SHR

The initial and final setting times of AAS and NPC pastes were measured using Vicat apparatus in accordance with TS EN 196-3 [17].

According to TS EN 1015-3 [18], flow table tests were conducted to determine mortar flowability, with and without admixtures, after 0, 15, 30, 45 and 60 min (end of mixing). For each mortar mixture, the diameter was measured in four directions following the flow of mortar onto the table of test apparatus.

Prismatic specimens with 40 × 40 × 160 mm dimensions and shrinkage specimens measuring 25 × 25 × 285 mm were prepared from both fresh NPC and AAS mortar mixes for the tests. After 24 h, the specimens were demoulded and cured in three ways until the time of testing; One group of specimens were placed in a humidity cabinet at 23 ± 2 °C with 95% relative humidity (moist curing–MC) while second group of specimens were cured in a humidity cabinet at 23 ± 2 °C with 50% relative humidity (dry curing–DC). In the third method, the specimens were immersed in water and the heater was turned on. The water temperature reached at 65 °C in 2 h and the water temperature was maintained at 65 °C for 5 h. Subsequently, the heater was turned off and, after the cooling periods of the specimens, they were placed in a humidity cabinet at 23 ± 2 °C with 50% relative humidity (heat curing–HC).

The strength tests of the specimens were conducted at 2, 7 and 28 days of age according to TS EN 1015-11 [19]. For flexural strength test, three prismatic specimens from each mixture were used and tested by one-point loading configuration with span of 10 cm. The compressive strength test was performed using six broken pieces of test prisms remained from flexural strength test. The flexural strengths were determined by taking the average of three test results whereas the compressive strengths were determined as the average of six test results.

Table 3
The initial and final setting times of OPC and AAS pastes.

Mix no.	Binder	Admixture	Initial setting time (min)	Final setting time (min)
1	OPC	–	390	475
2	AAS, 4% Na, $M_s = 0.75$	–	228	378
3	AAS, 6% Na, $M_s = 0.75$	–	83	167
4	AAS, 4% Na, $M_s = 1.00$	–	47	123
5	AAS, 6% Na, $M_s = 1.00$	–	51	102
6	OPC	SSRe	845	1150
7	AAS, 4% Na, $M_s = 0.75$	SSRe	392	638
8	AAS, 6% Na, $M_s = 0.75$	SSRe	116	184
9	AAS, 4% Na, $M_s = 1.00$	SSRe	50	135
10	AAS, 6% Na, $M_s = 1.00$	SSRe	47	90
11	OPC	SHR	468	588
12	AAS, 4% Na, $M_s = 0.75$	SHR	302	608
13	AAS, 6% Na, $M_s = 0.75$	SHR	105	160
14	AAS, 4% Na, $M_s = 1.00$	SHR	70	190
15	AAS, 6% Na, $M_s = 1.00$	SHR	55	100

A phenolphthalein method was used to follow the pH of mortar specimens in the carbonation experiments, and the tests were conducted at 2, 7 and 28 days of age. At the time of measurement, a 1% phenolphthalein solution in alcohol was sprayed on a broken surface of the mortar prism after flexural strength test, and the depth of neutralization was measured. The values were expressed by taking the average of three specimens.

The shrinkage values of AAS and NPC mortar mixes were measured until 180 days according to the relevant standard [20]. For shrinkage measurements, two prismatic specimens with 25 × 25 × 285 mm dimensions were prepared from each mixture and demoulded the day after. The initial length of the shrinkage specimens was measured before they were subjected to three different curing environments.

3. Results and discussion

3.1. Setting tests

The initial and final setting times for NPC and AAS pastes are presented in Table 3. It was observed that the setting times of AAS pastes decreased with an increase in the sodium concentration of the alkali activator. Furthermore, the setting times of AAS pastes also shortened with an increase in SiO_2/Na_2O ratio (M_s) and those of AAS pastes were much shorter than those of NPC paste. These results are in agreement with other researches [2,8,9,21].

As known, a binder should have an acceptable setting time long enough to allow the transportation and casting. If the setting time is too long, it is not economical. Contrary to, it may cause the irreversible mistakes if the setting time is very short. Usually, AAS binders show rapid-setting. Thus, SSRe and SHR chemical admixtures were used in this study to regulate the setting times of AAS pastes. It was observed that these admixtures generally showed no significant effect on the initial and final setting times of AAS pastes, with the exception of the sodium silicate-activated slag paste with $M_s = 0.75$ and 4% Na which exhibited the satisfactory setting times. The initial and final setting times of this paste in question were 392 and 638 min, respectively. Additionally, their efficiencies of SSRe and SHR admixtures decreased with an increase in the Na dosage and silicate modulus of activator. However, the effect of these chemical admixtures on the setting times of NPC paste was much greater than on AAS pastes and especially SSRe admixture lengthened the setting times of NPC paste. These results agree with those reported elsewhere [22].

Table 4
The flow values of AAS and OPC mortars.

Mix no.	Binder	Admixture	Flow values (mm)				
			0′	15′	30′	45′	60′
1	OPC	–	154	144	135	130	119
2	AAS, 4% Na, $M_s = 0.75$	–	149	128	127	120	116
3	AAS, 6% Na, $M_s = 0.75$	–	160	137	120	114	105
4	AAS, 4% Na, $M_s = 1.00$	–	151	135	124	115	108
5	AAS, 6% Na, $M_s = 1.00$	–	172	145	118	112	103
6	OPC	SSRe	172	168	146	139	131
7	AAS, 4% Na, $M_s = 0.75$	SSRe	165	147	137	130	125
8	AAS, 6% Na, $M_s = 0.75$	SSRe	173	148	130	122	109
9	AAS, 4% Na, $M_s = 1.00$	SSRe	156	144	132	126	116
10	AAS, 6% Na, $M_s = 1.00$	SSRe	183	159	126	100	100
11	OPC	SHR	157	145	135	131	122
12	AAS, 4% Na, $M_s = 0.75$	SHR	149	138	131	125	115
13	AAS, 6% Na, $M_s = 0.75$	SHR	160	137	128	122	110
14	AAS, 4% Na, $M_s = 1.00$	SHR	153	139	128	119	115
15	AAS, 6% Na, $M_s = 1.00$	SHR	177	137	127	119	113

3.2. Flow tests

The results of the flow table tests of mortar mixtures are presented in Table 4. As seen in Table 4, the flow values of mortars decreased in time due to some factors such as the evaporation of free water in mixture, the setting of binder paste. Additionally, the flow values of AAS mortars were less than NPC mortar and the workability of AAS mortars also decreased with an increase in the sodium concentration of the alkali activator. Similarly, M_s modulus increasing from 0.75 to 1 negatively affected the flowability in AAS mortars.

Compared to the slag mortars without admixtures, SSRe admixture increased the flow rate of AAS mortars during the test time of 60 min. and SSRe showed a positive effect on mortar workability. Bakharev et al. [13] indicated that slag activated by liquid sodium silicate had a reasonable response to retarders in terms of workability, and that no adverse effect on drying shrinkage was observed although the early strength was relatively impaired. These results are close to our findings. This improvement in the workability also conforms to the results of other researchers [9,23]. However, SSRe admixture reduced the workability of the slag mortar activated by liquid sodium silicate with 6% Na and $M_s = 1$ after half an hour. This could be attributed to the modification of the chemical structure of the admixture in high alkaline media [22]. In addition to this, SHR admixture slightly increased the flow values of AAS mortars. On the other hand, these chemical admixtures enhanced the workability of NPC mortar and the highest values in flowability for NPC mortar were observed in the case of SSRe admixture.

3.3. Compressive strengths

The compressive strength values of AAS and NPC mortars are given in Table 5. As seen in Table 5, AAS mortars were more sensitive to dry curing conditions with low relative humidity than NPC mortars. For the mortars with and without admixtures, AAS specimens

Table 5
Compressive strengths of AAS and OPC mortars (MPa).

Mix no.	Binder	Admixture	2-days			7-days			28-days		
			MC	DC	HC	MC	DC	HC	MC	DC	HC
1	OPC	–	22.63	20.94	24.31	27.72	24.81	32.69	35.91	27.39	41.19
2	AAS, 4% Na, $M_s = 0.75$	–	5.88	2.19	37.94	21.22	19.88	43.69	44.97	20.53	44.09
3	AAS, 6% Na, $M_s = 0.75$	–	25.28	16.59	60.19	47.34	33.00	65.44	84.09	34.25	65.88
4	AAS, 4% Na, $M_s = 1.00$	–	2.03	1.72	30.72	11.63	12.98	34.58	41.72	13.94	38.81
5	AAS, 6% Na, $M_s = 1.00$	–	16.25	12.97	56.72	43.25	28.75	59.19	79.69	28.94	60.25
6	OPC	SSRe	21.47	20.01	23.19	29.53	25.66	30.09	36.00	28.09	42.84
7	AAS, 4% Na, $M_s = 0.75$	SSRe	3.56	1.63	38.84	23.03	19.00	44.84	42.69	19.38	45.32
8	AAS, 6% Na, $M_s = 0.75$	SSRe	23.28	13.88	60.16	51.31	32.45	68.22	85.75	33.63	65.19
9	AAS, 4% Na, $M_s = 1.00$	SSRe	1.31	1.44	29.72	15.16	12.38	37.94	39.22	13.16	38.41
10	AAS, 6% Na, $M_s = 1.00$	SSRe	16.94	10.50	56.45	43.13	25.38	61.22	80.19	28.25	60.44
11	OPC	SHR	21.93	19.41	26.19	28.04	26.22	31.13	34.94	27.03	40.66
12	AAS, 4% Na, $M_s = 0.75$	SHR	4.44	1.03	32.19	24.94	21.44	41.06	42.53	19.28	42.22
13	AAS, 6% Na, $M_s = 0.75$	SHR	26.18	8.63	55.66	46.88	33.19	58.00	81.59	34.28	64.72
14	AAS, 4% Na, $M_s = 1.00$	SHR	1.09	0.63	27.09	12.34	14.56	37.06	41.50	15.04	39.56
15	AAS, 6% Na, $M_s = 1.00$	SHR	15.44	5.81	53.53	43.91	27.23	59.69	78.59	29.53	61.44

subjected to 23 ± 2 °C and 50% relative humidity showed the lowest compressive strengths compared to other curing conditions and these specimens did not exhibit a remarkable strength development at the end of 28 days of age. Additionally, depending on the lack of moisture, liquid sodium silicate with 4% Na could not provide an effective activation for the slag mixtures in terms of compressive strength during the tests. This event results from the loss of water in mixture due to dry curing conditions. When cured at high temperatures, AAS mortars developed strength quite rapidly, as reported earlier [24]. Accordingly, the highest compressive strength values at 2 and 7 days were obtained from AAS mortars subjected to 65 °C temperature in water for 5 h since heat treatment significantly accelerated the strength of AAS mortars at early ages. However, after the elevated temperature, the fact that the samples were kept under dry curing conditions at 23 ± 2 °C with 50% relative humidity which negatively affected the strength developments at later stages. Therefore, the highest compressive strengths of 28 days were obtained from AAS mortars exposed to 23 ± 2 °C with 95% relative humidity. These findings comply with those of other researches [11,25–28] reporting that the strength development of AAS binder was susceptible to the type of curing environment. They also stated that a lack of moist curing reduced compressive strength, increasing the level of micro cracking in comparison with moist curing.

The compressive strength values of AAS mortars also decreased with an increase in SiO_2/Na_2O ratio and maximum strength was obtained from the solution having low modulus ($M_s = 0.75$). However, the compressive strength of AAS mortars increased with an increase in the sodium concentration of the activator for each curing age without regard to curing conditions. These results agree with those reported by other authors [8,9,29,30]. This improvement in the compressive strengths can be explained in this way: The microstructural development of AAS binders largely results from the synthesis of the anions of slag and the cations in the alkaline activators. The chemical reaction occurring by ion exchange between the silicate anions of slag and cations of alkaline activators causes the formation of silica gel. By additional reacting with calcium ion of the slag, this silica gel may turn into silica-rich calcium silicate hydrates gel (CSH) [31].

The use of SSRe and SHR admixtures caused a decrease in the compressive strength of AAS mortars at the age of 2 days, retarding the strength development at early stages. However, this situation disappeared gradually and it was observed that neither of them showed any negative influence on the compressive strengths of AAS mortars at later ages. These strength findings obtained are similar to those reported by other researchers [13,22] although there are several variations in the literature [13–15,23] depending on some differences in the conditions were activated slag pastes, mortars or concrete were prepared (slag composition, alkali activator type and concentration, nature and dosage of admixture, etc.).

3.4. Flexural strengths

Table 6 shows the flexural strength values of AAS and NPC mortars. As seen in Table 6, the flexural strengths of AAS mortar increased when the sodium concentration of the activator increased for a constant SiO_2/Na_2O ratio. On the other hand, the SiO_2/Na_2O ratio at which the flexural tensile strengths of AAS mortars became the highest was 0.75 for a constant sodium concentration.

The unfavorable effect of dry curing conditions on the flexural strength was more serious than the compressive strength case. Contrary to expectation, the flexural strength values of the slag mortars activated by liquid sodium silicate with high Na concentration decreased in time. Namely, for the dry cured samples without admixtures, the flexural strengths at 2, 7 and 28 days for the slag mortar activated by liquid sodium silicate with 6% Na and $M_s = 0.75$ were 3.87 MPa, 2.73 MPa and 2.34 MPa, respectively. In terms of the flexural strength, the inferior performance of the AAS prisms results from lack of moist curing. The strength of AAS binders is sensitive to the relative humidity of the curing environment. Under the dry curing conditions, cracking is more frequent and intense for concrete with binders composed of AAS than NPC and slag blended cements [32]. Malolepszy and Deja [33] studying on AAS binder reported that when the AAS samples were exposed to 65% RH and 20 °C, microcracking was observed. Byfors et al. [12] also investigated microcracking on AAS binder and, following microscopic examination, concluded that the cracking frequency was greater when subjected to dry curing conditions, and reported that observed behavior leaded to the reduced flexural strength. The higher magnitude of drying shrinkage also contributes the development of these cracks in time. Finally, the expansion of cracks reduces a valid area of cross-sections, which causes a decrease in the flexural strength.

On the other hand, it was found that when cured at elevated temperatures, the flexural strength values of AAS mortars increased quickly at 2 days. But, the later age strength decreased since these mortars were exposed to 23 ± 2 °C with 50% relative humidity after heat treatment. So, the highest flexural strengths at 7 and 28 days were generally obtained from AAS mortars subjected to 23 ± 2 °C with 95% relative humidity.

SSRe and SHR chemical admixtures slightly reduced the flexural strength of AAS mortars at the age of 2 days, which was also found to valid for the compressive strength. However, these admixtures subsequently exhibited different effects. Namely, SSRe did not

Table 6
Flexural strengths of AAS and OPC mortars (MPa).

Mix no.	Binder	Admixture	2-days			7-days			28-days		
			MC	DC	HC	MC	DC	HC	MC	DC	HC
1	OPC	–	3.63	3.63	4.45	5.39	4.34	7.15	6.90	4.57	7.38
2	AAS, 4% Na, M_s = 0.75	–	1.64	0.70	4.14	5.63	2.62	5.16	8.20	2.73	5.39
3	AAS, 6% Na, M_s = 0.75	–	3.40	3.87	5.70	6.56	2.73	6.09	10.51	2.34	6.26
4	AAS, 4% Na, M_s = 1.00	–	0.94	0.47	3.79	3.28	2.44	4.34	6.50	1.76	4.20
5	AAS, 6% Na, M_s = 1.00	–	3.52	3.28	5.27	6.09	2.62	5.63	9.41	2.11	5.76
6	OPC	SSRe	3.48	3.41	4.69	5.86	4.45	7.50	7.03	4.59	7.85
7	AAS, 4% Na, M_s = 0.75	SSRe	1.17	0.47	4.45	5.74	2.46	5.27	8.25	2.58	5.43
8	AAS, 6% Na, M_s = 0.75	SSRe	3.59	3.28	5.74	6.80	2.81	5.86	10.55	2.34	5.98
9	AAS, 4% Na, M_s = 1.00	SSRe	1.06	0.47	4.34	3.63	2.46	4.57	6.56	1.88	4.69
10	AAS, 6% Na, M_s = 1.00	SSRe	3.52	2.58	4.81	6.37	2.70	5.27	9.30	2.11	5.04
11	OPC	SHR	3.52	3.52	4.69	5.51	4.22	6.79	6.56	4.87	7.15
12	AAS, 4% Na, M_s = 0.75	SHR	1.88	0.47	3.87	6.09	3.44	4.84	7.98	3.51	4.92
13	AAS, 6% Na, M_s = 0.75	SHR	3.87	3.31	5.78	7.07	4.45	6.33	10.55	4.22	6.45
14	AAS, 4% Na, M_s = 1.00	SHR	0.82	0.47	3.75	4.18	2.81	4.02	6.21	3.08	4.22
15	AAS, 6% Na, M_s = 1.00	SHR	3.63	3.08	5.16	6.68	3.69	5.52	10.00	3.87	5.86

have a distinctive impact on the flexural strength values of AAS mortars after 2 days. But SHR admixture, due to its restrictive effect on shrinkage, somewhat inhibited the crack development mentioned above by decreasing the surface tension of the water in the pores and by lowering the capillary tensile strength [34,35]. Thus, in the presence of SHR admixture, the flexural strengths of AAS mortars continued to increase with time and the flexural strengths of AAS mortars containing SHR were higher than those of AAS mixtures without SHR at the end of 28 days.

3.5. Carbonation

As known, carbonation is a continuous process progressing from the outer surface inward at a low rate of speed. The overall rate of carbonation is controlled by the physical properties of the solid binder, especially the porosity and pore size distribution of the material and the CO_2 diffusion through the porous network.

The carbonation values of AAS and NPC mortars under the different curing conditions are given in Table 7. The AAS mortars subjected to 23 ± 2 °C with 95% relative humidity did not show any carbonation during the test time of 28 days since the CO_2 penetration into the mortar was difficult due to the pores being filled with water on a large scale. Additionally, no carbonation was observed in the mortars subjected to other two curing conditions at age of 2 days. However, the depth of the carbonated layer in these mortars subsequently increased since CO_2 ingress increased with time depending on the dry curing conditions with 50% relative humidity.

It was observed from the samples cured in both dry curing and heat curing subsequently dry cured that the carbonation values of AAS mortars generally were higher than those of NPC mortars during the tests. For example, in the dry cured mortars without chemical admixtures, the carbonation depths of NPC mortar were 2.07 mm at the age of 7 days and 5.19 mm at the age of 28 days whereas the carbonation depths of AAS mortars ranged from 2.18 mm to 3.32 mm at the age of 7 days, and 6.10 mm to 11.80 mm at the age of 28 days. These values showed that carbonation was deeper and more intense in AAS mortar than in NPC mortars. This situation may be interpreted as follows: when AAS mortar is exposed to CO_2, the decalcification of the CSH gel takes place. The decalcification causes a loss of cohesion in the mortar and produces larger pore sizes. Consequently, the porosity of binder matrix increases and the CO_2 penetration into the mortar becomes easier. These findings also are in agreement with other researches [36–38] reporting that AAS binders have the higher susceptibility to carbonation compared to conventional cements. Additionally, in the mixtures, an increase in the Na content for a constant SiO_2/Na_2O ratio decreased the carbonation depths of AAS mortars at 7 and 28 days. This probably results from a denser and more compact mortar structure which the increment in the Na concentration accelerating the activation of slag produces. These results comply with other researches [30,39]. Nevertheless, carbonation depths of the dry cured AAS mortars were generally higher than those of the mortars subjected to heat curing for 7 and 28 days. This result may be attributed to the beneficial effect of heat treatment in decreasing the drying shrinkage [40]. Since carbonation in AAS develops mainly along shrinkage cracks occurring at the surface [36], heat curing reduces these shrinkage cracks, thereby causing the lower CO_2 ingress.

SSRe admixture did not make an obvious impact on the carbonation of AAS mortars while SHR admixture somewhat decreased the carbonation depths of AAS mortars. The possible reason for this situation may be explained in this way: AAS mortars significantly shrink during the drying process, leading to the development of a large number of cracks that facilitate the ingress of CO_2 into the mortar. SHR admixture inhibits the formation of these cracks because of its shrinkage-reducing effect and partly improves the resistance of AAS mortars to carbonation [38].

3.6. Shrinkage

The shrinkage results of AAS and NPC mortar specimens subjected to three different curing conditions are presented in Tables 8–10. As seen in all tables, the shrinkage of AAS mortars increased with an increase in the Na concentration of liquid sodium silicate activator for a constant SiO_2/Na_2O ratio. The increase in SiO_2/Na_2O ratio at a constant sodium concentration also increased the magnitude of shrinkage in AAS mortars. These findings are in conformity with those reported earlier in the literatures [8,9,29].

Since the curing condition with high relative humidity decelerated the amount of structural water loss, the shrinkage values of moist cured AAS mortars were lower than those of dry cured ones. In other words, the highest shrinkage values were obtained from the dry cured mortars. On the other hand, heat treatment considerably decreased the shrinkage values of mortars, especially at early ages, because curing at elevated temperatures made the concrete dimensionally more steady and less sensitive to shrinkage [24,41–43]. Reduced shrinkage by heat treatment may be attributed to the lower water content of CSH formed during heat treatment [24].

The chemical admixtures used within the scope of this study had a significant effect on volume stability of AAS mortars. The

Table 7
The evolution of carbonation in time for AAS and OPC mortars (mm).

Mix no.	Binder	Admixture	2-days			7-days			28-days		
			MC	DC	HC	MC	DC	HC	MC	DC	HC
1	OPC	–	0	0	0	0	2.07	1.06	0	5.19	2.10
2	AAS, 4% Na, M_s = 0.75	–	0	0	0	0	3.03	2.65	0	8.89	4.77
3	AAS, 6% Na, M_s = 0.75	–	0	0	0	0	2.04	1.23	0	6.10	2.45
4	AAS, 4% Na, M_s = 1.00	–	0	0	0	0	3.32	2.82	0	11.80	5.11
5	AAS, 6% Na, M_s = 1.00	–	0	0	0	0	2.55	1.87	0	7.19	2.64
6	OPC	SSRe	0	0	0	0	2.22	0.85	0	5.40	1.94
7	AAS, 4% Na, M_s = 0.75	SSRe	0	0	0	0	2.65	2.46	0	8.88	4.66
8	AAS, 6% Na, M_s = 0.75	SSRe	0	0	0	0	2.20	1.02	0	6.18	2.70
9	AAS, 4% Na, M_s = 1.00	SSRe	0	0	0	0	3.63	3.18	0	11.90	5.76
10	AAS, 6% Na, M_s = 1.00	SSRe	0	0	0	0	2.66	1.44	0	7.04	2.98
11	OPC	SHR	0	0	0	0	2.32	1.39	0	5.01	2.36
12	AAS, 4% Na, M_s = 0.75	SHR	0	0	0	0	1.94	2.80	0	8.48	5.08
13	AAS, 6% Na, M_s = 0.75	SHR	0	0	0	0	1.38	1.21	0	5.37	2.90
14	AAS, 4% Na, M_s = 1.00	SHR	0	0	0	0	2.81	3.11	0	11.36	5.90
15	AAS, 6% Na, M_s = 1.00	SHR	0	0	0	0	0	1.66	0	6.90	3.11

Table 8
Shrinkage values of AAS and OPC mortars subjected to moist curing (%).

Mix no.	Binder	Admixture	3 days	7 days	14 days	28 days	60 days	90 days	120 days	150 days	180 days
1	OPC	–	0.0189	0.0414	0.0263	0.0256	0.0262	0.0219	0.0252	0.0284	0.0276
2	AAS, 4% Na, M_s = 0.75	–	0.0239	0.2228	0.2270	0.2281	0.2249	0.2260	0.2312	0.2330	0.2351
3	AAS, 6% Na, M_s = 0.75	–	0.1165	0.3163	0.3246	0.3246	0.3367	0.3382	0.3421	0.3425	0.3430
4	AAS, 4% Na, M_s = 1.00	–	0.2004	0.2377	0.2447	0.2495	0.2521	0.2540	0.2551	0.2567	0.2574
5	AAS, 6% Na, M_s = 1.00	–	0.4629	0.4984	0.5088	0.5042	0.4907	0.4885	0.4886	0.4930	0.4978
6	OPC	SSRe	0.0186	0.0312	0.0321	0.0375	0.0326	0.0388	0.0430	0.0485	0.0464
7	AAS, 4% Na, M_s = 0.75	SSRe	0.0213	0.2015	0.2042	0.2050	0.2034	0.2035	0.2086	0.2097	0.2109
8	AAS, 6% Na, M_s = 0.75	SSRe	0.2737	0.2979	0.2993	0.2963	0.3086	0.3093	0.3096	0.3089	0.3094
9	AAS, 4% Na, M_s = 1.00	SSRe	0.0749	0.2435	0.2347	0.2333	0.2333	0.2400	0.2375	0.2354	0.2354
10	AAS, 6% Na, M_s = 1.00	SSRe	0.4356	0.4581	0.4689	0.4754	0.4705	0.4614	0.4612	0.4653	0.4667
11	OPC	SHR	0.0037	0.0146	0.0162	0.0192	0.0183	0.0199	0.0185	0.0183	0.0204
12	AAS, 4% Na, M_s = 0.75	SHR	0.0182	0.1167	0.1239	0.1288	0.1391	0.1377	0.1330	0.1395	0.1392
13	AAS, 6% Na, M_s = 0.75	SHR	0.1189	0.1930	0.2089	0.2092	0.2126	0.2130	0.2130	0.2121	0.2128
14	AAS, 4% Na, M_s = 1.00	SHR	0.0867	0.2014	0.2260	0.2207	0.2132	0.2102	0.2137	0.2102	0.2147
15	AAS, 6% Na, M_s = 1.00	SHR	0.4029	0.4168	0.4198	0.4120	0.4162	0.4188	0.4179	0.4162	0.4190

Table 9
Shrinkage values of AAS and OPC mortars subjected to dry curing (%).

Mix no.	Binder	Admixture	3 days	7 days	14 days	28 days	60 days	90 days	120 days	150 days	180 days
1	OPC	–	0.0428	0.0763	0.0942	0.0984	0.0993	0.1221	0.1518	0.1723	0.1819
2	AAS, 4% Na, M_s = 0.75	–	0.1826	0.3288	0.3632	0.3716	0.3746	0.3725	0.4132	0.4242	0.4282
3	AAS, 6% Na, M_s = 0.75	–	0.5582	0.5935	0.6130	0.6193	0.6140	0.6307	0.6433	0.6725	0.6752
4	AAS, 4% Na, M_s = 1.00	–	0.3365	0.4605	0.4895	0.4930	0.4919	0.5068	0.5367	0.5668	0.5711
5	AAS, 6% Na, M_s = 1.00	–	0.5956	0.6263	0.6721	0.6874	0.6823	0.6942	0.7196	0.7449	0.7502
6	OPC	SSRe	0.0274	0.0514	0.0689	0.0798	0.0918	0.1154	0.1296	0.1689	0.1691
7	AAS, 4% Na, M_s = 0.75	SSRe	0.0642	0.2882	0.3026	0.3037	0.2991	0.3312	0.3600	0.3754	0.3783
8	AAS, 6% Na, M_s = 0.75	SSRe	0.2768	0.4856	0.4963	0.4833	0.4791	0.5114	0.5346	0.5461	0.5498
9	AAS, 4% Na, M_s = 1.00	SSRe	0.2837	0.4247	0.4186	0.4203	0.4286	0.4340	0.4537	0.4754	0.4839
10	AAS, 6% Na, M_s = 1.00	SSRe	0.4653	0.6068	0.6381	0.6440	0.6389	0.6435	0.6595	0.6921	0.7025
11	OPC	SHR	0.0253	0.0463	0.0505	0.0568	0.0677	0.0993	0.1140	0.1431	0.1503
12	AAS, 4% Na, M_s = 0.75	SHR	0.1470	0.2386	0.2407	0.2418	0.2572	0.2818	0.3049	0.3189	0.3306
13	AAS, 6% Na, M_s = 0.75	SHR	0.3572	0.4118	0.4334	0.4421	0.4463	0.4493	0.4524	0.4582	0.4798
14	AAS, 4% Na, M_s = 1.00	SHR	0.1989	0.3289	0.3316	0.3326	0.3495	0.3625	0.3904	0.4002	0.4157
15	AAS, 6% Na, M_s = 1.00	SHR	0.4304	0.4940	0.5177	0.5247	0.5425	0.5616	0.5911	0.6126	0.6183

use of SSRe in the mixtures slightly reduced the shrinkage. This result conforms to that of another study [13] in the literature. In addition to this, an effective decrease in shrinkage was achieved with the SHR admixture under the dry curing conditions. The beneficial influence of SHR on the shrinkage mainly arises from the decrease in the surface tension of the pore water and the change in the pore structure induced by the admixture. Shrinkage-reducing admixtures, in turn, tend to decrease the surface tension of the water in the concrete pores, thereby lowering the capillary tension within the pore structure and therefore decreasing shrinkage when the water evaporates [22,34]. Nevertheless, for all mixtures with and without chemical admixtures, the shrinkage values of AAS mortars were considerably higher than those of NPC mortar. So, more researches on the shrinkage should be carried out to reduce it to the level of NPC mortar by using different types and dosages of admixtures. On the other hand, in the NPC mortars, SRH reduced the shrinkage as expected. But SSRe did not have a remarkable influence on the shrinkage as Al-Saleh and Al-Zaid [44] reported.

Table 10
Shrinkage values of AAS and OPC mortars subjected to heat curing (%).

Mix no.	Binder	Admixture	3 days	7 days	14 days	28 days	60 days	90 days	120 days	150 days	180 days
1	OPC	–	0.0193	0.0565	0.0611	0.0561	0.0684	0.0849	0.1007	0.1246	0.1386
2	AAS, 4% Na, $M_s = 0.75$	–	0.0684	0.1505	0.1975	0.2428	0.2537	0.2842	0.3007	0.3219	0.3353
3	AAS, 6% Na, $M_s = 0.75$	–	0.1793	0.2879	0.3286	0.3544	0.4060	0.4360	0.4546	0.4762	0.4825
4	AAS, 4% Na, $M_s = 1.00$	–	0.1325	0.2744	0.3174	0.3409	0.3749	0.4100	0.4270	0.4354	0.4418
5	AAS, 6% Na, $M_s = 1.00$	–	0.1804	0.3800	0.4388	0.4709	0.5077	0.5354	0.5688	0.5793	0.5864
6	OPC	SSRe	0.0101	0.0309	0.0330	0.0488	0.0725	0.0932	0.1225	0.1353	0.1364
7	AAS, 4% Na, $M_s = 0.75$	SSRe	0.0621	0.1353	0.1504	0.1814	0.2265	0.2572	0.2760	0.2894	0.3012
8	AAS, 6% Na, $M_s = 0.75$	SSRe	0.0509	0.1396	0.1539	0.1898	0.2325	0.2660	0.2828	0.3051	0.3169
9	AAS, 4% Na, $M_s = 1.00$	SSRe	0.0674	0.2049	0.2288	0.2537	0.2807	0.3119	0.3295	0.3465	0.3512
10	AAS, 6% Na, $M_s = 1.00$	SSRe	0.0782	0.2291	0.2602	0.3319	0.3814	0.4040	0.4252	0.4276	0.4306
11	OPC	SHR	0.0016	0.0125	0.0289	0.0463	0.0592	0.0677	0.0884	0.1025	0.1061
12	AAS, 4% Na, $M_s = 0.75$	SHR	0.0326	0.1128	0.1377	0.1677	0.2242	0.2516	0.2677	0.2719	0.2803
13	AAS, 6% Na, $M_s = 0.75$	SHR	0.0330	0.1316	0.1537	0.1846	0.2323	0.2579	0.2738	0.2961	0.3124
14	AAS, 4% Na, $M_s = 1.00$	SHR	0.0228	0.1768	0.1902	0.2253	0.2775	0.3014	0.3221	0.3350	0.3393
15	AAS, 6% Na, $M_s = 1.00$	SHR	0.0537	0.1891	0.2102	0.2613	0.2991	0.3239	0.3432	0.3514	0.3595

4. Conclusions

The following conclusions can be drawn from this study:

1. SSRe admixture increased the flow rate of AAS mortars while SHR admixture had a little effect on the flow values of AAS mortars. On the other hand, SSRe and SHR admixtures generally did not have an impact on the setting times of AAS pastes, with the exception for the sodium silicate-activated slag paste with $M_s = 0.75$ and 4% Na.
2. SSRe and SHR chemical admixtures reduced both compressive and flexural strength of AAS mortars at the age of 2 days. However, these admixtures subsequently exhibited different effects. After 2 days, SSRe had no impact on the flexural and compressive strength values of AAS mortars, whereas SHR increased the flexural strength of AAS, without making any change in the compressive strength behavior.
3. The carbonation depths of AAS mortars were higher than those of NPC mortar. The use of SSRe admixture had no impact on AAS mortars after carbonation whereas SHR admixture somewhat decreased the carbonation depths of AAS mortars.
4. Even though SSRe and especially SHR chemical admixtures reduced the shrinkage of AAS mortars, the shrinkage values of AAS mortars were higher than those of NPC mortars. Therefore, more research should be carried out on shrinkage by using several admixtures with different dosages.
5. When the findings of the tests performed within the scope of this study were considered, it was observed that curing conditions had a significant effect on the properties in the hardened state of AAS mortars. Heat curing considerably accelerated the early strength development and reduced the high shrinkage of AAS mortars. However, the best results were obtained from the AAS mortars cured at 23 ± 2 °C and 95% relative humidity throughout the test time since heat cured AAS mortars were subjected to dry curing conditions instead of moist curing after heat treatment of 65 °C for 5 h. On the other hand, dry curing condition with low relative humidity showed the worst curing performance on the mechanical properties of AAS mortars.

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