

Paper's No. BUE-FISC – 107

Properties of Sodium Alkali-Activated Ground Granulated Blast-Furnace slag(GGBS) Mortar

**Jin-Kyu Song, Keun-Hyeok Yang, Geon-Woo Kim,
Chan-Taek Lee, Byeong-Jo Kim, Jae-Hyeok Lee**

Dept. of Architecture Engineering, Chonnam National University, GwangJu,
South Korea, tel +82-62-530-1640 c.p 018-624-8700

Dept. of Architecture Engineering, Kyonggi University, Suwon, Kyonggi,
South Korea, tel +83-31-249-9843 c.p 010-4693-2839

Dept. of Architecture Engineering, Chonnam National University, GwangJu,
South Korea, tel +82-62-530-0260 c.p 010-8868-0412

Dept. of Architecture Engineering, Chonnam National University, GwangJu,
South Korea, tel +82-62-530-0260 c.p 010-2016-2352

Dept. of Architecture Engineering, Chonnam National University, GwangJu,
South Korea, tel +82-62-530-0260 c.p 010-9222-8982

Dept. of Architecture Engineering, Chonnam National University, GwangJu,
South Korea, tel +82-62-530-0260 c.p 010-4107-2041

jgsong@chonnam.ac.kr, yangkh@kgu.ac.kr, gwkim7210@lycos.co.kr,
tac3670@naver.com, www.jpnd@hanmail.net, leonhart2041@hanmail.net

ABSTRACT:

It has been increasing the demand of eco-friendly materials which have low environmental effects and consume low energy. Such demands have led the study of alkali-activated ground granulated blast furnace slag(AAG) mortar and concrete to be developing. And studies about non-cement alkali activated concrete using sodium silicate as an alkali activator are proceeding in field of concrete. Generally, if activator concentration is increased, concrete is hardened rapidly and increased production cost.

Purposes of this study are to compile data about effect for hydration of ground granulated blast furnace slag with activator concentration and to propose optimum activator combination. Strength test was conducted to develop economical efficiency, to apply in situ. In this study it was verified that 13 AAG mortar had been developed the compressive strength. 7 AAG mortar were set. Among them, sodium hydroxide and sodium silicate were shown high strength. So they were chosen to evaluate the compressive strength and the flow value of each batch. And sodium hydroxide or sodium silicate were mixed with another activators for improving performance. Compressive strength of AAG mortar with sodium hydroxide and sodium sulphate of sodium carbonate was increased as compared with AAG mortar

using only sodium hydroxide. But setting time was decreased. AAG mortar with sodium silicate and sodium sulphate was improved performance of compressive strength and setting time.

Conference Topic: The Earth/Desert/Green and Sustainable Buildings
Keywords: GGBS, Sodium Silicate, Alkali Activator

1. Introduction

Climate change and global warming caused by greenhouse gases are threatening seriously all over the world. Reduction of greenhouse gases is directly associated economic problem and environmental problem. Thus, the concrete industry is aware of this problem and is trying to reduce the quantity of cement. There have been researched on alkali activated concrete, which uses ground granulated blast furnace slag (GGBS) or fly ash (FA) as a substitute of cement. The plan “Vision 2030 : A vision for the U.S Concrete Industry” was proposed in U.S Concrete Industry. As a result, there has been a recent gradual growth in the application of concrete and mortar which are partially replaced with by-product materials, such as fly ash (FA) and ground granulated blast furnace slag (GGBS). The influence of dry curing conditions on ground granulated blast furnace slag (GGBS) concrete was marked as the replacement ratio of GGBS increased.

Another active effort is development of cementless paste synthesized from FA or GGBS as a source material and an alkaline inorganic material as an activator. There has been a significant increase in the number of studies into GGBS or FA-based alkali activated (AA) binders. Although, the mechanism of the alkali activation process is still a matter of controversy, it is commonly known that calcined materials with abundant silicon oxide (SiO_2) and aluminum oxide (Al_2O_3) are more suitable as source materials, and alkali hydroxide (MOH), non-silicate salts of weak acids (M_2CO_3 , M_2S , MF), or silicate salts of the $\text{M}_2\text{O}\cdot(n)\text{SiO}_2$ type can be the most effective activator, where M indicates an alkali metal ion such as Na, K or Li (Bin, 1988). A sodium hydroxide solution or sodium silicate solution is generally said to be a good application as an activator (Tang, 1988). In addition, a paste activated by alkali solutions experiences very short set times and subsequent shrinkage. This would cause rapid workability loss of the mortar or concrete with time, which would constitute an obstacle for practical application.

On the other hand, Yang and Song et al. analyzed properties of mortar and concrete with solid sodium silicate using water-binder ratio, fineness of source material, fine aggregate ratio as a variable. And they were proposed Alkali Quality Coefficient (Q_A) which combining the concentration of activator and main compositions in source materials (Yang, 2008). Mortar and concrete using sodium silicate as an activator were developed high strength but showed initial fluidity loss and cracking.

In the present study, properties of mortar using activator combination more than two were verified to solve weak point. 13 types of alkali activators were chosen from precedent study and literature review and conducted compressive strength. As a result, 7 activators were developed strength. Among them, sodium hydroxide and sodium silicate were shown high strength. So they were chosen to evaluate the compressive strength and the flow value of each content. And sodium hydroxide or sodium silicate were mixed with another activators for improving performance.

2. Experimental

2.1 Material

2.1.1 Ground Granulated Blast Furnace Slag

Ground granulated blast furnace slag (GGBS) as a source material is produced in a domestic G company which satisfied KS F CODE 2563 (Ground granulated blast furnace slag for use in concrete). The specific gravity and specific surface area of GGBS and basicity are 2.93 and 4204 (cm²/g) and 1.81, respectively. The chemical compositions of ground granulated blast furnace slag are given in Table. 1.

Table 1. Chemical Composition of GGBS

Source material	Chemical Composition							
GGBS	MgO	Al ₂ O ₃	SiO ₂	SO ₃	K ₂ O	CaO	TiO ₂	MnO
	43.8	13.8	34.7	0.95	0.48	44.6	0.74	0.24

2.1.2 Alkali activators

Glukovsky et al. classified alkali activators into six groups according to their chemical compositions (Glukovsky, 1980). 6 groups are caustic alkalis (MOH), non-silicate weak acid salts (M₂CO₃, M₂SO₃, M₃PO₄, MF etc), silicates (M₂O·nSiO₂), aluminates (M₂O·nAl₂O₃), aluminosilicates (M₂O·Al₂O₃·(2-6)SiO₂), non-silicate strong acid salts (M₂SO₄), where M indicates an alkali metal ion such as Na, K, Li. Among them, 4 alkali activator groups were used in this study. Sodium hydroxide (NaOH), calcium hydroxide (Ca(OH)₂), barium hydroxide (Ba(OH)₂), potassium hydroxide (KOH) of caustic alkali group and sodium sulfate (Na₂SO₃), sodium carbonate (Na₂CO₃), calcium carbonate (Ca₂CO₃), sodium fluoride (NaF), potassium carbonate (K₂CO₃) of non-silicate weak acid salt group are used. Sodium silicate (Na₂SiO₃), potassium silicate (K₂SiO₃) of silicate group and calcium sulfate (Ca₂SO₄), potassium sulfate (K₂SO₄) of non-silicate strong acid salt group were used in this study. Tests were conducted with total 13 alkali activators.

Table 2. Classification of Alkali Activators

Caustic alkali	Non-silicate weak acid salt	Silicates	Non-silicate strong acid salt
NaOH	Na ₂ SO ₃		
Ca(OH) ₂	Na ₂ CO ₃	Na ₂ SiO ₃	Ca ₂ SO ₄
Ba(OH) ₂	Ca ₂ CO ₃	K ₂ SiO ₃	K ₂ SO ₄
KOH	NaF		
	K ₂ CO ₃		

2.1.3 Aggregates

Locally available sand with a maximum size of 5mm (2in.) was used as the fine aggregate in saturated surface-dry condition. The specific gravity and grading of the sand were 2.54 and 2.97, respectively. And physical properties of sand were shown in Table 3.

Table 3. Physical Properties of Aggregate

Experimental item		Fine aggregate	
		Standard	Test result
Particle size	Fineness modulus (FM)	2.3 ~ 3.1	2.89
Physical properties	Unit weight (kg/l)	-	1.567
	Density (g/cm ³)	Oven dry density	Above 2.50
		Surface dry density	-
	Absorptivity (%)	Below 3.0	1.45
	Soundness (%)	Below 10	4.52
Harmful substance	0.08mm sieve passage mass (%)	Abrasion surface Below 3 Other surface Below 5	1.62
	Clay lump content (%)	Below 1.0	0.46
	Inorganic impurity	More lighter than standard solution	Lightness
	Chloride (%)	Below 0.4	0.003

2.2 Mix design

The source material, activator and fine aggregate were dry mixed in a mixer pan for 30 seconds, and water was then added and mixed for another 30 seconds. After initial flow testing, each mortar mixture was poured into 50 × 50 × 50 mm (1.97 × 1.97 × 1.97 in.) steel molds to measure the compressive strength. Immediately after casting, all specimens were cured at room temperature with a constant relative humidity of 70 ± 5% until tested at a specified age. All steel molds for slag-based AA mortars were removed at an age of 1 day. The test procedure of initial flow, setting time and compressive strength was conducted according to KS (Korea Standard) L 5105, KS F 2436 and KS F 2426. Initial flow of mortar was measured using a cone-shaped mold having a height of 50 mm (1.97in.), a bottom diameter of 100 mm (3.94in.), and a top diameter of 70 mm (2.76in.). immediately after filling the mold that was mounted rigidly on the flow table, the cone was slowly lifted and then the flow table was jolted 25 times. The mortar consequently spread out and the maximum spread parallel to the two edges of the table was recorded. The compressive strength development of the mortar was monitored by testing three mortar cube at 3, 7, 28 days using a 200kN capacity universal testing machine. Experimental conditions are shown in table 4.

Table 4. Experimental Conditions

Source material	W/B	Aggregate ratio	Curing temperature	Curing humidity
GGBS	50	1:2.45	23±2°C	70±5 %

3. Mix proportions and test results

3.1 Properties of strength development with alkali activator

Table 5. Test Results of AAG Mortar

Alkali activator		Strength with time		
		3Days	7Days	28Days
Caustic alkali	NaOH	10.40	13.70	16.20
	Ca(OH) ₂	5.54	11.49	14.32
	Ba(OH) ₂	4.00	4.50	5.65
	KOH	11.49	14.49	20.07
Non-silicate weak acid salt	Na ₂ SO ₃	-	-	-
	Na ₂ CO ₃	-	-	-
	CaCO ₃	-	-	-
	K ₂ CO ₃	-	-	-
	NaF	1.02	3.07	10.68
Silicates	Na ₂ SiO ₃	11.40	14.00	17.84
	K ₂ SiO ₃	-	-	-
Non-silicate strong acid salt	Ca ₂ SO ₄	-	0.51	1.05
	K ₂ SO ₄	-	-	-

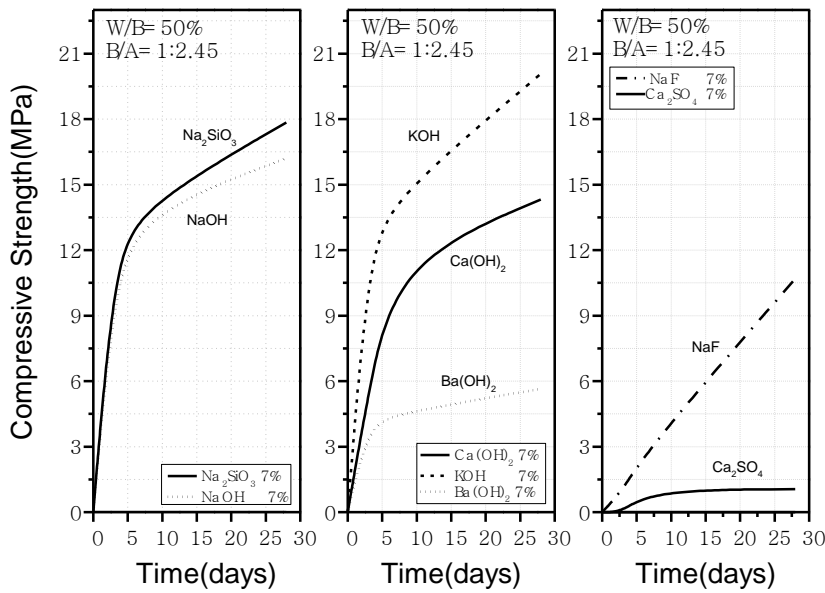


Figure 1. Compressive Strength Development

13 alkali activators were used. Alkali activators were chosen which reacted in variable curing condition from precedent study and literature review. Unlike present study, alkali activated mortars were cured under high temperature condition and steam curing condition in literature. After alkali activators were added to binder 7% by mass, compressive strength was measured as shown in Table 5 and Fig 1. All alkali activators of caustic alkali group were

developed strength. But sodium fluoride (NaF) of non-silicate weak acid salt group, sodium silicate of silicates group and calcium sulfate of non-silicate acid salt group were developed.

All the molds for alkali activated mortars were removed after 1 day. Then compressive strength test was measured at 3days but calcium sulfate was measured at 7days. Compressive strength of Calcium sulfate activated mortar was developed lower than other mortars. Six alkali activated mortars except calcium sulfate were developed strength more than 10 MPa. Among them, sodium fluoride activated mortar was increased linearly with time. Potassium hydroxide based mortar was developed the highest strength 20.06 MPa at 28 days. Caustic alkali group which developed strength contains hydroxyl ion (OH^-). Sodium silicate and sodium fluoride reacted with water produce hydroxyl ion (OH^-).

In result, hydrogen exponent (pH) of alkali activated mortar was present highly. As Alkali activators to induce reaction, alkali type is more effective than sulfate type. Alkali activators except sodium fluoride are strong acid salt what of pH is more than 12. Sodium fluoride is ionized into Na^+ and F^- . And F ion absorbs H ion, then is became acid. Although hydrogen exponent (pH) change of Sodium fluoride based binder was decreased with HF concentration, binder was hardened. On the other hand, property of initial strength development was somewhat low as 1 MPa at 3 days. From precedent study, initial hydration of slag is accelerated with hydrogen exponent (pH). Although hydrogen exponent (pH) of sodium fluoride activated mortar is about 10, sodium fluoride could accelerate ground granulated blast furnace slag (GGBS). As a result, hydration of alkali activated mortar is influenced by properties of anion group than initial hydrogen exponent (pH). And Jennings concluded that hydrogen exponent (pH) is influenced by silicon (Si), calcium (Ca), aluminum (Al) solution. Increasing hydrogen exponent (pH), concentration of silicon (Si), aluminum (Al) is increased and magnesium (Mg) is decreased. So, it is need to consider effect of alkali activator to ground granulated blast furnace slag (GGBS).

Wang et al. proposed that Na_2O to binder ratio is 3.3 ~ 5.0 % (by mass). Na_2O to binder ratio of binder added sodium hydroxide and sodium silicate 7% is 5.0 %, 3.3 % (by mass), respectively(Wang 2000). Result from flow test, calcium and potassium type activators except potassium hydroxide were shown loss of fluidity.

3.2 Properties of sodium hydroxide (NaOH) and sodium silicate (Na_2SiO_3)

Result from previous chapter, sodium hydroxide, potassium hydroxide, barium hydroxide, calcium hydroxide of caustic alkali group were developed strength, sodium fluoride of non-silicate weak acid salt group, sodium silicate of silicate group were also developed strength. Alkali activators which were developed high strength than others were potassium hydroxide, sodium silicate, sodium hydroxide order. Among them potassium hydroxide activated mortar was developed the highest strength. But production cost of potassium hydroxide based mortar is more expensive than others. So sodium hydroxide and sodium silicate were used as main activator. It is needs to analyze properties of sodium hydroxide - and sodium silicate based mortar. Tests of sodium hydroxide and sodium silicate mortars were conducted to analyze properties of alkali activator with 2 ~ 6% (by mass). At this time, Na_2O to binder ratio of sodium hydroxide and sodium silicate were about 1.5 ~ 4.5 %, 1.0 ~ 3.0 % (by mass), respectively. Compressive strength, flow, setting time of sodium hydroxide and sodium silicate based mortars are shown in table 6, fig 2, fig 3.

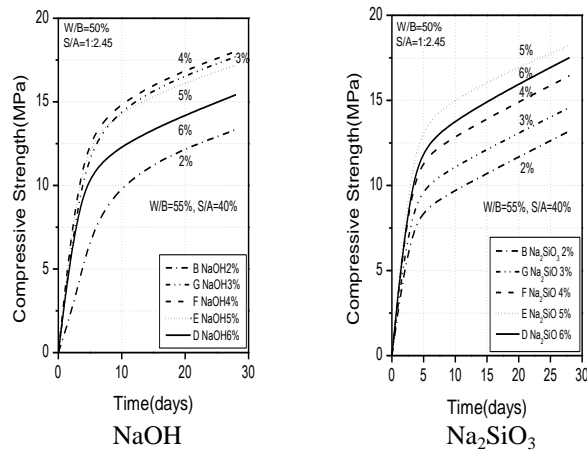


Figure 2. Compressive strength of NaOH and Na₂SiO₃ based mortar

Compressive strength of mortar was not increased linearly with sodium hydroxide concentration and was developed the highest strength with 4 % activator concentration. More than 4% activator concentration, compressive strength of mortar was decreased with activator concentration. Tang and Shi found that, regardless of activators, there is a critical activator dosage below which activators do not show activation effect on slag. Above the critical activator dosage, the strength of activated slag increases with activator dosage, reaches a maximum value, and then decreases with activator dosage (Tang, 1988). Bin proposed that compressive and flexural strength of activated slag cements increases almost linearly with activator dosage from 2 % to 8% (Na₂O) at ages from 3 to 180 days (Bin, 1988). On the other hand, compressive strength of sodium silicate activated mortar was developed the highest strength with 5 % activator concentration. And more than 5 %, strength was not increased.

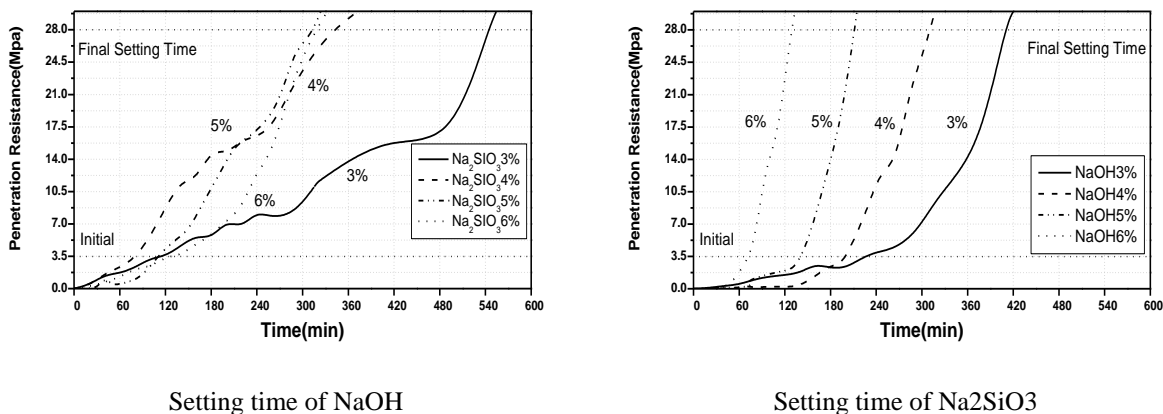


Figure 3. Setting time of NaOH and Na₂SiO₃

Setting time of sodium hydroxide and sodium silicate based mortar are shown in Fig 3. Setting time of sodium hydroxide based mortar was decreased with activator concentration. This result corresponds partially with proposal of Wu. He measured the setting times of slag cements with dosage of 2, 4, 6, 8, 10 % by Na₂O. When the dosage was 2 and 4 %, it did not show any obvious effect on both the initial and final setting times.

Table 6. NaOH and Na₂SiO₃ Mix Proportions and Test Result

Alkali activated mortar mixing detail					Flow(cm)			Compressive strength(MPa)		
Activator	Dosage(%)	SM	B/A	W/B	Initial	30min	60min	3Days	7Days	28Days
NaOH	2	GGBS	1:2.45	50	14.65	13.2	12.1	3.57	10.34	13.34
	3				16.15	15.3	14.6	9.38	14.72	17.70
	4				15.25	13.85	12.6	10.70	14.98	18.03
	5				17.25	15.65	15.15	10.62	14.40	17.20
	6				17.55	16.5	15.3	9.13	12.22	15.43
Na ₂ SiO ₃	2	GGBS	1:2.45	50	14.2	12.55	11.75	7.99	9.19	13.20
	3				14.9	13.5	11.2	9.17	10.63	14.59
	4				14.55	12.25	11.45	10.83	12.39	16.45
	5				15.7	15.65	12.95	12.15	14.84	18.23
	6				17.0	15.3	14.25	11.03	13.42	17.51

When the dosage was increased from 4 % to 6 %, it slightly decreased the final setting time from 90 minutes to 76 minutes, but significantly decreased the final setting time from 425 minutes to 245 minutes. But setting time of sodium silicate based mortar was not affected with activator concentration.

3.3 Activator combination to select assistant alkali activator

Sodium hydroxide and sodium silicate using as main activator were combined with other activators from 2 % to 8% (by mass). Mixing conditions of mortar are shown in table 7. Calcium sulfate, calcium carbonate, sodium sulfate, sodium carbonate, potassium silicate activated mortar were not developed strength, but when these activators were combined sodium silicate and sodium hydroxide, compressive strength was developed. Especially sodium hydroxide, when sodium hydroxide concentration 2 % was combined with sodium sulfate or sodium carbonate concentration 2 %, strength was developed higher than mortar using single activator. Mortar combined Sodium silicate concentration 2 % and sodium sulfate, sodium silicate 4 % and sodium sulfate, sodium silicate 4 % and sodium carbonate were developed higher strength than mortar using single activator. Li and Sun [4] noticed that the combination of sodium hydroxide and sodium carbonate gave a higher strength than those cement mortars activated either by sodium hydroxide or sodium carbonate alone. Krivenko noticed that the combination of any two types of anion or anion group gave higher strength than either one of them(Krivenko, 1992). But compressive strength of mortars was not increased in other combination.

3.4 Properties of activator combination

3.4.1 Properties of compressive strength development with activator combination

Combinations of sodium hydroxide and sodium carbonate, sodium silicate and sodium sulfate, sodium hydroxide and sodium sulfate were selected based test results. Properties of mortars were analyzed with alkali activator concentration from 2 % to 6 %.

Properties of compressive strength development with time are shown in Fig 4, Fig 5, Fig 6. Strength of sodium hydroxide and carbonate combination mortar was developed higher from 30 % to 100 % than either one of them. Among them, mortars combined of 3 % sodium hydroxide and 5 % sodium carbonate, 4 % sodium hydroxide and 5, 6 % sodium carbonate, 5 % sodium hydroxide and 6 % sodium carbonate were developed strength higher than 30 MPa. In case of 2, 3 % sodium hydroxide, compressive strength of mortars was increased to 5 % sodium carbonate. In case of 4, 5 % sodium hydroxide, strength of mortars was increased with

sodium carbonate concentration. In case of 6 % sodium hydroxide, sodium carbonated concentration was not affected compressive strength of mortars. Compressive strength of Sodium silicate and sodium sulfate combination mortars was increased slightly. But compressive strength of mortars was increased generally 20 % than sodium hydroxide activated mortar. These results are caused narrow range from 1.5 to 3 % of Na₂O concentration.

Sodium hydroxide and sodium sulfate combination mortars were developed lower strength in certain combinations than sodium hydroxide activated mortar. In case of 3, 4 % sodium hydroxide concentration, strength was developed lower strength than 2 % sodium hydroxide activated mortar. In case of 5, 6 % sodium hydroxide concentration, strength of mortars was developed about 30 MPa with combined 4, 5 % sodium sulfate. But mortar was set rapidly as sodium hydroxide concentration is increased. Compressive strength at 28 days of combination mortar is shown in Fig 7.

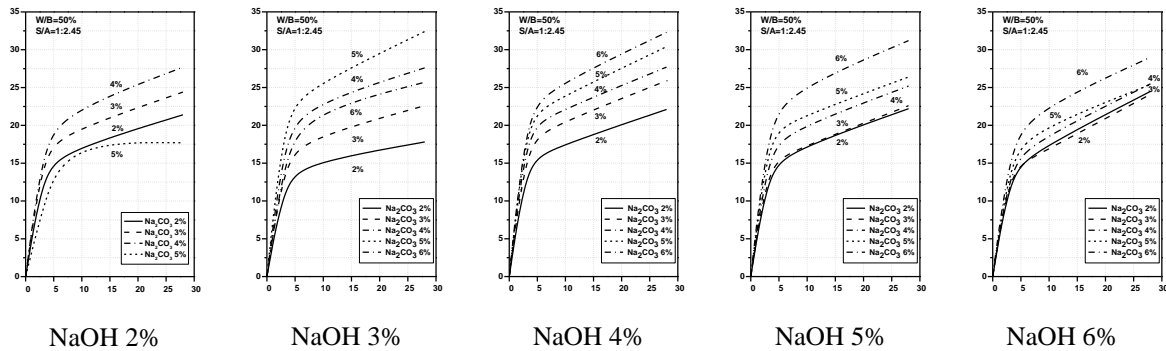


Figure 4. Compressive strength of NaOH + Na₂CO₃ combination

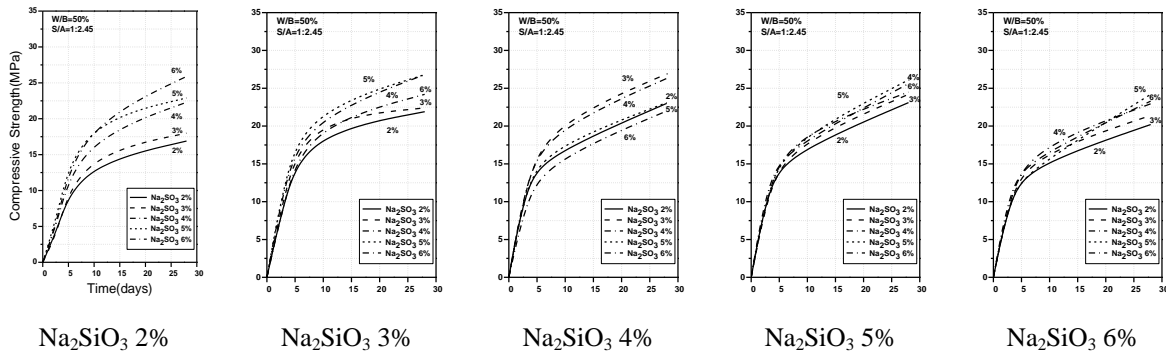


Figure 5. Compressive strength of Na₂SiO₃ + Na₂SO₃ combination

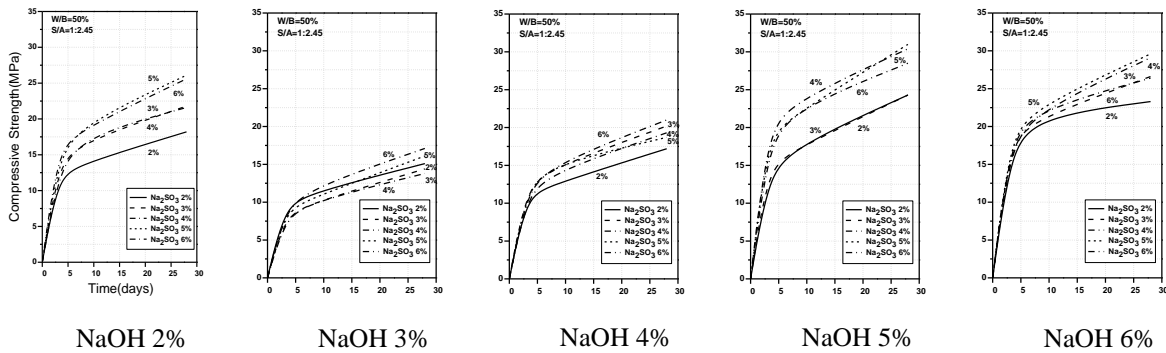


Figure 6. Compressive strength of NaOH + Na₂SO₃ combination

Table 7. Mix-proportion and Test results

Main Activator	Proportion of activator			Compressive strength(MPa)		
	Dosage(%)	Assistant Activator	Dosage(%)	3Days	7Days	28Days
NaOH	1	Na ₂ CO ₃	5	-	-	-
NaOH	2	Na ₂ CO ₃	2	12.61	16.61	21.41
NaOH	2	Na ₂ CO ₃	2	11.68	13.57	18.24
NaOH	2	Na ₂ SiO ₃	2	6.96	7.60	13.34
NaOH	2	K ₂ SO ₃	2	9.94	12.83	16.43
NaOH	2	Ca(OH) ₂	5	6.66	8.14	9.02
NaOH	2	Ca ₂ SO ₄	5	8.57	13.73	17.40
NaOH	2	Ca ₂ CO ₃	5	9.23	11.45	13.12
NaOH	4	Ca(OH) ₂	8	6.29	8.47	11.15
NaOH	4	Na ₂ CO ₃	8	21.62	27.04	30.14
Na ₂ SiO ₃	2	Na ₂ SO ₃	2	4.89	13.37	16.89
Na ₂ SiO ₃	2	Ca ₂ SO ₄	5	1.23	11.09	11.00
Na ₂ SiO ₃	2	Ca ₂ CO ₃	5	2.11	7.54	7.05
Na ₂ SiO ₃	4	Na ₂ CO ₃	2	11.01	14.56	19.62
Na ₂ SiO ₃	4	Na ₂ SO ₃	2	12.26	16.18	20.42
Na ₂ SiO ₃	4	Ca(OH) ₂	8	8.13	10.61	14.24

3.4.2 Properties of setting time with activator combination

Properties of setting time with combinations of sodium hydroxide and sodium carbonate, sodium silicate and sodium sulfate, sodium hydroxide and sodium sulfate are shown in Fig 7. Setting time of sodium hydroxide and sodium carbonate combination mortars was influenced activator concentration. Setting time of 3 % sodium hydroxide activated mortar was compared with sodium hydroxide 3 % and sodium carbonate 4, 5 % combination mortar. And setting time of 6 % sodium hydroxide mortar activated mortar was compared with 6 % sodium hydroxide and 4, 6 % sodium carbonate combination mortar. Setting time of mortar was increased sharply with sodium carbonate concentration. Decrease of setting time was occurred in case of 3 % sodium hydroxide, 6 % sodium hydroxide.

Setting time of sodium silicate and sodium sulfate combination mortars was not shown with sodium carbonate concentration in 3 % sodium silicate activated mortar. In case of 6 % sodium silicate, setting time of mortars was retarded with sodium sulfate concentration.

In case of sodium hydroxide and sodium sulfate combination mortar, setting time of mortars was retarded with sodium sulfate concentration. Setting time of 5 % sodium hydroxide and 4, 6 % sodium sulfate combination mortar was shown in Fig 8.

Setting time test of combination using sodium hydroxide and sodium silicate as main activators, sodium carbonate and sodium sulfate as assistant activators was conducted. As a result, setting time of mortar was retarded. Sodium sulfate is effective retarder in sodium silicate activated mortar, but sodium sulfate is not effective retarder in sodium hydroxide activated mortar.

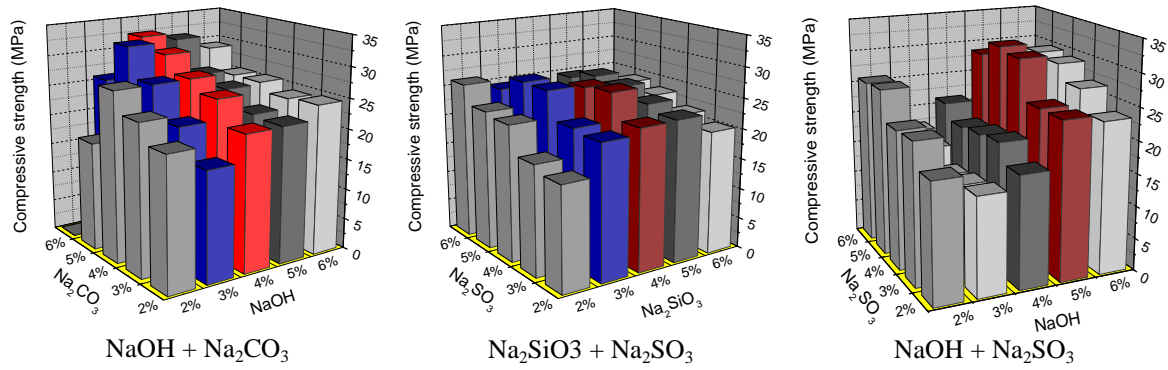


Figure 7. Compressive Strength at 28 Days of 3 Combinations

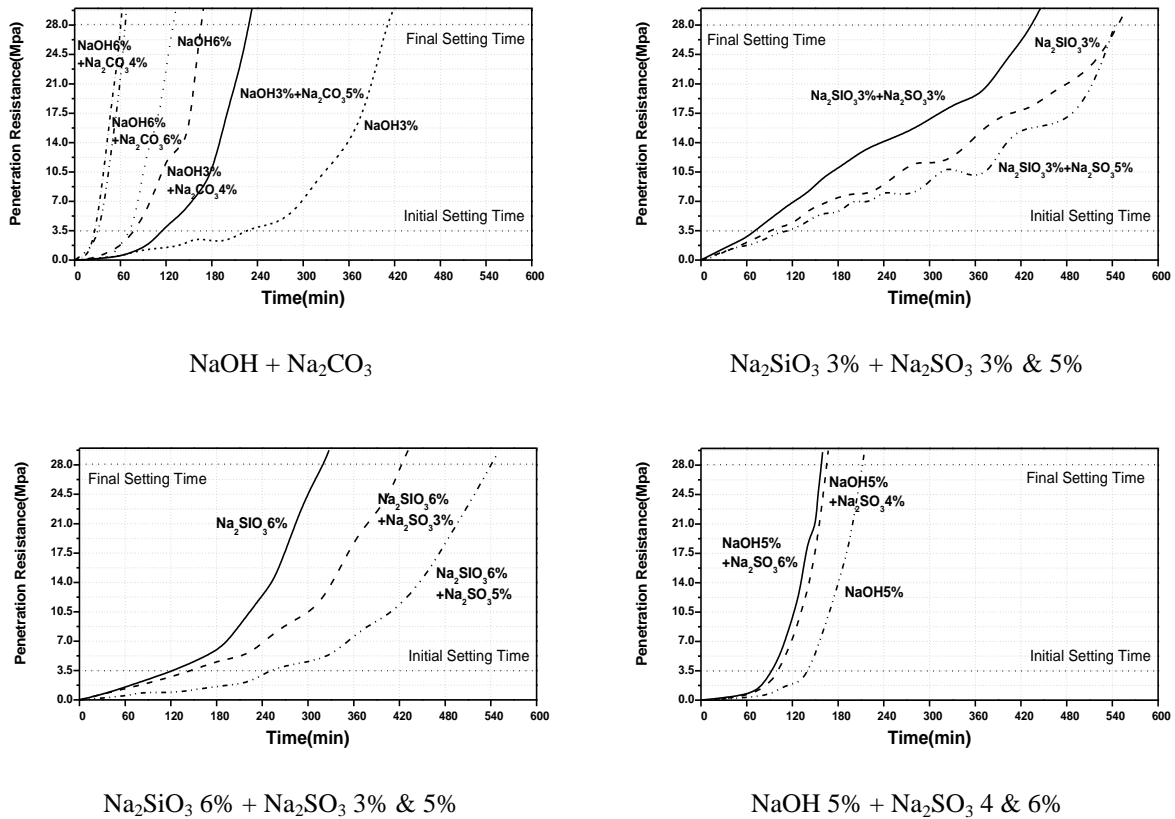


Figure 8. Setting time of combined mortars

4. Conclusion

In this study, compressive strength and setting time test were conducted using sodium hydroxide and sodium silicate as main activator, assistant activators to analyze properties of non-cement mortar with alkali activator combination.

13 types of alkali activators were used in present study. From the test results, alkali activated mortars developed strength were contained hydroxyl (OH⁻) ion and created hydroxyl (OH) ion as production of hydration.

Compressive strength of sodium hydroxide activated mortars was developed the highest strength with 4 % activator concentration. Setting time of sodium hydroxide activated mortar was decreased with activator concentration.

Compressive strength of sodium silicate activated mortars was developed with certain concentration. Setting time of sodium silicate activated mortar was not affected with concentration.

To select optimum combination, mortars added sodium hydroxide and sodium silicate as main activator were combined with assistant activators. Compressive strength of sodium hydroxide and sodium carbonate, sodium sulfate combination mortars was increased. And compressive strength of sodium silicate and sodium sulfate combination mortars was also increased than sodium silicate activated mortars.

As a result, three combinations of sodium hydroxide and sodium carbonate, sodium silicate and sodium sulfate, sodium hydroxide and sodium sulfate were selected. Compressive strength of mortars was increased with combination from 2 % to 6 % concentration. Especially, compressive strength of sodium hydroxide and carbonate combination mortars was largely increased. And optimum mix proportion is combination of 3 % sodium hydroxide and 5 % sodium carbonate.

Sodium carbonate and sodium sulfate are effective retarder in sodium hydroxide activated mortar. But setting time of mortar was retarded with sodium carbonate concentration but was not shown retardation with sodium sulfate concentration. In 6 % sodium silicate activated mortar, setting time was retarded with sodium sulfate concentration.

The activator combination is to reduce activator concentration, to improve performance, to increase cost problem and to retard setting time.

Acknowledgements

This work was supported by the Grant of the Korean Ministry of Education, Science and Technology (The Regional Core Research Program/Biohousing Research Institute) and The Biohousing Research Center.

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