*Article*

# A Study on the Sulfate Resistance of Alkali Activated FA Based Geopolymer and GGBFS Blended Mortar with Various Sulfate Types

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**Abstract:** In this study, the changes in mass, compressive strength and length were analyzed to investigate sulfate resistance according to ground granulated blast furnace slag (GGBFS) blending ratio and type of sulfate solution. All alkali activated mortars showed excellent sulfate resistance when immersed in sodium sulfate ($Na_2SO_4$) solution. However, when immersed in magnesium sulfate ($MgSO_4$) solution, different sulfate resistance results were obtained depending on the presence of GGBFS. Alkali activated GGBFS blended mortars showed a tendency to increase mass, increase length and decrease compressive strength when immersed in magnesium sulfate solution, but the alkali activated FA mortars did not show any significant difference depending on the types of sulfate solution. The deterioration of alkali activated GGBFS blended mortars in the immersion of magnesium sulfate solution was confirmed by the decomposition of C-S-H which is the reaction product by magnesium ion and the formation of gypsum ($CaSO_4 \cdot 2H_2O$) and brucite ($Mg(OH)_2$).

**Keywords:** alkali activated materials; fly ash; ground granulated blast furnace slag; sulfate resistance; cation accompanying sulfate

## 1. Introduction

As climate change has become a social issue since the 2000s, there is a growing need to develop new materials that can replace ordinary Portland cement (OPC) in order to reduce the amount of $CO_2$ generated during the cement manufacturing process. For this reason, studies on alkali activated materials (AAMs) such as ground granulated blast furnace slag (GGBFS), fly ash (FA) and metakaolin have been actively conducted [1-6]. In general, AAMs are divided into two types depending on the reaction products. First, a binder using calcium rich materials such as GGBFS is activated by an alkaline solution to produce a calcium silicate hydrate (C-S-H) similar to the OPC hydrate. Second is geopolymer, which is low in calcium and high in silica and alumina contents, such as metakaolin and FA, and mainly produces amorphous alkali aluminosilicates gel [7, 8]. Caustic alkalis (MOH), non-silicate ($M_2CO_3$, $M_3PO_4$, $M_2SO_4$ etc.) and silicates ($M_2O \cdot nSiO_2$) are used as activators. Of all these activators, NaOH, $Na_2CO_3$, $Na_2O{\cdot}nSiO_2$ and $Na_2SO_4$ are the most widely available chemicals. Some potassium compounds have been used in laboratory studies, but their potential applications will be very limited due to their costs [9]

Generally, the deterioration mechanism by the sulfate attack of concrete is represented by the following two explanations. First, as shown in Equation (1) and (2), $Ca(OH)_2$ in concrete reacts with sulfate ions ($SO_4^{2-}$) to form gypsum ($CaSO_4 \cdot 2H_2O$). Gypsum formation as a result of cation-exchange reactions is capable of causing expansion. Second, the reaction of gypsum with calcium aluminate hydrate as in Equation (3) causes the degradation of properties such as expansion and delamination due to the formation of ettringite [10, 11]

$$Na_2SO_4 + Ca(OH)_2 + 2H_2O \rightarrow CaSO_4 \cdot 2H_2O + 2NaOH \quad (1)$$

$$MgSO_4 + Ca(OH)_2 + 2H_2O \rightarrow CaSO_4 \cdot 2H_2O + Mg(OH)_2 \quad (2)$$

$$3CaO \cdot Al_2O_3 \cdot 12H_2O + 3(CaSO_4 \cdot 2H_2O) + 14H_2O$$

$$\rightarrow 3CaO \cdot Al_2O_3 \cdot 3CaSO_4 \cdot 32H_2O \quad (3)$$

Depending on the cation type associated with the sulfate solution (i.e., $Na^+$, $K^+$, or $Mg^{2+}$), both calcium hydroxide and C-S-H present in the hydrated Portland cement paste may be converted to gypsum by sulfate attack. In the case of magnesium sulfate attack, the conversion of calcium hydroxide to gypsum is accompanied by the simultaneous formation of magnesium hydroxide, which is insoluble and reduces the alkalinity of the system [12]. The AAMs concrete performed better than OPC in sodium sulfate ($Na_2SO_4$) solution, and similarly to OPC in magnesium sulfate ($MgSO_4$) solution [13]. In general AAMs are reported perform equivalently to or better than OPC, but the performance of the AAMs depends strongly on the chemistry of the source material (GGBFS, FA or others), on the type of the activator and on the composition and concentration of the sulfate solutions used for testing [9].

In this study, the sulfate resistance of alkali activated FA based geopolymer and GGBFS blended mortars was evaluated. Specifically, the sulfate resistance was evaluated by changing the mass, compressive strength and length of alkali activated mortars according to the types and concentrations of sulfate solution.

## 2. Experiment

### *2.1. Materials*

FA and GGBFS were used as source materials in alkali activation, and the chemical compositions and physical properties of FA and GGBFS are listed in Table 1. FA is classified as class F fly ash according to ASTM C 618 since $SiO_2 + Al_2O_3 + Fe_2O_3$ is 85.1% and CaO is 3.8%. GGBFS shows 43.0% of CaO, 34.3% of $SiO_2$ and 14.2% of $Al_2O_3$. The Blaine surface area is 381 $m^2/kg$ for FA and 428 $m^2/kg$ for GGBFS, and the density is 2,210 $kg/m^3$ for FA and 2,890 $kg/m^3$ for GGBFS, respectively. The fine aggregate is used in accordance with ISO 679. For the activation of FA, sodium silicate solution ($SiO_2$ 28.2%, $Na_2O$ 9.3%, $H_2O$ 62.55%), and pure sodium hydroxide are usually adopted. Sulfate solutions were prepared using magnesium sulfate ($MgSO_4$) and sodium sulfate ($Na_2SO_4$) reagents.

The X-ray diffraction pattern of the FA and GGBFS is shown in Figure 1. The major crystalline components found in FA are quartz and mullite. In case of GGBFS, it has an amorphous phase of 95% or more because it is precipitated in a rapid cooling while being completely melted in the steel making process. Therefore, only the Anhydrite ($CaSO_4$) added in the blast furnace slag grinding process is confirmed as the crystalline component. Both FA and GGBFS show the hump as an amorphous phase characteristic in the X-ray diffraction patterns and an amorphous hump was located between 15 degree and 35 degree 2θ.

**Table 1.** Chemical compositions and physical properties of alkali activated materials (FA and GGBFS)

| | **Chemical compositions (mass %)** | | | | | | | | | **Physical properties** | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | **$SiO_2$** | **$Al_2O_3$** | **$Fe_2O_3$** | **CaO** | **MgO** | **$K_2O$** | **$Na_2O$** | **$SO_3$** | **LOI** | **Density ($kg/m^3$)** | **Blaine ($m^2/kg$)** |
| **FA** | 58.9 | 20.9 | 5.30 | 3.80 | 1.31 | 0.74 | 1.69 | 0.50 | 4.87 | 2,210 | 381 |
| **GGBFS** | 34.3 | 14.2 | 0.47 | 43.0 | 2.71 | 0.50 | 0.20 | 3.64 | 0.01 | 2,890 | 428 |

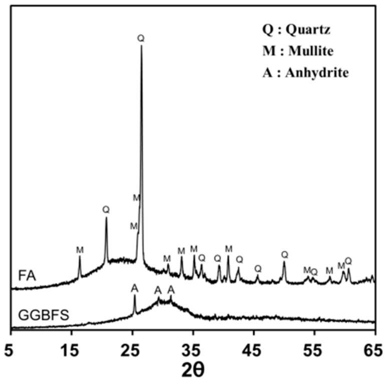


**Figure 1.** X-ray diffraction patterns of FA and GGBFS

### 2.2. Mix proportions

In mortar mixture design, the mass ratio of binder to fine aggregate was 1: 3 and the water/binder ratio was 0.5. The procedures for obtaining amount of activator and extra water are as follows. i) Determine $Na_2O$ content and $SiO_2/Na_2O$ molar ratios (Ms). ii) Calculate the amount of sodium silicate solution to be added from the determined Ms value. iii) Calculate the amount of $Na_2O$ by converting the required $Na_2O$ to NaOH excluding the amount of $Na_2O$ in the sodium silicate solution. iv) Add water to make water/binder ratio 0.5 including the amount of $H_2O$ in the sodium silicate solution. The overall mixture design used in this study is summarized in Table 2. The activator concentration was 8% of $Na_2O$ and 1.4 of Ms in FA based geopolymer and 4% of $Na_2O$ and 1.0, 1.5, 2.0 of Ms in GGBFS blended mortars. For each case, GGBFS was replaced with 30 wt.% or 50 wt.% of the FA amount, respectively. The mortar specimen preparation method given in ISO 679 was followed for mixing and placement [14]. After mixing, the FA based geopolymer specimens were cured at 70 °C, and GGBFS blended specimens were cured at 23 °C or 70 °C. After 24 hours curing, they have been kept until test periods in the constant temperature and relative humidity conditions (23℃ of temperature and 60% of R.H.). The size of the specimen for measuring the compressive strength and mass change was 40 × 40 × 160 mm and the specimen for length change was 25.4 × 25.4 × 295 mm.

**Table 2.** Mixture proportions of mortars

| Mix | Factors | | | | Mass proportions (g) | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | FA (%) | GGBFS (%) | $Na_2O$ (%) | Ms[a] | Cement | FA | GGBFS | NaOH | Sodium silicate solution | Water | Sand |
| FA100-1.4 | 100 | - | 8 | 1.4 | - | 450 | - | 25.4 | 175.8 | 114.4 | 1350 |
| S30-2.0 | 70 | 30 | 4 | 2.0 | - | 315 | 135 | 8.2 | 125.6 | 146.0 | 1350 |
| S50-1.0 | 50 | 50 | 4 | 1.0 | - | 225 | 225 | 15.7 | 62.8 | 185.5 | 1350 |
| S50-1.5 | 50 | 50 | 4 | 1.5 | - | 225 | 225 | 11.9 | 94.2 | 165.8 | 1350 |
| S50-2.0 | 50 | 50 | 4 | 2.0 | - | 225 | 225 | 8.2 | 125.6 | 146.0 | 1350 |
| OPC | - | - | | | 450 | - | - | - | - | 225 | 1350 |

[a] (SiO2/Na2O) molar ratio

### 2.3. Test methods

The mass change was measured by removing the water on the surface of the test specimen immersed in the sulfate solution with regarding to each age. The length change was measured by inserting studs at both ends of the specimen and using the length of the specimen cured for 28 days as a reference length. The length of the specimen immersed in the sulfate solution was measured according to KS F 2424 with regarding to each age and calculated by the Equation (4).

$$Length\ chagne(\%) = \frac{L_t - L_i}{L_i} \qquad (4)$$

Where, $L_t$ : Length(mm) at the immersion day (t), $L_i$ : length(mm) before immersion (28 days)

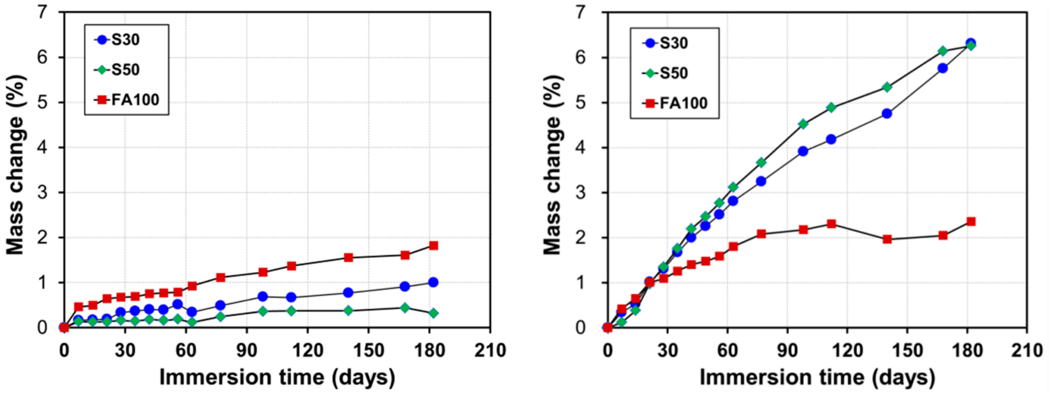
The compressive strength of specimen was measured according to ISO 679, and the loading rate of the compressive strength test was 2,400 N/s ± 200 N/s. XRD was analyzed using a Rigaku MiniFlex 600 diffractometer (40 kV and 20 mA). The scans ranged from 2θ angle equal to 5 degree to 65 with a step size 0.02 2θ.

## 3. Test results and discussion

### *3.1. Mass change*

In order to evaluate the sulfate resistance according to the amount of GGBFS in the alkali activated binder, the amounts of GGBFS were adjusted to 0%, 30% and 50% as shown in Table 2. The initial curing conditions of FA100 were 24 hours at 70 ℃, and S30 and S50 were 24 hours at 23 ℃ and 70 ℃, respectively. After initial curing, all specimens were cured in a chamber at 23 °C and 60% relative humidity for 28 days, and then immersed in 10% sodium sulfate ($Na_2SO_4$) and 10% magnesium sulfate ($MgSO_4$) solution. After that, the mass change of the specimen was measured by each age.

As shown in Figure 2, the difference in mass change with immersion time is significant depending on the type of sulfate solution. In case of immersion in 10% $Na_2SO_4$ solution, FA100 showed the largest mass increase of 1.8% at 181 immersion days, and the mass change tends to decrease as the amount of GGBFS increases.



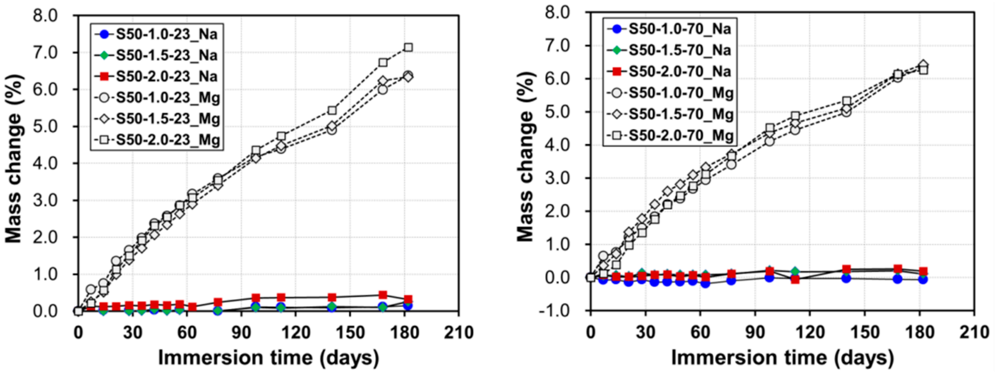
(a) 10% $Na_2SO_4$ solution (b) 10% $MgSO_4$ solution

**Figure 2.** Mass change by sulfate solution type and GGBFS content

In case of immersion in a 10% $MgSO_4$ solution, the mass increase of FA100 at 181 days of immersion was 2.3%, which is similar to that of 10% $Na_2SO_4$ solution. However, in case of S30 and S50, the mass increased continuously as the immersion age was increased in a 10% $MgSO_4$ solution, and mass increase at 181 days was 6.3% for S30 and 6.25% for S50, respectively. Therefore, it was confirmed that the sulfate resistance of the alkaline activated GGBFS blended mortar showed excellent performance to the sodium sulfate solution but the sulfate resistance to the magnesium sulfate solution was poor.

As is well known, Ms in alkali activated mortars has a great influence on the microstructure and compressive strength [15-17]. In this study, the mass change was measured with the duration of immersion by changing Ms (1.0, 1.5, and 2.0) and curing temperature (23 ℃ and 70 ℃) for S50. As shown in Figure 3, even if the immersion period is increased, the mass change is very small within 0.4% regardless of the initial curing temperature and Ms change when immersed in 10% $Na_2SO_4$ solution. In case of immersion in 10% $MgSO_4$ solution, the initial curing temperature of both 23 °C and 70 °C showed a large mass increase according to the immersion age. At an initial curing temperature of 23 °C, the mass increase of S50-1.0-23_Mg was 6.37%, S50-1.5-23_Mg was 6.33% and S50-2.0-23_Mg was 7.14% at 182 days. However, there is no difference in the mass change due to the initial curing temperature condition.

In summary, the sulfate resistance of alkali activated FA based geopolymer mortar shows good performance regardless of sulfate types, but the sulfate resistance of alkali activated GGBFS blended mortar has a greater influence on the sulfate types than Ms and curing temperature.



(a) GGBFS 50%, curing temperature 23℃ (b) GGBFS 50%, curing temperature 70℃

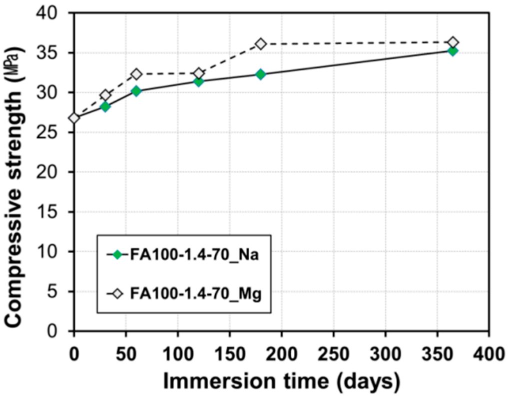
**Figure 3.** Mass change by sulfate solution type, Ms and curing temperature

### 3.2. *Compressive strength change*

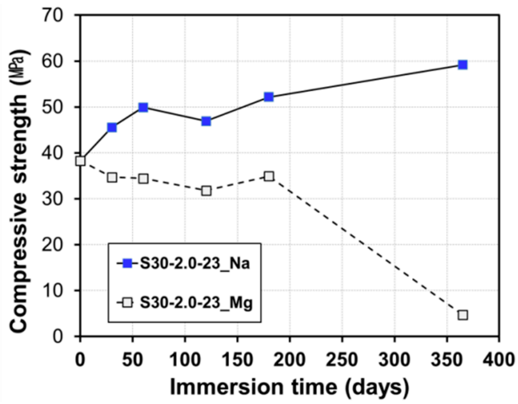
Figure 4 shows the change in compressive strength with the amount of GGBFS mixture (0%, 30% and 50%) and immersion of sulfate solutions (10% $Na_2SO_4$ and 10% $MgSO_4$). When the specimens cured up to 28 days were immersed in 10% $Na_2SO_4$ solution for one year, the compressive strength of the mortar was found to be higher than the strength before immersion in all specimens. Similar to the results of the mass change experiment, it was confirmed that the deterioration of the sulfate does not occur in the 10% $Na_2SO_4$ solution regardless of whether the GGBFS is mixed or not.

In the case of immersion in 10% $MgSO_4$ solution, the FA100 exhibited enhanced compressive strength of 36.3 MPa after one year immersion compared to before immersion (26.8 MPa). However, when S30 and S50 mixed with GGBFS were immersed in 10% $MgSO_4$ solution for one year, the compressive strength was greatly reduced to 12.3% (4.7 MPa) and 35.4% (20.2 MPa) of the strength before immersion.

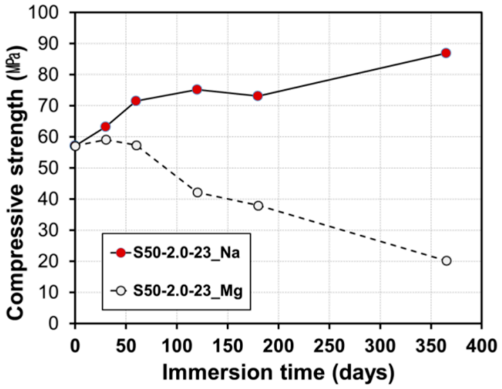
As a result of the mass and the compressive strength change experiment, FA based geopolymer mortars were not affected by the types of sulfate solution. However, when GGBFS is blended, deterioration occurs only in immersion of 10% $MgSO_4$ solution. This is because $Mg(OH)_2$ and $CaSO_4$ are formed by the penetration of magnesium ion and sulfate ion as shown in Equation (2), thus the mass increased and the compressive strength is decreased.



(a) fly ash 100%



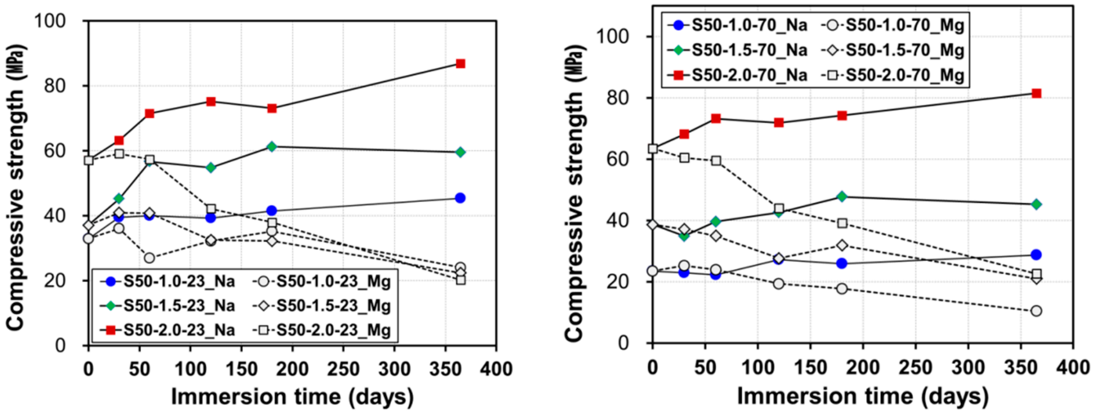
(b) GGBFS 30%



(c) GGBFS 50%

**Figure 4.** Compressive strength change by GGBFS contents and sulfate solution types

Ismail et al. have shown that the response of alkali activated GGBFS-FA blends to sulfate attack is strongly dependent on the nature of the cation accompanying the sulfate; $Na_2SO_4$ immersion caused little damage, while $MgSO_4$ caused loss of structural and dimensional integrity [18]. Figure 5 shows the compressive strength change of GGBFS blended mortars with respect to Ms and initial curing temperature. In case of immersion in 10% $Na_2SO_4$ solution, compressive strength was increased in all specimens irrespective of the change of Ms. The strength enhancement rate of the initial curing specimen at 23℃ was higher than that of the initial curing specimen at 70℃. Similar to the results of the mass change experiment, there was no decrease in compressive strength when immersed in 10% $Na_2SO_4$ solution regardless of initial curing temperature and Ms change. In case of immersion in 10% $MgSO_4$ solution, the compressive strength decreases consistently with the immersion period.

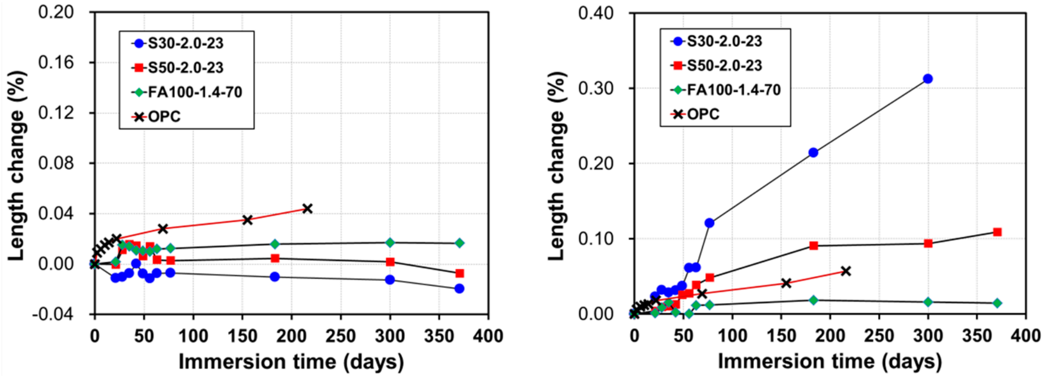


(a) GGBFS 50%, curing temperature 23℃ (b) GGBFS 50%, curing temperature 70℃

**Figure 5.** Compressive strength change by Ms, sulfate solution type and curing temperature

### 3.3. Length change

Experimental results on the length change of alkali activated mortar depending on the amount of GGBFS and sulfate solution types were shown in Figure 6. As shown in Figure 6 (a), the OPC mortar expands by 0.04% or more when immersed in 10% $Na_2SO_4$ solution, whereas the alkali activated mortars exhibit a smaller length change rate than OPC even after one year of immersion. This means that sulfate deterioration does not occur even when alkali activated mortars are immersed in a sodium sulfate solution, similar to the results of measurement of mass and compressive strength change. Although FA100 exhibits a similar rate of length change regardless of the sulfate types, the rate of length change is very large when S30 and S50 are immersed in 10% $MgSO_4$ solution. Specifically, S30 showed 0.31% expansion rate at 300 days of immersion and S50 showed 0.09% expansion rate at 300 days of immersion.



(a) 10% $Na_2SO_4$ solution (b) 10% $MgSO_4$ solution

**Figure 6.** Length change by sulfate solution type and binder types

Alkali activated mortar (S30-2.0-23) was adjusted to 2.5%, 5% and 10% of $MgSO_4$ concentration in order to measure the rate of length change with regarding to the immersion time. As shown in Figure 7, it can be seen that the rate of length change varies according to $MgSO_4$ concentrations. The maximum length change rate was 0.21% at $MgSO_4$ concentration of 10% at 180 days of immersion, and almost no expansion occurred at $MgSO_4$ concentration of 2.5%. Therefore, it can be seen that the deterioration due to the sulfate is greatly reduced in the low concentration $MgSO_4$ solution.

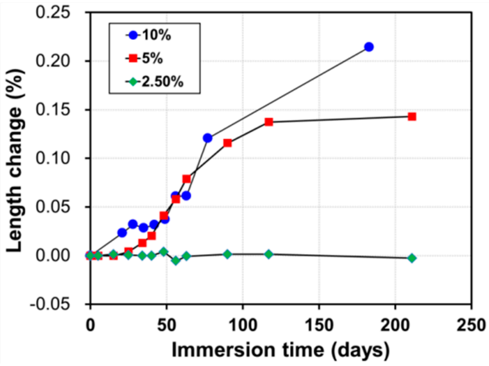
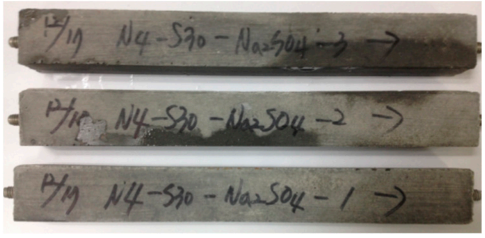


**Figure 7.** Length change by magnesium sulfate solution concentrations

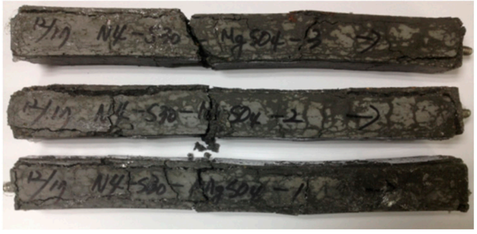
As shown in Figure 8, it can be seen that the specimen collapsed due to the expansion of the mortar immersed in the 10% $MgSO_4$ solution. This is because the gypsum is formed by equation (2) when alkali activated mortar is immersed in a 10 % $MgSO_4$ solution, and the generation of such gypsum results in an increase in mass, expansion and compression strength reduction.

Figure 8 shows the specimens of alkali activated mortars immersed in the sulfate solutions. As shown in Figure 8(c), the specimen collapsed due to the expansion of the mortar immersed in the 10% $MgSO_4$ solution. This is because the gypsum is formed by equation (2) when alkali activated mortar is immersed in a 10 % $MgSO_4$ solution, and the formation of gypsum leads to increase in mass, increase in length and decrease in compressive strength.

(a) FA100 - 10% $MgSO_4$ solution



(b) S30 – 10% $Na_2SO_4$ solution



(c) S30 - 10% $MgSO_4$ solution

**Figure 8.** Mortar specimens according to sulfate solution types

### *3.4. Mineral composition change*

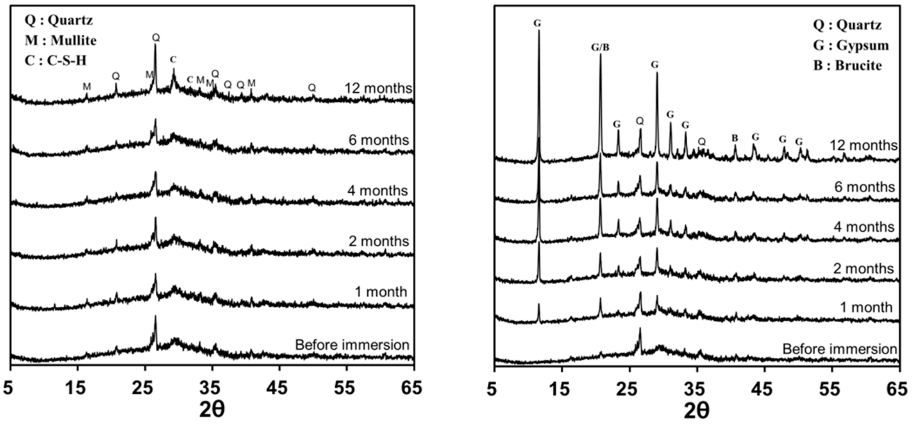
It was confirmed in the previous experiments that the sulfate resistance of the alkali activated mortar varies depending on the types of sulfate solution. In this experiment, the change of mineral composition of alkali activated mortar immersed in sulfate solutions was confirmed by X-ray diffraction analysis. Figure 9(a) shows the results of X-ray diffraction analysis of specimens immersed in 10% $Na_2SO_4$ solution. No change in mineral composition was observed until the one year of immersion. Quartz ($SiO_2$), mullite ($3Al_2O_3.2SiO_2$) and C-S-H were observed as main crystalline components of alkali activated GGBFS blended binder.

However, a very large difference can be seen from the results of x-ray diffraction patterns in the immersion of 10% $MgSO_4$ solution as shown in Figure 9(b). Peaks of gypsum ($CaSO_4.2H_2O$) and brucite ($Mg(OH)_2$) were observed from one month after immersion in 10% $MgSO_4$ solution, which is confirmed by the reaction of Equation (2). It is considered that the peaks of gypsum and brucite become higher as the immersion time is elongated because of the increase in the amount of their production. According to Ismail et al, $Mg^{2+}$ ions promote the decomposition of calcium compounds (Ca-rich gels)[18].

$$3MgSO_4 + 3CaO \cdot 2SiO_2 \cdot 3H_2O + 8H_2O$$

$$\rightarrow 3(CaSO_4 \cdot 2H_2O) + 3Mg(OH)_2 + 2SiO_2 \cdot H_2O \quad (5)$$

When alkali activated mortars were immersed in 10% $MgSO_4$ solution, the increase in mass, increase in length and decrease in compressive strength of alkali activated mortars were caused by the formation of gypsum and brucite due to the reaction of magnesium ion as shown in Equation (5) [12]. From the experimental results, it can be concluded that the alkali activated GGBFS blended mortar does not deteriorate in the presence of sulfate only, but deteriorates by the formation of gypsum and brucite when magnesium and sulfate ions are present together. In addition, FA based geopolymers that produce aluminosilicates gels that are not C-S-H as reaction products show excellent sulfate resistance regardless of the sulfate solution types.



(a) S30 - 10% $Na_2SO_4$ solution (b) S30 - 10% $MgSO_4$ solution

**Figure 9.** XRD patterns of sulfate solution types for GGBFS(S30)

## **4. Conclusions**

The following results were obtained from the evaluation of sulfate resistance of alkali activated FA based geopolymer and GGBFS blended mortars according to the types of sulfate solution.

(1) Alkali activated GGBFS blended mortars, unlike FA based geopolymer mortars, cause a significant mass increase immersed in 10% $MgSO_4$ solution.

(2) The compressive strength of alkaline activated mortars showed a great variation according to the cation accompanying sulfate. In 10% $Na_2SO_4$ solution immersion, the compressive strength does not decrease in all alkali activated mortars. However, in the immersion of 10% $MgSO_4$ solution, the alkali activated GGBFS blended mortars show a significant decrease in compressive strength.

(3) Alkali activated mortars were less expandable than OPC mortar immersed in 10% $Na_2SO_4$ solution, but alkali activated GGBFS blended mortars show a remarkable expansion immersed in 10% $MgSO_4$ solution.

(4) Ms plays an important role in the compressive strength of alkali activated mortars, but does not affect the sulfate resistance of alkali activated GGBFS blended mortars.

(5) XRD results showed that the deterioration of sulfate in the alkali activated GGBFS blended mortar was due to the decomposition of C-S-H by magnesium ion and the formation of gypsum and brucite. In addition, alkali activated FA based geopolymer mortar exhibits excellent sulfate resistance regardless of the sulfate solution types since there is no reaction product such as $Ca(OH)_2$ and C-S-H.

**Acknowledgments:** This research was supported by Korea Ministry of Environment(MOE) as “Public Technology Program based Environmental Policy”. This research was also supported by the Korea Institute of Energy Technology Evaluation and Planning (KETEP) and the Ministry of Trade, Industry & Energy (MOTIE) of the Republic of Korea (No. 20181110200070).

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