Assessment of salt stress resistance of concrete containing sodium silicate-based nano-silica

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ABSTRACT

This study proposes advanced sodium silicate-based nano-silica (SS) as a supplementary cementitious material for enhancing the physical properties and electrochemical performance of the concrete to increase the salt stress resistance of reinforced concrete structures located in the seashore. Moreover, the mixture of the proposed material was compared with ordinary Portland cement and binary-blended cement mixtures, which are conventionally used. In order to evaluate salt stress resistance of reinforced concrete, two different laboratory-scale salt stress environments are artificially designed and one natural place along the seashore is adopted, and reinforced concrete specimens are exposed to those environments. In addition to salt stress resistance properties of all mixtures the compressive strength of all concrete mixtures is conducted. The results of salt stress resistance test for all environments show that the mixtures containing SS have much better salt stress resistance than conventional mixtures. Meanwhile, the amount of SS between 5% and 10% of SS to cement ratio by weight do not significantly affect the salt stress resistance. For compressive strength, specimens containing SS show higher compressive strength than other concrete mixtures.

1. Introduction

Concrete is very vulnerable to the aggressive invasion of fluids and ions due to its porous microstructure (Xu, Jiang, and Wang 2009; Ghafoori and Najimi 2016; Almusallam et al. 2003). Recent studies have evaluated nano-sized mineral admixtures that can provide unique properties to concrete, replacing a portion of cement (Qing et al. 2007; Kim et al. 2004). These studies examined the effects of various nano-sized materials (e.g., nano-SiO$_2$, TiO$_2$, Al$_2$O$_3$, and Fe$_2$O$_3$) on the physical and mechanical characteristics of concrete. They have reported that particularly spherical nano-silica (SiO$_2$) among nano-sized materials improved the durability of concrete owing to its nano-filler effect and pozzolanic reaction (Ghafoori and Najimi 2016; Almusallam et al. 2003; Rahman et al. 2007; Senff et al. 2009). In the previous study of this, we compared two nano-silica types (i.e., sodium silicate-based nano-silica (SS) and ethyl silicate-based nano-silica (ES)) that were synthesized through the sol-gel process, which is a synthesis technique of silicon oxide and reported the effects of cement composites on pore characteristics (Kim et al. 2014). SS used in the previous study was synthesized based on sodium silicate and it was able to make the particle size of SS three times smaller than the particle size of ES that is synthesized based on tetraethyloxysilicate (Kim et al. 2014). Due to this characteristic, SS induces quicker pozzolanic reaction and better pore-filling effect than ES in cement composites. The pozzolanic reaction and filling effect commonly reduce the pores of cement composites as well as decrease the permeability of concrete at the same time. Therefore, it can “physically” block the invasion of external deterioration factors (Qing et al. 2007; Kim et al. 2014; Monticelli et al. 2016). Moreover, this phenomenon also accord with the required performance of concrete to prevent water and chloride ions from reaching the reinforcing steel of reinforced concrete (RC) structures, which is exposed to a marine environment. Meanwhile, even though the external deterioration factors can physically be blocked by concrete, the corrosion of rebar embedded in concrete is related to the electrochemical action of the chloride ions. Regarding this, many studies have investigated on binding chloride ions in concrete, preventing from reaching them to rebar embedded in concrete. In addition to binding chloride ions in concrete, some studies attempted to increase the service life of RC structures by utilizing electrochemical techniques such as electric extraction of previously infiltrated chloride ions from concrete (Elsener, Molina, and Böhni 1993; Andrade et al. 2001; Lambert, Page, and Vassie 1991; Miranda et al. 2006). However, in most studies above the researchers commonly concluded that it would be difficult to completely block deterioration factors such as chloride ions using simply physical methods such as improving strength and water tight-ness of concrete.
This study proposes a method to improve not only physical properties of concrete but also electromagnetic performance of concrete for preventing the invasion of chloride into RC structures exposed to a marine environment using sodium silicate-based nano-silica (SS). In order to investigate the resistance of chloride ion for SS, three different salt damage environments, two artificial environments and one actual coastal environment are established and all specimens are exposed to all environments. Afterwards, the chloride resistance tests in terms of corroded area rate, half-cell potential (HCP) of rebar embedded in concrete, and penetration depth of chloride ions are conducted for SS mixtures. In addition, the results of the chloride resistance tests for SS mixtures are compared to concrete specimens containing ordinary Portland cement (OPC) and ground granulated blast-furnace slag (GGBS).

2. Conceptual mechanism of SS in cement composite

Aforementioned, in the previous study of this, it was able to make the particle size of SS three times smaller than the particle size of ES as can be seen in Figure 1 (Kim et al. 2014). In this regard, it can be anticipated that SS induces quicker pozzolanic reaction and better pore-filling effect than ES in cement composites.

The following three expectations of SS in cement composites are anticipated; a quick pozzolanic reaction with cement composites and pore-filling within the cement matrix. These reactions physically create the dense concrete and prevent the intrusion of sea water and sea salt particles. Lastly, the final third reaction is to chemically prevent the diffusion of chloride ions (Cl\textsuperscript{−}) while inducing electrochemical equilibrium through the negative charge formation of the nano-silica surface and the inert halogen in the same group as the internal chloride ions as shown in Figure 2. The inert halogen group formed on the surface is synthesized by the nano-silica and organic oligomers. Denatured halogenated epoxy acrylate, used for synthesizing the organic oligomers, was synthesized through the half-esterification reaction from anhydride or halogenated anhydride. Figure 3 shows the chemical structure of the products generated from a half-esterification reaction.

3. Experimental programs

3.1. Establishment of an artificial marine environment

Three artificial marine environments that chloride ions could easily invade into the concrete from outside are set up as can be seen in Table 1. Among these environments, Type A and Type B were designed to have a similar condition to the coastal area and those were designed to have more extreme conditions than the general salt-stress condition in the coastal area to shorten the period of the experiments. Moreover, a control group (Type C) was exposed to the actual marine environment in South Korea up to 36 months to compare the group with the artificially exposed groups.

3.2. Materials

OPC with 3,150 kg/m\textsuperscript{3} of density, 346 m\textsuperscript{2}/kg of fineness, and meeting Korean Standard L 5201 (KS L 5201) are used (Association, K.S. 2016). GGBS with density of 2,940 kg/m\textsuperscript{3}, fineness of 435 m\textsuperscript{2}/kg, and equivalent to ASTM C989 is used (ASTM, ASTM C989 2012). River sand with density of 2,550 kg/m\textsuperscript{3} and maximum diameter of 5 mm is used as fine aggregates. Crushed natural granite aggregate sourced from southern Korea with density of 2,620 kg/m\textsuperscript{3} and maximum diameter of 25 mm is used as coarse aggregates. SS is incorporated into the cement composites as a mineral admixture. The density, viscosity, and the solid content by water weight of SS are 1,210 kg/m\textsuperscript{3}, 7 cp, and 30%, respectively.

3.3. Mixture proportion

It is specified in Korean Concrete Standard Specification that a water/binder ratio of marine concrete should not be greater than 0.5 as well as the

Figure 1. SEM micrographs of nano-silica.
amount of binders should be larger than 330 kg/m$^3$ (Institute, K.C. 2016). However, since this study not only uses a new type of binders, advanced sodium silicate-based nano-silica, to improve salt stress resistance of concrete but also compares its performance to concrete containing the existing materials such as OPC and GGBS, worse scenario than the regulation was taken account for concrete mix design. In this regard, OPC is used as a control mixture in this study and binary-blended cement with 60% of OPC and 40% of GGBS for the treatment in order to evaluate the effects of SS mixture. The mixture proportions of the applied concretes are presented in Table 3. The SS replacement of cement established 5% and 10% by binder mass. The water to binder ratio (w/b) of the concretes was fixed to 0.6 by mass for all mixtures.

### 3.4. Evaluation methods

In order to make specimens for tests, two rounded steel rebar with 150 mm in length and 10 mm in diameter are inserted into cylindrical concrete molds ($\Phi100 \times 200$ mm)

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**Table 1.** Exposure condition of specimens to salt stress.

| Type   | Established salt stress environment (1 cycle is composed of one wetting treatment and one drying treatment) | Number of Cycle Repetitions |
|--------|------------------------------------------------------------------------------------------------------|-----------------------------|
| Wetting |                                                                                                      |                             |
| ESS (A)| Spray saline solution* (Max. 65°C, R.H. 90%)

3days
| Drying |
|--------|--------------------------------------------------|
| ESS (A)| Spray saline solution† (Min. 15°C, R.H. 60%)

4days |

| ESS (A) | Up to 40 cycle |
|---------|----------------|
| ESC (B)| Exposed to 10% CO$_2$ (gas)

Up to 40 cycle |

| ESS (B)| Exposed to saline open environment which was within 1km from a seashore

(Mean monthly temperature: Max. 25.2°C and, Min. 0.5°C)

1month |

| ESO (C)| 36 months |

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*Chemical composition of artificial solution is shown in Table 2

ESS (A): Exposed to spray saline solution without CO$_2$ gas; ESC (B): Exposed to spray saline solution with CO$_2$ gas; ESO (C): Exposed to saline open environment.

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**Table 2.** Chemical composition of saline solution (1 L).

| Component                        | Mass (g) |
|----------------------------------|----------|
| Sodium chloride (NaCl)           | 24.5     |
| Magnesium chloride (MgCl$_2$ · 6H$_2$O) | 11.1 |
| Sodium sulfate (Na$_2$SO$_4$)    | 4.1      |
| Calcium chloride (CaCl$_2$)      | 1.2      |
| Potassium chloride (KCl)         | 0.7      |

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**Figure 2.** Conceptual diagram for chemical treatment on the surface of SS.

**Figure 3.** Chemical structure of half-esterification reaction product.

**Table 3.** Mixture proportions of the applied concretes.
and fresh concrete is placed into the molds, and the mold is kept in a mist room at 20 ± 3°C for 24 h until demolding. Afterwards, all reinforced concrete specimens are demolded and are cured for 28 days in a chamber room at a temperature of 20 ± 3°C and relative humidity of 50 ± 10%. Figure 4 shows the scheme of a reinforced concrete specimen.

Meanwhile, in order to evaluate the salt stress resistance of RC specimens, which were exposed to three marine environment types for a certain period of time, three different experimental methods are used in this study. Each method is described in the following subsections.

### 3.4.1. Measurement of corroded area ratio for rebar

The corrosion area of embedded rebar is measured by extracting the embedded rebar by splitting specimens exposed to salt stress for each predetermined cycle and month, marking the visually identified corrosion part. Finally, the marked areas are calculated using software. As a result, the corroded area ratio is presented in the corroded area over the total surface area of the rebar as can be seen in Equation (1). The process of measuring the corroded area ratio of the rebar embedded in the concrete is shown in Figure 5.

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\text{Corroded area ratio(%) = } \frac{A_{\text{corr}}}{A_{\text{total}}} \times 100
\]

### 3.4.2. Measurement of half-cell potential (HCP) for rebar

In order to measure the corrosion activity of the steel bar embedded in concrete specimens, an HCP test for uncoated rebar is conducted in accordance with ASTM C 876 (ASTM, ASTM C876 2012). Table 4 shows the corrosion probability evaluation criteria described in ASTM C876 on the potential probability for rebar. Figure 6 shows HCP measurement of rebar embedded in concrete specimens.

### 3.4.3. Measurement of penetration depth of chloride ions

In order to measure the penetration depth of chloride ions, RC specimens are split at the predetermined cycle and duration, which is same as conducting on measuring the corroded area ratio of rebar. Afterwards, 0.1 N AgNO₃ solution is sprayed on the cross-section of the RC specimens. The areas infiltrated with chloride ions are discolored to silver, which allows it to measure the depth of penetration.

### 3.4.4. Compressive strength

For compressive strength test, cylindrical specimens for all mixtures are produced by casting fresh concrete in cylindrical molds without rebar and kept in a mist room at 20 ± 3°C for 24 h until demolding. Once those are demolded, the specimens are placed in a water

| Class. | W/B | S/a | Water | Binder | C | GGBS | SS | Sand | Gravel | S₁ (mm) | A₁ (%) | F₂(t) (Days) | F₃ (MPa) |
|--------|-----|-----|-------|--------|---|------|----|------|--------|---------|--------|-------------|----------|
| OPC    | 0.6 | 0.45| 180   | 300    | -  | -    | -  | 772  | 970    | 215     | 5.1    | 13.9        | 27.0     |
| BSC    | 180 | 180 | 120   | 774    | 972| 230  | 4.6 | 13.8 | 30.1   | 13.8    | 27.4   | 37.6        |
| SS5    | 180 | 285 | -     | 777    | 976| 230  | 3.9 | 13.1 | 27.4   | 14.4    | 28.1   | 35.2        |
| SS10   | 180 | 270 | -     | 768    | 965| 225  | 3.9 | 13.1 | 27.4   | 14.4    | 28.1   | 35.2        |
bath at temperature of 25 ± 2°C for 28 days. After the bath curing, the specimens for compressive strength are exposed to each salt stress environment and are tested for compressive strength as specified in ASTM C39 at each predetermined cycle and exposure period. Three specimens per concrete mixture are conducted on compressive strength and the average value of three concrete specimens is reported.

4. Result and discussion

4.1. Corroded area ratio of rebar embedded in concrete

The surface corrosion shape and corroded area ratio of embedded rebar are evaluated for the specimens exposed to ESS and ESO. These were conducted on specimens under the ESS environment after 40 cycles and under the ESO environment for 36 months, which could be relatively the most corroded. Since this study visually examines the corroded areas of rebar and then manually marked on the area, accuracy, and reliability might not be high. However, it can be stated that the results of this study would be meaningful because it directly measures the degree of corrosion of embedded rebar. Eventually, the average value of two steel bars embedded in one specimen was used.

It is very difficult to precisely predict where initial corrosion occurs because the corrosion shape of the embedded rebar is irregular and various. However, the corrosion of rebar generally has a tendency to spread from the initial location to all directions once erosion begins. This is because once various factors that deteriorate concrete penetrate through weakened concrete cover parts, the deteriorated path makes easier to penetrate the exterior deterioration factors through itself.

Table 5 and Figure 7 show corroded area and ratio of rebar, respectively, for ESS and ESO. As can be seen
in Table 5 and Figure 7, the corroded area of OPC is slightly greater than BSC specimen. The corroded area of SS mixtures shows significantly lower than non-SS mixtures. For specimens exposed to the ESS environment, which was severer condition than the ESO environment, the total average surface areas of rebar for OPC, BSC, SS5, and SS10 showed 183.0, 150.6, 55.4, and 54.6 mm², respectively. These correspond to 3.88%, 3.20%, 1.18%, and 1.16% of the corroded area ratio for OPC, BSC, SS5, and SS10, respectively. These results imply that SS mixtures have more favorable properties in terms of chloride resistance than OPC and BSC mixtures. Also, these results might be attributed to that SS particles are easily able to fill in capillary spaces and pores in concrete matrix, which is in good agreement with other studies (Rao, Bhattacharyya, and Barai 2011; Alhawat and Ashour 2020).

### 4.2. HCP results of rebar embedded in concrete

The corrosion potentials of all specimens exposed to ESS, ESC, and ESO conditions at each cycle and month are shown in Figure 8. As can be seen in Figure 8, most specimens showed similar potential values around $-100$ mV in the period of cycle 4 to cycle 12 corresponding to the initial exposure. This indicates that the cover depth of concrete could prevent from invading chloride ions in the initial period. After 16 cycles, the resistance to salt stress varies with concrete mixing characteristics. In other words, it was observed that

### Table 5. Average corroded areas of two rebar.

| Class   | OPC| BSC| SS5| SS10 |
|---------|----|----|----|------|
| ESS     | Avg. 183.0 mm² | Avg. 150.6 mm² | Avg. 55.4 mm² | Avg. 54.6 mm² |
| ESO     | Avg. 114.2 mm² | Avg. 68.4 mm² | Avg. 28.4 mm² | Avg. 7.0 mm² |

Figure 7. Corroded area ratio of all mixtures under ESS and ESO conditions.
OPC and BSC were more vulnerable to the intrusion of salt stress than SS mixtures since 16 cycles. In addition, for both ESS and ESC conditions at 32 cycles, OPC and BSC specimens exceed −350 mV that is classified as high corrosion probability specified in ASTM C876. On the other hand, half-cell potential values of SS series were kept above −300 mV, which can be classified as uncertain level in terms of corrosion probability.

As can be seen in Figure 8(c), even though the increase rate of corrosion probability for specimens under ESO condition was lower than the specimens exposed to the artificial marine environment conditions, it was clearly shown that SS series improved the resistance to salt stress compared to other mixtures. Meanwhile, based on results at 36 months, the maximum measurement duration conducted in this study, the half-cell potential of SS series showed relatively more favorable value than OPC and BSC specimens. It is plausible to conclude that SS-based nano-silica might attribute the reduction of half-cell potential values to a quick pozzolanic reaction with cement composites and pore-filling within the cement matrix as well as the chemical reaction that prevents the diffusion of chloride ions while inducing electrochemical equilibrium through the negative charge formation of SS surface and the inert halogen in the same group as the internal chloride ions (Kim et al. 2014; Snehal, Das, and Akanksha 2020). The amount of SS did not significantly affect half-cell potential value.

**4.3. Infiltration resistance of chlorine ions**

Figure 9 shows the infiltration depth of chloride ions for specimens exposed to ESS, ESC, and ESO environment at each cycle and month. As can be seen in Figure 9, under ESS condition, chloride penetration depth of OPC and BSC specimens at 40 cycles reached up to 23 and 22 mm, respectively. The chloride ions for SS5 and SS10
specimens at 40 cycles were penetrated up to 13 and 11 mm, respectively. For ESS condition, chloride penetration depth of all specimens at each cycle showed a similar tendency with specimens under ESC condition. Meanwhile, comparing chloride penetration depth of SS series to OPC and BSC, SS series decreased the penetration depth up to 44%. Based on this, it could be stated that adding SS in concrete has good effect on controlling the penetration of chloride ions, agreeing with other studies (Chalee, Jaturapitakkul, and Chindaprasirt 2009; Li et al. 2019, 2017). However, the amount of SS does not show a significant effect on controlling the penetration of chloride ions.

For concrete specimens under ESO condition, as the exposure time increases, the depth of chloride ions penetration gradually increases. Chloride penetration depth for OPC and BSC concrete specimens exposed to ESO condition at 36 months reached about 14 to 15 mm, while SS5 and SS10 specimens approximately showed 8.2 and 5.5 mm of chloride penetration depth, respectively. According to the concrete standard specification of Korean Concrete Institute 2004 ((KCI), K.C.I. 2006), it is stipulated that RC structures nearby the seafront should have at least 40 mm of concrete cover depth. Regarding this, based on the data obtained from ESO specimens, the estimation time to reach 40 mm of chloride ions penetration depth was calculated using linear regression analysis as can be seen in Figure 10. Figure 11 shows the estimation time that chloride ions reach into the concrete cover depth. As can be seen in Figure 11, the estimation time to reach rebar in concrete, 40 mm, for OPC, BSC, SS5, and SS10 turns out to be 93 (7.8 years), 97 (8.1 years), 173 (14.4 years), and 253 (21.1 years) months, respectively.

4.4. Compressive strength

The results of compressive strength tests for each concrete mixture are shown in Figure 12. As can be seen in

5. Conclusion

This study proposes advanced sodium silicate-based nano-silica as a supplementary admixture to enhance the physical properties and electrochemical performance of concrete in terms of salt stress. Regarding this, two artificial marine environments in laboratory scale and
actual marine environment were established to evaluate salt stress resistance of concrete containing the sodium silicate-based nano-silica, comparing to concrete specimens containing ordinary Portland cement and ground granulated blast-furnace slag. Throughout this study, the following summaries can be drawn:

Based on results obtained from this study in terms of resistance performance to chloride ions, it can be clearly mentioned that SS is able to improve resistance performance to salt stress in comparison with ordinary Portland cement and ground granulated blast-furnace slag. However, the effects of the amount of SS are not significant. This means that a relatively small amount of SS can sufficiently develop the electromagnetic actions in concrete composite, improving its resistance performance to salt stress. Also, it has been found that SS can improve the physical property of concrete such as compressive strength. Throughout this study, it can be concluded that SS can develop favorable properties of concrete as a salt stress resistance material; therefore it can be a useful material for structures that salt stress resistance performance is required.

**Disclosure statement**

No potential conflict of interest was reported by the authors.

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![Figure 12. Results of compressive strength.](image_url)
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