

Effects of Combined Salt-Damage Resistant Agent on the Shrinkage, Chloride Penetration and Chemical Erosion of Mortar



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Abstract: Reinforced concrete structures located on coastal landfill frequently adjoin sea-water environment, and are exposed to sea water and humid environment during construction. Particularly, in the case of large-scale structures like dams, their drying shrinkage is accompanied by fatal cracking, and thus chlorine ion penetration becomes easier. The present study develops a salt damage-resistant agent (SRA) to which aluminum salts, oligomer condensate, and amino alcohol derivatives with the alkyl group are applied as binding inducers. SRA performs the roles of reducing the drying shrinkage of cement composites, binding chlorine ions, and preventing erosion by sulfate ions. This study tests and evaluates its resistance to degradation factors that may occur to structures constructed on coastal landfill and so on.

As a result of evaluating shrinkage cracking properties by performing the restrained shrinkage cracking test, SRC showed the shrinkage reduction compared with BSC. As for the performance of resistance to chlorine ion and the chemical sulfate erosion rate, SRC showed the highest resistance performance, followed by BSC and OPC, regardless of the concentration of aqueous solutions for immersion. In addition, as for the rate of mortar weight change by sulfate erosion, the SRA-intermixed SRC mixture showed a weight reduction rate at the level of 1/3 of BSC and 1/6 of OPC, respectively.

Keywords: Aluminum salts, Oligomer Condensate, Chloride Penetration, Dry Shrinkage, Chemical Erosion.

I. INTRODUCTION

Salt damage to concrete structures arises from external factors such as the penetration of chlorine ions from the outside or the intermixture of sea water. Particularly, it directly results in the corrosion of embedded reinforced bars, and it has been known that the degree or frequency of the damage is very serious [1, 2]. The chloride ion, which affects concrete performance degradation, has the form of free chloride or exists in the phase equilibrium state of binding ion within concrete [3, 4]. The chloride binding methods include the physical method that shuts up salt damage factors in cement micro-pores through the water-tightness and densification of concrete itself, and a chemical method for intermixing cations such as Na^+ , K^+ , Si^{2+} and Mg^{2+} into concrete and inducing reaction with anions such as Cl^- and SO_4^{2-} .

Such chemically bound chloride ions do no longer exist as salt damage factors (CaCl_2 , NaCl , and CaSO_4) that damage structures, but turn into the phase equilibrium state including Friedel's salt, and consequently decrease the occurrence of salt damage [5, 6]. In XRD assessment, Zhu [7] showed the order of binding ratios (or reaction ratios) among the cations of Na^+ , K^+ , Ca^{2+} , and Mg^{2+} ($\text{Ca} > \text{Mg} > \text{Na} \approx \text{K}$) through their reaction with chloride ion, and reported that CaCl_2 was generated at the highest ratio. Arya [8] showed the quantified binding ratio for each of chlorides NaCl (43%), CaCl_2 (65%), and MgCl_2 (61%), which include the chlorine ion. In addition, in the case of the hydration products of cement, most researchers including Zibara [9] reported that the aluminate (C_3A) had the greatest effects on the chloride binding ratio, and it is known that this results from the generation of $\text{C}_3\text{A} \cdot \text{CaCl}_2 \cdot 10\text{H}_2\text{O}$ (Friedel's salt). Like this, research on chemical binding to induce the binding of chloride ions and the phase equilibrium state of chlorides have been carried out in various ways, and in Korea also, relevant products have been developed and applied mostly in the form of admixtures.

The present study reviewed chemical equations by applying as binding inducers aluminum salts, oligomer condensate, and amino alcohol derivatives with alkyl group for the purpose of chemical binding of the free chloride ion (Cl^-). The expected chemical equations and mechanisms of their binding are as follows:

II. ION BINDING ALGORITHMS

As for aluminum inorganic salts, they show chemical binding reaction with the free chloride ion by reacting like Equation (1) within concrete [10]. In addition, it is expected that some aluminum ions dissociated in unreacted state perform the role of inducing the generation of chelate complex compounds and $\text{Al}(\text{OH})_3$ (expansive-hydration) and reduce the drying shrinkage of concrete. As applicable aluminum salts, it is deemed that aluminum salts such as aluminum sulfate ($\text{Al}_2(\text{SO}_4)_3$), aluminum hydroxide ($\text{Al}(\text{OH})_3$), and aluminum oxide (Al_2O_3) as well aluminum nitrate ($\text{Al}(\text{NO}_3)_3$) can be equally applied [11].



Also, in the case of the oligomer condensate, it is an oligomer-type compound ($\text{C}_{20}\text{H}_{20}\text{N}_4\text{O}_9\text{S}$) with the molecular weight of 1,500 – 2,000 (Mw), and has the physical properties that can improve the rheology of concrete and minimize bleeding that causes shrinkage,

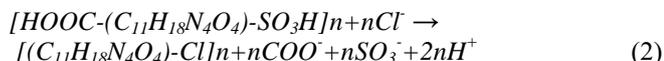
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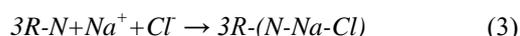
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and chemical properties that can bind chloride ions in the concrete during the substitution reaction (Equation (2)). The binding of chloride ions appears in such a form that the sulfonate group and carboxyl group of the compound are separated and then combine with chloride ions, and is characterized by increasing in proportion to the condensation degree (n) of oligomer condensate.



where, n is the degree of condensation (in the range of 3-5).

Lastly, in the case of amino alcohol derivatives (3R-N, R: alkyl group), they are acyclic compounds having basic molecular structure. It is deemed that chemical adsorption of chloride ions will be possible as shown in the chemical equation below.



The developed aluminum inorganic complex salts, the oligomer condensate of organic matter, and the amino alcohol derivatives are manufactured in the form of liquid admixture, together with other compounds. After then, they are intermixed with cement composites and act as admixtures to improve durability against salt damage attack and drying shrinkage.

III. SURVEY OF ENVIRONMENTAL CONCENTRATIONS IN COASTAL LANDFILL

The ratios and concentrations of chloride ion and sulfate ion contained in sea water vary greatly according to the chemical composition of soil and sea water in countries or regions. It is deemed necessary to conduct the prior review of soil and salt damage environmental concentrations [12-14]. Hence, this study randomly selected 27 coastal landfills, and measured the chloride ion (Cl⁻) and sulfate ion (SO₄²⁻) concentrations and pH of their underground water according to vertical distances from the ground (EL), which are shown in Figures 1 – 3.

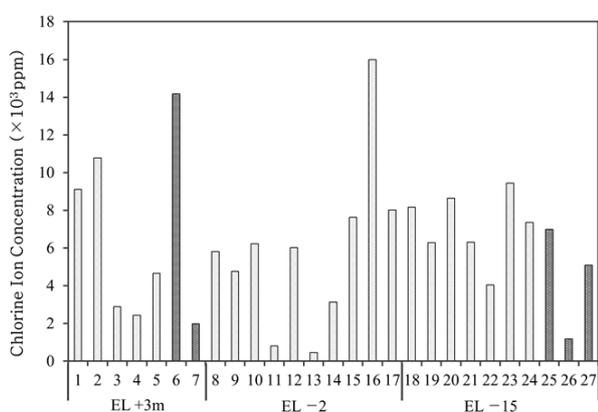


Figure 1. Chlorine ion concentration of coastal landfill

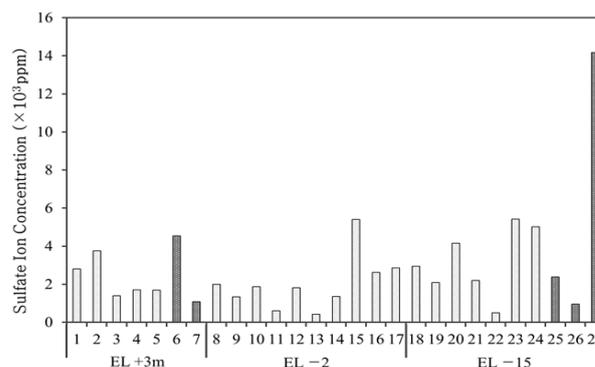


Figure 2. Sulfate ion concentration of coastal landfill

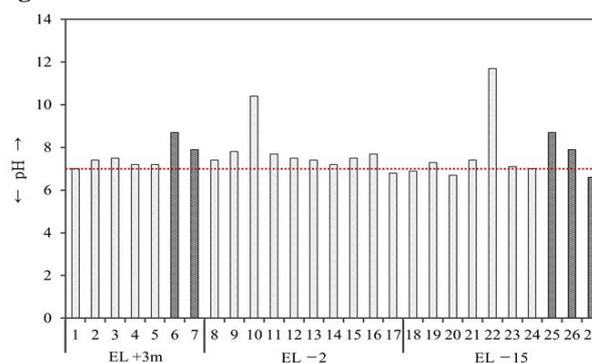


Figure 3. Hydrogen ion concentration (pH) of coastal landfill

In the case of chloride ions, minimum 364.5 ppm (mg/l) to maximum 16,094 ppm were detected, albeit varying more or less with survey locations. They not only exceeded the regulation criterion (300 mg/l) of Korean Concrete Standard Specification [15], but also chloride ions of high concentration reaching maximum 50 times the criterion were found. In the case of sulfate ions as well, most exceeded 1,500mg/l, a criterion for using sulfate-resistant Portland cement recommended by the sulfate ion standard specification, and even the maximum 14,180mg/l was surveyed. On the other hand, in the case of pH, the hydrogen ion concentration scale, it was surveyed that mostly the neutral state of 7 or above was maintained. It is deemed that the situation is in danger of damage, given that most chemical erosion of concrete is caused by the chemical action of chlorine ion or sulfate ion, not pH. Particularly, the ratio of sulfate ion was more or less higher than the existing reviews, and it was judged that erosion could be caused by sulfate as well as salt damage.

IV. EXPERIMENTAL PROGRAM

The present experiment conducts the experimental review of the performance of salt damage resistant agent (SRA) developed to prevent chemical erosion by chlorine ion or sulfate ion and cracks caused by the drying shrinkage of concrete. The review items include whether performance for compressive strength decreases or not, the quantitative assessment of cracking, and the assessment of resistance to chlorine ion and sulfate ion. For the mixture of mortar, the water-binder ratio (W/B) was set to 0.47, and the mass ratio of cement and sand to 1:2:5, respectively.

As for the binder, two types of 100% ordinary Portland cement (OPC) and the blast furnace slag cement (BSC), which has 1:1 mixture of ground granulated blast furnace slag and OPC, were used.

In addition, SRA, 0.6% of binder weight, was mixed into BSC that is widely used for marine structures or SOC structures. Thus, mixtures of 3 types in total were reviewed. The evaluation items of the present experiment are drying shrinkage cracking under restrained condition, the depth of chlorine ion penetration, and the degree of erosion caused by sulfate. Among them, as for evaluation relating to chlorine ion and sulfate, experiments were performed with two different ion concentrations of Cl^- and SO_4^{2-} .

A. Materials and Mixture proportion

OPC with the density of 3,140 kg/m³ and the fineness of 343 m²/kg, which meets Korean Standard (KS) L 5201, was used. GGBS with the density of 2,940 kg/m³ and the fineness of 435m²/kg, which is equivalent to ASTM C 989, was used. And river sand with the density of 2,550 kg/m³ and the maximum diameter of 5mm was used as fine aggregates. Lastly, the physical and chemical properties of liquid SRA are shown in Table 1, and the mixture proportion of mortar for the present experiment in Table 2, respectively.

Table 1. Physical and Chemical properties of SRA

Density (kg/m ³)	Appearance	Chemical composition (%)			
		Silicon inorganic salt	Aluminum inorganic salt	Oligomer Condensate	The others
1,124	Dark brown solution	21±2	46±1	20±1	13

Table 2. Mixture proportion of mortar

Class	W/B (%)	Binder : Sand (weight ratio)	Binder (%)		SRA dosage ratio (B×%)
			OPC	GGBS	
OPC	47.0	1:2.5	100	-	-
BSC			50	50	-
SRC			50	50	0.6

B. Evaluation methods

To identify the restrained shrinkage property of mortar, the plate-ring type test was conducted. The test of restrained shrinkage cracking properties is a method partly applying the drying shrinkage cracking test suggested by the existing ASTM 1581-04 and AASHTTO 34-99 specifications, and enables the quantitative evaluation of shrinkage crack occurrence.

The specimen was set by a ring-type mold with the external diameter of Ø400mm and the internal diameter of Ø150mm (Figure 4 a, b)), and was placed with the height of 50 mm as standard. It



Figure 4 a). Restrained shrinkage cracking test mold

was cured under the environment of 20±3°C and 60±5% R.H. for 28 days. Then, the width and length of cracks on the concrete surface were measured with a crack scale. In addition, grid lines that divided the surface of the 125mm-wide specimen into five equal parts were set according to ASTM C 457, and portions where cracks adjoin the lines were defined as crack points, and their number was calculated.

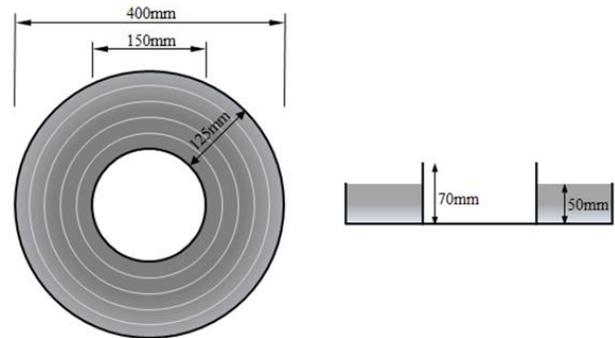


Figure 4 b). Designed restrained shrinkage cracking mold

As an experiment to evaluate the resistance of mortar to salt damage and sulfate, the depth of chlorine ion penetration and the rate of change in mortar weight caused by sulfate were reviewed, respectively. First, to check the depth of chlorine ion penetration according to whether SRA is intermixed or not and the kinds of binders, the mortar specimen of Ø70×150mm were fabricated, and standard water curing was carried out for 7 days after placement. After then, to promote the penetration of chlorine ion, NaCl aqueous solutions of 3 (w/w)% and 15(w/w)% were prepared, respectively, and the fabricated specimen were immersed into the solutions. As for the method for measuring the depth of penetration, the specimen were split on the 7th, 28th, and 56th days after immersion, the indicator was sprayed on check surfaces, and the depth of discolored portions was measured. As for the chlorine ion indicator to induce reaction with chloride contained in concrete, it was prepared at the concentration of 0.1N after drying AgNO₃ for more than one hour at 130°C according to KS M ISO 6353-2.

After waiting for the sufficient reaction of the indicator with chlorine ion on the check surface, the indicator of uranine (C₂₀H₁₀Na₂O₅) was prepared at 1(w/w)% and was sprayed, and then the penetration depth was measured.

On the other hand, for the measurement of the rate of change in mortar weight caused by sulfate, the aqueous solution of H₂SO₄ was used according to ASTM C 267. Similarly to the above chloride penetration test, H₂SO₄ aqueous solutions were prepared with two concentrations of 5 and 10 (w/w)%. Specimen were taken out on the 7th, 14th, and 28th days after immersion. And their weight was measured after they were dried at room temperature, and was compared with their initial weight to measure the rate of reduction in weight.

V. RESULT AND DISCUSSION

A. Compressive strength

Figure 5 shows the compressive strength of mortar according to its ages pursuant to KS L 5105. As a result of measuring the compressive strength for three mixtures of OPC, BSC, and SRA, while BSC and SRA showed a little high strength at the age of 56 days, OPC showed a little bit high strength at the initial age of 7 days. This resulted from differences in binders for the mixtures, and it is deemed that GGBS in substitution for OPC had positive effects on the development of long-age strength. Besides the effects of the binders, the compressive strength property for all ages showed no particular difference due to the intermixture of SRA, and thus it may be deemed that SRA does not become a factor of damage to the strength.

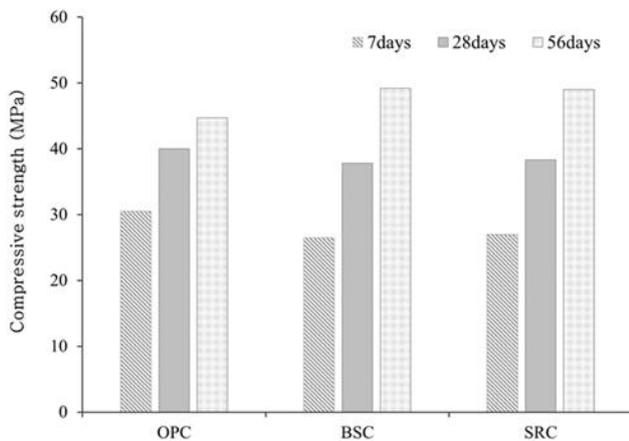


Figure 5. Compressive strength of the mortar

B. Plate-ring type drying restrained shrinkage cracking

Table 3 shows the shapes, gross area and points of specimen cracks according to mixtures found by the plate-ring type restrained shrinkage test at the age of 28 days. First, among the three types of mixtures set according to the kinds of binders and whether SRA is intermixed or not, SRA intermixed mortar (SRC) shows the most excellent shrinkage control performance. As a result of measuring the width and length of cracks occurring at the age of 28 days, and calculating their areas, while OPC showed a high cracking area of 228.1mm², the cracking area of SRC was found to be 125.1mm², which is 45.2% of OPC and 39.8% of BSC.

Overall, results of the present test using mortar showed a little higher quantity of cracks than the test using concrete. This is because coarse aggregates in concrete reduce shrinkage, and the comparatively large amount of evaporation is also another factor.

On the other hand, referring to ASTM C 457, the intersecting points of cracks on the surface and four lines dividing the longitudinal direction of the ring were set as crack points, and their number was counted. Overall, the cracks showed the shape of extending from the center towards the outside due to the phenomenon of stress concentration at the center of the ring. In OPC and BSC that had relatively wide cracking areas, a relatively large quantity of crack points appeared at the ring located at the center (Ø125, 150). The crack points of SRC were a total of 24, showing the occurrence rate of 44.4% of OPC and 53.3% of BSC.

Table 3. Shape, gross area and points of the surface crack

Class		OPC	BSC	SRA
Crack shape and crack point				
	Gross area of crack (mm ²)	228.1	207.7	125.1
	Crack point			
Crack point	Ø100mm	10 (18.5%)	7 (15.5%)	6 (25.0%)
	Ø125mm	17 (31.5%)	9 (20.0%)	8 (33.4%)
	Ø150mm	17 (31.5%)	17 (37.8%)	5 (20.8%)
	Ø175mm	10 (18.5%)	12 (26.7%)	5 (20.8%)
	Total	54 (100%)	45 (100%)	24 (100%)

The above findings result from the chemical action of aluminum salts and multi-cyclonic oligomer condensate included in SRA, and are attributed to the action of minimizing bleeding, which is a factor of initial mortar shrinkage, the expansion action of aluminum itself, and the formation of chelate complex compound.

C. The depth of chlorine ion penetration in mortar

The depth of chlorine ion penetration in mortar immersed for 7 to 56 days in NaCl aqueous solution prepared to evaluate the resistance of mortar to chlorine ion was reviewed. The depth of chlorine ion depth is depicted in Figure 6 according to the age of immersion and the concentration of aqueous solution, and the photos of the relevant specimen are shown in Table 4.

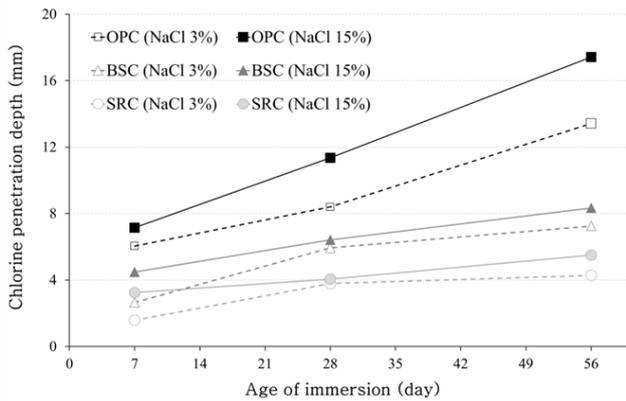


Figure 6. Chlorine penetration depth in the mortar

Similarly to the above shrinkage and cracking evaluation, SCR showed the most excellent results in resistance to chlorine ion penetration, followed by BSC and OPC. Particularly, as of the 56th day after immersion, BSC showed 8.33 mm and OPC 17.42 mm, respectively, and thus it was found that the resistance of BSC to chlorine ion is two times or more excellent compared to OPC. Given that the compressive strength of OPC and BSC showed similar results, it is deemed that this is because the micro-pores of BSC mortar were filled to form physically dense structures owing to the pozzolanic reaction of GGBS itself.

SRC specimen immersed in the NaCl aqueous solution of relatively high concentration (15%) showed lower penetration depth than OPC and BSC specimen immersed in that of low concentration (3%). This indicator shows that even the environment of severe salt harm attack can be overcome by material resistance performance of the cement composite itself. Although the concentration of aqueous solutions for immersion showed five-fold differences, the chlorine ion penetration depth in SRC (NaCl 15%) showed 40.9% of OPC (NaCl 3%) and 75.8% of BSC (NaCl 3%), on the basis of the age of 56 days. It is deemed that the chlorine ion binding action of material is a very important determinant of mortar durability under salt attack, and it was experimentally proved that the action of SRA in mortar shows excellent resistance to salt harm.

On the other hand, according as immersion in chlorine ion continues, the penetration depth showed generally constant increase. This is a result showing that chlorine ion penetrates continuously over time, but for through crack or other fatal

defect in structure. It is deemed that this result may become basic data for calculating the time for chlorine ion to reach reinforced bar or the penetration rate of chloride according to the thickness of concrete encasement.

Table 4. Chlorine ion penetrated section (NaCl 15%)

Class	OPC	BSC	SRC
Immersion 7 days			
Immersion 28 days			
Immersion 56 days			

D. Mortar weight reduction by sulfate ion

Unlike chlorine ions that penetrate mortar and affect rebar corrosion or neutralization, sulfate ions have structural effects on the cement composite itself, and thus its weight reduction or strength decrease is observed [16]. Figure 7 shows the rate of mortar weight reduction due to surface stripping and scaling at some ages measured after mortar was cured in water for 7 days and immersed in 5% and 10% H₂SO₄ solution. The reduction rate was represented by the percentage of reduced weight to mortar weight immediately before immersion, and was measured after mortar was taken out of a tank and was dried for more than 3 hours to remove moisture weight.

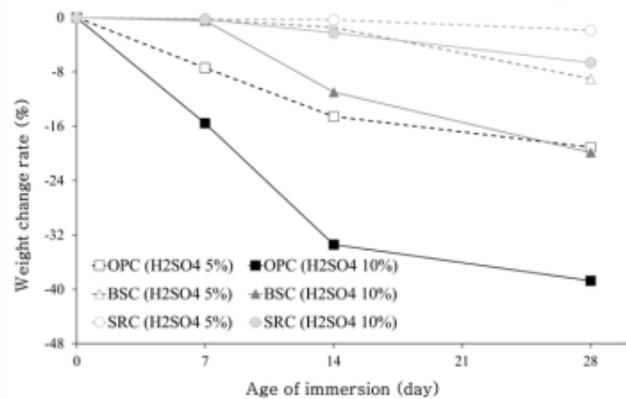


Figure 7. Weight change rate of the mortar by sulfuric acid

At the age of 7 days in the initial stage, all the results except for OPC (10%) showed almost no change, and showed sharp differences after 28 days. Sulphate first reacts with Ca(OH)₂, a cement hydrate, to generate gypsum, and the gypsum in turn reacts with ettringite-group compound to result in volume expansion and surface detachment.

It is deemed that owing to this required elapse of time, the influence was trivial at the initial immersion stage, but brought about great change rates after then in OPC where the residual amount of $\text{Ca}(\text{OH})_2$ was high. On the basis of specimen aged 28 days after immersion in a highly concentrated H_2SO_4 aqueous solution (10%) that causes high sulfate erosion attack, OPC showed the weight reduction rate of 38.75%, BSC 19.88%, and SRC 6.63%, respectively. Compared with SRC, BSC showed three times weight reduction, and OPC six times weight reduction, respectively. As a result of testing the resistance to sulfate, BSC showed high resistance to sulfate by securing watertightness due to the pozzolanic effect of GGBS, compared with OPC showing a high residual rate of calcium hydroxide. As for SRC where durability improvement agent was applied to BSC, however, it is deemed that soundness against salt harm could be secured according as the transition zone of salt harm binding was formed due to the complex action of aluminum salts and oligomer condensate.

VI. CONCLUSION

This study developed a salt damage resistant agent (SRA) by applying as binding inducers aluminum salts, oligomer condensate, and amino alcohol derivatives with the alkyl group, for the purpose of the chemical binding of chloride and sulfate and the securing of resistance to cracking due to shrinkage. It was applied to mortar, and its resistance to degradation factors that can occur in coast or landfill was evaluated by tests.

1) As a result of randomly selecting Korean coastal landfills and conducting a survey of environmental concentrations, it was found that the concentrations of chlorine ion and sulfate ion greatly exceeded criteria of the standard specification. In case of planning the construction of structure at landfill or coast, prior review will have to be carried out regarding salt harm concentration in the relevant region.

2) As a result of intermixing the developed SRA in mortar and evaluating the compressive strength, there was no great effect of SRA intermixture, and thus it was found that it is not a harmful factor in strength development.

3) As a result of evaluating shrinkage cracking properties by performing the restrained shrinkage cracking test, SRC showed the shrinkage reduction rate of 39.8% compared with BSC. In addition, it was found that its crack points adjoining lines had high shrinkage compensation performance, showing 44.4% of OPC and 53.3% of BSC.

4) As for the performance of resistance to chlorine ion and the chemical sulfate erosion rate, SRC showed the highest resistance performance, followed by BSC and OPC, regardless of the concentration of aqueous solutions for immersion. As of the age of 56 days, SRC was found to have excellent chloride binding performance and chemical adsorption performance by showing the low chlorine ion penetration depth of 34% and 68% compared with BSC and OPC, respectively. In addition, as for the rate of mortar weight change by sulfate erosion, the SRA-intermixed SRC mixture showed a weight reduction rate at the level of 1/3 of BSC and 1/6 of OPC, respectively.

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