

## A Study on Chloride Binding Property of Concrete using $\text{CaO}\cdot 2\text{Al}_2\text{O}_3$ as Admixture

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### ABSTRACT

A basic study is carried out to evaluate the effects of a newly developed mineral admixture based on  $\text{CaO}\cdot 2\text{Al}_2\text{O}_3$ , (“ $\text{CA}_2$ ”) a kind of calcium aluminate, on properties of concrete. The admixture is expected to have two important functions for protecting chloride attack on concrete structures; immobilization of chloride ions and to make concrete denser by filling pores. To examine the effectiveness of the admixture, tests on mechanical properties of  $\text{CA}_2$  concrete and its ability to reduce chloride penetration under marine environment are conducted. In the experiment, four types of concrete with ordinary Portland cement partially replaced by  $\text{CA}_2$  with percentages of 0%, 5%, 7%, 9% of the cement are used. From experimental results, strength of concrete tends to decrease with increasing replacement rate of  $\text{CA}_2$  in binder. It is clearly confirmed that chloride penetration into concrete is controlled and has high ability to immobilize the chloride ions.

**Keywords.** Calcium Aluminate, Chloride Attack, Chloride Permeability, Rebar Corrosion, Neutralization.

### INTRODUCTION

Recently in Japan, many structures, which were built in the rapid economic growth period during 1955 to 1973, are under serious conditions and deteriorated. They have to be demolished soon. Especially on these structures which are constructed near sea or in the coastal environment, earlier progress of the deterioration due to chloride attack made their endurance fall off and urgent repair is required. On the other hand, a project to extend the life of new structures under such severe environment is also currently in progress. However, the techniques which are put to practical use for repair of existing structures or extension of service life of newly constructed ones in the chloride attack environment have not been familiarized to the public because of its cost and technical problems, except for the important structures. Therefore, in future, extending the life of the concrete structure in large scale and

a wide variety of applications is important. The development of a new technology that combines low cost and simple workability is necessary as soon as possible.

In this study, as a countermeasure method to reduce the damage from the chloride attack with the low cost performance, a mineral admixture based on  $\text{CaO}\cdot 2\text{Al}_2\text{O}_3$ , a kind of calcium aluminate (herein-after-called as “CA<sub>2</sub>”), is newly developed. As shown in the Eq. (1), the admixture reacts with calcium hydroxide in cement and produces Hydrocalumite (herein-after-called as “HC”) when it is mixed in concrete (Tabara, 2010). HC is expected to have two important functions for protecting the chloride attack; one of which is to combine with chloride ions in concrete to produce Friedel’s salt as shown in the Eq. (2), and another is to make concrete more dense by filling effect in pores (Tabara, 2011).



Therefore, by replacing CA<sub>2</sub> with cement before making concrete, would increase the resistance of concrete to chloride attack. However, so far, performance evaluation of concrete with CA<sub>2</sub> has not been done and remains unclear in terms of strength and durability.

In this study, resistance to chloride attack, such as anti-corrosion effect and the effect of immobilization of chloride ions of concrete where cement is replaced partially by CA<sub>2</sub> is studied. The concrete with CA<sub>2</sub> is also examined by exposure test in marine environment, for properties like carbonation and strength. The replacement ratio of CA<sub>2</sub> in the binder is varied to determine optimum replacement level.

## OUTLINE OF EXPERIMENT

**Materials and Mix Proportions.** Material used in the study are, ordinary Portland cement (here-in-after called “OPC concrete”), CA<sub>2</sub>, of which density and specific surface area are 2.93g/cm<sup>3</sup> and 3100cm<sup>2</sup>/g, respectively, is used as a mineral admixture with different replacement ratios to make concretes. Fly-ash (2.25g/cm<sup>3</sup> density, 3990cm<sup>2</sup>/g specific surface area,) and blast furnace slag (2.90g/cm<sup>3</sup> density, 4180cm<sup>2</sup>/g specific surface area) are also used as the mineral admixture, of which properties are compared with the one of CA<sub>2</sub>. sea sand with density of 2.52g/cm<sup>3</sup> and water absorption of 2.66%, in which chloride

**Table 1. Chemical composition**

Material	contents (mass%)									
	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	CaO	MgO	SO <sub>3</sub>	K <sub>2</sub> O	TiO <sub>2</sub>	MnO	Na <sub>2</sub> O
CA <sub>2</sub>	0.62	67.60	8.85	22.30	0.27	0.01	0.01	0.02	0.08	0.19
OPC	18.00	4.35	4.32	66.90	1.56	3.49	0.58	0.31	0.10	0.26
GGBS	30.70	14.10	0.40	44.10	7.39	1.68	0.34	0.63	0.40	0.27
FA	65.00	19.20	5.73	5.03	1.12	0.73	1.65	0.94	0.10	0.43

**Table 2. Mix proportion of concrete**

Sample	W/B (%)	s/a (%)	Unit Weight (kg/m <sup>3</sup> )							(B×wt%)		Slump (cm)	Air (%)
			W	C	CA <sub>2</sub>	GGBS	FA	S	G	AE1	AE2		
OPC	50	42.5	185	370	-	-	-	721	991	0.30	0.006	7.5	4.3
CA2-5%				352	19	-	-	721	990	0.35	0.006	9.0	4.8
CA2-7%				344	26	-	-	720	990	0.40	0.006	7.5	5.0
CA2-9%				337	33	-	-	720	989	0.45	0.006	7.0	5.5
BB				185	-	185	-	715	979	0.20	0.006	9.0	4.0
FA				296	-	-	74	711	990	0.25	0.010	9.0	3.0

ions are completely removed, is used as fine aggregate. Crushed stone with density of  $2.56\text{g/cm}^3$  and water absorption  $0.96\%$  is used as coarse aggregate. Air entraining water reducing agent (AE1) and air entraining agent (AE2) are also used for controlling the workability and air content in fresh concrete.

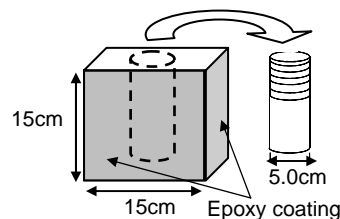
Table 1 shows results of the chemical composition of  $\text{CA}_2$  and other binders determined by X-ray fluorescence spectrometers (XRF). The percentage of  $\text{Al}_2\text{O}_3$  is very high and  $\text{SiO}_2$  is very low in  $\text{CA}_2$  compared to fly-ash, ground granulated blast furnace slag (GGBS) and OPC. Table 2 shows the mix proportions of concrete used in the study. OPC concrete is made using only the ordinary cement as binder. Three types of concrete using  $\text{CA}_2$  are made with the cement in which 5%, 7% or 9% is replaced by  $\text{CA}_2$ , respectively. Concrete using GGBS (here-in-after called “BB concrete”) is made with cement of which 50 % is replaced by GGBS. Concrete with fly-ash (herein-after-called as “FA concrete”) is made with cement of which 20 % is replaced by fly ash.

In the preparation of specimen, water binder ratio for all concretes is fixed at 50%. The target slump is set at  $8.0 \pm 1.5\text{cm}$  and target air amount is set at  $4.0 \pm 1.5\%$ .

**Test methods.** In the examination, accelerated carbonation test and exposure tests in marine environment for concretes are carried out.

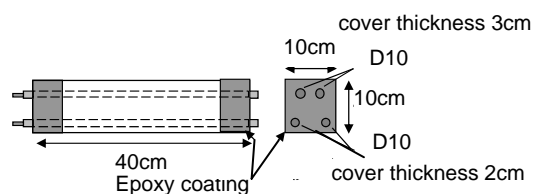
For the carbonation test, cylindrical specimens with  $\phi 10 \times 20\text{cm}$  are used. the specimens are cured in normal water at  $20^\circ\text{C}$  for 28 days, then subjected to the test conditions with 5% of  $\text{CO}_2$  concentration, 60 % of humidity and  $20^\circ\text{C}$  of temperature. After the predetermined acceleration period of time, the carbonation depth is measured by phenolphthalein method at the split surface of specimen.

In the exposure test in tidal zone of marine environment, chloride ion penetration property and steel corrosion protection ability of concrete are examined. In the case of chloride ion penetration test, the cubic specimen having  $15\text{cm} \times 15\text{cm} \times 15\text{cm}$  in size is used. The surface of the specimen are coated with epoxy resin except of the test surface as shown in Figure 1, because the chloride ion penetration should be limited to a one-way direction. After the specimens are cured in water during 28 days, they are stored in the air for 3 months before the exposure tests. To measure chloride ion, a dry concrete core of diameter 5.0cm is taken from the specimen, and it is sliced in several thin pieces having 1.0cm thickness from the top surface, while only the first slice has 0.5cm of thickness. The chloride ion content in each concrete slice is measured to comply with JCI-SC4.



**Figure 1. Specimen for chloride penetration test**

A shape of specimen for examining corrosiveness of rebar in concrete is shown in Figure 2. It is a prismatic specimen of  $10 \times 10 \times 40\text{cm}$  in size with four deformed rebars having 10mm diameter, in which two are set at 2cm cover thickness and other two at 3cm thickness. During the exposure test, natural potential of rebar is measured periodically in accordance with JSCE-E-601-2000 "Test method for half-cell potential of uncoated rebars in concrete



**Figure 2. Reinforced concrete specimen for testing on corrosion of rebar**

structures". After the expiration of test period of time, specimens are dismantled to examine the amount of rebar corrosion.

In the experiment, compressive strength of concrete used is measured in accordance with JIS A 1108-2006 "Method of test for compressive strength of concrete".

## RESULTS AND DISCUSSION

**Pore Structure.** Figure 3 shows the measurement results of the pore size distribution of OPC concrete and CA<sub>2</sub> ones which are cured in the normal water for 28 days. Volume of pore in CA<sub>2</sub> concrete, of which diameter is larger than 3500nm, is relatively large comparing with OPC concrete in spite of CA<sub>2</sub> contents. However, the cumulative pore volume of all CA<sub>2</sub> concretes is smaller than in OPC one. This means that concrete can be dense by adding CA<sub>2</sub> as an admixture.

**Compressive Strength.** Figure 4 shows compressive strength test results for concretes cured in under normal water and the ones exposed in the tidal zone. The strength increases up to 1 year with increase of curing duration. At early ages of curing, OPC concrete and concrete with CA<sub>2</sub> show higher strength than FA and BB concrete. However, three months later, regardless of the replacement ratios of CA<sub>2</sub>, strength of CA<sub>2</sub> is lower than BB, FA and OPC concretes. Furthermore, OPC

concrete has gained strength up to 2 years of exposure period, while CA<sub>2</sub> concretes have not increased in strength between 1 year and 2 years of exposure. With regard to specimens exposed to tidal zone for 1 year, strength of CA<sub>2</sub> concrete is also somewhat lower than BB, OPC and FA concretes. However, the difference of strength between CA<sub>2</sub> concretes and OPC one is smaller in tidal zone than in the normal water curing condition. In the case of concrete under tidal zone environment, the strength of CA<sub>2</sub> concretes have increased slightly between exposure periods of 1 year and 2 years. From the results, lower strength of CA<sub>2</sub> concrete compared to OPC one can be attributed to the fact that replacement of cement by CA<sub>2</sub> relatively increase the water to cement ratio in the mix due to the reduction of substantial cement content. In tidal zone condition, chloride ion diffusing into concrete may produce Friedel's salt due to react with HC. Therefore, there is a possibility that the pore

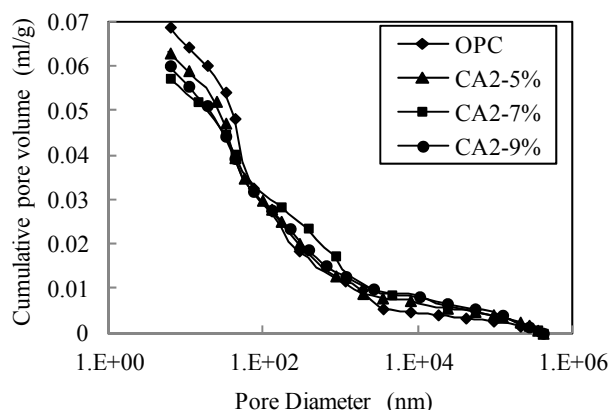


Figure 3. Cumulative pore volume

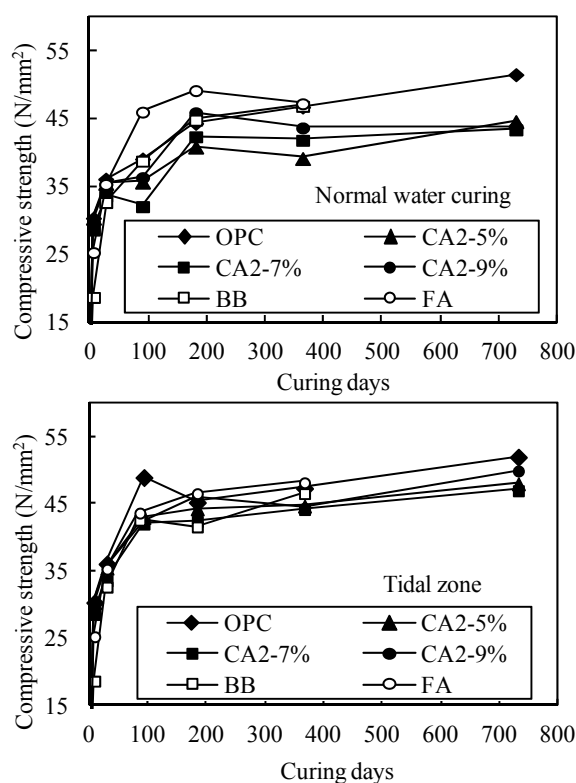
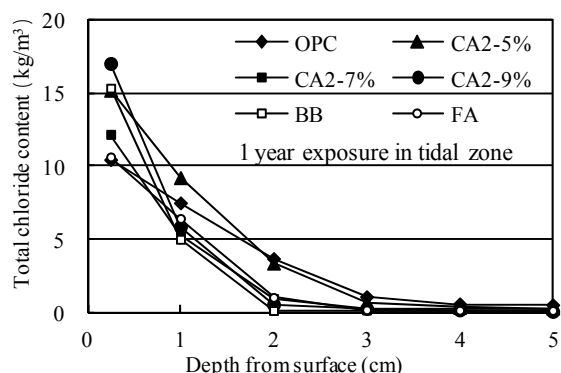


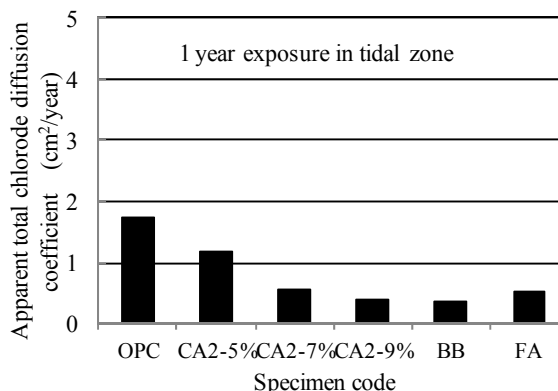
Figure 4. Compressive strength results

structure densification would lead to the higher strength condition comparing to the concrete under the normal water curing. So, the densification of concrete in tidal zone can suppress strength reduction in concrete due to reduction in cement.

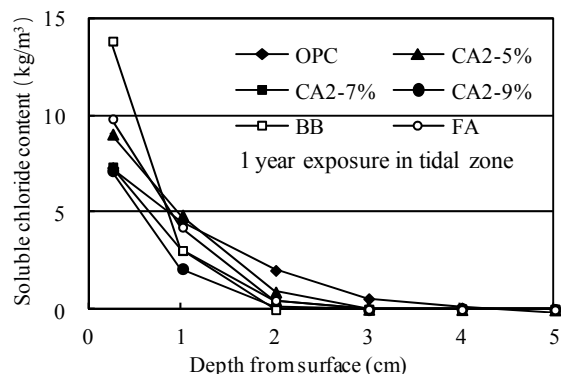
**Properties of Chloride Ion Penetration.** Figure 5 shows the distribution of total chloride ion content in the specimen. The specimens for the test are cured in normal water for 28 days after casting and then exposed to tidal zone. In concrete near the surface, the amount of chloride ions is more in case of CA<sub>2</sub> concretes for all replacement levels and BB one compared to OPC concrete. At deeper positions of specimens, the penetrated chloride content in CA<sub>2</sub> and BB concretes is lower compared to OPC one. The resistance against chloride ingress increases with increase in replacement ratio of CA<sub>2</sub>. At 1cm depth or more from surface, concretes with 7% and 9% of CA<sub>2</sub> contents are less in total chloride content compared to the ones with OPC or 5% of CA<sub>2</sub>. In particular, for the replacement level of 7% of CA<sub>2</sub>, the chloride content is almost same or lesser than FA concrete. In case of 9% of CA<sub>2</sub>, the content is almost same as of BB concrete. Therefore, replacement levels of 7% or more, show remarkable reduction of amount of chloride ion penetration. On the other hand, in the case of 5% of CA<sub>2</sub> replacement level, the chloride content is same as in OPC concrete. The results means that the 5 % of CA<sub>2</sub> replacement does not help in sufficient inhibitory effect of chloride penetration. Therefore, sufficient replacement of CA<sub>2</sub>, to some extent, is necessary to obtain inhibitory effect for chloride penetration. it is because, only 5% of CA<sub>2</sub> does not generate enough amount of HC necessary for densification of concrete.



**Figure 5. Total chloride ion content**



**Figure 6. Apparent chloride diffusion coefficient**



**Figure 7. Soluble chloride ion content**

Figure 6 shows the apparent diffusion coefficient of chloride ion after 1 year of exposure. It is calculated based on the results of Figure 4 in accordance with JSCE-G573-2003 (JSCE Standards, 2004). The apparent diffusion coefficient of chloride ions tend to decrease with increasing replacement ratio of CA<sub>2</sub> and approach the value of BB. Apparent chloride diffusion coefficient in OPC is 1.734cm<sup>2</sup>/year and for concrete replaced by 5% CA<sub>2</sub>, reduction of about 2/3rd of OPC in diffusion is found. As the replacement increases to 7% and 9%, the reduction is about 1/3rd and 1/4th of OPC respectively. These results show a

clear improvement of salt preventive property with replacement of  $CA_2$ . From the result of diffusion coefficient only, it can be seen that the value for concrete with  $CA_2$  7% or more is slightly higher than BB but almost same as FA.

Figure 7 shows the measurement results of soluble chloride ion content that are directly attributable to the corrosion of rebar. The soluble chloride ion content in OPC concrete near the surface is the same compared to the one in  $CA_2$  concretes. At

the depth of more than 1cm from the surface,  $CA_2$  concretes have less amount of soluble chloride ion compared to OPC. These results indicate that the concrete mixed with  $CA_2$  are clearly superior in immobilization ability of chloride ion compared to the OPC one. In case of BB and FA concretes, the difference in total chloride ion and soluble chloride is slight compared to the ones of  $CA_2$  concrete, especially at the surface area. This difference is high for  $CA_2$  9% and the soluble chloride ion is lesser compared to BB at all depths from surface. This effect is due to immobilization ability of  $CA_2$  9% concrete. Figure 8 shows fixed chloride ion ratio of all concretes for 1 year exposure under tidal zone. The amount of fixed chloride ion is regarded as the difference in amount of soluble chloride ion and total amount of chloride ion. A fixed chloride ratio is the ratio of fixed chloride ion to total chloride one. The fixed ratio is calculated for each concrete considering the depth to which the total chloride ion penetrated. At the surface, where chloride content is high, the fixed chloride ratio for  $CA_2$  concrete is higher than for OPC one. The fixed ratio of OPC concrete is 30%, whereas  $CA_2$  5% and 7% are about 40%. Especially  $CA_2$  9% concrete has fixed chloride ratio of around 60%. Replacement of  $CA_2$  in concrete increases the fixing ability of concrete. On the other hand, BB and FA have very less fixed chloride content of less than 10%. At the depths of 1cm and above, the fixed ratio of  $CA_2$  concretes are high compared to OPC and BB ones. FA concrete is similar to the OPC. Hence,  $CA_2$  concrete has two effects, one is densifying concrete and other is immobilization of chloride ions. These can increase the resistance against penetration of chloride into concrete. On the other hand, it is thought that BB and FA have only one effect of making concrete denser thereby suppressing chloride ingress.

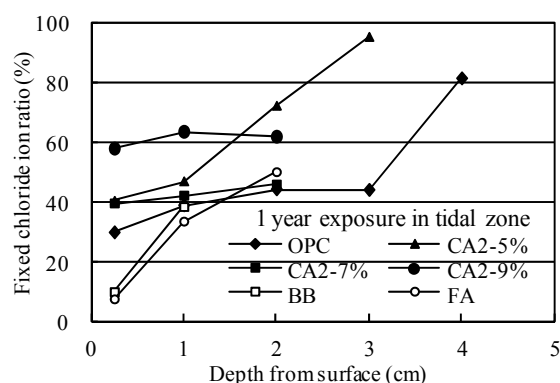


Figure 8. Fixed soluble chloride ratio

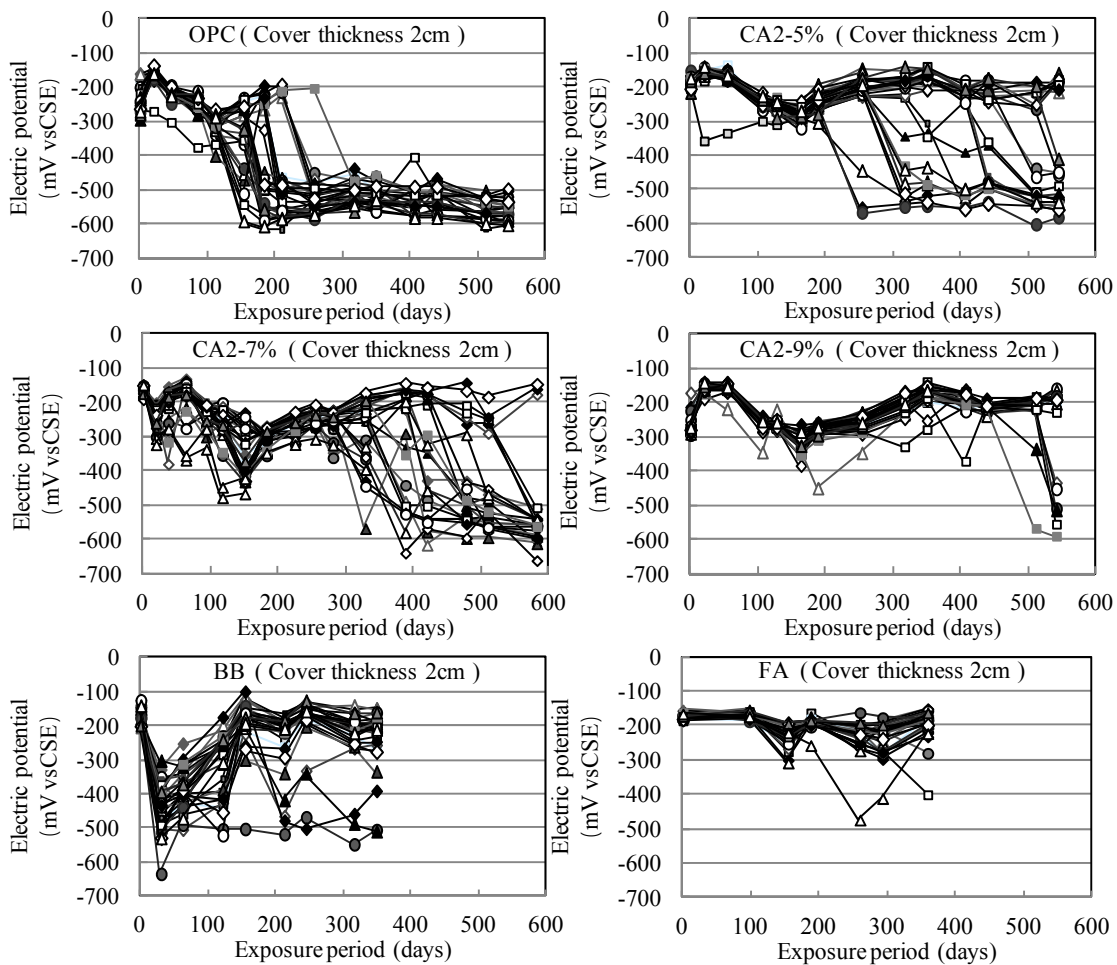
**Protection Ability against Rebar Corrosion.** Figure 9 and Figure 10 show the changes of electric natural potentials of rebars placed at 2cm and 3cm cover thickness in reinforced concrete specimens with dimension of 10\*10\*40cm exposed to tidal zone. In each type of concrete the total of 15 rebars are set with each cover thickness, and after all, the natural potentials of 30 rebars for each type of concrete is obtained. All the results are tabulated because of deviation resulting in measurement. The results of natural potentials are measured during exposure period of 560 days for OPC and  $CA_2$  concretes and 360 days for BB and FA ones.

For the rebars in OPC concrete with 2cm of cover thickness, electric natural potentials of the almost all rebar have been more negative than -350mV vs. CSE at which corrosion of rebar is expected to initiate on rebar in concrete, during the exposure period of about 100 days. All rebars in the OPC concrete have the negative potential more than -350mV about 300 days of exposure, then show a trend of constant decrease of potential after 300 days. In cases of  $CA_2$  concrete, as the replacement level increases, the exposure period of time when the electric

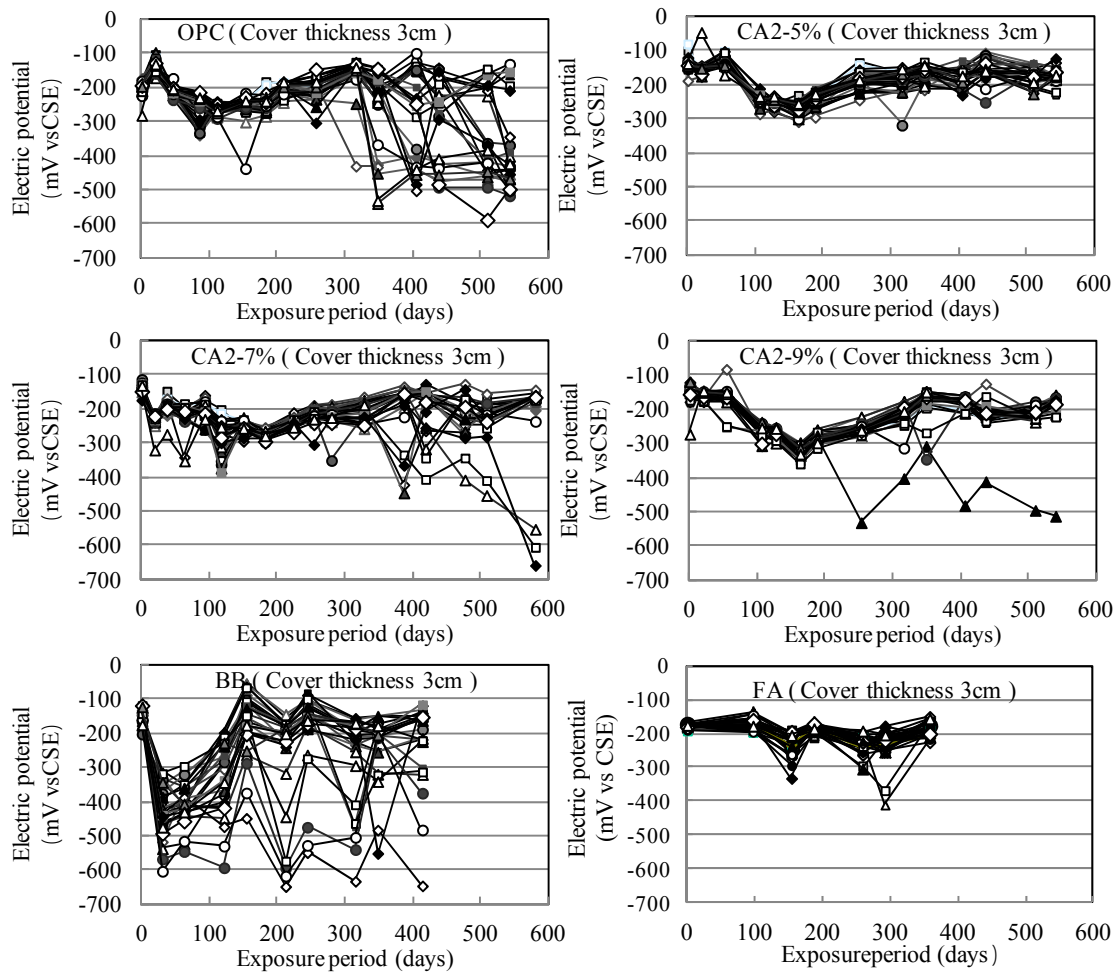
potential of rebars is more negative than  $-350\text{mV}$  extends further. Especially, for concrete with 9% of  $\text{CA}_2$ , almost all rebars have electric potentials around a higher value of  $-200\text{mV}$ . In the case of rebars having 3cm of the cover thickness, electric natural potentials of a few rebars in OPC concrete have changed to more negative than  $-350\text{mV}$  vs. CSE around an exposure period of 350 days. Whereas, potential of  $\text{CA}_2$  concrete rebars, irrespective of replacement ratio, have not decreased until the exposure period of 550 days.

In case of BB concrete, potential of rebars, in spite of the cover thicknesses, drops at early ages of exposure, but recovers to more positive potential at later stages. The peculiar phenomenon in the potential change with time on rebars in concrete using GGBS is due to the fact that the oxidation of iron including in GGBS temporarily results in oxygen deficiency in concrete and then the potential changes to negative direction. However, this may not show the actual corrosion initiation of rebars in concrete. On rebars with 3cm of cover thickness in the GGBS concrete, large fluctuation in potential is observed although potential of few rebars have very low values. As the reason is not yet been confirmed, more attention on future trend of potential and corrosion determination is necessary.

In case of FA, even though a few rebars show a tendency of negative in potential, almost all rebars have relatively positive values and the rebars are forecast to be in good condition.



**Figure 9. Electric potential of rebars at 2cm cover thickness of concrete**



**Figure 10. Electric potential of rebar at 3cm cover thickness of concrete**

**Probability of Corrosion Initiation with Exposure Time.** Figure 11 shows the cumulative probability of corrosion initiation for rebar at both cover thicknesses. The data is obtained based on the measurement results of the natural potential of which value changes to more negative than  $-350\text{mV vs. CSE}$  which means the start of corrosion on rebar in all types of concrete. Firstly for rebar at 2cm cover thickness, for OPC concrete, average cumulative probability of 50%, which means 50% of rebar corroded at this stage, is reached at 120 days of exposure periods,. But for CA<sub>2</sub> 5% concrete, average of 50% is reached earliest at 480 days of exposure, and for CA<sub>2</sub> 7% concrete at 400 days. On the other hand, CA<sub>2</sub> 9% have about 20% probability even after an exposure period of 550 days, which means that the corrosion protection ability of concrete is improved by using CA<sub>2</sub>.

For rebar with cover thickness of 3cm, 50% of the rebar corrodes in OPC concrete after 420 days of exposure. However, in case of CA<sub>2</sub> concretes, the probability is under 20% up to 550 days of exposure. CA<sub>2</sub> concrete is confirmed to reduce corrosion probability. In case of BB and FA concretes, even if the exposure period is about 360 days at this time, probability of corrosion initiation has not been observed.

**Corrosion Conditions of Rebar.** Ratio of corrosion area in the rebar surface is shown in Figure 12, where the reinforced concrete specimens are exposed to tidal zone. For OPC concrete, the corrosion area is larger in both rebar having 2cm and 3cm of cover thicknesses.



Especially at 2cm cover thickness the corrosion area ratio of rebars in OPC concrete is 4% and clear corrosion on rebars have been observed. In the specimen replaced by 5% and 7% CA<sub>2</sub>, corrosion area ratio is slightly less than 0.2% in rebar at 2cm, and almost no corrosion has occurred in case of CA<sub>2</sub> 9% at 2cm. As the replacement ratio of CA<sub>2</sub> increases, the incidence of rebar corrosion decrease. Also for BB, only spot corrosion is observed where the corrosion area ratio is under 1%. FA rebars have not shown any corrosion appearance. Regardless of substitution rate of CA<sub>2</sub>, the incidence of corrosion is less in case of CA<sub>2</sub> concretes.

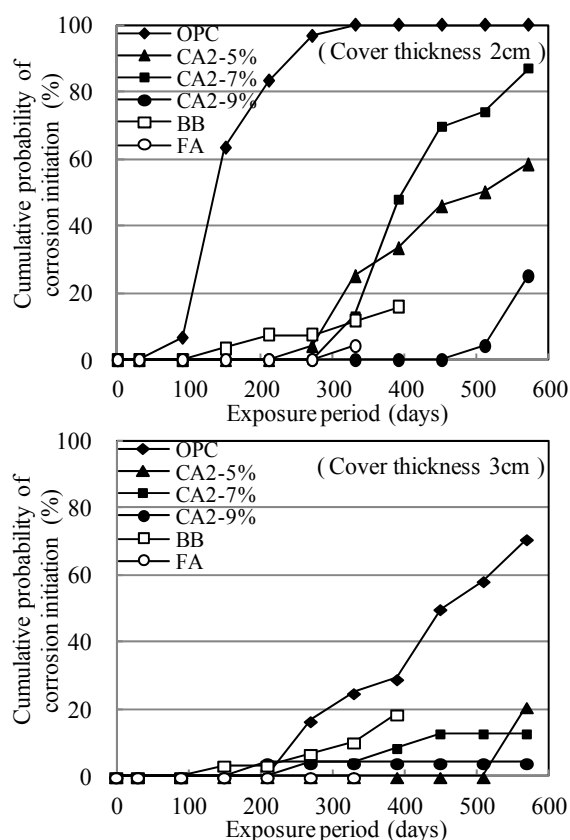
Regarding rebars with 3cm cover thickness, rebars in OPC and BB concretes show only spot rusting. These results are generally consistent with the electric potential measurement ones. It is also shown clearly on the rebars with 3cm cover thickness, incubation period until corrosion initiation on rebar can be prolonged when CA<sub>2</sub> is mixed in concrete.

**Resistance to Carbonation.** Figure 13 shows carbonation depth of each specimen after 280 days of acceleration test. In CA<sub>2</sub> concretes, the carbonation depth is significantly less compared to the ones of BB and FA concrete, and behaves similar to OPC concrete concretes. However, with increasing the replacement level of CA<sub>2</sub>, the carbonation depth increases a little comparing to the ones with OPC concrete. Carbonation depth of CA<sub>2</sub> 9% concrete is 50% of that of BB concrete and 25% of FA one. From the results mentioned above, in concrete mixed with CA<sub>2</sub>, chloride penetration resistance of concrete can be improved without any obstruction on the resistance to carbonation.

## CONCLUSION

In this study, in order to grasp the fundamental characteristics of the concrete mixed with CaO·2Al<sub>2</sub>O<sub>3</sub> fine powder, an experimental study is conducted on strength properties, salt penetration characteristics and carbonation and the following conclusions are obtained;

- (1) Concrete mixed with CaO·2Al<sub>2</sub>O<sub>3</sub>, generally has lesser strength compared to OPC concrete, because of relative reduction of cement. However, the difference tend to be very less when exposed to marine conditions.
- (2) By mixing CaO·2Al<sub>2</sub>O<sub>3</sub> in concrete, the resistance to chloride penetration improves. In addition, replacement up to 9% of CaO·2Al<sub>2</sub>O<sub>3</sub>, is also possible to get good results.



**Figure 11. Cumulative probability of corrosion initiation**

(3) The immobilization of chloride ion is superior with  $\text{CaO}\cdot 2\text{Al}_2\text{O}_3$  replacement in concrete compared to OPC.

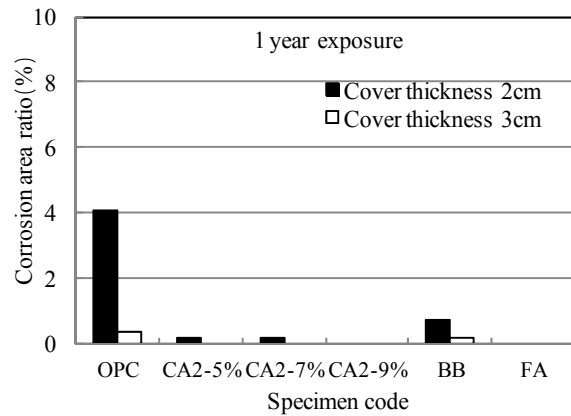
(4) Immobilization of chloride ions and penetration resistance of concrete can be obtained by using  $\text{CaO}\cdot 2\text{Al}_2\text{O}_3$  without being affected by the method of initial curing.

(5) Compared to concrete using fly ash and GGBS, concrete with  $\text{CaO}\cdot 2\text{Al}_2\text{O}_3$  has higher neutralization resistance and its performance is same as the ordinary cement concrete.

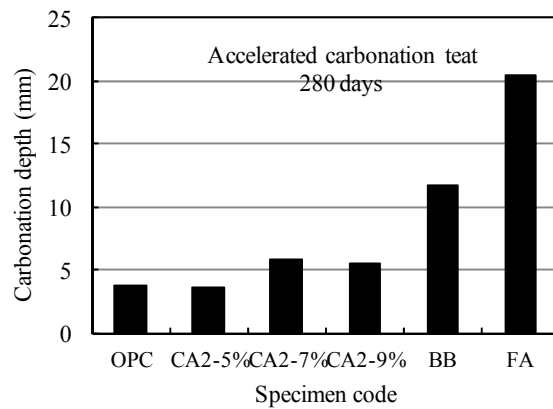
(5) When 5 to 9 % of cement is replaced with fine powder of  $\text{CaO}\cdot 2\text{Al}_2\text{O}_3$  in concrete, both the incubation period until rebar corrosion starts and the propagation period which is decided by rebar corrosion rate during the life of marine concrete structure can be extended in the reasonable manner.

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**Figure 12. Corrosion area ratio**



**Figure 13. Carbonation depth**