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Deterioration characteristics of cement stabilized soils with different fine fraction content under seawater environment

R. Ishikura[1], N. Yasufuku[2], A. Alowaisy[3], T. Kono[4], and T. Fujisawa[5]

[1]Associate Professor, Kyushu University, Fukuoka, Japan, email: ishikura@civil.kyushu-u.ac.jp

[2] Professor, Kyushu University, Fukuoka, Japan, email: yasufuku@civil.kyushu-u.ac.jp

[3]Assistant Professor, Kyushu University, Fukuoka, Japan, email: a.adel@civil.kyushu-u.ac.jp

[4]Senior Chief Researcher, Takenaka Corporation, Inzai, Japan, email: koono.takao@takenaka.co.jp

[5]Researcher, Nippon Steel Blast Furnace Slag Cement Co.,Ltd, Kitakyushu, Japan, email: t-fujisawa@kourocement.co.jp

ABSTRACT

Soft ground is widely distributed in the coastal areas of Japan, and the cement stabilization method is commonly used as a countermeasure to the associated problems. However, recently, it was reported that cement stabilized soil might deteriorate when exposed to seawater for an extended time in such regions. The deterioration mechanism of cement stabilized soil is still not well understood, and a general method for predicting the deterioration progress is lacking. In this study, the influence of the fine fraction content of the cement stabilized soils' base material on the seawater resistance was investigated, and the deterioration characteristics when exposed to seawater were discussed. The results of chemical analysis by the µXRF test showed that the leaching of Ca, penetration of Mg, and penetration of sulfate (S) increased with depth for the specimens with higher fine fraction. Finally, the penetration of sulfate (S) in seawater was confirmed as one of the causes of the strength deterioration of cement stabilized soil. Furthermore, the distribution of S concentration through the specimens was expressed using the diffusion equation, assuming that the penetration of S into the specimens proceeds in the same way as the diffusion of material with concentration. The predicted deterioration depths were compared with the results obtained in the laboratory.

Keywords: Deterioration, sulfate, cement stabilized soils, blast furnace slag, fine fraction content

1 INTRODUCTION

Soft ground is widely distributed in coastal areas of Japan. So far, ground improvement technologies using cement stabilization, such as the deep mixing method, have been commonly applied in soft ground. However, several decades have passed since the construction, and studying the long-term durability is necessary. Recently, it has been reported that cement stabilized soils deteriorate under long-term exposure to seawater (Miao et al., 2012; Ishikura et al.,2014; Hara et al.,2014; Van, N. P. et al.,2017). Since the sea level may rise due to climate change, it is crucial to evaluate the deterioration characteristics of cement stabilized soils when exposed to seawater. However, the deterioration mechanism of cement stabilized soil is still in the research stage, and a reliable method for predicting the deterioration progression is lacking. The deterioration characteristics due to contact with sea water of cement stabilized soil mixed with 70 kg/m^3 cement with different slag replacement ratios and cured in artificial seawater for around 7 months were investigated (Ishikura et al., 2020). It was reported that the larger the slag replacement ratio (BA=25%, BB=45%, BC=65%, BD=85%), the lower the CO_2 emissions during cement production and the more environmentally friendly the cement becomes. Needle penetration tests and X-ray fluorescence analysis (hereafter referred to as µXRF tests) were conducted on the cured specimens to elaborate on the strength and elemental mapping with distance from the seawater contact surface (Figure1).

Figure 1(a) shows the strength properties of specimens made with different types of cement. µXRF test results (Figure 1(b)) are shown for specimens made with BD cement, which has a higher slag replacement ratio. Compared to the strength properties, the values of the needle penetration gradient are larger in the sound zone for specimens made with cement that contains a higher slag ratio. However, the extent of the decrease in needle penetration gradient near the seawater contact surface is greater for specimens with larger slag ratios.

From the µXRF test results of the BD cement specimens, the Ca concentration is smaller, while the Mg concentration is larger in the areas where the strength decreased, as illustrated in the depth profiles. This can be associated with the ion exchange reaction due to Ca leaching and Mg intrusion, as shown in Equations (1) and (2).

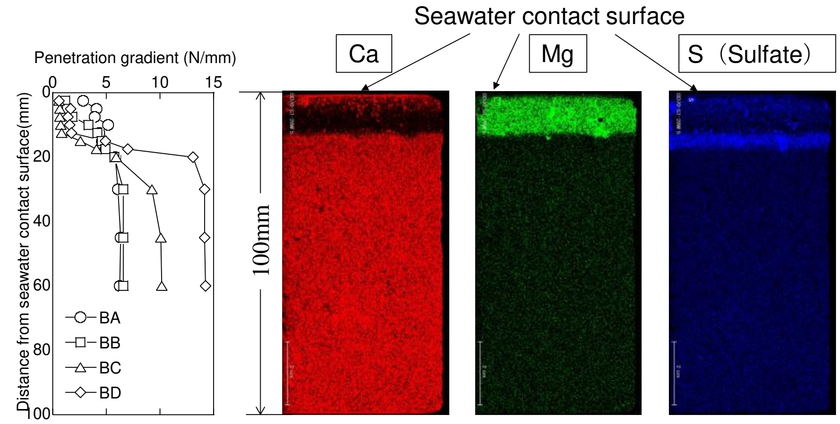
$$\mathrm{MgCl_2 + Ca(OH)_2 \rightarrow Mg(OH)_2 + CaCl_2} \tag{1}$$

$$\mathrm{MgSO_4 + Ca(OH)_2 \rightarrow Mg(OH)_2 + CaSO_4 \cdot 2H_2O} \tag{2}$$

Furthermore, the Sulfate (*S*) concentration increases in the transition zone of strength reduction, and the formation of ettringite ($3CaO \cdot Al2O3 \cdot 3CaSO4 \cdot 32H2O$) due to the *S* intrusion may also be responsible for the strength reduction.

The effect of sulfate on the strength of marine clays (Rajasekaran, 2005) has also been studied, and the intrusion characteristics of S due to seawater contact should also be considered. Most previous studies on the deterioration characteristics of cement stabilized soils exposed to seawater focused on improved soils solidified from base materials consisting mainly of cohesive soils. However, it is important to understand the influence of different base materials on the deterioration of cement stabilized soil, since there are various types of base materials in actual situations.

In this study, the influence of the fine fraction content of the base material on the seawater resistance of cement stabilized soil was investigated. Cement stabilized soil samples mixed at different mass ratios with Toyoura sand and kaolin clay were tested, where the deterioration characteristics when exposed to artificial seawater were studied. Since the penetration of sulfate in seawater is considered to be one of the causes of strength deterioration, it is assumed that the penetration of *S* is analogous to the diffusion of material with concentration, and the S concentration distribution can be expressed using the diffusion equation to predict the progression of deterioration.



(a) Needle penetration gradient

(b) Comparison of elemental mapping with depth in specimen cross section (BD cement)

Figure 1. *Comparison of strength properties of cement stabilized soils and mapping of various element concentrations (BD cement)*

2 EXPERIMENTAL OUTLINE

2.1 Specimen preparation and curing

This section describes the preparation and curing methods of the specimens used in this study. Toyoura sand and kaolin clay were adopted as the base materials. The grain size distribution curves of the specimens are shown in Figure 2. The fine fraction content F_c is 0% for Toyoura sand and 100% for Kaolin clay. The base materials were mixed at various mass ratios to vary the fine fraction content. Table 1 shows the preparation conditions of the specimens. The sandy soil-based and clay-based specimens were prepared separately, with a water content of 18% and 80%, respectively. Blast furnace cement Class B (BB Cement) was used, where 150 kg/m^3 was added. The mixed sample was filled into a plastic container (ϕ50mm × h100mm), sealed and left for one day, and then cured in direct contact with artificial seawater for the designated period. Only one end surface of the specimen was in contact with artificial seawater. The main components of the artificial seawater are shown in Table 2. Furthermore, the curing artificial seawater was replaced every 30 days.

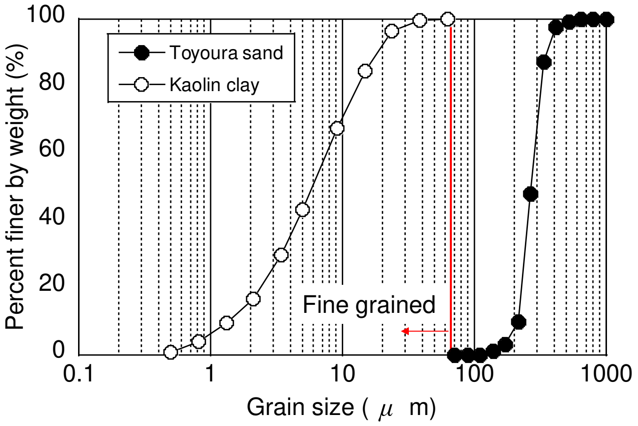


Figure 2. *Grain size distribution of soils*

Table 1. *Specimen preparation conditions*

Base material (Mass ratio) Toyoura sand : Kaolin clay	Toyoura sand base (Water content w = 18%) 100 : 0, 95 : 5, 90 : 10, 85 : 15, 80 : 20
	Kaolin clay base (Water content w = 80%) 20 : 80, 15 : 85, 10 : 90, 5 : 95, 0 : 100
Specimen inch method (mm)	ϕ 50×h100
Cement type	BB
Amount of cement added (kg/m^3)	150
W/C (%)	80
Sealing and curing time (day)	1
Curing environment	Artificial seawater (Aquamarine)
Curing time in seawater (month)	1, 3, 6, 10
Number of specimens under the same conditions	3

Table 2. *Main Components of Artificial Seawater*

Analyzed ion (unit)	Artificial seawater
Ca^{2+} (mg/L)	420
Mg^{2+} (mg/L)	1320
SO_4^{2-} (mg/L)	2770

2.2 Test method

Figure 3 shows the dimensions of the specimens and measuring points for the needle penetration test. The needle penetration test (e.g., Dipova, 2018) was conducted on specimens that were cured for a predetermined period of time. The test involves a needle penetrating vertically from the side surface to measure the penetration gradient (N/mm) at each depth, while the penetration resistance (N) and the penetration distance (mm) are used to determine the strength deterioration of the specimens. A higher value of the needle penetration gradient implies a higher strength. The normalized penetration resistance is defined as the needle penetration gradient at a given depth divided by the maximum needle penetration gradient along the sample. The same experiments were conducted on three specimens under the same conditions, and the average value was used throughout the analysis. The deterioration depth was used to summarize the penetration test results and compare the deterioration degree and progression. As shown in Figure 4, the range where the normalized penetration resistance increases rapidly from the seawater contact surface and the range where it converges are approximated by straight lines. The intersection of the two lines is defined as the experimental value of the deterioration depth obtained by the needle penetration test. It is assumed that the depth at which the strength deterioration initiates coincides with the lower end of the layer at which the S concentration is increasing, which allows determining the depth experimentally.The specimens cured in seawater for 6 months were tested using the µXRF test, and the distribution of each element concentration in the specimens was analyzed.

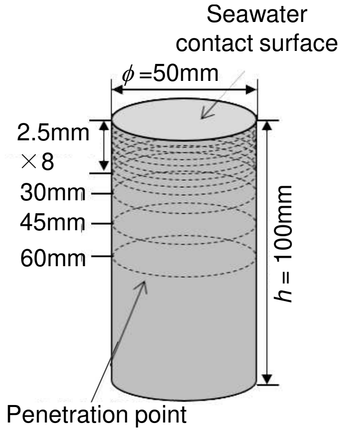


Figure 3. *Measuring points for the needle penetration test*

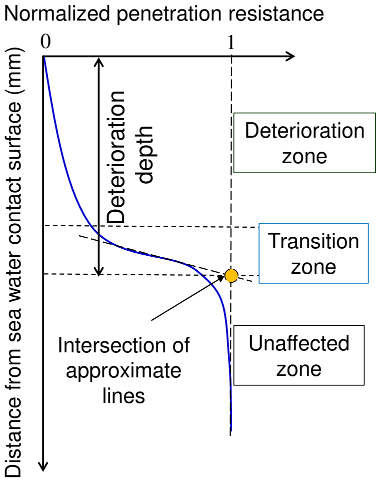


Figure 4. *Definition of deterioration depth from needle penetration test*

2.3 Test results

2.3.1 Deterioration characteristics using the needle penetration test

This section discusses the results of the needle penetration tests conducted on specimens cured in seawater for specific periods. Figure 5 shows the needle penetration gradient and the normalized penetration resistance of a sand-based specimen after 10 months of curing in seawater. Figure 6 illustrates the needle penetration gradient and the normalized penetration resistance of the clay-based specimens cured for 10 months in seawater. Figure 5 shows that among the sand-based specimens, specimen of sand mass ratio 100% showed the highest progress of deterioration. It can be associated with the material separation, referred to as the “breathing” phenomenon, which occurs when the specimens are filled into the plastic container, and the upper part of the specimen does not develop sufficient strength. A comparison of Figures 5 and 6 shows that the sand-based specimens have a greater overall needle penetration gradient, i.e., greater strength, than the clay-based specimens. Figure 7 shows the relationships between fine fraction content F_c and the deterioration depth of the specimens after 10 months of seawater curing. The deterioration depth was determined based on the experimental results using the definition illustrated in Figure 4. The deterioration depth for the specimens with 0% and 5% fine fraction content are remarkably larger due to the effect of breathing. However, excluding those

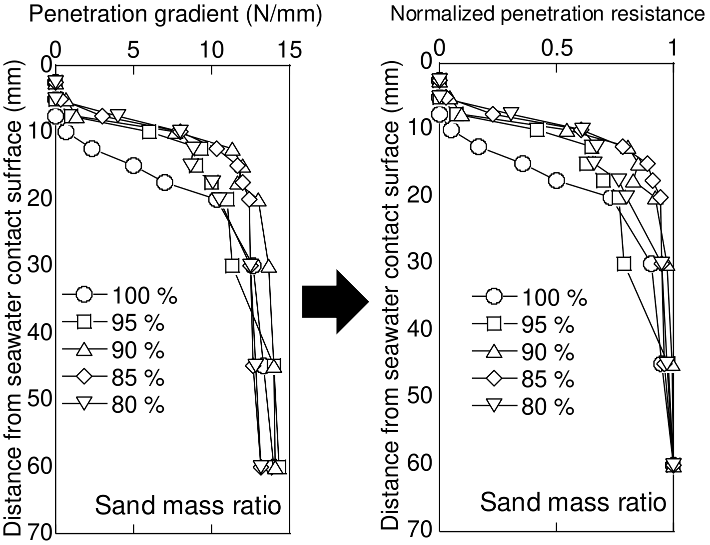


Figure 5. *Needle penetration gradient and normalized penetration resistance of sand-based specimens (10 months of seawater curing)*

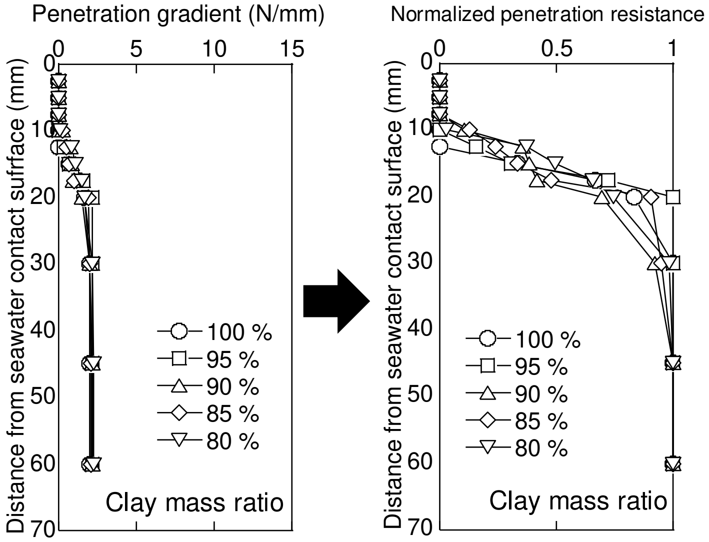


Figure 6. *Needle penetration gradient and normalized penetration resistance of clay-based specimens (10 months of seawater curing)*

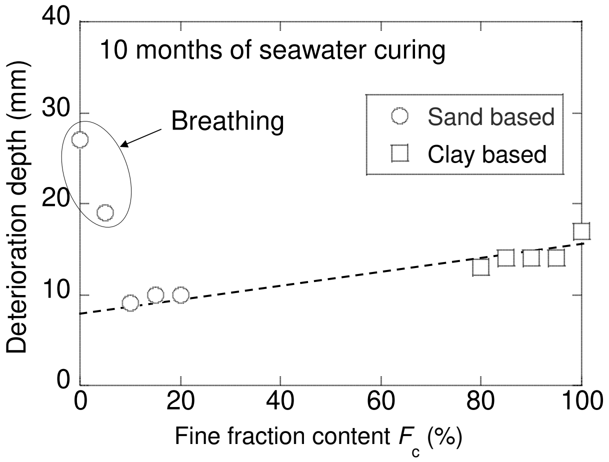


Figure 7. *Relationships between fine fraction content and experimental deterioration depth*

samples, the deterioration depth generally tends to increase for specimens with higher fine fraction content.

2.3.2 Concentration distribution of various elements using the µXRF test

This section describes the chemical analysis results of the µXRF test. Figure 8 shows the results of various elemental mapping of Kaolin clay 80% (Clay mass ratio 80%) and Kaolin clay 100% (Clay mass ratio 100%) specimens that had been cured in seawater for six months. The elemental mapping figures show the distribution of Ca, Mg, and S concentrations, with lighter colours indicating greater elemental concentrations. Figure 9 shows the relative intensity results for the same specimen depicted in Figure 8. The relative intensity is a numerical representation of the elemental concentration mapping results obtained from the µXRF tests. It shows the concentration distribution as a normalized value relative to the maximum concentration measured along the tested sample, where 100% corresponds to the maximum detected elemental concentration. Comparing the two specimens, it can be observed that the specimen with a fine fraction content of F_c = 100% (Clay mass ratio 100%) exhibits higher leaching of Ca in the vertical direction and a greater penetration depth of Mg and S compared to the specimen with a Clay mass ratio of 80%. In addition, compared to Figure 1 reported in a previous study (mapping of element concentrations in the specimen with 70 kg/m^3 cement and cured for about 7 months seawater), the dissolution of Ca and the penetration depth of Mg are both smaller in the specimen prepared with 150 kg/m^3 cement and cured for 6 months in seawater, as shown in Figure 8. Despite the slight difference in the curing period in seawater, it is suggested that the leaching of Ca and the accompanying penetration of Mg may be suppressed by increasing the amount of cement added.

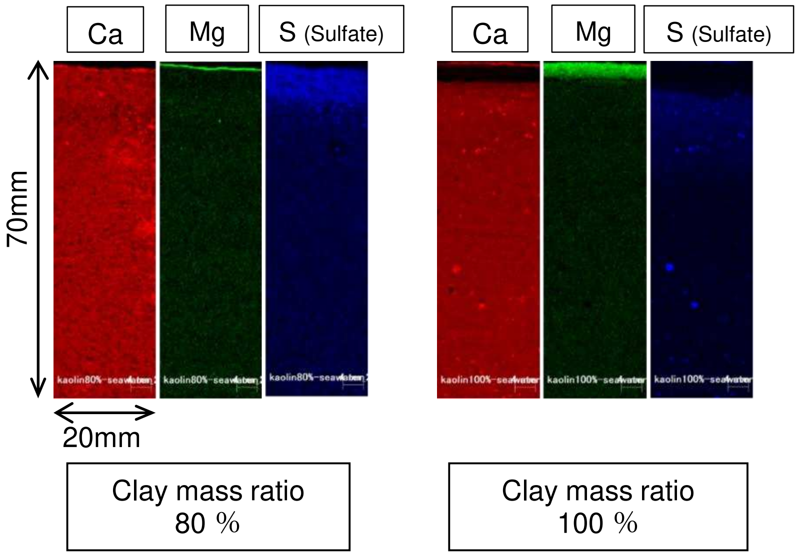


Figure 8. *Mapping results of various element concentrations (6 months of seawater curing)*

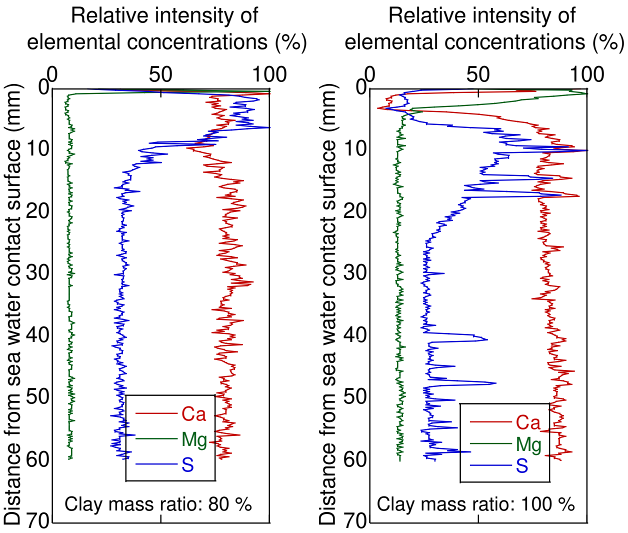


Figure 9. *Relative intensity for various elemental concentrations (6 months seawater curing)*

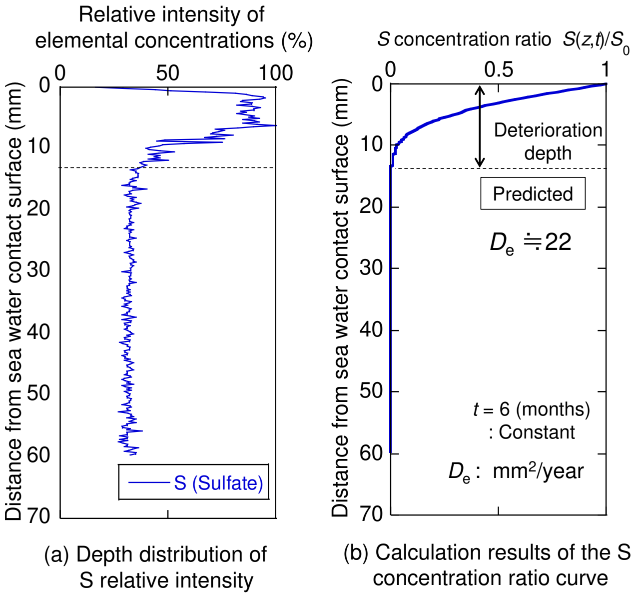
2.4 Diffusion equation for S concentration distribution and prediction of deterioration progression of cement stabilized soil

In this study, the penetration of *S* into the specimen was considered to contribute to the deterioration. In addition, it was assumed that the penetration of S into cement stabilized soil proceeds in the same manner as the diffusion of material concentration. Therefore, it is proposed to express the *S* concentration distribution in cement stabilized soil using Fick's diffusion equation (Equation (3)), which is commonly used to describe the migration of Cl^- ions in concrete (e.g. Zhang et al, 2010).

$$S(z,t) = S_0\left\{1 - erf\left(\frac{z}{2\sqrt{D_e t}}\right)\right\} \tag{3}$$

Where *S*(z,t): *S* concentration (mol/m^3), S_0: surface *S* concentration (mol/m^3), *z*: distance from seawater contact surface (mm), *t*: time (years), D_e: diffusion coefficient of *S* (mm^2/year), erf(x): error function. Figure 10(a) shows the depth distribution of S (sulfate) relative intensity for a specimen with a Clay mass ratio of 80% cured in seawater for 6 months. In this study, the depth at which the *S* concentration in the specimen begins to increase rapidly based on the depth distribution of *S* relative intensity is defined as the deterioration depth based on the µXRF test (chemical analysis).

An example of the *S* concentration ratio (S(z,t)/S_0) curve obtained from Equation (3) is depicted in Figure 10(b). Using Equation (3), the depth at which the curve representing the depth distribution of the S concentration ratio (S(z,t)/S_0) begins to converge to 0 is defined as the predicted depth of deterioration. D_e, the diffusion coefficient of *S*, is varied to match the deterioration depth shown in Figure 10(a) (in this case, *t*=6 months is constant), and D_e is calculated for each specimen by fitting. The calculated D_e is substituted back into Equation (3), and each specimen's predicted deterioration depth is calculated over time. The value of D_e was found to be larger for specimens with more advanced S (sulfate) penetration based on the µXRF test (chemical analysis). The D_e diffusion coefficient of *S* for a specimen with a Clay mass ratio of 80% predicted by the above method was about 22 (mm^2/year).



(a) Depth distribution of S relative intensity

(b) Calculation results of the S concentration ratio curve

Figure 10. *S concentration ratio curve and definition of predicted deterioration depth (Clay mass ratio 80%)*

2.5 Comparison of experimental and predicted deterioration depths

Figure 11 shows an example of a comparison between the experimental deterioration depth of the specimen and the predicted deterioration depth obtained from the µXRF test results after 6 months of curing in seawater, where De was calculated based on the results of the µXRF test. The predicted deterioration depths showed that the deterioration rate decreased with time under both conditions but did not converge to a constant value. Figure 12 compares the experimental and predicted deterioration depths of specimens after 1, 3, 6, and 10 months of curing in seawater. It must be noted that specimens affected by breathing have been excluded. Both experimental and predicted values tended to be larger for the clay-based specimens than for the sand-based specimens. Generally, the predicted results were smaller than the experimental results for the sand-based specimens and larger than the experimental results for the clay-based specimens. Figure13 shows the relationship between the base soil's fine fraction content Fc (%) and the diffusion coefficient of *S*, D_e (mm^2/year), calculated based on the µXRF test results. It can be inferred that the cement stabilized soil containing higher fine fraction in the base soil has a greater rate of *S* penetration into the cement stabilized soil in contact with seawater, consequently resulting in a larger deterioration progression associated with the strength.

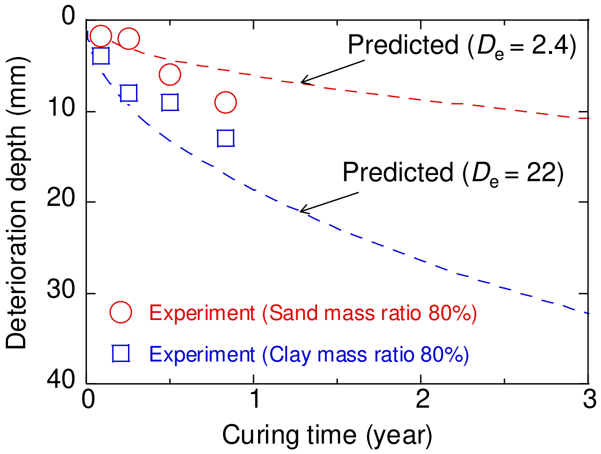


Figure 11. *Comparison of predicted and experimental deterioration progress*

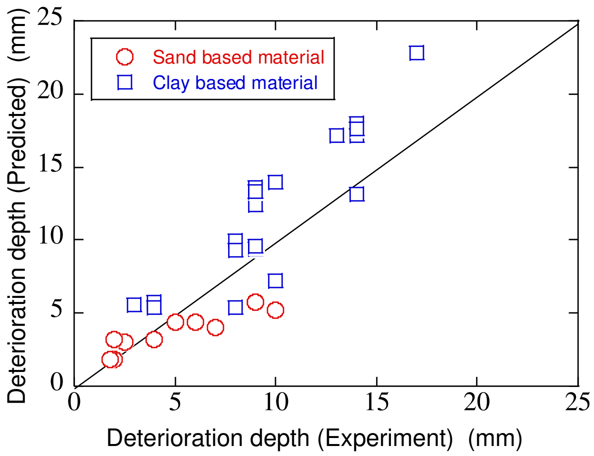


***Figure 12.** Comparison of experimental and predicted deterioration depth*

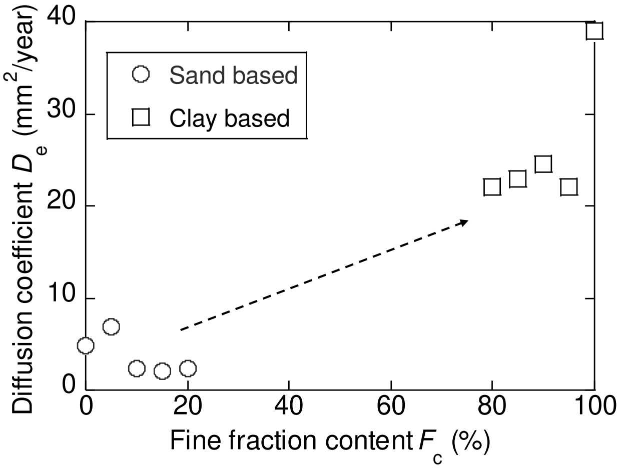


***Figure 13.** Relationships between fine fraction content F_c and Diffusion coefficient D_e*

3 CONCLUSIONS

The effect of exposing cement stabilized soil to a seawater environment on the deterioration characteristics was studied. Specimens with different fine fraction content of base material were prepared, cured in the laboratory for a specified period, and investigated using the needle penetration test and chemical analysis (µXRF test). The main conclusions can be outlined as follows:

1) It was experimentally confirmed that the needle penetration gradient (strength) of sand-based specimens with lower fine fraction content was greater than that of clay-based specimens mixed with the same cement amount. The depth of deterioration of the specimens during the same curing period tended to increase for specimens with higher fine fraction contents.

2) The results of the needle penetration test confirmed that the range of strength loss from the seawater contact surface of the specimens increased with the curing period. The results of the chemical analysis using the µXRF test showed that the *Ca* leaching, *Mg* penetration and *S* penetration depths increased with the curing time in the vertical direction below the seawater contact surface. In particular, it is believed that the penetration of *S* (sulphate) into the specimen influences the strength deterioration.

3) It was assumed that the penetration of S into the specimen proceeds in the same way as material concentration diffusion. Fick's diffusion law was used to predict the distribution of S (sulphate) concentration within the cement stabilized soil, where the predicted changes with time in the depth of deterioration using the diffusion coefficient D_e of *S* was calculated based the µXRF tests. The

predicted values generally agree well with the experimental values. The diffusion coefficient of S, D_e, was found to be larger for specimens with higher fine fraction content.

4 ACKNOWLEDGEMENTS

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