# Assessment of Frost Resistance of Concrete Exposed to Deicing Salts by Using Different Freeze Thaw Test Methods

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## Abstract

Observation and researches reveal that the scaling damage of concrete increase quickly along with the increment of the amount spread of chloride due to deicing salts. However, the regulation for concrete scaling evaluation is still remained unsolved in Japan. In order to provide more basic information for establishing such regulation, the present paper reports a comparison study of three different test methods, which are already standardized in the United States and some European countries. The study shows that the test results measured by the three different test methods are similar. To simplify the test processes and saving test time, the authors modified the test method of *ASTM C 672* (American Society for Testing and Materials standard) by automatic temperature control and using the bottom surfaces of the specimens as the test surfaces instead of the upper ones.

***Keywords***: De-icing salts, Scaling, *ASTM C 666 A*, *ASTM C 672*, *RILEM CDF*

## 1. Introduction

After the law prohibiting the use of studded tires was executed in 1991 in Japan, the amount of de-icing salts spread on slip road has been increased to prevent ice forming to help its thawing ice and snow.[1] Consequently, the spreading of chlorides became one of the most serious reasons causing scaling damage of concrete. But, there is still not a standard for concrete scaling test in Japan, and, of course, to establish economical and suitable standards of scaling test method becomes an urgent topic.

Pioneer researches on the test method of scaling resistance were carried out in the United States and European countries in early 1970s. The *ASTM C 672*[2] for evaluating scaling resistance was standardized in the USA in 1971, and is known as a most common test method for laboratory among various standardized methods to assess the scaling resistance of concrete surface exposed to deicing chemicals. Referring to the *ASTM C 672*, *RILEM CDC2*[3] was developed in France in 1995 and *SS 137244*[4] in Sweden in 1992, and then RILEM (Reunion Internationale des Laboratories d'essays Etde recherches sur les Materiaux et les constructions) promoted an assessment method named by CDF (Capillary suction of Deicing chemicals

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and Freeze thaw test)[5] in 1995.

In order to obtain more information for establishing the Japanese standards of scaling resistance assessments, the authors studied and compared three methods of ASTM C 666 A, ASTM C 672 and RILEM CDF by experiments. Moreover, the authors tried simplification to omit the test procedure, adopting ASTM which is simulating actual freeze-thaw environment. For the test method based on ASTM C 672, the authors demonstrated that the test processes could be simplified and test time could be saved by introducing an automatic temperature controlling system and using the bottom surfaces of the specimens as the test surface. The authors have also investigated the influences of the water cement ratio and types of de-icing salts on the concrete scaling features using modified ASTM C 672, and these results will be reported in detail following sections.

## 2. Experiment

### 2.1 Materials and Mixture Proportions of the concrete

Main materials and mixture proportions of the concrete used for the experiments were ordinary Portland cement with specific gravity of 3.16 g/cm$^3$; the coarse aggregate (crushed stone) of 25 mm in maximum size, 6.90 in fineness modulus and 2.69 g/cm$^3$ in density; the fine aggregate of pit sand with 2.73 in fineness modulus and 2.59 g/cm$^3$ in density. An air entraining agent with the main ingredient of natural resin, was mainly used as the chemical admixtures, and high range water reducing agent was used for some concrete mixtures in test. Table 1 indicates the mixture proportions, and Table 2 the cylinder strength and air void systems of the tested concretes.

### 2.2 Speciment and curing method

Test specimens for each of the three test methods were made and cured according to the three different freeze-thaw standards respectively.

**(1) ASTM C 666 A**

For the test with ASTM C 666 A, fifteen specimens of 100×100×400 mmwere made, cured

Table 1 Mixture proportions in the concrete

| W/C (%) | Target Slump (mm) | Target Air (%) | s/a (%) | Unit weight (kg/m$^3$) | | | | AE agent (C×wt%) | HWR* (C×wt%) |
|---|---|---|---|---|---|---|---|---|---|
| | | | | W | C | S | G | | |
| 55 | 80 | 5.0 | 44.7 | 169 | 307 | 819 | 1,025 | 0.025 | — |
| 55 | 80 | 6.0 | 43.0 | 164 | 298 | 802 | 1,036 | 0.030 | — |
| 45 | | | 41.9 | | 364 | 742 | 1,040 | 0.043 | — |
| 35 | | | 39.9 | | 469 | 671 | 1,022 | 0.067 | 0.75 |

*: High range water reducing agent

Table 2 Cylinder strength and air void characteristics of the concrete

| W/C (%) | Air content (%) | | Compressive Strength 28 days (Mpa) | Spacing factor ($\mu$m) |
|---|---|---|---|---|
| | Fresh | Hardened | | |
| 55 | 5.8 | 5.3 | 27.9 | 239 |
| 55 | 5.6 | 6.2 | 28.6 | 231 |
| 45 | 6.4 | 5.9 | 38.9 | 202 |
| 35 | 6.0 | 6.6 | 51.2 | 172 |

in water of 20°C for 14 days, and then stored in an air surrounding of 20°C and 60% in relative humidity for additional 14 days prior to testing.

**(2) ASTM C 672**

For the test of ASTM C 672, sixty specimens were cast into wood forms of 270×270×75 mm, and all the inside surfaces were coated with urethane resin, and tinplate dike were set up on the upper surface to retain to de-icing salt solution. The specimens were initially cured in water of 20°C for 14 days, and then stored in an air surrounding of 20°C and 60% in relative humidity for additional 14 days prior to testing.

**(3) RILEM CDF**

Nine specimens of 150×150×530 mm were initially cured in water of 20°C for 7 days, and then sliced into 150×150×75 mm. The sliced specimens pieces is shown in Fig. 2. Then, the sliced pieces were stored in air surrounding of 20°C and 60% in relative humidity for 21 days.

### 2.3 Freezing and thawing test methods

The freezing and thawing tests according to the three standards are introduced in following sections respectively. The chloride based solutions used for the tests are shown in Table 3.

**(1) ASTM C 666 A**

The test was started from 28th days after the specimens were moved out from the forms. The specimens were put into a gum container and the spaces between the specimens and the

Table 3 Experiment solutions

| Chloride | Applied solution | | |
|---|---|---|---|
| | Conc. (%) | Cl-conc. (%) | Symbol |
| Ion Exch. Water | — | 0 | IEW |
| NaCl | 3.0 | 1.82 | NaCl |
| $CaCl_2$ | 3.0 | 1.92 | CaCl |
| $MgCl_2$ | 3.0 | 1.05 | MgCl |
| Art. Sea Water | — | 1.98 | ASW |

container were full of chloride solution. The freezing and thawing test was carried out up to 300 cycles of temperature changing between −18°C and +5°C, and the dynamic modulus of elasticity and mass change were measured in every 30 cycle of freezing-thawing.

**(2) ASTM C 672**

The test was started from 28th days after the removal of the formwork. As shown in Fig. 1, the chloride solution was poured on the upper surface of the specimens and the absorption of the solution for 6 hours was made before testing. Then, the specimens controlled a temperature by the move between the 20°C chamber and the −20°C chamber for 50 cycles, which the curve of temperature changing was shown in Fig. 3. For every five cycle, the degree of damage of the test surface was evaluated according to the visual rating designated by ASTM C 672, and the mass of scaled-off particles was measured.

In order to simplify the test processes and saving testing time, the authors introduced an automatic temperature control system, and the scaling characters due to such an automatic temperature changes were investigated. In addition, the bottom surfaces of the specimens were used as the test surfaces, such that the surface finishing procedure as well as the problems of surface treatment can be omitted. The surface temperature of the specimens by the automatic temperature control method was shown in Fig. 3. Based on the automatic method, the influence of the water cement ratio and the types of the deicing salts on scaling damage were examined.

The chloride solution used for the test are sodium chloride solution (NaCl), calcium chloride ($CaCl_2$), calcium chloride (PPL) and calcium magnesium acetate (CMA ; in the mole ratio $Ca(CH_3COO)_2/Mg(CH3COO)_2$ of 1/1 was selected). The concentration of these solutions is 3%. Ion exchange water was used to confirm the mass of scaled-off particles in the case where chloride doesn't exist.

**(3) RILEM CDF (Tentativeness for RILEM in 1994)**

The test was started from 28th days after the removal of formwork. As shown in Fig.2, the specimen surfaces were soaked in the chloride solution for 7 days. According to the freeze-thaw temperature condition shown in Fig. 4, 80 cycles of freezing and thawing were carried out. For every eight cycle, the degree of damage of the test surface was evaluated by measuring of the mass of scaled-off particles.

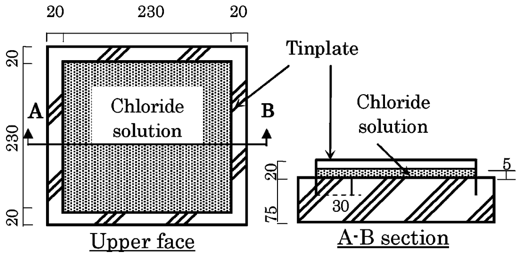

Fig. 1 Test specimen for *ASTM C 672*

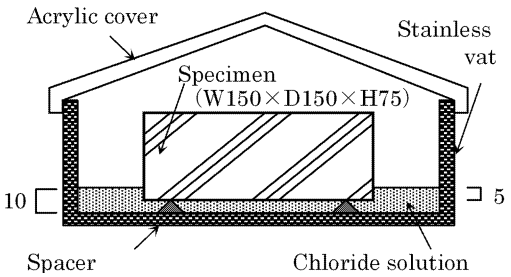

Fig. 2 Test specimen for *RILEM CDF*

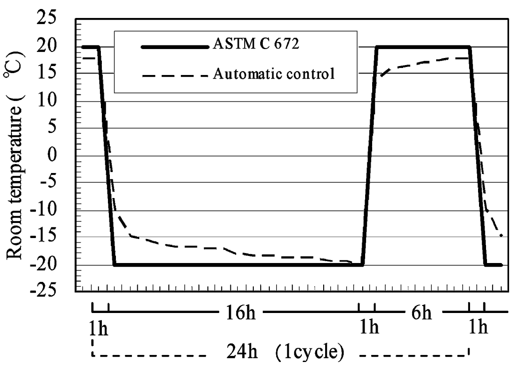

Fig. 3 temperature condition of *ASTM C 672*

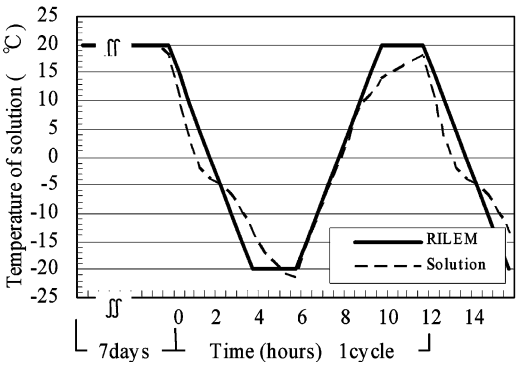

Fig. 4 temperature condition of *RILEM CDF*

## 3. Test results and discussion

### 3.1 The scaling property by the three test methods

### (1) ASTM C 666 A

The relationships of the dynamic modulus of elasticity and the mass change with the freezing-thawing cycles are shown in Fig. 5 and 6, respectively. The decrease of dynamic modulus of elasticity is small, and a little difference in the scaling damage caused by different types of the chloride based solutions. Along with the increasing of the freezing-thawing cycles, the mass changes of the specimens increase, and the difference of the mass reduction due to different types of chloride can be observed exactly. Therefore, the mass change of the specimens can be considered to be available for the evaluating factor for the damage due to concrete scaling. The rank of the mass change due to the types of chloride based solution are presented as: NaCl>$CaCl_2$>ASW>$MgCl_2$>IEW.

### (2) ASTM C 672

Fig. 7 and 8 show the mass of scaled-off particles with respect to 50 freezing-throwing

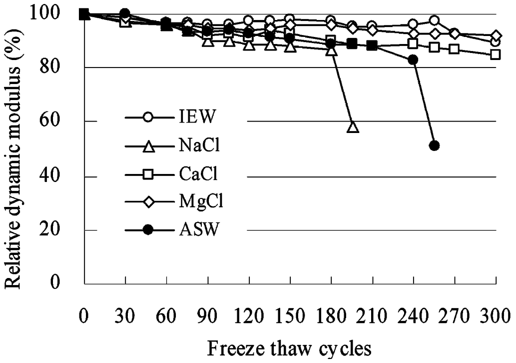

Fig. 5 Relation between relative dynamic modulus and freeze-thaw cycles

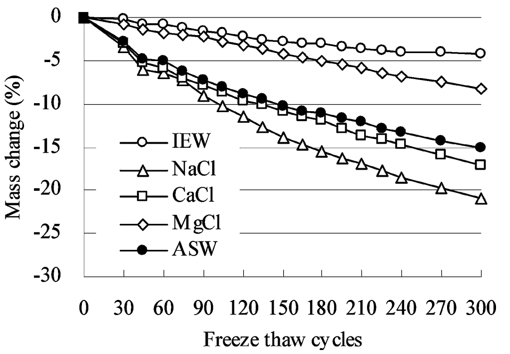

Fig. 6 Relation between weight change and freeze-thaw cycles

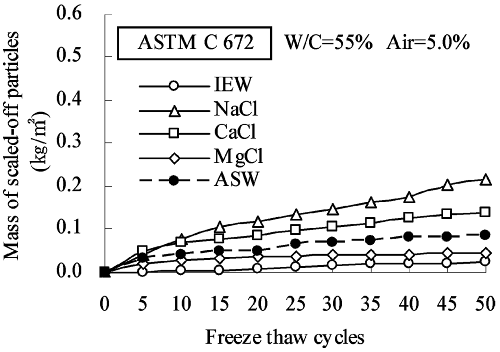

Fig. 7 Relation between Mass of scaled-off particles and freeze-thaw cycles by ASTM C 672

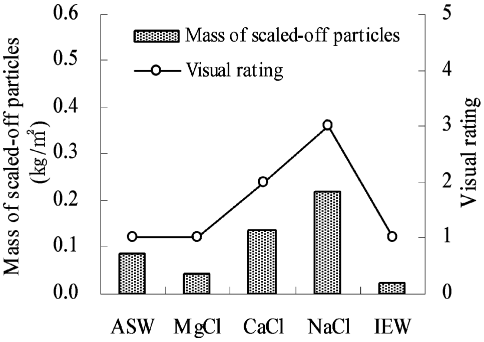

Fig. 8 Visual rating and mass of scaled-off particles after 50 cycles by ASTM C 672

cycles and the visual rating according to the ASTM C 672 respectively. By the visual rating after 50 cycles of freezing thawing, the scaling damage degrees were evaluated with respect to the types of chloride solutions, and the result presented the rank as NaCl : rate 3>$CaCl_2$ : rate 2>ASW : rate 1=$MgCl_2$ : rate 1=IEW : rate 1. When adopting visual rating, the strict evaluation of the scaling damage by chloride solution was difficult. However, the rank of the mass of scaled-off particles due to the types of solutions are presented as : NaCl>$CaCl_2$> ASW>$MgCl_2$>IEW, and this result is as same as that observed by the test based on ASTM C 666 A. Therefore, the method by visual rating based on ASTM C 672, seems a little available to make a quantitative evaluation for the scaling, but the stricter assessment of the scaling resistance will be possible by observing mass of scaled-off particles.

### (3) RILEM CDF

Fig. 9 shows the mass of scaled-off particles versus freezing-thawing cycles, and calcium chloride caused more mass change than sodium chloride, $CaCl_2$>NaCl, which is different from that obtained by the test methods according to ASTM C 666 A and ASTM C 672. Such differences may be probably caused by the scaling from the side surfaces of the specimens. In order to improve the accuracy of the examination, it is necessary to seal up the side surface of the specimens to prevent the side surface scaling.

## 3.2 Comparing the test results

The mass changes and mass of scaled-off particles obtained by ASTM C 666 A, ASTM C 672 and the RILEM CDF were shown in Fig. 10. As for the three test methods, test results showed the same tendency in the scaling damage by the types of chloride solutions.

## 3.3 The simplification of the ASTM C 672

### (1) The automation of the temperature condition

Figs. 11 and 12 show the comparison of characteristics of scaling based on conventional ASTM, which the specimens are moved between two thermostatic chambers with temperature

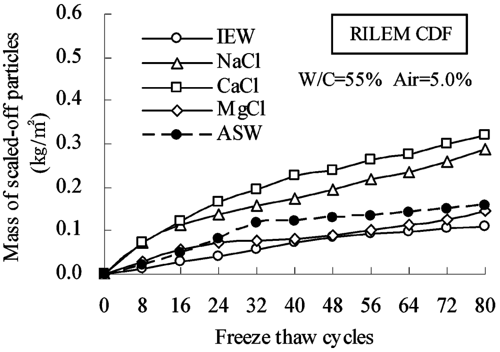

Fig. 9 Relation between Mass of scaled-off particles and freeze-thaw cycles by RILEM CDF

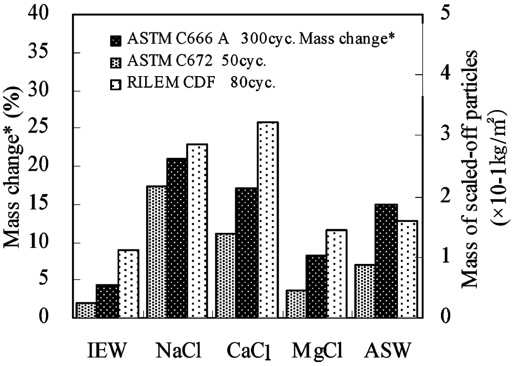

Fig. 10 The tendency of the scaling damage by three kinds of test methods

of +22°C and −21°C respectively, and the modified method by automatic temperature control and using the bottom surface of the specimens. As show in Fig. 3, although the result differences between the conventional ASTM method and modified method, mass of scaled-off particles and mass changes were similar. Therefore, moving the specimens between two thermostatic chambers is not necessary, and the method with automatic temperature control is very efficient.

### (2) Influence of testing surface

As shown in Figs. 11 and 12, the mass of scaled-off particles by using the bottom surface is about 40% of that by using the upper surface. The small scaling for testing the bottom surface were considered to be primarily caused by the heterogeneity due to differences of water cement ratio and air-void system in the vertical direction, which are attributed to the consolidation at the bottom part and the bleeding at the top part. According to the ASTM standard

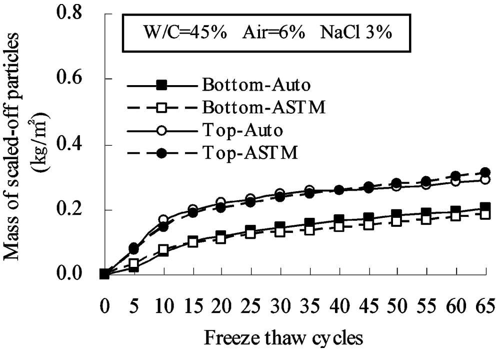

Fig. 11 Mass of scaled-off particles versus number of freeze-thaw cycles for comparing conventional ASTM method and automatic temperature control method

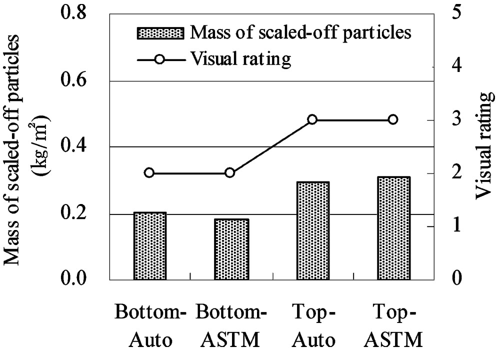

Fig. 12 Visual rating and mass of scaled-off particles after 50 cycles for comparing conventional ASTM method and automatic temperature control method

which the top surface is taken as the test surface, the top surface should be finished correctly. While, the surface treatment procedure can be omitted if the bottom surface of the specimen is taken as the test surface.

### 3.4 The scaling property by the simplification of the ASTM C 672

#### (1) Influence of water-cement ratio

Figs. 13 and 14 show the mass of scaled-off particles due to the water cement ratio, which are the results obtained by using of the bottom surface of the specimens. The rank of scaling damage due to the water cement ratio was presented as: W/C 55%>W/C 45%>W/C 35%, and it was confirmed that the scaling resistance increased at the low water cement ratio. Consequently, strong scaling resistance can be obtained when the water cement ratio was equal to or less than 45%.

#### (2) Influence of deicing salts type

Figs. 15 and 16 show the mass of scaled-off particles due to the water cement ratio, which

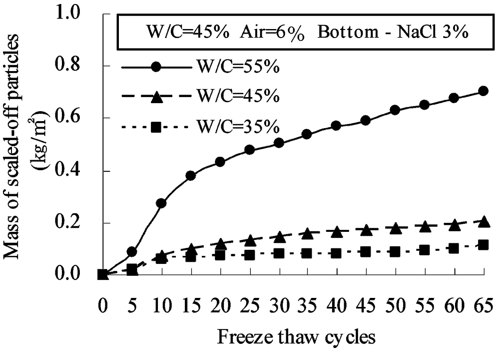

Fig. 13 Mass of scaled-off particles versus number of freeze-thaw cycles for comparing water-cement ratio

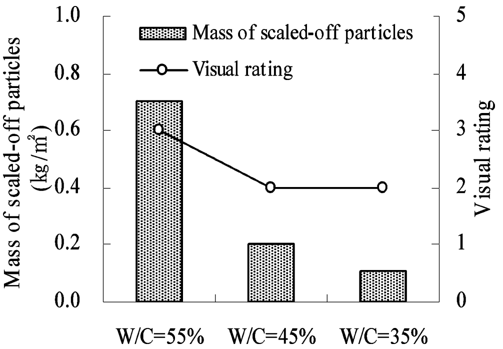

Fig. 14 Visual rating and mass of scaled-off particles after 50 cycles for comparing water-cement ratio

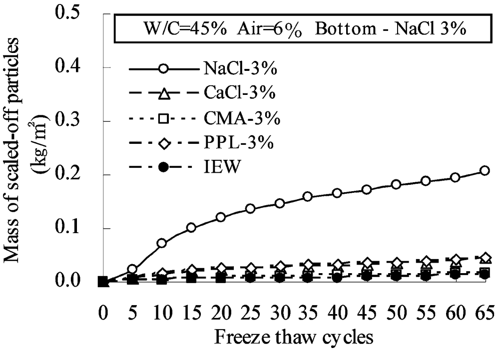

Fig. 15 Mass of scaled-off particles versus number of freeze-thaw cycles for comparing de-icing salt types

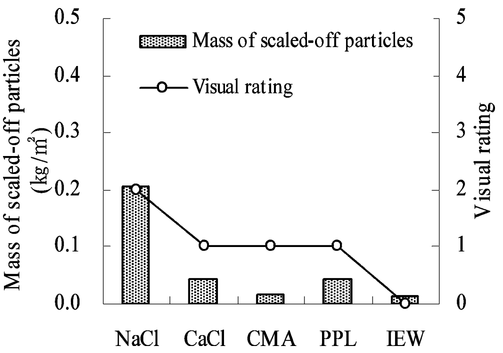

Fig. 16 Visual rating and mass of scaled-off particles after 50 cycles for comparing de-icing salt types

are the results obtained by using of the bottom surface of the specimens. The rank of scaling damage due to the water cement ratio was presented as: W/C 55%>W/C 45%>W/C 35%, and it was confirmed that the scaling resistance increased at the low water cement ratio. Consequently, strong scaling resistance can be obtained when the water cement ratio was equal to or less than 45%.

## 4. Conclusions

(1) As for the test based on ASTMC 666 A, the dynamic modulus of elasticity decreased slightly though large mass changes were observed. Therefore, a mass change will be preferable as a factor evaluate of the scaling resistance of concrete.

(2) As for the test according to the visual rating of ASTM C 672, the evaluation of the degree of the scaling damage is difficult. It will be considered to be affected by the operator's subjectivity and the differences between individual experiences. However, the stricter assessment of the scaling resistance will be judged to be possible in observing mass of scaled-off particles.

(3) The RILEM CDF method can evaluate the scaling quantitatively. In order to improve the accuracy of the examination, the authors proposed to seal up the side surface of the specimen for mass loss from the side surface.

(4) Similar relationships between mass change and mass of scaled-off particles were obtained by test methods according to three different standards, ASTM C 666 A, ASTM C 672 and RILEM CDF.

(5) The mass of scaled-off particles obtained from the automatic temperature control method was almost similar to that of the conventional ASTM C 672. Therefore, transporting the specimens between two thermostatic chambers is not necessary, and the method with automatic temperature control is very efficient and available.

(6) As for the effect of the testing surface of concrete specimen, the greater scaling damage was observed in the top surface of specimen than that in the bottom surface of specimen. The bottom surface of specimen, which can exclude the problems accompanying the finishing operation and the surface treatment as the test surface, can be adopted in case of the relative comparison.

(7) Scaling resistance can be improved when the water cement ratio was equal to or less than 45%.

(8) The scaling damage was increased pronouncedly due to the application of sodium chloride. Therefore, a suitable strategy will be necessary to prevent the application of sodium chloride to concrete structures.

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