- Technical Paper -

# WATER ABSORPTION PROPERTIES OF CONCRETE USING SURFACE PENETRANTS INFLUENCED BY VARIOUS FREEZING AND THAWING CONDITIONS

Qinqing QUAN\*<sup>1</sup>, Osamu SENBU\*<sup>2</sup>, Tomoko FUKUYAMA\*<sup>3</sup> and Dequn Ma\*<sup>4</sup>

# ABSTRACT

In this study, for the purpose of clarifying the influence of surface penetrants on water absorption properties and frost damage deterioration in concrete under the different freezing and thawing conditions, bottom surface water absorption and freeze-thaw test were carried out in which the freezing direction was changed by heat insulation method, the water absorption properties of concrete specimens using surface penetrants were investigated. According to the results, the silane-based penetrants whose water absorption was suppressed in the water absorption test got higher water absorption rates than that of the no coating ones under the condition of upper heat insulation after the freeze-thaw test started, the water absorption got accelerated.

Keywords: concrete, surface penetrants, freezing and thawing, water content, water absorption rate

## 1. INTRODUCTION

In order to prevent frost damage of concrete structures, surface penetrants having water absorption suppressing effect are applied to the concrete surface. The surface penetrants are colorless and transparent and have the feature of protecting the building without changing the appearance of the structures [1].

As representatives of surface penetrants, silane type with the impregnated area get hydrophobized and water absorption can be suppressed [2,3], and silicate type with the surface layer get densified by filling the interspaces and cracks in the concrete with the C-S-H gel are widely used. While these surface penetrants are reported to be effective against frost damage [4,5], there are reports that the surface penetrants have large variation in the water absorption suppressing effect, even water absorption accelerated under the freezing and thawing conditions [6], there are also reports that the surface deterioration were more serious in the concrete coated with silane type surface penetrants than the untreated ones under the freezing and thawing conditions [7], the influence of surface penetrants on frost damage deterioration of concrete is unclear.

In freezing and thawing conditions, if the water freezes first, it assumes that the water can be pushed into the unfrozen concrete by freezing pressure and pushing force raised by frozen water. If the concrete freezes first, it assumes that the water can be attracted by frozen concrete by freezing pressure and drawing force raised by unfrozen concrete. It depends on the freezing direction, and the water absorbing properties in these cases are thought to be different [8].

In this study, in order to clarify the influence of the surface penetrants on water absorption properties and frost damage deterioration of concrete under different freezing and thawing conditions, a bottom surfaced water absorption and freeze-thaw test were carried out in which the freezing direction was changed by using heat insulation material to simulate different freezing and thawing conditions, the water absorption properties and deterioration situations of concrete were investigated.

Specimen typesCondition of bottom surface freeze-thaw testExperiment contentsMeasurement itemsSilane type ×2Upper insulationI : bottom surface water absorption test (11 days) (20 · 60%R.H.)• Water absorption test : mass, moisture meterSilicate type ×1Bottom insulationI : bottom surface freeze- thaw test (100 cycles) (-20°C · 2.5h, 20°C · 2.5h)• Water absorption test : mass, moisture meter				
Silane type ×2Upper insulationI : bottom surface water absorption test (11 days) (20 · 60%R.H.)• Water absorption test : mass, moisture meter • Freeze-thaw test : mass, moisture meter, visual appearance observation, ultrasonic wave propagation velocity	Specimen types	Condition of bottom surface freeze-thaw test	Experiment contents	Measurement items
	Silane type ×2 Silicate type ×1 No coating type ×1	Upper insulation Bottom insulation No insulation	I : bottom surface water absorption test (11 days) (20 $\cdot$ 60%R.H.) II : bottom surface freeze- thaw test (100 cycles) (-20°C $\cdot$ 2.5h, 20°C $\cdot$ 2.5h)	<ul> <li>Water absorption test : mass, moisture meter</li> <li>Freeze-thaw test : mass, moisture meter, visual appearance observation, ultrasonic wave propagation velocity</li> </ul>

Table 1 Experimental plan

<sup>\*1</sup> Graduate School of Engineering, Hokkaido University, JCI Student Member

<sup>\*2</sup> Prof., Faculty of Engineering, Hokkaido University, Dr.E., JCI Member

<sup>\*3</sup> Assistant Prof., Faculty of Engineering, Hokkaido University, Dr.E., JCI Member

<sup>\*4</sup> Graduate School of Engineering, Hokkaido University, JCI Student Member

# 2. OUTLINE OF EXPERIMENT

### 2.1 Experimental plan

The experimental plan is shown in Table 1. Concrete test specimens using three types of penetrants and one without coating were used. After the bottom surface water absorption test finished, the bottom surface water absorption freeze-thaw test using three kinds of heat insulation methods as shown in Figure 1 was carried out.

For simulating different freezing and thawing conditions during bottom freeze-thaw test, the freezing direction was changed by using heat insulation methods as showed in Figure 1. It assumes that water can be pushed into the unfrozen specimens as water in the container froze first in the upper insulation, and water can be drawn into the frozen specimens as the upper part of the specimen frozen first in the bottom insulation, the no insulation is a general condition of bottom surface freeze-thaw test.

After 11 days of bottom surface water absorption test, the water content of the concrete specimens got stable, then bottom surface water absorption freeze-thaw test was carried out. The measurement items were the mass (water content), the water content by the moisture meter on the water absorption surface and the top surface, the ultrasonic wave propagation velocity, and the visual appearance observation.

### 2.2 Specimen overview

Table 2 shows the mixture proportion of concrete specimens, and Table 3 shows the outline of surface penetrants. The specimens were cut from prismatic concrete of  $100 \times 100 \times 400$  mm, Cn, Mr and Rd were coated on the water absorption surface (bottom surface) and four side surfaces, the opposite surface (upper surface) was kept exposed state (no treatment). Non was the specimens that keeps exposed condition (no treatment) on all sides.

#### 2.3 Experimental method

Figure 2 shows the flow of the experiment. After 2 weeks of curing in water, the prismatic concrete was cut into test specimens. The specimens were subjected to 4 weeks of curing in air, and penetrants were coated on the water absorption surfaces and the side surfaces, then cured in a thermostatic chamber at  $20^{\circ}$ C · 60%R.H. for 2 weeks. The condition was taken as the starting condition of test.

The bottom surface water absorption freeze-thaw test was one in which freezing and thawing was performed under the condition of absorbing water from bottom surface. Here, degreasing cotton and water were placed in stainless steel containers ( $240 \times 160 \times 30$  mm), the specimens were immersed in water on the wet degreasing cotton and kept the water level about 5mm above the bottom surface.

The 11 days of bottom surface water absorption test was carried out as shown in Fig.1c) under the condition of no insulation, Since it assumed that the upper surface kept drying state during bottom surface water absorption test, after 114 hours of water absorption test, the top of the container was covered with plastic wrap. Then put each container in a program temperature control tank after treated with heat insulation material according to Figure.1, the bottom surface water absorption freeze-thaw test was conducted.

The ambient temperature setting of the program temperature control tank was based on keeping the central ministry between the specimens and degreasing cotton of the specimens in no insulation at the level of  $-20^{\circ}$ C · 2.5h,  $20^{\circ}$ C · 2.5h, and all the specimens in the three conditions of heat insulation were tested in the same environment, 5h/cycle was conducted by about 100 cycles. After completion of the tests, the specimens were dried at 105 °C, and the absolute dry weight was measured.



a)Upper insulation b)Bottom insulation c)No insulation Fig.1 Heat insulation methods during freeze-thaw test

Table 2 Mixture pro	portion of	concrete
---------------------	------------	----------

W/C	s/a	Unit amount (kg/m <sup>3</sup> )			Air content	Slump	
(%)	(%)	W	С	S	G	(%)	(cm)
60	49.8	180	300	952	961	1.3	21.0
*C : cement(Ordinary Portland cement, density : 3.16g/cm <sup>3</sup> ),							
S : land sand (density in saturated surface-dry condition : $2.55$ g/cm <sup>3</sup> ),							

G : rubble (density in saturated surface-dry condition : 2.67/cm<sup>3</sup>)

Table 3 Outline of s	urface penetrants
----------------------	-------------------

Surface penetrants type	Designation in this paper	Amount of use $(kg/m^2)$	Impregnation depth (mm)
Silane	Cn	0.30	5.3
Silane	Mr	0.20	5.5
Silicate	Rd	0.25	-
No coating	Non	-	-

\* Impregnation depth: the silane types were measured according to JSCE-K571-2003 6.2. the silicate type has not been measured.



Fig.2 Flow of the experiment



# 3. RESULTS AND DISCUSSION

3.1 Temperature change of upper and bottom surfaces of specimens

The temperature was measured by thermocouples. The upper surfaces temperature was measured by burying the temperature measurement contacts in the center of the specimens, and the bottom surfaces temperature was measured by putting the temperature measurement contacts on the central ministry between the specimens and degreasing cotton.

Figure 3 shows the temperature change of the upper and bottom surfaces of the specimens in the freeze-thaw test. In the upper insulation, the water froze first, the freezing of the concrete was delayed about 10 minutes, it assumed that water can be pushed into the unfrozen specimens. In the bottom insulation, the concrete froze first, and the freezing of the water was delayed about 30 minutes, it assumed that water can be drawn into the frozen specimens. Also, in no insulation, the freezing of the water was delayed by about 10 minutes. Although freezing of the concrete was completed in a short time, it takes 20 to 50 minutes for the water from the start of freezing to the end of freezing.

The freezing periods of the upper and bottom surfaces in three heat insulation methods kept 2.5h-3h, and the thawing periods kept 2h-2.5h. In upper insulation, the minimum freezing temperature of upper and bottom temperature were about  $-10^{\circ}$ C. In bottom insulation, while the minimum freezing temperature of upper temperature was about  $-10^{\circ}$ C, the minimum bottom temperature was only  $-2^{\circ}$ C. In no insulation, the minimum freezing temperature of upper temperature was only  $-2^{\circ}$ C. In minimum freezing temperature were about  $-15^{\circ}$ C, the minimum freezing temperature was lower than the other conditions.

# 3.2 Appearance observation results in freeze-thaw test

The visual appearance observation result in the bottom surfaced water absorption freeze-thaw test is shown in Figure 4. There was no deterioration observed in the bottom insulation, it seemed to be difficult to absorb water even though frozen concrete contacted with water. In upper insulation and no insulation, the crack occurred at early stage in Rd and Non that showed severe deterioration, the scaling was observed from about 60th to 70th cycle in the silane types.



Fig.4 Visual appearance observation results

The silane type Cn and Mr showed intense scaling in the upper insulation and no insulation, even cracks occurred in the upper insulation. For silane surface penetrants, it showed more severely deteriorated in upper insulation than in no insulation.

In all the test specimens of Rd and Non, crack occurred in upper insulation at 29th cycle and in no insulation at 13th cycle. In addition, these specimens collapsed from 60th to 80th cycle.

# 3.3 Ultrasonic propagation velocity measurement result

Figure 5 shows the change in ultrasonic velocity during the freeze-thaw test. In bottom insulation, no decrease in ultrasonic wave propagation speed was observed, it is considered that no deterioration occurred, and this result corresponded to the visual appearance observation result. With respect to Rd and Non, the ultrasonic wave propagation velocity began to decrease from 29th cycle in upper insulation, and from 13th cycle in no insulation. This corresponded to the occurrence timing of cracks in visual appearance observation.

In the upper insulation, the ultrasonic wave propagation velocity of Cn and Mr were seen to decrease since about 70th cycle. This corresponded to the occurrence timing of scaling and cracks in visual appearance observation. In the upper insulation, it was considered that the water pushing force at the time of freezing was greater than the force that can hold the contact angle of surfaces with silane-based penetrants, then the water was pushed into the concrete and the cracks occurred when freezing.





3.4 Water absorption properties of water absorption test and freeze-thaw test

In the water absorption test and the freeze-thaw

test, the volume water content was determined from the masses by the following formula, and water absorption curves were made.

$$V=m_{a}-m_{w}$$

$$\rho = (m-m_{0})/V *100$$
where,
$$V : \text{volume (cm}^{3})$$

$$m_{a} : \text{mass in air (g)}$$

$$m_{w} : \text{mass in water (g)}$$

$$\rho : \text{volume water content (\%)}$$

$$m : \text{mass in air at the time of measurement (g)}$$

$$m_{0} : \text{absolute dry mass (g)}$$

The curves of the water absorption and freeze-thaw test are shown in Figure 6. As a characteristic of these water absorption curves, the water absorption rates are calculated as shown in Figure 7. Water absorption rates are obtained in four periods: (1) initial water absorption, (2) the water absorption get stable, (3) water absorption at the start of freeze-thaw test and (4) water absorption from the 5th cycle of freeze-thaw test to the time of deterioration. These water absorption rates of each period are shown in Figure 8. The following tendencys are recognized from Figure 6 and Figure 8.

(1) At the beginning of the water absorption test, compared with the no coating specimens, it is obviously that the water absorptions ware suppressed in the specimens coated with surface penetrants. Especially, water absorption was largely suppressed in silane-based penetrants. Although the water absorption suppressing effect of Rd was recognized, the water content was somewhat lower than Non.

(2) After 11days of water absorption test, the water absorption were almost stable, the water content of Cn, Mr still remained low.

(3) When the freeze-thaw test began, the water content of silane-based Cn and Mr showed a tendency of increasing along with the freeze-thaw cycle, however, the water absorption rates and water content still remained lower than Non in any heat insulation methods At the beginning of freeze-thaw test, Rd showed higher water absorption rate than Non in any heat insulation methods, as a result, the water content got higher than Non.

(4) As for the water absorption curve from the 5th cycle of freeze-thaw test to the time of deterioration (as the deterioration was not found in bottom insulation,



Fig.7 Example of calculating water absorption rate at each period

the rates were calculated from 5th cycle to the end of freeze-thaw test), the increasing of water contents of Cn,Mr in upper insulation were remarkable.

In the upper and bottom insulation the water absorption rates of Cn and Mr exceed the values of Non, and it can be said that the water absorptions of Cn and Mr under these conditions were promoted, on the other hand, in no insulation, the water absorption rates of Cn and Mr still remained lower than Non.

As for Rd, the water absorption rate and water content still kept higher than Non after freeze-thaw test began in any heat insulation methods.

Figure 9 shows the relationship between the water content and the water absorption rate at the start of freeze-thaw test. Figure 10 shows the relationship between the water content and the water absorption rate from the5th cycle of freeze-thaw test to the time of deterioration. These figures were made because it was thought that the water content would rapidly increase if the water content was low when the freeze-thaw started, however, there was no clear trend in these relationships. From these figures, it can be seen that the water absorption rate of Rd rapidly increased at the start of the freeze-thaw test and for Cn and Mr, it got higher than that of Non after 5th cycle in the upper insulation.

### 3.5 Moisture meter measurement result

Figure 11 shows the water content (moisture meter measurement value) of the water absorption surface (bottom surface) and the upper surface of the specimens in the freeze-thaw test.

Before the start of freeze-thaw test .The water content of the water absorption surface and the upper surface in Cn and Mr were lower than Non. In the case of Rd, the water content of the bottom surface was equivalent to Non, some of the values on the upper surface were small, which seemed that the water absorption was suppressed.

After freeze-thaw test started, the water content of the upper surface which remained low in Rd rose sharply, it was considered that the water absorption was promoted in freeze-thaw test.



Fig.8 Water absorption rates of each period



Fig.9 Relationship between water absorption rate and water content at the start of freeze-thaw test



Fig.10 Relationship between water absorption rate and water content from the 5th cycle of freeze-thaw test to the time of deterioration

In Cn and Mr, the water content showed a tendency to rise after freeze-thaw test started, and this tendency was remarkable especially in upper insulation. It seemed that in upper insulation the water pushing force at the time of freezing was greater than the force which can hold the contact angle of surfaces with silane penetrants, so the water content rose when freezing. In addition, some of the rising trends of the water content are larger on the upper surface, and it is conceivable that water is pushed into the upper part at a state where the water content of the impregnation layer is low.



### 4. CONCLUSIONS

In order to clarify effective of surface penetrants against frost damage, the water absorption properties and deterioration situations of concrete coated with surface penetrants were studied under the different freezing and thawing conditions, the following result have been obtained.

- (1) During freeze-thaw process, for those using silane penetrants, the surface deterioration was suppressed compared with the no coating ones in any heat insulation methods, the effect against frost damage could be seen. For those using silicate penetrants, the surface deterioration was almost the same with the no coating ones in any heat insulation methods, the effect of frost damage suppression could not be seen.
- (2) For the specimens using silicate penetrants, although the water absorption suppressing effect coud be found in the water absorption test, but in freeze-thaw test, the water content suddenly increased, it became water absorption promoted.
- (3) In no insulation, the water absorption rates of the silane penetrants were suppressed compared with the no coating ones in the freeze-thawing test, but in the upper insulation, the water absorptions rates exceeded that of the no coating ones, the water absorption became accelerated.
- (4) In bottom insulation, no deterioration and significant change in water absorption can be found, for the minimum freezing temperature of bottom temperature was only -2°C.

#### REFERENCES

[1] Tittarelli F, "The effect of silance-based hydrophobic admixture on corrosion of reinforcing steel in concrete," Cement and Research, Vol. 38, No.1, 2008, pp1354-1357.

- [2] Y G ZHU, "Influence of silance-based water repellent on the durability of recycled aggregate concrete," Cement & Concrete Composite, Vol. 35, Issue 1, 2013, pp32-38.
- [3] Hager, R, "Silicones for concrete protection," Proceedings of the International Conference held at the University of Dundee, Scotland, 1996, pp361-367.
- [4] Hirotake ENDOH, "Effect of protection of concrete structure in cold region by penetrating sealers (type of silane)," Proceedings of the Japan Concrete Institute, Vol.28, No.1, 2006, pp2081-2086.
- [5] Hirotake ENDOH, "Basic evaluation on scaling durability of concrete with surface penetrate material (silicate type)," Proceedings of the Japan Concrete Institute, Vol.29, No.1, 2007, pp1203-1208.
- [6] Kazuaki SUGIURA, "Influence of coating and penetrants on the preventive effect against water absorption and the moisture distribution in applied concrete during freezing and thawing test under various lowest temperature," Proceeding of AIJ Hokkaido Architectural Research Conference, No.89, 2016, pp17-20.
- [7] Masato TERAZAWA, "Experimental study on durability of surface penetrants used in cold region," Proceedings of the Japan Concrete Institute, Vol.29, No.2, 2007, pp553-558.
- [8] Seisei ZEN, "Water absorption properties of concrete coated with surface penetrants influenced by various freezing and thawing conditions," Proceeding of AIJ Hokkaido Architectural Research Conference, No.95, 2017, pp381-384.