Effect of application timing of silicate-based surface penetrants on the mass transport properties of concrete

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ABSTRACT

This paper presents an experimental study examining the effect of the application of silicate-based surface penetrants. The penetrants were applied to either early-age specimens or aged specimens containing micro-cracks, and the mass transport properties (durability) were evaluated by the carbonation rate and water absorption. The results showed that in the case of early age materials, application of the sodium-type silicate at 14 days accelerated the hydration process more than application at 7 days. This resulted in higher quality of the concrete surface and improved durability against water and carbonation. After applying silicate-based surface penetrants to aged concrete with micro-cracks, the micro-cracks were observed to close and the durability was thus improved. From these results, it can be shown that silicate-based surface penetrants can improve the durability of both early-age concrete as well as aged concrete containing micro-cracks.

Keywords. silicate-based surface penetrants, water absorption, carbonation, surface air permeability

INTRODUCTION

When the performance of a concrete structure is less than required performance due to aging and deterioration, the performance needs to be restored through measures such as repair or reinforcement. Surface penetrants can improve the durability of concrete and delay the degradation of the structure. In addition, when applying penetrants at an early age for preventive maintenance, the progress of deterioration can be reduced from the beginning (JSCE, 2005). silicate-based surface penetrants are one type of surface protection construction methods, and their application to the concrete surface can improve the durability of the concrete. The application of silicate-based surface penetrants has several
benefits such ease of maintenance and the ability to visually check the surface after application.

In past studies, it was found that the effect of silicate-based surface penetrants varies depending on the quality of the concrete beginning (Hirotake, 2010, Hirotaka, 2010), and it was also shown that the penetrants could improve the durability of concrete containing micro-cracks and protect against carbonation. However, it is believed that the state of the concrete to which the penetrant will be applied is important, and the effect of application timing on the mass transport properties of concrete is unclear.

This paper presents an experimental study examining the effect of penetrants which were applied to early-age specimens and aged specimens containing micro-cracks. The study on early-age specimens examined the influence on hydration reaction and the difference due to application timing. The study on aged specimens examined the durability recovery in the presence of the micro-cracks.

**MEASUREMENT**

**Experiments on early-age materials**

To understand the effect of application timing on the durability, penetrants were applied to specimens aged 7 and 14 days, after which the specimens were placed in a controlled environment (20°C, R.H. 60%) for 4 weeks. Mass transport properties (durability) were then evaluated by the carbonation rate and water absorption. In addition, a two-chamber vacuum cell was also utilized to evaluate the changes in the concrete outer layer department over time.

**Experiments on aged concrete with micro-cracks**

In this series of experiments, all specimens were demolded 24 hours after casting and then were cured under standard conditions for 56 days. After this initial curing, micro-cracks (0.03mm - 0.08mm) were introduced into the specimens by three cycles of wet-dry exposure which consisted of water submersion for 3 days followed by then 80°C drying for 4 days per one cycle. Penetrants were then applied to the surface of the specimens with micro-cracks. After application, specimens were cured under dry conditions for 14 days, after which the mass transport properties (durability) were evaluated by the carbonation rate and water absorption (similar to the early-age specimens). The flow for the experiments on early-age specimens and aged specimens is shown in Figure 1.
Material and Specimens

Table 1 show the two types of silicate-based surface penetrants. The application method was carried out depending on the penetrant quality. Concrete was made with ordinary portland cement (density: 3.15 g/cm³, Blaine fineness: 3440 cm²/g) or ordinary portland cement incorporating fly ash (density: 2.28 g/cm³, Blaine fineness: 3920 cm²/g, JIS A 6201 II grade ash). The fine aggregate was river sand (density: 2.61 g/cm³, fineness modulus: 2.91) while the coarse aggregate was crushed sandstone with Gmax of 20 mm (density: 2.73 g/cm³, fineness modulus: 6.61). The mix proportions are shown in Table 2. The slump of fresh concrete was 8±2.5 cm and air content was 4.5±1.5%. The water-cement ratio was 0.55. The water absorption test was carried out on an area of 15×15×15 cm, and the carbonation rate test carried out on an area of 15×15×10 cm. Specimens were given a surface treatment to limit the mass transport surfaces. Table 3 shows the number of application sides and measurement area of the specimens.

Table 1. Silicate-based surface penetrants types

<table>
<thead>
<tr>
<th>Ingredient</th>
<th>Application quantity (g/m²)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Na Sodium silicates</td>
<td>150</td>
</tr>
<tr>
<td>Li lithium silicate</td>
<td>120</td>
</tr>
</tbody>
</table>

Table 2. Concrete mix mixture proportions

<table>
<thead>
<tr>
<th>Code</th>
<th>W/C (%)</th>
<th>s/a (%)</th>
<th>W (kg/m³)</th>
<th>C (kg/m³)</th>
<th>F (kg/m³)</th>
<th>S (kg/m³)</th>
<th>G (kg/m³)</th>
<th>AE</th>
</tr>
</thead>
<tbody>
<tr>
<td>N</td>
<td>55</td>
<td>45</td>
<td>804</td>
<td>1016</td>
<td></td>
<td></td>
<td></td>
<td>C×0.02%</td>
</tr>
<tr>
<td>FA</td>
<td>55</td>
<td>46</td>
<td>698</td>
<td>1001</td>
<td>107</td>
<td></td>
<td></td>
<td>(C+F)×0.05%</td>
</tr>
</tbody>
</table>
Table 3. Number of application side measurement area of specimens

<table>
<thead>
<tr>
<th>Measurements (cm)</th>
<th>Application side</th>
</tr>
</thead>
<tbody>
<tr>
<td>Test for water absorption</td>
<td>15×15×15</td>
</tr>
<tr>
<td>Test for carbonation</td>
<td>15×15×10</td>
</tr>
</tbody>
</table>

**Test methods**

**Water absorption test**

The water absorption test was carried out according to JSCE-K 572-2012. The specimen was removed from the testing container seven days after the start of the test and the mass was measured (Wai) after removing the surface water. Using this value the water absorption rate (Wa) was calculated using equation (1). The reported absorption rate is the mean of three values.

\[
Wa = \frac{Wai - Wa_0}{Wa_0} \times 100
\]

where, \(Wa\) : absorption(\%), \(Wa_0\) : mass before the examination start(g), \(Wai\) : mass at the time of the measurement(g)

**Carbonation test**

The carbonation test was carried out according to JSCE-K 572-2012. The examination condition used a temperature of 20\(^\circ\)C, R.H. 60%, 5% CO\(_2\) density, and the carbonation depth was measured after 7 and 28 days. The carbonation depth was measured by applying phenolphthalein on the fracture surface, and the reported value was calculated as the mean of six data points, which were measured twice per specimen (the depth from two opposing sides) and from three specimens.

**Air permeability test**

The coefficient of air permeability was measured on the concrete cover side of the concrete specimen using the Torrent method for evaluating air permeability of concrete (Torrent, 1992). Before conducting the test, all specimens were dried to avoid the influence of water content of specimens on the test results.

**Degree of hydration test**

Degree of hydration was evaluated following the manual of the Japan Concrete Institute using the "degree of hydration test" (JCI, 2005). Specimens were prepared from mortar with a water-cement ratio of 0.55. Penetrants were applied to specimens at 3, 7 and 10 days, after which the specimens were placed in a controlled environment (20\(^\circ\)C, R.H. 60%) until 28 days. The degree of hydration was determined 28 days after casting.
Ma=((Mi-M_0)/M_0) \times 100

where, Ma: degree of hydration(\%), Mi: mass after 105^\circ C drying (g), M_0: mass after 1000^\circ C drying (g)

RESULTS AND DISCUSSIONS

Early age materials experiments

Water absorption test

The results showed that, in the case of early age materials, application timing of the penetrant at 7days and 14 days affected the water absorption ratio (calculated as the ratio of the test result to the water absorption rate in the case that no penetrant was applied) as shown in Figure2. Ordinary portland cement specimens experienced improved durability against water absorption, and the absorption rate was lower when the penetrants were applied at 14 days than at 7days.

With regards to the difference between penetrant types, it was found that the water absorption ratio was smaller for the sodium-type silicate than for the lithium-type silicate. Specimens using ordinary portland cement with fly ash also experienced improved durability against water absorption, and the absorption rate was less when penetrants were applied at 7days than when applied at 14days, and there was no difference between the types of penetrants. When comparing OPC specimens with FA specimens, it could be seen that application was more effective at 14 days for OPC specimens but more effect for FA at 7 days.

Degree of hydration test

The results of the hydration test given in Figure3 show that, in the case of sodium-type silicate, the hydration rate is higher at 10 days compared to the standard material since the amount of calcium hydroxide is higher at 10 days compared to 3 and 7days. No application effect could be seen, and the hydration rate was for the lithium-type silicate was at the same level as when no penetrant was applied regardless of application timing. When comparing FA with OPC, it could be seen that the hydration process of OPC was roughly 1.2 times higher than FA. From the water absorption rate and degree of hydration test results, it was thought that application of the sodium-type silicate penetrant at 14 days to the OPC specimen resulted in the most-improved concrete surface.

Air permeability test

The coefficients of air permeability (KT) test results are shown in Figure 4. It was found that OPC and FA follow a similar trend. After the water absorption tests, KT varied from 0.1 to 0.01 \times 10^{-16} \text{ m}^2. It is believed that the degree of hydration increased during the water absorption test, which led to a decrease in KT.
Carbonation test

Test results for the carbonation of concrete (28 days) are shown in Figure 5. Carbonation ratio for both OPC and FA became less than half of the ratio of the standard specimen regardless of application timing or type. The depth of carbonation over time is shown in Figure 6. It can be seen that the carbonation depth at 56 days is not a linear function, and it is possible that the carbonation depth exceeded the reforming depth. The reforming depth \( Y \) of concrete by applying silicate-type penetrants can be calculated using equation (3).

\[
Y = \left( \frac{B_p}{B_c} \right) H
\]  

(3)

where, \( Y \): reforming depth (≤ penetration depth) (mm), \( H \): carbonation depth for no penetrant application (mm), \( B_c \): carbonation rate for no penetrant application (mm/√year), \( B_p \): carbonation rate for penetrant application (mm/√year)

The reforming depth became roughly 2 to 3 mm for OPC and roughly 1 to 2 mm for FA. In the case of sodium-type silicate application, the difference between types of impregnation is seen to be very large. In previous studies, the penetration depth was reported to be around 20 mm from the surface (Takayuki, 2011). Therefore, the reforming depth found in this experiment was lower than the penetration depth in the previous studies.
Aged concrete with micro-cracks experiments

Water absorption test

Results for the water absorption test are shown in Figure 7. No difference between sodium-type and lithium-type silicate penetrants for OPC could be seen. For FA, application of lithium-type silicate reduced the water absorption ratio compared to the sodium-type silicate. Furthermore, the water absorption ratios for OPC and FA were only 80% of that in the standard specimen with no impregnation.

Carbonation test

The carbonation ratio results (at 28 days) are shown in Figure 8. The carbonation ratio of sodium-type silicates for both OPC and FA was half that of the standard specimen. It was thought that OPC specimens with sodium-type penetrants experienced improved durability against water absorption and carbonation. In the case of the FA specimens, calcium hydroxide may have been used up in the pozzolanic reaction, but the carbonation depth of FA specimens with sodium-type and lithium-type silicates was the same as that of OPC specimens.

Finally, the change in the coefficient of air permeability (KT) shown in Figure 9. It was shown that the KT level became smaller after the carbonation test.

Table 4. Reforming depth

<table>
<thead>
<tr>
<th></th>
<th>OPC</th>
<th>FA</th>
</tr>
</thead>
<tbody>
<tr>
<td>Na7</td>
<td>3.2</td>
<td>0.9</td>
</tr>
<tr>
<td>Na14</td>
<td>3.2</td>
<td>1.0</td>
</tr>
<tr>
<td>Li7</td>
<td>1.8</td>
<td>2.0</td>
</tr>
<tr>
<td>Li14</td>
<td>2.2</td>
<td>1.9</td>
</tr>
</tbody>
</table>

Figure 6. Depth of carbonation aging
CONCLUSIONS

The effect of the application timing of silicate-based surface penetrants on the mass transport properties of concrete was experimentally conducted. The penetrants were applied to either early-age specimens or aged specimens containing micro-cracks. The results are summarized as follows.

・In the case of early age materials, when comparing OPC specimens with FA specimens, it could be seen that application was more effective at 14 days for OPC specimens but more effect for FA at 7 days.

・In the case of aged concrete with micro-cracks experiments, the water absorption rates for OPC and FA were only 80% of that in the standard specimen with no impregnation.

・The carbonation rate of sodium-type silicates for both OPC and FA was half that of the standard specimen.

・Silicate-based surface penetrants were found to have a good resistance against carbonation.
REFERENCES


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