ABSTRACT: In recent years, surface penetration has attracted attention as a repair method for concretes structures. Silicate based penetration materials have been used in this method. In this study, visual appearance, water permeability, water absorption, water-vapor permeability, neutralization (carbonation) and chloride ion penetration testing of a silicate based surface penetration material are performed. The protection and waterproofing effects of a silicate based surface penetration material are evaluated based on the results. The silicate based surface penetration material penetrates deeply into the concrete surface, shows self-repair functionality, is effective to the neutralization (the carbonation) of the concrete and improves the performance of waterproofing. The penetration depth of the silicate based surface penetration materials plays an important role in the surface penetration method. Therefore, permeability testing of a mortar specimen, penetration depth testing of the mortar specimens with various silicate based surface penetration materials, viscosity testing of the materials and modelling of the one dimensional penetration into the mortar specimens are performed. The results of viscosity testing and coefficient of permeability suggest that these silicate based surface penetration materials will perform well surface penetration materials.

Keywords: Coefficient of permeability, Potassium silicate, Surface penetration material, Viscosity

1. INTRODUCTION

Recently, maintenance of concrete structures has been widely studied; one technique for performing this maintenance is the surface penetration method, wherein the concrete surface is protected by coating it with surface penetration materials [1]. Surface penetration materials are classified into silane based, silicate based and others. Silane based materials form layers and prevent water from seeping into concrete. Silicate based surface penetration materials fill voids in the surface layers of concrete [2], [3].

Surface penetration materials with low viscosity have recently been developed. However, the properties of such materials remain largely unknown. The main components of silicate based surface penetration materials are sodium silicate and lithium silicate, whereas for the potassium silicate based surface penetration material with low viscosity developed in this study, the main component is potassium silicate [4], [5], [6]. This study aims to clarify the performance of the potassium silicate based surface penetration material with low viscosity.

In this study, visual appearance, water permeability, water absorption, water-vapor permeability, neutralization (carbonation) and chloride ion penetration testing of the potassium silicate based surface penetration material with low viscosity were performed and the results were evaluated.

The penetration depth of silicate based surface penetration materials in concrete is important; the deeper the penetration depth, the greater are the modification effects. Therefore, this paper also focuses on the permeability of a mortar specimen using various silicate based surface penetration materials. Permeability testing on the mortar specimens, penetration depth testing on the mortar specimens with various silicate based surface penetration materials, viscosity testing on the surface penetration materials and modelling on the one dimensional penetration into the mortar specimens were performed. The relationship between the penetration depth at the surface of mortar specimens and viscosity of the silicate based surface penetration materials were evaluated.

2. SILICATE BASED SURFACE PENETRATION MATERIALS

2.1 Types and Characteristics

Silicate based surface penetration materials can be classified as reaction type or solidification type.
type material for silicate based surface penetration materials. This type produces calcium silicate hydrates (CSH) by the reaction of calcium hydroxides in the concrete with silicate in the silicate based surface penetration materials. These effects occur when the reaction type material is in wet condition. Furthermore, when exposed to external water, the reaction type material can restore itself by producing further calcium silicate hydrates (CSH). Thus, the duration over which its effects are exercised is long. Potassium silicate based surface penetration material with low viscosity has been developed to be of the reaction type material.

Figure 2 shows the characteristics of the solidification type material for silicate based surface penetration materials. This solidification type material hardens on the concrete, exercising its effects in the dry condition.

2.2 Chemical Reaction

Equation (1) shows the reaction between potassium silicate (reaction type) and the concrete components, while Eq. (2) shows the reaction between sodium silicate (solidification type) and the concrete components [7].

\[
K_2O \times SiO_2 + xCa(OH)_2 + yH_2O \rightarrow xCaO \times SiO_2 \times zH_2O + 2KOH + (x + y - z - 1)H_2O
\]

(1)

\[
Na_2O \times SiO_2 + xCa(OH)_2 + yH_2O \rightarrow xCaO \times SiO_2 \times zH_2O + 2NaOH + (x + y - z - 1)H_2O
\]

(2)

The performance of the materials varies depending on their main component.

2.3 Silicate Based Surface Penetration Materials with Low Viscosity

Table 1 shows the components of the potassium silicate based surface penetration material with low viscosity. Potassium silicate solution changed viscosity by consistency of potassium silicate. A Potassium silicate solution containing 15 to 20% consistency of potassium silicate obtains low viscosity.

Table 1 Composition of the potassium silicate based surface penetration material with low viscosity

<table>
<thead>
<tr>
<th>Components (chemicals)</th>
<th>Contents (weight %)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Potassium silicate</td>
<td>15 - 20</td>
</tr>
<tr>
<td>Ancillary materials</td>
<td>≤ 1.5</td>
</tr>
<tr>
<td>Purified water</td>
<td>Remainder</td>
</tr>
</tbody>
</table>

Table 2 Composition of the mortar sample

<table>
<thead>
<tr>
<th>Materials</th>
<th>Portland cement</th>
<th>Standard sand</th>
<th>Tap water</th>
</tr>
</thead>
<tbody>
<tr>
<td>Compounded amount (kg/batch)</td>
<td>38.25</td>
<td>114.75</td>
<td>19.125</td>
</tr>
</tbody>
</table>

Table 3 Methods for coating the surface penetration material onto the mortar sample

<table>
<thead>
<tr>
<th>Coating process</th>
<th>Coating count</th>
<th>Coating material</th>
<th>Coating method</th>
<th>Amount coated (kg/m²)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>2</td>
<td>Potassium-silicate-based</td>
<td>Brush coating</td>
<td>Average: 0.012</td>
</tr>
<tr>
<td>2</td>
<td>1</td>
<td>Tap water</td>
<td>Brush coating</td>
<td>Approximately the amount of the coating face is covered with water</td>
</tr>
</tbody>
</table>
composition of the mortar specimen. The water-cement ratio was 50%; the sand-cement ratio was 3.

The mortar specimens used were based on those described in “JSCE-K 571-2010 5.2”. Silicate based surface penetration materials with low viscosity coat to the top surface of the mortar specimens. Table 3 shows the coating method and the amounts of potassium silicate based surface penetration material with low viscosity used.

The coated mortar specimens were produced as described by “JSCE-K571-2010 6” and the visual appearance testing, the water permeability testing, the water absorption testing, the water-vapor permeability testing, the neutralization (carbonation) testing and the chloride ion penetration testing were conducted [8].

3.2 Evaluation of Penetration Depth and Viscosity of Silicate Based Surface Penetration Materials

3.2.1 Permeability testing of mortar specimens

Table 4 shows the composition of the mortar specimens. The water-cement ratio is 50%. The sand-cement ratio is 3. Mortar specimens used in the permeability testing were cylindrical (H: 5.1 cm, φ: 5.0 cm). The mortar specimens used in the penetration depth testing were also cylindrical (H: 10.0 cm, φ: 5.0 cm). The curing period was 1 day in air following 27 days in water. The mortar specimens were examined in the saturated condition.

Figure 3 shows the permeability testing device for the mortar specimens. The device was composed of a graduated tube, a net plate, a water pen and a mortar specimen which the upper and the bottom plates of the mold are removed. During the testing, water was injected into the graduated tube and $h_1$ (m) was determined. Then, $h_2$ (m) was measured 1, 5, 10, 15, 20, 25 and 30 minutes later.

The coefficient of permeability was calculated by Eq. (3) [9]:

$$k = \frac{2.303 \times a}{A(t_2 - t_1)} \log_{10}$$

The cross section of the tube is $a$ (m²). The height of the mortar specimens is $L$ (m). The cross section of the mortar specimens is $A$ (m²). The start time is $t_1$ (s), whereas the end time is $t_2$ (s).

3.2.2 Penetration depth testing of the mortar specimens with various silicate based surface penetration materials

Figure 4 shows the depth and direction of penetration of various silicate based surface penetration materials into the mortar specimens. In this testing, the top faces of the mortar specimens were coated. Silicate based surface penetration materials were mixed with phenolphthalein solution, and 4 g of each of these mixtures were used to penetrate the mortar specimens. After 30 min, the mortar specimens were broken, as in Fig. 4. The penetration depth value was the average of the deepest and shallowest distances from the top face.
Four types of silicate based surface penetration materials were used in this testing. Each material was used to penetrate three mortar specimens.

Table 5 shows the main components of the silicate based surface penetration materials used. The main component of “A” is potassium silicate, while that of “B”, “C” and “D” is sodium silicate.

### 3.2.3 Viscosity testing of silicate based surface penetration materials

A rotational viscometer was used for determining the viscosities; it determines the coefficient of viscosity by measuring the viscosity–resistance torque of the liquid that works its spindle. Figure 5 shows the device for viscosity testing of silicate based surface penetration materials. Silicate based surface penetration materials (500 mL) were set in the beaker. The spindle was in cylindrical form, and its rotation speed was 200 cycles/min. The coefficient of viscosity was determined after 3 min of operation. The materials used were the same as those used in the penetration depth testing.

### 3.2.4 Modelling of one dimensional penetration

Mortar specimens were assumed to be in the saturated condition. A model was constructed based on Darcy’s law. The penetration depth of each material was calculated by Eq. (4) [9]:

\[
h = \frac{k i t}{\eta} t
\]

Where, the penetration depth is \( h \) (m). The coefficient of permeability for mortar specimens, \( k \), after 30 min (m/s). The ratio of the viscosity of water to that of the material in question is denoted by \( \eta \). The penetration time, \( t \), was 1,800 (s). The hydraulic grade, \( i \), is assumed to be 1.

### 4. RESULTS OF TESTING AND MODELLING

#### 4.1 Visual Appearance Testing

Table 6 shows the observations of visual appearance. Figure 6 shows the specimens subjected to the visual appearance testing, which were observed in scattered daylight. In Fig. 6, the upper three specimens were coated in silicate based surface penetration materials (hereafter, coated specimens), while the bottom three were not coated in silicate based surface penetration materials (hereafter, uncoated specimens).

#### 4.2 Waterproof Performance

Table 7 shows the results of the water permeability, water absorption and water-vapor permeability tests. The averages of each quantity for the three coated specimens were compared with the respective averages for the three uncoated specimens.

<table>
<thead>
<tr>
<th>Materials</th>
<th>A</th>
<th>B</th>
<th>C</th>
<th>D</th>
</tr>
</thead>
<tbody>
<tr>
<td>Main components</td>
<td>K₂O·SiO₂</td>
<td>Na₂O·SiO₂</td>
<td>Na₂O·SiO₂</td>
<td>Na₂O·SiO₂</td>
</tr>
</tbody>
</table>

Table 5: Components of applied silicate based surface penetration materials

<table>
<thead>
<tr>
<th>Samples number</th>
<th>Appearance</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>No change</td>
</tr>
<tr>
<td>2</td>
<td>No change</td>
</tr>
<tr>
<td>3</td>
<td>No change</td>
</tr>
</tbody>
</table>

Table 6: Observation of visual appearance

Fig. 5: Viscosity testing device for silicate based surface penetration materials

Fig. 6: Coating mortar samples and Non-coating mortar samples
The average water permeability ($W_p$) of the coated specimens was 8.30 mL, while that of the uncoated specimens was 9.38 mL, resulting in a $W_p$ ratio of 88%; the average water absorption ($W_a$) of the coated specimens was 0.8%, while that of the uncoated specimens was 1.7%, resulting in a $W_a$ ratio of 47%; the average water-vapor permeability ($W_v$) of the coated specimens was 0.35 g, while that of the uncoated specimens was 0.30 g, resulting in a $W_v$ ratio of 117%.

4.3 Resistance Against Deterioration

Table 8 shows the results of testing of resistance against neutralization (carbonation) and chloride ion penetration. For both resistance testing, the averages of the three coated and three uncoated specimens are recorded. The average neutralization (carbonation) depth of the coated specimens was 6.1 mm, while that of the uncoated specimens was 7.2 mm, resulting in a ratio of 85%. The average chloride ion penetration depth of the coated specimens was 11.7 mm, while that of the uncoated specimens was 10.8 mm, resulting in a ratio of 92%.

4.4 Permeability Testing

Figure 7 shows the permeability coefficient of the mortar specimens for elapsed time. The maximum permeability coefficient was $6.32 \times 10^{-6}$ m/s, whereas the minimum was $6.12 \times 10^{-6}$ m/s. This coefficient is thus essentially constant.

4.5 Penetration Depth Testing

Table 9 shows the penetration depth of each silicate based surface penetration material into the mortar specimens. The penetration depth decreases in order from “A” to “D”.

4.6 Viscosity Testing

Table 10 shows the coefficient of viscosity for each silicate based surface penetration material. The coefficient of “A” is the lowest of all silicate based surface penetration materials. Table 11 shows the content ratio by weight of the main components of each silicate based surface penetration material.

4.7 Modelling of One Dimensional Penetration

Table 12 shows the penetration depths from the one dimensional model simulating the penetration of each silicate based surface penetration material into the mortar specimens.
5. DISCUSSIONS OF TESTING AND MODELLING

5.1 Evaluation of the Visual Appearance Testing

None of the three coated specimens underwent changes in color or overall appearance due to impregnation. Thus, silicate based surface penetration materials with low viscosity do not change the appearance of a mortar specimen.

5.2 Chemical Components

At present, the main component of silicate based surface penetration materials is often sodium silicate. Table 13 compares the chemical formulas and characteristics of silicate components [10]. The chemical formula and characteristics of potassium silicate are similar to that of sodium silicate. The appearance of concrete remained the same in both cases because the water solutions were clear. The difference between the properties of these two silicates was observed with variation of temperature and concentration. The properties of sodium silicate changed more than those of potassium silicate. Sodium silicate reacted unstably and decreased its reaction products. In contrast, potassium silicate reacted stably. Furthermore, potassium silicate was easily soluble because of its large ionization tendency. Therefore, potassium silicate could effectively repair itself with the addition of a little water. The speed of chemical reaction of potassium silicate was slower than that of sodium silicate, and potassium silicate had a lower viscosity. Thus, potassium silicate penetrated more deeply into the concrete surface, offering an improved waterproofing performance. The cost of potassium silicate is greater than that of sodium silicate because there are no sources of potassium in Japan. However, neither component is particularly expensive.

Table 14 compares the chemical formulas and characteristics of the hydroxide components [10]. The characteristics of potassium hydroxide are similar to those of sodium hydroxide. The appearance of concrete did not change in either case because the water solutions were clear. These components had high deliquescence, implying that they absorbed water in concrete and became water solutions. They showed differences in their absorption of CO₂, with potassium hydroxide absorbing more CO₂ than sodium hydroxide. Accordingly, potassium hydroxide inhibited neutralization (carbonation) to a greater extent than sodium hydroxide.

Table 15 compares the characteristics of carbonate components [10]. Potassium carbonate is more alkaline than sodium carbonate and is also more soluble in water and absorbs more CO₂. Thus, potassium carbonate inhibited neutralization (carbonation) to a greater extent than sodium carbonate. Potassium hydrogen carbonate was formed by a chemical reaction between CO₂ and potassium carbonate. Potassium hydrogen carbonate and sodium carbonate are both white in color. However, blanching of the surface was not observed in Fig. 3. It is thus inferred that the silicate based
surface penetration material penetrated deeply into the concrete surface.

### 5.3 Waterproofing Performance

Coating the specimen with the potassium silicate based surface penetration material with low viscosity decreased $W_p$ and $W_a$ by 15% and 53%, respectively, thus implying the effect of the silicate based surface penetration material mentioned above. In other words, calcium silicate was formed by a reaction of potassium silicate with calcium hydrate and caused densification of the mortar specimens, suppressing water penetration.

Voids formed due to entrapped air when mixing ingredients may provide passages for water migration within concrete, thereby advancing deterioration. It is assumed that coating concrete with a silicate based surface penetration material can eliminate or reduce the size of these voids, leading to the densification of concrete.

Coating with the potassium silicate based surface penetration material with low viscosity decreased $W_v$ to increase by 17% because existing moisture decreased as the voids were filled.

### 5.4 Resistance to Deterioration

Coating with the silicate based surface penetration material caused the resistance to neutralization (carbonation) to increase by 15% and the resistance to chloride ion penetration to increase by 8%. Because neutralization (carbonation) occurs when the specimen makes contact with carbon dioxide in the air, it is considered to progress further if the specimen has more voids, allowing the transmigration of more moisture and gases. This reasoning also applies to chloride ion penetration; more movement of chloride-ion containing water or gas within the specimen causes more significant deterioration. Coating with the silicate based surface penetration material improved the resistance against neutralization (carbonation) or chloride ion penetration in the testing, because the mortar specimens achieved densification, offering better waterproofing performances, including water absorption.

These resistances are related to the degree of progress of neutralization (carbonation), salt damage and steel corrosion in concrete structures. They, together with waterproofing performance, are very important factors when considering deterioration control of concrete.

### 5.5 Relationship Between Penetration depth and Viscosity

Figure 8 shows the relationship between the penetration depth predicted by the Modelling of one dimensional penetration and observed experimentally. The theoretical curve and the values of penetration depth testing are approximate. The horizontal axis represents the penetration depth (mm); the vertical axis represents the coefficient of viscosity ($\text{Pa} \cdot \text{s}$). The penetration depth decreases with increase of the coefficient of viscosity. Accordingly, evaluation of the viscosity of silicate based surface penetration materials enables evaluation of the penetration depth and performance of these silicate based surface penetration materials.
6. CONCLUSIONS

This study found results concerning the performance of silicate based surface penetration materials including low viscosity one. They are listed as follows:

(1) Because it is low viscosity, the potassium silicate solution penetrates deeply into the surface of concrete.

(2) Potassium silicate reacts stably because the properties of potassium silicate change less with temperature and concentration compared with those of sodium silicate.

(3) Because the ionization tendency of potassium silicate is large, it can effectively repair itself when small quantities of water are added.

(4) Potassium hydroxide and potassium carbonate form from chemical reactions with concrete and absorbed CO₂, allowing the silicate based surface penetration material to effectively resist neutralization (carbonation). Although these substances are white in color, no bleaching of the concrete was observed because they penetrated deeply.

(5) Penetration of the potassium silicate based surface penetration material with low viscosity improved the performance of waterproofing and the deterioration control performance of concrete structures.

(6) Evaluating the viscosity of silicate based surface penetration materials enables the evaluation of penetration depth and performance of silicate based surface penetration materials.

7. REFERENCES


