



Effects of inorganic surface treatment on water permeability of cement-based materials



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ABSTRACT

The permeability of the cement-based materials can be used as an important indicator of their durability. Surface treatment is a simple way to reduce permeability and improve durability of cement-based materials. This paper studied the effects of fluosilicate and sodium silicate surface treatments on the permeability of cement-based materials using the Autoclave water permeability and water absorption testing method. The experimental results showed that both fluosilicate and sodium silicate surface treatments could effectively reduce the permeability of cement-based materials. However, fluosilicate worked within the first 28 days after treatment, while sodium silicate showed more obvious effect at later ages. Autoclave water permeability index exhibited an exponential relationship with the water absorption of the cement-based materials. In addition, mercury intrusion porosimetry result suggested that these inorganic surface treatment agents could reduce the porosity of surface layer of cement-based materials.

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1. Introduction

The permeability of concrete affects the transport of gases, liquids, and ions through it under gradient of pressure, chemical potential, or electric potential [1,2]. Since many aggressive substances can transport by water, water permeability is a key property of concrete governing its long-term durability [3–8].

Many methods, such as reducing water to binder ratio and adding mineral admixtures etc., are often used for decreasing the permeability of concrete by reducing its porosity [9]. However, these methods often cannot be used for existing concrete structures. Compared with reducing water to binder ratio and adding mineral admixtures, protective surface treatment wins favor in that it does not interrupt construction work, and is hence cost-effective [10,11]. Surface treatment has been proven to be effective in limiting water penetration [12,13]. In very aggressive environments, they also can dramatically reduce the material degradation and steel corrosion which affect strength and stability of concrete structures [14,15]. In addition, some surface treatment agents can also extend service life of cracked concrete, while admixtures cannot prevent ingress of aggressive substances in cracked concrete

[10,14]. Concrete surface treatment shares at least one of two objectives: to make the concrete cover zone less permeable to aggressive substances or to reduce its moisture content and thereby to increase the concrete resistivity with appropriate barrier characteristics. Both effects can increase the service life of the structures [17].

Surface treatment agents can be classified into inorganic and organic categories. The organic treatments do not react with the concrete substrate, but create a physical barrier against the ingress of aggressive substances. The service life of organic treatment agents depend largely on their adhesive ability with the concrete substrate and resistance of weathering. There were many encouraging results demonstrated their good performance, but they still showed some drawbacks. Acrylic coatings could significantly reduce the permeability of concrete [18]. However, its poor resistance to chloride ion penetration limits its application in the marine structures [19,20]. The resistance of organic sealers to aging is so poor that they cannot be used in long-life structures such as dams and bridges. Though the silane performs well in reducing the permeability of concrete, it is difficult to ensure its longevity. Some organic treatment agents have poor fire resistance, possibly leading to cracking and detachment, limited service life and hard to remove after losing effectiveness [21,22].

The inorganic surface treatment agents are mainly aqueous solutions of sodium silicate, also known as “waterglass”, and, to a

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much lower extent, potassium silicates and fluosilicates [23]. These silicates are one of pore-blocking treatments which react with cement paste forming calcium silicate hydrate (C–S–H) gel, and increase hardness and impermeability of concrete surface layer [23]. However, the published results on the effect of sodium silicate surface treatment are very limited [10]. Some results [10] show that sodium silicate hardly penetrates into concrete substrate and is quite ineffective in preventing water absorption and chloride penetration. Furthermore, waterglass may increase the risk of alkali–silica reaction due to the increase of alkali content. Others reported that it performed well in preventing water adsorption and resisting of carbonation [24]. Kagi showed that its performance could be significantly improved by post-treatment with cationic surfactants (alkylquaternary ammonium salts) [25].

This paper investigated the effects of fluosilicate and silicate surface treatments on water permeability of concrete using Autoclam method and water absorption. Mercury intrusion porosimetry (MIP) was also used to investigate the porosity of concrete surface layer after surface treatments. The purpose was to examine the effectiveness of those inorganic surface treatment agents, and the interactions between those surface treatment agents and concrete.

2. Raw materials and testing methods

2.1. Materials

P·I 52.5 Portland cement with a specific surface area of 336 m²/kg, and an ignition loss of 2.49% was used. It gave compressive strength of 26.6 MPa at 3 days and 57.3 MPa at 28 days. Its chemical composition is shown in Table 1. Medium size sand and gravel from Xiang River, Hunan, China, after sieving with 40 mm sieve were used as fine and coarse aggregates.

An industrial grade waterglass with modulus (n) of 3 was used. NaOH was added to the waterglass to obtain a solution with modulus of 2. Their physical and chemical properties are shown in Table 2. Waterglass N₂ and N₃ and water were mixed in a mass ratio of 1:4 to obtain waterglass solutions for surface treatment.

Because sodium fluosilicate is often used as an accelerator of waterglass, three sodium fluosilicate solutions with concentrations of 1%, 2% and 3% were used for pretreatment to enhance the efficiency of waterglass treatment. Magnesium fluosilicate solutions with concentrations of 10%, 20%, and 30% were prepared and used as surface treatment agents.

2.2. Mixture proportions and preparation of specimens

The mixture proportion of the concrete is given in Table 3. The concrete mixture was prepared in the laboratory using a 60 L mixer. Cylinders of $\Phi 110$ mm \times 100 mm were cast for water absorption testing (three cylinders/group), slabs of 300 \times 230 \times 75 mm were cast for water permeability testing (one slab/group). Mortar with the same mixing ratio of cement, sand and water was casted in $\Phi 110$ mm \times 100 mm PVC pipes for porosity measurement. All the molds with specimens were compacted using a vibration table, then covered with plastic films and placed in a lab room at T = 20 \pm 1 °C for 24 h. Then concrete specimens were demolded and cured in a moist curing room at T = 20 \pm 1 °C and RH \geq 98%, while the mortar specimens were cured with the mold in same

Table 1
Chemical composition of cement (%).

SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	K ₂ O	Na ₂ O	SO ₃
21.91	5.30	3.67	65.10	1.51	0.62	0.19	2.03

Table 2
Chemical composition and parameters of waterglass.

Code	Modulus	Na ₂ O (%)	SiO ₂ (%)	Baume degree	Density (kg/m ³)
N ₃	3	9.05	26.5	38.4	1369
N ₂	2	12.9	25.0	44.1	1440

environment. The mortar samples could not be demolded until surface treatment, because the molds could prevent treatment agent from penetrating into lateral faces during treatment.

2.3. Surface treatments

After 6 days of curing in the curing room, cylinder specimens were cut into 50 mm length from the middle. Then all the concrete specimens were placed in an environment of T = 20 \pm 2 °C, RH = 55 \pm 5% for 24 h before surface treatment to ensure that moisture condition of the specimen surface was dry as recommended in the publication [25]. The treatment methods are summarized in Table 4. The top side of concrete slabs, all faces of concrete cylinders would be treated, while the treatment agents was only applied on top face of mortar cylinders. From groups 1–2 to 1–6, the treated faces were brushed with surface treatment agents using a nylon brush every two hours for four times according to Table 4. In term of groups 1–7 to 1–12, the treated faces were brushed with sodium fluosilicate every two hours for four times. After 24 h, the waterglass treatment agents were applied on the treated surface for 4 times. After treatment, they were put back into the curing room T = 20 \pm 1 °C and RH \geq 98% until testing ages.

2.4. Testing methods

2.4.1. Water permeability

Concrete slabs were placed in laboratory at T = 20 \pm 2 °C and RH = 55 \pm 5% for 24 h before Autoclam permeability measurement which is quite sensitive to moisture condition [26]. The water permeability indexes were measured 14d, 28d and 56d after surface treatment. It took about 5 min for the concrete samples to achieve saturation and for the instrument to reach a steady state [27]. Thus only data between 5th and 15th minutes were recorded. Criteria of water permeability are shown in Table 5 [28].

2.4.2. Water absorption test

The water absorption were measured at 0d, 14d, 28d and 56d after surface treatment. The 0 day measurements were tested immediately after surface treatment. Concrete specimens of $\Phi 110$ mm \times 50 mm were first vacuumed for 4 h, and then immersed in saturated calcium hydroxide solution for 24 h. After wiping the surface water, the mass (m₁) of specimens were measured under surface-dry condition, then the samples were dried in an oven at 105 °C until constant mass (m₂). The water absorption of the specimen can be calculated as following equation. The means of three specimens were presented in the results.

$$\rho = \frac{m_1 - m_2}{m_2} \times 100\%$$

2.4.3. Pore structure

The mortar specimens were cut into thin-disc about 5 mm of thickness below the treated surface 56d after treatment. The discs were crushed into fragments about 0.5 cm size by a plier. The small fragments were immersed in anhydrous ethanol at least 7d to stop further hydration, then dried in an oven at 60 °C for 24 h. Pore

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