

Research Article

Carbonation of Water Repellent-Treated Concrete

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Water repellent treatment has been considered an effective preventive method against water and aggressive ions penetration into concrete and consequently can improve the durability of concrete structures. In reality, many concrete structures are exposed to conditions with high risk of carbonation. In this contribution, one type of ordinary concrete had been prepared and surface impregnated by 400 g/m² silane cream and 100 g/m² and 400 g/m² silane gel. In addition, integral water repellent concrete was produced by adding 2% silane emulsion. Then, the specimens were exposed to accelerated carbonation for 7, 28, and 72 days. The effect of water repellent treatment on carbonation of concrete has been investigated. The results indicate that surface impregnation reduced carbonation depth of concrete under RH 70%, but integral water repellent concrete increased carbonation. Carbonation reaction started behind the hydrophobic layer in the surface-impregnated concrete. The coefficient of carbonation can be described better by a hyperbolic function of time. Treatment by 400 g/m² silane gel and silane cream showed better efficiency on reducing carbonation than usage of 100 g/m². Coefficient of water capillary suction was decreased significantly by both surface impregnation and integral water repellent treatment. It is an effective method to protect concrete from water penetration into the material.

1. Introduction

Ordinary cement concrete is a cheap and versatile building material which has been applied for nearly 200 years with great success and in most diverse structures. It is hardly possible to imagine the construction of the infrastructure in all industrialized nations today without concrete. But pure cement concrete is rather brittle with a small ultimate strain capacity. This often leads to early crack formation. Moreover, cement concrete is porous, which allows aggressive substances to migrate into the material. This is at the origin of early deterioration of concrete in aggressive environment and of limited protection of embedded steel reinforcement.

Moisture transport in cement-based materials is an important physical process with direct influence on service life and durability. If the surface of concrete is in direct contact with water-soluble compounds, hydration products may be washed out by leaching. In this way, near-surface zones are weakened and become more frost sensitive. If the liquid in contact with the surface is an acid, this deteriorating process is even accelerated. In case water gets in contact with

a predried surface of concrete, it will be absorbed quickly and deep into the pore space. Aggressive compounds dissolved in water can be transported deep into the pore space of concrete. Drying of concrete always imposes shrinkage of the dry zones and leads to crack formation. Gases of the environment may be dissolved in the aqueous pore solution of concrete. In this way, acids are formed, for instance, by dissolution of CO₂, which could react rapidly with the hydration products of cement.

It is obvious that all these deterioration mechanisms act from the surface of concrete. Throughout history, a range of protective materials have been applied on the exposed surfaces of structural concrete elements to prevent the ingress of water. Nowadays, water repellent treatment has proved to be effective to prevent concrete from water penetration and consequently can improve the durability of concrete structures [1–8]. Application of hydrophobic treatment with an approved product has even become standard practice on new bridge decks in the Netherlands [9]. For broader information about the studies on water repellent treatment, the proceedings of the Hydrophobe

conference series (Hydrophobe I–VIII) can be referred [10–17].

When applying the water repellent agents on actual structures, however, many concrete surfaces are exposed to carbonation condition, and some had been carbonated already before the surface treatment. Heinrichs et al. [18] and Gerdes [19] reported that water repellent treatment alters the drying attitude of concrete. This in turn mainly influences the process of carbonation. They had simulated the transport, storage, and chemical reaction of the main influencing variables for carbonation of treated concrete. It was believed that the carbonation process can be delayed by surface impregnation under proper conditions. The study from Courard and Lucquiaud [20] also showed that the application of a water repellent delayed the propagation of the carbonation front. However, there is some concern that the gradual drying out due to hydrophobic treatment causes deeper carbonation [9]. Besides, very little study has focused on the influence of integral water repellent treatment on the carbonation process of concrete. Therefore, it has to be clarified if the cement-based materials could be protected by water repellent treatments any more when carbonation reaction occurs simultaneously.

In this contribution, one type of ordinary concrete with a water-to-cement ratio of 0.5 has been produced. Two types of water repellent treatment were applied. One was surface impregnation. The surface of the samples was covered by different amounts of silane, in this study, 400 g/m² silane cream and 100 g/m² or 400 g/m² silane gel. Another type was integral water repellent treatment by adding 2% of silane emulsion in fresh concrete during mixing. All surface-impregnated and integral water repellent concrete were exposed to accelerated carbonation for 7, 28, and 72 days. In this way, the effect of water repellent treatment on carbonation of concrete has been investigated. Another emphasis had been concentrated on the water capillary absorption behavior of water repellent-treated concrete after carbonation.

2. Basic Mechanism of Water Repellent Treatment

There are basically two different technologies to make concrete water repellent: (1) surface impregnation and (2) integral water repellent treatment. In the first case, the surface of hardened concrete is impregnated with a reactive agent, and in the second case, the reactive agent is added to the fresh mix. Different organic compounds have been used in the past as water repellent agents. Historically, natural polymers such as oils, fats, and waxes have been used to protect statues and buildings. Today, in many cases, silanes are applied to make concrete or stones water repellent [15, 16]. The general composition of higher silanes can be written as H-(SiH₂)_n-H with $n = 1, 2, 3, \dots$. Similar to alkanes, silanes with more than three Si atoms can form structural isomers. In the presence of water, silanes react to form siloxanes and finally silicon resin. The resin forms a network, which is firmly fixed on the huge surface of the hydration products of Portland cement. In Figure 1, the

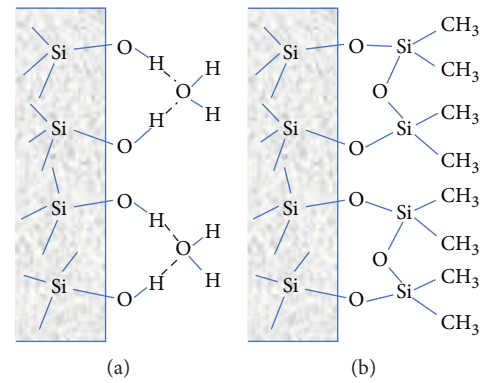


FIGURE 1: Schematic representation of (a) adsorbed water layer on the surface of hardened cement paste and (b) silicon resin fixed on the surface replacing the water layer.

replacement of an adsorbed water layer by the silicon resin network is shown schematically.

In the case of surface impregnation, liquid silane penetrates into the pore space of concrete by capillary action. In order to achieve a sufficient penetration depth, the contact time between the concrete surface and the liquid silane must be long enough [21–23]. On a horizontal surface, this can be achieved easily, but vertical surfaces have to be sprayed or brushed with liquid silane for several times. This problem can be overcome by applying a so-called cream, a silane emulsion which can be applied once with the necessary thickness or silane gel which is a suspension of fine clay articles in silane. The gel is stiff enough to be applied with the necessary thickness. If integral water repellent concrete is to be produced, silane emulsion can be added to the fresh mix. After hardening and initial drying, the emulsion breaks and silicon resin is formed on the pore walls. In principle, the distribution of silicon resin in integral water repellent concrete should be uniform all over the volume. This is, however, not automatically achieved.

The effect of water repellents is essentially based on their low surface tension. The behavior of water when contacting the surface of a material is governed by the surface tension which can be measured by the contact angle. The intensity of the water repellent property is associated with the contact angle between water and the treated surface. Contact angles of a water droplet of more than 90° represent hydrophobic property while less than 90° hydrophilic property. The organofunctional groups will reduce the critical surface tension of the material surface and thus provide hydrophobicity.

3. Materials and Methods

3.1. Materials and Preparation of Specimens. The composition of the concrete matrix has been the same for all specimens used in this project, which is shown in Table 1. Portland cement 42.5 type II with a compressive strength at 28 days of no less than 42.5 MPa, local crushed granitic aggregates with a maximum diameter of 20 mm and density of 2620 kg/m³, and river sand with a maximum grain size of

TABLE 1: Composition of the concrete used in this project.

Type	W/C	Cement, kg/m ³	Sand, kg/m ³	Aggregate, kg/m ³	Water, kg/m ³	Compressive strength at 28 d, MPa
Concrete, C	0.5	320	653	1267	160	40.1

TABLE 2: The type of water repellent agents used in this project and their amounts.

Ref.	Type	Component	Amount of use	Note
Ref.	—	—	—	No treatment, reference sample
C400	Silane cream	Alkylalkoxysilane	400 g/m ²	Surface impregnation
G100	Silane gel	Triethoxysilane	100 g/m ²	Surface impregnation
G400	Silane gel	Triethoxysilane	400 g/m ²	Surface impregnation
IW	Silane emulsion	Octyltriethoxysilane	2%	Adding to the fresh mixture

5 mm and density of 2610 kg/m³ were used. The water-to-cement ratio was 0.5. To improve workability, 0.5% superplasticizer of naphthalene type, related to the mass of cement, had been added to the fresh concrete. In addition, some integral water repellent concrete specimens were prepared with addition of 2% (related to the mass of cement) silane emulsion (octyltriethoxysilane) into the fresh concrete during mixing. It shall be noticed that the compressive strength of integral water repellent concrete at 28 days was reduced by approximately 11%, from 40.1 MPa for reference-untreated concrete to 35.7 MPa.

Prismatic specimens with the dimensions of 100 × 100 × 400 mm³ and cubic specimens with the edge length of 100 mm were produced. All specimens were compacted in steel forms and cured for one day before demoulding. Then, the specimens were stored in the curing room at a temperature of 20 ± 2°C and relative humidity of 95% until an age of 14 days. After that, all specimens were taken out of the curing room and further stored under laboratory conditions at 20 ± 2°C and 60% RH for another 7 days before applying surface impregnation.

3.2. Water Repellent Treatment. After drying in the lab for 7 days, all the samples were divided into 5 groups. One group was maintained as reference specimens, that is, without water repellent treatment. One group was integral water repellent concrete, as produced above. The other three groups of concrete specimens were treated by different types and amounts of water repellent agents, as listed in Table 2. When one of the moulded surfaces was treated by covering 400 g/m² of silane cream, the series was named C400, while the specimens treated by 100 and 400 g/m² of silane gel were called G100 and G400, respectively. After surface impregnation, the specimens were exposed to laboratory atmosphere again for another 7 days in order to allow sufficient polymerization of silane. Then, both water repellent-treated and -untreated concrete specimens were ready for accelerated carbonation test.

3.3. Accelerated Carbonation. All the water repellent-treated and -untreated specimens were then submitted to accelerated carbonation for 7, 28, and 72 days. According to

the method in Chinese standard [24], the concentration of CO₂ gas had been maintained constant at 20 ± 2%, relative humidity in the carbonation box was about 70%, and the temperature was 20 ± 3°C. Four surfaces except for the treated surface and its opposite surface of concrete specimens had been sealed with wax before placing in the carbonation box. After 7, 28, and 72 days, carbonation depth for each sample was determined by spraying phenolphthalein solution of 1% in ethanol. It was measured every 10 mm along a side length of 100 mm. Except for the two values from the border, the average value of the other nine measurements was the carbonation depth of each sample. For each case, three specimens were measured. Then, the average value of these three samples represented the carbonation depth for each case.

3.4. Water Capillary Absorption. In addition, in order to study the effect of water repellent treatment on water capillary absorption of concrete before and after carbonation, the water capillary absorption test has been performed according to the partial immersion method as explained in the ISO standard [25]. The specimens were cut into two halves first and then were dried in an aerated oven at a temperature of 50°C for 7 days until mass equilibrium. When the specimens cooled down to room temperature, they were put in contact with water. The amount of absorbed water was then measured by weighing the specimens at different time. In this test, it was at 0.5, 1, 2, 4, 8, and 24 hours in the first day and then measured every day until 7 days or so.

4. Results and Discussion

4.1. Carbonation in Water Repellent-Treated and -Untreated Concrete. Carbonation depth in water repellent-treated and -untreated concrete has been measured after exposure to 7, 28, and 72 days of carbonation. The results are shown in Figure 2. It is obvious that surface-impregnated concrete had lower carbonation depth than untreated samples. But the integral water repellent concrete has comparatively higher carbonation depth. The possible reason is that the addition of silane emulsion can retard the hydration process of cement [26]. This also explains why

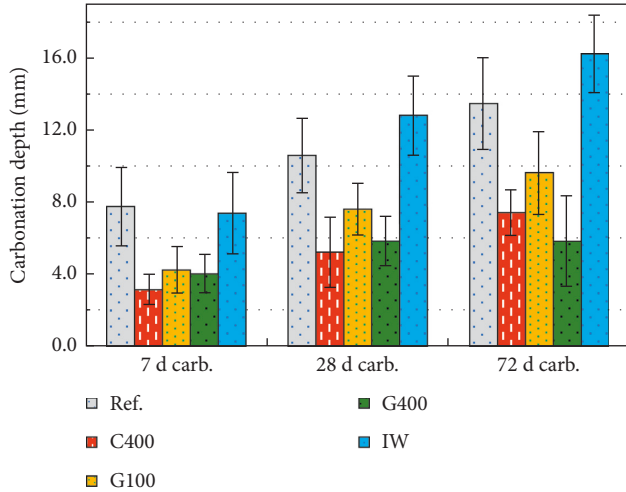


FIGURE 2: Carbonation depth of water repellent-treated and -untreated concrete after 7, 28, and 72 days of accelerated carbonation.

the compressive strength of integral water repellent concrete was reduced slightly. In this case, the penetration of carbon dioxide would be quicker and then cause higher carbonation depth.

For the surface-impregnated concrete, application of 400 g/m² silane cream and silane gel reduced approximately one-half of the carbonation depth compared to the reference-untreated samples. For instance, the carbonation depth of untreated concrete was 7.7 mm after 7 days of carbonation. After surface impregnation, carbonation depth of the samples C400 and G400 was 3.1 mm and 4.1 mm, respectively. The efficiency of reducing carbonation action is better than 100 g/m² covering usage of silane.

After surface impregnation, the silane penetrated by capillary transport into the concrete. Consequently, the silicon functional group reacts with water and yields a reactive silanol group (hydrolysis stage); further, condensation by cross linking to the hydroxyl group forms polysiloxane as the active water repellent film which is linked to the inorganic substance by way of covalent siloxane bounds.

The rate of carbonation process depends very much on the moisture content of concrete. The neutralization between CO₂ gas and calcium hydrate or C-S-H gel requires the presence of water because carbon dioxide needs to dissolve in water first forming carbonic acid. If the concrete is too dry, CO₂ gas may enter the material rapidly. But it cannot dissolve, and no carbonation occurs. If, on the other hand, it is too wet, CO₂ is not able to enter the pore and no carbonation occurs, either. The optimal conditions for carbonation occur at a relative humidity of 50%–80% [27–29]. In this test, all the treated and untreated specimens were kept in carbonation box under relative humidity of 70%. After 7, 28, and 72 days, the carbonation depth had been measured by spraying the phenolphthalein solution on the fractured surface. The typical results are shown in Figure 3. During the measurements, it has been found that surface-impregnated specimens G400 had three different layers, as shown in Figure 3 (the left photo). The first layer

was pink. This means the near surface region was not carbonated as the water content in this layer was very low [30]. Carbonation reaction did not occur in this hydrophobic layer as it is too dry. But CO₂ gas certainly migrated through this layer and reached the inner part where there was necessary water for carbonation reaction. Thus, the second layer without colour indicates that the carbonation process started behind the water repellent layer. However, the carbonation process in this untreated region was delayed because its drying rate was lowered by the hydrophobic layer, and consequently, a high content of water in the pores hindered the carbonation process. Therefore, the carbonation depth in surface-impregnated concrete was reduced in comparison with the reference untreated concrete.

The carbonation mechanism has been studied by many researchers, and various mathematical models have been developed with the purpose of predicting carbonation depth [31–34]. For the sake of simplicity, however, the carbonation kinetics can be described as a first approximation to be a diffusion process of CO₂ through porous material with given moisture content. By applying Fick's law, (1) can be found after integration:

$$x = \sqrt{\frac{2D \cdot c}{d}} \sqrt{t}, \quad (1)$$

where x is the carbonated depth at time t , D is the effective diffusivity of CO₂, c is the CO₂ concentration in the atmosphere, and d is the concentration of the reactive compounds. Diffusivity D is not a constant but a function of numerous parameters. It mainly depends on time (degree of hydration), location (curing effect), and on the moisture content. Equation (1) can be written in the well-known form as

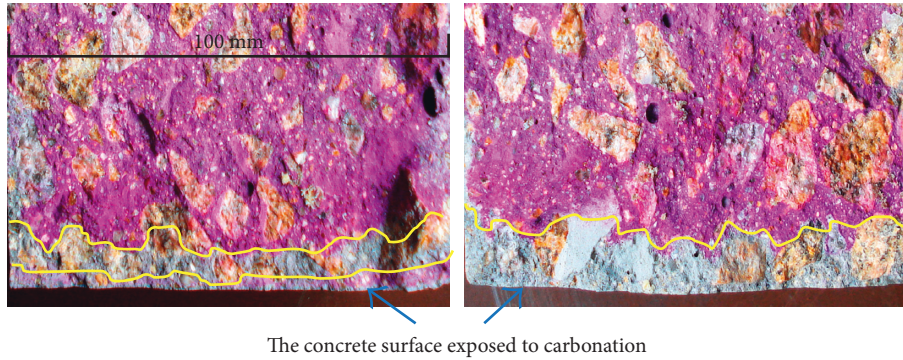
$$x = K \sqrt{t}, \quad (2)$$

where K denotes the coefficient of carbonation. Assuming the coefficient of carbonation rate changes with carbonation time, K can be described by a hyperbolic function, as shown in (3), in which P_1 , P_2 , and P_3 are parameters fitted from the experimental results. Another exponential model can also be used to describe the decrease of carbonation coefficient, as shown in (4), in which a and b are parameters fitted from the experimental results:

$$K = P_1 - \frac{P_2 t}{P_3 + t}, \quad (3)$$

$$K = a \cdot b \cdot e^{-b\sqrt{t}}. \quad (4)$$

It has been learned already from Figure 2 that with increasing time, the carbonation depth increases. The relationship between carbonation depth x and the square root of time t is shown in Figure 4. According to (3) and (4), the coefficient of carbonation for water repellent-treated and -untreated concrete can be calculated. The results are shown in Figure 5. It can be seen from the results that the carbonation coefficient was decreased significantly by surface impregnation. With the increasing time, finally the coefficient values would reach constant. This proves again that



The concrete surface exposed to carbonation

FIGURE 3: Determination of carbonation depth in surface-impregnated (left: G400) and reference untreated concrete specimens (right: Ref.).

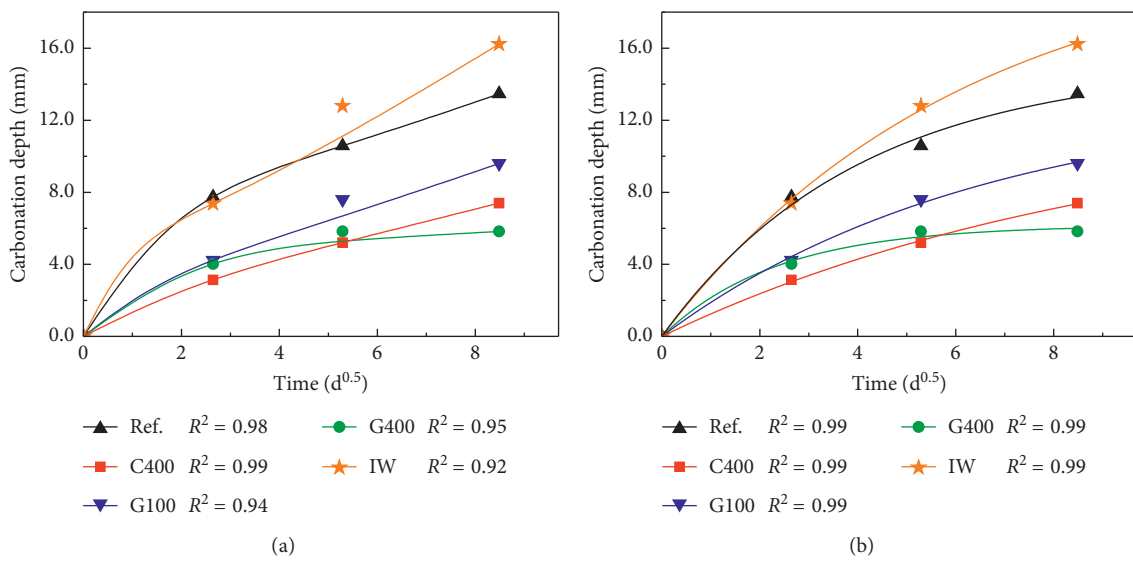


FIGURE 4: Carbonation depth versus square root of carbonation time. (a) Fitted by (3). (b) Fitted by (4).

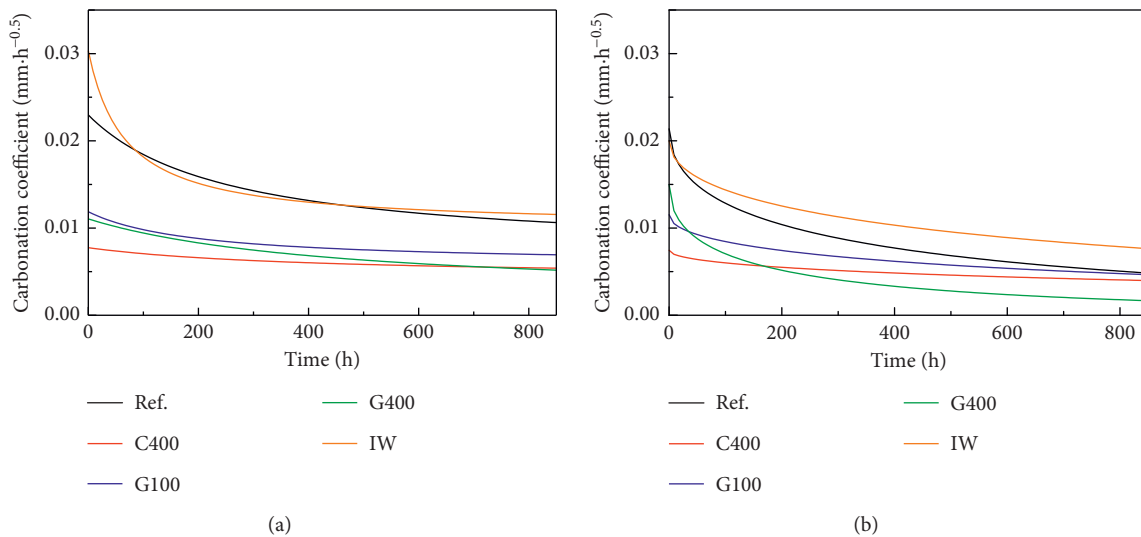


FIGURE 5: Changes of carbonation coefficient with increasing time. (a) Results obtained from (3). (b) Results obtained from (4).

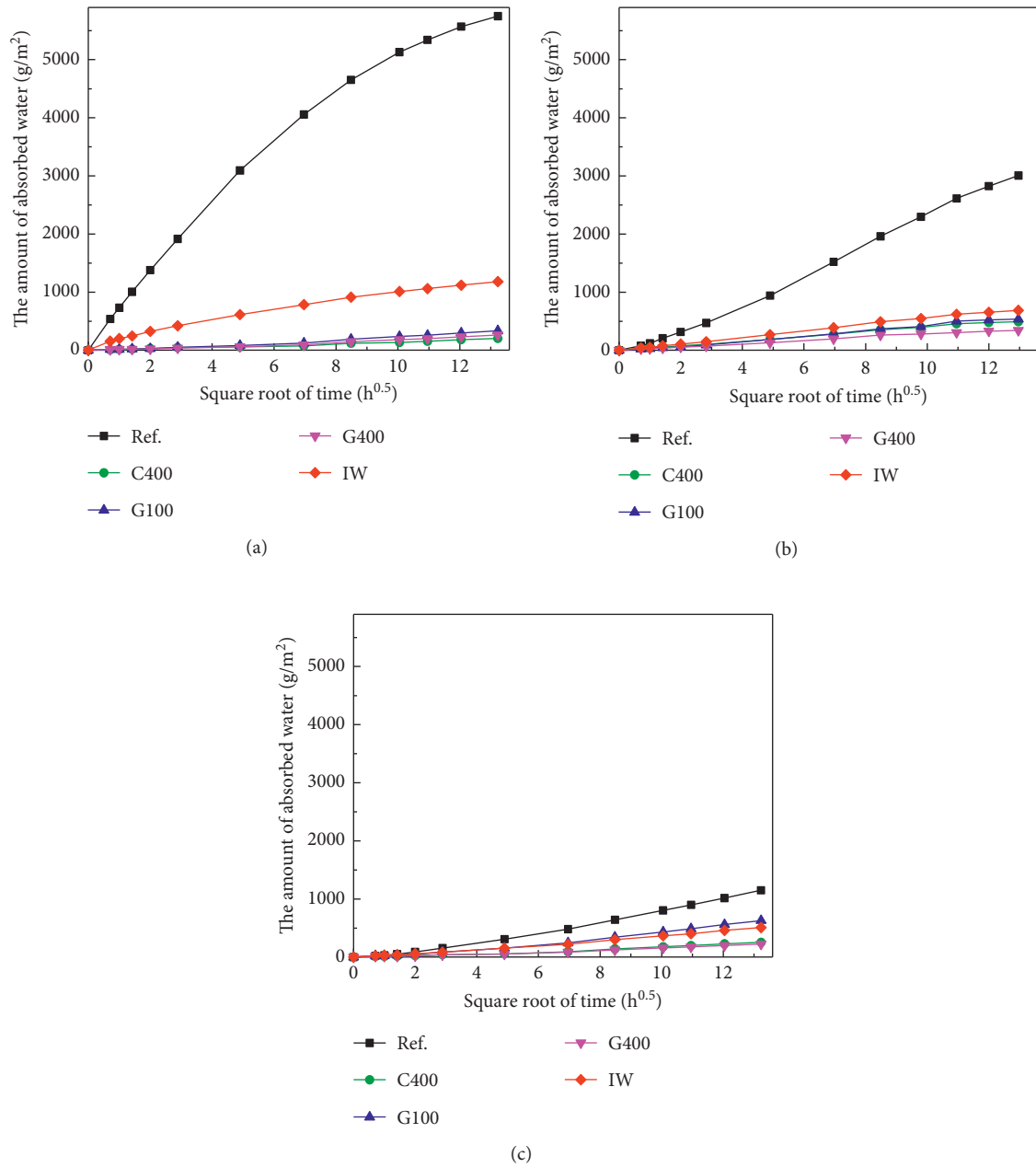


FIGURE 6: Water capillary absorption of water repellent-treated and -untreated concrete before and after 7 and 28 days of carbonation. (a) No carbonation. (b) 7 days of carbonation. (c) 28 days of carbonation.

surface impregnation can slow the carbonation process and consequently reduce carbonation depth. Application of 400 g/m^2 silane cream and 400 g/m^2 silane gel shows better efficiency than 100 g/m^2 silane gel. Comparing the two models, it can be found that the exponential model achieves better agreement with the experiment results.

4.2. Water Capillary Absorption of Treated and Untreated Concrete after Carbonation. If the surface of a porous material is put in contact with a liquid, the liquid will be absorbed by capillary action. Within a limited range of time and as first approximation, the amount of absorbed

water as function of duration of contact time can be expressed by

$$\Delta W = A\sqrt{t}, \quad (5)$$

where ΔW is the amount of absorbed water; A stands for the coefficient of capillary suction ($\text{g/m}^2\text{h}^{0.5}$), which depends both on the properties of the absorbed liquid and the porous structure; and t is the absorption time.

If the microstructure of concrete changed due to carbonation of CH or C-S-H gel, or the surface of capillary pores was water repellent treated, the capillary suction would be changed consequently. The coefficient of capillary suction is considered to characterize this change. In this

TABLE 3: Coefficient of capillary absorption (A ($\text{g}/\text{m}^2\text{h}^{0.5}$)) by linear fitting of the results in Figure 6.

Condition/concrete	Ref.	C400	G100	G400	IW
No carbonation	584.4	12.9	19.5	15.1	116.9
After 7 days of carbonation	214.1	40.1	40.7	29.0	56.3
After 28 days of carbonation	71.0	14.6	16.7	13.3	33.0

test, this value for each type of specimen had been obtained experimentally.

The results of capillary suction for all the water repellent-treated and -untreated concrete, which were imposed to accelerated carbonation for 7 and 28 days, are shown in Figure 6. With (5), the coefficient of capillary suction within the first 72 hours can be calculated. The results are compiled in Table 3. The results clearly indicate that both surface impregnation and integral water repellent treatment can reduce considerably the water absorption of concrete.

In the surface-impregnated concrete samples C400, G100, and G400, the silane molecules ($d \approx 1-3$ nm) penetrated into concrete microstructures and made the surfaces of capillary pores hydrophobic. The coefficient of capillary suction was reduced enormously compared to the reference untreated concrete. With respect to the integral water repellent concrete IW, the coefficient of capillary suction also decreased. But the reduction is relatively lower. The origin of this phenomenon is due to the size of silane emulsion. Because the particle size of silane emulsion is approximately 100 nm, nanopores of C-S-H gel are not penetrated. Consequently, the fine pores will not become water repellent by the addition of silane emulsion. If the surface is in contact with water, some water can enter the porous structure of cement-based materials.

It shall be noticed that the carbonation resistance of integral water repellent concrete was lower than the reference untreated concrete mainly because the addition of silane emulsion delayed the hydration process of cement as mentioned previously. But this would not change the situation that the particle of silane emulsion still can break and form a siloxane network on the internal surface of some capillary pores in hardened cement paste. Therefore, the water capillary absorption can be still reduced by internal hydrophobic treatment in comparison with the reference untreated concrete.

After exposure to 7 and 28 days of carbonation, water absorption of concrete was also reduced by surface impregnation and integral water repellent treatment. This implies that water repellent treatment can still protect concrete from water absorption effectively even under carbonation condition. But the carbonation reaction can destroy the hydrophobic film inside the capillary pores and consequently may cause slightly more water absorption. For instance, the coefficient of capillary suction of the surface-impregnated concrete G400 was $15.1 \text{ g}/\text{m}^2\text{h}^{0.5}$ before carbonation. While after 7 days of carbonation, it became $29.0 \text{ g}/\text{m}^2\text{h}^{0.5}$. However, after 28 days of carbonation, water capillary absorption became lower again since the carbonation products filled the pores in the near-surface zone.

5. Conclusions

Based on the results presented herein, the following conclusions can be drawn:

- (1) Carbonation depth of concrete can be reduced by one-half by surface impregnation. But for integral water repellent concrete, carbonation depth increased compared to the untreated concrete. One has to be careful while using integral water repellent concrete under severe carbonation atmosphere concerning steel corrosion.
- (2) Three layers with different colours can be found when determining the carbonation depth of surface-impregnated concrete by means of phenolphthalein solution. CO_2 migrated through the impregnated layer quickly without carbonation as it is very dry. Carbonation process started behind the hydrophobic layer in this case.
- (3) The coefficient of carbonation can be described better by an exponential function than by a hyperbolic function of time. Surface impregnation by silane cream and silane gel with the amount of $400 \text{ g}/\text{m}^2$ has better efficiency on reducing carbonation than using $100 \text{ g}/\text{m}^2$ silane gel.
- (4) Coefficient of water capillary absorption of concrete can be reduced significantly by both surface impregnation and integral water repellent treatment, even under carbonation condition. It is an effective method to protect concrete from water penetration despite carbonation reaction.

It must be noticed that in this study accelerated carbonation test was performed with relatively high CO_2 concentration as the natural carbonation process was too slow. There might be different trends between the two types of test. This has to be further clarified.

Conflicts of Interest

The authors declare no conflicts of interest.

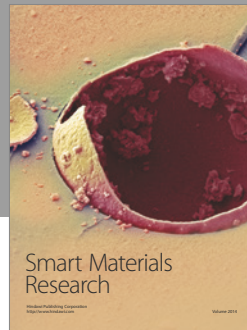
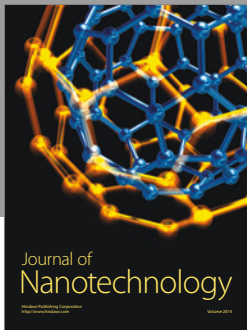
Acknowledgments

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