

Article

Analysis of the Effect of Nano-SiO₂ and Waterproofing Agent on the Water Transportation Process in Mortar Using NMR

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Received: 9 September 2020; Accepted: 3 November 2020; Published: 6 November 2020



Abstract: Moisture diffusion in cement-based materials significantly impacts its durability. In this study, we analyzed the effect of adding a waterproofing agent and nano-SiO₂ (NS) on the water transportation process in mortar using capillary water absorption tests and nuclear magnetic resonance (NMR) technology. The results indicate that the combined action of the waterproofing agent and nano-SiO₂ drastically reduce the capillary water absorption coefficient and have a more significant impact than only adding the waterproofing agent. The moisture diffusion in different sections of the mortar during the water absorption process is obtained from the NMR test results and two function models. Comparative studies indicate that the NMR test results have good correlation with the capillary water absorption test results and provide accurate process data. The NMR analysis results offer a new analytical method to characterize the porosity of cement-based materials by continuously monitoring small-pitch sections.

Keywords: moisture diffusion; nanomaterials; waterproofing agent; cement-based materials; NMR

1. Introduction

As a complex porous medium containing solid, gas, and liquid phases, concrete is vulnerable to several types of damage, with water content being one of the main influencing factors [1,2]. Several deleterious substances like chloride can enter cement-based materials through moisture diffusion. Glanville studied the permeability of concrete in the 1930s [3]. The Feldman Sereda model suggests that only free water (capillary water), adsorbed water, and structural water (interlayer water) can transport inside cement, whereas crystal water cannot [4]. However, in reality, other than in underwater engineering applications, concrete material rarely reaches a saturated state wherein its internal structure is full of liquid water. Considering the process of capillary flow, permeability cannot be used to suitably describe the transport of water in unsaturated porous materials, and gas phase transportation must be considered [5,6]. The total transmission effect is represented by Darcy's law, which considers the water saturation degree to be a descriptive variable [5,7,8].

Analyzing the moisture content in concrete by conventional methods is difficult. Wormald and Britch were the first to evaluate the moisture content in construction materials using neutron and gamma-ray diffraction [9]. Subsequently, accurate and non-destructive experimental methods, such as the hygrometer method [10], wave propagation [11–13], thermal transmittance testing [14–16], electrical property testing methods [17,18], and nuclear magnetic resonance (NMR) [19–22], have gradually been developed. However, each of these methods has its own disadvantages. The gamma-ray test method requires a dry sample and cannot be used for on-site or in-situ testing. The hygrometer method requires

many test holes to be prepared on the sample and the accuracy is not high. NMR technology is a fast, accurate, non-destructive, and continuous measurement method. Moreover, samples can be tested without drying or injecting liquids. Consequently, NMR technology can be used for in-situ detection and continuous monitoring. It measures the relaxation behavior of hydrogen protons in water molecules inside the material to obtain information such as the state of water, total moisture content [19,20], and distribution of water in the pores [21–23]. Nunes et al. analyzed NMR- T_2 relaxation to obtain pore water distribution at different distances from the material interface [24]. Stelzner et al. found the reconfiguration process of moisture from smaller gel pores to larger interhydrate pores by ^1H -NMR—relaxometry [25].

Studying the moisture transfer process of ordinary concrete is insufficient. In practical engineering applications, various waterproofing agents are added to concrete to prevent capillary action and reduce water absorption or water permeability under hydrostatic pressure and achieve a waterproof state. Based on their function, waterproofing agents are divided into three categories: (i) thin-coating surface forming additives, (ii) concrete pore lining forming additives, and (iii) pore blocking additives, all of which have good water resistance [26–30]. In addition to waterproofing agents, novel admixtures called nano-materials that have good physical and chemical properties have also been used in cement-based materials. The most common types of nano-materials are nano silica, nano clay, and nano alumina. Nano silica can improve the cement microstructure of the interfacial transition zone and increase the density of micro pores [31–33]. The resulting cement has higher strength, better durability, and improved mechanical properties [34,35]. Nano clay can enhance chloride penetration resistance in ultra-high-performance concrete [36]. It also improves thermal behavior and increases the compressive and tensile strength of cement [37–39]. Nano alumina can accelerate the hydration process by providing a path for the silica component to penetrate the internal structure of the hydration gel [40–42].

Although several studies have analyzed the advantages of waterproofing agents and nano-materials independently, few have analyzed cement-based materials that incorporate both. In particular, the influence of nano-materials on the water-absorption of cement-based materials with waterproofing agents has not been studied. This is primarily because current technologies can only measure internal water distribution and cannot accurately measure the changes in mass or density in various parts of the material. We used an advanced NMR technology to determine the water content in different sections of concrete. In addition, the capillary water absorption test was conducted to study the effect of a Krystol internal membrane (KIM) waterproofing agent and nano- SiO_2 (NS) on the water-absorption process of cement-based materials.

2. Materials and Methods

2.1. Materials and Samples

In this study, P.O. 42.5 cement manufactured by Anhui Conch Cement Co., Ltd. (Wuhu, China) (see Table 1 for composition), and ISO standard sand procured from Xiamen Aisiou Standard Sand Co., Ltd. (Xiamen, China) were used to prepare cement mortar samples. The nano material used herein was VK-S01B nano- SiO_2 (NS) dispersion material manufactured by Hangzhou Wanjiang New Material Co., Ltd. (Hangzhou, China); its performance characteristics are shown in Table 2. Table 3 provides the general information of the KIM waterproofing agent from The Kryton Group of Companies (Vancouver, BC, Canada), which was used as the waterproofing agent. A polycarboxylic acid superplasticizer (PC) manufactured by Jiangsu Zhaojia Building Materials Technology Co., Ltd. (Suzhou, China) was used, with a water reduction ratio of 32%. More details about the samples can be found in [43]. The mix proportion of the mortar is shown in Table 4.

Table 1. Composition of cement.

CaO/SiO ₂	C3S + C2S (%)	SO ₃ (%)	MgO (%)	Chloride (%)	Active Mixed Materials (%)
≥2.2	≥75	≤3.5	≤5.0	≤0.06	>5 and ≤20

Table 2. Performance characteristics of nano-SiO₂.

Type	Exterior	Average Particle Size (nm)	Content (%)	Solvent	pH
VK-S01B	Translucent liquid	13	30.1	Water	10.2

Table 3. General information of KIM waterproofing agent.

Color	Texture	Particle Size (μm)	Relative Bulk Density (g/cm ³)	Specific Gravity
Gray	Powder	40–150	≈1.4	≈2.8

Table 4. Mix proportion of cement mortar (g).

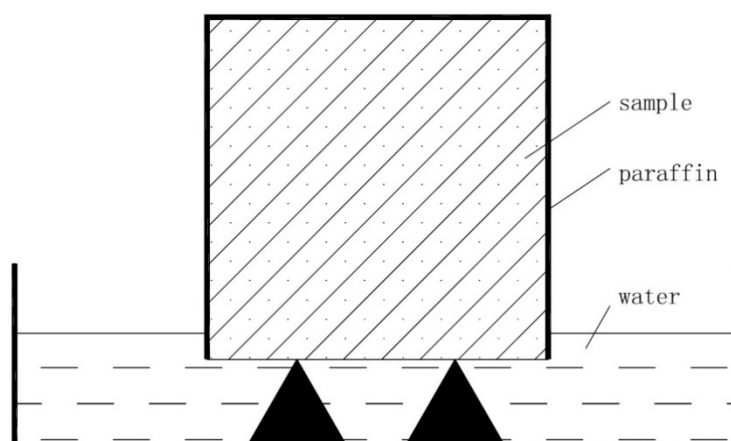
Group	Cement	Water	Sand	KIM	NS	PC
J	450	225	1350	0	0	0
K	450	225	1350	9	0	0.2
KS	450	225	1350	9	6.75	0.8

Note: The dosage of PC was adjusted to maintain the fluidity at about 160 mm.

2.2. Experimental Methods

2.2.1. Capillary Water Absorption Test

Two cube specimens with a side length of 70.7 mm were prepared according to the mixing ratio of each group and maintained in a curing room at a temperature of 20 ± 2 °C with a relative humidity of more than 95% for 28 days. The average of the test results of the two specimens was considered to be the absorption mass per unit area of each group at different intervals of time. Once the test specimens had dried to a constant weight at 105 °C, five faces of each sample were sealed using paraffin, and the remaining surface (bottom surface) was reserved as a water absorption surface (see Figure 1). The original mass M_0 (g) of all the test samples was recorded before the test. The water level was maintained at about 3–5 mm above the water absorption surface, the ambient temperature was 20 ± 2 °C, and the relative humidity was about $75\% \pm 5\%$. The mass M_t (g) of each specimen was recorded at regular intervals until it stabilized.

**Figure 1.** Schematic diagram of capillary water absorption test.

2.2.2. NMR-Based Stratification Saturation Test

The test instrument was the MesoMR60 manufactured by New Ma Electronic Technology Co., Ltd. (Suzhou, China), with a resonance frequency of 23.315 MHz, magnetic strength of 0.50 T, and magnet

temperature of 32 °C. The processing procedure of the test specimens was the same as the capillary water absorption test. Standard samples were used to calibrate the machine before the test. During the experiment, the cement mortar samples were immersed in water and placed in the probe coil to detect the y -direction signal. The MSE imaging sequence was used to record the signal values. The measuring time intervals were 1, 4, 9, 25, 49, and 81 h. After 81 h, all samples were vacuum-saturated and the signal values were recorded under saturated conditions.

2.3. Calculation Process

2.3.1. Capillary Water Absorption Coefficient

Capillary water absorption refers to the process wherein liquid phase water enters an unsaturated porous material due to capillary action. The capillary water absorption coefficient S ($\text{kg}/(\text{m}^2 \cdot \text{h}^{1/2})$) is an important parameter of the capillary water absorption process and represents the rate of water absorption by the porous material under capillary action. In the early 1980s, Hall et al. [44,45] carried out numerous experiments on capillary absorption in bricks and rocks, and concluded that the water absorption of the test samples had a linear relationship with the square root of the duration of water absorption. This ratio is the capillary water absorption coefficient S . Subsequently, after numerous experiments, the following formula was proposed [45]:

$$i = S \sqrt{t} + a \quad (1)$$

where i is the cumulative water absorption per unit area, S is the capillary water absorption coefficient, t is the duration of water absorption, and a is the intercept on the y -axis.

2.3.2. Expression and Empirical Formula of Water Diffusion Coefficient

When saturation is used as the descriptive variable, the total transmission effect of the isotropic porous material with gaseous water and liquid water can be expressed by the Richards equation [46]:

$$\frac{\partial \theta}{\partial t} = \nabla(D(\theta)\nabla\theta) \quad (2)$$

where θ is the saturation of the material and $D(\theta)$ is the moisture diffusion coefficient that is mathematically related to θ . Saturation can be obtained from the volumetric water content Θ :

$$\theta = \frac{\Theta - \Theta_i}{\Theta_s - \Theta_i} \quad (3)$$

where the subscripts i and s denote the initial unsaturated state and fully saturated state, respectively.

Generally, the one-dimensional diffusion equation is used to describe the total transmission effect. Considering the initial and boundary conditions:

$$\begin{cases} x = 0, & t > 0, & \Theta = \Theta_s \\ x \geq 0, & t = 0, & \Theta = \Theta_i \\ x \rightarrow \infty, & t > 0, & \Theta = \Theta_i \end{cases} \quad (4)$$

Equation (2) can be simplified to [5,19]:

$$\frac{\partial \theta}{\partial t} = \frac{\partial}{\partial x} \left(D(\theta) \frac{\partial \theta}{\partial x} \right) \quad (5)$$

where x denotes the distance from the inflow surface at which water is applied.

Considering one-dimensional diffusion with constant boundary saturation and an infinite half-plane, the Boltzmann transformation can be used to obtain the analytical expression of the water diffusion coefficient. Substituting the Boltzmann variable $\varphi = \frac{x}{t^{1/2}}$ into Equation (4), we obtain:

$$-\frac{1}{2}\varphi\left(\frac{d\theta}{d\varphi}\right) = \frac{d}{d\varphi}\left(D(\theta)\frac{d\theta}{d\varphi}\right) \quad (6)$$

where $\varphi(\Theta = \Theta_i) = \infty, \varphi(\Theta = \Theta_s) = 0$.

The expression for $D(\theta)$ can be written as:

$$D(\theta) = -\frac{1}{2}\frac{d\varphi}{d\theta}\int_0^\theta \varphi d\bar{\theta} \quad (7)$$

Considering the form of $D(\theta)$, calculating the above equation is difficult. Therefore, a relatively simple form of the water diffusion coefficient is required. Currently, the most common formulae are the exponential function and the power function, which are as follows [47,48]:

$$\text{Exponential function } D(\theta) = D_0 e^{n\theta} \quad (8)$$

$$\text{Power function } D(\theta) = D_0 \theta^n \quad (9)$$

where D_0 is the diffusion coefficient of completely dry concrete and n is the empirical constant. According to [49], for concrete materials, n is generally equal to 6.

2.3.3. Relationship between Capillary Water Absorption Coefficient and Water Diffusion Coefficient

In addition to Equation (1), the cumulative water absorption of the test sample can be calculated by the distribution law of water content as follows:

$$i = \int_{\Theta_i}^{\Theta_s} x d\Theta = t^{1/2} \int_{\Theta_i}^{\Theta_s} \varphi d\Theta \quad (10)$$

Then, the capillary absorption coefficient S can be expressed as:

$$S = \int_{\Theta_i}^{\Theta_s} \varphi d\Theta = (\Theta_s - \Theta_i) \int_0^1 \varphi d\Theta \quad (11)$$

Parlange et al. [50] proposed a high-precision approximate analytical solution for the nonlinear diffusion Equation (6) as follows:

$$2 \int_\theta^1 \frac{D(a)}{a} da = s\varphi + \frac{A}{2}\varphi^2 \quad (12)$$

where s is the relative water absorption ratio that can be calculated from the capillary water absorption coefficient S as:

$$s = S/(\Theta_s - \Theta_i) \approx \left(\int_0^1 (1 + \theta)D(\theta)d\theta\right)^{1/2} \quad (13)$$

and A is given by:

$$A = 2 - \frac{s^2}{\int_0^1 D(\theta)d\theta} \quad (14)$$

After substituting Equation (8) into Equation (13), we can obtain Equation (15), which was proposed by Lockington et al. [51].

$$D_0 = \frac{n^2 s^2}{e^n (2n - 1) - n + 1} \quad (15)$$

It is also possible to calculate the expression of the coefficient A as:

$$A = \frac{e^n n^{-1} - 1 - n^{-1}}{e^n - 1} \quad (16)$$

For power,

$$D_0 = \frac{s^2 (1 + n)(2 + n)}{3 + 2n} \quad (17)$$

and

$$A = 2 - \frac{(1 + n)s^2}{D_0}. \quad (18)$$

3. Results and Discussion

3.1. Capillary Water Absorption Test Analysis

The relationship between the cumulative absorption per unit area and the square root of the capillary absorption time was obtained from the results of the capillary water absorption test. The results are illustrated in Figure 2.

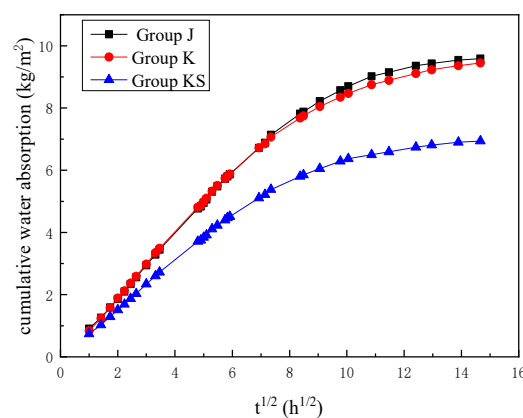


Figure 2. Capillary water absorption test

As shown in Figure 3, the linear correlation between the water absorption mass per unit area and the time square root of each test group was strong, with a correlation coefficient above 0.999. This result agrees with the theoretical derivation. The capillary water absorption coefficient of group K was slightly smaller than that of group J. The difference between the two groups was discernible, which indicated that the waterproof effect of the KIM waterproof agent during the initial stage of capillary water absorption is not obvious. The capillary water absorption coefficient of group KS was significantly lower than that of groups J and K. This indicated that nano-SiO₂ causes crystal nucleation and micro-aggregation, which considerably improved the compactness of the test samples. The modification effect of nano-SiO₂ in the cement mortar with waterproof agent was significant, and the capillary water absorption rate was slower.

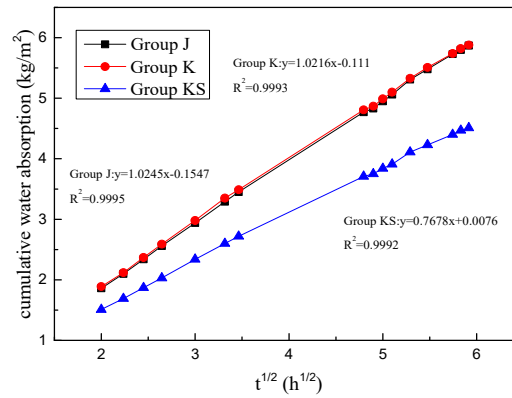


Figure 3. Fitting result of early stage.

According to the linear expression obtained from the fitted curve, the capillary water absorption coefficients of the J, K, and KS groups were 1.0245, 1.0216, and 0.7678 (kg/m²)/h^{1/2}, respectively.

3.2. NMR Saturation Test Analysis

Before using NMR technology to visually obtain the internal moisture distribution of the material, calculation and simulation were carried out according to Equation (12) to predict the saturation at a long distance from the water absorption surface.

From the root formula, the value of φ can be expressed by Equation (19):

$$\varphi = \frac{-s + \sqrt{s^2 + 4A \int_{\theta}^1 \frac{D(a)}{a} da}}{A} \quad (19)$$

Then, the relationship between the coordinate x of a point in the test sample and its relative water content is:

$$x = \varphi \sqrt{t} = \frac{-s + \sqrt{s^2 + 4A \int_{\theta}^1 \frac{D(a)}{a} da}}{A} \sqrt{t} \quad (20)$$

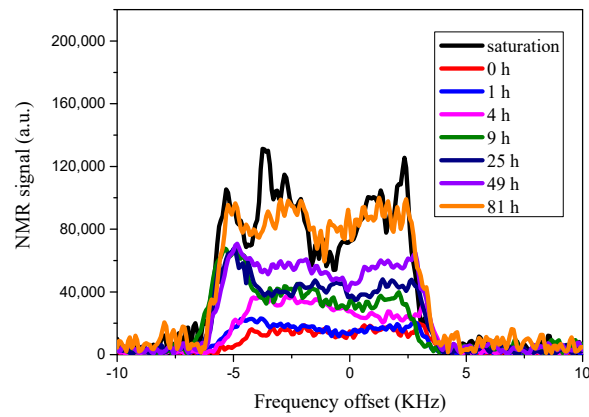
In the first half, the saturation of the function gradually decreases with distance, whereas in the second half, it decreases rapidly. The curve of the function is parabolic. The data in [49] also perform well with this function. However, there is a limitation in formula simulation. In the prediction of Equation (20), x has a maximum value. When the distance exceeds the maximum value, its saturation defaults to 0.

The NMR technique uses the MSE sequence to perform spatial encoding by reading the gradient. The acquired data images are shown in Figure 4.

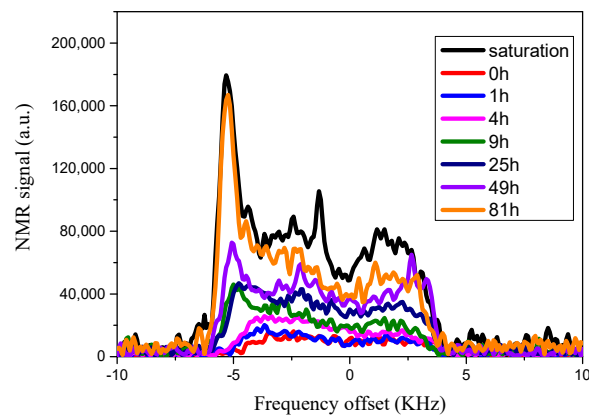
After the cement mortar samples were saturated with water, the saturation was set to 100%, while that at 0 h was set to 0%. Then, the saturation curve as a function of the spatial position was calculated from the signal value ratio.

As shown in Figure 5, the internal moisture distribution of the material obtained by the NMR technique is significantly different from that obtained by the formula calculation simulation. Moreover, the curve does not show a downward trend as the water absorption distance increases. Although the magnitude of the nuclear magnetic signal on the ordinate cannot be directly substituted into the calculation, it can be used to reflect the quality of pure water in the sample. After fitting, the relationship between the semaphore and pure water quality was roughly a function. As the test conditions changed, the scale factor changed as well. Here, the unknown coefficient was defined as γ and was retained, not specifically calculated. From signal value of 0 h and saturated state, the water content $\Theta_s(\gamma)$ in the saturated state of each test group was calculated. The capillary water absorption coefficient S of each group and the water content $\Theta_s(\gamma)$ in the saturated state of each group were

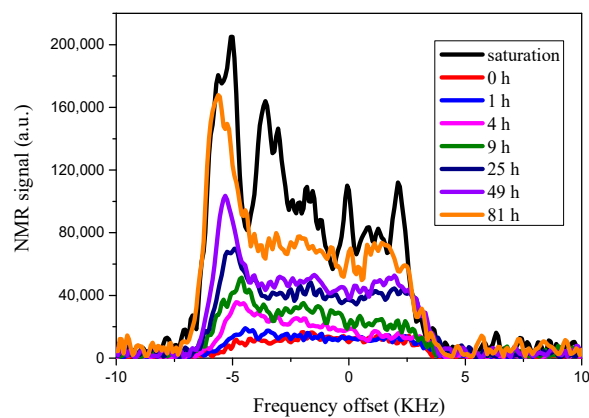
substituted into Equation (13) to obtain s . Subsequently, the D_0 of the exponential function and power function were obtained using Equations (15) and (17), respectively (see Table 5).



(a) Group J

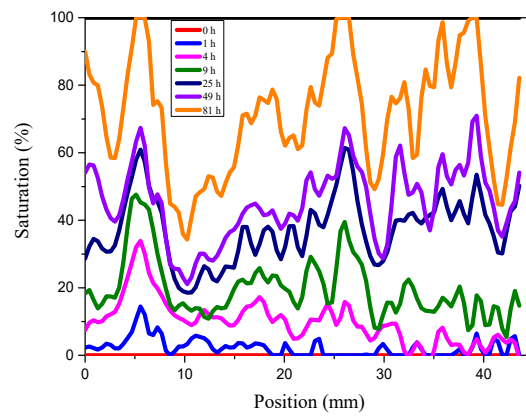


(b) Group K

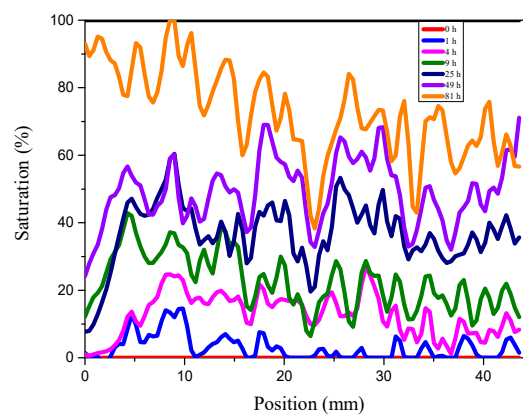


(c) Group KS

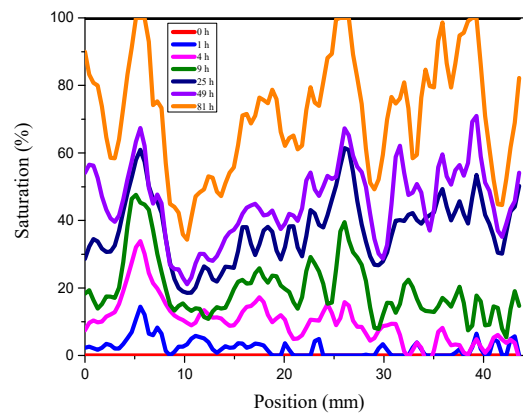
Figure 4. Signal values of the water absorption process obtained by the NMR test.



(a) Group J



(b) Group K



(c) Group KS

Figure 5. Saturation of water absorption process.**Table 5.** D_0 values of exponential and power functions.

Group	Capillary Water Absorption Coefficient S	Saturated Water Content $\Theta_s(\gamma)$	Exponential D_0	Power D_0
J	1.02454	10.22809899	8.15×10^{-5}	0.037460
K	1.02159	9.692812897	9.02×10^{-5}	0.041472
KS	0.76777	12.742302	2.95×10^{-5}	0.013554

Substituting the saturation value calculated from the signal value obtained in the NMR test into Equations (8) and (9), the water diffusion coefficient $D(\theta\gamma)$ at each depth section at different time intervals was obtained, as shown in Figures 6 and 7.

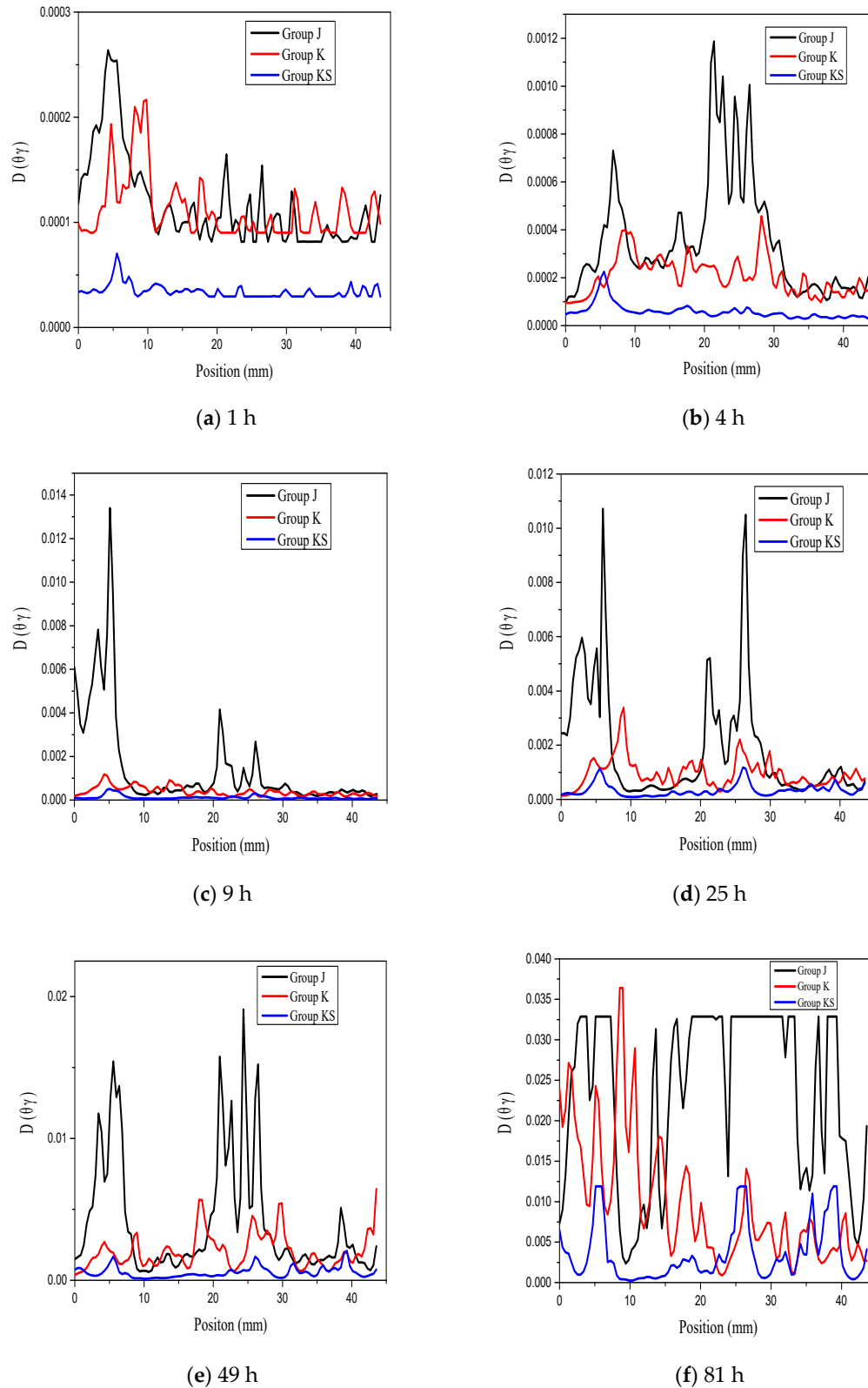
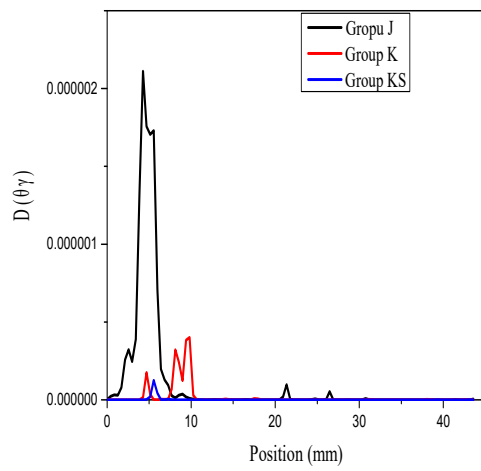
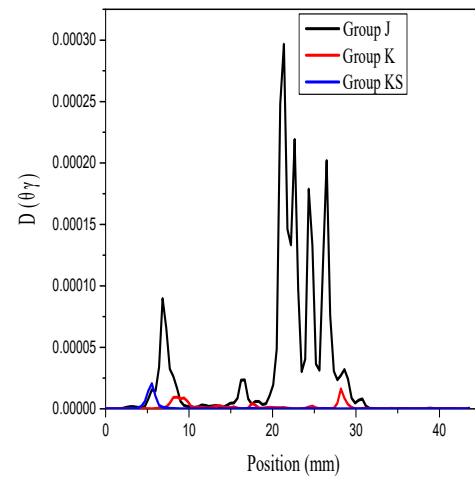


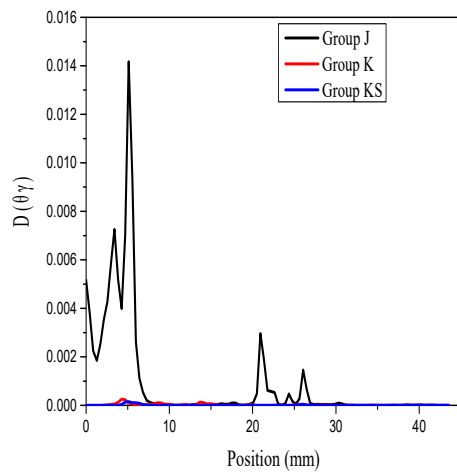
Figure 6. Comparison of the exponential $D(\theta\gamma)$ at different time intervals.



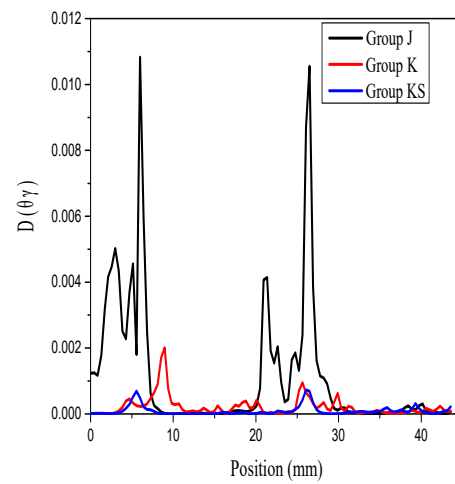
(a) 1 h



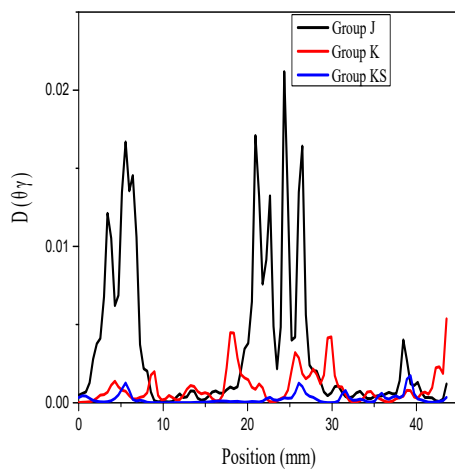
(b) 4 h



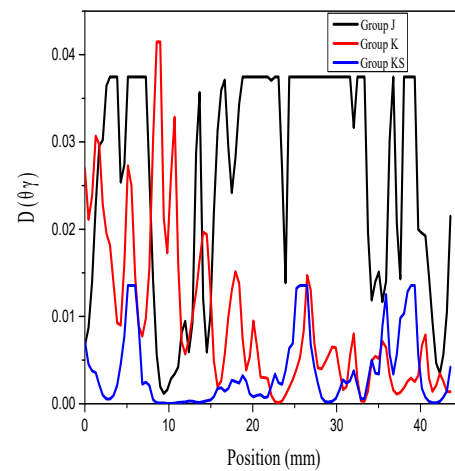
(c) 9 h



(d) 25 h



(e) 49 h



(f) 81 h

Figure 7. Comparison of the power $D(\theta\gamma)$ at different time intervals.

Figures 6 and 7 indicate that the amount of water absorption in the concrete and the water diffusion coefficient $D(\theta\gamma)$ increase. The water diffusion coefficient curves of group J are above the curves of groups K and KS, which indicate that the water diffusion capacity of group J was relatively strong. This implies that the impermeability of cement-based materials is improved by the addition of a waterproofing agent or a combination of a waterproofing agent and nano-SiO₂. Comparing the curves of groups K and KS, the water diffusion coefficient of group KS was significantly lower than that of group K at each section position, which suggested that after nano-SiO₂ is added to the mixture, the cement mortar structure is more uniform and denser than that with only the waterproofing agent. Moreover, there were fewer connected pores, which weakened the water diffusion ability.

$D(\theta\gamma)$, the water diffusion coefficient, is a function that describes the strength of water transport and is positively correlated to porosity. Consequently, the number of pores in the section where the peak surface appears can be assumed to be high. Comparing the curves of group J at 1 and 4 h, as shown in Figure 7, a new peak appeared at 20–30 mm; at 49 h, another new peak appeared near 40 mm. Essentially, as the test time increases, the surface moisture continuously spreads into the material. When it is transmitted to a relatively loose section, it appears as a more obvious peak on the curve. Therefore, by observing the position and time of each peak in the graph, the distribution of the sample density can be evaluated and the approximate depth of water infiltration can be determined. The group K curve also followed a similar law. However, the undulation of the curve was small, which implied that the waterproofing agent effectively improved the compactness of the cement mortar. The group KS curve showed almost no obvious peak during the entire test period, which indicated that nano-SiO₂ further improved the pore distribution of the cement mortar sample and reduced the pore size.

The average water diffusion coefficient $D(\theta\gamma)$ of each group exhibited three curves—high, medium and low—related to the degree of modification of the material, as shown in Figure 8. Although the initial waterproofing effect of the KIM waterproofing agent was not obvious, the test sample had lower saturation during the initial stage of the water absorption test, and the curve differed from the capillary water absorption fitting curve. However, as the test proceeded, the effect of the waterproofing agent was more pronounced and, consequently, the growth rate of the curve was lower than that of group J. The presence of nano-SiO₂ further increased the waterproofing effect in group KS, and the curve increased even more slowly than that of group K. Compared to Figure 2, the slope of the curve began to change after about 49 h of the capillary water absorption test, which indicated that the cumulative water absorption gradually entered a stable state and the water content of the test sample began to saturate. In Figure 8, the three curves have a noticeable transition at about 50 h, indicating that the internal saturation of the test samples changed significantly. This indicates that the water content was at a higher level, which confirms the results of the capillary water absorption test.

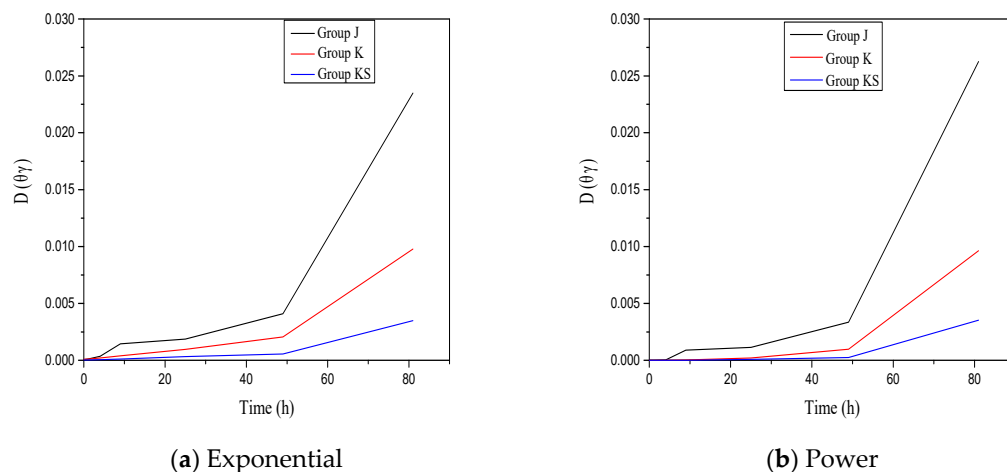


Figure 8. Relationship between $D(\theta\gamma)$ and time.

Figure 9 shows that the relationship between the water diffusion coefficient of the three test groups. The curves of groups J and K are very close, and the growth rate is significantly larger than that of group KS. This corroborates the curve fitting result of the capillary water absorption test (Figures 2 and 3). Overall, the results of the two tests are in good agreement with each other.

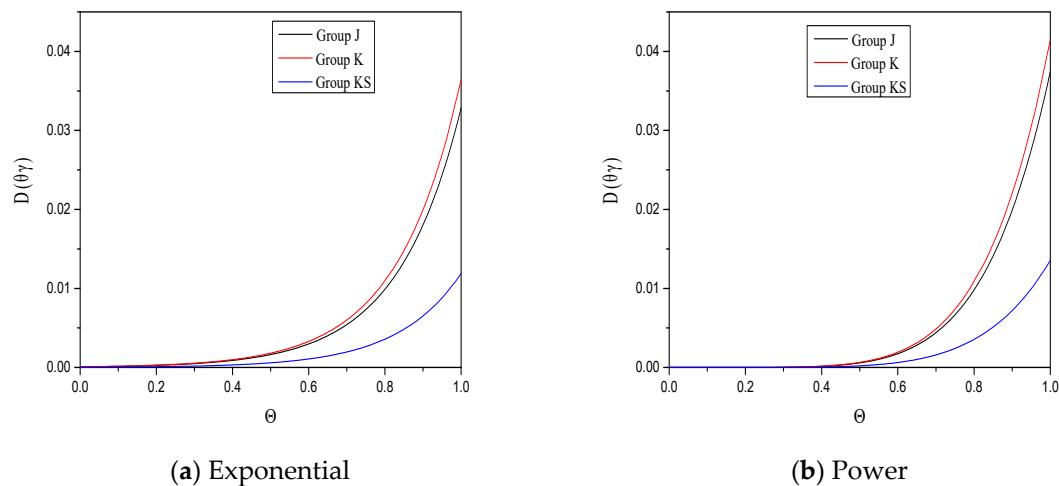


Figure 9. Relationship between $D(\theta\gamma)$ and saturation.

4. Conclusions

KIM waterproofing agent has no obvious effect on the capillary water absorption coefficient S , although it can slightly improve the impermeability of cement mortar.

Adding KIM waterproofing agent with nano-SiO₂ can significantly reduce the capillary water absorption coefficient S by 25% and improve the impermeability.

The presence of nano-SiO₂ helps to further enhance the effect of KIM waterproofing agent on reducing the water diffusion coefficient $D(\theta\gamma)$ and delay the process of water transportation in cement mortar.

The water diffusion coefficient $D(\theta\gamma)$ of cement mortar is affected by saturation level, which increases gradually at low saturation levels but increases rapidly at high saturation levels. Adding nano-SiO₂ can improve the saturation inflection point when the water diffusion coefficient $D(\theta\gamma)$ increases rapidly.

The depth of water infiltration can be estimated based on the time at which a peak occurs in the water diffusion coefficient curve and the compactness of the section can be inferred from the position of the peak. The relative results of adding nano-SiO₂ showed that the compactness of cement mortar could be significantly improved.

Author Contributions: Methodology, M.W.; Data curation, H.W. and M.W.; Validation, T.M.; Investigation, M.W.; Writing—original draft, H.W.; Project administration, T.M.; Writing—review & editing, H.W. and K.Y.; Supervision, T.M. All authors have read and agreed to the published version of the manuscript.

Funding: The authors want to thank the support by Zhejiang Key Research & Development Program (2020C04013) and National Natural Science Foundation of China (52078453).

Conflicts of Interest: The authors declare no conflict of interest.

Abbreviations

NS	Nano-SiO ₂
NMR	Nuclear magnetic resonance
KIM	Krystol internal membrane
PC	Polycarboxylic acid superplasticizer

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