Impacts of various factors on the rehydration of cement-based materials with a low water–binder ratio using mathematical models

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Abstract

When cement-based materials with a low water–binder ratio are exposed to water, the internal unhydrated cementitious components undergo rehydration. Rehydration tests of paste samples with a low water–binder ratio were conducted to determine the hydration degree under water immersion conditions. A modified rehydration model was developed based on a thermodynamic model for cement hydration and used to predict the hydration degrees of samples at different times. The modified rehydration model closely agreed with the test data. The ambient temperature significantly impacted material rehydration. The temperature effect on the rehydration reaction rate constant follows the Arrhenius law.

Keywords: rehydration; hydration degree; model; ultrahigh performance concrete; bound water

1 Introduction

With the continuous development of cement-based materials, ultrahigh strength and performance concrete has attracted widespread attention for practical applications. A low water–binder ratio (w/b) is necessary to obtain ultrahigh strength and performance. Generally, a lower water–binder ratios results in higher strength, lower hydration degree, lower generation of hydration gel, and more unhydrated cementitious material. According to the theory proposed by Powers [1], cement cannot be completely hydrated when the water–cement ratio is less than or equal to 0.38. For sealed conditions, the minimum water–cement ratio is 0.44 for the complete hydration of cement [2]. The water–cement ratio of ultrahigh performance concrete is commonly less than 0.38; the unhydrated cementitious material is a major factor that affects the long-term performance of concrete. In this paper, “rehydration” mainly refers to the hydration reaction between external water entering cement-based materials with a low water–binder ratio and the existing unhydrated cementitious materials. Yang and Guan [3] and Guan et al. [4] indicated that the rehydration of concrete with a low water–cement ratio could lead to concrete damage, an obvious decline in the compressive strength, and poor durability. A lower water–cement ratio increases the damage generated by the rehydration of unhydrated cementitious materials. Their specimens with water–cement ratios of 0.22, 0.25, 0.28, 0.31, and 0.47 all showed a decline in strength after 7 d of hydration, which was accelerated by high-temperature water. In particular, the specimen with a water–cement ratio of 0.22 exhibited a decrease in strength of 21.0%. However, it has also been suggested that once moisture enters concrete cracks, the hydration products from the rehydration reaction of unhydrated cementitious materials tends to repair these cracks to a certain extent. Yao et al. [5] suggested that the self-healing process of damaged concrete essentially involves the hydration of unhydrated cementitious materials at damaged locations and the generation of new hydration products to bridge the cracks. Zhu et

al. [6] studied the change in the loading performance of damaged engineered cementitious composite (ECC) materials after freeze–thaw cycles. Their results demonstrated that the rehydration reaction during the freeze–thaw cycles can help heal the cracks generated during preloading to a certain extent. Huang et al. [7, 8] showed that the rehydration of unhydrated cement can repair microcracks and thus help repair the damage to concrete. Sahmaran et al. [9] investigated the chloride permeability of concrete damaged during preloading under the three curing conditions of water, air, and freeze–thaw cycle. Their results suggested that unhydrated cementitious materials have significant effects on the self-healing of concrete cracks. Granger et al. [10] prefabricated cracks by utilizing the three-point loading method and subsequently studied the changes in the bending strength and integral stiffness of the damaged concrete after water immersion. Although the product of a calcium–silicate–hydrate (CSH) gel generated from unhydrated cementitious materials during the rehydration reaction from immersion in water may differ from the CSH gel generated in the early hydration process, the values of the stiffness are close.

The hydration process of cement is highly complex. Mathematical models describing the hydration process have long been a popular topic of research. Various approaches to hydration models have been developed. For example, numerical models [11] calculate the product composition of cement hydration from the chemical equations of the reaction between each component of cement and water. Mathematical expression models establish the relationship between the hydration rate and the hydration degree based on the bound water, the variation in the hydration heat, etc. Examples include the model of three basic processes proposed by Krstulovic and Dabic [12] and the shrinking-core model proposed by Tomosawa [13]. Although the rehydration process has a significant impact on the long-term performance of concrete, there have been few studies related to rehydration models. Huang et al. modeled the rehydration of unhydrated cement on the basis of the hydration of single clinker

components and nuclear magnetic resonance (NMR) tests [14]. The results showed that the hydration degree of the cement paste increased from 67% to approximately 82%, which was much higher than that for the case without extra water. Moreover, the capillary porosity of the paste adjacent to the crack surfaces decreased to less than 4%.

In this study, rehydration tests of paste samples with low water–binder ratios were performed to examine the effects of silica fume, the curing conditions, and the ambient temperature on the rehydration degree. The rehydration degree refers to the increase in the hydration degree of specimens that were placed in water. In addition, it can be calculated by the difference between the hydration degree after rehydration and the hydration degree before rehydration. The variation in the compressive strength of undamaged ultrahigh performance concrete after natural immersion was measured, and the potential impacts of rehydration on the macroscopic performance were analyzed. These results were used to develop a rehydration model. The above contributions should provide a reference for research on the long-term performance of cement-based materials with low water–binder ratios in water.

2 Experiments

2.1 Mixing ratio

In this test, 42.5# ordinary Portland cement (OPC) was adopted; the C_3A , C_4AF , C_2S , and C_3S contents were 7.4%, 8.9%, 18.1%, and 60.5%, respectively. The microsilica fume contained more than 90% SiO_2 with an average diameter of 0.31 μm . The superplasticizer had a water-reducing rate of 29% and a solid content of 31%. The three diameter ranges of quartz sand particles were 0.16–0.315 mm, 0.315–0.625 mm, and 0.625–1.25 mm with a mixing ratio of 1:4:2. The tensile strength, diameter, and length of the short thin steel fibers were 2800 MPa, 0.22 mm, and 13 mm, respectively. Six different mixing ratios were used in this test, as presented in Table 1. The concrete sample with the mixing ratio LWB0 had dimensions of 100 mm $\times$ 100 mm $\times$ 100 mm and was used to determine the compressive

strength. Paste samples with other mixing ratios had dimensions of R10 mm $\times$ 20 mm and were used to measure the bound water.

Table 1 Mixing ratios in tests

Mix.	Cement	Microsilica fume	Quartz sand	Steel fiber	Superplasticizer	Water	Water–binder ratio
LWB0	0.815	0.185	1.449	0.185	0.085	0.141	0.20
LWB1	0.815	0.185	0.000	0.000	0.085	0.091	0.15
LWB2	0.815	0.185	0.000	0.000	0.085	0.141	0.20
LWB3	0.815	0.185	0.000	0.000	0.085	0.191	0.25
LWB4	0.815	0.185	0.000	0.000	0.085	0.240	0.30
LWB5	1.000	0.000	0.000	0.000	0.085	0.141	0.20

2.2 Test methods

The molds of the formed samples were removed after 24 h of curing at room temperature. Then, the samples were placed in a steam-curing box at 75 °C for another 72 h of curing; both the heating and cooling rates were maintained at 15 °C/h. The samples were finally placed and stored in a standard curing room (T = 20 °C and RH > 95%) for curing for up to 28 d. When the curing was finished, the samples were immersed in water for the rehydration tests. The samples were removed for measurements after a certain period of time and then immersed in acetone to suspend the hydration reaction.

The bound water was measured by the loss-on-ignition method at a calcination temperature of 950 °C. The amounts of bound water were measured in samples immersed for 0, 7, 14, 28, 56, 90, and 180 d. The results were used to develop a rehydration model and analyze the effects of the silica fume, water–binder ratio, and ambient temperature on rehydration. In order to further analyze the impact of rehydration on the performance of ultrahigh performance concrete, a natural immersion test of the LWB0 concrete sample was conducted in an

immersion environment at 20 °C. After a certain immersion time, the compressive strength was measured.

3 Kinetic model of the hydration of cement-based material

The kinetic model of the hydration reaction proposed by Krstulovic and Dabic [12] describes the relationship between the hydration degree of cement and the reaction time. In general, the early stage of the hydration reaction is considered to be controlled by nucleation and crystal growth. The intermediate stage is controlled by a phase-boundary reaction, and the final stage is mainly regulated by diffusion. Eq. (1) expresses the model controlled by diffusion:

$$d\alpha / dt = 3 / 2k_3(1-\alpha)^{2/3} / [1-(1-\alpha)^{1/3}] \quad (1)$$

where α , t , and k_3 are the hydration degree, rehydration time, and hydration reaction rate constant, respectively. k_3 ignores the impact of the particle diameter but is affected by factors including, the hydration conditions, etc.

Compared with cement hydration, rehydration mainly differs in two aspects. First, rehydration occurs in hardened cement, and unhydrated cement particles may be surrounded by the gel generated during the initial hydration. Second, the water for the rehydration reaction penetrates from the outside. Therefore, the rehydration reaction is primarily dominated by diffusion, and the rehydration process can be described by a model in which k_3 is the rehydration reaction rate constant. However, the rehydration model does not include parameters that measure the diffusion rate. Hence, the effective diffusion coefficient of water in the CSH gel D_e was introduced to further modify the model for the diffusion process. D_e is essentially affected by the hydration degree and satisfies the equation $D_e = D_{e0} \ln(1/\alpha)$ [13, 15]. Moreover, the ambient temperature of the rehydration influences the chemical reaction rate and the state of the reaction products. To consider the impacts of temperature, a new rehydration model was developed, where k_3 is replaced by k_r , expressed as follows:

$$d\alpha / dt = 3 / 2k_r(1-\alpha)^{2/3} / [1-(1-\alpha)^{1/3} D_{e0} \ln(1/\alpha) / r_0] \quad (2)$$

where α , t , k_r , and r_0 are the hydration degree, the rehydration time, the rehydration reaction rate constant, and the diameter of the unhydrated cement particles, respectively. D_{e0} is the effective diffusion coefficient of water in a CSH gel prior to rehydration and is influenced by factors such as the water–cement ratio and mineral admixture. The parameter k_r in Eq. (2) varies with the temperature and satisfies the Arrhenius law as follows:

$$k_r = k_{r20} \exp(-\beta(\frac{1}{T} - \frac{1}{20})) \quad (3)$$

where k_{r20} is the value of k_r at 20 °C, and β is a constant relevant to the material itself [16].

4 Analysis of the factors affecting the rehydration degree

Bound water is usually adopted to evaluate the hydration degree of concrete. According to the amount of bound water after complete hydration, the hydration degree of a sample can be calculated. Thus, the rehydration model can be built by measuring the hydration degree of rehydrated samples.

4.1 Effects of silica fume on the rehydration degree

Fig. 1 illustrates the variation in the hydration degree of samples with silica fume (LWB2) and without silica fume (LWB5). The hydration degrees of the samples increased with the immersion time relatively quickly in the early stage and slowly afterwards. After 180 d, the hydration degree of the sample with silica fume increased from 0.443 to 0.498 for an increase of 0.055 (12.43%). The hydration degree of the sample without silica fume increased from 0.353 to 0.420 for an increase of 0.067 (18.84%). The addition of silica fume can trigger a pozzolanic reaction that consumes the CH generated during the cement hydration process as well as the gel water but not the bound water [17]. According to the principle of equilibrium, this promotes cement hydration to a certain extent. Therefore, it can be concluded that silica fume partly accelerates the hydration reaction, and the initial hydration degree of the sample with silica fume was significantly higher than that without silica fume. However, because the sample without silica fume had a low initial hydration degree, it had a large quantity of

unhydrated cement particles and exhibited a more significant increase in the hydration degree during the rehydration process.

Fig. 1 Effect of silica fume on the hydration degree (w/b=0.20)

4.2 Effects of the water–binder ratio on the rehydration degree

Fig. 2 shows the hydration degree curves of four samples with various water–binder ratios. The water–binder ratio influenced the rehydration degree of the sample as well as the initial hydration degree. The hydration degrees of samples with water–binder ratios of 0.15, 0.20, 0.25 and 0.30 increased from 0.389, 0.443, 0.465, and 0.531 to 0.442, 0.498, 0.509, and 0.575 when immersed for 180 d for increases of 0.052, 0.05507, 0.045, and 0.044, respectively. This suggests that the rehydration degree increases and then decreases as the water–binder ratio increased. The rehydration degrees were relatively close between the samples with water–binder ratios of 0.15 and 0.20 and between the samples with water–binder ratios of 0.25 and 0.30. The increases in the hydration degree were 13.48%, 12.43%, 9.70%, and 8.28%, respectively; thus, it gradually decreased as the water–binder ratio increased. At a low water–binder ratio, even when unhydrated cementitious materials were abundant, the rehydration degree was primarily affected by water owing to the dense internal structure of the sample and the slow water transport and diffusion. As the water–binder ratio increased, the internal structural density of the sample decreased, the water transport and diffusion increased, and the rehydration degree substantially improved. However, a higher water–binder

ratio means that there is less unhydrated cementitious material within the sample and a lower rehydration degree. Thus, rehydration is mainly influenced by unhydrated cementitious materials.

Fig. 2 Effect of the water–binder ratio on the hydration degree

4.3 Effects of the ambient temperature on the rehydration degree

To consider the effect of the ambient temperature on the rehydration process, samples were placed in thermostatic water tanks at temperatures of 20, 40, and 60 °C to observe the variations in the hydration degree for different immersion times, as shown in Fig. 3. The hydration degrees of all samples increased over time. The ambient temperature showed a remarkable effect on rehydration. With 90 d of immersion, the hydration degrees of the samples at ambient temperatures of 20, 40, and 60 °C increased by 0.044 (9.87%), 0.112 (25.22%), and 0.126 (28.32%), respectively. Thus, the rehydration degree clearly increased as the temperature increased compared with the values before immersion. At 20 °C, the hydration degree significantly increased at 0–14 d, whereas it only slightly increased at 14–90 d. At 40 °C, the variation in the hydration degree at 0–14d resembled that at 20 °C, whereas the hydration degree linearly increased at 14–90 d. At 60 °C, the increase in the hydration degree significantly decreased at 56–90 d.

Fig. 3 Effect of the ambient temperature on the hydration degree (w/b=0.20)

5 Modeling rehydration

5.1 Rehydration model

The test results were analyzed by using Eq. (1). Tables 2 and 3 present the analysis results. All of the correlation coefficients were greater than 0.95, which indicates that the proposed rehydration model based on cement hydration can be applied to describe the rehydration process with and without silica fume. The effects of the silica fume, water–binder ratio, and ambient temperature can all be represented by k_3 . The value of k_3 increased when silica fume was added to a sample. There were no remarkable variation patterns for k_3 in samples with different water–binder ratios; the smallest and largest values were observed for samples with water–binder ratios of 0.15 and 0.30, respectively. The value of k_3 substantially increased as the ambient temperature increased. Figs. 4–6 plot the rehydration curves according to the values of k_3 obtained by fitting the results described in section 4 using equation (1). In the early stage of rehydration, the hydration rate of the sample without silica fume was clearly higher than that with silica fume. However, for the same hydration degree, the rehydration rate of the sample with silica fume was greater than that without silica fume. The initial rehydration rate of the sample with a water–binder ratio of 0.20 was significantly higher than those with the other three water–binder ratios. The rehydration rate increased with the ambient temperature.

Table 2 Fitting results using the rehydration model for various water–binder ratios

Water–binder ratio	0.15	0.20		0.25	0.30
		With silica fume	Without silica fume		
$k_3 (\times 10^{-5})$	5.3488	8.1174	6.1877	6.6592	8.9310
Correlation coefficient	0.986	0.956	0.980	0.988	0.982

Table 3 Fitting results using the rehydration model for various ambient temperatures

Ambient temperature	20 °C	40 °C	60 °C
$k_3 (\times 10^{-5})$	12.6659	30.7097	43.6157
Correlation coefficient	0.972	0.984	0.931

Fig. 4 Effect of silica fume on the rehydration rate (w/b=0.20) **Fig. 5** Effect of the water–binder ratio on the rehydration rate

Fig. 6 Effect of the ambient temperature on the rehydration rate (w/b=0.20)

Yan and Zheng [18] presented a reaction rate constant for cement hydration during the diffusion process. This was used to plot the hydration reaction rate curves, which were then compared to the rehydration test results in this study, as shown in Fig. 7. The rehydration rate was far less than the initial hydration rate of cement with a difference of three orders of magnitude. Hence, for cement-based materials with low water–binder ratios, the rehydration of unhydrated cementitious components is rather slow, which would affect the long-term performance of concrete.

Fig. 7 Comparison of the proposed model and the model in [18]

5.2 Modified rehydration model

A modified rehydration model was employed for a fitting analysis of the test results. Tables 4

and 5 present the parameters obtained from fitting. The correlation coefficients were all higher than the corresponding ones in Tables 2 and 3. This suggests that the modified rehydration model better matched the test data compared with the rehydration model. Tables 4 and 5 indicate that an increase in the water–binder ratio had little effect on the variation in k_r . However, the values of k_r of the sample without silica fume were clearly greater than those with the silica fume in contrast to the rehydration model. k_r increased with the ambient temperature. Eq. (3) was employed to fit the test data of k_r in Table 5. The results are shown in Fig. 8. $\beta = 60.53$, and the correlation coefficient was 0.998; this indicates that the effect of the temperature on k_r in the rehydration process can be described by the Arrhenius law. D_{e0} increased with the water–binder ratio, as shown in Fig. 9; thus, the initial density of the sample decreased as the water–binder ratio increased, and the effectiveness of the rehydration model was further demonstrated to a certain extent.

Table 4 Fitting results of the modified rehydration model for various water–binder ratios

Water–binder ratio	0.15	0.20		0.25	0.30
		With silica fume	Without silica fume		
$k_r (\times 10^{-5} \text{ m/d})$	2.2803	3.9735	4.5169	2.3484	3.1109
$D_{e0} (\times 10^{-8} \text{ m}^2/\text{d})$	3.7454	4.4805	3.2587	4.8133	6.0916
Correlation coefficient	0.998	0.985	0.996	0.998	0.995

Table 5 Fitting results of the modified rehydration model for various ambient temperatures

Ambient temperature	20 °C	40 °C	60 °C
$k_r (\times 10^{-5} \text{ m/d})$	5.2416	22.5958	39.9616
$D_{e0} (\times 10^{-8} \text{ m}^2/\text{d})$	4.4805	4.4584	4.4700
Correlation coefficient	0.991	0.999	0.984

Fig. 8 Effect of the ambient temperature on k_r **Fig. 9** Effect of the water–binder ratio on D_{e0}

Figs. 10–12 plot the variation in the rehydration rate according to the fitted values. In contrast to the rehydration model curves, the modified rehydration model curves are clearly divided into two stages: the initial stage, where the rehydration rate rapidly declined, and the late stage, where the decrease in the rehydration rate slowed and eventually leveled off. The initial hydration degree of the cement sample without silica fume was low, whereas its initial rehydration rate was significantly higher than that with silica fume. The initial rehydration rate decreased as the water–binder ratio increased. Similar values were observed for samples with water–binder ratios of 0.15 and 0.20 and those of 0.20 and 0.30. A high temperature was found to accelerate the rehydration reaction. Therefore, the rehydration rate increased with the temperature. Fig. 13 compares the two models for the sample with a water–binder ratio of 0.20. At the beginning of rehydration, the hydration rate with the modified rehydration model was far greater than that with the rehydration model, but this lasted for a fairly short time. Afterwards, the hydration rate with the rehydration model was substantially higher than that with the modified rehydration model. This indicates that the modified rehydration model better matches actual situations.

Fig. 10 Effect of silica fume on the rehydration rate (w/b=0.20)

Fig. 11 Effect of the water–binder ratio on the rehydration rate

Fig. 12 Effect of the ambient temperature on the rehydration rate (w/b=0.20)

Fig. 13 Comparison of the diffusion and modified rehydration models (w/b=0.20)

5.3 Prediction of the rehydration degree

The rehydration degree gradually increased over time, whereas the rehydration rate continuously decreased. According to the modified rehydration model, the hydration degree of concrete can be calculated at any moment. Fig. 14 shows the variation in the rehydration degree over time for the sample with a water–binder ratio of 0.20 at an ambient temperature of 20 °C; this was calculated by using the modified rehydration model with MATLAB. The rehydration rate was relatively high in the first year and then decreased; a hydration degree of 0.73 was obtained in the 10th year.

Fig. 14 Curve for predicting the rehydration degree (w/b=0.20)

Fig. 15 shows the variation in the compressive strength of the ultrahigh performance concrete (LWB0) for natural immersion at 20 °C and the variation in the hydration degree over time. The compressive strength of the ultrahigh performance concrete tended to decrease as the

immersion time increased, which agrees with the research results of Guan et al. [4]. The compressive strength decreased to 128.62 MPa after 2 years of immersion for a decrease of 14.5%. The water–binder ratio of the ultrahigh performance concrete is usually low. Therefore, even under steam-curing conditions, its initial cementitious materials cannot experience complete hydration. During the immersion process, the penetration of water can provide the necessary conditions for rehydration. A cubic centimeter of cement occupies a volume of approximately 2.1 cm^3 after hydration [19] owing to the increase in the volume of the hydration products and the generation of a swelling pressure. In addition to the poor gelling properties of the rehydration products, the compressive strength is further reduced. Therefore, the effects of rehydrating unhydrated cementitious materials on the long-term performance of concrete with a low water–binder ratio cannot be neglected.

Fig. 15 Relationship between the compressive strength and the variation in the hydration degree (w/b=0.20)

6 Conclusions

This work investigated the rehydration degree of cement samples with a low water–binder ratio under various conditions. A modified rehydration model was developed based on the conventional cement hydration model. The effects of silica fume, the water–binder ratio, and the ambient temperature on rehydration were analyzed. The variation in the compressive

strength of concrete under natural immersion conditions was considered to examine the impact of rehydration on the performance of cement-based materials. The following conclusions can be drawn:

1) Cement-based materials with a low water–binder ratio are associated with the rehydration process when exposed to water; the hydration degree gradually increases with the immersion time. Paste samples with water–binder ratios of 0.15, 0.20, 0.25, and 0.30 exhibited increases in the hydration degree of 0.052, 0.055, 0.045, and 0.044, respectively, after an immersion period of 180 d. The rehydration degree increases and then decreases with an increasing water–binder ratio.

2) The addition of silica fume to the sample improves the initial hydration degree but not the rehydration degree for specimens with a water–binder ratio of 0.20. The ambient temperature has a significant impact on hydration during 0–90 d. After an immersion period of 90 d, the hydration degrees of the samples increased by 0.044, 0.112, and 0.126 at ambient temperatures of 20, 40, and 60 °C, respectively. The rehydration degree also substantially improves with an increase in the temperature.

3) The hydration diffusion model of cement-based materials can also be applied to the rehydration process. By introducing the effective diffusion coefficient of water in a CSH gel D_e and considering the effect of the ambient temperature, a modified rehydration model was developed based on the conventional hydration diffusion model. The numerical results of the modified model were in good agreement with the experimental data. The model parameters (D_{e0} , β) obtained through simulations matched practical situations well and thus provide a reference for future studies on rehydration models.

4) The rehydration rate of unhydrated cementitious materials decreased over time. The hydration degree of the sample with a water–binder ratio of 0.20 would be 0.73 in 10 years. The rehydration of unhydrated cementitious materials may degrade the concrete strength. The

ultrahigh performance concrete sample exhibited a slight decrease in the compressive strength after being immersed in a water. This suggests that rehydration has a nonnegligible effect on the long-term performance of concrete.

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