

PAPER • OPEN ACCESS

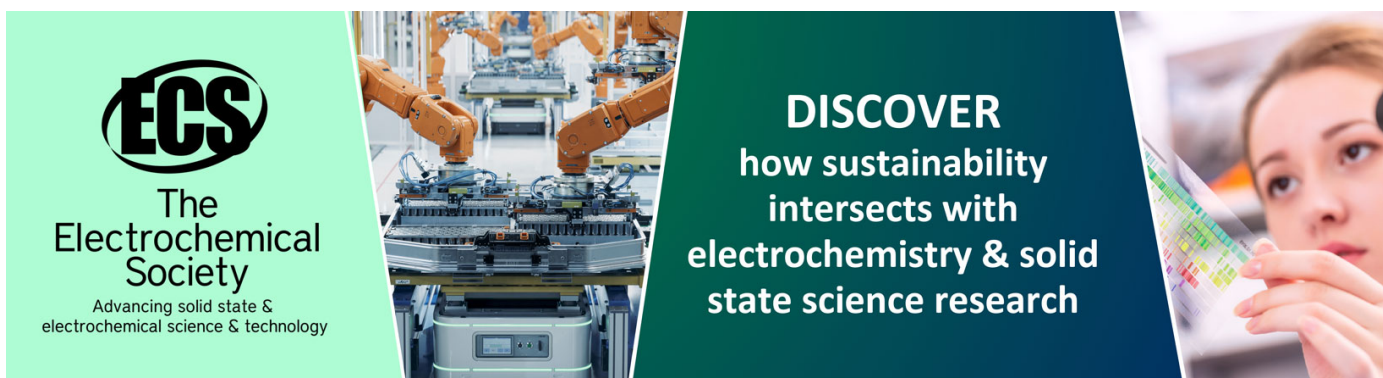
The Hydration Simulation and Strength Prediction of New Grouting Materials

To cite this article: Jun-wu Xia *et al* 2021 *J. Phys.: Conf. Ser.* **2044** 012170

View the [article online](#) for updates and enhancements.

You may also like

- [Gold as a catalyst. Part I. Nucleophilic addition to the triple bond](#)
Sergey B Alyabyev and Irina P Beletskaya
- [Hydration water dynamics around a protein surface: a first passage time approach](#)
Shivangi Sharma and Parbati Biswas
- [Chloride diffusivity in hardened cement paste from microscale analyses and accounting for binding effects](#)
P Carrara, L De Lorenzis and D P Bentz



ECS
The
Electrochemical
Society
Advancing solid state &
electrochemical science & technology

DISCOVER
how sustainability
intersects with
electrochemistry & solid
state science research

The Hydration Simulation and Strength Prediction of New Grouting Materials

Jun-wu Xia^{a*}, En-lai Xu^{b*}, Lin-li Yu^{c*}, You-min Han^{d*}

Jiangsu Key Laboratory of Environmental Impact and Structural Safety in Engineering, China University of Mining and Technology, Xuzhou 221116, Jiangsu China

^{a*}1657@cumt.edu.cn, ^{b*}TS19030087A31@cumt.edu.cn, ^{c*}2446603663@qq.com, ^{d*}18332178823@163.com

Abstract—The new grouting material is a kind of material which can realize rapid condensation at high w/c and has a broad development prospect in many engineering fields. In order to simulate the hydration process and predict the compressive strength, we establish the hydration model by using CEMHYD 3D program to simulate the hydration process. Based on the hydration simulation results, the strength prediction model was established by using the strength formula of "cement-air ratio". The results show that CEMHYD 3D program can be used to simulate the hydration reaction process of new grouting materials. The strength prediction model based on the hydration simulation results has high reliability with prediction error of less than 10%.

1. Introduction

Since the 1980s, the new grouting materials have been widely used in China due to their obvious advantages of early strength and high w/c^[1]. With the development of research, hydration simulation and strength prediction become the focus of grouting materials. Using computer technology to simulate the diffusion, dissolution and reaction stages of materials, hydration simulation is a process based on hydration mechanism. Yan Peiyu^[2] discussed the hydration mechanism of Portland cement in detail. Xia Junwu^[3,4] studied the hardening mechanism of new grouting materials and obtained the hydration reaction equations at different stages. The study found that the particle size distribution of Portland cement and the new grouting material are similar and the hydration products are basically the same. Currently, many scholars^[5-7] use CEMHYD 3D model to simulate the hydration process of Portland cement, while few studies apply it to the hydration simulation of grouting materials. In addition, there are many methods for strength prediction, including fitting formula^[8], establishing neural network model^[9-10], and strength formula-based prediction^[11]. Compared with the previous two methods, the method based on the strength formula does not rely on the test data, and its theoretical basis is hydration information, so the prediction model obtained more research significance.

Based on the study of hydration mechanism of digital image analysis, this paper uses CEMHYD 3D program to simulate the hydration and hardening process of new grouting material, and explores the applicability of this program. Then, the strength prediction model of new grouting material is established based on the strength formula of "cement- air ratio".



2. Test preparation and method

2.1 Raw materials and sample preparation

The new grouting material used in this test is a mixture of A and B. The composition is shown in Fig.1. Additive A (AA) and B (BB) mainly control the initial and final setting time and increase the dispersion of solid materials in the slurry. XRF analysis was performed on the new grouting material, and its chemical composition was obtained as shown in Table 1.

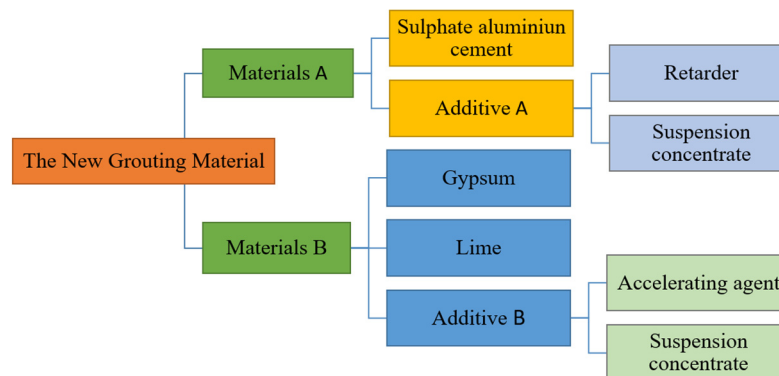


Fig.1 Composition of new grouting materia

Table 1 Mineral composition of new grouting materials

Sample	Mass fraction/%												
	CaO	Al ₂ O ₃	SiO ₂	SO ₃	Fe ₂ O ₃	MgO	SrO	K ₂ O	Na ₂ O	TiO ₂	P ₂ O ₅	CO ₃	Others
CSA	41.12	35.36	7.31	9.10	2.31	0.89	0.07	0.31	0.17	1.38	0.19	3.59	0.21
Gypsum	36.23	0.18	1.06	45.58	0.05	2.84	0.26					13.67	0.03
Lime	88.12	0.49	1.15	0.15	0.13	9.87	0.06	0.02					0.02

Sample A: Specimen with size of 40mm×40mm×160mm was prepared for compressive strength test, and the w/c was 1.5. Considering only the effects of age (5h, 1d, 3d, 7d, 28d), the test ratio was CSA: AA: Gypsum: BB=1: 0.1: 0.85: 0.15: 0.1. Three specimens of each group were prepared and cured in the standard curing box to the corresponding age.

Sample B: In this study, in order to obtain the distribution state that the material just contacts with water and does not participate in the reaction, use epoxy resin with viscosity similar to water but not reacting for curing. Except that the water was replaced with epoxy resin, other components were identical with the hydration reaction test to ensure that the characteristic information of 2D images obtained in the test was consistent with the actual information. The epoxy resin used is GCC135 type A and GCC137 type curing agent, and the size of the silica gel mold is 50mm×50mm×50mm. The production process of the sample is shown in Fig.2.

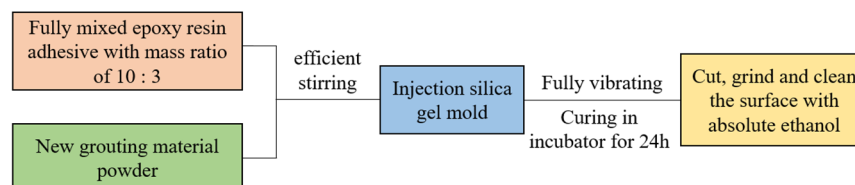


Fig.2 Sample preparation process

Sample C: In order to obtain the particle size distribution of the new grouting material, about 100g of powder sample C was prepared according to the ratio.

2.2 Test Method

Test of compressive strength: Shanghai wuxi AEC-201 automatic cement strength testing machine was used to carry out the strength test on sample A, and the test was carried out according to “method of

testing cements-determination of strength” to determine the compressive strength value of the material under different hydration ages.

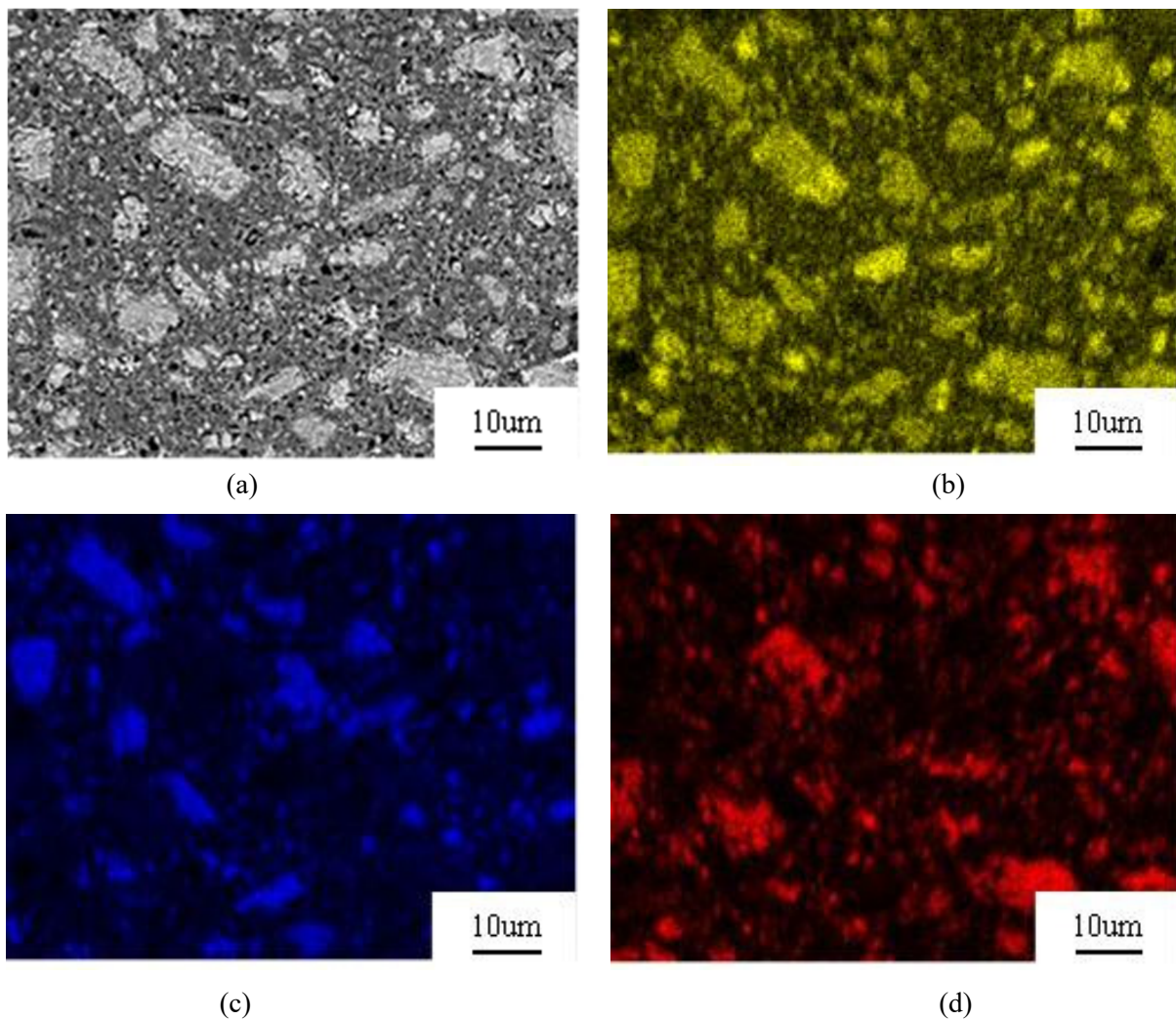
Acquisition of Scanning Electron Microscope Image: Sample B was prepared according to the requirements of SEM scanning electron microscope, and low vacuum mode image acquisition was used for testing. In the SEM scanning process, for backscattered electron imaging, the acceleration voltage is 25 kV, the magnification is 800 times, and the scanning process lasts 120 minutes.

Establishment of hydration model: According to the area fraction and perimeter fraction extracted from 2D images, the 3D hydration model was established based on digital image model, and the hydration process was set to simulate hydration.

3. Hydration Simulation and Strength Prediction

3.1 Initial 2D image digitization

Analyze the SEM and BSE images of sample B. After obtaining SEM images, preprocessing operations such as smoothing and filtering^[12-13] to make the images clearer. The BSE images of the same region of the sample and the X-ray images of Ca, S, Al, Fe and Si are shown in Fig.3.



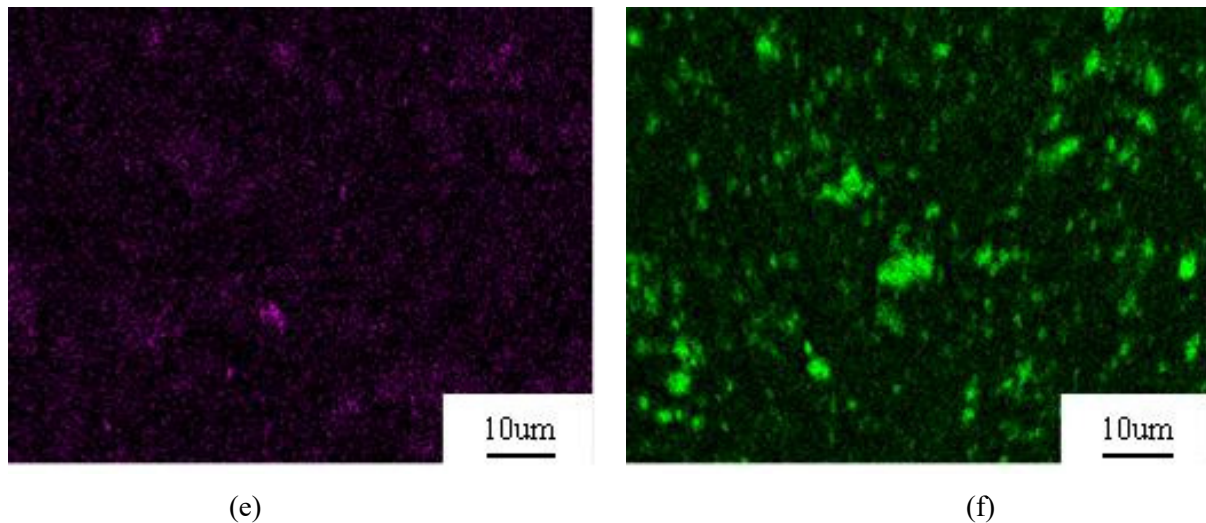


Fig.3 BSE image and X-ray images of major elements (a) BSE image (b) Ca (c) S (d) Al (e) Fe (f) Si

In order to speed up the image processing, the obtained x-ray color image is transformed into black and white image. The gray value of each pixel in the image represents the relative density of an element at the pixel, and the threshold of gray value is set to determine whether the region contains an element^[14]. The processing is shown in Fig.4, where X^* represents the gray threshold of X in the image.

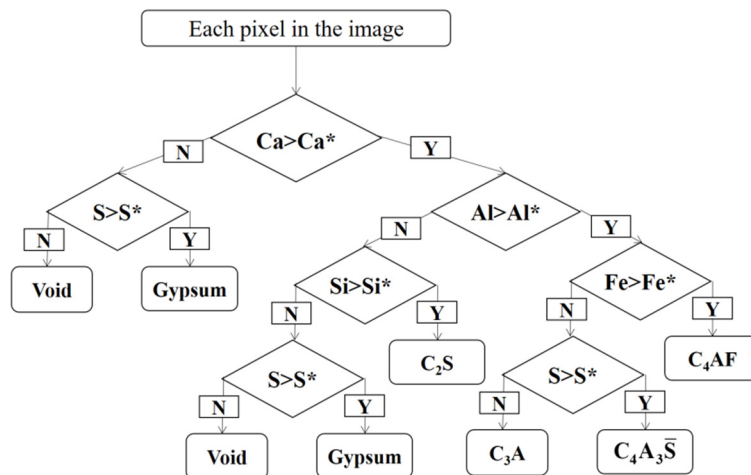


Fig.4 Image segmentation flow chart based on judgment tree

In order to improve the clarity of the image, Matlab software is used to filter the initial 2D image to reduce the error caused by equipment. Finally, the initial 2D image of the new grouting material is obtained, as shown in Fig.5.

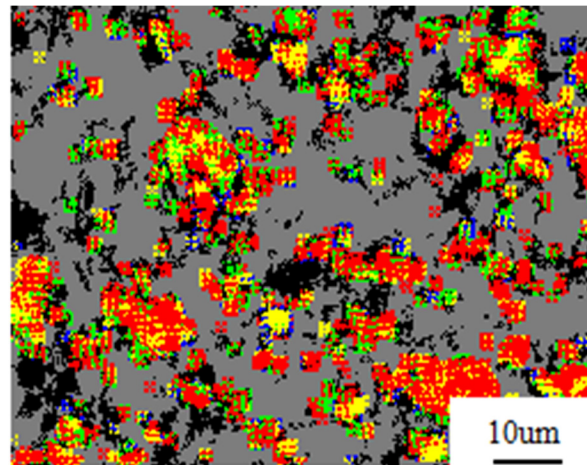


Fig.5 Initial 2D image of new grouting material
(red- $C_4A_3\bar{S}$, yellow- C_4AF , green- C_3A , blue- C_2S , gray-gypsum, black-epoxy)

For the initial image binarization, the gray value greater than the threshold is white, and the gray value less than the threshold is black. Calculate the number of pixels according to the requirements and extract the eigenvalues. The results are shown in table 2. Eigenvalue refers to the area fraction and perimeter fraction of each phase in 2D state. According to stereology principle, it corresponds to volume fraction and surface area fraction in 3D state respectively, which provides a basis for the establishment of hydration model.

Table 2 Area and perimeter fraction of new grouting materials

Composition	$C_4A_3\bar{S}$	C_4AF	C_3A	C_2S
Area fraction	0.563	0.193	0.195	0.048
Perimeter fraction	0.667	0.162	0.138	0.032

3.2 Computer simulation of hydration reaction

The particle size distribution of sample C was measured by Jinan Winner 3005 dry laser particle size analyzer, and the measurement results are shown in Table 3. It was found that the particles with particle size less than 33 μm accounted for 95.24% of the total volume fraction. Since the Bentz model only places particles within 33 μm , only particles smaller than 33 μm are placed^[15]. The CEMHYD 3D program simulates hydration in the cube of $100\mu m \times 100\mu m \times 100\mu m$, that is, cement and water occupy $10^6 \mu m^3$ space. The pixel of the new grouting material is $1/(1+\rho_c(w/c)) \times 10^6$, where $\rho_c = 3200 \text{ kg/m}^3$. Xia^[6] established the corresponding relationship between particles and pixels under different rules. It is found that according to the rule that the distance from the body center to the particle center is less than or equal to the particle radius, that is, the pixel is considered to be contained by the particle, the relation between the particle and the pixel is consistent with the Bentz model. Based on this, the calculation results are shown in Table 3. The number of particles placed is 173394, which is greater than the actual pixel volume of 172416. The reason is that when large particle sizes are placed, the number of pixels contained in the particles is greater than the pixel volume, so the number of pixels placed is relatively large.

Table 3 Particle size distribution and pixel calculation

Particle size/ μm	Volume fraction /%	Cumulative volume fraction /%	Volume / μm^3	Number of single particle pixels	Particle number	Total particle pixels
1	6.167	6.167	10 633	1	10 633	10 633
3	4.666	10.833	8 045	19	423	8 037
5	5.783	16.616	9 971	81	123	9 963
7	6.621	23.237	11 416	179	64	11 456
9	9.464	32.701	16 317	389	42	16 338
11	8.529	41.230	14 705	739	20	14 780

13	7.557	48.787	13 029	1 189	11	13 079
15	5.870	54.657	10 121	1 791	6	10 746
17	4.627	59.284	7 978	2 553	3	7 659
19	3.906	63.190	6 734	3 695	2	7 390
21	4.884	68.074	8 421	4 945	2	9 890
23	5.512	73.586	9 503	6 403	1	6 403
25	5.011	78.597	8 640	8 217	1	8 217
27	4.048	82.645	6 979	10 395	1	10 395
29	4.203	86.848	7 247	12 893	1	12 893
31	5.069	91.917	8 740	15 515	1	15 515
33	3.323	95.240	5 729	18 553	0	0
35 ~ 61	4.760	100	8 208	22 575 ~ 119 009	0	0
total			172 416		11 334	173 394

According to the number of pixels the particle contains, the particles are placed in the order from large to small. Phase labeling was performed on the pixel points during placement, and only the new grouting material (except $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$) and $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ were preliminarily distinguished. The identifier of the former was 1, and the latter was 2. After the initial release, the number of pixels put was counted, and the missing part was supplemented in the form of single pixel particles. Based on the stereology principle, the particles designated as new grouting materials are divided into various phases of the constituent materials by using the extracted 2D image eigenvalues. The program uses the autocorrelation function between two pixels to adjust the volume fraction of each phase, and uses the hydraulic radius to adjust the surface area fraction. Assign different ID values to pixels to represent corresponding phases, and set mask values-2^{ID} for different phases to facilitate subsequent programming, gypsum-2⁰, $\text{C}_4\text{A}_3\bar{\text{S}}$ -2¹, C_2S -2², C_3A -2³, C_4AF -2⁴.

For the 2D image, the area fraction (pixel ratio) of the specified phase or composite phase can be obtained by calculating the autocorrelation function. The size of the 2D image is $M \times N$, and the autocorrelation function is expressed as:

$$S(x, y) = \sum_{i=1}^{M-x} \sum_{j=1}^{N-y} \frac{I(i, j) \times I(i+x, j+y)}{(M-x) \times (N-y)} \quad (1)$$

Where, $S(x, y)$ represents the area fraction of the desired phase in the range from coordinates (x, y) to (M, N) in the image. The calculation of the autocorrelation function of 3D structure is the same as that of 2D structure. The expression of the autocorrelation function is:

$$S(r) = \frac{1}{2r+1} \sum_{i=0}^{2r} S(r, \pi/4r) \quad (2)$$

$S(r, \theta)$ is obtained from $S(x, y)$ through linear interpolation. Each pixel is assigned a random number that follows a normal distribution, denoted as $N(x, y, z)$. Define:

$$F(x, y, z) = \frac{S(r = \sqrt{x^2 + y^2 + z^2}) - S(0)^2}{S(0) - S(0)^2} \quad (3)$$

The autocorrelation function of each pixel in 3D is:

$$R(x, y, z) = \sum_{i=0}^{30} \sum_{j=0}^{30} \sum_{z=0}^{30} N(x+i, y+j, z+k) \times F(i, j, k) \quad (4)$$

In the phase distribution, the new grouting material particles were first divided into silicate and aluminate by using the autocorrelation function of silicon phase (C_2S), and then the pixels contained in each phase were adjusted by hydraulic radius to make them consistent with the circumference fraction in the 2D image. Then, the aluminate salts were changed to C_3A , and C_4AF , and the hydraulic radius was adjusted. By inputting the inherent information such as dissolution probability, curing condition and

induction time, the 3D model of the new grouting material is basically established.

The hydration simulation was carried out in three stages, namely: material and water dissolution, slurry diffusion and hydration reaction. Hydration occurs in strict accordance with the given chemical reaction equation. The system simulates the actual hydration process by controlling the time required for the hydration model to complete a reaction cycle. Since the kinetics generated by the hydration model is close to that of the Nusen linear model, the actual hydration reaction time and the number of hydration cycles are related by the following formula: $T = t_0 + \text{number of cycles} \times \text{the time of one cycle}$, where t_0 is induction time, the time of one cycle is 0.00025s. The corresponding cycle number and hydration degree at each age are obtained as shown in Table 4.

Table 4 Relationship between hydration degree and cycle number

age	cycle number	hydration degree
5h	141	0.269 4
1d	310	0.355 6
3d	537	0.493 2
7d	820	0.621 7
28d	1 640	0.751 3

3.3 Establishment of strength prediction model

Through hydration simulation of the new grouting material, the hydration degree of different ages was obtained. Based on the strength formula of "cement-air ratio" ^[16,17], the strength of materials at different ages is predicted. The formula is:

$$f_c = f_{c0} \cdot x^n \quad (5)$$

Where f_{c0} is the compressive strength of the stone body when the cement-air ratio is 1, and the value is 238 MPa^[18]. n is a constant, usually 2.6~3.0. x is cement-air ratio, which is the ratio of the volume of cement gel to the sum of the volume of cement gel and pores.

$$x = \frac{2.14 \frac{1}{\rho} \cdot \alpha}{\frac{1}{\rho} \cdot \alpha + \frac{w}{c}} \quad (6)$$

Where ρ -dry density(g/cm³). α -hydration degree/%.

At the same time, the strength test was carried out to determine the compressive strength at different ages under the same ratio, and the predicted strength was compared with the experimental strength. The experimental data are shown in Table 5. The comparison shows that the prediction error of age within 3 days and later is less than 8%, which is basically consistent with the experimental intensity. The early prediction value is far less than the experimental strength, which fails to reflect the early strength of the material. The reason may be that in the test, the slurry was stirred separately and then mixed, which accelerated the initial hydration reaction. In addition, the early strength agent in the ethyl material improves the hydration reaction rate.

Table 5 Comparison of measured and predicted strength

age	5 h	1 d	3 d	7 d	28 d
measured value/ MPa	2.03	2.81	3.36	5.82	8.84
	2.14	2.78	3.26	5.57	8.76
	2.01	2.75	3.38	5.94	8.93
mean value/ MPa	2.06	2.78	3.33	5.78	8.84
predicted value / MPa	0.84	1.65	3.60	6.17	9.49
error/%	+145.2	+68.5	-7.5	-6.3	-6.8

In order to predict the compressive strength more accurately, the strength prediction formula must be fitted to correct the error caused by the early strength of the material at the early stage of hydration. By introducing the influence coefficient k , the fitting formula for predicting the compressive strength is

selected as follows:

$$f_c = f_{c0} \times x^n \times k = f_{c0} \times x^n \times (a_0 + a_1T + a_2T^2 + a_3T^3) \quad (7)$$

Where T-hydration, age/h.

The least square method is used for polynomial fitting, and the coefficients of the fitting equation are solved by Matlab software. The final strength prediction formula is obtained by substituting them into the fitting formula:

$$\begin{aligned} f_c &= f_{c0} \times x^n \times k(T) \\ &= (620.37 - 10.33T + 0.088T^2 - 2.44 \times 10^{-4}T^3) \cdot x^{2.6} \end{aligned} \quad (8)$$

The modified strength is compared with the measured strength and predicted strength, as shown in Fig.6. The data above the modified strength is the error value between the modified strength and the measured strength. It can be seen from the figure that after correction, the overall prediction error is within 10%, which proves that the prediction model established based on the strength formula of "cement-air ratio" can predict the compressive strength of the new grouting material well after correction by Matlab software.

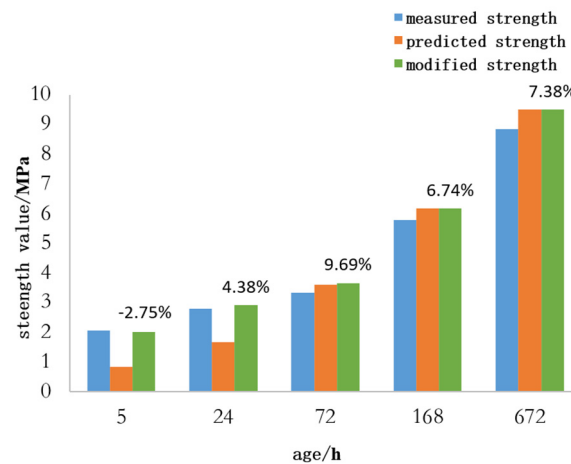


Fig.6 Strength values at different ages

4. Conclusion and prospect

In this study, the CEMHYD 3D model was used for hydration simulation, and the strength prediction model with hydration degree and hydration age as parameters was established based on the strength formula of "cement-air ratio". The model has high reliability and the error between the predicted value and the measured value is within 10%. It can be used to predict the compressive strength of new grouting materials.

Through the study, it is found that when CEMHYD 3D program is directly used in the hydration simulation of new grouting materials, the early hydration degree is predicted to be low, resulting in a large error in early prediction. Therefore, in the subsequent research, it can be considered to modify the program by introducing parameters and other operations, and add the programming part of the additive to more accurately simulate the hydration process of new grouting materials.

Acknowledgments

This work was financially supported by the National Natural Science Foundation in china(52074270)

References

- [1] XIAO Rongjiu. Development and application of foreign chemical grouting technology [J]. Northwestern Geology, 1994: 16-21, 29.
- [2] YAN Peiyu, ZHANG Zengqi. Mechanism of hydration and hardening of composite cementitious materials[J]. Journal of the Chinese Ceramic Society, 2017, 45(08): 1066-1072.
- [3] Xia J, Zhao W, Liu D, et al. Study on the hydration and hardening mechanism of the high-water rapid-setting material[J]. IOP conference series. Materials Science and Engineering, 2018, 439: 42044.
- [4] Xia J, Su Q, Liu D. Optimal gypsum-lime content of high water material[J]. Materials Letters, 2018, 215: 284-287.
- [5] WU Danlin, WANG Peiming. Computer Simulation of Cement Hydration Process-Analysis and Simulation Realization of CEMHYD3D System[J]. Materials Reports, 2007(04): 100-103.
- [6] XIA Junwu, SUN Kewei, ZHAO Haitao. Study on the Relationship between Particle and Pixel in Microscopic Analysis Model of Cement Based Materials[J]. Journal of Hydraulic Engineering, 2016, 47(07): 865-872 .
- [7] WU Dajiang, SHE Wei, MIU Changwen, et al. Simulation of cement hydration microstructure evolution based on improved CEMHYD3D model[J]. Journal of Building Materials, 2020, 23(01): 11-17.
- [8] ZHAI Fengrui, SONG Huanbin, ZHANG Lili, et al. Experimental Study on Early Strength Prediction of Dust Cement Based Materials[J]. Journal of Kunming University of Science and Technology (Natural Science), 2010, 35(02): 47-50.
- [9] Alexandridis A, Triantis D, Stavrakas I, et al. A neural network approach for compressive strength prediction in cement-based materials through the study of pressure-stimulated electrical signals[J]. Construction and Building Materials, 2012, 30: 294-300.
- [10] DONG Yue, YANG Zhiqiang, GAO Qian. Influence of Steel Slag on Properties and Strength Prediction of Slag-based High Water Filling Material[J]. Bulletin of the Chinese Ceramic Society, 2017, 36(11): 3841-3847.
- [11] SUN Kewei. Study on Compressive Strength Prediction of Cement-Based Materials Based on the Microstructure[D]. Master's thesis, China University of Mining and Technology, China, 2016.
- [12] SU Qiong. Study on the Strength and Microstructure of New Type of Grouting Material[D]. Master's thesis, China University of Mining and Technology, China, 2018.
- [13] TU Wangming, WEI Youguo, SHI Shaomin. Application of MATLAB in Digital Image Processing[J]. Microcomputer Information, 2007(06): 299-300+23.
- [14] MIAO Deyu, BAI Xiaohong. SEM image processing method of soil based on MATLAB[J]. Hydrogeology & Engineering Geology, 2014, 41 (06): 141-146.
- [15] LI Guangyao. Research and Application of Image Threshold Segmentation Method and Edge Detection Method. Information & Communications, 2013(04): 19 .
- [16] LIU Chongxi. Mathematical Model of Strength of Cement Stone Structure[J]. Journal of the Chinese Ceramic Society, 1995(06): 635-643 .
- [17] WANG Yanwen. Cement Hydration Simulation and Algorithm in Cellular Automata Environment[D]. Master's thesis, Wuhan University of Technology, China, 2011.
- [18] YANG Shuzhen, SONG Hantang, XIE Rong. Study on the Reaction Rate of Cement Hydration by XRD[J]. Journal of Instrumental Analysis, 1996: 73-76