



Article Pore Structure Quantification and Fractal Characterization of MSA Mortar Based on 1H Low-Field NMR

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Abstract: With the gradual depletion of natural sand due to over-exploitation, alternative building materials, such as manufactured sand aggregate (MSA), have attracted much attention. In order to interpret the evolution of pore structure and fractal characteristics in MSA mortar over long-term water saturation, the 1H low-field nuclear magnetic resonance (LF-NMR) relaxation method was used to investigate the temporal evolution of the pore structure in five single-graded MSA mortars and synthetic-graded mortars with small amplitudes in particle size. MSA presents a fresh rock interface characterized by a scarcity of pores, which significantly reduces the porosity of the mortar. The surface-to-volume ratio (SVR) is employed for characterizing the MSA gradation. Through an analysis of parameters, such as total porosity, pore gradation, pore connectivity, and pore fractal dimension of mortar, a correlation model between pore structure parameters and aggregate SVR is constructed. The fractal characteristics of pores and their variations are discussed under three kinds of pore gradations, and the correlation model between fractal dimension and porosity is established. These results demonstrate the high impermeability and outstanding corrosion resistance of synthetic-graded mortar. The fractal model of the pore structure evolution of MSA mortar has a guiding effect on the pore distribution evolution and engineering permeability evaluation of MSA mortar in engineering.

Keywords: manufactured sand aggregate (MSA); pore structure; NMR; fractal analysis; water-saturated mortar; permeability

1. Introduction

The durability and permeability of materials such as concrete and mortar are important issues in engineering. They are determined by pore structure at the micro scale, and the study of mortar pore structure and its impact on durability is gaining attention. The pore structure of mortar immersed in water for a long time will undergo complex changes [1,2]. Therefore, the study of pore evolution is necessary to ensure the long-term performance of mortar structure. Additionally, the excessive use of natural sand has led to a growing interest in utilizing manufactured sand as an alternative aggregate in mortar production. This trend is driven by the need to conserve natural resources.

Aggregate gradation plays a significant role in the pore structure of mortar. Considerable progress has been made in studying aggregate gradation [3–6]. The usage of an aggregate that is too large or too small would adversely impact the uniformity [5], permeability, and microstructure of mortar [4,7]. The size and content of aggregates have a significant impact on porosity and the interfacial transition zone (ITZ) [8]. The overall surface area of the aggregate is determined by the particle size [9], which in turn affects the paste film thickness and water film thickness [10]. A larger aggregate surface area results



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Copyright: © 2024 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). in a thinner paste film but a thicker water film. The research [11] has shown a correlation between the Talbot gradation and the shear resistance of cemented paste, highlighting the influence of aggregate size on the peak strength of cemented paste [12]. Therefore, it is imperative to investigate the influence of aggregate particle size on the pore structure of mortar [13]. Limited research has been conducted on the quantitative characterization and modeling of pore structure in single-graded MSA (mortar containing small variation amplitudes in aggregate size) mortar, as well as the impact of MSA on the microstructure of mortar using a single variable.

The durability of mortar is influenced by its pore structure [14,15]. The pore structure undergoes changes under the conditions of a water saturation environment or a dry–wet cycle [1]. The hydration and calcium dissolution of mortar contribute to the changes in mortar performance [16]. In addition, the aggregate size also affects the effective water–binder ratio involved in the hydration process in mortar [17], where the water–binder ratio primarily impacts the compactness of the spatial accumulation of hydrated calcium silicate (C-S-H) gel particles [18]. Moreover, the aging of C-S-H gel is accelerated by hot water curing, leading to a significant increase in both the porosity and critical pore size of saturated mortar [19]. Additionally, the pore structure becomes coarser, which in turn enhances water permeability [20]. Currently, there are limited reports available concerning changes in the pore structure of MSA mortar under long-term water saturation.

The fractal method effectively characterizes the state of pore structure. The fractal dimension describes the irregularity of mortar mesh pores, with more complex pore characteristics resulting in a higher fractal dimension [21–23]. The complex pore structure in mortar is impossible to effectively describe using traditional geometric methods, but it exhibits self-similarity due to its wide range of pore sizes [22]. Consequently, fractal theory can analyze the complex pore structure with self-similarity [24,25]. Fractal theory is applied to the PSD spectrum obtained by NMR to obtain the fractal dimension pores with different gradations [26], and to quantitatively analyze the pore size characteristics [27]. As a new non-destructive analysis method, NMR has been used to determine the fractal dimension of porous materials such as cement, tailings mortar [28], natural sand aggregate mortar [7], and rock [24,29]. Additionally, the pore fractal dimension characteristics of MSA mortar deserve attention.

This study aims to interpret the evolution of pore structure in MSA mortar at water saturation. Five types of single-graded mortars and a synthetic-graded mortar were prepared. The parameters of mortar porosity, pore gradation, pore connectivity, and pore fractal dimension were analyzed using LF-NMR technology. A correlation model between the pore structure parameters and aggregate SVR was constructed, and the pore fractal characteristics and differences under three pore gradation divisions were explored.

2. Experiments and Theory

2.1. Materials and Experimental Procedures

The procedures for material preparation, specimen fabrication, and testing process are shown in Figure 1.

(1) Aggregate preparation. Manufactured sands were obtained by crushing limestone. To investigate the impact of aggregate particle size on the pore structure of mortar, five different graded manufactured sands were used: 0.1–0.5 mm, 0.5–1 mm, 1–2 mm, 2–4 mm, and 4–7 mm, the bulk densities of aggregates were 1334.76 kg/m³, 1346.48 kg/m³, 1358.70 kg/m³, 1362.88 kg/m³, and 1430.82 kg/m³, respectively. Particle size distribution followed a Gaussian distribution. Samples were prepared and marked as M1, M2, M3, M4, and M5 groups with three parallel samples. Additionally, to simulate realistic natural environments or conditions, the five types of aggregates were mixed to form a synthetic-graded aggregate that was used to create the M-syn group. The M-syn group encompassed the entire aggregate size range of the individual M groups (M1, M2, M3, M4, and M5).



Figure 1. Experimental instrument and flow chart.

(2) Cement mortar was prepared by mixing pure water, P.O. 42.5 Portland cement, and manufactured sand in proportions of 0.28: 0.77: 1, respectively. The water-cement ratio was 0.36. The composition of the mortar is illustrated in Figure 2b. Specimens were shaped into cylinders with a diameter of 50 mm and a height of 100 mm according to the Rock Test Rules for Water Conservancy and Hydropower Projects (SL/T 264-2020). After filling, tamping, initial curing, and demoulding, mortars were placed in a curing chamber at 22 °C with a relative humidity of 98% for 28 days.



Figure 2. (**a**). Aggregate particle size and SVR distribution; (**b**). Schematic diagram of aggregate size affecting mortar structure.

- (3) Water saturation treatment. Prior to conducting the NMR test, a vacuum saturation device operating at a pressure of 0.1 MPa was used to saturate the specimen's pores with water for a duration of 48 h.
- (4) NMR test. The NMR tests on each specimen were conducted using the AiniMR-150 NMR system manufactured by Suzhou New-market Analytical Instruments Co., Ltd. [30]. The CPMG sequence was conducted with 0.1 ms echo time (TE = 0.1 ms), 3000 echos (NECH = 3000), 1000 ms waiting time (TW = 1000 ms), and 16 scans. NMR tests obtained the T_2 distribution and porosity of the pores.
- (5) Centrifugation and drying. Saturated specimens were centrifuged at 4000 rpm for 90 min and then tested for NMR. Next, the specimens were dried at 50 °C for 20 h and tested again for NMR.
- (6) Water saturation curing. The mortars after 28 days of hydration were removed from the curing box and fully immersed in water within a container for 365 days.

NMR tests were conducted following procedures (3), (4), and (5) after 28 days and 365 days of hydration, respectively, to observe the changes in the mortar's pore structure in a water saturation environment.

The surface-to-volume ratio (SVR) of aggregate particles is the ratio of surface area to volume. This paper is used to quantitatively characterize the gradation of aggregates [30]. The laser particle size analysis shows that the aggregate size distribution under 0.1–0.7 mm gradation conforms to the normal distribution, so the SVR is obtained by weighted average calculation. Figure 1 shows the ideal aggregate SVR distribution curve and the average SVR value obtained by the test. SVR and aggregate size distribution of each group are shown in Figure 2a. A series of tests were carried out on the mortar to obtain the solid density ρ_{28d} , ρ_{365d} and its density loss $\Delta \rho$ of mortar, and the porosity Φ_{28d} at 28d and Φ_{365d} at 365d are shown in Table 1.

Gradation of Aggregates	Aggregate Size/mm	SVR /cm ⁻¹	No.	Solid Density ρ _{28d} /kg⋅m ⁻³	Solid Density ρ _{365d} /kg⋅m ⁻³	Density Loss Δρ/kg·m ⁻³	Φ_{28d} /%	$\Phi_{ m 365d}$ /%
Synthetic gradation M-syn Single gradation M groups	0.1–7.0		M-syn-1	1395.33	1421.78	4.49	3.12	3.82
			M-syn-2	1407.87	1404.41	3.46	2.94	3.42
		75.02	M-syn-3	1389.71	1382.39	7.32	2.84	3.7
			M-syn-Ave	1397.64	1402.86	5.09	2.96	3.64
	0.1–0.5		M1-1	1423.03	1412.35	10.69	3.02	4.56
		212.02	M1-2	1396.31	1382.62	13.69	3.12	4.46
		213.03	M1-3	1383.24	1361.01	12.06	3.52	4.52
			M1-Ave	1400.86	1385.33	12.15	3.22	4.52
	0.5–1.0		M2-1	1415.08	1403.65	11.43	2.64	4.12
		05 41	M2-2	1396.16	1386.53	9.63	3.10	3.84
		85.41	M2-3	1406.21	1389.76	1.07	3.00	4.22
			M2-Ave	1405.82	1393.32	7.38	2.92	4.06
	1.0–2.0	43.12	M3-1	1446.55	1436.61	9.93	2.46	3.64
			M3-2	1412.85	1406.98	5.87	2.88	3.66
			M3-3	1383.40	1383.65	-1.25	3.08	3.84
			M3-Ave	1414.27	1409.08	4.85	2.82	3.72
	2.0-4.0		M4-1	1433.21	1423.19	10.02	2.58	3.58
		21.68	M4-2	1426.27	1421.71	4.56	2.82	3.54
			M4-3	1407.31	1407.34	-0.03	2.72	3.50
			M4-Ave	1422.26	1417.42	4.85	2.7	3.54
	4.0–7.0	11.77	M5-1	1401.65	1400.82	0.83	2.64	3.22
			M5-2	1417.75	1409.93	7.83	2.64	3.22
			M5-3	1427.52	1422.25	5.28	2.56	3.40
			M5-Ave	1415.64	1411.00	4.64	2.62	3.28

Table 1. Physical parameters of cement mortar.

2.2. Background Theory

2.2.1. NMR Theory

By employing NMR technology, the presence of hydrogen 1H in water molecules within porous materials, such as mortar and concrete, enables the acquisition of pore distribution information and the visualization of pore structure [30,31]. The NMR analysis of saturated mortar provides comprehensive insights into the entirety of its pores [32]. Assuming uniformity in the magnetic field during testing, the contributions of volume and diffusion relaxation of water within the void can be disregarded [30,33]. Consequently, the T_2 relaxation rate is solely dependent on the surface relaxation rate, allowing for simplification, as follows:

$$\frac{1}{T_2} = \rho_2 \left(\frac{S}{V}\right) = \rho_2 F_s \frac{1}{r}$$
(1)

where T_2 is the decay time of transverse relaxation component (ms); r is the pore radius (μ m); *S*/*V* denotes the specific surface area of the pore (m⁻¹). F_s refers to the pore shape factor (spherical pore, $F_s = 3$); ρ_2 is the surface relaxation strength of the mortar, primarily influenced by the mineral composition, to be determined as 0.01 μ m/ms based on the literature [30,34–36]. Therefore, the relationship between r and T_2 can be simplified and expressed as follows:

$$r = 0.03T_2$$
 (2)

2.2.2. Fractal Theory Based on NMR

According to research on fractal theory [22,26,37], it is evident that porous materials like cement and concrete display pronounced fractal characteristics in their internal pore structure, establishing a correlation among T_2 , pore radius r, and fractal dimension D. The subsequent equations can be employed to determine pore sizes larger than r [30,38]:

$$N_{\rm r} = \int_r^{r_{max}} f(r)dr = ar^{-D} \tag{3}$$

where N_r represents the count of pores with sizes exceeding r, r denotes the pore radius in micrometers (μ m), r_{max} signifies the maximum pore radius in micrometers (μ m), a denotes the fractal factor, D represents the fractal dimension, and f(r) represent the density function of the pore radius expressed as a percentage (%), given by

$$f(r) = \frac{dN_r}{dr} = -Dar^{-D-1} \tag{4}$$

The total volume of pores smaller than *r* can be calculated using

$$V_r = \int_{r_{min}}^r f(r)ar^3 dr = a' \left(r^{3-D} - r_{min}^{3-D} \right)$$
(5)

Here, V_r denotes the cumulative volume of pores with radii lesser than r in cubic micrometers (μ m³), a' represents a proportional constant equal to $-Da^2/(3-D)$, and r_{min} signifies the minimum pore radius in micrometers (μ m). Consequently, the total pore volume V_t of the mortar can be obtained from Equation (6):

$$V_t = \int_{r_{min}}^{r_{max}} f(r) a r^3 dr = a' \left(r_{max}^{3-D} - r_{min}^{3-D} \right)$$
(6)

The cumulative volume fraction of pore radius less than r can be obtained by Equations (5) and (6) [30,38].

$$S_{v} = \frac{V_{r}}{V_{t}} = \frac{\left(r^{3-D} - r^{3-D}_{min}\right)}{\left(r^{3-D}_{max} - r^{3-D}_{min}\right)}$$
(7)

When r_{min} is considerably smaller than r_{max} , simplification of Equation (7) results in Equation (8). Furthermore, Equation (8) can be further revised as Equation (9):

$$s_{v} = \frac{r^{3-D}}{r_{max}^{3-D}} = \frac{T_2^{3-D}}{T_2^{3-D}}$$
(8)

$$lg(s_v) = (3 - D)lgr + (D - 3)lgr_{max} = (3 - D)lgT_2 + (D - 3)lgT_{2max}$$
(9)

If the pores in the mortar exhibit self-similarity and fractal characteristics, Equation (9) establishes a linear relationship between lg(r) and $lg(s_v)$ [39]. A higher fractal dimension D indicates greater complexity in the pore structure and increased heterogeneity in the mortar sample [22].

3. Results

3.1. Evolution of T₂ Spectr\$um and Porosity

The T_2 spectra of three parallel samples had good reproducibility. The PSD of six groups of mortars with different gradations have similar distribution patterns, which have four peaks (P1, P2, P3, and P4) and three valleys (V1, V2, and V3). As shown in Figure 3a, the peak represents the most probable pore size. The method of double T_2 cut-off (T_{2c}) value is shown in Figure 3b [30], which is obtained through a procedure of saturation, centrifugation, and drying, and the T_2 spectrum distribution is shown in Figure 4.



Figure 3. Pore division based on: (a). spectral valleys, (b). T_{2c}.

The double T_{2c} value method reflects the connectivity of pores in mortar [40]. According to the fluidity of water in pores, the pores are divided into clay-bound fluid pores (*CBF*), capillary-bound fluid pores (*CAF*), and movable fluid pores (*MF*). The *CBF* pores include micropores such as C-S-H interlayer voids, gel pores, and internal hydration pores [41]. The *CAF* pores are mainly large gel pores. The fluid in these pores is mainly adsorbed by the van der Waals force of the solid matrix. It can be seen that the T_2 spectrum of test sequences S-28d and S-365d in Figure 4 characterizes the distribution of full-size pores, the T_2 spectrum of C-28d and C-365d characterizes the distribution of *CBF* and *CAF* pores, and the T_2 spectrum of D-28d and D-365d only contains *CBF* pores.



Figure 4. Effect of water saturation environment on T_2 spectrum distribution of mortar: (**a**). M1; (**b**). M2; (**c**). M3; (**d**). M4; (**e**). M5 group; (**f**). The explanation for subfigures.

In the case of single-graded mortar, mortars with varying aggregate sizes exhibit equal most probable pore radii under identical curing times. Additionally, the porosity fraction of the most probable pore and the maximum pore size are positively correlated with the aggregate SVR [42]. This demonstrates that the aggregate size has an impact on the maximum micron pores and the number of characteristic pores.

The mechanism of the aggregate size affecting the pore structure is as follows: Aggregate with a large SVR has a large amount of water adsorption on the surface, resulting in a decreased effective water–cement ratio [43], and the mortar forms a smaller paste film thickness in the rapid hydration stage (initial setting 2 to 20 h), which will increase the number of C-S-H interlayer pores, gel pores, and hydration pores in the P1 region. In addition, the aggregate SVR is positively correlated with the total volume of the ITZ and incompletely hydrated loose bodies [8], while macropores are mainly produced in the ITZ and loose bodies [9]. Therefore, aggregates with a larger SVR also increased the number of pores in the range from P2 to P4 region, and the pore components of the most probable pores and maximum pore radii also increased [3].

After servicing in water saturation for 365 days, the T_2 spectral distribution of each mortar is broadened. Additionally, the most probable pore size of P1 decreased, while their respective components increased by 41.7%, 45.5%, 27.3%, 22.6%, and 30% for groups M1 to

M5. The most probable pore radius of P1 decreased from 0.025μ m to 0.01μ m, indicating that the formation of a dense cementitious structure within the mortar [18]. Despite the decrease in the most probable pore radius of P4 from 35μ m to 10μ m, pores larger than 120 µm were observed.

The T_2 spectrum is divided and statistically analyzed according to the method in Figure 3a; cumulative porosity components and their proportions of the spectrum peaks are obtained, as shown in Figure 5. It was found that the pore variation is manifested by the increase in P1 and P4, that is, the increase in hydration micropores and erosion macropores. One of the reasons for this variation is the continuous hydration of the internal unhydrated cement (the peak value of P1 increases). The other reason is that the mortar is eroded by water [33,44] and the calcium hydroxide dissolves [45] (the peak value of P4 increases and produces pores larger than 120µm).



Figure 5. Porosity component evolution divided by spectral valleys.

3.2. Evaluation of Pore Connectivity

The double T_{2c} value that characterizes the connection connectivity of mortar is shown in Figure 6, which conforms to the function $T_{2c} = a \times SVR^b$ with aggregate SVR. T_{2c1} and T_{2c2} exhibit a negative correlation with SVR, indicating that the smaller aggregate size of MSA leads to greater pore richness and stronger pore connectivity. These experimental findings are supported by Figure 2b, which illustrates that ITZ dominates the seepage path influencing mortar permeability. The aggregate SVR is proportional to the total volume of ITZ [19]. Therefore, small aggregate (large SVR) makes the mortar form rich pores, resulting in a small T_{2c} value according to the principle of T_{2c} in Figure 3b. In addition, after 365 days of water saturation, T_{2c1} decreases and T_{2c2} increases, providing evidence of enhanced connectivity in *CBF* pores (T_{2c1} decreases) and weakened connectivity in *CAF* pores (T_{2c2} increases) [33]. The cause of this phenomenon is supported by Figure 7, where an increase in *CBF* porosity and a decrease in *CAF* porosity confirm that the pore-throat structure connecting the pores primarily consists of *CBF* pores.



Figure 6. T_{2c} value of single-graded mortar: (a). T_{2c1}; (b). T_{2c2}.



Figure 7. Effect of aggregate SVR on various pores: (**a**). Total pores; (**b**). *CBF* pores; (**c**). *CAF* pores; (**d**). *MF* pores.

All the pores detected in the saturated mortar included *CBF* pores, *CAF* pores, and *MF* pores. Subsequent centrifugation facilitated the detection of fluid-bound *CBF* and *CAF* pores. Upon complete drying, solely small and poorly interconnected *CBF* pores were observable. Through calculations and the analysis of the PSD spectrum depicted in Figure 4, a connection can be established between aggregate SVR and the presence of *CBF*, *CAF*, and *MF* pores, as indicated in Figure 7. Porosity escalates in tandem with SVR, and its relationship broadly conforms to the power function $\Phi = a \times SVR^b$. Both parameters a and

b exhibited higher values following 365 days of hydration. Evidently, there is an increase in pores and alterations in pore connectivity.

In addition, the *MF* pores are mainly located within ITZ and unhydrated cementitious material, which is distant from the aggregate [46]. The hydration leads to the densification of ITZ and the hydration of unhydrated material. Hence, the *MF* porosity in the M1 to M5 groups has a decreasing trend. Once the mortar has been immersed in water for a specific period, the cementitious material becomes fully hydrated. Consequently, hydraulic erosion causes the maximum size of *MF* pores to incrementally expand over time. Which can be verified by the increase in the maximum pore size in Figure 4. It was also found that needle-like crystal substances appeared on the surface of samples and gradually increased.

3.3. The Permeability Variation

Permeability characterizes the ability of porous media materials to be passed by liquid, and its size is related to porosity, pore density, and pore connectivity [39,47]. The Coates model is widely used to calculate permeability in the porous media of water-bearing systems. The simplest form of permeability K (mD) is as follows:

$$K = \left(\frac{S_M}{S_{CB} + S_{CA}}\right)^n \left(\frac{\Phi}{C}\right)^m \tag{10}$$



Figure 8. Permeability of single-graded mortar: (a). hydration 28d; (b). hydration 365d.

After 365 days in a water saturated environment, the permeability of the mortar increases, which confirms the enhancement of pore connectivity [16]. The permeability change is mainly due to the increase in *MF* pores, and the *CBF* pores and *CAF* pores as pore throats are further increased.

It can be seen from Figure 7 that *SCB* gradually increases, and *SCA* and *SM* gradually decrease. It can be seen that in the process of mortar service to 365 d, the effect of the initial water saturation environment on mortar is mainly to enhance the hydration of mortar. With the continuous saturation time, the unhydrated mortar will be completely hydrated [18], which leads to the densification of the C-S-H structure, and the cumulative hydraulic erosion of mortar will increase, and the mortar with large aggregate SVR is more strongly affected by hydraulic erosion [49].

3.4. Fractal Characteristics of Pore Structure

In the NMR-based fractal characterization method [22,26], the fractal dimension is usually calculated according to the pore gradation, but the entire pore range of the cement-based material does not necessarily have fractal characteristics [21]. Pores with each size exhibit a distinct inner surface morphology. Therefore, in this study, three methods, namely, the spectral valley-based pore fractal calculation (F–sv method), the T_{2c} –based pore fractal calculation (F– T_{2c} method) [30], and the pore size-based pore fractal calculation (F–ps method) [23,29], are employed to analyze the fractal characteristics across varying pore gradations (Figure 9).



Figure 9. The fractal dimension based on (a). F-sv method; (b). $F-T_{2c}$ method; (c). F-ps method.

According to the PSD spectral valley, T_{2c} value, and pore size characteristics, the pore fractal dimensions of five types of single-graded mortars are obtained (see Figures 10 and 11, Table 2). The pore structures of the mortars exhibit favorable fractal characteristics within their respective ranges. The fractal dimension D represents the self-similarity and scale invariance of the pore structure. With D values less than 3 and approaching 3, the pore surfaces demonstrate increased complexity.

The linear relationship between porosity (φ 1 to φ 4) and the corresponding fractal dimension (D1 to D4) is depicted in Figure 10 according to the F-sv method. Upon saturating the mortar for 365 days, the fractal dimension of each pore gradation generally declines, with the exception of an increase in D1 for the M4 and M5 groups. This observation, in conjunction with Figure 4, highlights the formation of a dense structure resulting from long-term water saturation conditions and cement hydration.



Figure 10. The correlation analysis between porosity and fractal dimension under F–sv method for (a). D1; (b). D2; (c). D3; (d). D4.



Figure 11. The correlation between porosity and fractal dimension under F-ps method for (**a**). D; (**b**). D_{s1} ; (**c**). D_{s2} ; (**d**). D_{s3} .

No.	Hydration	CBF Pores		CAF Pores		MF Pores	
	Time	Φ_{cb}	D _{cb}	Φ_{ca}	D _{ca}	$\Phi_{\rm m}$	D _m
M-syn	28 d	2.120	1.497	0.432	2.724	0.408	2.976
	365 d	2.910	1.907	0.420	2.986	0.316	2.988
M1	28 d	1.488	1.490	0.692	2.250	1.036	2.590
	365 d	3.048	1.787	0.512	2.966	0.952	2.967
M2	28 d	1.528	1.603	0.688	2.229	0.702	2.432
	365 d	3.046	1.834	0.444	2.962	0.568	2.981
M3	28 d	1.518	1.629	0.684	2.188	0.608	2.351
	365 d	2.894	1.784	0.418	2.945	0.404	2.985
M4	28 d	1.474	1.670	0.676	2.176	0.506	2.309
	365 d	2.750	1.782	0.400	2.946	0.392	2.984
M5	28 d	1.460	1.723	0.662	2.169	0.458	2.297
	365 d	2.670	1.765	0.298	2.949	0.288	2.986

Table 2. The porosity and fractal dimension are divided under the $F-T_{2c}$ method.

The fractal dimensions D1 and D4 exhibit significant changes; D1 is directly related to the extent of hydration reaction, displaying a higher value when the porosity is greater. After 365 days, the P1 region shifts to the left, reducing the complexity of inner pore surfaces, leading to a decrease in fractal dimension. On the other hand, the P4 region consists of incompletely hydrated material and large pores within the ITZ. During the incomplete hydration at 28 days, the pore size is substantial, and the unhydrated material forms a loosely structured complex inner surface. After 365 days, due to dissolution and leaching of calcium, the complexity of the inner surface remains largely unchanged (indicated by minimal change in the D4 value) but is lower compared with the initial period.

The fractal dimension of the pores in the P2 and P3 zones gradually decreases with the increase in the porosity $\varphi 2$ and $\varphi 3$, indicating that the distribution and structural complexity of these pores are slightly reduced. The primary factor contributing to this decrease is the reduction in the most probable pore size. These pores are predominantly transition pores formed during the hydration process [50].

The porosity (φ cb, φ ca, and φ m) and corresponding fractal dimensions (Dcb, Dca, and Dm) were determined using the F–T_{2c} method, as presented in Table 2. If D > 3 or D < 2, it is considered non-physical and lacks fractal characteristics from the perspective of surface geometry [51]. The selF–similarity and scale invariance of the mortar pore surface only exist within a specific range. It is evident that *CBF* pores lack self-similarity; however, after 365 days of water saturation, there is an increase in the fractal dimension, suggesting an enhancement in the self-similarity and scale invariance of *CBF* pores.

The porosity of *CAF* pores and *MF* pores shows a positive correlation with the D at 28d or 365d; φ ca and φ m increase with the increase in SVR. At 365 d of water saturation, the Dca and Dm increased significantly, while φ ca decreased significantly, which was related to the decrease in T_{2c1} and the increase in T_{2c2}.

To sum up, after 365 days of water saturation, the internal pores of the mortar experience densification resulting from complete hydration. This process increases the fractal dimension of *CBF* pores. Conversely, hydration causes a decrease in the porosity of *CAF* pores and *MF* pores. Simultaneously, hydraulic dissolution causes calcium precipitation and enlarges the maximum pore size of *MF* pores, resulting in an increased fractal dimension of both *CAF* pores and *MF* pores. The fractal dimension of the pores, obtained through the $F-T_{2c}$ method, is appropriate for evaluating the fractal evolution characteristics of pore connectivity.

According to the F-ps method, Figure 11 illustrates a linear relationship between the fractal dimension and the porosity of total pores and pores at various levels, except for pores less than 0.1 μ m. After 365 days of water saturation, the fractal dimension generally increases in each mortar. This can be attributed to the formation of more abundant pores within the mortar under saturated conditions, resulting in a higher density of pore distribution and the formation of interconnected pore structures. Furthermore, it is observed that the change in fractal dimension with SVR remains consistent before and after 365 d of water saturation. Specifically, the fractal dimension of total pores increases as SVR increases, while the fractal dimension of mesopores and macropores exhibits an inverse trend. This indicates that SVR not only influences porosity and PSD but also affects the inner surface of pores. A higher surface area of MSA (larger SVR) leads to a chaotic morphology in aggregate distribution [50], resulting in the formation of more complex micropores and an increase in the inner surface area of the pore.

In summary, the F-ps method differs from the F-sv method and the F- T_{2c} method. The F-ps method employs a fixed pore gradation division approach, whereas the F-sv method and the F- T_{2c} method adopt a variable pore gradation division method, owing to the alteration in spectral peak and T_{2c} value following 365 d of water saturation. These three methods are utilized to analyze changes in the fractal dimension of pore characteristics from various perspectives.

3.5. Pore evolution Characteristics of Synthetic-Graded Mortar

Pore connectivity can be qualitatively evaluated by the magnitude of water displacement in pores by external forces. The T_2 spectrum evolution trend of the pore structure in the synthetic-graded mortar is similar to that of the single gradation, and its T_2 spectrum components and porosity values are all in the middle of the five groups of single gradation [30]. The most probable pore radius of P1 obtained after centrifugation and drying is significantly reduced but basically unchanged after 365 days. By combining the peaks' decrease in T_2 and the PSD spectrum after centrifugal drying technology, it was found that the pore connectivity in the P1 range is stronger when hydrated for 28 days. However, the pore connectivity in the range from P2 to P4 is stronger when hydrated 365 days. Otherwise, for the synthetic-graded mortar M-syn, the variation trend of the T_{2c} value is the same as that of the single-graded mortar. T_{2c1} shows a decrease from 0.668 ms to 0.572 ms, while T_{2c2} decreases from 0.922 ms to 0.756 ms, representing reductions of 14.4% and 18.0%, respectively. The porosity of the synthetic grade increases from 2.957% to 3.646%, representing a 23.0% increase, which is lower than the increase observed in the single-grade mortar as illustrated in Figure 9a.

Figure 12 demonstrates a decrease in the peak value of *MF* pores in the P4 area after 365 days in water, accompanied by a reduction in the maximum pore size. However, the permeability decreases from 340.8×10^{-5} mD to 257.6×10^{-5} mD; synthetic-graded mortar exhibits a decrease in permeability, indicating enhanced impermeability and corrosion resistance. According to Table 3, the change trend in the porosity and fractal dimension of the M-syn group mortar closely aligns with that of the single-graded mortar prior to and after 365 days. The fractal dimension and porosity of the M-syn group mortar fall between the values observed in the M group mortars, close to the value of the M2 group. In comparison with the single-graded M groups, it is evident that the synthetic-graded mortar exhibits superior resistance to hydraulic erosion attributable to its well-structured gradation, resulting in diminished impact on macropores under prolonged water saturation.



Figure 12. *T*₂ spectrum (PSD spectrum) of synthetic-graded mortar M-syn: (**a**). hydration 28d; (**b**). hydration 365d.

Fractal	Туре	28	3 d	365 d		
Method		Φ	D	Φ	D	
	Total	2.957	2.886	3.646	2.898	
	Φ_1 -D ₁	2.574	2.617	3.290	2.333	
F-sv method	Φ_2 -D ₂	0.214	2.980	0.174	2.972	
	Φ_3 -D ₃	0.114	2.989	0.068	2.985	
	Φ_4 -D ₄	0.058	2.995	0.114	2.994	
Г	Φ_{s1} -D _{s1}	2.558	2.264	3.328	2.473	
F-ps	Φ_{s2} -D _{s2}	0.218	2.956	0.153	2.986	
method	Φ_{s3} -D _{s3}	0.181	2.987	0.165	2.993	

Table 3. Porosity and fractal dimension of M-syn group.

4. Discussion

4.1. Porosity Variation Characteristics of Mortar

The aggregate size affects the PSD and pore connectivity in the mortar. The total porosity and the pore $\varphi 1$ in the P1 zone increase and the variation increases with the positive correlation power function of the aggregate SVR as illustrated in Figure 13a. Similarly, *CBF*, *CAF*, and *MF* pores also have the same change trend as illustrated in Figure 13b. In the PSD distribution of mortar, the pore $\varphi 3$ in the P3 area has a decreasing trend and the decrease increases with the increase in SVR. It can be seen that the evolution of pores in mortar has been affected by aggregates, and similar studies have been reported [30]. Changes in the porosity of total pores and pores with different gradations result in alterations in the overall pore structure. Consequently, further confirmation is needed regarding the relationship between the change in pore fractal dimension and porosity.



Figure 13. The aggregate SVR affects the variation in porosity for (a). F-sv method; (b). $F-T_{2c}$ method.

4.2. Fractal Correlation Model of Pore Evolution

A correlation model was constructed to examine the fractal variable amplitude characteristics of pores with different gradations, specifically analyzing the relationship between porosity variable amplitude and fractal dimension variable amplitude. Figure 14 depicts the models of fractal dimension variable value for pores with different gradations under the F-ps, F-sv, and $F-T_{2c}$ methods. The linear relationship between the variable amplitude of fractal dimension and the variable amplitude of porosity can be observed in the F-svand $F-T_{2c}$ methods, as depicted in Figure 14a,b.



Figure 14. Correlation model of porosity variation and fractal dimension variation for (**a**). F–sv method; (**b**). F– T_{2c} method; (**c**). F–ps method.

There is no evident correlation between the variable amplitude of fractal dimension and variable amplitude porosity under the F–ps method, as indicated in Figure 14c. At a significance level of 0.05, p > 0.05, the slope is not significantly different from zero, and the correlation coefficient (R²) is less than 0.6. The variable quantities of porosity and fractal dimension are deemed to lack a linear relationship.

Under the F-sv and $F-T_{2c}$ methods, which are classification methods based on pore size range, a correlation model between porosity and fractal dimension can be constructed, and a correlation model between the variable amplitude of porosity and the variable

amplitude of the fractal dimension can also be constructed. However, the F–ps method only allows obtaining the correlation between porosity and fractal dimension.

The fractal dimension increases with the increasing aggregate SVR, and the fractal dimension decreases with the decreasing SVR, indicated that the inner surface of larger pores is more complex than that of smaller pores [27]. Moreover, the aggregate gradation influences the complexity of the pore structure [23,25]. From the perspective of fractal geometry, aggregates with larger SVR result in increased structural complexity and irregularity.

5. Conclusions

This study employed the LF–NMR relaxation method to analyze both single gradation and synthetic gradation specimens. Additionally, we explored the influence of long-term water saturation on the pore structure. Furthermore, we analyzed the fractal characteristics of pore structure during the hydration process for different gradations of mortar. An established correlation model between pore structure parameters and aggregate SVR is presented, leading to the following conclusions:

- 1. MSA has a fresh rock surface, which greatly reduces the number of pores in the mortar, especially the number of large pores. MSA mortar exhibits excellent compactness and small porosity.
- 2. The particle size of MSA significantly affects the pore structure distribution in mortar. The SVR of the aggregate shows a positive power-exponential relationship with mortar porosity, and a strong correlation exists between varying gradations of pores and SVR.
- 3. Long-term water saturation causes an increase in mortar porosity, primarily manifested by the growth of micropores, indicating further hydration reactions. Additionally, dissolution leads to an upward trend in both the maximum size and porosity of macropores.
- 4. The permeability of single-graded mortar increases proportionally with SVR, and the permeability increases after 365 days of water saturation. However, synthetic-graded mortar exhibits a decrease in permeability, indicating enhanced impermeability and corrosion resistance.
- 5. Mortar pores exhibit favorable fractal characteristics. Pore gradation in fractal analysis is determined using the F-sv method, $F-T_{2c}$ method, and F-ps method. All three methods establish a correlation model between porosity and fractal dimension, representing classification based on a variable pore size range, enabling the construction of a correlation model between porosity variable amplitude and fractal dimension variable amplitude.

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