

Original Paper

Influence factors of long-term stability of silica-enriched Portland cement under ultrahigh temperatures

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ABSTRACT

Downhole high-temperature and high-pressure (HTHP) conditions can cause significant deteriorations of well cement, posing a major risk to wellbore integrity. A comprehensive experimental program is designed to investigate the long-term physical and mechanical property stability of silica-enriched Portland cement (SEPC) systems and various influencing factors. Formulations representing three different densities and two silica dosages are designed for two different curing regimes commonly adopted in the literature: 1) direct high-temperature curing at 240 °C and 2) 80 °C low-temperature pre-curing followed by high temperature curing at 240 °C. Under the direct-curing regime, SEPC systems containing 70% silica undergoes progressive microstructural coarsening and strength retrogression over time. The gradual transformation of tobermorite into xonotlite and other crystal phases, as well as potential structural changes in amorphous C–S–H, both causing reductions in bound water content in hydration products, are the principal factors driving strength retrogression. In contrast, under the pre-curing regime, the tobermorite and bound water contents remain stable or increase progressively with curing time, which lead to significant improvements in their physico-mechanical properties during long term curing. Compared to SEPC systems containing 70% silica, the SEPC systems with 40% silica exhibit improved strength stability and comparable long-term strength but lower initial strength, higher porosity and permeability, due to the generation of large amounts of xonotlite as the main hydration product. Decreasing cement slurry density by increasing water to solid ratio promotes the formation of more crystalline phases, which leads to significantly higher porosity and permeability as well as lower strengths with similar long-term retrogression behavior.

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1. Introduction

Deep oil and gas resources have emerged as the primary contributors to newly proven reserves (Wang et al., 2017; Zhao et al., 2025a). Due to temperature and pressure gradients in the ground formation, deep and ultra-deep wells are typically subjected to high-temperature and high-pressure (HTHP)

environments (Liu et al., 2022). For instance, the bottom-hole temperature (BHT) and pressure of the deepest well in China (Well TaKe-1 in Tarim Oilfield) reached approximately 220 °C/145 MPa (Zhang et al., 2024). In several other deep wells and geothermal wells with higher temperature gradients, BHT has approached or exceeded 240 °C (Wang et al., 2024). Maintaining wellbore integrity under such extreme HTHP environments poses significant challenges. Strength retrogression of cementitious materials at elevated temperatures is a key concern, as it can lead to annular sealing failures, ultimately compromising long-term zonal isolation and overall wellbore integrity (Dai et al., 2023a, 2023b). In severe cases, wellbore integrity failures can not only result in production losses but may also

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necessitate complete well abandonment. Therefore, proper design of well cement formulations to mitigate strength retrogression and achieve long-term zonal isolation in wellbore annulus are highly critical for the development of deep petroleum resources.

The primary mineral phases in well cement include tricalcium silicate (C₃S), dicalcium silicate (C₂S), tricalcium aluminate (C₃A), and tetracalcium aluminoferrite (C₄AF), with C₃S and C₂S comprising approximately 80% of the total mass content. Under low-temperature conditions, the hydration of C₃S and C₂S predominantly yields amorphous C–S–H gels and calcium hydroxide, forming a dense microstructure with favorable mechanical properties. However, above 110 °C, the C–S–H gels gradually transforms into crystalline hydration products with reduced specific surface area, leading to the development of large pores and substantial declines in mechanical strength. The most widely adopted method to mitigate high-temperature strength retrogression involves the incorporation of silica sand into the cement formulation (Qin et al., 2021; Liu et al., 2021; Zhang et al., 2023; Zhao et al., 2025b). Nevertheless, recent studies have demonstrated that, even with silica additions, cement cured at temperatures above 200 °C could continue to exhibit notable strength retrogression and microstructural coarsening (Pang et al., 2021; Qin et al., 2023; Sun et al., 2023).

The research on high-temperature strength retrogression of well cement has spanned more than half a century. However, most previous studies have employed a curing regime where the cement is first set under low-temperature conditions (pre-curing) before it is subsequently exposed to high-temperature curing. This approach mainly reflects the conditions encountered in steam injection wells, where the low-temperature cured cement (typically at shallow depth) encounters high temperature during steam injection. It may also represent the upper sections of high-temperature wells, where low-temperature cured cement experiences elevated temperatures during production of hot fluids. Clearly, this curing regime does not adequately represent the conditions in the lower sections of high-temperature wells, where the cement is directly placed under high-temperature conditions (direct-curing) and remains in such environments over the long term. Summaries of representative studies employing both curing regimes are presented in Table 1. Clearly, cement systems enriched with silica sand exhibit relatively stable long-term mechanical performance when precured under a low-temperature setting condition (pre-cure) before high-temperature exposure. In contrast, when the silica-enriched Portland cement (SEPC)

systems are directly set and cured under high-temperature conditions, they tend to suffer from severe long-term strength retrogression. Recently, Sun et al. (2024) have shown that, compared to direct curing, pre-curing can make a significant difference in the hydration products of pure alite (i.e. C₃S, which is the primary mineral in well cement). However, whether such observed phenomenon applies to SEPC systems remain unclear, and related studies are extremely limited. In addition, previous studies primarily focused on conventional density cement systems (approximately 1.9 g/cm³); a few short-term studies (Feng, 2014) have indicated that low-density cement systems often exhibit more severe strength retrogression under high-temperature conditions; however, the long-term performances of such systems remain uninvestigated. In deep or geothermal wells where natural fracture networks are extensively developed, the formation cannot sustain the hydrostatic pressure exerted by conventional-density cement slurries and low-density cement slurries are often needed. Therefore, it is critical to study the influence of cement slurry density on its long-term strength retrogression performance.

Based on previous analysis, the lack of systematic investigations on the influences of setting temperature, slurry density and long-term curing (above 30 days) on the properties of high-temperature cured cement is several of the main factors currently limiting our understanding of the strength retrogression mechanism of well cement. To address these knowledge gaps, long-term curing laboratory experiments were conducted to investigate and directly compare the strength and other property evolutions of SEPC systems under two distinct curing regimes. Considering that bottom-hole temperatures in deep and ultra-deep wells can reach or exceed 240 °C, and that cement at shallow depths of steam injection wells and geothermal wells may experience similar temperatures, 240 °C was selected as the long-term curing temperature. Eight cement formulations with varying densities were prepared. The two curing regimes were: (i) low-temperature curing followed by high-temperature curing, in which slurries were initially cured at 80 °C for 2 days (pre-curing) followed by long-term curing at 240 °C, 30 MPa; and (ii) direct high-temperature curing, in which slurries were directly cured at 240 °C, 30 MPa for the entire duration. To explore the mechanisms of strength evolution and microstructural changes under different curing regimes, comprehensive characterization techniques were employed to analyze both macroscopic and microscopic properties of the sample after 2, 30, and 90 days of curing.

Table 1
Summary of cement strength retrogression studies employing pre-curing regime and direct-curing regime

References	Silica dosage, %	Pre-curing condition	HTHP-curing condition	Strength decline
Eilers and Root (1974)	20–50	107 °C/20 h	232 °C/2 y	No
Iverson et al. (2014)	40–80	25 °C/1 d	343 °C/300 d	No
Pernites and Santra (2016)	35	87.8 °C/1 d	287 °C/30 d	No
Costa et al. (2017)	40	38 °C/1 d	300 °C/3 d	No
Xie (2019)	43–67	80 °C/7 d	260 °C/28 d	No
Zhang et al. (2020)	50	70 °C/3 d	350 °C/30 d	No
Jiang et al. (2021)	35	25 °C/7 d	320 °C/21 d	No
Geng et al. (2022)	35	80 °C/1 d	260 °C/180 d	No
Dai et al. (2023a)	40	50 °C/1 d	300 °C/3 d	No
Geng et al. (2023)	25–35	80 °C/2 d	260 °C/28 d	No
Wang et al. (2017)	35	N/A	150 °C/28 d	6%
Pang et al. (2021)	60–80	N/A	200 °C/142 d	78%–83%
Qin et al. (2022)	60–80	N/A	200 °C/180 d	70%–83%
Sun et al. (2023)	35	N/A	200 °C/2 d	33%
Zhang et al. (2023)	50–80	N/A	240 °C/21 d	28%

2. Materials and experiment routines

2.1. Raw materials

The materials used in this study include Shengwei Class G oil well cement, silica sand, and several commercially available high-temperature cementing additives, such as retarder, dispersant, suspension agent, fluid loss additive, defoamer, etc. The materials and chemical additives used in this study were provided by the PetroChina Tarim Oilfield Company and CNPC Engineering Technology R&D Co., Ltd. The particle size distribution (PSD) curves of the cement and silica sand are shown in Fig. 1, indicating that the two materials share very similar PSD profiles. Key PSD parameters and specific gravity values are summarized in Table 2. The main oxide compositions of the cement and silica, determined via X-ray fluorescence analysis, are provided in Table 3 (minor components with mass content below 0.2% are not listed).

2.2. Formulation design

A total of eight cement slurry formulations were designed in this study, as summarized in Table 4. Three target slurry densities were selected: 1.90, 1.75, and 1.60 g/cm³, corresponding to the formulation code names “h/H”, “m/M”, and “l/L”, respectively. The numerical designation “70” or “40” refers to the mass percentage of silica sand by weight of cement in the formulation. Lowercase letters (“h/m/l”) denote formulations subjected to direct high-temperature curing at 240 °C, 30 MPa, while uppercase letters (“H/M/L”) indicate formulations that initially cured at 80 °C for 2 days, followed by long-term curing at 240 °C, 30 MPa. For the low-temperature pre-cured formulations (H/M/L), no retarder was incorporated to ensure complete setting at 80 °C. In contrast, appropriate dosages of high-temperature retarders were added into the direct-curing formulations (h/m/l) to avoid premature setting before the slurry reaches the target temperature of 240 °C. The setting time of the cement slurry formulations were measured using a high-temperature high-pressure consistometer, and the detailed testing procedure can be found in the relevant research (Guo et al., 2023).

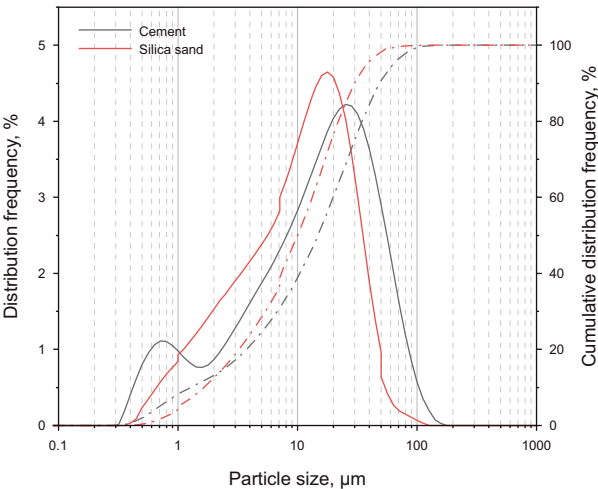


Fig. 1. Particle size distribution analysis of cement and silica.

Table 2
Summary of materials properties: particle size and specific gravity.

Materials	D10, μm	D50, μm	D90, μm	Specific gravity
Cement	1.245	14.775	49.107	3.17
Silica sand	2.071	14.190	42.307	2.62

Table 3
Main oxide of bulk materials.

Materials	Cement, %	Silicand, %
SiO ₂	19.082	99.556
Al ₂ O ₃	2.656	0.324
CaO	65.275	
Fe ₂ O ₃	4.744	
K ₂ O	0.721	
MgO	2.396	
SO ₃	4.312	

Table 4
Densities and cement formulation designs.

Formulation	h70	h40	m70	l70	H70	H40	M70	L70
Cement	100	100	100	100	100	100	100	100
Silica sand	70	40	70	70	70	40	70	70
Retarder	3.5	3.5	3	2.5	0	0	0	0
Dispersant	5	4.5	4	3	5	4.5	4	3
Suspending agent	0.15	0.2	0.25	0.6	0.15	0.2	0.25	0.6
Fluid loss additive	3.5	3.5	3	3	3.5	3.5	3	3
Defoamer	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
Water	51.6	45	76	114	56.7	50	81	119
Design density, g/cm ³	1.90	1.90	1.75	1.6	1.9	1.9	1.75	1.6
Setting time, min	200	284	240	252	/	/	/	/

2.3. Sample preparation

The overall process for cement slurry preparation, curing, and testing in this study is illustrated in Fig. 2. Initially, the dry solid materials were mixed with water containing the designated additives using a high-speed mixer following the API standard (API, 2013). The dry materials were gradually introduced into the mixing cup at 4,000 rpm, followed by high-speed mixing at 12,000 rpm for 35 s to ensure homogeneity. The resulting slurry was then poured into previously prepared stainless steel molds (approximately 70 mm in height and 25 mm in internal diameter). To eliminate entrapped air, the slurry was gently puddled with a glass rod. For the pre-curing regime, the filled molds were placed in a water bath at 80 °C and cured for 48 h, then the samples were demolded, processed (end cutting and gridding to a final size of 50 mm in height and 25 mm in diameter) and finally placed inside a HTHP curing autoclave. For the direct high-temperature curing regime, the filled molds were directly placed inside the HTHP curing autoclave. To minimize experimental variations, the pre-cure and direct-cure samples were placed inside the same autoclave for the high temperature curing process. After a designated curing period, temperature and pressure of the autoclave were gradually brought to the atmospheric condition and the direct-cure samples were demolded and processed (end curing and grinding) for further evaluations. For each formulation and curing regime, five samples were prepared: three specimens were subjected to uniaxial compressive strength testing, while two specimens were reserved for permeability measurements. Fragments of

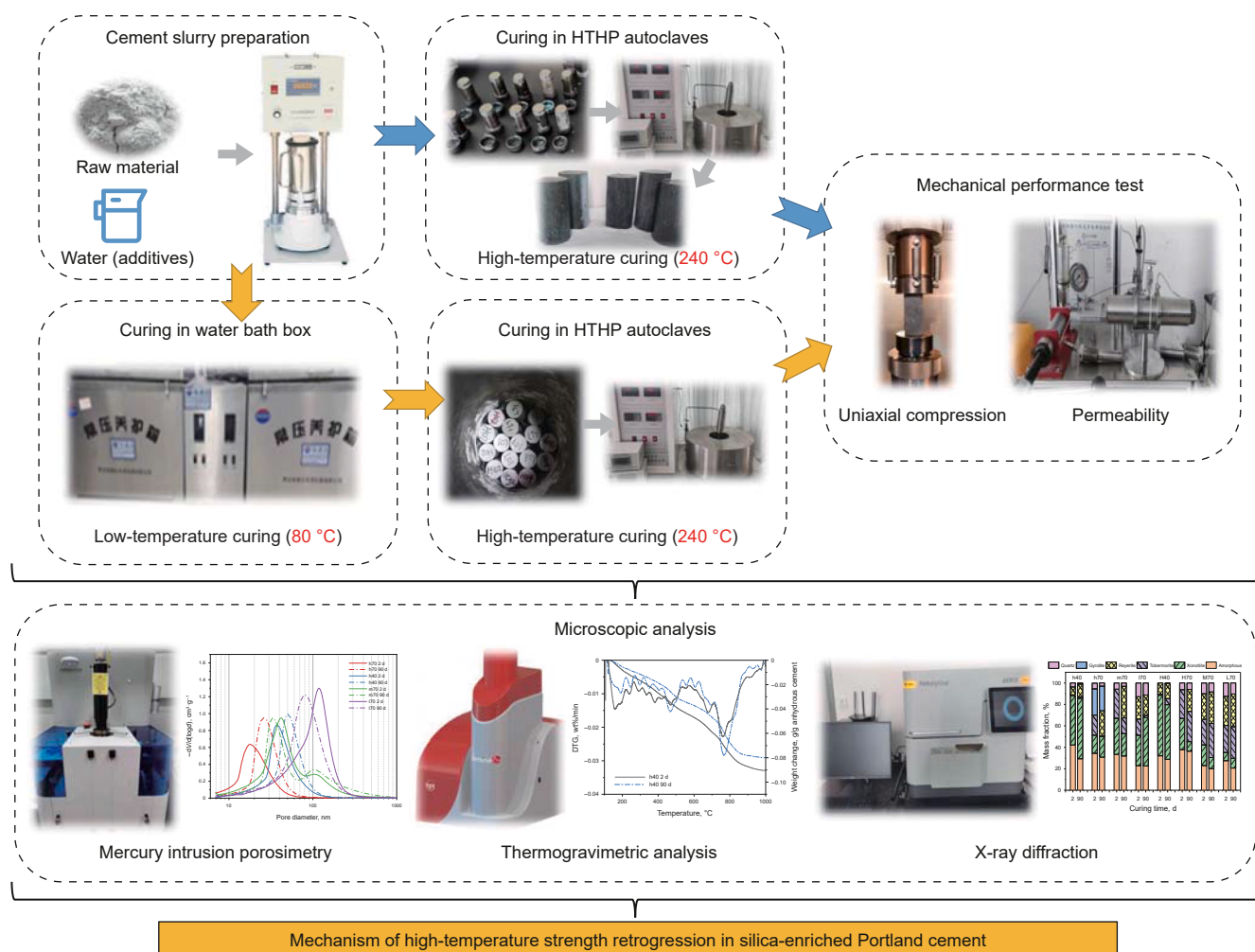


Fig. 2. Cement sample preparation, curing, and testing process.

set cement were dried and prepared for microstructural analyses according to the testing protocols of X-ray diffraction (XRD), thermogravimetry (TG) and mercury intrusion porosimetry (MIP).

2.4. Test methods

2.4.1. Sedimentation stability test

Sedimentation stability was evaluated by measuring the maximum density variation within cylindrical cement specimens (25 mm in diameter and 70 mm in height). Each specimen was sectioned into four equal parts using a precision cutting machine. The density of each section was determined according to API standard (API, 2013), i.e. by following the Archimedes' principle.

2.4.2. Mechanical property test

UTM5105X microcomputer-controlled electronic universal testing machine from Shenzhen SUNS Technology Company was used for the compressive strength test. The set cement samples were cut to a final size of 50 mm in height and 25 mm in diameter with a cutter, having their end surfaces polished with a grinder. The samples were kept in water and blot dry with a wet paper towel to achieve the "saturated surface dry condition" before each test. The loading rate employed was 150 N/s, which is equivalent to 0.30 MPa/s for the cylindrical samples used in this study with a

diameter of 25 mm. For each formulation, three replicate specimens were tested to check data reliability and reproducibility.

2.4.3. Water permeability test

Permeability of set cement samples were evaluated using water permeability tests, as cement in the wellbore is generally under saturated condition due to the presence of formation water. Water permeability tests were performed using a testing apparatus designed following the principle of Darcy's Law and manufactured by Haian Huacheng Company, Nantong, Jiangsu, China. The sample was placed inside a core holder and the upstream pressure was maintained at 3 MPa using an automatic-control piston pump while the confining pressure was maintained at 5 MPa using a hand-control piston pump. The water flow rate was measured using a balance monitoring the mass of water flowing out the sample holder at atmospheric pressure. The water permeability was calculated with Eq. (1):

$$K = -\frac{Q\mu L}{A\Delta P} \times 1000 \quad (1)$$

where K is the water permeability of the sample, mD; Q is the measured water flow rate, $\text{cm}^3 \cdot \text{s}^{-1}$; ΔP is the pressure difference for water permeability test, atm (or ~ 0.1 MPa); μ is the viscosity of water, 1 mPa·s; L is the sample length, cm; A is the sample cross-sectional area, cm^2 .

2.4.4. XRD test

Following compressive strength testing, selected fragments were collected from the interior of the crushed specimens, which were vacuum-dried for three days, and then ground into fine powders using an agate mortar. XRD analysis was conducted using a Panalytical Aeris benchtop X-ray diffractometer over a scanning range of 7° to 70° , with a scan time of 31 min and a step size of 0.01° , to identify mineralogical phases. Phase quantification analysis was performed using the Highscore Plus software (version 5.0), employing the Rietveld refinement strategy along with the external standard method, where a monocrystalline silicon was used as the external standard. The calculation formula for the external standard method is shown as follows:

$$W_j = \frac{S_j \rho_j V_j^2}{S_s \rho_s V_s^2} W_s \frac{\mu_m}{\mu_s} \quad (2)$$

In Eq. (2), W_j represents the mass fraction of phase j in the sample; S_j is the scale factor obtained from the Rietveld refinement of phase j ; ρ_j is the unit cell density of phase j ; and V_j denotes the unit cell volume of phase j . Similarly, S_s , ρ_s , and V_s represent the corresponding parameters of the standard material. W_s is the weight fraction of the standard phase in the external standard (100% in this study). μ_m and μ_s are the mass absorption coefficients of the sample and the standard material, respectively. More detailed description about this method can be found in our previous publications (Qin et al., 2022; Pang et al., 2022).

2.4.5. MIP test

For MIP analysis, cylindrical cement samples were initially sliced into thin discs and further crushed into particles smaller than 5 mm in their largest dimensions, totaling approximately 1 g in mass. Testing was performed using a Quantachrome Mercury Intrusion Pore Size Analyzer (Model PM 33), with a maximum intrusion pressure of 32,000 psi (220 MPa). Pore size distributions were derived using standard calculations, assuming the mercury surface tension to be 0.485 N/m and a contact angle of 140° between mercury and the cement matrix.

2.4.6. TGA

TGA was conducted using a Setaram Thermal Analyzer (Model Setline STA). Sample preparation closely followed similar procedures used for XRD analysis. Approximately 60 mg of ground sample was placed in an alumina crucible (90 mm^3). The temperature was increased from 28 to 105°C at a rate of $5^\circ\text{C}/\text{min}$ and held for 1 h at 105°C to remove evaporable water. Subsequently, the temperature was ramped from 105 to 1000°C at the same rate to remove non-evaporable water. Only the decomposition of the non-evaporable water (i.e. the heating stage from 105 to 1000°C) was analyzed.

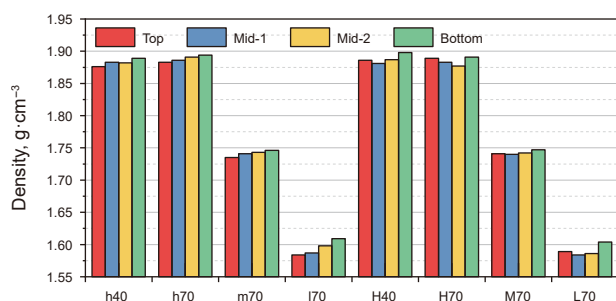


Fig. 3. Sedimentation stability test results for different formulations.

3. Results and discussion

3.1. Sedimentation stability

Fig. 3 presents the results of the sedimentation stability tests of all formulation. The direct high-temperature cured samples were demolded and sectioned after 2 days of curing at 240°C , while the low-temperature cured samples were processed after 2 days of curing at 80°C . All specimens exhibited excellent sedimentation stability, with maximum density variations below $0.02 \text{ g}/\text{cm}^3$. Moreover, the average measured densities closely aligned with the corresponding design values for each formulation. This high degree of homogeneity ensured that vertical segregation was negligible, ensuring the reliability of subsequent mechanical and microstructural evaluations.

3.2. Compressive strength

Fig. 4 illustrates the compressive strength evolution of the SEPC systems under different curing regimes. Under direct high-temperature curing at 240°C (Fig. 4(a)), the slurry with 40% silica (h40) exhibited strong stability over time: although its 2-day strength is relatively low at 30 MPa, it showed no strength degradation and the strength even increased slightly (to approximately 35 MPa) at 90 days. In contrast, all of the slurries with 70% silica (h70, m70, and l70) exhibited strength retrogression. As expected, both short-term and long-term strengths of these slurries decreased with decreasing slurry density. The 2-day strength of slurry h70 is significantly higher than that of h40 (reaching 44 MPa), but it decreased significantly to 31 MPa after 30 days, rendering its long-term strength slightly similar to that of h40. It should be noted that no medium and low-density slurries prepared with 40% silica dosage were studied here due to poor performances under direct-cure condition.

As shown in Fig. 4(b), under the condition of low-temperature curing followed by high-temperature curing (pre-cure), the strength evolution trends differed markedly. Across all formulations, compressive strength either stabilized or improved after the transition to high-temperature curing. Notably, slurries H40 and L70 exhibited the most significant strength gains after the temperature increase. Long-term strength developments of all slurries are significantly improved in this curing regime. For example, the strength of slurry H70 increased from 28 MPa at 2 days to over 50 MPa at 90 days. The strength of slurry H40 (which had a higher cement content than slurry H70) achieved 45 MPa at 2 days and 55 MPa at 90 days. Although slurries M70 and L70 exhibited similar 2-day strengths (18–20 MPa), their long-term behaviors diverged significantly: the strength of slurry M70 reached 40 MPa at 90 days, while that of slurry L70 remained at around 21 MPa.

3.3. Water permeability

Fig. 5 illustrates the influences of curing regimes on the water permeability of SEPC systems. Under direct high-temperature curing at 240°C (Fig. 5(a)), the permeability of slurry h70 was extremely low at 2 days, indicating a relatively dense microstructure at early age. However, after 30 days of curing, permeability increased by approximately one order of magnitude, reflecting microstructural coarsening. From 30 to 90 days, permeability of slurry h70 stabilized, suggesting that a plateau in structural degradation was reached. Slurry h40 exhibited consistently greater permeability than slurry h70 at all curing times but showed no significant increase over time, consistent with its stable compressive strength. The permeability of slurry m70 exhibited progressive increases with time over long-term curing, aligning

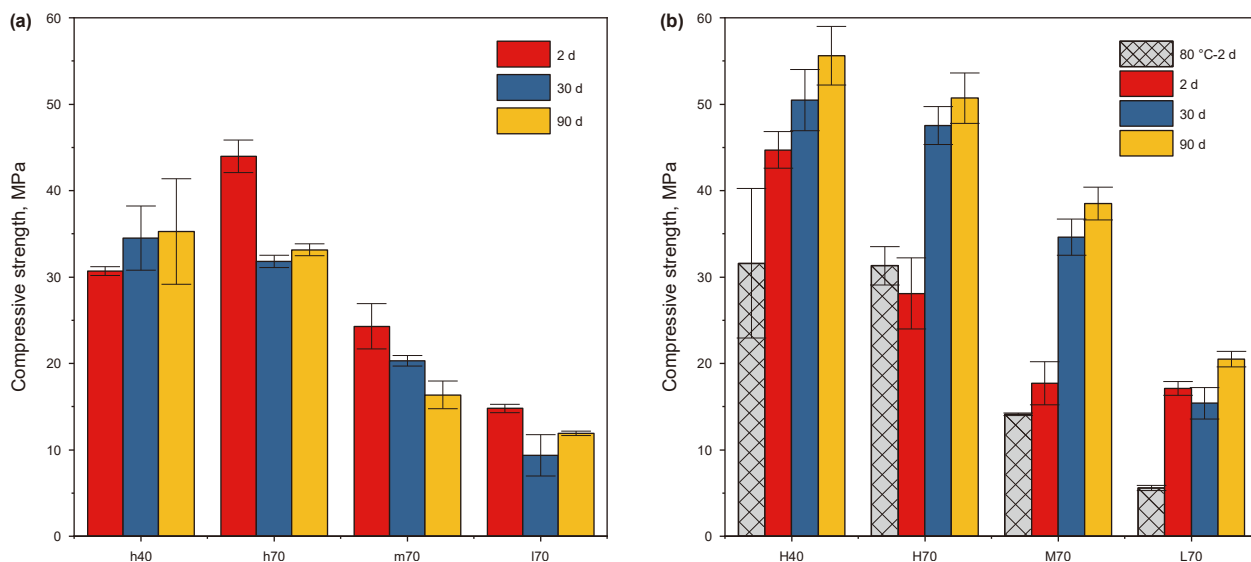


Fig. 4. Variation of compressive strength of SEPC with curing time. (a) Direct high-temperature curing at 240 °C; (b) pre-curing at 80 °C followed by further curing at 240 °C.

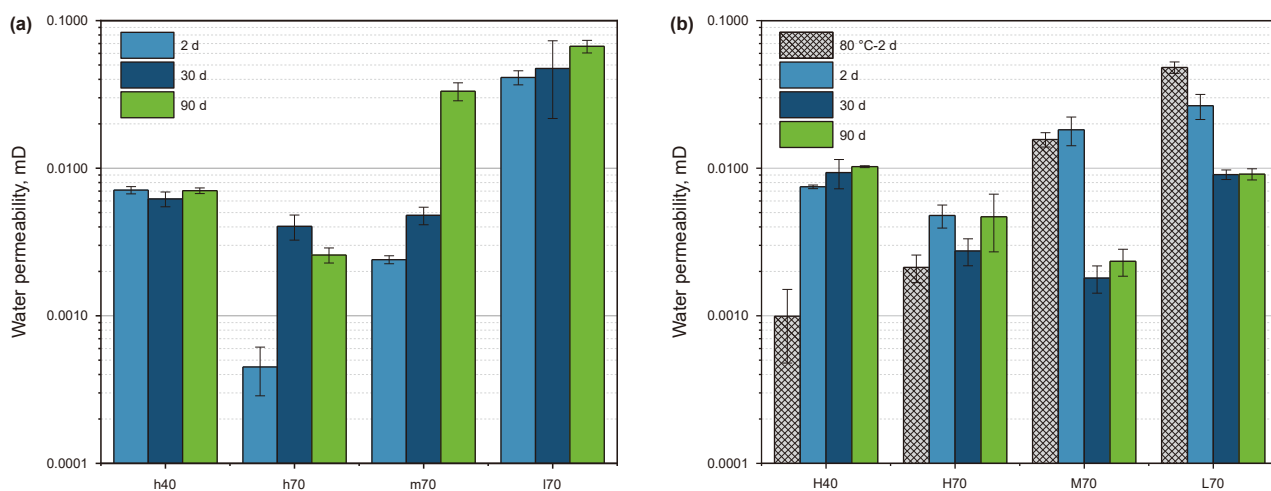


Fig. 5. Variation of cement permeability with curing time under different setting temperatures. (a) Direct curing at 240 °C; (b) pre-curing at 80 °C and 240 °C further curing.

with the observed strength retrogression. The low-density slurry l70 showed persistently high permeability (>0.04 mD) from 2 to 90 days, indicating poor microstructural integrity. In contrast, permeability evolution trends were markedly different for pre-cured samples (Fig. 5(b)). Slurries H40 and H70 exhibited relatively low initial permeability after pre-curing for 2 days, which increased after high temperature exposure, with slurry H40 exhibiting a much stronger increase. However, permeability of both slurries remained relatively stable throughout the subsequent 90-day curing period. Slurries M70 and L70 displayed relatively high initial permeability after pre-curing, which did not increase after high temperature exposure for 2 days and even showed significant reductions during further exposure to 30 days, indicating densification of the cement matrix with extended high-temperature curing. When comparing the final permeability values at 90 days, the high-density formulations (H70 and h70, 1.9 g/cm^3) exhibited similar permeability levels under both curing regimes, with slightly lower values observed under direct high-temperature curing. However, for the medium-density (1.75 g/cm^3) and low-density (1.6 g/cm^3) formulations, direct high-

temperature curing resulted in significantly higher permeability than the low-temperature pre-curing approach, further confirming the stabilizing effect of pre-curing.

3.4. MIP

Fig. 6 presents the MIP results of the cement samples pre-cured at 80 °C for 2 days. The higher-density samples H40 and H70 exhibited similar total porosities of approximately 35% after curing at 80 °C for 2 days, along with comparable pore size distributions. As the slurry density decreased, the total porosity of M70 and L70 increased to about 39% and 51%, respectively, accompanied by significant increases in median pore size, which is consistent with the compressive strength (Fig. 4) and permeability (Fig. 5) results. The detailed evolution of pore size distribution will be further discussed later.

Fig. 7 presents the MIP test results of SEPC formulations under different curing regimes. It can be observed that, for the same slurry density of 1.9 g/cm^3 , higher silica dosage led to lower porosity under both curing regimes. At the same silica dosage of

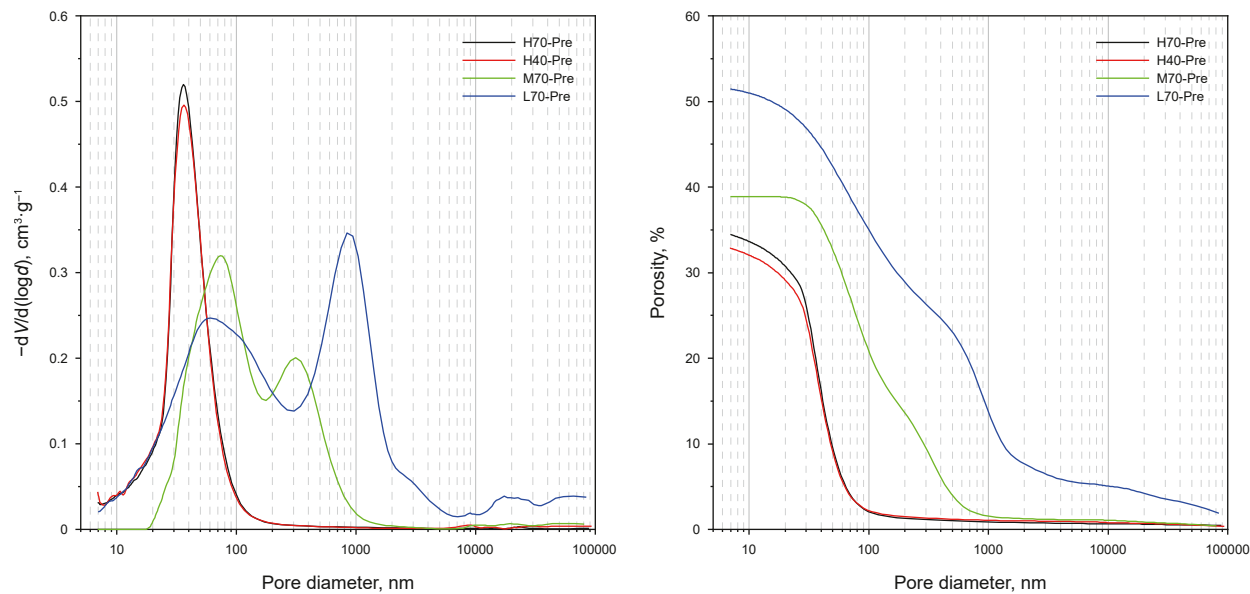


Fig. 6. MIP analysis of cement pre-cure at 80 °C.

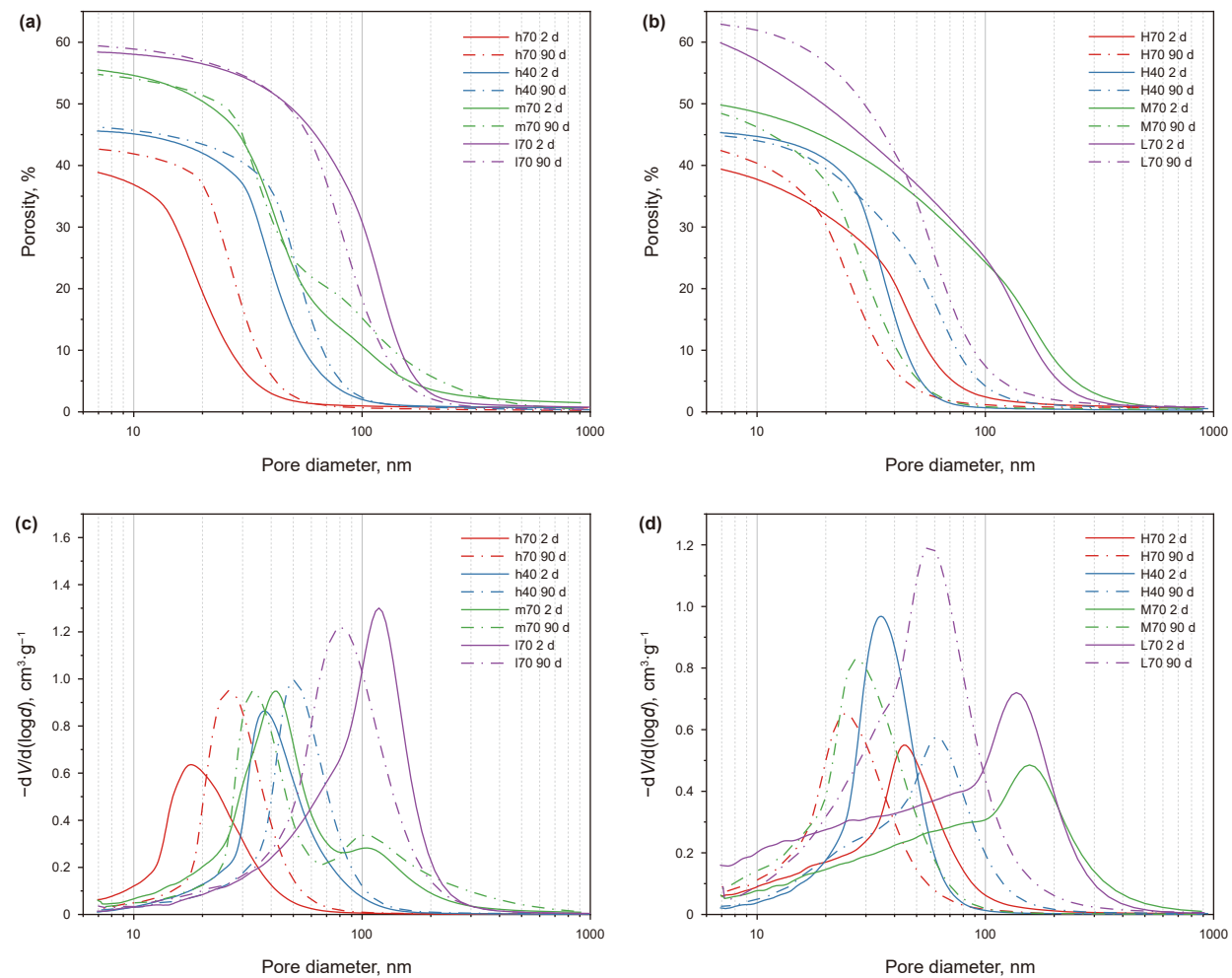


Fig. 7. MIP analysis of cement under different curing regimes. (a, c) Direct curing at 240 °C; (b, d) pre-curing at 80 and 240 °C further curing.

70%, the total porosity increased with decreasing slurry density due to higher water contents. Under direct high-temperature curing, the total porosity of all formulations (except for h70) remained largely unchanged with extended curing time. Specifically, slurry h70 exhibited a moderate increase in total porosity, from 39% to 43%, while slurries h40, m70, and l70 maintained constant porosity values of approximately 46%, 55%, and 59%, respectively. In terms of pore size evolution, the median pore diameters of the high-density slurries h70 and h40 (1.9 g/cm^3) increased by around 10 nm, indicating structural coarsening. In contrast, slurries m70 and l70 showed slight reductions in median pore diameter by roughly 5 nm and 25 nm, respectively. The total porosities of the pre-cure samples after initial high temperature exposure for 2 days were similar to those of the direct-cure samples at 2 days except for the medium density slurries, with M70 exhibiting an obviously lower total porosity than m70. Slurries H70 and L70 exhibited slight porosity increases with increasing curing time, while slurries H40 and M70 maintained nearly constant porosity values. At 70% silica dosage, the pore diameter showed a decreasing trend with time in all pre-cure samples. However, the slurry with 40% silica dosage (H40) exhibited relatively significant pore diameter increase with time. Overall, the pore size distribution curves of the pre-cure samples changed more significantly with increasing curing time than those of the direct-cure samples.

According to the pore size classification proposed in previous studies (Shen et al., 2018; Cheng et al., 2024a, 2024b), pores in cementitious materials can be categorized as: harmless pores (<20 nm), less-harmful pores (20–50 nm), harmful pores (50–200 nm), and more-harmful pores (>200 nm). A refined pore structure analysis was conducted based on the pore classification method, and the results are presented in Fig. 8. Under direct high-temperature curing, pronounced pore coarsening was observed in the high-density systems h70 and h40. However, the permeability and strength of slurry h40 were stable probably because the contents of more-harmful pores remained low. Meanwhile, the medium- and low-density slurries m70 and l70 exhibited relatively small changes in pore size distribution over long-term curing, yet slurry m70 exhibited apparent strength retrogression and permeability increase, which may be explained by the relatively significant increase in the contents of more-harmful pores (from 3.49% to 5.3%). Under the pre-curing regime, as the curing temperature increased from 80 to 240 °C, the total porosity of all samples increased to some extent; however, the contents of

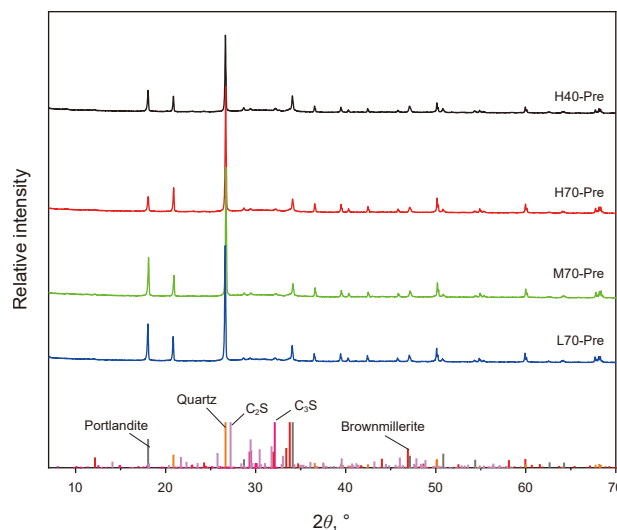


Fig. 9. XRD analysis of SEPC (after pre-cured at 80 °C for 2 days).

harmful and more-harmful either remain stable or decreased significantly (e.g. low-density sample L70), which explained its significant strength gain after high temperature exposure. With further increase in curing time, pre-cured slurries with 70% silica dosage all exhibited significant pore refinement trends. In particular, the contents of more-harmful pores in medium- and low-density slurries M70 and L70 decreased dramatically, explaining their significantly decreased permeability with time.

3.5. XRD

Fig. 9 displays the XRD patterns of SEPC formulations after curing at 80 °C for 2 days. The main crystalline phase in the hydrated cement are unreacted silica, or quartz (COD: 96-500-0036), unreacted C4AF, or brownmillerite (COD: 96-901-6158), and hydration product portlandite (COD: 96-500-0036). Unreacted C₃S (COD: 96-901-6126) and C₂S (COD: 96-210-3317) were also detected. As mentioned previously, the main hydration product of well cement at such a low temperature is amorphous C–S–H gel, which has very weak diffraction peaks. Obviously, the high

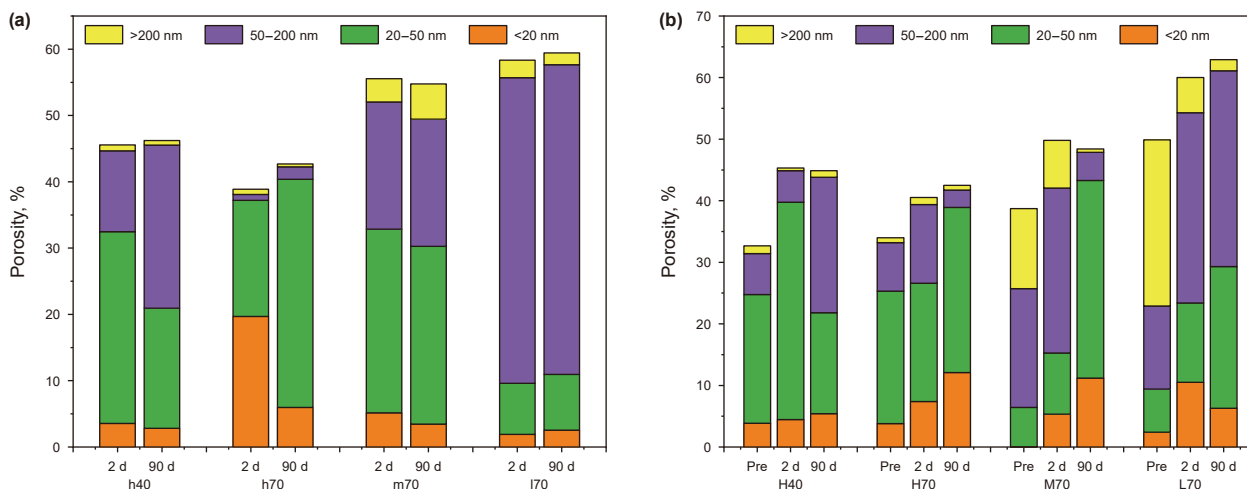


Fig. 8. MIP pore distribution analysis under different curing regimes. (a) direct curing at 240 °C; (b) pre-curing at 80 °C and 240 °C further curing.

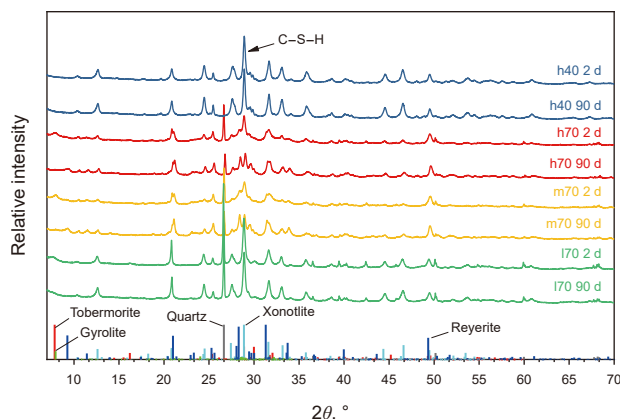


Fig. 10. XRD analysis of SEPC (direct curing at 240 °C).

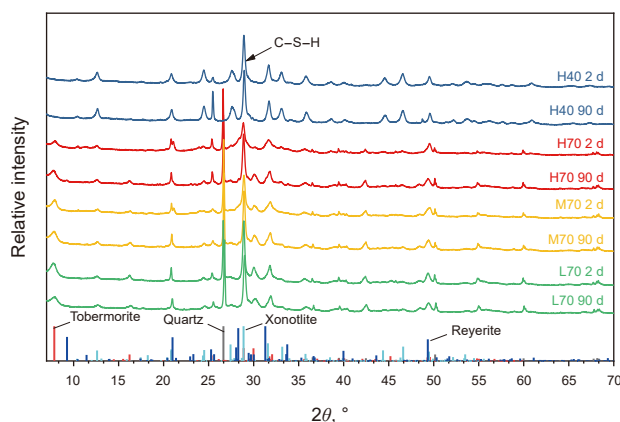


Fig. 11. XRD analysis of SEPC (pre-curing at 80 °C followed by 240 °C further curing).

crystalline quartz peak had made diffraction peaks of hydration products less apparent.

Figs. 10 and 11 display the XRD patterns of SEPC formulations under different curing regimes after high temperature curing. Across all formulations, the primary crystalline hydration products identified were tobermorite (COD: 96-900-5498), xonotlite (COD: 96-900-8440), reyerite (COD: 96-900-9471), and residual quartz derived from unreacted silica. Notably, quartz peaks were absent in the 40% silica formulations, suggesting complete consumption of the silica sand. The broad hump peak observed between 28.5° and 31°, typically attributed to the amorphous C-S-H, became

progressively narrower and more intense with increasing curing duration and with decreasing slurry density. This trend suggests a gradual transformation of amorphous C-S-H into more ordered crystalline phase (Sun et al., 2023). Therefore, it appears that decreasing slurry density has a similar effect as increasing curing time in transforming amorphous C-S-H into crystalline phases, possibly due to the fact that higher water content promoted the crystallization process, which involves dissolution and precipitation.

Fig. 12 presents the quantitative XRD analysis results, highlighting the hydration product phase evolution under various conditions. Under direct high-temperature curing, the tobermorite content in the 70% silica formulations decreased markedly over time, with xonotlite and reyerite becoming the dominant crystalline phases by 90 days. Conversely, in samples initially set at low temperatures and subsequently cured at high temperatures (pre-cure samples), the tobermorite content increased progressively, while xonotlite content declined, which is the main reason for their long-term strength gains. After 90 days, these formulations were predominantly composed of tobermorite and reyerite. In low-silica (40%) formulations, the tobermorite content remained low throughout the curing period regardless of the setting temperature. Although a slight increase in tobermorite was observed at 90 days in pre-cured samples, xonotlite and reyerite continued to dominate. Notably, gyrolite formation was identified only in the directly cured high density slurry h70, suggesting a unique crystallization pathway facilitated by high silica content, relatively low water content, as well as elevated setting temperature. Additionally, higher residual quartz content was observed in the low-density formulations (e.g., l70 and L70), indicating incomplete silica dissolution and less extensive pozzolanic reactions compared to the higher-density formulations. Specifically, the degradation in compressive strength under direct high-temperature curing is closely associated with the reduction of tobermorite and its transformation into coarser crystalline phases, such as xonotlite and gyrolite, which contribute to microstructural degradation. In contrast, strong retention and continued formation of tobermorite in pre-cured samples contribute to significantly improved strength and pore structure stability. For low-silica (40%) formulations, no clear phase transition was observed throughout the curing process. As a result, compressive strength remained relatively stable. However, microstructural coarsening and increased permeability were still evident, likely attributed to the gradual crystallization of initially formed amorphous C-S-H or the phase transitions of xonotlite and reyerite.

3.6. TGA

Fig. 13 presents the TG and DTG curves of SEPC formulations subjected different curing regimes. The DTG profiles exhibit two decomposition ranges and three primary decomposition peaks, each corresponding to the dehydration or thermal decomposition of specific hydrated phases: (i) two primary peaks were identified in the temperature range of 105–600 °C, with a relatively sharp peak at approximately 200 °C, and a wider peak near 400 °C, which can be primarily attributed to the dehydration of semi-crystalline C-S-H and other minerals such as tobermorite (Eisinas et al., 2012; Lothenbach et al., 2022; Qin et al., 2023; Cheng et al., 2024a); (ii) A third peak at approximately 780 °C was identified in the decomposition range of 600–1000 °C, which is associated with the decomposition of thermally more stable crystalline phases such as xonotlite and reyerite (Lothenbach et al., 2022; Cheng et al., 2024b). For the samples pre-cured at 80 °C for 2 days, the decomposition peak observed at around 450 °C can be attributed to the decomposition of portlandite (Pang et al., 2022). However,

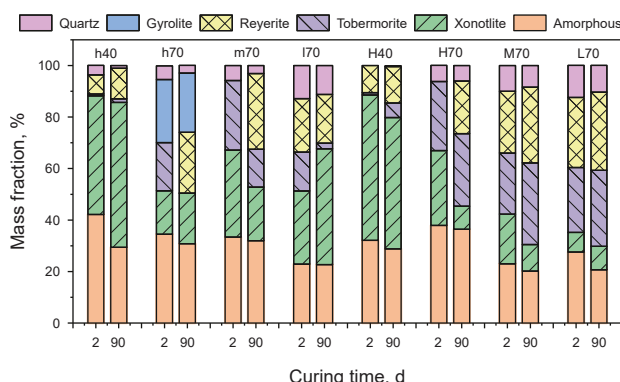


Fig. 12. Quantitative XRD analysis of SEPC.

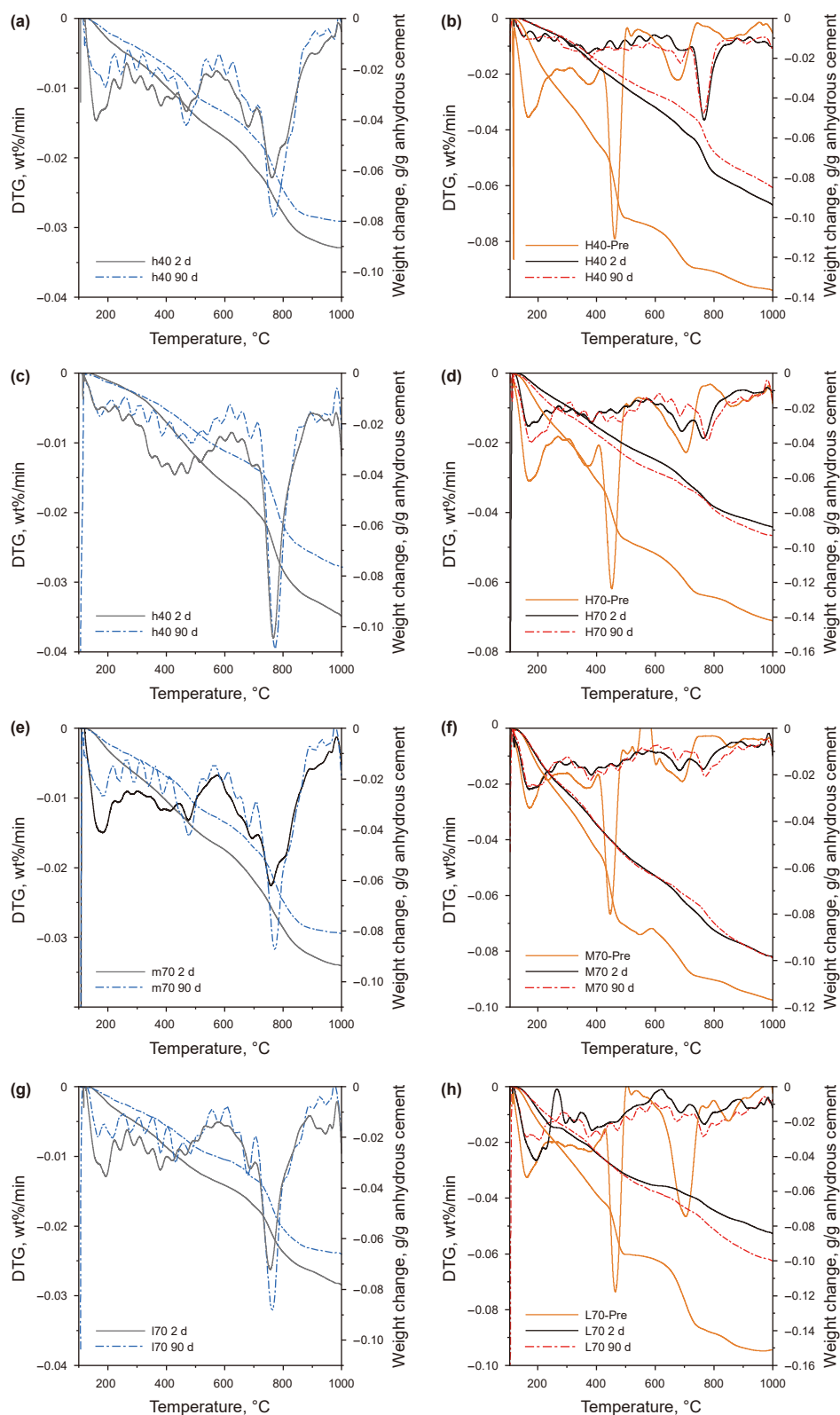


Fig. 13. TGA of cement under different curing regimes. (a, c, e, g) Direct curing at 240 °C; (b, d, f, h) pre-curing at 80 and 240 °C further curing.

when the curing temperature was increased to 240 °C, the decomposition peak of portlandite almost completely disappeared, and the main hydration products as well as the DTG curves became similar under both curing regimes.

Under direct high-temperature curing at 240 °C, all SEPC formulations exhibited prominent DTG peaks near 780 °C, corresponding to the high-temperature dehydration of xonotlite and reyerite. These peaks intensified noticeably from 2 to 90 days for

the formulations with 70% silica, indicating continued formation of these thermally stable crystalline phases. Formulations with high xonotlite content tend to show significantly narrower peaks than those with low xonotlite content, indicating its unique decomposition behavior. The formulation with 40% silica (h40) exhibited apparently different decomposition peak profiles compared with those with 70% silica, despite similar hydration product compositions. For example, formulation h40 showed almost no peak at 200 °C with only a broad peak at approximately 400 °C, while all formulations with 70% silica showed strong decomposition peaks at 200 °C and the peaks spanned from 200 to 450 °C. Such differences are possibly attributed to different structural morphologies of the C–S–H formed in the two types of formulations. The intensity of both peaks decreased over time, reflecting the structural transformation or consumption of C–S–H and tobermorite. Concurrently, the total bound water content in all direct-cured formulations decreased with curing time.

For pre-cured samples with 40% silica (H40), the decomposition profiles were very similar to those of the direct-cured samples (h40), with a strong peak near 780 °C, further confirming the dominance of the stable xonotlite phase in both samples. However, the reduction in total bound water content was less significant in the former (0.009 g in sample H40 compared with 0.02 g in sample h40, normalized based on per gram of anhydrous cement). For pre-cured samples with 70% silica (H70, M70 and L70), the DTG decomposition profiles exhibited less prominent peaks near 780 °C, but stronger peaks near 200 °C, compared with those of the direct-cured samples (h70, m70, l70), possibly due to increased tobermorite content and decreased xonotlite content; additionally, the peaks at both 200 and 780 °C were also more stable than the direct-cured samples with increasing curing time, leading to almost no changes or even increases in total bound water content with increasing curing time. Specifically, bound water per gram of anhydrous cement increased from 0.089 to 0.094 g in sample H70, remained constant at 0.099 g in M70, while it increased from 0.084 to 0.1 g in L70. These results further support the hypothesis that the low-temperature pre-curing facilitates the retention of tobermorite phase and promotes microstructural refinement during subsequent high-temperature curing.

Fig. 14 presents the variation in bound water content (i.e. dehydrated water during TG tests) of cement samples under different curing regimes. For the samples directly curing at 240 °C, it is clear that the variations in total bound water content were strongly correlated with the dehydration of amorphous C–S–H and tobermorite over the 105–600 °C range. Sample h40 showed the most significant decline in bound water content from 2 to 90 d

curing, with a reduction of approximately 0.013 g (per gram of dry cement) in the range of 105–600 °C, and a further decrease of about 0.0067 g/g in the range of 600–1000 °C. For the samples with 70% silica sand, the bound water content decreased uniformly by approximately 0.01 g/g in the range of 105–600 °C, whereas almost no change was observed in the range of 600–1000 °C (extremely small reductions of 0.001, 0.002, and 0.0023 g/g, were recorded in samples h70, m70 and l70, respectively). For the pre-cured samples, a decline in bound water content was observed only in the 40% silica sand sample (H40), and the magnitude of reduction was significantly lower than that of h40. Conversely, the 70% silica sand samples exhibited either stable or slightly increased bound water contents with increasing curing time, which is consistent with their increasingly refined microstructure.

4. Discussion

The high temperature strength retrogression problem of Portland-cement-based materials has been studied for several decades and SEPC has long served as the most commonly used well cementing material in the industry for high temperature applications. Studies dealing with the long-term performances of SEPC systems are very limited due to the stringent requirement on the test environment. Only in recent years, the long-term strength retrogression phenomenon of SEPC systems subjected to direct high temperature curing regime has been revealed. It is reported that the early compressive strength of SEPC systems cured under 200 °C is strongly dependent on silica dosage and fineness, which can reach up to 80 MPa when relatively fine silica was added at dosages of 60% to 100% (Qin et al., 2021). Further analysis indicates that, increasing silica dosage can significantly decrease the xonotlite content and increase the contents of amorphous C–S–H and tobermorite phases in SEPC systems cured at 200 °C, which is the primary reason for these systems to achieve high early strengths. However, due to the relatively poor stability of amorphous C–S–H at high temperatures, the high-strength SEPC systems experience severe long-term strength retrogression during long term curing, with strength declines of 60%–80% typically observed over curing periods of 142 to 180 d (Pang et al., 2021; Qin et al., 2023). In contrast, silica dosage is found to have little effect on the compressive strength of SEPC systems directly cured under 260 °C, the early compressive strength was relatively low at approximately 30 MPa, but remain highly stable with increasing curing time (Pang et al., 2023). The lower initial strength as well as better stability can be primarily attributed to the generation of high contents of xonotlite phase, low contents of amorphous C–S–H phase as well as absence of the tobermorite phase. Therefore, the long-term strength retrogression of SEPC systems does not necessarily intensify with increasing curing temperature. When the direct curing temperature is set at 240 °C (as in this study), it is found that the silica dosage has a moderate effect on the early strength of SEPC systems and that the system prepared with high silica dosage (h70) exhibited moderately high early strength at approximately 44 MPa and moderate degree of strength retrogression (declined to 31 MPa over 90 d curing). Clearly, the findings of this study are consistent with previous studies and further confirm that the long-term strength retrogression of SEPC systems shows nonlinear evolution behavior.

Compared to cement slurries directly set and cured at 240 °C, slurries pre-cured at 80 °C and subsequently cured at 240 °C exhibit completely different strength evolution profiles. The high-density low-silica slurry (H40) and the low-density high-silica slurry (L70) exhibited significant immediate strength gain upon only 2 d high temperature exposure, indicating faster pozzolanic reactivity. In contrast, both the high-density and medium-density

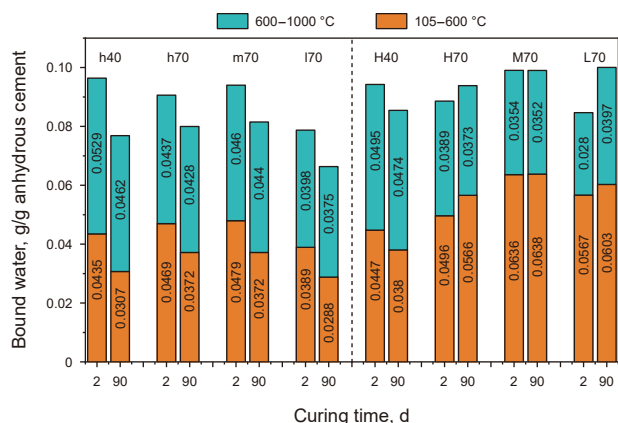


Fig. 14. Bound water change under different curing regimes.

slurry with high silica content (H70 and M70) exhibited significant strength gain from 2 to 30 d of curing, suggesting relatively slow pozzolanic reactivity. In any case, the long-term high-temperature stability of the pre-cure samples were significantly better, with their 90 days strength 57% to 137% higher than those of the direct-cure counterparts. In particular, slurry H40 has almost identical composition, porosity and permeability as slurry h40 after high temperature curing, but its compressive strengths are on average 34% higher than those of h40 across all curing ages. When the test results of the slurries with different densities were compared, it is clear that both the early-age and long-term strengths decreased with decreasing slurry density due to increased water content, which lead to higher porosity. Interestingly, decreasing slurry density led to the formation of higher xonotlite phase and lower reyerite/gyrolite phases in the direct-cure samples, whereas the same process led to the formation of lower xonotlite phase and higher reyerite content in the pre-cure samples. Under direct high-temperature curing conditions, the crystalline phase could originate from both conversion from amorphous C–S–H phase (formed during hydration) and direct nucleation-growth of crystalline nucleus. The high free water content in low density slurries probably favored the formation of xonotlite nucleus and promoted the formation of more xonotlite phase. In pre-cured samples, the crystalline phase primarily come from transformation of amorphous C–S–H gel previously formed at 80 °C curing temperature, and such process appear to favor the formation of reyerite phase with increasing water content.

Along with other published (Pang et al., 2021, 2023) studies in recent years, the results of this study further confirmed that the long-term strength degradation of SEPC is primarily driven by the transformation of amorphous C–S–H and tobermorite into other crystalline phases such as xonotlite and reyerite, as well as the coarsening of amorphous C–S–H structure itself. It appears that this process can be at least partially reversed for the curing regime that adopts a low initial setting temperature, which lead to strength enhancement with increasing curing time instead of strength retrogression. Under the curing regime that adopts direct high temperature curing, decreasing silica dosage (from 70% to 40%), increasing curing temperature (from 200 to 240 or 260 °C) and increasing mixing water content (slurry density decreasing from 1.9 to 1.6 g/cm³) can all lead to less amorphous C–S–H and more xonotlite phase contents in the SEPC systems. This could be attributed to the enhanced ion dissolution and mobility resulting from the decreased silica dosage, elevated curing temperature, and increased water content, which collectively promoted the direct nucleation and growth of xonotlite nucleus during hydration. These changes lead to relatively poor initial physical and mechanical properties of the hardened cement, but enhance its long-term stability.

5. Conclusions

To evaluate the long-term stability of silica-enriched Portland cement (SEPC) at ultra-high temperatures, several influencing factors were investigated, including curing regime (direct high temperature curing versus low temperature pre-curing followed by high temperature curing), curing temperature (240 °C in this study compared with 200 and 260 °C in the literature), slurry density (from 1.6 to 1.9 g/cm³), and silica dosage (70% vs 40%, by weight of cement). The following key conclusions can be drawn:

- (1) For SEPC systems with high silica content, setting temperature has a strong influence on their long-term stability: SEPC systems directly set and cured at 240 °C experience apparent strength retrogression over time, while those pre-

cured at 80 °C before 240 °C exposure experience significant strength gain over time. The macroscopic strength retrogression phenomenon is accompanied by permeability increase, microstructural coarsening, bound water content reduction, as well as phase transformations (primarily from tobermorite to xonotlite and reyerite). In contrast, pre-cured systems with strength gain over time exhibit stable or decreased permeability, microstructure refinement, stable or increased bound water content, as well as stable phase contents (favorable phase changes are observed in selected systems).

- (2) For SEPC systems with low silica content, setting temperature has relatively minor influence on their long-term stability, possibly due to their high contents of the stable xonotlite phase. However, pre-cured systems consistently exhibited higher strengths (about 34% higher) than direct-cured systems at all curing ages. The evolution of the microstructure, hydration products, and bound water content under both curing regimes was largely comparable, which explained the similar development of samples' physico-mechanical properties. Although the mechanical performance remained relatively stable over 90 days, the observed microstructural coarsening and reduction in bound water content indicated a potential risk of strength retrogression during further curing.
- (3) For the SEPC systems with 70% silica sand, a decrease in cement slurry density led to an increase in total porosity due to higher water content, thereby deteriorating both the early-age and long-term physical and mechanical properties. Under direct curing at 240 °C, more pronounced deterioration of mechanical performance was observed with decreasing density. Slurry I70 (with the density of 1.6 g/cm³) exhibited extremely low early strength, leaving little capacity for further retrogression at later ages. Overall, the medium-density slurries showed the most significant strength changes with time under both curing conditions, with slurry m70 exhibiting a 33% decline while slurry M70 exhibiting a 54% increase. The 90 days strength of M70 was 137% higher than that of m70, despite their similar mineral compositions.
- (4) Comparing the test results of this study with those published in the literature, it can be concluded that increasing curing temperature from 200 to 240 or 260 °C lead to significant reductions in amorphous C–S–H content and significant increases in xonotlite content in direct-cured SEPC systems with high silica content. These changes resulted in apparent deteriorations in early-age physical and mechanical properties of the set cement, but with the benefit of increased long-term stability. Therefore, at ultrahigh temperatures above 200 °C, while the generation of a higher content of amorphous C–S–H may contribute to greater early strength, its relatively poor structural stability will eventually lead to poor performances of the set cement over long-term curing.

CRediT authorship contribution statement

Yang Li: Writing – original draft, Formal analysis, Data curation. **Xue-Yu Pang:** Writing – review & editing, Funding acquisition, Formal analysis, Conceptualization. **Yong-Jin Yu:** Writing – original draft, Project administration, Investigation. **Sheng-Lai Guo:** Writing – review & editing, Data curation. **Jia-Pei Du:** Writing – review & editing. **Hai-Ge Wang:** Project administration, Investigation. **Kai-He Lv:** Writing – review & editing, Project

administration. **Jin-Sheng Sun:** Writing – review & editing, Project administration, Funding acquisition.

Declaration of competing interests

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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