Hydration and pore structure characteristics of concrete incorporating internal curing materials in a dry and large-temperature-difference environment

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Abstract

The climate in the Central Asian area is extreme dry, with a large temperature difference between day and night, and the concrete is easy to crack during the construction period under the conditions of traditional external curing. Therefore, this study concerns understanding the application of internal curing materials in a dry and large-temperature-difference environment. The effects of three internal curing materials, namely, Super Absorbent Polymer (SAP), Light Weight Aggregate (LWA) and Perforated Cenospheres (PCs), on hydration and pore structure characteristics of concrete were investigated. Scanning electron microscope (SEM) and x-ray diffractometer (XRD) were used to observe the microscopic morphology and physical phase composition of concrete, and the evolution of pore characteristics was analyzed using mercury intrusion porosimeter (MIP) to explore the mechanism of pore structure refinement. Results show that the pores formed by the release of water provide space for the accumulation of hydration products, thereby accelerating the formation of ettringite crystals. Results indicate that the continuous release of internal curing materials allows the interior of the concrete to continue to be hydrated, which greatly promotes the overall hydration of the cement. The effect of supplementary cementitious materials (GGBS, FA) and implications of large-temperature-difference environment on concrete durability are discussed.

1. Introduction

The implementation of China’s ‘One Belt One Road’ has increased infrastructure construction in Central Asia. However, the characteristics of extreme dryness, large temperature difference between day and night, and strong sunshine in Central Asia seriously threaten the infrastructure construction in the region. These harsh climatic environmental characteristics can easily lead to cracking of concrete, especially for the newly poured concrete [1]. During the construction period, windy weather and dry weather conditions caused serious water loss on the surface of the concrete [2, 3]. Coupled with the large local temperature difference, the concrete is very prone to cracks under such an environment. Cracks are a kind of discontinuity in concrete materials [4]. Concrete cracks not only affect the beauty of the structure, but also become channels for corrosive media to penetrate into the concrete, and ultimately affect the safety and durability of the concrete structure [5]. In addition, for the repair of later cracks, not only is labor and time consuming, but the repair cost will also be a huge expenditure. Therefore, in this harsh climate, how to reduce or even avoid the occurrence of early cracks in concrete structures and prolong its service life has important theoretical and practical significance.

The curing method of concrete is usually cured from the outside, that is, spraying water on the surface of the concrete, and then covering the surface of the concrete to prevent water volatilization [6], as shown in figure 1. However, for the high-performance concrete in the dry northwestern region with large temperature differences, the external curing moisture is easily evaporated due to environmental influences, and the concrete itself has a
relatively high density [7, 8]. Therefore, it is difficult for the unevaporated moisture to enter the interior of the concrete, and the curing effect is poor [9, 10]. In addition, for some hidden parts of concrete buildings, external maintenance is difficult to achieve the desired effect.

In 1991, the concept of concrete internal curing was first proposed by Philreo [7], and then many scholars have done a lot of research in this area. After the concrete is poured, with the evaporation of internal water and the continuous progress of cement hydration, the internal relative humidity gradually decreases, causing negative pressure in the capillary pores, which causes the pre-saturated internal curing material to generate water release power [11]. Therefore, it supplements the internal water consumption and plays a role in reducing the shrinkage, thereby improving the durability of the concrete and extending the service life of the concrete structure [4, 12]. Indeed, internal curing technology has made significant progress in shrinkage and cracking of concrete [13]. However, the existing investigations lack pore structure and hydration characteristics results, and their conclusions partially disagree since the type and content of internal curing materials have a significant impact on the property of concrete [14–17]. Studies on how internal curing agents influence microstructure of concrete, in particular those concretes containing supplementary cementitious materials (SCMs), are also lacking [18–20]. Therefore, the research on internal curing concrete is not sufficient. Additionally, many scholars have carried out most of the experimental studies on the shrinkage and cracking of internally cured concrete under normal temperature environmental conditions, and rarely consider the impact of changes in environmental conditions, which is inconsistent with actual projects [21, 22]. In fact, the external construction environmental conditions have an important influence on the shrinkage and cracking of concrete, especially in a dry and large-temperature-difference environment.

Based on the characteristics of the climate and environment in Central Asia, this study used an environmental simulation chamber to simulate a large temperature difference and dry environment. Scanning electron microscope (SEM) and x-ray diffractometer (XRD) were used to observe the microscopic morphology and physical phase composition of concrete, and the evolution of pore characteristics was analyzed using mercury intrusion porosimeter (MIP) to explore the mechanism of pore structure refinement. Our primary aim is to find a proper dosage of internal curing materials for such implications. The results will help design an alternative strategy for concrete structure in harsh environment.

2. Experimental program

2.1. Materials

Ordinary Portland cement P.O. 42.5 with specific surface area 374 m$^2$ kg$^{-1}$ were used in this study. Specific gravities of the cement and fly ash was equal to 3.06 and 2.34 respectively. The fine aggregate was local river sand with a fineness modulus of 2.87. Coarse aggregate with a diameter 5–13 mm were used as for samples preparation. Fly ash with a fineness 10.8% and slag had a specific surface area 429 m$^2$ kg$^{-1}$ were added in concrete mixture. Polycarboxylate superplasticizer with water reducing ratio 25%–30% was used to ensure workability. Tap water was adopted in the preparation process.

Figure 2(a) shows the images of the three internal curing materials: superabsorbent polymers (SAP), lightweight aggregate (LWA), and perforated cenospheres (PCs). This test uses 120 mesh super absorbent resin SAP, which has better water absorption rate and water retention capacity. Saturated porous lightweight aggregate (LWA) uses clay ceramsite, and most of its appearance features are round or oval spheres, as shown in figure 2. The particle size of clay ceramsite is between 5 mm and 20 mm. Perforated cenospheres (PCs) are hollow fly ash.
particles that are off-white, with thin and hollow walls, with a particle size of about 0.1 mm and a smooth surface. It is an industrial waste produced in the high temperature and high pressure environment of the coal burning process in thermal power plants. Since the perforated cenospheres particles are hollow spheres, they can be used as internal curing materials for concrete, playing the role of pre-absorption, retention and release of water, so as to achieve efficient use of materials. The appearance of the floating beads before and after absorbing water is shown in figure 2(a). The detail preparation process of perforated cenospheres is shown in figure 2(b).

2.2. Mix proportion and curing

Ten different batches of concrete were prepared in this study, as shown in table 1. The concrete samples, namely cubes with the dimensions 100 mm × 100 mm × 100 mm and the corresponding paste prisms with the dimensions 40 mm × 40 mm × 160 mm were prepared according to the mix proportion described in table 1. The wetted internal curing materials introduced additional water into the concrete, which increased the initial w/c by (w/c)$_{IC}$. The total water/cement ratio ((w/c)$_{total}$) thus changed for each batch of concrete.

A simulated environment test box is used for simulation experiments, as shown in figure 3. Taking Central Asia region as an example, combined with local meteorological data and survey reports, the simulated temperature is determined to be $-5^\circ C$–$40^\circ C$ and the relative humidity is 35% according to the specifications. The daily temperature changes are: the temperature rise stage from 6 to 14 o’clock, the high temperature and
constant temperature stage from 14:00 to 20 o’clock, the cooling stage from 20 o’clock to 1 o’clock, and the low
temperature constant temperature stage from 1 to 6 o’clock.

2.3. Experimental methods
Scanning electron microscope (SEM) was used to observe the microstructure of selected sample. Mercury
intrusion porosimetry (MIP) was used to determine the pore size distribution of mortar samples after exposure
to dry environment. X-ray diffraction (XRD) were used to evaluate the evolution of phase composition in
selected sample.

3. Results and discussion
3.1. Micromorphology by SEM
Figure 4 shows the micro morphology of concrete at 28 days. Figure 3(a) shows the microscopic morphology of
LWA micro-regions. Due to the feature of multiple openings on the surface of the LWA, a large amount of
cement paste aggregates on the surface. During the internal curing process, the internal moisture of the LWA is
released externally, which makes the cement hydration process more fully, and the interface transition zone is
more uniform and dense. This indicates that the LWA improves the transition zone of the concrete interface and
improves the bond between the LWA and the cement paste. In the process of cement hydration, the high
porosity of LWA can fully retain water and release water. The difference in humidity caused by cement hydration
allows the internal moisture of the LWA to be released into the surrounding slurry under the capillary pressure,
so that the cement near the LWA is more fully hydrated [23–25]. However, the nature of high porosity for LWA
may reduce the strength of concrete.

Figure 3. Appearance picture of simulated environment test chamber.

Table 1. Mix proportions of concrete (kg m$^{-3}$).

| Group | Water | Cement | Fly ash | Slag | Sand | Stone | Superplasticizer | Internal curing material | Name | Quantity | (w/c)$_{IC}$ | (w/c)$_{total}$ |
|-------|-------|--------|---------|------|------|-------|------------------|--------------------------|------|-----------|-------------|--------------|
| J0    | 160   | 280    | 60      | 60   | 700  | 1140  | 1.2             | /                         | SAP | 0.4       | 0.01        | 0.41         |
| S0.1  | 160   | 280    | 60      | 60   | 700  | 1140  | 1.2             | SAP                      | 0.4  | 0.01      | 0.41        |
| S0.2  | 160   | 280    | 60      | 60   | 700  | 1140  | 1.2             | SAP                      | 0.4  | 0.02      | 0.42        |
| S0.3  | 160   | 280    | 60      | 60   | 700  | 1140  | 1.2             | SAP                      | 0.4  | 0.03      | 0.43        |
| L10   | 160   | 280    | 60      | 60   | 700  | 1026  | 0.8             | LWA                      | 22   | 0.077     | 0.477       |
| L20   | 160   | 280    | 60      | 60   | 700  | 912   | 0.6             | LWA                      | 44   | 0.154     | 0.534       |
| L30   | 160   | 280    | 60      | 60   | 700  | 798   | 0.4             | LWA                      | 66   | 0.231     | 0.631       |
| P3    | 160   | 280    | 60      | 60   | 700  | 1140  | 1               | PCa                      | 12   | 0.066     | 0.466       |
| P6    | 160   | 280    | 60      | 60   | 700  | 1140  | 0.8             | PCa                      | 24   | 0.132     | 0.532       |
| P9    | 160   | 280    | 60      | 60   | 700  | 1140  | 0.6             | PCa                      | 36   | 0.198     | 0.598       |
The C-S-H gel in SAP concrete has a higher degree of compaction and a lower porosity. The structure of the middle hole is irregular, and there is a layer of film material inside the hole, the structure of the hole wall is fold-like, and the density near the hole is relatively high. Therefore, it is judged that the reason for the formation of the hole is the release of water from the SAP. This also shows that SAP can provide water for the hydration of cement in the concrete after absorbing water.

The hydration products of the PCs concrete are denser, with fewer pores, and there are more ettringite crystals near the pore walls. The ettringite crystals of needle-shaped rods tend to grow in places with less restriction, and the greater the water-cement ratio, the greater the amount of formation. The water-cement ratio near PCs is relatively large, which can promote the mass production of ettringite. Further, the PCs itself is a sphere with an inner hollow shell, which provides enough space for the growth of ettringite, so the ettringite grows densely near the pore wall, filling the holes formed by the PCs after releasing water to a certain extent.

### 3.2. Mineral phases by XRD

It can be seen from the XRD patterns of each group of concrete that the selected samples contain silicon dioxide (SiO$_2$), ettringite (AFt), calcium hydroxide (Ca(OH)$_2$), calcium carbonate (CaCO$_3$), and gypsum (CaSO$_4$) and unhydrated cement clinker (C$_3$S and C$_2$S).

The overall observation of the XRD patterns of each group of concrete shows that the SiO$_2$ diffraction peaks of each group of concrete are the highest. This is because the concrete contains a large amount of sand. It is inevitable that there will be different amounts of sand when selecting concrete samples to grind the powder. The composition is SiO$_2$, which leads to the highest SiO$_2$ peak value, so the change rule of SiO$_2$ peak value is not considered. The diffraction peak intensities of ettringite (AFt), gypsum (CaSO$_4$) and calcium carbonate (CaCO$_3$) of each group of concrete powder are not significantly different, while calcium hydroxide (Ca(OH)$_2$) and unhydrated cement clinker (C$_3$S and C$_2$S) diffraction peak intensity has obvious changes, which indicates that Ca(OH)$_2$ and CnS are significantly different in each group of concrete.

It can be seen from figure 5 that compared with the reference group, the concrete with internal curing has enhanced diffraction peak intensity in Ca(OH)$_2$ (characteristic peaks at 18.1°, 34.0° and 47.2°), and the intensity of the diffraction peaks in CnS (characteristic peaks at 29.4°, 32.2° and 34.5°) decreased. It shows that at the age of 28, under dry and large temperature differences, the concrete without internal curing materials gradually

![Figure 4. Microstructure of internal curing concrete. (a) LWA, (b) PCs, (c) SAP, (d) PCs.](image-url)
stops hydrating because of the lack of additional moisture, while the concrete with internal curing materials releases its internal pre-absorbed by the internal curing materials. Moisture allows the cementitious material to continue the hydration reaction, and the addition of internal curing materials has a certain promotion effect on the degree of cement hydration and the secondary hydration of mineral admixtures.

In figure 5(a), comparing the results of different SAP content, the calcium hydroxide diffraction peak intensity in S0.2 group is the strongest, and the CnS diffraction peak intensity of unhydrated cement clinker is the weakest, indicating this group is more thoroughly hydrated. In figure 5(b), it is obvious that compared with the benchmark group, the addition of pre-absorbed LWA has a significant effect on the hydration degree of concrete. Comparing the results of different amounts of LWA, the diffraction peak intensity of calcium hydroxide in the L10 group is significantly stronger than that of the other groups, and the CnS diffraction peak intensity of the unhydrated cement clinker is the weakest, indicating that this group has the most complete hydration. In figure 5(c), comparing the results of different PCs content, the P3 group has the strongest calcium hydroxide diffraction peak intensity, indicating that this group has the most complete hydration.

3.3. Pore structure by MIP

This section illuminates the influence of internal curing materials and admixtures on concrete in a Nano-scale via MIP test. The pore structure results of the mortar in each group of concrete at the age of 28 days are shown in table 2.

The porosity of concrete refers to the percentage of the pore volume in the concrete material to the total volume of the concrete in its natural state [27]. The porosity is closely related to the compactness of the concrete structure. It can be seen from the table that in a dry environment with a large temperature difference, the porosity of the SAP group is not significantly different from that of the reference group. The porosity of the LWA group increased with the increase of the dosage. Compared with the J0 group, the porosity of the L10 group, the
The porosity of the PCs group increased with the increase of the dosage, compared with the J0 group, the porosity of the P3, P6, and P9 concrete increased by 29%, 42%, 62%, respectively.

The differential curve of concrete pore size distribution has a peak point. The pore size corresponding to this point is called the maximum aperture. Its physical meaning is the pore with the largest change rate of pore volume with pore size. It can be considered that the most probable pore size represents the most probable pore size distribution.

![Evolution of pore size in selected samples at 28 d curing: (a) SAP; (b) LWA; (c) PCs.](image)

**Table 2.** Characteristic parameters of pore structure in concrete under dry environment with large temperature difference.

| Code | Porosity(%) | Maximum aperture(nm) | <20nm | 20~50nm | 50~200nm | >200nm |
|------|-------------|-----------------------|-------|---------|----------|--------|
| J0   | 14.16       | 120.87                | 9.19  | 11.40   | 37.16    | 42.25  |
| S0.1 | 14.13       | 151.09                | 12.86 | 10.49   | 43.07    | 33.58  |
| S0.2 | 14.07       | 120.86                | 5.81  | 12.66   | 56.43    | 25.10  |
| S0.3 | 14.15       | 120.82                | 10.20 | 14.19   | 59.77    | 15.84  |
| L10  | 14.30       | 120.88                | 4.05  | 11.39   | 64.01    | 20.55  |
| L20  | 17.30       | 151.13                | 6.52  | 9.59    | 44.28    | 39.61  |
| L30  | 20.77       | 226.82                | 8.73  | 8.25    | 31.28    | 51.74  |
| P3   | 18.24       | 151.14                | 6.93  | 9.68    | 37.74    | 43.65  |
| P6   | 20.16       | 183.21                | 6.24  | 12.07   | 42.23    | 39.46  |
| P9   | 22.89       | 227.09                | 7.51  | 12.65   | 32.42    | 47.42  |

L20 group, and the L30 group increased by 1%, 22%, and 47%, respectively. The porosity of the PCs group increased with the increase of the dosage, compared with the J0 group, the porosity of the P3, P6, and P9 concrete increased by 29%, 42%, 62%, respectively.

The differential curve of concrete pore size distribution has a peak point. The pore size corresponding to this point is called the maximum aperture. Its physical meaning is the pore with the largest change rate of pore volume with pore size. It can be considered that the most probable pore size represents the most probable pore size distribution.
size in concrete. The developed pore size range should be regarded as an important characteristic parameter of
the concrete pore structure.

As one of the significant indexes of concrete pore structure, porosity is closely related to concrete
performance. However, as a highly heterogeneous body, the internal pore structure of concrete is very
complicated, and the porosity is not enough to accurately characterize the internal pore structure of concrete.
Therefore, it is necessary to classify the pores of concrete with different pore diameters. We divide the pores are
into Four types of harmless pores \((d \leq 20 \, \text{nm})\), less harmful pores \((20 \, \text{nm} < d \leq 50 \, \text{nm})\), harmful holes
\((50 \, \text{nm} \leq d < 200 \, \text{nm})\), and harmful holes \((d \geq 200 \, \text{nm})\).

Figure 6 shows the changing trend of the pore size range of internal curing concrete. It can be seen that the
harmful pores decrease with the increase of SAP content. Compared with the J0 group, the harmful pores of the
S0.1, S0.2 and S0.3 concrete were reduced by 21%, 41%, and 63%, respectively. The degree of refinement is the
highest. A larger pore volume in the pore diameter above 200 nm (harmful pores) is observed for the samples
with more LWA content because the high porosity nature for LWA. As shown in figure 6(b), sample L10 has the
highest degree of aperture refinement, with a peak of 64% at pore diameter range of 50–200 nm. It can be seen
from figure 6(c) that the proportion of harmful pores in the PCs group is not significantly different from that of
the reference group. Therefore, in a dry environment with a large temperature difference, the PCs group does
not contribute to the refinement of concrete.

4. Conclusions

By experimentally determining the evolution of the phase composition and pore structure characteristics of
hydration products via SEM, XRD and MIP tests, our results elucidate the mechanism for the effect of different
amounts of SAP, LWA and PCs on the microstructure of concrete in a dry environment with large temperature
differences. The main conclusions are as follows:

(1) Internal curing can greatly promote the hydration of cement by slowly releasing water. The pores formed
after the internal curing material releases water can provide space for hydration products, thereby
accelerating the formation of ettringite crystals.

(2) The XRD results show that the calcium hydroxide of internal curing concrete increases significantly, and the
unhydrated cement clinker decreases significantly. Ordinary high-performance concrete gradually stops
hydration after 28 due to lack of additional water, while internal curing concrete can release water
continuously. Consequently, the interior of the concrete can continue to be hydrated. The addition of
internal curing materials greatly promotes the degree of cement hydration.

(3) The MIP results show that the pore structure of the S0.3 concrete and the L10 concrete has the most obvious
degree of refinement, and the concrete is more uniform and dense. The porosity, most probable pore size
and harmful pores of the PCs group have increased significantly, and the concrete density is relatively low.

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Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

Declaration of competing interest

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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