Influence of Coarse Aggregate Concentration on the Frost Resistance of Non-Air-Entrained Concrete

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Aggregate-interlocking concrete is a kind of low-carbon and high-performance concrete, while the aggregate-interlocking concrete prepared by the distributing-filling coarse aggregate (DFCA) process worsens frost resistance than ordinary concrete. In order to better apply the aggregate-interlocking concrete, it is necessary to figure out the relationship between the frost resistance and the coarse aggregate concentration. In the study, three series of concretes, C30, C40, and C50, are designed, and the charge passed, scaling mass, water uptake, and penetration depth of NaCl solution are tested as indicators to account for the relationship between frost resistance and coarse aggregate concentration. The results indicate that aggregate still has a strong restraint effect on the permeability of concrete after freezing-thawing cycling, but compared to the unfrozen concrete, the charge passed of the concrete with higher aggregate concentration tends to increase more intensively. The scaling happens at both bulk mortar and interfaces between aggregate and mortar. The increase in aggregate concentration could restrain the scaling of mortar, but excessive aggregate concentration results in a significant increase in scaling mass. The concrete with higher coarse aggregate concentration could absorb more water during the freezing-thawing cycling, and the penetration depth of NaCl solution also increases with the increase of coarse aggregate concentration. The results reveal that the increase in aggregate concentration degrades the frost resistance of concrete, and the DFCA process could be applied in high-strength concrete to mitigate the negative effect of coarse aggregate on the frost resistance of concrete.

1. Introduction

Ordinary aggregate, such as crushed limestone, basalt, and pebbles, generally has higher strength and elastic modulus and better durability than cement paste, and the positive effects of aggregate on the properties of concrete have been widely confirmed. Stock et al. [1] claimed that the strength of concrete increased with the increase of aggregate content when the aggregate volume fraction is larger than 40%. de Larrard [2] revealed that compressive strength presented a good correlation with maximum paste thickness (MPT), and the smaller MPT, the higher the compressive strength. A number of classic models have been proposed for elastic modulus and could well illustrate the essential skeleton effect of aggregate [3]. Besides, aggregate presents dilution and tortuosity effects on permeability [4, 5] and restraint effect on shrinkage [6, 7] of concrete. Hence, aggregate-interlocking concrete tends to present better performances than conventional concrete. Besides, due to the significant reduction of cement paste, aggregate-interlocking concrete is also a promising type of low-carbon concrete.

The distributing-filling coarse aggregate (DFCA) process is proposed by Shen [8] to prepare aggregate-interlocking concrete. DFCA concrete consists of the reference concrete and DFCA (additional coarse aggregate), and the reference concrete is designed and prepared with the conventional methods. During the DFCA process, DFCA is added into the mold with the casting of fresh reference concrete. Then the compacted concrete owns a higher aggregate concentration than the reference concrete. Based on the previous studies, the DFCA process significantly improves the compressive strength and elastic modulus and minimizes the chloride ion penetration and drying shrinkage. With the DFCA ratio of 20%, the reference concretes with C30–C80 strength grades are improved by 20% in compressive strength [9]. Self-compacting concrete generally has high paste content, which
would lead to significant drying shrinkage. Using the DFCA process, 20% DFCA make the drying shrinkage decrease by about 32%, and meanwhile, the penetration resistance is also improved by about 35% [10]. The benefits of the DFCA process also are applied to prepare high-performance recycled aggregate concrete [11], and the DFCA process has been successfully applied to the pavement and pipe piles [12, 13]. However, in a previous study [14], it was found that the DFCA concrete with 20% DFCA had worse frost resistance than the reference concrete. The DFCA treated by microfines could further improve the compressive strength of concrete, which implied that the ITZs around DFCA were enhanced, but it still led to the degradation of frost resistance.

Most of the investigations concentrated on studying the influence of inherent properties of aggregate and ITZs induced by aggregate on frost resistance of concrete. For instance, because the residual mortar on recycled aggregate results in the double ITZs in recycled aggregate concrete [15], recycled aggregate concrete generally has inferior frost resistance to natural aggregate concrete [16, 17]. Due to the high water absorption of coal gangue aggregate, the replacement of natural aggregate with coal gangue aggregate led to the obvious reduction of frost resistance of concrete [18]. Pang et al. [19] compared the different effects of natural aggregate (NA), steel slag aggregate (SSA), and carbonated steel slag aggregate (CSA). The concrete prepared with CSA had wider ITZs than that with NA but had better frost resistance. The reason would be that the initial cracks on the edge of CSA could remit the stress concentration induced by ice formation nearby the aggregate surface. ITZs are prone to fracture under freezing condition, and Pan et al. [20] claimed that ITZ debonding was also related to the differences between frost expansion and thaw contraction in sandstone and cement paste. As for the influence of aggregate concentration, Zarauskas et al. [21] found that the increase of aggregate had a negative effect on the frost resistance of concrete, but the mechanism was not deeply discussed, and more relevant information was rarely reviewed in other literature.

Frost resistance may be a limiting factor in the application of aggregate-interlocking concrete. In comparison to ITZs, the mesostructure may be a more important factor in the frost resistance of concrete. The aim of this study is to find out the relationship between frost resistance of concrete and coarse aggregate concentration and sufficiently illustrate the inherent mechanism from a mesostructure point of view. In this paper, three series of concretes with strength grades of C30, C40, and C50 are designed, and the coarse aggregate concentration varies from 0% to about 50%. Using the change of charge passed, scaling mass, water uptake, and penetration depth of NaCl solution as indicators, the effect of aggregate concentration on frost resistance is further illustrated.

2. Materials and Methods

2.1. Materials. Cementitious materials included PO 42.5 ordinary Portland cement complying with GB175-2007 [22] and class II fly ash. River sand with an apparent density of 2,610 kg/m$^3$ and crushed limestone with an apparent density of 2,700 kg/m$^3$ were used as fine aggregate and coarse aggregate, respectively. The coarse aggregate had water absorption of about 1% by mass and a crushing value of 12.7%. The coarse aggregate was divided into three gradations by sieve: 5–10 mm, 10–20 mm, and 20–25 mm, denoted as CA1#, CA2#, and CA3#, respectively. The coarse aggregate with the size of 5–20 mm is generally used to prepare conventional concretes, especially the concretes with high fluidity, and the mixed aggregate of 30% CA1# and 70% CA2# had maximum packing density and was used to prepare the reference concretes in this study. CA3# was used as DFCA, which had a smaller specific surface area and was conducive to the compaction of DFCA concrete. The gradation curves of fine and coarse aggregate are shown in Figure 1.

2.2. Mix Proportions and Specimen Preparation. Three reference concretes with strength grades of C30, C40, and C50, noted as Ref30, Ref40, and Ref50, were first designed. Table 1 lists the mix proportions of the reference concretes. Based on the mix proportions of the reference concretes, only the coarse aggregate was varied to obtain three series of concrete mixtures with different coarse aggregate concentrations. All of the designed concrete mixtures and their volume fractions of coarse aggregate are listed in Table 2, and C303, C403, and C503 correspond to the reference concretes of Ref30, Ref40, and Ref50, respectively. The reference concretes and the concrete with smaller volume fractions of coarse aggregate than the reference concretes were prepared by the conventional mixing process, during which the raw materials were mixed in succession. The concretes with larger coarse aggregate concentration than the reference concretes were prepared by the DFCA process during the DFCA process,
after the fresh reference concretes were obtained, the weighed DFCA was added into the mold three times along with the casting of fresh concrete. The different DFCA ratios (replacement ratio of DFCA to the reference concrete mixture by volume) were set for each series of concrete mixtures, and based on the previous studies, the maximum volume fraction of coarse aggregate in DFCA concretes should not exceed 50% to guarantee that the fresh concrete could be well compacted in the laboratory. Because DFCA had a larger size than the coarse aggregate in the reference concretes and the concrete with smaller volume fractions of coarse aggregate than the reference concrete, the gradations of the coarse aggregate in every concrete mixture are also stated in Table 2.

2.3. Test Methods

2.3.1. Charge Passed. Two cylindrical specimens with 50 ± 2 mm thickness and 100 mm diameter for each mix proportion were prepared for the rapid chloride penetrability test (RCPT) according to GB/T 50082-2009 [23]. The specimens were saturated by water under vacuum for 18 h; then the specimens were removed from the water; and their lateral surfaces were coated with silica gel. After that, the specimens were mounted on the voltage cell with one side immersed in a sodium chloride solution (3 wt% NaCl) and the other side in a sodium hydroxide solution (0.3 mol/L NaOH). The total charge (in Coulombs) that passes through the specimen for 6 hours under 60 V voltage was used to reflect the permeability of concrete.

2.3.2. Frost Resistance. Theoretically, the cement paste within concrete becomes more and more porous with the freezing-thawing cycling. In addition, under the freezing-thawing environment, the concrete will be scaled and absorb water from the surroundings. Hence, the change of charge passed after the rapid freezing-thawing test and the scaling mass and water uptake after the salt freezing test were used to illustrate the damage degree of concretes with different coarse aggregate concentrations. The used freezing-thawing tests were conducted with reference to GB/T 50082-2009 [23].

(1) Change of Charge Passed after Rapid Freezing and Thawing. The cylindrical specimens with 50 ± 2 mm thickness and 100 mm diameter were first tested to obtain the initial charge passed. Then the specimens were immersed in the water in the rubber bucket. The temperature in the rapid freezing-thawing testing machine ranges from −18 ± 2°C to 5 ± 2°C, and each freezing-thawing (F-T) cycle lasted about 3 h. After a number of F-T cycles, the charge passed of the specimens was tested, and the relative change to the initial charge passed was regarded as one of the indicators of frost resistance.

| Table 1: Mix proportions of the reference concretes (kg/m³). |
|-------------------------------|---------------------|-----------------|----------------|-----------------|----------------|
| Notation | Cement | Fly ash | Coarse aggregate | Sand | Water | Superplasticizer |
| Ref30 | 400 | — | 1,207 | 650 | 172 | — |
| Ref40 | 300 | 129 | 1,120 | 716 | 161 | 4.3 |
| Ref50 | 448 | 124 | 945 | 724 | 179 | 7.5 |

| Table 2: Mix proportions of concrete with different coarse aggregate concentrations. |
|-------------------------------|---------------------|-----------------|-----------------|-----------------|
| Notation | DFCA ratio (%) | Volume fraction of coarse aggregate (%) | CA1#:CA2#:CA3# |
| C301 | — | — | — |
| C302 | — | 20.0 | 30%:70%:0% |
| C303 (Ref30) | — | 44.7 | 30%:70%:0% |
| C304 | 8 | 49.1 | 25.1%:58.6%:16.3% |
| C401 | — | — | — |
| C402 | — | 20.0 | 30%:70%:0% |
| C403 (Ref40) | — | 41.5 | 30%:70%:0% |
| C404 | 10 | 47.4 | 23.8%:55.4%:20.8% |
| C405 | 15 | 50.3 | 20.6%:48.1%:31.3% |
| C501 | — | — | — |
| C502 | — | 20.0 | 30%:70%:0% |
| C503 (Ref50) | — | 35.0 | 30%:70%:0% |
| C504 | 10 | 41.5 | 22.8%:53.1%:24.1% |
| C505 | 20 | 48.0 | 17.5%:40.8%:41.7% |

![Figure 2: F-T cycling scheme of salt freezing test.](image-url)
Scaling and Water Uptake after Salt Freezing Test. The prism specimens of 100 mm × 100 mm × 400 mm were sawed into 150 mm length and 70 mm thickness. Two specimens were prepared and tested for each mixture. The side surfaces of obtained specimens were sealed with aluminum foil; then the specimens were put in the steel box; and only the sawed cross-section contacted with the 3 wt% NaCl solution. After presaturated for 7 days, the specimens were frozen and thawed with the scheme as shown in Figure 2. The scaling after every 4 F-T cycles was collected with the filter paper and then dried at the temperature of 105°C for 24 h. The mass of water uptake by specimen was calculated as follows:

$$m_{ui} = m_i + m_s - m_0,$$

where $m_{ui}$ is the mass of water uptake, g; $m_i$ is the mass of specimen after i F-T cycles, g; $m_s$ is the mass of scaling, g; and $m_0$ is the mass of specimen after presaturated, g.

Since the coarse aggregate has good frost resistance and the designed mixtures have different aggregate concentrations, the scaling mass ($m_s$) and water uptake ($m_{ui}$) would mislead the results on the frost resistance of different concrete mixtures. Hence, the area of mortar on the frozen surface of the specimen was measured with the image process method, and the scaling mass per mortar area and water uptake per mortar area were better for evaluating the damage degree within the specimen. Besides, when the freezing and thawing finished, the specimens were split, and the penetration depth of NaCl solution was determined by spraying 0.1 mol/L AgNO₃ solution on the cross-section.

2.3.3. Pore Characteristics. Volumetric water absorption was determined to account for the open porosity of concrete, and the water absorption test was conducted in accordance with GB-T 50081-2019 [24]. The cube specimen of 100 mm × 100 mm × 100 mm was used and was dried at 105°C for 24 h first; then they were saturated with water under vacuum. The volumetric water absorption of the specimen was calculated in accordance with the following equation:

$$\omega_w = \frac{m_w - m_0}{\rho_w V_c} \times 100,$$

where $\omega_w$, $m_0$, $m_w$, and $\rho_w$ are the water absorption in volume (%), the oven-dried weight (g), the water-saturated weight (g), and the density of water (g/cm³), respectively, and $V_c$ is the volume of the concrete specimen (cm³).

Pore size distribution was measured by a mercury intrusion porosimeter (AutoPore IV 9500). The mortar samples with a diameter of about 5 mm were taken from the inside of the concrete. They were immersed in ethanol to clear the fine particles on the surface and then dried at 45°C for 24 h.
2.3.4. Image Processing. Two-dimensional image processing is widely used to quantitatively characterize the mesostructure of concrete [25, 26]. It was used to measure the aggregate concentration and mortar thickness between two adjacent aggregate particles on the frozen surface of concrete specimens. Figure 3 shows the schematic diagram of the image processing method, and the detailed procedures are concluded as follows: (1) recognition of aggregate: due to the varied colors of aggregate, the aggregate was highlighted manually using the cutout tool in Photoshop software. (2) Binarization of images: the highlighted aggregate and mortar were colored with black and white, respectively, as shown in Figure 3(b). The area of mortar and perimeter of total aggregate could be measured by Image-Pro Plus software in this step. (3) Delaunay triangulation of the binary images: centroid function located the centroid coordinates of each coarse aggregate, and as shown in Figure 3(c), the centroids of the neighboring coarse aggregates were connected to each other with the Delaunay triangulation algorithm. After removing the coarse aggregates, the remaining line segments as shown in Figure 3(d) were the mortar thickness between coarse aggregates, and they were measured by Image-Pro Plus software.
3. Results and Discussion

3.1. Charge Passed. Figure 4 shows the charge passed of concrete specimens after different F-T cycles. For the concrete without F-T cycles, the increase of aggregate concentration is effective to improve the impermeability of concrete. When the volume fraction of coarse aggregate is increased from 0% to 20%, the charge passed decreases by 54.1%, 20.0%, and 22.2% for C30, C40, and C50, respectively. This is attributed to the dilution and tortuosity effects of aggregate on the permeability of concrete. The increase in aggregate concentration reduces the volume fraction of cement paste and makes the transport path more tortuous. Although subjected to the F-T environment, the concrete with high aggregate concentration could maintain a lower charge passed.

In order to better show the influence of freezing and thawing on the permeability of concrete, the relative change of charge passed with reference to the unfrozen concrete is calculated. As shown in Table 3, after a number of F-T cycles, the relative changes of charge passed present increase trends with the increase of aggregate concentration, while the relative changes of charge passed appear negative values for the concretes with low aggregate concentrations, which is also found in the study conducted by Wang et al. [27]. After more F-T cycles, the charge passed of the concretes increases in varying degrees, and the concretes with high aggregate concentration tend to have a larger relative change of charge passed, which is more obvious for C30 and C40 concretes. This indicates that the cement paste in concrete with higher aggregate concentration becomes more porous after F-T cycling.

3.2. Scaling Mass. The scaling mass of concrete at 28 d curing age with F-T cycles is shown in Figure 5. It can be seen that the scaling mass is nearly linear to the F-T cycle, while the concretes with different strength grades have significant differences in scaling rate. For C30 concretes, C301 has the lowest scaling rate with F-T cycles. After 12 F-T cycles, the scaling mass of C301 is 2.29 kg/m², while C302, C303, and C304 have the scaling mass of 4.28 kg/m², 4.31 kg/m², and 3.47 kg/m², respectively, which are much higher than that of C301. With the increase of concrete strength grade, the scaling mass is significantly minimized, but the scaling mass with aggregate concentrations of C40 and C50 concretes is not similar to that of C30 concrete. It can also be seen that C401 and C501 have moderate scaling mass compared to the concretes with the same strength grade, and the scaling mass of the other concretes distributes on both sides of the fitted
line of C401 and C501. Figure 6 shows the scaling mass of C40 and C50 concretes after being cured for 90 d. The sequence of scaling rate of C40 concretes with aggregate concentration has no significant change after the longer curing age. As for C50 concretes, the scaling resistance of C501 and C504 is enhanced more significantly than that of other concrete mixtures.

The relationship between the slopes (scaling rate) of fitted lines in Figures 5 and 6 and the volume fraction of coarse aggregate are plotted in Figure 7. The strength grade and curing age of concrete are two critical factors in the scaling of concrete, while aggregate also significantly affects the frost resistance of concrete. The increase in aggregate concentration is obviously harmful to the frost resistance of C30 concrete. But, for C40 and C50 concretes, the low volume fraction of coarse aggregate only slightly affects the scaling of concrete. With the increase of volume fraction of coarse aggregate, the scaling rate presents decrease trends and then increases drastically when the volume fraction of coarse aggregate is larger than 42%, especially for C40 concrete. Generally, the increase in aggregate concentration has a more deleterious effect on the scaling resistance of lower-strength concrete, and excessive aggregate concentration would also aggravate the scaling phenomenon.

3.3. Water Uptake. The cracks generate from the surface and spread to the inside of concrete during F-T cycling, which increases the porosity and makes the specimens absorb water [28]. The water uptake of concrete at 28 d and 90 d curing age is plotted in Figures 8 and 9, respectively. It can be seen that the water uptake instantly increases after 4 F-T cycles; then the increase of water uptake becomes steady with the F-T cycle. C30 has been severely damaged after 12 F-T cycles. At the 12th F-T cycle, C301 has a mass of absorbed water of 1.3 kg/m². The water uptake increases with the increase of aggregate concentration, and C303 has the highest mass of absorbed water, which is almost 2 times that of C301. Compared to C30 concretes, C40 and C50 concretes have a relatively lower mass of absorbed water, and their water uptakes present a strong correlation with the aggregate concentration whether the curing age is 28 d or 90 d. Water uptake...
uptake accounts for the cracking rate of concrete, so the results indicate that the increase in volume fraction of coarse aggregate would accelerate the frost damage. After the F-T test, the penetration depth of NaCl solution into the specimen is determined by spraying AgNO₃ solution on the cross-section. As shown in Figure 10, the penetration depth of NaCl solution obviously increases as the aggregate concentration increases for C30 and C40 concretes. The C50 concretes with different aggregate concentrations have a close penetration depth of NaCl solution ranging from 3.5 mm to 4.0 mm, and only a slight increase is seen with the increase of coarse aggregate concentration. This indicates that lower w/c could weaken the impact of aggregate concentration on frost resistance of concrete. Although the water uptake of C505 is about 3 times that of C501, the cracking is condensed within a relatively narrow region.

3.4. Discussion. All of the experimental results of charge passed, scaling mass, water uptake, and penetration depth of NaCl solution generally illustrate that the concretes with high coarse aggregate concentration have poor frost resistance. The variation of aggregate concentration may result in the different content of entrapped air in cement paste and
affects the distribution of aggregate in concrete, so the formation mechanism of frost resistance is analyzed from two aspects: pore characteristics in mortar and mesostructure of concrete.

Volumetric water absorption reflects the open porosity of concrete. The volumetric water absorption against aggregate concentration is plotted and regressed as shown in Figure 11. It can be seen that higher water to binder (w/b) ratio results in higher open porosity, and the water absorption decreases with the increase of coarse aggregate concentration. Good linear relationships exist between water absorption and aggregate concentration, which indicates that the decrease in water absorption mainly depends on the "dilution effect" of aggregate. The frost resistance of concrete is also related to the pore distribution within the cement paste. The pores in cement paste could be classified as more harmful pore (>200 nm), harmful pore (100–200 nm), less harmful pore (20–100 nm), and harmless pore (<20 nm) [29]. The MIP results of C301, C401, and C403 are shown in Figure 12. From Figure 12(a), the critical pore size of C301 is 76.97 nm, while C401 has much lower critical pore diameters, which account for the better frost resistance of concrete with lower w/b. The threshold diameters of pores in C401 and C403 are about 40 nm and 100 nm, respectively.

Figure 12: Pore characteristics: (a) pore size distribution and (b) percentage of different pores.

Table 4: Scaling of concretes with different strength grades after 6 F-T cycles.

| Position | C30 | C40 | C50 |
|----------|-----|-----|-----|
| Mortar   | ![Mortar_C30](image) | ![Mortar_C40](image) | ![Mortar_C50](image) |
| Interface| ![Interface_C30](image) | ![Interface_C40](image) | ![Interface_C50](image) |
However, from Figure 12(b), the percentage of pores with the size of 200–10,000 nm in C401 is larger than that in C403. Compared to C401, C403 has less harmless pores, but it also has less more harmful pores. Hence, it is limited to interpret the significant differences in frost resistance induced by the variable aggregate concentrations.

The surfaces of concretes after 6 F-T cycles are observed by a microscope at 80 times magnification. As shown in Table 4, the scaling of concretes is mitigated with the increase of concrete strength grade, and C50 concrete shows no obvious cracking. In terms of C30 and C40 concretes, the scaling happens in both bulk mortar and the interfaces between aggregate and mortar simultaneously, and the aggregate on the specimen surface still maintains the intact surface, so the degradation of frost resistance with an increase of aggregate concentration may be greatly related to the mesostructure and the amount of interface. A schematic diagram as shown in Figure 13 is conceived to distinctly describe the interactions among aggregate, mortar, and pores containing water. The formation of ice in pores first generates high pressure exerting on the wall of pores, which results in the cracking and expansion of cement paste [30]. Coarse aggregate generally has a higher elastic modulus than mortar. As a result, the expansion of mortar is restrained by aggregate so that the stress concentration would happen at the interfaces between coarse aggregate and mortar [31]. Moreover, as shown in Figure 14, the increase in aggregate concentration results in the reduction of the mortar thickness and the increase of the interface length. The thinner mortar thickness would intensify the stress concentration around aggregate, and due to the low porosity of aggregate, more interfaces are not conducive to dissipating the concentrated stress [3]. In addition, the ITZs in concrete generally have a higher porosity than the bulk cement paste, which implies that the interfaces between aggregate and mortar will be easily attacked by the ice formation [32]. In summary, the increase in aggregate concentration increases the risk of frost damage of concrete.

4. Conclusions

The influence of aggregate concentration on the frost resistance of concrete is investigated in the study. Three series
of concretes, C30, C40, and C50, are designed, and the charge passed, scaling mass, water uptake, and penetration depth of NaCl solution as indicators are determined to illustrate the relationship between frost resistance and coarse aggregate concentration.

The increase of aggregate concentration is beneficial to reducing water absorption and charge passed by concrete. After a number of $F$-$T$ cycles, the charge passed still presents a decrease trend with the increase of aggregate concentration, but compared to the unfrozen concrete, higher aggregate concentration results in a higher increase of charge passed. The scaling happens both in bulk mortar and interfaces between aggregate and mortar in concrete. The increase in aggregate concentration could restrain the scaling of mortar, but excessive aggregate concentration would aggravate the scaling phenomenon. The concrete with higher coarse aggregate concentration can absorb more water during the $F$-$T$ cycling, and the penetration depth of NaCl solution also increases with the increase of volume fraction of coarse aggregate. All of the test results indicate that the increase in aggregate concentration has a deleterious effect on the frost resistance of concrete.

Aggregate-interlocking concrete sufficiently utilizes the skeleton effect of coarse aggregate and has high strength and elastic modulus, good penetration resistance, and volumetric stability. It is a promising low-cement concrete, but the frost resistance may be the shortcoming that limits the wide application of aggregate-interlocking concrete. Based on the experimental results, the DFCA process is preferable to preparing high-strength aggregate-interlocking concrete to mitigate the deleterious effect of aggregate on its frost resistance. Besides, using an air-entraining agent is an essential approach to improving the frost resistance of concrete, while the results obtained by the present study may not be appropriate for the air-entrained concrete, and the combined effect of coarse aggregate concentration and air-entraining agent should be further studied.

Data Availability

The data used to support the findings of this study can be obtained from the corresponding author upon reasonable request.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

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