Rheological and Mechanical Properties of High-Performance Fiber-Reinforced Cement Composites with a Low Water–Cement Ratio

Baojun Cheng, Xiaowei Gu,* Yuxin Gao, Pengfei Ma, Wen Yang, and Jing Wu

ABSTRACT: High-performance fiber-reinforced cement composites (HPFRCCs) have been widely used in structural engineering due to their excellent performance. With the trend of lightweight construction, these materials, which can be used in prefabricated components, are becoming more and more important. This study investigated the influence of the water–cement (w/c) ratio, within the 0.19–0.28 range, on the rheological and mechanical properties of HPFRCCs; the pore structure and microstructure were observed to evaluate its effect. An elastic modulus test showed that a smaller w/c ratio would result in a higher rigidity of the material. Both the yield shear stress and plastic viscosity decreased to significantly different degrees with an increasing w/c ratio; a decrease in the yield shear stress and plastic viscosity was conducive to air discharge from the composite and, hence, reduced the air content. Most of the internal pores had a diameter of 20–100 nm or larger than 200 nm, while the proportion of those with a diameter of 100–200 nm was relatively low. When the w/c ratio was below 0.22, the flexural and compressive strengths barely increased due to an increment in the number of larger pores (i.e., diameter >200 nm). The results showed that the yield shear stress, plastic viscosity, pore uniformity, and the number of pores with a diameter above 200 nm are the dominant factors affecting the HPFRCC performance at a low w/c ratio.

1. INTRODUCTION

To avoid concrete brittle cracking, high-performance fiber-reinforced cement composites (HPFRCCs) are widely used in structures because of their excellent mechanical properties, self-compacting capacity, and good crack resistance. They can effectively prevent cracking and improve the strength and durability of structures; when the fiber content reaches 2%, the tensile, impact, and bending strengths of HPFRCCs can be increased by 1, 10–20, and 1–2 times, respectively. With the recent increasing demand for good mechanical properties and durability of engineering structures, the application of high-performance concrete is gradually rising. The use of HPFRCCs in earthquake-resistant structures provides higher shear strength, better deformable capacity, and damage tolerance. Unlike normal concrete, the HPFRCC has no coarse aggregates, and the water–cement (w/c) ratio is reduced to improve the density. Furthermore, the tensile, flexural, and compressive strengths and the impact resistance of HPFRCCs are improved by adding toughening fibers, enhancing their overall permeability, frost resistance, and carbonation resistance. However, their w/c ratio is too low to obtain appropriate plastic viscosity, which is not conducive to construction applications and promotes the shrinkage of concrete structures.

In general, besides the interfacial transition zone, porosity is another important factor affecting the concrete strength. In the case of high-porosity concrete, water, oxygen, CO₂, corrosive media, and other harmful substances can enter the material through the pores, which is the main cause of concrete structure deterioration. Porosity and the pore size, morphology, and distribution are collectively referred to as the pore structure of concrete. Some studies have shown that the pore structure determines the mechanical properties of concrete and its non-mechanical properties associated with durability, such as frost resistance, impermeability, and carbonation resistance. The internal pores of the concrete are divided into four levels according to their size: <4.5, 4.5–50, 50–100, and >100 nm. Based on the research on the fractional porosity of each pore level conducted by Wu and Lian, the pores having a size of <20, 20–100, 100–200, and >200 nm can be defined as harmless, slightly harmful, harmful, and very harmful, respectively.

With the recent rise of labor costs, the demand for prefabricated buildings is increasing; accordingly, the system-
atic research about HPFRCCs, which can be used in prefabricated components, is becoming more and more important. To obtain a material with high strength, durability, and crack resistance, the present study focused on manufacturing HPFRCCs with a relatively low w/c ratio (0.19–0.28). The rheological and mechanical properties of the new mixture were tested. Its characteristic pore structure parameters, such as pore morphology, porosity, and pore size distribution, were characterized via scanning electron microscopy16 and mercury intrusion porosimetry (MIP); to assess the influence of the microscopic pore structure on the mechanical and rheological properties, the average pore size and uniformity were also analyzed through water absorption dynamics measurements. Compared with other studies, the systematic research on HPFRCC materials presented in this paper provides a more practical guide for the commercial development of building components having high strength, durability, and crack resistance.

2. MATERIALS AND METHODS

2.1. Materials. The cement used in this study was type I ordinary Portland cement (OPC); semi-encrypted silica fume (SF) and class I fly ash (FA) served as supplementary cementitious materials. Table S1 summarizes the chemical compositions of these raw materials, while the physical and mechanical properties of OPC alone are listed in Table S2. According to an X-ray diffractometry analysis (Figure 1), the main phases in OPC were C3S, C2S, and CaSO3, while those in FA were SiO2 and gismondine. The strength of HPFRCCs is sensitive to the aggregate amount and size;17 here, quartz powder with a fineness modulus 200 mesh was selected as the fine aggregate. The water-reducing agent was a homemade polycarboxylic acid (PC) superplasticizer with a solid content of 50%. Tap water was also added to the mixture. The defoamer used in this study was from Sant’ro. Because the material properties of the PVA fiber influence the HPFRCC performance,18 they are summarized in Table S3.

2.2. Mixture Proportions. Based on packing density theory and preliminary rheological and mechanical tests, different HPFRCC mixtures having a w/c ratio ranging from 0.19 to 0.28 were prepared (Table S4).

2.3. Methods. 2.3.1. HPFRCC Preparation. A double-horizontal shaft mixer with a capacity of 110 L was used to prepare the HPFRCC specimens as follows: The dry components (i.e., OPC, FA, SF, and quartz powder) were mixed for 3 min at a low speed; then, water and the PC superplasticizer were added and mixed for 2 min at a low speed, followed by the addition of the PVA fiber and further mixing for 5 min. The as-obtained HPFRCC mixtures (Figure S1) were poured into the molds by layering and compacted by inserting and ramming.

2.3.2. Measurements of Pressure Bleeding Rate and Rheological Properties. The pressure bleeding rate was measured using a pressure bleeding meter. The rheological properties were tested with an ICAR Plus rheometer (Germann Instruments), as shown in Figure S2. Two types of tests were performed. The first was a stress growth test where the vane was rotated at a constant slow speed of 0.025 rev/s; the initial torque increase was measured as a function of time, and its maximum value was used to calculate the static yield stress. The other one was a flow curve test, conducted to determine the dynamic yield stress and plastic viscosity. It began with a breakdown period during which the vane was rotated at maximum speed; this was done to break down any thixotropic structure present in the mixture and provide a consistent shearing history before measuring the Bingham parameters. Then, the vane speed was decreased within a specified number of steps selected by the user (a minimum of six steps is recommended). At each step, the vane speed was held constant, and the average speed and torque were recorded. The Bingham parameters were derived from the flow curve, which is the plot of torque versus speed of vane rotation.

2.3.3. Measurement of Mechanical Strength. To determine the mechanical strength, all the specimens were covered with a plastic film and stored at a temperature of 20 ± 2 °C and relative humidity of 65 ± 2% for 24 h. Then, they were all demolded and placed in a standard maintenance room. The flexural and compressive strengths were tested with the

![Figure 1](https://doi.org/10.1021/acsomega.1c05068)
HPFRCC specimens aged 7 and 28 days, while those aged 28 days were used to evaluate the tensile performance and elastic modulus.

2.3.4. Measurement of Pore Structure and Microstructure.
The pore structure of the hardened HPFRCC was measured via MIP. Before testing, a broken HPFRCC block with a size of ∼6 mm was dried in an oven at 60 °C until constant weight. Its crystal morphology was observed through SEM.

3. RESULTS AND DISCUSSION

3.1. Rheological Properties. The high specific surface of the raw materials and the low w/c ratio generally lead to a decreased workability of the final composite.20–26 Therefore, investigating the rheological properties of fresh HPFRCCs is important. As shown in Table S5, as the w/c ratio of the prepared specimens increased, the \( t_{500} \) slump flow, and modified slump slightly rose, whereas the inverted slump time significantly decreased; this indicates that the w/c barely influences the \( t_{500} \) slump flow, and modified slump. Note that the air content of the specimens ranged from 3.9 to 2.6%, which is much smaller than the values reported by Kobayashi et al.27 The fresh slurry can be defined as a non-Newtonian thixotropic fluid.28–33 Moreover, according to the study by Kuder et al.,33,34 the rheological properties of fresh HPFRCCs can be described by the Bingham model as follows

\[
\tau = \tau_0 + \eta \dot{\gamma}
\]  

Figure 2. Rheological parameters of the HPFRCC specimens for different water–cement ratios (w/c). (a) Yield shear stress and (b) plastic viscosity values.

Figure 3. (a) Flexural strength and (b) compressive strength as functions of the water–cement ratio (w/c) for the HPFRCC specimens aged 7 and 28 days.

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Both the compressive and compressive strength was initially stable and then decreased. The specimens aged 7 and 28 days.

3.2. Mechanical Properties. 3.2.1. Flexural and Compressive Strengths. The addition of fiber improves flexural strength but barely influences compressive strength. The HPFRCC flexural and compressive strengths measured in the present study are plotted in Figure 3. As the w/c increased, the flexural strength first increased and then decreased, showing a maximum value of 26.0 MPa at a w/c ratio of 0.22. At a w/c ratio of 0.19–0.22, the flexural strength of the 7 day aged specimens was 24.0 and 25.7 MPa, respectively, while that of the 28 day aged ones was 24.3 and 26.0 MPa, correspondingly; thus, the flexural strength of the latter increased less (1.2%) than that of the former (1.3%). At a w/c ratio of 0.25–0.28, the flexural strength of the 7 day aged specimens was 15.9 and 14.3 MPa, respectively, and that of the 28 day aged ones was 21.9 and 20.9 MPa, correspondingly. This indicates a higher flexural strength for the specimens aged 28 days at a w/c ratio of 0.19–0.22 (respectively, 37.7 and 41.2% larger than that of those aged 7 days). As for the compressive strength of the specimens aged 7 and 28 days, it decreased from 70 and 100.3 to 40 and 72 MPa, respectively, with an increasing w/c. However, when the w/c was increased from 0.19 to 0.22, interestingly, the compressive strength of the 28 day aged specimens hardly decreased, but it was always ~40% higher than that of the 7 day aged ones.

This slightly differs from what was reported by Zhou et al. and Yaowarat et al., who stated that increasing the w/c may cause a continuous loss of strength. Within the investigated range of the w/c from 0.19 to 0.28, the flexural strength of the specimens first increased and then decreased, while the compressive strength was initially stable and then decreased. Both the compressive and flexural strengths showed the highest values when the w/c was 0.22; at a lower w/c, the compressive strength was not significantly higher, while the flexural strength was lower. This might be caused by the high air content and plastic viscosity of the specimens; besides, based on these results, the air was difficult to release when preparing the specimens with a w/c below 0.22. Figure 4 displays the trend in the ratio between flexural and compressive strengths, which was lower for the 28 day aged specimens than for the 7 day aged ones. It increased along with the w/c, indicating that this parameter has a less adverse effect on the flexural strength than on the compressive one.

3.2.2. Tensile Performance. Figure 5 shows the specimen size and experimental layout used to investigate the ultimate tensile stress and elongation of the 28 day aged specimens with different w/c ratios. The displacement control method was adopted for the loading; the speed was 0.5 mm/min, and the loading was ended when the tensile strength of the specimen decreased to 80% of the ultimate tensile strength. Based on the research conducted by Lu et al., as the w/c increases, the initial crack strength and ultimate tensile strength of PVA fiber-reinforced cement composites decrease, while the ultimate tensile strain increases. Choi et al. observed a reduction in the tensile strength from 7.9 to 5.9 MPa and an increase in the tensile strain capacity from 5.3 to 5.9% as the w/c of HPFRCCs increases from 0.34 to 0.38. Furthermore, Xue et al. reported that the PVA fiber can significantly improve the flexural toughness of the concrete, resulting in a higher elongation compared with polypropylene fiber-reinforced concrete.

The results of the tensile test conducted in the present study are displayed in Figure 6. The tensile strength fluctuated around 6.7 MPa in the w/c range of 0.19–0.28; this might be caused by the high strength of the cement matrix resulting from a too low w/c. This indicates that the w/c ratio barely influences the tensile strength when the ratio is relatively low. In contrast, the specimen elongation continued to increase along with the w/c, which is consistent with the results reported.

3.2.3. Elastic Modulus. Figure 7 illustrates the experimental setup used to investigate the elastic modulus of the HPFRCC specimens. The results are displayed in Figure 8, showing a reduction of 35.7% in the elastic modulus (from 27.7 to 17.8 GPa) when increasing the w/c from 0.19 to 0.28. This demonstrates that the w/c significantly influences the elastic modulus of HPFRCCs; a smaller w/c corresponds to higher rigidity of the specimen.

![Figure 4](https://example.com/fig4.png)  
**Figure 4.** Ratio between flexural and compressive strengths as a function of the water–cement ratio (w/c) for the HPFRCC specimens aged 7 and 28 days.
3.3. Pore Structure. 3.3.1. Water Absorption Rate and Dynamics. The water absorption property of concrete can be characterized by its dynamic parameters. Under the condition of temperature at 20 °C adopted in this study, when capillary pore adsorption occurs, the water absorption curve of the HPFRCC specimen has the characteristics of a stable exponential function, which can be expressed as

\[ w_t = w_{\text{max}} (1 - e^{-\alpha t}) \]  

where \( w_t \) is the water absorption rate of the specimen during the soaking time, \( w_{\text{max}} \) is the maximum water absorption by mass, \( \lambda \) is the average diameter of the capillary pores (a larger \( \lambda \) indicates a greater average pore size), and \( \alpha \) indicates the homogeneity of the capillary diameter (0 < \( \alpha < 1 \)). Table S6 summarizes the calculated water absorption rate and kinetic parameters of the HPFRCC specimens. When the w/c ratio was changed from 0.19 to 0.28, the water absorption rate at 48 h first decreased and then increased; when the ratio was 0.22, the water absorption rate at 48 h reached the lowest value (6.2%). The calculated overall pore size of HPFRCCs decreased when the w/c ratio reduced from 0.28 to 0.19, and the calculated hole uniformity coefficient also decreased accordingly from 0.34 to 0.28. Moreover, the calculations revealed a positive correlation between homogeneity of the capillary diameter and w/c; with decreasing the w/c, the pore uniformity became worse.

The relationship between the water absorption rate and strength of the HPFRCC specimens is illustrated in Figure 9. The higher the water absorption rate, the lower the strength, indicating a negative correlation between the compressive strength and water absorption rate. At the same time, the changing pattern of strength with water absorption explains why the strength decreased when the w/c was lower than 0.22; the plastic viscosity of the mixture increased, and the internal air became difficult to discharge. Hence, the number of open pores that could absorb the water in the specimen increased, adversely influencing the strength.

Aparna et al. reported a close correlation between pore size and compressive strength, and the experiment conducted in the present study confirmed this conclusion. The relationships between the flexural and compressive strengths of the HPFRCC with the average internal pore diameter displayed in Figure 10 showed close correlations. With the continuous increase in the average pore diameter, the flexural and compressive strength was decreased correspondingly. The effect of the average pore diameter on the flexural strength was greater for the 7 day aged specimens than for the 28 day aged ones, while there was no significant difference for its effect on the compressive strength. Figure 11 shows a linear relationship between the capillary diameter homogeneity and w/c. Therefore, the relationship between capillary diameter...
homogeneity and strength should be similar to that between the w/c and strength.

3.3.2. MIP Results. The gas and water transport, frost resistance, shrinkage, and mechanical properties of cementitious building materials are influenced by their porosity. The MIP results are shown in Figure 12 and summarized in Table S7. In the w/c range tested, the median pore size, average pore size, and threshold aperture decreased along with the w/c ratio. However, when the w/c was reduced from 0.22 to 0.19, the median and average pore sizes increased significantly, and the threshold aperture also showed a slight increase. The median pore size and threshold aperture at a 0.19 w/c were much larger (121.18 and 319.04 nm, respectively) than those at the other w/c values; in contrast, the total porosity decreased when the w/c reduced from 0.28 to 0.19. Note that the w/c ratio is the main factor affecting the compressive strength of concrete due to its direct effect on the final porosity. The smaller porosity observed at the w/c of 0.19 and 0.22 w/c (12 and 13.76%, respectively) indicates a higher compressive strength, confirming the results shown in Figure 5. All these results show that the number of pores decreased at a w/c of 0.22 or lower, while the pore diameter

Figure 9. Effect of the water absorption rate on the (a) flexural and (b) compressive strengths.

Figure 10. Effect of the average pore diameter on the (a) flexural and (b) compressive strengths.

Figure 11. Relationship between the capillary diameter homogeneity and water–cement ratio (w/c).
increased and the pore uniformity became worse. These results are consistent with the conclusion drawn using eq. 2. Furthermore, the average pore size value measured at a w/c of 0.19 (1.37 μm) is close to the calculated one (0.32 μm).

The pore size distribution of the HPFRCC specimens is presented in Figure 13 according to the classification method proposed by Wu and Lian.16 Most of the internal pores had a diameter of 20–100 nm or above 200 nm, while the proportion of those with a diameter of 100–200 nm was relatively low. The w/c significantly influenced the pore size distribution. When the w/c was 0.19, pores of a diameter of 100–200 nm only accounted for ~5% of the total pores, which is far less than its proportion when the w/c was within the range of 0.22–0.28. Furthermore, when the w/c was further reduced from 0.22 to 0.19, the number of pores with a diameter larger than 200 nm increased sharply. In plain concrete, the porosity widely depends on the chemical composition of the raw materials, the hydration processes, and the packing properties.49 Nevertheless, the distribution of pores larger than 200 nm observed for the HPFRCC specimen having a w/c ratio of 0.19 depended mostly on the undischarged air. These experimental results explain why, when the w/c decreased from 0.22 to 0.19, the compressive strength did not continue to increase, the flexural strength decreased, and the water absorption rate rose.

3.4. SEM Results. The SEM images (Figure 14) showed that as the w/c increased from 0.19 to 0.28, the number of pores increased, and their maximum diameter decreased to 200 μm. In other words, the number of small pores increased, the number of large pores decreased, and the pore size distribution became more and more uniform. These observations are consistent with the results of the mechanical, water absorption dynamics, and MIP measurements. Moreover, the findings of the water absorption dynamics calculations and MIP can effectively reflect the internal pore distribution of the HPFRCC specimens, demonstrating that the pore structure and number are important factors determining the macroscopic strength.

According to the theory of central mass, with decreasing the water–binder ratio, the pore size of hydrated cement and the porosity of hardened paste decrease, while the number of unhydrated cement particles increases. In the present study, the FA content in the adhesive system exceeded 50%. With the extension of the curing age, the FA needed to consume the Ca(OH)₂ produced after cement hydration. Thus, the generated Ca(OH)₂ was continuously reduced, greatly reducing its further formation at the interface and consequently improving the local microstructure. The porosity also decreased with extending the curing age; the pore diameter was continuously reduced, and the mixture presented an overall dense microstructure, enhancing its mechanical properties and durability (in terms of impermeability, carbonation resistance, and frost resistance).

4. CONCLUSIONS

The rheological and mechanical properties of HPFRCCs with a low water–cement ratio (0.19–0.28) were experimentally investigated, leading to the following conclusions:

(1) The yield shear stress and plastic viscosity decreased with an increasing w/c. Their smaller values were conducive to the air discharge from the mixture, reducing the air content.

(2) The ratio between flexural and compressive strengths increased along with the w/c. The tensile test results indicated that when the w/c is relatively low, it has little
effect on the tensile strength; however, the specimen elongation increased along with the w/c. The elastic modulus test results showed that the smaller the w/c is, the more rigid the specimen is.

(3) Most internal pores had a diameter of 20−100 nm or above 200 nm, while the proportion of those with a diameter of 100−200 nm was relatively low. The flexural strength was more sensitive than the compressive strength to the changes in the water absorption rate and kinetic parameters, pore size distribution, and pore structure morphology. The specimen with a w/c of 0.19 exhibited the lowest total porosity, the worst internal pore uniformity, and the largest number of pores with a diameter above 200 nm.

**ASSOCIATED CONTENT**

**Supporting Information**
The Supporting Information is available free of charge at https://pubs.acs.org/doi/10.1021/acsomega.1c05068.

Appearance of the prepared HPFRCC; layout for the rheological tests; chemical composition of the OPC, FA, and SF used in this study; physical and mechanical properties of the OPC used in this study; properties of the polyvinyl alcohol fiber; mixture proportions (kg/m³) of the HPFRCC specimens; rheological test results (w/c: water−cement ratio); water absorption rate and kinetic parameters; and pore structure parameters (PDF)

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**Figure 14.** Scanning electron micrographs of the HPFRCC specimens.
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Notes
The authors declare no competing financial interest.

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