Performance evaluation of steel-polypropylene hybrid fiber reinforced 1 concrete under supercritical carbonation 2 3 4 Hao Bao<sup>a</sup>, Min Yu<sup>b,\*</sup>, Yin Chi<sup>b</sup>, Yu Liu<sup>b</sup>, Jianqiao Ye<sup>c,\*</sup> 5 a. College of Civil Engineering & Architecture, China Three Gorges University, Yichang, China 6 b. School of Civil Engineering, Wuhan University, Wuhan, China 7 c. Department of Engineering, Lancaster University, Lancaster LA1 4YR, UK 8 \* Correspondence author: ceyumin@whu.edu.cn (M. Yu), j.ye2@lancaster.ac.uk (J. Ye) 9 Abstract: In this paper, systematic supercritical carbonation tests of steel-polypropylene hybrid fiber 10 reinforced concrete (SPFRC) were carried out to evaluate the performance of SPFRC under supercritical 11 12 condition. The effects of the length-diameter ratio of steel fiber, volume fraction of steel fiber, and polypropylene fiber on the carbonation depth and compressive strength of concrete under supercritical 13 14 condition were studied. A one-dimensional mathematical model for the physical-chemical coupling process of 15 supercritical carbonation of cement-based materials was established. The relational model between the equivalent porosity and the compressive strength of fully carbonated SPFRC was also proposed. Results 16 17 indicate that whether the addition of steel fibers or polypropylene fibers or the inclusion of fibers can 18 accelerate the carbonation process by the increase of porosity. The carbonation depths of SPFRC increase with 19 the increase of the addition of steel fibers and polypropylene fibers. The compressive strength after 20 carbonation is significantly increased. The maximum relative compressive strength was obtained when the 21 volume fraction of steel fibers and polypropylene fibers were 1.5% and 0.0% and the length-diameter ratio of 22 steel fiber was 60, respectively. Furthermore, a mathematical model was proposed to evaluate the equivalent 23 initial porosity of SPFRC.

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Keywords: Steel fibers; Polypropylene fibers; Supercritical carbonation; Carbonation depth; Compressive
 strength.

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## 28 **1. Introduction**

Fiber-reinforced concrete (FRC), a typical engineering material <sup>[1]</sup>, has been extensively utilized in tunnel 29 linings<sup>[2]</sup>, military engineering<sup>[3]</sup>, etc. There are significant advantages in the utilization of FRC. The cracking 30 is prevented by toughening of steel and polypropylene fiber reinforced concrete <sup>[4-6]</sup>, the microstructures of 31 32 concrete are changed <sup>[7, 8]</sup>, tensile and flexural strength are improved <sup>[9, 10]</sup>. The material limitations of traditional concrete such as low tensile strength, brittleness, and poor ductility can be avoided by the addition 33 of steel fibers in concrete [11]. The dry shrinkage deformation of concrete can be reduced by the addition of 34 polypropylene fibers in concrete <sup>[12]</sup>. For reinforced concrete structures, the transport of CO<sub>2</sub> in concrete and 35 36 the carbonation reaction will convert Ca(OH)<sub>2</sub> into CaCO<sub>3</sub>, reduce the pH value of pore solution, and destroy 37 the passivation film on the surface of steel bars in concrete structures, cause the corrosion of reinforcements <sup>[13,</sup> 38 <sup>14]</sup>. The maximum carbonation rate will be obtained when the relative humidity used for carbonation in a controlled environment ranges from 55% to 70% <sup>[15]</sup>. However, FRC is a cementitious composite, when FRC 39 40 structures are subjected to carbonation penetration, the carbon dioxide will react with the alkaline substances in the FRC. The carbonation products, which had a higher molar volume than the original phases <sup>[16]</sup>, could 41

42 decrease the pore volume and permeability of the concrete products <sup>[17]</sup>. The capillary porosity, moisture 43 contents and microcracking of FRC are reduced after the CO<sub>2</sub> treatment. CO<sub>2</sub> curing yields better flexural 44 strength <sup>[18]</sup>. Then the compressive strength was increased and the porosity was reduced <sup>[9]</sup>.

The accelerated carbonation technology had been extensively applied to study the carbonation 45 performance of fiber-reinforced concrete <sup>[19]</sup>. However, the research period of the accelerated carbonation test 46 ranges from 3 to 24 months, and the corresponding carbonation depth varies between 1 to 10 mm <sup>[20]</sup>. 47 Meanwhile, with the development of techniques of carbon dioxide storage <sup>[21]</sup>, supercritical carbonation 48 techniques have been applied in the treatment of cement-based materials <sup>[22-31]</sup> and the seal of heavy metal <sup>[16,</sup> 49 <sup>32-34]</sup>. CO<sub>2</sub> will be in a supercritical fluid condition with the temperature and pressure exceed 304.12 K and 50 7.38 MPa, respectively <sup>[35]</sup>. The accomplishment of the carbonation test of concrete can be sharply shortened to 51 almost 7.5 hours with a carbonation depth of 10 mm <sup>[36]</sup>. Supercritical carbonation techniques have become a 52 53 promising and time-saving method in the study of carbonation resistance of fiber reinforced concrete. Furthermore, the mechanical properties, microstructural stability [37], and toughness of fiber reinforced 54 concrete can be improved after the supercritical carbonation  $CO_2$  treatment <sup>[38]</sup>. The supercritical carbonation 55 treatment significantly increased the designed strength and toughness of the fiber-reinforced cement and 56 greatly increased the fiber-matrix bond <sup>[39]</sup>. However, the addition of steel fibers and polypropylene fibers can 57 58 increase the  $CO_2$  penetration path and decrease the compactness of the matrix, and then reduce the carbonation 59 resistance of SPFRC. The strengthening mechanism of the supercritical carbonation treatment of SPFRC is indeterminate. The optimal contents of steel and polypropylene fibers are controversial. 60

61 Carbonation depth, compressive strength and  $Ca(OH)_2$  into  $CaCO_3$  conversion are the three main indicators to be concerned for the performance evaluation of the carbonated SPFRC<sup>[40, 41]</sup>. Whatever natural 62 carbonation, accelerated carbonation, and supercritical carbonation, the average carbonation depth was 63 concerned as one of the factors to evaluate the carbonation degree of concrete [42, 43]. For the corrosion of 64 reinforced concrete, the maximum carbonation depth is more important than the average carbonation depth. 65 Experimental research has shown that under both natural <sup>[44]</sup> and supercritical <sup>[36, 41, 45]</sup> conditions, the boundary 66 topography of a carbonation zone is irregular, characterized by a random distribution of depth along the 67 68 boundary with distinctive maximum and minimum. Meanwhile, the compressive strength of concrete increases 69 after the treatment of carbonation. Due to carbonation, the compressive strength of concrete cured in the 70 accelerated carbonation chamber was higher than that of concrete cured in the general air environment <sup>[46]</sup>. The 71 compressive strength of concrete increases rapidly at the initial stage of carbonation and increases gradually 72 with the increase of carbonation depth. When the carbonation depth exceeds a certain value, the compressive strength of concrete increases slowly <sup>[47]</sup>. There is no systematic and in-depth study on the variation of 73 74 compressive strength and carbonation depth of SPFRC after the supercritical carbonation treatment.

Due to the complex nature of the problem, this paper attempts, as a pioneer work <sup>[36, 45, 48]</sup>, to focus on investigating the performance of steel-polypropylene hybrid fiber reinforced concrete under supercritical carbonation. The supercritical carbonation test of SPFRC with different mix proportions was carried out. The effects of length-diameter ratio of steel fiber, the volume fraction of steel fiber, and polypropylene fiber on the carbonation resistance were discussed. The compressive strength of SPFRC specimens after and without supercritical carbonation was tested. The variation of compressive strength without and after the carbonation of SPFRC specimens with different mix proportions was analyzed. The supercritical carbonation model of SPFRC was established based on the previous work <sup>[41]</sup>. The variation of average carbonation depth over time in the process of supercritical carbonation of concrete was simulated and the value of equivalent initial porosity of the matrix of SPFRC was estimated. The influence of types and contents of fibers on the carbonation depth was obtained. The carbonation depth of SPFRC at different initial porosity was studied and the equivalent porosity of SPFRC with different mix proportions was obtained. Besides, the mathematical model of the relationship between the compressive strength and the equivalent porosity of SPFRC after full carbonation was proposed.

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# 90 2. Experimental program

## 91 2.1. Materials and mix design

92 In the test, Portland cement type P.O 42.5 was used as the binder for the mixes. The chemical components 93 of cement were determined by an X-ray fluorescence spectrometer (XRF) are listed in Table 1. Natural river 94 sands with a fineness modulus of 2.69 and gravels with a size range of 5 mm to 20 mm were used as the fine 95 and coarse aggregates, respectively. The polycarboxylic acid high-performance water reducing agent with a 96 water-reducing rate of 25%~35% was used. The concrete was designed according to the specification for the mix proportion design of ordinary concrete <sup>[49]</sup> and technical specification for reinforced concrete structures <sup>[50]</sup> 97 with the designed cube compressive strength of 40 MPa. The corresponding designed mix proportions of 98 99 concrete are shown in Table 2.

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**Table 1** Chemical components of cement, in mass percent (%).

	SiO <sub>2</sub>	$Al_2O_3$	$Fe_2O_3$	CaO	MgO	$SO_3$	$R_2O$		
	21.7	4.84	3.19	62.26	2.35	3.57	2.09		
101	Remarks: R <sub>2</sub> O defines the general term for oxides with very little content.								
102	2 <b>Table 2</b> Mix proportions of concrete.								
	Components	Cement	Gravel	Sand	Wate	r	Water-binder ratio		
	Unit $(kg/m^3)$ (k			$(kg/m^3)$ $(kg/m^3)$		n <sup>3</sup> )	-		
	Concrete	417	1086	724	175		0.42		

<sup>103</sup> 

The shear wave steel fibers with the length-diameter ratio of 30, 60, 80, polypropylene fibers with a length of 11 mm, and the coarse aggregates were prepared for the experiment. The physical and mechanical properties of steel fibers and polypropylene fibers were shown in Table 3.

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 Table 3 Physical and mechanical properties of steel fibers and polypropylene fibers.

	r r r r		JF FJ F F F F F F F F F F F F F F F F F
Type of fibers	Equivalent diameter	Density (g/cm <sup>3</sup> )	Compressive strength (MPa)
Shear wave fiber	0.55 mm	7.8	≥600
Bunchy monofilament fiber	18μm ~48μm	0.91	≥450

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109 2.2. Specimens preparation

110 To study the effects of the supercritical carbonation on the carbonation mechanism of steel-polypropylene 111 hybrid fiber reinforced concrete, the parameters considered in this study include fiber type, the volume fraction 112 of fiber, length-diameter ratio of steel fiber. The compressive strength of concrete and the specimen's design of

steel-polypropylene hybrid fiber reinforced concrete (SPFRC) were summarized and shown in Table 4.

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 Table 4 Specimens design of SPFRC.

	Length-diameter ratio of	Volume fraction of steel	Volume fraction of polypropylene
Group number	steel fiber, $\lambda_{SF}$	fiber, $\rho_{SF}$	fiber, $\rho_{PF}$
SPFRC0000(60)	60	0	0
SPFRC0500(60)	60	0.5%	0
SPFRC1000(60)	60	1.0%	0
SPFRC1500(30)	30	1.5%	0
SPFRC1500(60)	60	1.5%	0
SPFRC1500(80)	80	1.5%	0
SPFRC0005(60)	60	0	0.05%
SPFRC0505(60)	60	0.5%	0.05%
SPFRC1005(60)	60	1.0%	0.05%
SPFRC1505(60)	60	1.5%	0.05%
SPFRC0010(60)	60	0	0.1%
SPFRC0510(60)	60	0.5%	0.1%
SPFRC1010(60)	60	1.0%	0.1%
SPFRC1510(30)	30	1.5%	0.1%
SPFRC1510(60)	60	1.5%	0.1%
SPFRC1510(80)	80	1.5%	0.1%
SPFRC0015(60)	60	0	0.15%
SPFRC0515(60)	60	0.5%	0.15%
SPFRC1015(60)	60	1.0%	0.15%
SPFRC1515(60)	60	1.5%	0.15%

115 *Remarks*: As shown in SPFRC1505(60), SPFRC represents the steel-polypropylene hybrid fiber reinforced concrete. 15 116 represents the volume fraction of steel fiber is 15%. 05 represents the volume fraction of polypropylene fiber is 5%. 60 represents 117 the length-diameter ratio of steel fiber is 60.

118 A standard slump flow test was conducted to measure the slump of all mixtures <sup>[51]</sup>. The top diameter, 119 bottom diameter, and height of the slump cone were 100 mm, 200 mm, and 300 mm, respectively. The slump 120 cone was placed on the plate and the fresh SPFRC was cast into the slump cone fully. Then, the slump cone 121 was left vertically and smoothly to allow the concrete mixture to flow out freely. The lifting process of the 122 slump cone was completed in 5 seconds. The final diameter of the concrete mixture after slump flow was 123 measured by the steel ruler, and the measurement was carried out in two directions perpendicular to each other, 124 and the average value of the two measured diameters was taken as the value of slump flow. The target slump is 125 30 mm.

126 Steel-polypropylene hybrid fiber reinforced concrete cubes with a dimension of  $100 \text{ mm} \times 100 \text{ mm} \times 100$ 127 mm were designed for the experiments of supercritical carbonation. 20 groups of SPFRC specimens with four 128 different volume fractions of steel fiber ( $\rho_{SF} = 0, 0.5\%, 1.0\%, 1.5\%$ ), four different volume fractions of 129 polypropylene fiber ( $\rho_{PF} = 0, 0.05\%, 0.10\%, 0.15\%$ ) and three different length-diameter ratios of steel fiber 130  $(\lambda_{SF} = 30, 60, 80)$  were cast. There are nine SPFRC specimens in each group and the total number of specimens is 180. Three of them were carbonated for the measurement of carbonation depth. Another three were 131 carbonated for the measurement of compressive strength after the carbonation of concrete. The remaining three 132 133 specimens were not carbonated for the measurement of the compressive strength of concrete without 134 carbonation. After casting, the test specimens were covered with plastic sheets and left in the casting room for 135 24 hrs. The specimens were then demolded and placed into a standard curing room with a constant temperature of 20 °C and humidity of 95% until 28-day strength was achieved. The specimens were taken out and placed in 136 the temperature and humidity control chamber for further curing for 28 days after the 28 days of curing in the 137 138 standard curing room. The relative humidity was set at 70% to control the initial water retention rate of the 139 concrete specimens. The average value of the recorded relative humidity was 68.4%.

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- 141 2.3. Experimental program
- 142 2.3.1. Supercritical carbonation of SPFRC

To monitor the supercritical carbonation test process of steel-polypropylene hybrid fiber reinforced 143 144 concrete more efficiently, the supercritical carbonation device was applied in the test. CO<sub>2</sub> can be compressed 145 into the supercritical condition and the steel-polypropylene hybrid fiber reinforced concrete can be carbonated. The details of the closed-cycle carbonation device are shown in Fig. 1 (a). During supercritical carbonation of 146 147 SPFRC specimens, the temperature and pressure in the reaction still were controlled by temperature and 148 pressure controller as shown in Fig. 1 (b) and (c), respectively, and detected by the temperature sensor and the 149 pressure gauge, respectively. The recirculated water and heating rods can be utilized to control the temperature in the reaction still. More details of this supercritical carbonation system are shown in previous work <sup>[36]</sup>. 150







c) Pressure controller

Fig. 1. Closed-cycle carbonation setup.

151 The main experiment procedure of supercritical carbonation of steel-polypropylene hybrid fiber 152 reinforced concrete is shown as follows <sup>[36]</sup>:

- 153 1) Six concrete cubes were placed on the bracket as shown in Fig. 2 (a).
- 154 2) The bracket with the specimens was placed in the reaction still as shown in Fig. 2 (b).
- 3) The reaction still was vacuumed to almost -1.00 bar after the seal of the chamber as shown in Fig. 2(c).
- 4) The injection of  $CO_2$  in the  $CO_2$  tanks to the reaction still by an air compressor until the accomplishment and maintaining of the target pressure and temperature in the reaction still.
- 159 5) The CO<sub>2</sub> gas was driven from the reaction still to the CO<sub>2</sub> tanks by the air compressor to realize the 160 recycling of CO<sub>2</sub>.
- 161 6) The six carbonated steel-polypropylene hybrid fiber reinforced concrete cubes were removed from the162 reaction still.







a) Specimens on the bracket b) Specimens in the reaction still c) Negative pressure gauge **Fig. 2.** Preparation of the supercritical carbonation of SPFRC.

163 In the supercritical carbonation test, the designed pressure and temperature are 8.0 MPa and  $40^{\circ}$ C, 164 respectively. From the pre-carbonation results, it is observed that a minimum of 5-6 hours of supercritical 165 carbonation time was required for reliable data processing, longer carbonation time would lead to complete 166 carbonation with no or very short carbonation boundary to be analyzed. Therefore, the designed supercritical 167 carbonation time was six hours. During the procedure of supercritical carbonation of steel-polypropylene hybrid fiber reinforced concrete, the temperature and pressure were monitored. Without loss of generality, only 168 169 partial recorded temperature and pressure of SPFRC specimens during the supercritical carbonation process 170 were shown in Fig. 3. As can be seen from Fig. 3, the pressure and temperature in the reaction still can be 171 controlled stably by the closed-cycle carbonation setup.



Fig. 3. Carbonation condition of SPFRC: Carbonated for 7.17 h (supercritically carbonated for 6 h).

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#### 173 2.3.2. SEM observation

174 To study the effect of steel fibers and polypropylene fibers on the carbonation boundaries 175 microcosmically, the SPFRC samples without and after the achievement of supercritical carbonation were 176 tested by Scanning Electron Microscopy (SEM). The microtopography of steel fiber, polypropylene fiber, 177 matrix, and cracks was also observed. Before the observation, some carbonated SPFRC samples were received cutting and sanding with the power tool and then the fresh cuts of the cubes were polished and cleaned to 178 179 avoid the effect of dust on observation results. The effect of the distribution of steel fibers and polypropylene 180 fibers near the boundary between the carbonation zone and non-carbonation zone of concrete on the 181 carbonation depth is the focus of the observation. Finally, the samples were prepared to observe the 182 microstructure of matrix, steel fibers, and polypropylene fibers by the SEM test. The prepared samples were dried in an oven to a constant weight and the surfaces of the samples were coated with gold powder in an ion 183

spatter to increase their conductivity. The samples were vacuumed after the placement of samples on the stage of a scanning electron microscope. Then the contrast, brightness, the best multiple and rate of scanning were adjusted and determined. The images were taken on a JSM-7500F equipped with a tungsten filament. In the process of samples preparation and testing, the integrity and health of the samples were guaranteed.

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189 2.3.3. Compressive strength testing

After the accomplishment of the supercritical carbonation test of SPFRC, partial specimens were prepared to obtain the compressive strength of SPFRC after the carbonation. The compressive strength test of SPFRC cubes was measured using a 3000 kN MTS YAW6306 testing machine according to the test methods used for steel fiber reinforced concrete (CECS 13: 2009) <sup>[51]</sup>. The loading rate is 1.2 MPa/s. The ends of the specimens were polished smoothly to avoid the influence of stress concentration on the compressive behavior of the specimens before the compressive strength testing. The compressive loads and corresponding displacements were automated recorded by the testing system. The ultimate compressive loads were also obtained.

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# 198 **3. Results and discussion**

### 199 3.1. Carbonation results

200 The carbonation depth of concrete specimens was measured and compared in this Section. After the achievement of the supercritical carbonation of SPFRC specimens, the carbonated concrete cubes were 201 202 removed from the reaction still. Then some of the specimens were cut into two halves with a precise power 203 cutting tool. The appropriate cutting speed was determined as 1 mm/s according to the preliminary experiment. 204 The steel fibers and polypropylene fibers in concrete can be completely cut off. The pullout and fracture of 205 fibers and the debonding of the interface between cement matrix and fibers can be avoided. The fresh cuts of 206 the samples were polished and cleaned. Then the phenolphthalein solution was sprinkled homogeneously on 207 the cutting surface. The carbonation boundary can be identified after the accomplishment of the color reaction. 208 The carbonation profiles can be obtained by a scanner <sup>[36]</sup>. The enlarged details of the carbonation profile of 209 partial SPFRC specimens were shown in Fig. 4. As shown in Fig. 4, the random nature of carbonation boundaries not only can be affected by the random distribution of porosity and coarse aggregates <sup>[36, 42]</sup> but also 210 211 affected by the existence of steel fibers. However, the effect of polypropylene fibers on the distribution of 212 carbonation depth cannot be macroscopically observed.



(a) SPFRC0000(60)
 (b) SPFRC0500(60)
 (c) SPFRC1510(80)
 Fig. 4. Zoomed-in details of test carbonation profile of partial SPFRC specimens.

respectively. Fig. 5 shows the carbonation depth of steel-polypropylene hybrid fiber reinforced concrete cubes with various volume fractions of steel fibers and a fixed length-diameter ratio of steel fibers of 60. Fig. 6 shows the carbonation depth of steel-polypropylene hybrid fiber reinforced concrete cubes with a various volume fractions of polypropylene fibers and a fixed length-diameter ratio of steel fibers of 60. Fig. 7 shows the carbonation depth of steel-polypropylene hybrid fiber reinforced concrete cubes with various length-diameter ratios of steel fibers. As shown in Figs. (5-7), there are irregular carbonation boundaries around the non-carbonated zone.



Fig. 5. The carbonation depth of steel-polypropylene hybrid fiber reinforced concrete cubes with various volume fractions of steel fibers.







- 223 It can be generally concluded that the carbonated areas increase with the increase of the volume fraction of steel fibers or polypropylene fibers according to the carbonation results in Fig. 5 and Fig. 6. The effect of 224 225 the addition of fibers on the random nature of the carbonated areas cannot be ignored. The increase of volume 226 fraction of fibers increases the length and area of the weak interface transition zone between the fibers and 227 cement matrix, leading to faster transport of CO<sub>2</sub> and more rapid carbonation in SPFRC<sup>[8, 52]</sup>. As shown in Fig. 228 7, the maximum carbonated areas were obtained when the length-diameter ratio of steel fibers is 60.
- 229 To statistically and quantitatively analyze the effect of steel fibers and polypropylene fibers on the 230 carbonated areas, the distribution of the measured carbonation depths along the carbonation zone was obtained 231 by the imaging processing technique <sup>[36]</sup>. The illustration of the process of carbonation results of SPFRC was 232 concluded and shown in Fig. 8. More details about the image processing of the irregular carbonation profiles can be found in the previous work [36]. Finally, the results of the carbonation depth of SPFRC after 233 234 supercritical carbonation were shown in Table 5.
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Fig.	8.	Illustration	of the	process	of	carbonation	results	of SPFRC.
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Table 5 Results of supercritical carbonation depth of SPFRC.							
Spacimons number	Average carbonation	Coefficient	Spacimons number	Average carbonation	Coefficient		
specifiens number	depth (mm)	of variation	specifiens number	depth (mm)	of variation		
SPFRC0000(60)	23.47	0.20	SPFRC0010(60)	24.98	0.23		
SPFRC0500(60)	23.71	0.11	SPFRC0510(60)	27.90	0.11		
SPFRC1000(60)	24.41	0.11	SPFRC1010(60)	28.99	0.12		
SPFRC1500(30)	28.04	0.19	SPFRC1510(30)	25.93	0.18		
SPFRC1500(60)	25.86	0.16	SPFRC1510(60)	30.53	0.13		
SPFRC1500(80)	30.04	0.20	SPFRC1510(80)	30.94	0.12		
SPFRC0005(60)	23.80	0.12	SPFRC0015(60)	28.68	0.14		
SPFRC0505(60)	26.66	0.18	SPFRC0515(60)	32.51	0.14		
SPFRC1005(60)	25.66	0.17	SPFRC1015(60)	34.26	0.11		
SPFRC1505(60)	28.27	0.15	SPFRC1515(60)	39.56	0.14		

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3.2. Effect of supercritical carbonation on the microstructure of SPFRC 241

242 The SEM images of a typical area of the steel fiber, polypropylene hybrid fiber, cracks, and the matrix with the magnification times of 100 are shown in Fig. 9. The microstructure of steel fiber reinforced concrete 243 with the number of the specimen of SPFRC1500(60) without and after the carbonation treatment are shown in 244 245 Figs. 9 (a) and (b), respectively. The microcracks (Fig. 9a) occurred along the narrow gap between the matrix and steel fiber without carbonation can be seen, while no cracks along the boundary between matrix and steel 246 fiber can be observed (Fig. 9b) and the matrix attached to the steel fiber firmly after carbonation. This indicates 247 248 that the porosity or cracks between the matrix and steel fiber can be reduced by the supercritical carbonation 249 treatment. The microtopography of polypropylene hybrid fiber reinforced concrete (SPFRC0015(60)) without

250 and after carbonation is shown in Figs. 9 (c) and (d), respectively. The cracks around the polypropylene hybrid 251 fiber can be observed without and after carbonation. However, the width of the cracks was reduced after supercritical carbonation treatment. Without loss of generality, the SEM images of SPFRC1515(60) without 252 and after carbonation treatment were shown in Figs. 9 (e) and (f), respectively. Also, the supercritical 253 254 carbonation treatment can reduce the cracks between the matrix and steel fibers or polypropylene hybrid fibers to some extent. The weakness of the interfacial transition zone between the cement matrix and fibers may 255 shorten the transport time of CO<sub>2</sub>, leading to more rapid carbonation in the ITZ<sup>[8, 52]</sup>. The existence of steel 256 fibers and polypropylene fibers in the SPFRC may result in the random nature of carbonation boundaries. 257



a) Steel fiber reinforced concrete without carbonation (SPFRC1500 (60))



c) Polypropylene hybrid fiber reinforced concrete without carbonation (SPFRC0015(60))



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b) Steel fiber reinforced concrete after carbonation (SPFRC1500 (60))



d) Polypropylene hybrid fiber reinforced concrete after carbonation (SPFRC0015(60))



- e) Steel-polypropylene hybrid fiber reinforced concrete f) Steel-polypropylene hybrid fiber reinforced concrete without carbonation (SPFRC1515(60)) after carbonation (SPFRC1515(60))
  - Fig. 9. SEM micrographs of steel-polypropylene hybrid fiber reinforced concrete samples without and after supercritical carbonation.

#### 260 3.3.1. Effect of steel fibers

261 The comparison of the carbonation depth of SPFRC with different volume fractions of steel fibers under 262 supercritical condition is shown in Fig. 10. It can be concluded that the carbonation of concrete is generally accelerated by the addition of steel fibers in the SPFRC for the average carbonation depth of concrete increases 263 264 with the increase of volume fraction of steel fibers. In Fig. 10, the colored lines and symbols show the average 265 carbonation depth and the error bars represent the minimum and maximum carbonation depth. The maximum 266 and minimum carbonation depths are discrete with the increase of volume fraction of steel fibers, which may 267 be caused by the random nature of the distribution of carbonation depth and limited experimental data. The 268 maximum and minimum carbonation depth are fluctuant with the increase of volume fraction of steel fiber for the experimental error, positive or negative hybrid effect <sup>[53]</sup> or the deterioration of liquidity, increase of 269 imperfection and porosity of concrete by the caking and sinkage of steel fibers and floating of coarse 270 271 aggregates <sup>[54]</sup>. The fluctuation of minimum and maximum carbonation depth may also be caused by the variation of consistency and workability of SPFRC with different volume fractions of steel fibers and 272 273 polypropylene fibers and the change of porosity during the curing and supercritical carbonation of SPFRC. 274



**Fig. 10.** Influence curves of volume fraction of steel fibers on carbonation depth ( $\lambda_{SF}$  =60).

SPFRC1500 and SPFRC1510 were taken as examples to analyze the effect of the length-diameter ratio of 275 276 steel fiber on the supercritical carbonation depth of concrete. The influence curve of length-diameter ratio of steel fibers on carbonation depth was shown in Fig. 11. The carbonation resistance is enhanced with the 277 278 increase of the length-diameter ratio of steel fibers within a certain range from 30 to 60, but a further increase 279 of the length-diameter ratio of steel fibers from 60 to 80 will reduce the carbonation resistance. The 280 carbonation resistance of a length-diameter ratio of 60 is better than that of 30 or 80. The error bars show that 281 the effect of the length-diameter ratio of steel fibers on the maximum and minimum carbonation depth is not 282 obvious for the random distribution of coarse aggregates and fibers. As shown in Fig. 11, the maximum 283 carbonated areas were obtained when the length-diameter ratio of steel fibers is 60. It is possible that when the length-diameter ratio of steel fibers is 30, the effect of fibers on the carbonated areas is not as great as that of 284 coarse aggregates. In addition, when the length-diameter ratio of steel fibers is 80, the ITZ between fibers and 285 286 matrix is filled with calcium carbonates produced by the reaction of calcium hydroxide and CO<sub>2</sub>. Then the reaction rate of carbonation and the carbonated areas are reduced. The hybrid of steel fibers and polypropylene 287 288 fibers increases the defects of the matrix and leads to the significant carbonation of concrete.



Fig. 11. Effect curves of the length-diameter ratio of steel fibers on carbonation depth ( $\rho_{SF} = 1.5\%$ ).

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#### 290 3.3.2. Effect of polypropylene fibers

The comparison of the carbonation depth of SPFRC with different volume fractions of polypropylene fibers under supercritical condition is shown in Fig. 12. The colored lines and symbols also show the average carbonation depth and the error bars represent the minimum and maximum carbonation depth. In general, the average carbonation depth increase with the increase of the addition of polypropylene fibers. When the volume fraction of polypropylene fibers is below 0.05%, the effect of polypropylene fibers on the average carbonation depth is not obvious. Also, there is no significant effect of the volume fraction of polypropylene fibers on the maximum and minimum carbonation depth of SPFRC.



Fig. 12. Influence curves of volume fraction of polypropylene fibers on carbonation depth ( $\lambda_{SF}$  =60).

298 3.4. Compressive strength of SPFRC after supercritical carbonation

299 3.4.1. Results of compressive strength before and after supercritical carbonation

300 Without loss of generality, the failure pattern of typical SPFRC specimens was shown in Fig. 13. It can be 301 concluded that the addition of steel fibers or polypropylene fibers can prevent the disintegration of specimens 302 to some extent by comparing the failure pattern of specimens in Figs. 13 (a-c). In the meantime, the integrity of 303 the specimen can be better ensured by the addition of polypropylene fibers than steel fibers. This may be 304 attributed to the more uniform distribution of polypropylene fibers, which decreases the propagation of the 305 fracture plane <sup>[55]</sup>. As shown in Fig. 13, the failure pattern of SPFRC specimens demonstrated that the fracture 306 plane propagates through the cement paste. The compressive strength of SPFRC depends strongly on the 307 cement paste. The toughness of concrete was improved by the addition of steel fibers or polypropylene fibers for the bridging effect of fibers across cracks <sup>[56]</sup>. The crack propagation was retarded, and the fracture plane 308

309 was bridged by fibers <sup>[57]</sup>.



Fig. 13. Failure pattern of SPFRC.

The relative compressive strength was proposed to evaluate the effect of carbonation on the compressive properties of a cement-based material. It is a specific value of the compressive strength of concrete cubes after carbonation and without carbonation. It is defined as the following equation <sup>[58]</sup>:

$$k_{RC} = \frac{f_{CC}}{f_{CO}} \tag{1}$$

where  $k_{RC}$  is the parameter of relative comprehensive compressive strength.  $f_{CO}$  and  $f_{CC}$  are the comprehensive compressive strength of specimens without and after carbonation with the unit of MPa, respectively. The partially carbonated cubes can be regarded as the combination of a fully carbonated shell and a non-carbonated core. Therefore,  $f_{CC}$  is the comprehensive compressive strength of carbonated parts and non-carbonated parts after the carbonation of concrete.

The effect of carbonation on the compressive properties of cement-based material cannot be accurately calculated by Eq. (1). To calculate the compressive strength of concrete cubes after carbonation more accurately, the compressive strength of concrete was defined comprehensively in Eq. (2).

$$f_{cc} = \frac{f_{co}A_{co} + f_{cc}A_{cc}}{A_{co} + A_{cc}}$$
(2)

where  $f_{CC}$  is the compressive strength of fully carbonated concrete cubes with the unit of MPa.  $A_{CO}$  is the area of the non-carbonated zone with the unit of m<sup>2</sup> and the value was obtained by the calculation of the area circled by the red curve as shown in Figs. (5-7);  $A_{CC}$  is the sectional area of the fully carbonated shell with the unit of m<sup>2</sup> and the value was obtained by the difference of the sectional area of the cube and  $A_{CO}$ .

325 It is worth emphasizing that the compressive strength of fully carbonated concrete,  $f_{CC}$ , can be calculated 326 according to Eq. (2) and the other parameters can be obtained from the test results. The compressive strength 327 of SPFRC without and after carbonated were obtained and shown in Table 6.

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Table o Complessive suchgul of SITIKC	Table 6	Compressive	strength	of SPFRC
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Specimens number	fco (MPa)	fcc (MPa)	fcc/fco	$A_{CO} \times 10^{-3} (\mathrm{m}^2)$	$A_{CC} \times 10^{-3} \text{ (m}^2\text{)}$	fcc' (MPa)	fcc'/fco
SPFRC0000(60)	39.6	55.8	1.41	2.478	7.522	61.1	1.54
SPFRC0500(60)	37.6	49.5	1.32	1.221	8.779	51.2	1.36
SPFRC1000(60)	33.4	46.1	1.38	1.428	8.572	48.2	1.44
SPFRC1500(30)	36.2	45.8	1.27	1.820	8.180	47.9	1.32
SPFRC1500(60)	34.3	56.4	1.64	2.045	7.955	62.1	1.81
SPFRC1500(80)	39.6	52.8	1.33	1.515	8.485	55.2	1.39

SPFRC0005(60)	35.0	49.4	1.41	1.756	8.244	52.5	1.50
SPFRC0505(60)	37.5	47.7	1.27	1.999	8.001	50.2	1.34
SPFRC1005(60)	40.9	51.8	1.27	2.367	7.633	55.2	1.35
SPFRC1505(60)	35.3	52.2	1.48	2.015	7.985	56.5	1.60
SPFRC0010(60)	41.1	53.3	1.30	1.993	8.007	56.3	1.37
SPFRC0510(60)	35.6	47.7	1.34	1.555	8.445	49.9	1.40
SPFRC1010(60)	38.8	51.2	1.32	1.829	8.171	54.0	1.39
SPFRC1510(30)	41.9	54.3	1.30	1.862	8.138	57.1	1.36
SPFRC1510(60)	36.1	55.1	1.53	1.324	8.676	58.0	1.61
SPFRC1510(80)	31.2	46.1	1.48	1.298	8.702	48.3	1.55
SPFRC0015(60)	24.9	38.7	1.55	1.505	8.495	41.1	1.65
SPFRC0515(60)	24.3	30.8	1.27	1.187	8.813	31.7	1.30
SPFRC1015(60)	24.7	29.7	1.20	0.872	9.128	30.2	1.22
SPFRC1515(60)	22.9	30.3	1.32	0.369	9.631	30.6	1.34

330 As shown in Table 6. The compressive strength value of fiber reinforced concrete varies with the 331 variation of volume fraction of steel fibers, volume fraction of polypropylene fiber, and length-diameter ratio 332 of steel fiber. The ultimate compressive strength of SPFRC decreased with the increase of the addition of steel 333 fibers, polypropylene fibers, and the length-diameter ratio of steel fiber before the supercritical carbonation. 334 This may be attributed to the increase of ITZ between the fibers and matrix with the addition of fibers in SPFRC<sup>[8]</sup>. However, the ultimate compressive strength of SPFRC increased by 22% to 81% after the 335 336 supercritical carbonation. This may be attributed to the micro-pores of the matrix and ITZ were filled with the calcium carbonate after the carbonation reaction of fiber reinforced concrete, which makes the composite more 337 compacted and increases the fiber-matrix bond [39]. Thus, the ultimate compressive strength of SPFRC 338 339 increases to some extent. The compressive strength of concrete is affected by supercritical carbonation. The 340 value of the relative compressive strength of concrete is greater than 1. The compressive strength of concrete 341 has been greatly improved after carbonation. The compressive strength and the relative compressive strength 342 of carbonated parts are greater than the overall compressive strength and overall relative compressive strength.

343

344 *3.4.2. Establishment of mathematical model for the supercritical carbonation of SPFRC* 

The effect of the initial porosity of hybrid fiber reinforced concrete on the carbonation depth was analyzed in this section. The initial porosity of different hybrid fiber concrete was calibrated through the comparison of test and numerical average carbonation depth. A one-dimensional mathematical model for the physical-chemical coupling process of supercritical carbonation of cement-based materials was established. The change of average carbonation depth over time in the process of supercritical carbonation of concrete was simulated by using multi-physics coupling simulation software. The theoretical model is validated by the test results.

The governing equations for the supercritical carbonation of SPFRC are shown in Eqs. (3-7). The rate of chemical reaction, mass conservation for gas-liquid two phase flow, diffusion and dispersion of  $CO_2$  in water, energy conservation for porous medium, and the solubility of  $CO_2$  in water are all considered in the mathematical model.

$$\frac{\partial R_c}{\partial t} = \alpha_1 \times f_1(h) \times f_2(g_v) \times f_3(R_c) \times f_4(T)$$
(3)

$$\frac{\partial(g)}{\partial t} = \frac{\partial(m_{co_2})}{\partial t} \tag{4}$$

$$\frac{\partial(nS_{\alpha}\rho_{\alpha})}{\partial t} + \nabla \cdot (\rho_{\alpha}\vec{u}_{\alpha}) = q_{\alpha}$$
(5)

$$\vec{u}_{\alpha} = -\frac{k_0 \left(\frac{n}{n_0}\right)^3 \cdot \left(\frac{1-n_0}{1-n}\right)^2 k_{r\alpha}}{\mu_{\alpha}} (\nabla P_{\alpha} - \rho_{\alpha} \vec{g})$$
(6)

$$\left(\rho C_{q}\right)_{eff}\frac{\partial T}{\partial t} = \nabla \cdot \left(k_{eff}\nabla T\right) - \left(C_{g}\rho_{g}\vec{u}_{g} + C_{w}\rho_{w}\vec{u}_{w}\right)\nabla T$$

$$\tag{7}$$

where  $R_c$  is the degree of carbonation; g is the mass concentration of CO<sub>2</sub> in water; n is the porosity of material that decreases during carbonation;  $P_{\alpha}$  is the pressure of phase  $\alpha$ ; subscript  $\alpha$  refers to w for the liquid phase and g for the gaseous phase. The detailed information about the other parameters can be found in Zha<sup>[41]</sup> and Yu [45].

The initial and boundary conditions were applied to solve the governing equations. In this study, the conditions are introduced and shown in Eqs. (8-10).

$$R_{c} = R_{c0} = 0, \ P_{g} = P_{g0}, \ P_{w} = P_{w0}, \ g = g_{0} = 0, \ T = T_{0}, \ t = 0 \ on \ \Omega$$
(8)

$$\vec{n} \cdot \nabla R_c = 0, \ \vec{n} \cdot \nabla g = 0 \ on \ \Gamma_2$$
 (9)

$$P_g = P_{g,sur}, P_w = P_{w,sur}, T = T_{sur} on \Gamma_1$$
(10)

where  $R_{c0}$  is the initial conditions specifying the degree of carbonation;  $P_{g0}$  and  $P_{w0}$  are the initial water pressure and initial gas pressure, respectively;  $g_0$  is the initial concentration of dissolved CO<sub>2</sub> in water;  $T_0$  is the initial temperature;  $\vec{n}$  is the normal vector of the boundary;  $\Gamma_2$  and  $\Gamma_1$  are the boundary using Neumann's conditions and Dirichlet's conditions, respectively;  $P_{g,sur}$  and  $P_{w,sur}$  are the surrounding gas and liquid pressure, respectively;  $T_{sur}$  is the surrounding temperature. The detailed information about the other parameters can be found in Zha <sup>[41]</sup> and Yu <sup>[45]</sup>.

368 The cement-based materials can be considered as a porous medium. The transformation of carbon dioxide 369 in the pore and the ultimate carbonation results are affected by its related material physical parameters, such as initial porosity, intrinsic permeability rate, and initial saturation. The physical characteristic parameters of 370 371 cement-based materials in the numerical model are evaluated and shown in Table 7. The control equations, initial conditions, and boundary conditions are given <sup>[41, 45]</sup>. According to the test procedure, the total 372 373 carbonation time is 7.17 hours and the supercritical carbonation time is 6 hours. The recorded temperature and 374 pressure of SPFRC specimens during the supercritical carbonation process were applied in the numerical 375 carbonation model.

376

Table 7 Parameters for SPFRC.

Porous porosity	Value	References
Intrinsic permeability, $k_0$	10×10 <sup>-21</sup> m <sup>2</sup>	[59]
Initial average porosity, $n_0$	0.13	[59]
Capillary pressure curve coefficient, $\alpha$	5.3695×10 <sup>-8</sup> Pa <sup>-1</sup>	[60]
Relative permeability coefficient, m	0.4396	[60]
Relative humidity, $h_0$	0.684	

378 As the porosity of concrete range from 0.09 to 0.21 <sup>[61]</sup>, the simulation results of average carbonation depth of SPFRC under supercritical condition with the assumed initial porosity of 0.12, 0.13, 0.14, 0.15, 0.16, 379 0.17, 0.18, 0.19, 0.20 were calculated as 23.02 mm, 25.26 mm, 27.5 mm, 29.74 mm, 31.98 mm, 34.22 mm, 380 36.46 mm, 38.7 mm and 40.94 mm, respectively. The test results of the average carbonation depth of the 20 381 382 kinds of mixed steel-polypropylene hybrid fiber reinforced concrete under supercritical condition were 383 compared with the numerical results. After the comparison and analysis of experimental and numerical 384 carbonation depth of SPFRC under supercritical carbonation, the value of the initial porosity of hybrid fiber concrete can be estimated by using linear interpolation. The estimated value of the equivalent porosity was 385 obtained and shown in Table 8. 386

2	0	
э	o	1

**Table 8** Estimated value of equivalent initial porosity.

Specimens number	Estimated value of equivalent initial porosity, $n_0$	Specimens number	Estimated value of equivalent initial porosity, $n_0$
SPFRC0000(60)	0.122	SPFRC0010(60)	0.129
SPFRC0500(60)	0.123	SPFRC0510(60)	0.142
SPFRC1000(60)	0.126	SPFRC1010(60)	0.147
SPFRC1500(30)	0.142	SPFRC1510(30)	0.133
SPFRC1500(60)	0.133	SPFRC1510(60)	0.154
SPFRC1500(80)	0.151	SPFRC1510(80)	0.155
SPFRC0005(60)	0.123	SPFRC0015(60)	0.145
SPFRC0505(60)	0.136	SPFRC0515(60)	0.162
SPFRC1005(60)	0.132	SPFRC1015(60)	0.170
SPFRC1505(60)	0.143	SPFRC1515(60)	0.194

388

389 3.4.3. Relationship between carbonated compressive strength and equivalent initial porosity of SPFRC

In this section, the experimental supercritical carbonation depth of SPFRC in Table 5 was compared with the numerical results to calibrate the equivalent initial porosity of the 20 groups of SPFRC specimens. Then the relationship between carbonated compressive strength and the equivalent initial porosity of SPFRC was obtained.

394 The compressive strength of concrete was affected by the change of pore structure after the carbonation 395 treatment. The molar volume of calcium carbonate is larger than that of calcium hydroxide after carbonation, 396 and the solubility is smaller than that of calcium hydroxide in water. The porosity was reduced, the density and 397 compressive strength of concrete increased after the deposition of calcium carbonate in pores. Then the 398 relationship between the equivalent porosity without the carbonation of SPFRC and the compressive strength 399 of full carbonation were studied in this section. A mathematical model for the relationship between the 400 equivalent initial porosity and the compressive strength of SPFRC was established. The steps of proposing the 401 mathematical model can be summarized as follows. Firstly, the compressive strength of fully carbonated 402 concrete cubes was estimated according to Eq. (2). The one-dimensional mathematical model for the 403 physical-chemical coupling process of supercritical carbonation of cement-based materials was established. 404 Then the equivalent initial porosity was calculated by linear interpolation according to the comparison of 405 carbonation depth of experiment and numerical simulation. Finally, the mathematical model for the relationship between the compressive strength of SPFRC and equivalent initial porosity was proposed as 406 407 shown in Eq. (11).

408

The relationship between the compressive strength of concrete and porosity of concrete was proposed by

409 the regression analysis <sup>[62]</sup> based on the mathematical model <sup>[63]</sup> as shown in the following equation:

$$f_C = f_{C0} \times \left(1 - kn_0\right) \tag{11}$$

410 where  $f_C$  is the fitted value of compressive strength when the initial porosity is  $n_0$ ,  $f_{C0}$  is the value of 411 compressive strength when the initial porosity is 0, *k* is a coefficient to be determined,  $n_0$  is the equivalent 412 initial porosity.

413 The relationship between the compressive strength of concrete without carbonation and porosity of 414 concrete was obtained by the regression analysis of the estimated value of equivalent initial porosity in Table 8 415 and the comprehensive compressive strength of specimens without carbonation,  $f_{CO}$ , in Table 6, as shown in 416 Eq. (12).

$$f_c = 82.5 \times (1 - 4.07n_0) \tag{12}$$

417 in which,  $f_C$  is the fitted value of compressive strength when the initial porosity is  $n_0$  without carbonation.

418 The regression analysis of the compressive strength of SPFRC after the full carbonation and equivalent 419 initial porosity was performed by the estimated value of equivalent initial porosity in Table 8 and the 420 compressive strength of fully carbonated concrete,  $f_{CC}$ , in Table 6, as shown in Eq. (13).

$$f_{c}' = 119.4 \times (1 - 4.07n_{0}) \tag{13}$$

421 in which,  $f_{C}$  is the fitted value of the compressive strength of SPFRC after full carbonation when the initial 422 porosity is  $n_0$ .

The comparison of the calculated results by Eq. (12), Eq. (13), and the test results are shown in Fig. 14. As shown in Fig. 14, Eq. (12) and Eq. (13) can be used to describe the relationship between the compressive strength without and after the full carbonation and initial equivalent porosity of SPFRC, respectively. There's a linear relationship between the equivalent initial porosity and compressive strength without carbonation and after full carbonation.



Fig. 14. Regression curves of equivalent initial porosity and compressive strength.

428

## 429 **4. Conclusions**

430 In this paper, the supercritical carbonation of steel-polypropylene hybrid fiber reinforced concrete was

431 studied. The main conclusions are as follows:

- (1) The compressive strength after carbonation is significantly increased by at least 20%. The maximum
  relative compressive strength was obtained when the volume fraction of steel fiber and polypropylene fiber
  was 1.5% and 0.0% and the length-diameter ratio of steel fiber was 60, respectively.
- 435 (2) An evaluation formula was proposed to estimate the compressive strength of fully carbonated436 concrete.

(3) The equivalent initial porosity of SPFRC can be evaluated by the mathematical model of compressivestrength after fully carbonated and the equivalent initial porosity of SPFRC.

(4) Further experiments will be carried out to investigate the effects of supercritical carbonation on the
 porosity of SPFRC and the ITZ between the fibers and matrix. The post cracking behavior of the composite
 and the mechanical properties are to be evaluated.

442

### 443 **Data Availability Statement**

444 Some or all data, models, or code that support the research findings of this study are available from the 445 corresponding author by reasonable request, including the experimental and numerical data.

446

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