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Methods study on dispersion of fibers in CFRC

Yuanxia Yang*

Civil Engineering College, Central South University, 22 Shaoshan South Road, Changsha, Hunan 410075, China

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Abstract

Through the study of the effect of MKC (a fiber dispersant) on the dispersion of carbon fibers in cement, this paper presents four methods of characterizing fiber dispersion. The fresh mixture method (FM method), which can show the degree of fiber breakage during mixing, is an efficient means to compare fiber dispersion in different fresh mixtures. The scanning electron microscope method (SEM method) is adequate to analyze the microstructure of composite. The measurement of electrical resistance (ERM method) can indirectly suggest the fiber dispersion of composite. The simulation experiment (SE method) is the best method to determine the effect of various fiber dispersion agents. Different methods are fit for different conditions and they can also be used together to gain the true dispersion of fibers.

Keywords: Carbon fiber; Electrical resistance; Dispersion; Method

1. Introduction

With the development of relatively low-cost, pitch-based carbon fibers in recent years, the commercialization of short carbon fiber-reinforced cement (CFRC) has become possible. Reinforcement with carbon fibers can lead to great improvement in flexural strength and toughness, tensile performance, impact resistance and durability of cementitious materials [1–8]. In addition, there has been an increasing interest in use of CFRC as functional sensibility [9–12]. The effectiveness of the fiber added depends on the degree of fiber dispersion. Many previous works were focused on the improvement of fiber dispersion [4,13,14]. Electrical resistivity measurement has been used to assess the degree of steel fiber dispersion [15], but comparison of different methods has not been reported. This paper introduces four methods—the fresh mixture method (FM method), the measurement of electrical resistance (ERM method), the scanning electron microscope method (SEM method) and the simulation experiment (SE method). Furthermore, the characteristics of every method are illustrated and the methods are experimentally validated.

2. Methods and theory

2.1. FM method

Firstly, to get some pieces of mixture of about the same weight from different areas of fresh CFRC, wash the fibers clean, dry up and weigh them, then calculate the content of carbon fiber. Standard deviation (S),variation coefficient (Ψ) and dispersion coefficient (β) are calculated as follows:

$$\beta = e^{-\Psi(X)} \tag{1}$$

$$\Psi(X) = \frac{S(X)}{\overline{X}} \times 100\% \tag{2}$$

$$S(X) = \sqrt{\frac{\sum_{i=1}^{n}(X_i - \overline{X})^2}{n-1}} \tag{3}$$

where X_i = carbon fiber content of sample i (g/100 g cement); $\bar{X}$ = average fiber content of the mixture (g/100 g cement); n = number of samples; β = dispersion coefficient of carbon fiber; $S(X)$ = standard deviation; $\Psi(X)$ = variation coefficient. In accordance with Eqs. (1)–(3) , if carbon fibers are uniformly dispersed or X_i is the same (from $i=1$ to $i=n$), then $\Psi(X)=0$ and $\beta=1$. If carbon fibers are in one

* Tel.: +86-731-558-5211x74784; fax: +86-731-557-1736.
E-mail address: bjliu@crsu.edu.cn (Y. Yang).

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sample only, then $\Psi(X) \to \infty$ and $\beta \to 0$. Therefore, at normal conditions, $\Psi(X)$ is always over zero and β varies from 0 to 1. And the less $\Psi(X)$ is or the higher β is, the better dispersed the fibers are.

It is quick, simple and applicable to determine the dispersion of fibers in CFRC. To some degree, the dispersion of carbon fibers can be determined by β. In addition, it can give the degree of fiber breakage influenced by mixing [2]. It is useful to determine the applicable mixing process, but fails to suggest the fiber dispersion in any of the samples. The result is influenced by the number of samples and the weight of each sample, and cannot respond to fiber dispersion in hardened CFRC because time of vibration may lead to change of fiber dispersion more or less.

2.2. SEM method

The dispersion of fibers, whether they are dispersed one by one, is observed by a SEM and by using hardened samples.

It is intuitionistic and in point to analyze the dispersion of fibers in small area and is also applicable for CFRC of any types. The result is actual but limited by the number of sample and the area of sampling. It cannot reflect the macroscopical dispersion of fibers.

2.3. ERM method

Electrical resistance of different groups of specimens was measured, then the average resistivity and Ψ were calculated, respectively. For specimens of the same fiber fraction, the carbon fibers may be dispersed the best in which the electrical resistivity and Ψ are the lowest, respectively. This is because carbon fibers are more conductive than the hardened cement matrix [16]. In case of carbon fibers at a volume fraction below the percolation threshold, the electrical conductivity of the composite is highly dependent on the degree of fiber dispersion. The greater the degree of fiber dispersion is, the higher the conductivity of the composite is. At the same time, the resistivity of CFRC is also sensitive to the air void content and the fiber–matrix interface. Although the electrical conductivity of composite decreases with the increase of the air void content or the poor fiber–matrix interface structure, the variation coefficient must be low while the fibers are dispersed uniformly. On the contrary, if the variation coefficient is high, whether the electrical resistivity is high or not, conclusion, as carbon fibers were not well dispersed in CFRC, can be drawn.

Table 1
The properties of carbon fibers

Item	Diameter (μm)	Tensile strength (GPa)	Tensile modulus (GPa)	Resistivity (Ω/m)
Index	7 ± 0.2	≥ 1.95	≥ 175	310

Table 2
Mix proportions of CFRC (wt.%)

Mix number	Fiber/ cement	Water/ cement	Dispersant/ cement
A-1	0.5	30	0
A-2	0.5	30	0.5

The result of ERM method provides a relative indication of degree of fiber dispersion. It costs less than SEM analysis does. However, it cannot tell the actual dispersion of fibers in any of the specimens. Factors, such as structure of cement paste, content and length of fibers, may lead to great change of electrical resistivity. To determine the dispersion of carbon fibers influenced by mixing or molding, the other factors must be changeless. The result is not very reliable. If the true dispersion of fibers is required, whether it is macroscopically or microcosmic, this method may be used together with other methods.

2.4. SE method

The fiber dispersion agent is first dissolved in water in a glass beaker. Then the short carbon fibers are added and stirred by hand. Through glass, the dispersion of carbon fibers can be seen clearly. In this experiment, the dispersion of carbon fibers, whether they are dispersed singly or not, can be well observed. It economizes a significant amount of raw materials and time while determining a fiber dispersant. It can only represent dispersion of fibers in fresh CFRC and cannot represent the dispersion of fibers in hardened CFRC, if molding changed the dispersion of fibers.

3. Experiment

3.1. Materials and experimental methods

The carbon fibers used were short (nominally 5 mm in length) and PAN-based, as provided by Shanghai Carbon Ltd. Co. (Shanghai, China). Their properties are shown in Table 1. The matrix was 425 Portland blast-furnace slag cement. The dispersion agent used was homemade and named MKC and its main component is cellulose.

Table 2 shows the proportions of CFRC as to validate the FM, SEM and ERM methods. The additions were dissolved in water and carbon fibers were added and stirred by hand

Table 3
The result of ERM method

	I		II		III	
Number	ρ (kΩ cm)	Ψ (%)	ρ (kΩ cm)	Ψ (%)	ρ (kΩ cm)	Ψ (%)
A-1	14.12	81.0	8.25	125.8	6.83	58.0
A-2	16.02	2.6	13.90	47.7	12.35	17.0

Table 4
Result of FM method

Mixing time (min)		1	2	3	4	5	6	7	8
β	A-1	0.9397	0.9613	0.8758	0.8680	0.8251	0.8449	0.8695	0.8777
	A-2	0.9666	0.9798	0.9186	0.8765	0.8788	0.9043	0.9070	0.9290
Ψ (%)	A-1	6.22	3.95	13.26	14.16	19.22	16.85	13.98	13.04
	A-2	3.40	2.04	8.49	13.18	12.92	10.06	9.76	7.36

for about 2 min. Then this mixture and cement were mixed in a cement mixer for 1–8 min and eight pieces of sample were used every time as to investigate the effect of mixing time on fiber dispersion by FM method. For the purpose of comparing ERM and SEM methods, the mixture was poured into molds (40×40×40 mm) after mixing for 2 min. Then a vibrator was used to decrease the amount of air bubbles. The specimens were demolded after 1 day and then cured at room temperature in water for 28 days. For each type of experiment, six specimens were tested. For SE method, two glass beakers were used. Small amounts of fibers were added into water with MKC and without MKC, respectively, to observe fiber dispersion while and after stirring.

3.2. Measurement of electrical resistance

The electrical resistance was measured in three different conditions. (I) The specimens were air-dried after 28-day curing. The DC volume electrical resistance was measured using two graphite electrodes embedded in it and a known low voltage. (II) The specimens were air-dried after 28-day curing. Then two copper electrodes were stuck on the opposite surface of each specimen by a graphite-conductive adhesive. The resistance was read by a direct-reading digital resistance meter. (III) Seven days after measurement of (II), the current across the same specimen was measured by applying a known higher voltage than that of (I) and (II). Then the electrical resistivity (ρ) and variation coefficient (Ψ) were calculated as follows (Eqs. (4) and (5)):

$$\rho = \frac{RS}{l} \tag{4}$$

$$\Psi = \frac{\sqrt{\sum_{i=1}^{n} \frac{(\rho_i - \overline{\rho})^2}{n-1}}}{\overline{\rho}} \times 100\% \tag{5}$$

where R = electrical resistance (kΩ); S = area of electrodes (cm^2); l = space between two electrodes of a specimen (cm); ρ_i = electrical resistivity of the number i specimen; $\overline{\rho}$ = the average of electrical resistivity of specimens measured in a same condition; n = number of specimens measured in a same condition. Because the DC current was so small that polarization can be neglected, the two- and four-probe methods gave the same results.

4. Results and discussion

Table 3 gives the results of ERM method. For any of the conditions, the electrical resistivity of A-2 is higher than that of A-1 and the variation coefficient is lower than that of A-1. This suggests that MKC is helpful in improving the dispersion of fibers. The fact that the electrical resistivity of A-2 is higher than that of A-1 may due to the higher air content or the change of fiber–matrix interface resulting from the addition of MKC.

Table 4 shows the results of FM method. The β increased during the first 2 min and decreased during the later 3 min. Then β increased slightly again for each type of mixture. The β of mixture with MKC was higher than that of mixture without MKC as for any of the same mixing time. Relationship between Ψ and mixing time is similar to that of β, except that the decrease in β was increase in Ψ and the increase in β was the decrease in Ψ. This may due to fibers being dispersed, conglomerated and then broken and dispersed a little better [2]. In addition, the result suggests that MKC has improved the dispersion of fibers.

Table 5 shows the results of SE method. Carbon fibers were dispersed singly only in water containing MKC. This is due to that MKC can soak carbon fibers well and disperse them by single thread. Furthermore, MKC can avoid the assembling of carbon fibers by forming a layer of film on the surface of fibers. The results indicated that MKC is efficient to make uniform dispersion of carbon fibers.

Results of SEM method were shown in Ref. [17]. There are many biggish pores in specimens of A-2, while the structure of A-1 is more compact. At the same time, carbon fibers were dispersed singly in specimen A-2 but fasciculi

Table 5
Result of SE method

Mixture	State at beginning	Add shot fibers and stir for 2 min	Two minutes after stirring
Water	colorless and transparent	single fiber and fasciculus exists	fibers deposit at the bottom of beaker
Water containing MKC	colorless and sticky	fibers exist single by single	fibers deposit slightly

exist in A-1. It explains why electrical resistivity of A-2 is higher than that of A-1, and substantiates that MKC can truly increase the dispersion of fibers.

5. Conclusion

All methods introduced here are useful in evaluating fiber dispersion in cement. Each of them has its characteristics and is fit for different conditions. SE method is the best in comparing various fiber dispersants. In addition, FM method is a better choice while a better mixing or molding technology or a better dispersant is being chosen. Though it is difficult to gain some comprehensive macrographic information, the result of SEM method can give the actual micrograph of fiber dispersion. With all other factors being the same, the ERM method may be used to determine a better mixing time or mixing sequence, but the result is less accurate. The best method must be selected according to the matter of fact or several methods may be hung together to get the actual result.

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