# MIXING SEQUENCE EFFECT OF CEMENT COMPOSITES WITH CARBON FIBRES

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**ABSTRACT:** Carbon fibres are widely recognised as reinforcement materials that effectively control cracks in concrete structures. Nonetheless, these fibres do not disperse uniformly inside the cement matrix, disrupting the mixture homogeneity. To address this concern, this study investigated two distinct mixing sequences of cement composites with carbon fibres. Two mixing sequences were investigated including the addition of fibres after cement (AC-CF) and the addition of fibres before cement (BC-CF). The surface topography of carbon fibres and the engineering properties of the cement paste were also examined. Consequently, carbon fibres in cement composite produced lower flowability due to the surface roughness. The AC-CF specimen demonstrated the highest hardened density at 28 days with 2679.22 kg/m<sup>3</sup> followed by BC-CF and the control specimen with 2386.08 kg/m<sup>3</sup> and 2278.36 kg/<sup>3</sup>, respectively. The AC-CF specimen also had the highest compressive strength at 28 days with 69.91 MPa, followed by BC-CF and the control specimen with 65.92 MPa and 63.20 MPa, respectively. Further, the flexural strength of the AC-CF specimen exhibited the highest strength with 10.86 MPa, followed by BC-CF and the control specimen with 9.35 MPa and 9.17, respectively. The fibre dispersion in AC-CF was also superior to BC-CF. Therefore, it can be concluded that the best mixing sequence is the addition of fibre after cement (AC-CF) because it had better fibre dispersion and engineering properties compared to the addition of fibre before cement (BC-CF).

ABSTRAK: Gentian karbon lebih dikenali sebagai bahan bantuan yang berkesan dalam mengawal keretakan pada struktur konkrit. Walau bagaimanapun, gentian ini tidak tersebar secara seragam di dalam matrik simen dan akan mengganggu kehomogenan campuran. Bagi mengatasi masalah ini, kajian ini mengkaji tentang dua susunan campuran berbeza simen komposit dengan gentian karbon. Dua susunan campuran ini adalah melalui penambahan gentian selepas simen (AC-CF) dan penambahan gentian sebelum simen (BC-CF). Permukaan topografi gentian karbon dan sifat kejuruteraan pes simen turut diperiksa. Kajian mendapati bahawa gentian karbon dalam komposit simen mengurangkan kebolehaliran pes simen disebabkan oleh kekasaran pada permukaan gentian. Spesimen AC-CF menunjukkan ketumpatan pengerasan tertinggi pada hari ke-28 dengan 2679.22 kg/m<sup>3</sup> diikuti spesimen BC-CF dan spesimen kawalan sebanyak 2386.08 kg/m<sup>3</sup> dan 2278.36 kg/m<sup>3</sup>, masing-masing. Spesimen AC-CF juga mempunyai kekuatan mampatan tertinggi pada hari ke-28 dengan 69.91 MPa, diikuti oleh spesimen BC-CF dan spesimen kawalan sebanyak 65.92 MPa dan 63.20 MPa, masing-masing. Seterusnya, kekuatan lenturan spesimen AC-CF menunjukkan kekuatan tertinggi dengan 10.86 MPa, diikuti spesimen BC-CF and spesimen kawalan dengan 9.35 MPa dan 9.17 MPa, masing-masing. Penyebaran gentian dalam AC-CF juga lebih baik daripada BC-CF. Oleh itu, kajian ini merumuskan bahawa susunan campuran terbaik adalah dengan penambahan gentian selepas simen (AC-CF) kerana ia mempunyai kekuatan lenturan

gentian terbaik dan sifat kejuruteraan berbanding penambahan gentian sebelum simen (BC-CF).

KEYWORDS: mixing sequence; cement composite; carbon fibre; engineering properties

### 1. INTRODUCTION

Fibres are a fibrous material type commonly employed to improve the properties of concrete. Moreover, fibres mitigate cracks from plastic with drying shrinkage and restrict the permeability of concrete [1]. Fibres have been widely used in several applications, including pavements, bridge decks, offshore structures, and machine foundations. Generally, these fibres are categorised into three distinct types: steel, glass, and synthetic [2]. Carbon fibres are widely recognised for their favourable elastic modulus, tensile strength, and thermal and electrical conductivities [3]. When fibres are added to concrete, several mechanical properties are enhanced [4]. These properties include compressive strength, flexural strength, tensile strength, durability, and cracking resistance. Nevertheless, multiple parameters, including fibre percentage, diameter, and length, can affect these characteristics.

Numerous studies demonstrated that gradually adding carbon fibre into concrete reduced workability [5-8]. This outcome occurred due to the fresh concrete movement obstruction from the fibre interaction with other concrete constituents [9]. Previous studies also demonstrated a positive correlation between the higher fibre percentage and fibre length in improving the compressive and flexural strength of concrete [6-12]. These improved concentre properties were attributed to the fibres, serving as a bridge to maintain the proximity of concrete particles [13]. Nonetheless, lower concrete strength was presented when the fibre percentage was 1.0% due to fibre agglomerations [10,12].

One of the primary concerns associated with fibres is their tendency to aggregate, leading to non-uniform dispersion within the cement matrix and lower strength. Conversely, the fibre mixing sequence can impact their dispersion inside the matrix. According to the American Concrete Institute (ACI) 6.44-3R, fibres should be incorporated into a fluid mixture in the final mixing stage or added to the mixer with aggregates [14]. Several studies have supported this reasoning, recording enhanced fibre dispersion within the matrix when the fibres are introduced into a fluid mix [15-18]. To the authors' knowledge, the correlation between the fibre dispersion inside the hardened cement matrix and their engineering properties has not been explored. Thus, this study focused on the investigation of synthetic carbon fibres.

This study evaluated the surface topography of carbon fibres. A carbon fibre blending sequence assessment was conducted inside a cement paste based on several engineering properties, including flowability, hardened density, compressive strength, and flexural strength. This study investigated two different mixing sequences: fibres added after cement (AC-CF) and fibres added before cement (BC-CF). The surface topographies of the specimens were used to compare the fibre dispersions in the hardened AC-CF and BC-CF. Finally, the optimal mixing sequence of a cement composite with carbon fibres was identified.

## 2. MATERIALS AND METHODS

#### 2.1 Materials

The cement composite utilised in this study consisted of Ordinary Portland Cement (OPC), water, and carbon fibres (see Fig. 1). Three specimens were assessed in this study: cement paste without fibres (control), AC-CF, and BC-CF. The AC-CF denotes fibres added after cement,

while BC-CF was defined as fibres added before cement. Table 1 tabulates the mixture proportions of the specimens. For example, 1014 g of AC-CF contained 778 g of cement, 233 g of water, and 3 g of carbon fibres.



Fig. 1: The (a) OPC, (b) water, (c) carbon fibre components used in this study.

Specimen	Water/Cement Ratio	Cement (g)	Water (g)	Fibre Content (g)
Control	0.30	778	233	0
AC-CF	0.30	778	233	3
BC-CF	0.30	778	233	3

Table 1: Summary of specimen designations and mixture proportions

The methodology employed for the control specimen adhered to the American Society for Testing and Material (ASTM) C305 guidelines [14]. Meanwhile, the specimen preparation containing carbon fibres was conducted following the method described by Gao et al. [15]. Figure 2 depicts the mixing sequences and duration methodologies for all specimens.



Fig. 2: Schematic flow chart indicating the mixing methods for (a) control, (b) AC-CF, and (c) BC-CF specimens.

### 2.2 Methodology

This study involved the surface topography acquisition of carbon fibres using Atomic Force Microscopy (AFM) and Field Emission Scanning Electron Microscopy (FESEM). Both experiments were conducted at the Centre for Instrumentation and Science Services, Universiti Malaysia Sabah (UMS). The carbon fibre dispersion in hardened cement composite was also achieved using Scanning Electron Microscopy (SEM). These characterisations were performed at the Biotechnology Research Institute, UMS. Figure 3 illustrates the instruments employed in this study.



Fig. 3: The (a) AFM, (b) FESEM, and (c) SEM instruments used to examine the carbon fibre properties.

Approximately 54 cement-paste specimens were fabricated, of which 36 specimens (50 mm  $\times$  50 mm  $\times$  50 mm) were utilised for hardened density and compression strength tests. An additional 18 specimens (40 mm  $\times$  40 mm  $\times$  160 mm) were applied for flexural strength tests. The mixing procedure was initially performed using a mechanical mixer, following the guidelines outlined in ASTM C305 [19]. The cement paste was then carefully introduced into the cube and the prism moulds and allowed to cure for one day. Subsequently, the specimens were demoulded and cured through water tank immersion for 7, 28, and 56 days. The flowability, hardened density, compressive strength, and flexural strength of the cement composite were evaluated using standardised test methods, including ASTM C1437, BS EN 1015-10, ASTM C109, and ASTM C348, respectively [20-23]. Alternatively, the flowability, hardened density, and compressive strength tests were conducted in the Concrete Lab and Material, UMS. The flexural strength test was also performed at the Faculty of Tropical Forestry, UMS. Figure 4 portrays the instrumentation employed in the study.



Fig. 4: The (a) flowability, (b) hardened density, (c) compressive strength, and (d) flexural strength test instruments used to test the engineering properties of the cement composites.

# 3. RESULTS AND DISCUSSION

### 3.1 Surface Topographies of the Carbon Fibres

Figure 5(a) and 5(b) present the surface topographies of carbon fibres using FESEM at low and high magnifications, respectively. The diameters varied from 36  $\mu$ m to 40  $\mu$ m, while extrusion lines along the longitudinal axis of the carbon fibre (red circle) indicated a smooth surface.



Fig. 5: The FESEM topographies of carbon fibres at (a) low and (b) high magnifications.

The FESEM images were compared with the AFM images (see Fig. 6). A distinct presence of extrusion lines on the fibre surface was observed, suggesting the low fibre roughness. Moreover, a discernible hairy structure in the fibres was demonstrated. A study by He and Yang proposed that this characteristic could improve the interfacial binding strength between the fibre surface and the surrounding cement-paste matrix by introducing additional Van der Waals forces [24].



Fig. 6: The AFM topography of the carbon fibres indicating the (a) extrusion lines on the surface and (b) the hairy texture.

### 3.2 Flowability Values of the Cement Composites

Table 2 lists the flow diameters of the control and cement composites. The results indicated that the control specimen exhibited the largest flow diameter, followed by the AC-CF and BC-CF specimens. These measurements were 205 mm, 204 mm, and 202 mm, respectively. According to the ASTM standard [20], all diameters fell within the permitted range from 205 mm to 215 mm (105% to 115%). The AC-CF and BC-CF produced lower diameters due to the carbon fibre roughness, which was proven by the FESEM and AFM images. These features

were widely recognised to significantly impact the fibre dispersion within the cement matrix [25]. Therefore, the flowability of the matrix was restricted.

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Specimen	Control	AC-CF	BC-CF
Flow diameter (mm)	205	204	202

Table 2: Summary	of the	flow	diameters	for	the specimens
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#### **3.3 Hardened Densities of the Cement Composites**

A higher hardened density in cement composites is vital for proper fibre dispersion. If the fibres are not adequately dispersed, their ability to occupy the vacant spaces is compromised. This process develops a lower hardened material density [26]. Consequently, the hardened densities of all samples presented an upward trend with increased curing duration (7, 28, and 56-day periods) (see Fig. 7). The results also demonstrated that the hardened density of the AC-CF specimen was higher than the BC-CF and control specimens at all curing durations. For example, the measured hardened densities of the AC-CF, BC-CF, and control specimens on day 56 were 2679.22 kg/m<sup>3</sup>, 2386.08 kg/m<sup>3</sup>, and 2278.37 kg/m<sup>3</sup>, respectively.



Fig. 7: The hardened densities of various specimens.

#### 3.4 Compressive Strengths of the Cement Composites

Figure 8 presents the compressive strengths of the control, AC-CF, and BC-CF specimens. The results indicated a positive correlation between the curing duration and the compressive strengths of the specimens across the 7, 28, and 56-day periods. Additionally, the AC-CF specimen exhibited the highest compressive strength, followed by the BC-CF and the control specimens. Considering that the experimental conditions for AC-CF and BC-CF were fixed at 3 g, these findings aligned with previous studies that demonstrated improved compressive strength of cement composites when the fibre percentage was below 0.75% [6,7,9]. Denser cement composites typically develop smaller void amounts, leading to higher compressive strength [27].



Fig. 8: The compressive strengths of the specimens.

Figure 9 reveals the correlation between the hardened density and compressive strength of the specimens at days 28 and 56. The analysis presented that the coefficient of determination  $(R^2)$  exceeded 0.90, suggesting a strong, positive, and linear relationship between the two variables [28].



Fig. 9: The correlation between hardened density and compressive strength at the (a) 28<sup>th</sup> and (b) 56<sup>th</sup> day periods.

#### 3.5 Flexural Strengths of the Cement Composites

Figure 10 depicts the flexural strengths of the control, AC-CF, and BC-CF specimens. Similar to the compressive strength, the flexural strength of all specimens exhibited an upward trend with increasing curing days (7, 28, and 56-day periods). Furthermore, the AC-CF specimen acquired the highest flexural strength, followed by the BC-CF and the control specimens.

A study by Paul et al. discovered that the fibres in cement composites formed bridges that effectively inhibited crack opening, which improved the flexural strength of the cement composites [13]. Figure 11 portrays the conditions of the specimens after failure. After failure, the control specimen was split into two sections, while a small crack was observed in the AC-CF specimen. Thus, the fibres present in the specimen contributed to its flexural residual strength, which prevented the crack expansion.



Fig. 10: The compressive strengths of the specimens.



Fig. 11: The conditions of the (a) control and (b) AC-CF specimens after failure.

### 3.6 Carbon Fibre Dispersions in the Cement Composites

The engineering properties of cement-based composites are significantly influenced by fibre dispersion. This property can be improved by using a more uniform fibre dispersion. Figure 12 presents the carbon fibre dispersions in AC-CF and BC-CF specimens using SEM images, respectively. The carbon fibres were scattered in the AC-CF specimen, suggesting a homogeneous distribution. Entangled fibres were observed in the BC-CF specimen, indicating inadequate dispersion. These findings demonstrated that the AC-CF was superior and acquired a more uniform fibre dispersion than BC-CF. This discovery was also consistent with earlier research that reported well-dispersed fibres could successfully contribute to higher cement-based composite strengths [29,30]. Therefore, fibre additions in the fluid cement mixture were concluded to facilitate the uniform fibre dispersion process.



Fig. 12: The low and high magnification of SEM images for (a, b) AC-CF and (c, d) BC-CF specimens.

# 4. CONCLUSION

This study successfully evaluated two distinct mixing sequences (AC-CF and BC-CF) by investigating the engineering properties of the cement composites and carbon fibre dispersions. Based on the findings and analysis presented in this study, the conclusions are as follow:

- a) The flowability values of AC-CF and BC-CF were lower than the control, attributed to surface roughness on the carbon fibre.
- b) Carbon fibres improved the hardened density, compressive strength, and flexural strength values of the cement composites.
- c) The AC-CF fibre dispersion was superior to the BC-CF specimen. This outcome increased the hardened density, compressive strength, and flexural strength of the cement composite.
- d) The AC-CF specimen demonstrated the optimal mixing sequence for a cement composite containing carbon fibres.

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