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Mechanical Characteristics and Self-Monitoring Technique of Smart Cementitious Mixtures with Carbon Fiber and Graphite Powder as Hybrid Functional Additives

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HIGHLIGHTS

- Self-sensing and mechanical behavior of ECC with hybrid functional fillers were assessed.
- Carbon fibers and graphite powder were utilized to create the conductive network.
- Steel, polyolefin, and polypropylene fibers were used to enhance the mechanical properties of ECC.
- The mixtures with low dosages of graphite and high carbon fibers have more electrical conductivity with satisfactory compressive strength.
- The self-sensing behavior is better for mixtures containing a high dosage of graphite (wt.%, 2) with a low dosage of CF (vol.%, 0.25).

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ABSTRACT

In this paper, the self-sensing properties of cementitious composites under compressive loads were investigated by utilizing hybrid functional fillers, which are responsible for creating an electrical network that is used to build the self-sensing capability within the traditional cement-based mixtures. Most of the previous works depict the self-sensing capability with the aid of one type of functional fillers or fibers. The present paper attempts to utilize two types of functional fillers, representing a gap filling within the subject. Four hybrid proportions of Graphite (G) (wt.%) and carbon fibers (CF) (vol.%) were introduced. These are (0.5, 1.0), (1.0, 0.75), (1.5, 0.50) and (2.0, 0.25) respectively. In addition to carbon-based materials, polypropylene, polyolefin, and steel fibers are used as reinforcing fibers. One type of fiber was utilized in each manufactured mixture with a constant rate of 2% by volume of the mixture. A plain mixture without functional fillers has also been manufactured. The samples were non-destructively tested by an ultrasonic device before a uniaxial compression test was performed. To verify the self-sensing properties of the manufactured samples, the electrical resistance of the samples was recorded during load application each second. The self-sensing behavior was better for mixtures containing high dosages of graphite with respect to the fractional change in electrical resistivity (FCER). Steel and polyolefin fibers showed good results in terms of compressive strength behavior. However, polypropylene fibers showed the lowest compression strength among all types of reinforcing fibers used.

1. Introduction

Although it is well known that conventional concrete has no electrical properties. Still, it has been developed recently in various fields, such as monitoring stress changes on structures by detecting changes in electrical resistance under applied load. As a result, the newly developed cement compounds with structural and non-structural properties are known as "Multifunctional Cementitious Composites - MCC" [1]. These MCCs, in addition to their structural functions, act as intelligent sensors capable of detecting the subsequent deformations inside them and, therefore, instantaneously monitoring the structural health [2]. As a result, providing multiple advantages that can extend the service life of these structures/infrastructures to the greatest extent possible can be implemented by acting as stress sensors. In addition, savings on expenses, greater toughness, and advantages include improved functional volume, simplicity in design, and the absence of mechanical property degradation, which happens when inserted electronics are used in place of multi-use structural components. All that is enabled by the use of smart cement-based materials [3-6].

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Conductive cementitious composites are made by mixing certain quantities of different conductive fillers. These functional fillers include graphite powder, steel slag, fly ash, carbon fibers, and carbon black.

Many important factors are taken into consideration in manufacturing cement-based mixtures that contain the hybrid conductive fillers involve lowering the cost of these components by partially substituting a substance that is also electrically conductive [3, 7].

Most research dealing with self-sensing cement-based materials utilizes only one type of functional filler responsible for creating the electrical network within the cementitious composites. To fill a part of this gap, in the present paper, two types of functional fillers were embedded: graphite (G) as particles and carbon fibers (CF). The optimal percentage among four pairs of these materials is investigated. Moreover, three types of reinforcing fibers were utilized to enhance the mechanical properties of the manufactured mixtures. These are polypropylene (PP), polyolefin (PO), and steel fibers (SF).

The investigation of the mechanical and self-sensing properties of MCC by hybrid carbon-based fillers with optimization of the fractional change in electrical resistivity under uniaxial compression loads is presented in this paper. This was addressed by incorporating carbon fiber (CF) and graphite powder (G) into the cementitious mortar matrix. The mechanical and self-sensing properties were investigated and compared to those of plain mortar.

2. Experimental Procedures

2.1 Materials

ASTM Type I Ordinary Portland cement (PC) (Figure 1-c), Class F fly ash (FA) (Figure 1-d) that meets the requirements of ASTM C 618 was utilized in the present work. It has pozzolanic properties, 0.3 % water absorption capacity silica sand with a maximum aggregate size of 0.3 mm and a specific gravity of 2.6 (Figure 1-e), and high range water reducing admixture based on polycarboxylate ether (HRWRA). Potable water was used to produce the mixtures. The chemical and physical characteristics of PC, FA, and silica sand are shown in Table 1.

Hybrid-conducting particles and fibers were employed to make the arrays electrically conductive for the self-sensing test. The surface area of the graphite (G) was 10–35 (m^2/g), the Particle size was 40-150 µm, and the bulk density (g/cm^3) was 1.9–2.3 (Figure 1-b) [8, 9]. As a small-scale electrical conductor, 12 mm chopped carbon fiber (CF) with aspect ratios of 1600 was employed (Figure 1-a). CF had a tensile strength of 4150 MPa, a modulus of elasticity of 252 GPa, an elongation of 1.8 percent, a density of 1.7-2.0 (g /cm^3), and a diameter of 7 µm. Polypropylene (Figure 1-f), polyolefin (Figure 1-g), and steel fibers (Figure 1-h) with diameters of 18, 900, and 200 microns, nominal tensile strength 400, 500, and 2850 MPa, modulus of elasticity 4, 20, and 200GPa, and specific weights of 0.91, 0.91, and 7.8, respectively, were used to construct ECC mixes for reinforcing issues. The application rate was 2% by the total volume of mixtures to improve mechanical characteristics, which comprise numerous carbon-based conductive fillers, as shown in Figure 1. SEM images of the graphite and carbon fibers used are presented in Figures 2-a and b respectively.

Table 1: Chemical properties and physical characteristics of PC, FA, and silica sand

Chemical composition, %	PC (wt.%)	FA (wt.%)	Silica sand
CaO	62.35	7.98	--
SiO2	21.33	52.22	>= 50 - <= 100 %
Al2O3	3.74	16.58	--
Fe2O3	4.76	6.60	--
MgO	3.73	2.10	--
SO3	2.01	0.02	--
Loss of ignition (LOI)	2.07	10.36	--
Insoluble residue	0.68	-	-
Lime saturated Factor	0.9	-	-
Physical properties			
Specific gravity	3.15	2.10	2.65
Blaine fineness (cm2 g−1)	3940	2690	----

Figure 1: Samples of materials used

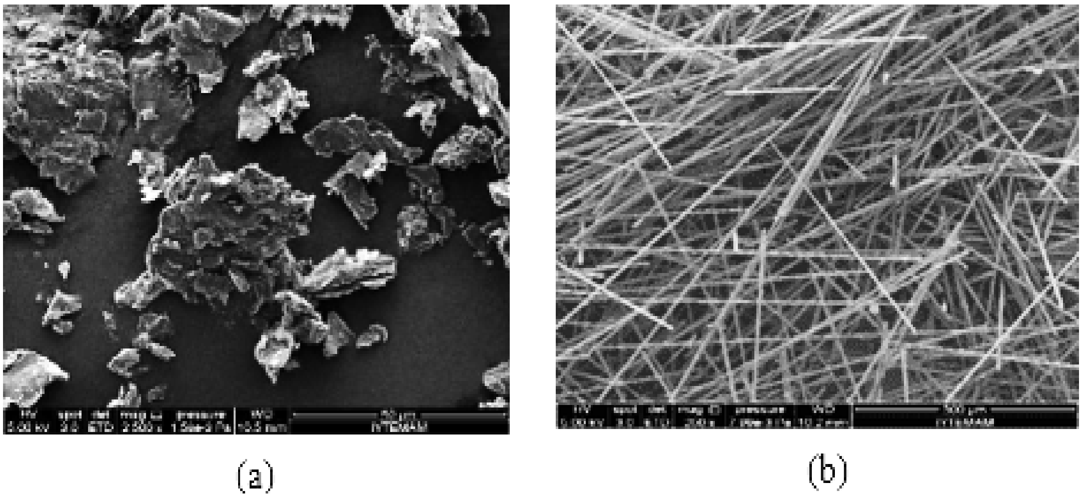

Figure 2: SEM photos of (a) graphite, G and (b) carbon fiber, CF

2.2 Preparation of Mixtures and Proportions

The ratio of water to cementitious materials (W/CM) and fly ash to Portland cement (FA/PC) was 0.27 and 1.2, respectively, and were used to manufacture the cementitious composites (with and without reinforcing fibers). These ratios were used according to the recommendations of [1, 2, 6, 10].

Table 2 shows the percentages used of the carbon-based electrically conductive hybrid composites. The number of micro-scale materials (CF) was a proportion to the full volume of the mortar specimen. In contrast, graphite powder material (G) was a dosage by the complete weight of cementitious materials (PC + FA). This research's carbon-based electrically conductive filler ratios are based on previous studies [2, 11].

Previous research showed that a continuous electrical network capable of sensing stress in real-world applications requires strong contact between electrically conductive components via the regular distribution of these materials in cementitious systems [2, 12-14].

The electrically conductive carbon-based particles (the graphite, G) are mixed with the full amount of mixing water and HRWRA using a hand blender at 3000 rpm for 15 min. Meanwhile, the dry raw materials (PC, FA, and silica sand) were mixed with carbon fibers (CF) (see Figure 3-a, b) for 10 minutes at 100 rpm in a mortar mixer (4-liter capacity) to disperse the carbon fibers within the cementitious system homogeneously.

The preprepared graphite solution was then gradually added to the premixed raw materials over 10 seconds at 100 rpm. Next, the speed is increased to 300 rpm after completely adding the (Graphite-HRWRA-water) solution for an additional 10 minutes. Finally, the mixer was slowed down to 100 rpm once again to add the reinforcing fibers (steel, polypropylene, or polyolefin fibers) (see Figure 3c-d), and the mixing of all components continues for 10 more minutes at 300 rpm [1], as shown in Figure 3.

Table 2: Proportions used of fibers and electrically conductive particles

Type of Material	Amount of materials utilization, %[a]			
	1st ratio	*2nd ratio*	*3rd ratio*	*4th ratio*
Carbon fibers (CF)	0.25	0.50	0.75	1.00
Graphite (G)	0.50	1.00	1.50	2.00
Proportions of pairs of fillers (CF + G)	1.00, 0.50	0.75, 1.00	0.50, 1.50	0.25, 2.00
Polypropylene fibers	2.00	2.00	2.00	2.00
Polyolefin fibers	2.00	2.00	2.00	2.00
Steel Fibres	2.00	2.00	2.00	2.00

a, by total weight of cementitious materials (PC+FA) for graphite and total volume of mixtures for carbon fiber.

Figure 3: Mixing steps of cementitious composites

In order to ensure acceptable and equal workability of the manufactured cementitious composites, a mini-slump flow test was carried out to the produced mixtures. The dosage of HRWRA was variable due to the use of hybrid functional fillers with different dosages to attain the same flow deformation levels for all types of the mixtures. Figure 4 - a, b, c and d exhibits the deformation levels of the mixtures contains (0.25% vol. CF+2.0% wt. G.), (0.50% vol. CF+1.5% wt. G), (0.75% vol. CF+1.0% wt. G) and (1.00% vol. CF+0.5% wt. G) respectively.

Figure 4: Deformation levels of the produced mixtures

2.3 Laboratory Tests

2.3.1 Compressive testing and electrical resistivity measurement

Samples were made using a 2-inch (50 mm) cubic mold (Figure 5-a), and compression testing was performed in line with [15]. A digital electrical testing machine (ELE) with a capacity of 2000 kN and a loading rate of 0.36 MPa/sec was used to perform the compression test under stress control. Three specimens from each type of mixtures were examined throughout the test, carried out under continuous loading until failure.

During the compression test, the DC electrical resistance along the stress axis was measured using binary probes embedded in the fresh mortar in two symmetrical planes with respect to midway along with the sample height and perpendicular to the stress axis in order to acquire reliable results. DC measurements were performed using the Pro'sKit MT-1860 Multimeter. A wood cover was placed between the loading machine and the samples to prevent an electrical connection, which might impact the data's accuracy. The electrical resistivity data is generated as a result of the preceding, which is then transformed into the fractional change in electrical resistivity (FCER, percent) using Equation 1.

$$\mathrm{FCER}\left(\frac{\Delta\rho}{\rho o}\right)\% = \left(\frac{\rho T - \rho o}{\rho o}\right) \times 100 \quad (1)$$

where FCER, ρT, ρo is the fractional change in electrical resistivity, the electrical resistivity during compression loading, and the initial electrical resistivity, respectively.

Two electrodes are used in this work. Strips of brass plate with a 0.1 mm thickness, 0.3 resistance, 75mm length (50 mm on the inside of the cube and 25 mm outside it), and 10 mm width were implanted symmetrically around the midpoint of the sample. The distance of 40 mm between them is perpendicular to the length of the sample for the measurement of the electrical resistance of the sample, as presented Figure 5a for electrode positions, Figure 5b for an electrode sample and Figure 5c for a poured cubic specimen . This method was based on what Al-Dahawi [10] and Azhari [16] achieved regarding the methods of using the electrodes and their types. Figure 6 presents the whole compression and self-sensing test setup. To instantaneously record the electrical resistance, a control unit was utilized (Figure 6a). Moreover, the electrical resistance values were recorded with the aid of a DC source meter as exhibited in Figure 6b. Due to the inability to electronically record the applied compression load on the specimens, a digital camera was utilized (see Figure 6c).

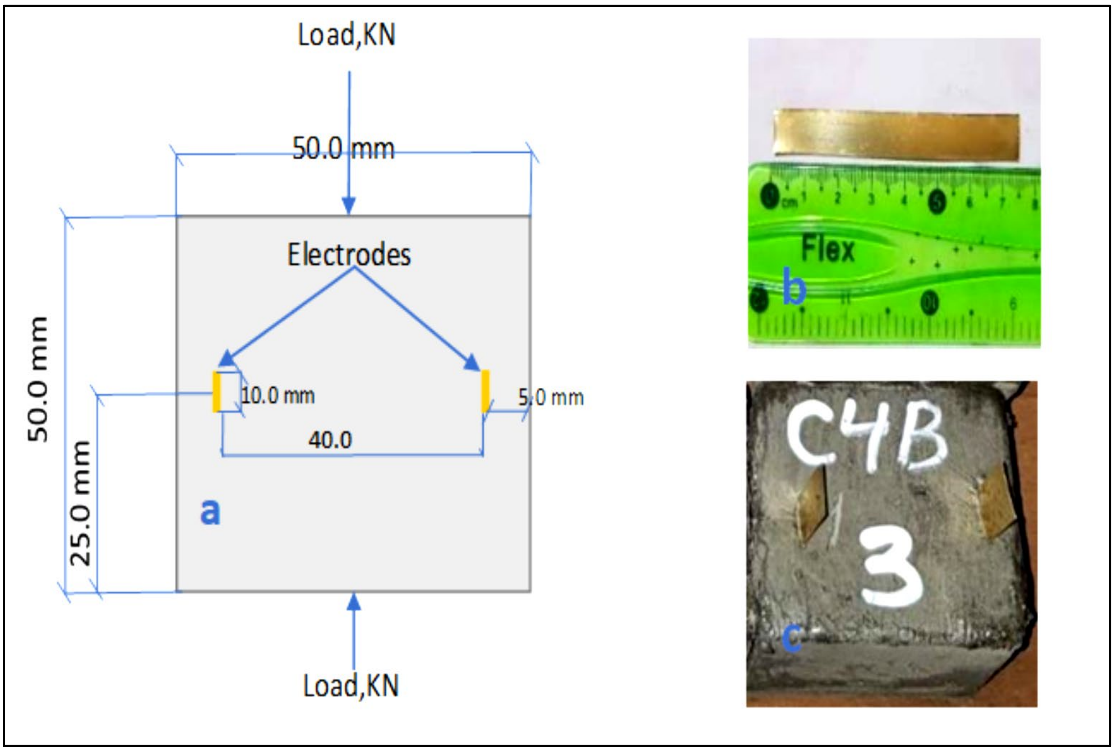

Figure 5: Cubic sample: (a) geometric dimensions, (b) brass plate electrodes, and (c) a poured cubic specimen with coding and electrodes

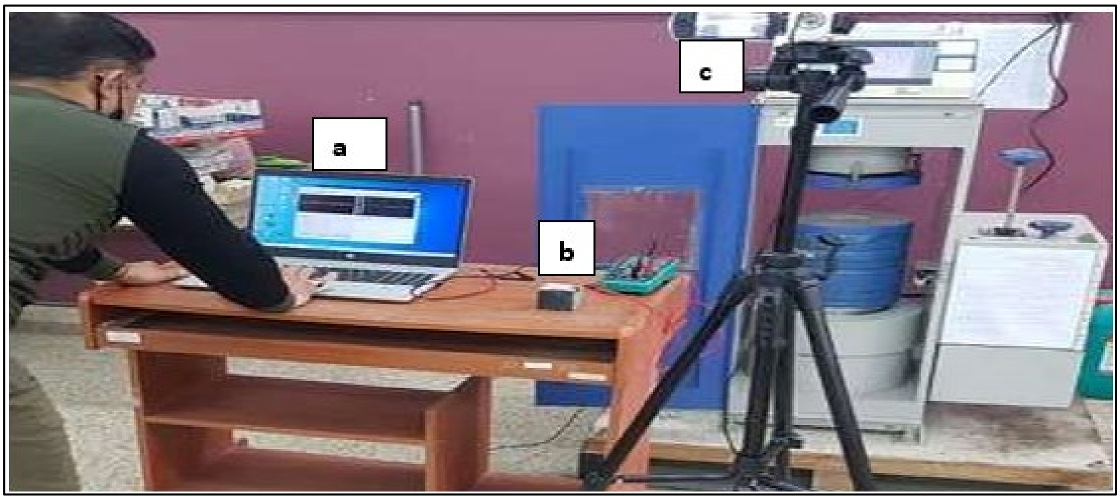

Figure 6: Electrical resistivity test settings, (a) resistivity measurement software, (b) Pro'sKit MT-1860 electrical resistance meter, and (c) digital camera to record compression value every second

2.3.2 Ultrasonic pulse velocity (UPV) test

An ultrasonic velocity test of 2-inch (50 mm) 28-day-old cubic specimens was performed in line with [17]. An acoustic electro transducer is mounted in contact with a single concrete surface under test, and longitudinal stress wave pulses are created by it. The pulses are received and transformed into electrical energy by a second transducer placed at a distance L from the transmitted transducer after passing the concrete. T stands for transit time, and it is measured electronically. Pulse velocity V is determined by dividing L by T.

Figure 7 shows the test procedures by the Proceq Pundit Lab device. After calibrating the instrument according to the manufacturer's instructions [18] (Figure 7a), three preparation samples were greased (Figure 7b) to prepare them for testing (Figure 7c). By measuring the time it takes for ultrasonic pulses to travel through concrete (Figure 7c) and comparing it to authorized standards such as BSI, the samples' quality and the mixture's homogeneity may be determined. For example, slower rates indicate that the concrete has numerous cavities and fractures, whereas higher speeds show that the material's quality and consistency are good [19].

Because the pulsation velocity in saturated concrete is 5% greater than that of dry concrete and less affected by variations in its relative quality, the degree of saturation of the samples was taken into consideration by treating them with heat at 60 ° C for 24 hours with lubrication of the opposite sides of the samples to achieve more trustworthy findings. The direct transmission method is used in this test according to the standard specifications, defined as the propagation of ultrasonic waves along a straight-line path between the two opposing sides of the sample in this investigation [17, 19, 20].

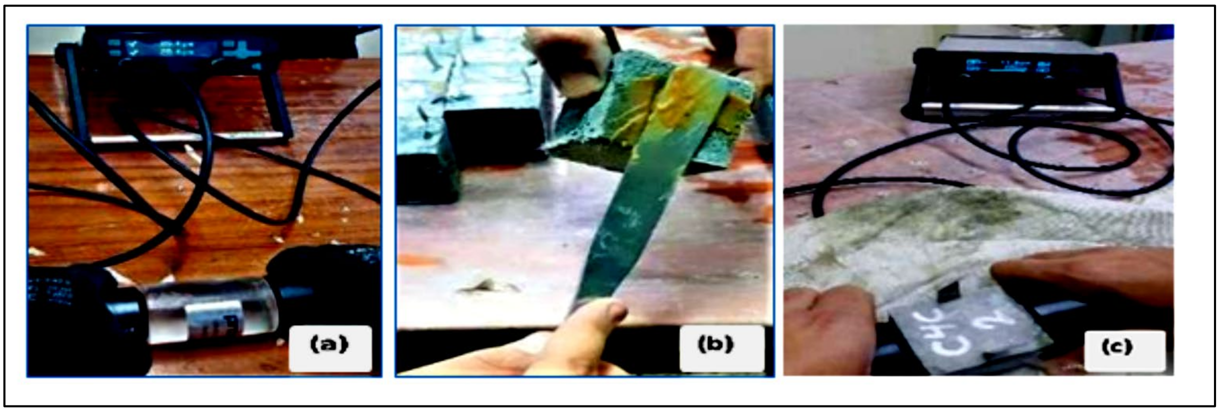

Figure 7: (a) Ultrasonic device calibration, (b) Greasing surfaces of the specimen, and (c) Ultrasonic test of a sample

3. Results and Discussion

3.1 Mechanical Properties of Compression Test

The compressive strength test results were conducted for specimens aged 28 days. Their effect on the mechanical properties of the hybrid mixtures was mechanically supported by one out of three types of fibers with a fixed ratio by the total volume of the mixture of 2% was investigated.

Figure 8 (a-d) presents the compressive strength in MPa of the different types of electrically conductive-based mixtures in addition to the mixtures without any conductive fillers reinforced with polypropylene fibers, polyolefin fibers, steel fibers and those mixtures without reinforcing fibers respectively.The results exhibits that the (CF+G) hybrid functional fillers mixed with polypropylene fibers showed a significant decrease in the compressive strength as in the previous sections with an increasing dosage of graphite powder compared to other fibers, with a peak of 22 MPa regarding the mixture C1A shown inTable 3, when the full 1.0 vol.% CF and a quarter of the suggested percentage of G 0.5% wt. are used. Moreover, the compressive strength of the plain mixture with and without polypropylene fibers were 34 and 27 MPa, respectively. To explain that, the weak interaction between the matrix of the mixture and the hydrophobic graphite particles, and the lamellar graphite structure can act as a stress center for fracture development [21].

The same hybrid ratios have different compressive strength behavior when reinforced with polyolefin fibers. It registered an average value of 36 MPa in the third ratio (Table 3) of the hybrid mixture with proportions of graphite powder and carbon fibers of 1.5 %wt. and 0.5 %vol., respectively. The improvement rate of this hybrid mixture was 64% over that reinforced with polypropylene fibers, while the improvement percentages over the plain mixture (with and without polyolefin fibers) were 21% and 32%, respectively.The highest compressive strength values were achieved with steel fibers compared to the rest of the fibers, with a value close to 56 MPa when the proportions of graphite powder and carbon fibers are 1.0 wt.% and 0.75 vol.%, respectively. This superiority in mechanical properties enhances what the researchers achieved and mentioned earlier in several studies about the behavior of carbon fibers and graphite particles with steel fibers [22-27]. These studies confirmed that combining short carbon fibers with short steel fibers in the same matrix of cementitious composites enhances the mixture's mechanical properties by continuing the compressive strength network. Moreover, the presence of the graphite particles in the cementitious matrix enhances the distribution of steel fibers in a large way [26, 28]. The improvement ratios over mixtures reinforced with polyolefin, polypropylene, and plain mixtures (with and without steel fibers) were 56%, 155%, 24%, and 105 %, respectively. Generally, all the hybrid ratios containing steel fibers have better compressive strength than others, especially when carbon fibers are utilized in the same group. Previous studies concluded that in blends reinforced with steel and carbon fibers, a strong fiber-to-fiber bond is generated [23], especially if compared to carbon-polypropylene fiber combinations. It demonstrated a noticeable drop in the mixture's strength, which may be justified due to the lower elastic modulus and tensile strength of PP fibers [29].

Table 3: Coding utilized for mixtures of carbon-based conductive materials and reinforcing fibers

Type of mixture	Coding			
	1st ratio	*2nd ratio*	*3rd ratio*	*4th ratio*
Coding of (CF+ G), (C)	C1	C2	C3	C4
Coding of (CF+ G) with (PP) fibers	C1A	C2A	C3A	C4A
Coding of (CF+ G) with (PO) fibers	C1B	C2B	C3B	C4B
Coding of (CF+ G) with (SF) fibers	C1C	C2C	C3C	C4C

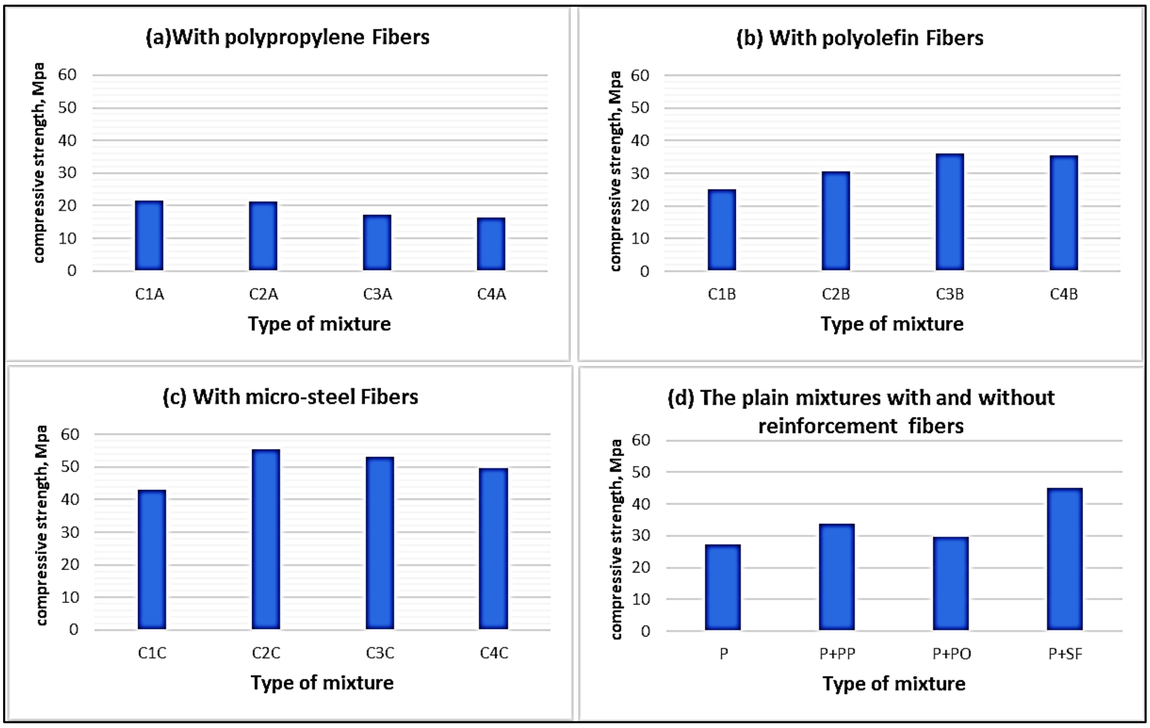

Figure 8: Compressive strength results from different types of mixtures

3.2 Self-Sensing Behavior of Mixtures Under Uniaxial Compression Test

Figure 9 compares the best self-sensing behavior (in terms of fractional change in electrical resistivity (FCER)) obtained by the different mixtures. They were manufactured in the present paper using different dosages of functional fillers (CF and G) and reinforced with one type of reinforcing fibers (PP, PO, or SF) versus testing time under uniaxial compression stress. The mixtures in this figure are coded according to what was previously detailed in Tables 2 and 3.

The results show a clear difference in the self-sensing behavior of the manufactured mixture under compression load. As can be seen in Figure 9, the specimens reinforced with polypropylene and polyolefin fibers have the highest fractional change in electrical resistivity (FCER, %) under loading when the pairs of the hybrid functional fillers CF, %vol. and G, %wt. utilized are (0.75, 1.0) and (0.25, 2.0) respectively compared to the other mixture types. Furthermore, the plain mixture without conductive fillers showed a significant self-sensing behavior. This is due to the fly ash presence with a high percentage of Fe_2O_3 that enhances the self-sensing of the plain mixtures [6]. Moreover, the figure exhibits the modestly self-sensing behavior of the mixture reinforced with steel fibers.

Accordingly, to create a state of balance between FCER and compressive strength together to achieve the desired goal of the research, which is the early monitoring of the health of concrete structures and the preservation of their mechanical properties if they are not improved, the mixture contains 0.25 %vol. CF and 2.0 %wt. G and reinforced with 2.0 %vol. Polyolefin fibers have the best self-sensing behavior with enhanced mechanical properties in compressive strength. This finding is closed to what was concluded by previous works [30, 31].

This can be justified due to the theory of tunnels formed by carbon fibers, which are crucial in the properties of electrical resistance under stress [32]. Furthermore, the electrical resistance may sometimes decrease after starting the compression load application. This may be attributed to the reduced contact resistance between the carbon fibers when they are forced to converge due to the elastic volumetric stress of the material. Moreover, the sample conduction length is also reduced due to this stress [33].

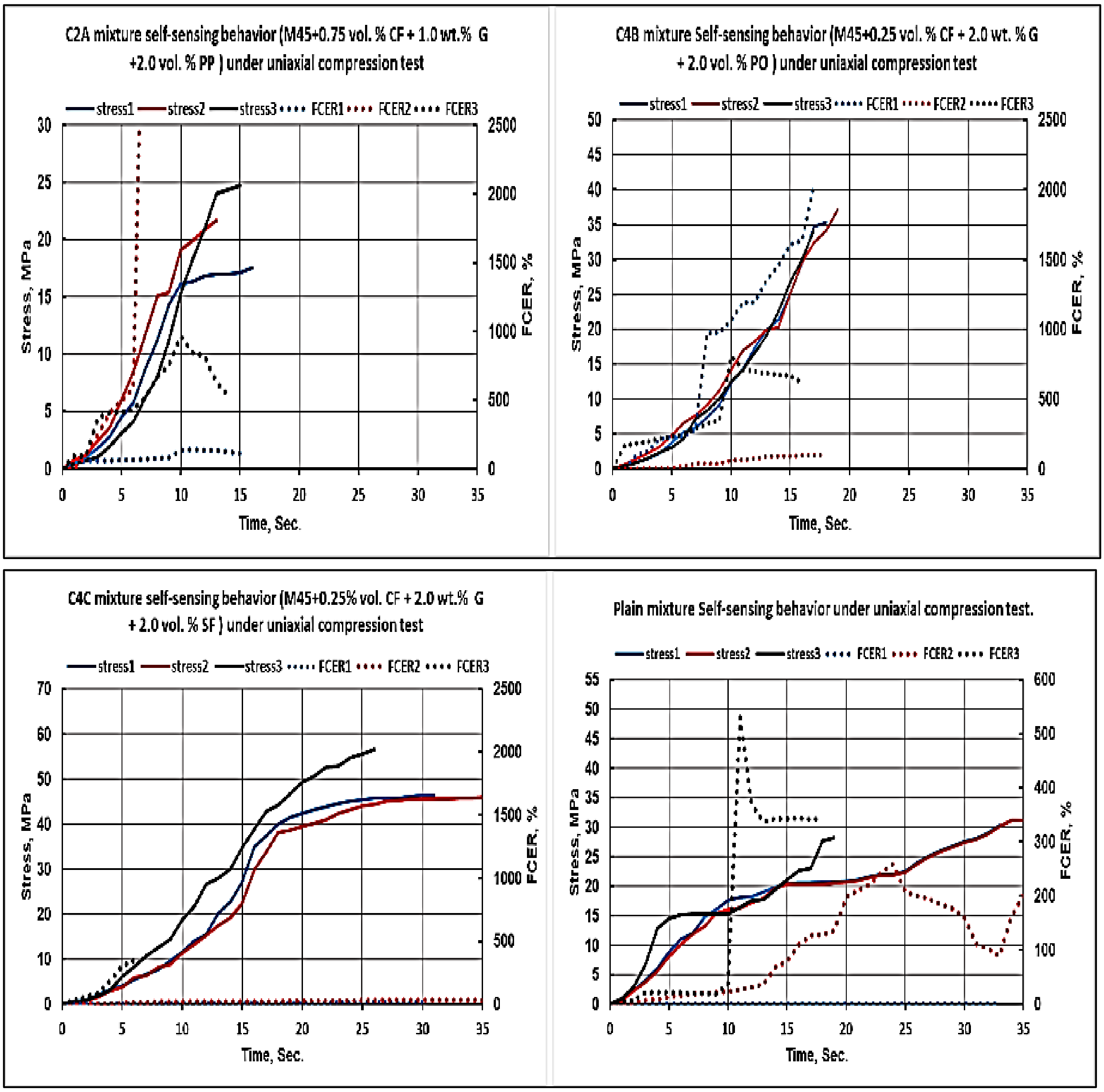

Figure 9: Self-sensing behavior of hybrid mixtures under uniaxial compression test

3.3 Ultrasonic Pulse Velocity Test Results

Table 4 and Figure 10 present the pulse transmission velocity results in the ultrasonic test of cubes (2 inches) for all types of mixtures, even the plain ones. Carefully, the sample size clearly impacts the accuracy of the data obtained from the UPV test. This was confirmed by a previous study [34], which indicated that the sample size should be no less than 50 mm and not more than 100 mm because the pulse is either large at small distances or small at large distances. Therefore, deviations in the values of the results of the tests may occur. The test values were compared with British Standards (BS) to determine the mixtures' quality. The BS Standards classify the ultrasonic pulse velocity through samples as very poor, weak, dubious, good, and excellent for the values of 2000 m/s and less, 3000-3500, 3500-4500, and 4500 m/s and above, respectively [19]. By taking a careful look at Figure 10, the UPV values of the mixtures reinforced with steel fibers are the best in pulse speed (4328 m/s), followed by those reinforced with polyolefin fibers at a speed of 4129 m/s. These results are consistent with what was reached by [35] at 28 days, while all the mixtures reinforced with polypropylene showed a significant decrease in pulse speed, even with the plain mixtures, which reached 3,639 m/s. However, all mixtures can be regarded as good homogeneous mixtures. Moreover, this distinction in average pulse speeds results from the presence of hybrid conductive fillers gave the impression that the electrically conductive materials affect the homogeneity and strength of the mixture, and this is consistent with what was achieved by [20]. They showed that the types of materials and their proportions in the cement-based mixture affect the speed of the pulse.

Table 4: Ultrasonic Testing Results

No.	Types of mixture	Pulse time, µs	UPV, m/s
1	C1A	12.1	4144
2	C2A	12.4	4032
3	C3A	12.9	3876
4	C4A	12.7	3927
5	C1B	12.7	3927
6	C2B	12.9	3876
7	C3B	12.6	3979
8	C4B	11.7	4261
9	C1C	11.4	4386
10	C2C	11.4	4386
11	C3C	11.4	4386
12	C4C	11.6	4323
13	P	12.4	4032
14	P+PP	12.4	4032
15	P+PO	12.4	4032
16	P+SF	12.4	4032

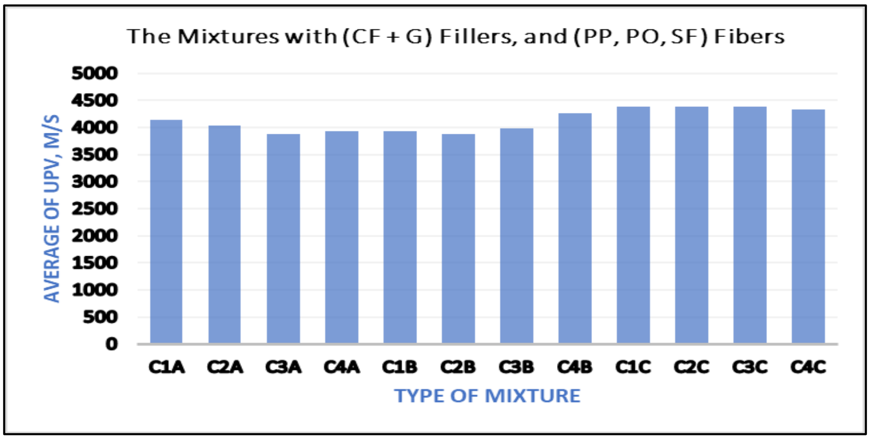

Figure 10: Results of UPV test

4. Conclusions

Some conclusions from this work can be summarized as follows:

1) The formation of cementitious composite mixtures from conductive hybrid materials (carbon fibers and graphite powder) has clear effects on their piezoresistive properties through the noticeable fractional change in the electrical resistivity under the applied load over time. It is very useful in various applications, particularly in monitoring the structural health of highway structures.
2) Among 12 types of mixtures, which were fabricated within this paper, the mixture consists of (2.0 vol.% polyolefin (PO), 0.25 vol.% carbon fibers (CF), and 2.0 wt.% graphite (G)). It is coded by (C4B) gave the study's desired objective, which is the early self-sensing of damage with an average fractional change in electrical resistivity exceeding the 700% limit. Furthermore, the mechanical properties in terms of compressive strength of this mixture were enhanced by 30% over the plain mixture.

Author contribution

All authors contributed equally to this work.

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Data availability statement

The data that support the findings of this study are available on request from the corresponding author.

Conflicts of interest

The authors declare that there is no conflict of interest.

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