Effect of curing age on the self-sensing behavior of carbon-based engineered cementitious composites (ECC) under monotonic flexural loading scenario

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Abstract. The potential effects of curing age on the self-sensing (piezoresistivity) capability of carbon-based Engineered Cementitious Composites (ECC) specimens are under focus in the present study. This non-structural feature can be regarded as one of the best solutions for continuously monitoring of infrastructures in terms of damage and deformations. Carbon fibers which are a micro-scale electrically conductive material were added to the ECC matrix and well dispersed to create the electrically conductive network. This network is responsible for sensing the applied loads on the prismatic specimens. The self-sensing behavior of electrically conductive prismatic specimens under four-point monotonic flexural loading was investigated and compared with dielectric ECC specimens at four ages of curing (7, 28, 90 and 180 days). The results showed that the developed multifunctional cementitious composites can sense the changes in the applied flexural stresses and the resultant mid-span deflection along the adopted curing ages with an improvement in the later ages.

1 Introduction

The concrete, which is the main material in the construction of several structures and infrastructures around the world, suffers from of a lot of risks resulting from the applied loads, as well as, from the surrounding weather conditions, which caused, as a whole, a decline in the serviceability index of these structures. Cementitious composites have demonstrated outstanding performance in many civilian facilities such as roads, bridges, dams, sanitary facilities and others. In order to continue working with these materials within the economic boundaries, it was incumbent on the civil engineer to accept the challenge of the conditions referred above in order to ensure the continuity of the work of concrete structures and infrastructures along the economic construction age at the lowest cost. This cannot be achieved easily without incessant monitoring of the structural performance of these cementitious composite constructions in terms of stress, strain, and damage indications to remove the potential defects quickly and economically by the application of the rehabilitation systems opportunely.

To achieve the above goal, and as an initial step, many types of sensors have been used either to be embedded inside the structural members, or attached with them [1]. The only advantage that has been obtained through the use of these sensors is to achieve control in the area of concrete structures. On the other hand, the high cost, the non-homogeneity of the cementitious composites, the limited monitoring of concrete members hampered the use of these sensors. Moreover, the limited durability and the weak resistance of these devices to weather conditions in addition to their contribution in reducing the mechanical properties of concrete infrastructures/structures are the main reasons that led to search for effective, precise, durable and cheap structural health monitoring tools.

The self-sensing technology that appeared in the nineties of the last century is an alternative technology to continuously monitor the behavior of carbon-based cementitious composites effectively without any negative impact on their mechanical properties. This technique is based on the employment of non-structural properties of the cementitious composites to self-sense the distresses suffered by the structure by embedding electrically conductive carbon-based materials within these composites to create semi-homogeneous conductive networks. This gives the ability to sense the development of distresses in terms of strain, stress and damage by means of changes in the electrical resistivity. So, in this case, the cementitious composite material will sense itself without embedded or attached sensors.

This behavior has been found to be more effective in cement-based paste and mortar than in concrete [2]. Many researchers in this field studied the effects of static compression loadings on the self-sensing cementitious composites, besides few studies highlighted on the behavior of these composites under static flexure. It is

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important to mention that most of the previous studies concentrated the efforts on clarifying the self-sensing behavior under loadings for only one curing age, and this factor is not included in those studies except the study that was carried out by Fu and Chung [3]. The self-sensing behavior of carbon fiber reinforced mortar was studied under static compression up to failure for the curing ages from 7 to 28 days. It was found in that study that, after 7 days of curing, the electrical resistance increased monotonically with the increase in compressive strain versus decrease in the electrical resistance after 14 and 28 days of curing ages. It was concluded that the reason of this behavior is due to the weakening of fiber-cement interconnection with the progress of curing age. It was presented figuratively that the fractional change in electrical resistance reached up to around 225% at compressive strain of 1.9×10-3 and compressive stress of 39 MPa at 7 days of curing age. These results can be seen, also, within the study of Chung [4].

On the other hand, many studies have applied their tests at only one age of curing. Chen and Chung [2] examined the capability of mortars reinforced by 0.2-4.2 vol.% and concretes contain 0.2-1.1 vol.% of short carbon fiber (CF) to act as a strain sensor via electrical probing under static and cyclic compression, tension and flexure loadings at 7 days age. It was reported that the fractional change in electrical resistivity (FCER) increases with the applied stresses by up to 2100% under static compression up to failure, 5.3% under static tensile loading up to failure and 18.4% under static flexure loading up to failure. It was concluded that the small increase in FCER in the cases of loading under tension and flexure is due to much lower ductility under tension and flexure than compression.

Han and Ou [5] used a 6mm CF with carbon black (CB) as conductive materials to prepare a cement-based mortar and explore its piezoresistivity under single compressive loading after 28 days of curing. The manufactured specimens have dimensions of 50×40×30mm3. Embedded four gauzes for each specimen were used as electrodes to measure the 4-probe electrical resistivity in the perpendicular direction with respect to load direction. It was noted that the FCER decreased with the increase in the applied stress/strain to reach up to 27% at an ultimate stress and strain of 40MPa and 0.0022 respectively.

The damage in carbon fiber reinforced concrete was monitored under uniaxial compression and three-point bending by electrical resistance measurement within a study carried out by Chen and Liu [6]. Pitch-based carbon fibers with 5mm length were used to fabricate the conductive cubic specimens ($70.7\times70.7\times70.7mm^3$) and prismatic specimens ($40\times40\times160\ mm^3$). DC with 4-probe method was used to measure the electrical resistivity. The electrical contacts were copper wires attached around the whole perimeter of the cubes or around the four planes of the prisms with the aid of silver paint. The four wired planes of the four electrical contacts were perpendicular to the stress axis in the case of cubes and parallel to the stress axis in the case of beams. It was observed that the fractional change in resistance with respect to cubes under uniaxial compression decreases when the stress is increased at low level of stress which was equal to the 57% of the fracture stress. The fractional change in resistance at this point reached up to around 38%. After this point, when the damage in the specimen begins due to high level of stress, the fractional change in resistance increased rapidly and reached up to 50% with stress increasing. The researchers divided the resulted load-displacement-FCER curve into three zones. The first one refers to the elastic stage with a deflection range of greater than zero and less than 0.01mm. Within this zone, it was noted that with the increase of deflection, the FCER increased up to around 1.2%. In the second zone, when the deflection is bounded between 0.010mm and 0.015mm, visible cracks occurred and the FCER increased from 1.2% to 1.5%. The end of this zone represented the point of maximum applied load. When moving to the third zone with a deflection of more than 0.015mm, it was seen that the FCER remained approximately constant due to breaking of the conductive network.

Yeh et al. [7] used a 15mm chopped CF to fabricate a cement-based self-sensing material for measuring the strain and detecting the damage of civil engineering structures. The researchers tested the specimens under compression and bending at 28 days age. To measure the electrical resistivity, 4-probe method was used by attaching copper wires around the perimeters of the specimens with the aid of silver paint. Cylindrical samples with dimensions of (100mm diam.×200mm h.) and prismatic samples with dimensions of ($550\times150\times150mm^3$) were prepared for uniaxial compression and 3-point bending tests, respectively. The experimental results of uniaxial compression test showed that the fractional change in electrical resistance reached up to 14% corresponding with 4.2×10^{-3} compressive strain. The experimental results of cyclic loadings of all fabricated types of three-point bending specimens showed that the relationships between fractional change in electrical resistance and strain measured at top and bottom of specimens were linear. At a cyclic tension strain level of 0.05×10^{-3}, the fractional change in electrical resistance reached up to around 3.5%, whereas, the value of this parameter reached up to around 5% at a cyclic compression strain level of 0.08×10^{-3}.

In the present study, the effects of curing age on the self-sensing behavior of cementitious composites were experimentally investigated under four-point monotonic flexural test at 7, 28, 90 and 180 days of curing. Moreover, electrically conductive chopped carbon fibers (CF) in micro-scale were used to fabricate the multifunctional cementitious composites (MCC).

2 Experimental program

2.1 Ingredients of carbon-based cementitious composites

The fabricated mixtures, in the present work, comprise mainly the common ingredients which are used in the composition of the cement-based mortar. These are: CEM I 42.5R ordinary Portland cement (PC) (similar to ASTM Type I); Class-F fly ash (FA); fine silica sand with maximum aggregate size of 0.4 mm, specific gravity of 2.60 and water absorption capacity of 0.3%; potable mixing water and polycarboxylate ether based high range water reducing admixture (HRWRA). The chemical and physical properties of Portland cement, FA and silica sand are illustrated in Table 1, and the particle size distributions of these ingredients are represented in Figure 1.

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

Chemical composition, %	PC	FA	Silica sand
CaO	61.43	7.98	0.02
SiO_2	20.77	52.22	99.79
Al_2O_3	5.55	16.58	0.06
Fe_2O_3	3.35	6.60	0.02
MgO	2.49	2.10	0.01
SO_3	2.49	0.02	-
K_2O	0.77	1.53	0.01
Na_2O	0.19	0.86	0.02
Loss on Ignition (LOI)	2.20	10.36	0.07
$SiO_2 + Al_2O_3 + Fe_2O_3$	29.37	75.40	99.87
Physical properties			
Specific gravity	3.06	2.10	2.60
Blaine fineness (cm^2/g)	3250	2690	-

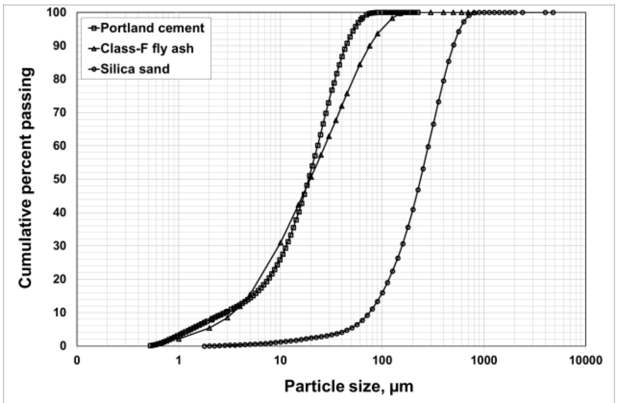


Fig. 1. Particle size distributions of Portland cement, class-F fly ash and silica sand.

The carbon-based electrically conductive materials, which were employed to create an electrically conductive network within the cementitious composites, were chopped carbon fibers with 12mm length and aspect ratio of 1600 (CF12). CF were with the tensile strength of 4200 MPa, elastic modulus of 240 GPa, elongation of 1.8%, density of nearly 1.7-2.0 g/cm3 and diameter of 7.5 µm.

Polyvinyl Alcohol fibers (PVA) of 8mm length, 39 µm diameter and specific gravity of 1.3 were also used to enrich the mechanical properties of the developed materials against the applied loads, in spite of its negative impact on reducing the electrical conductivity of the developed MCC because it is a dielectric material. The nominal tensile strength and elastic modulus of these fibers were 1620 MPa and 42.8 GPa, respectively.

In order to remove any undesirable bubbles that may be created due to the use of HRWRA, a defoamer powder was used as a percentage by weight of cementitious materials.

2.2 Electrical measuring setup

To measure the electrical resistivity of the specimens under 4-point flexural tests, four brass plates were embedded within 360×75×50mm3 prismatic specimens. The positions of electrodes and the test configuration setup can be seen in the last study carried out by the author [8]. Alternating current (AC) with 2-probe method and direct current (DC) with 4-probe method were used to measure the electrical impedance between the electrodes in the case of AC and to measure the voltage values between the internal electrodes due to a constant applied current on the external electrodes in the case of DC. Engineered Cementitious Composites (ECC) mixtures were used in the present study. ECC was firstly developed by Li [9] to be the unique construction material that has a flexible application together with low cost and weight, isotropic properties and high performance in terms of strength, ductility, fracture toughness and pseudo strain-hardening. ECC is formed from cement-based mortar with a high modulus fiber such as PVA. Since its composition is similar to that of conventional types of concrete and fiber reinforced concrete (FRC), the ECC can be regarded within the non-electrically conductive materials. To add the electrical conductivity property, as an additional function to the ECC without losing its properties, carbon-based materials, which are essentially electrically conductive materials, can be utilized.

2.3 Mixture proportions and mixing methods

Water to cementitious materials (PC + FA) ratio (W/CM) and fly ash to Portland cement ratio (FA/PC) were kept constants as 0.27 and 1.2, respectively during the preparation of mixtures. The dosage of CF as it was reported by Al-Dahawi et al. [10]. This dosage was 1.00% by volume of mixture. Moreover, for the same reasons that were previously mentioned, the PVA fiber was used with a 2.00% dosage by the volume of the mixture [9]. In addition to that, the high range water reducing admixture (HRWRA) was used to get the same liquidity of the MCC. It is well known that the differences in the microstructural properties of the carbon-based ECM that were utilized, in the present study, led to difficulties in obtaining the same workability by the use of the same amount HRWRA. To keep the specimens in the same acceptable workability, different dosages of HRWRA were, therefore, used for the different carbon-based MCC. A defoamer dosage of 0.3% by weight of cementitious materials was utilized to remove the undesirable air bubbles.

During the present study, the best methods that were suggested by Al-Dahawi et al. [11] to mix the micro-scale materials (CF) were utilized here for the achievement of the best distribution of the electrically conductive carbon-based materials in order to obtain the best self-sensing capability. Mixing of CF within the cementitious matrix was made in accordance with the findings of that study to get the highest electrical conductivity. Accordingly, CF was first mixed with the dry raw materials (PC, FA and silica sand) in a 20-liter-capacity mortar mixer at 100 rpm for 10 minutes. After slowly adding the mixing water at 100 rpm over 10 seconds, speed was increased to 300 rpm, all of the HRWRA was added over 30 seconds, and mixing of all materials was continued for additional 10 minutes at 300 rpm in the mortar mixer.

3 Preparation and testing of specimens

The fabricated specimens were poured, according the mixing procedures that have been recommended by Al-Dahawi et al. [11], in the oiled molds and have been placed in their molds at 50 ± 5% RH, 23 ± 2 ^{o}C and covered with plastic sheets for 24 hours. After demolding process, all the specimens were, then, moved into isolated plastic bags to be cured at 95 ± 5% RH, 23 ± 2 ^{o}C until 24 hours before the pre-specified test ages, regarding to the specimens that were tested within 28 days. To remove the potential effects of the internal moisture on the electrical measurements due to polarization, the specimens were dried in an oven at 60 ^{o}C for 24 hours before testing [12, 13]. The prepared specimens to test after 90 and 180 days have been removed from the plastic bags after 28 days of curing and then kept at 50 ± 5% RH, 23 ± 2 ^{o}C till their pre-specified testing dates. All specimens were tested at room temperature.

4 Results and discussion

4.1 Mechanical properties of the developed MCCs under flexure

The most important mechanical features of the prismatic specimens under the flexural loading are the ultimate flexural strength (also known as a modulus of rupture) and the deflection hardening that can be obtained from the multiple cracking behavior (vs single cracking and localization) as mentioned by Naaman [14] to define the high performance of the fiber reinforced cement composites. The typical stress/deflection curve of the prismatic specimens that behave with the deflection hardening contains many important points. These are the first cracking position; the points of load-carrying capacity greater than the load at the first cracking point and the ultimate flexural strength point which is represent the modulus of rupture value as mentioned above and as defined by the ASTM C 78 – 02. All these points were described in detail in the previous ASTM standard C 1018 – 97. However, the researchers faced a difficulty in correctly distinguishing the first cracking position and the corresponding strength for the materials exhibited a deflection hardening behavior according to the new ASTM standard C 1609 [15]. Therefore, in the present study, the method that was described in ASTM standard C 1018 – 97 and suggested by kim et al. [15] was utilized to identify this point at the stress/deflection curve. This method takes into account the fact that the point which is located at the end of the straight line and the beginning of the non-linear line of the stress/deflection curve is the point of the occurring of the first crack. The simplest way that was explained by Naaman [14] to represent the deflection hardening behavior was used in the present study. He showed that the deflection hardening of the prismatic specimens can be measured by utilizing the ductility index (DI) which is the ratio of the mid-span deflection at the ultimate strength point (δult.) and the mid-span deflection at the first cracking point (δfc) based on a condition that the ultimate stress (σult.) must be greater than the first cracking stress (σfc).

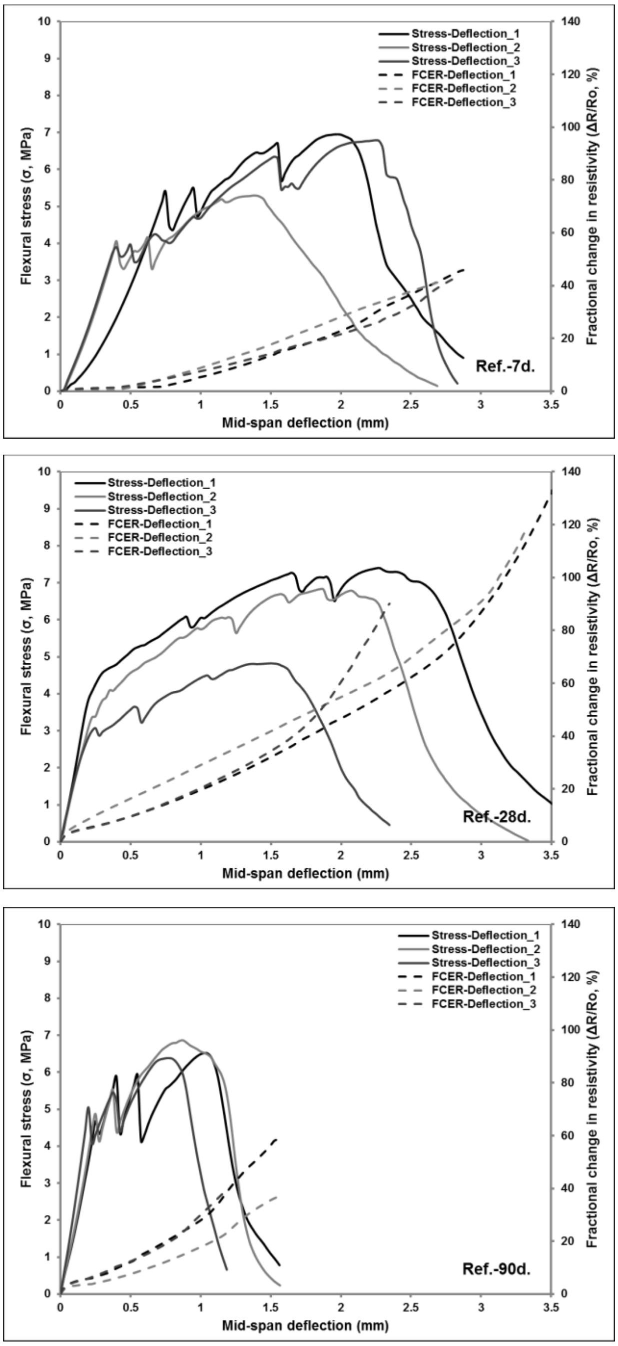
The stress/deflection curves of the developed MCC, in the present study, for different curing ages together with the self-sensing behavior can be seen in Figures (2 and 3). In this section, the discussion is limited to demonstrate the mechanical behavior of the developed prismatic mixtures, while the next section will cover the self-sensing behavior exhibited by them. From the stress/deflection curves for the different curing ages above, the previously mentioned desirable characteristics were specified and plotted. Within Figure 4, the mid-span deflection values at the first cracking positions of the different manufactured mixtures at different curing ages can be seen. On the other hand, the mid-span deflection values of the corresponding mixtures at the ultimate flexural strength were demonstrated within Figure 5. The corresponding stresses at the first and post cracking points were presented in Figures 6 and 7, while the ductility index and the ratio of the ultimate strength to the first cracking strength were showed in Figures 8 and 9, respectively. From Figure 4 it can be noticed that the general behavior trend of the first cracking deflection values is the starting with small values of around 0.10 to 0.15mm at 7 days old with an increase up to around 0.20 and 0.30mm at the 28 and 90 days, respectively followed by some decrease at 180 days of curing ages. This trend encompasses the manufactured mixtures except the control mixture at 7 days old. The same trend of deflection values can also be seen in Figure 5 but with the deflection values at the ultimate strength positions except the behavior of the control specimens, which exhibited the inverse trend. This case reflects the enhancement characteristics of adding the micro-scale carbon-based material within the cement-based matrices with the progression of their ages to extend their ductility with multiple cracking behaviors under flexural loadings. Like what has been observed in Figure 4, the trend of the corresponding stress values at the first cracking positions were similar as can be noticed in Figure 6. The stress values at these positions for the earlier ages (7 and 28 days) were around 3 MPa while those at late ages were around 5 MPa. Unfortunately, the high values of the first crack deflections and strengths are not desirable for achieving the deflection hardening behavior and this is what will be seen in the next figures. With approximately constant values of the control specimens against increasing values with the progression of their curing ages with respect to the carbon-based MCC, Figure 7 clears this behavior of the ultimate strengths of the manufactured mixtures. As previously mentioned, the control specimens get only few flexural strength development across their curing ages to reach up to 18% at the end of 180 days old. Whereas the CF - based MCC get gains reached up to 36% of their flexural strength at 180 days old from their original 7-days of curing age values. In brief, it can be concluded that the CF-based MCC registered an enhancement in the flexural strength of about 20% compared with the control specimens at the end of 180 days.

From the calculated values of ductility index (DI) (Figure 8) it can be seen easily that all of the fabricated mixtures exhibited a deflection hardening behavior at all the adopted curing ages. In addition to the DI criterion, it can be seen in Figure 9 another representation of the developed mixtures in terms of deflection hardening which is the ratio of the ultimate flexural strength to the flexural strength at the first cracking position. This figure reflects the stress development with the curing ages together with Figure 7. All mixtures crossed the limit of 1, which is the condition of deflection hardening behavior of the prismatic specimens under flexural loading. As an average, all mixtures reached up to around 1.5 at all the adopted curing ages.

4.2 Self-sensing behavior under flexural test

Generally, the FCER/mid-span deflection curves can be divided into three parts according to their slopes that have been obtained from the results shown in Figures (2 and 3) which are due to the progressive load levels. It can be seen that the first slow slope is bounded between the starting point of loading and the first cracking position which is located at the end of the linear section of the stress/mid-span deflection curve. The second part of the FCER/mid-span deflection curve can be specified from the end of the first part up to the end of the load carrying ability of the stress/mid-span deflection curve before occurrence of failure which can be distinguished by fast loss in the ability of the specimen to carry the applied flexural stress. The slope of this part of the FCER/mid-span deflection curve is higher than the first part of the same mixture. The third part of this curve begins at the end of the second part up to the end of the loading process which has generally the highest rate of change in FCER.

Figure 10 abstracts the average values of FCER of the different mixture specimens at the considered curing ages at the first crack position of each specimen, which can be regarded as the most important part within the loading process. From this figure, it can be noticed clearly the ability of CF-based MCC on diagnosing and sensing the first crack position in terms of FCER at most curing ages. At 7 days old, the developed CF-based MCC exhibited an improvement of 171% in FCER as compared with the control specimens. After 28 days of curing, it can be seen that the control specimens achieved the highest value of FCER. An improvement in FCER of 103% was registered at 90 days of curing for



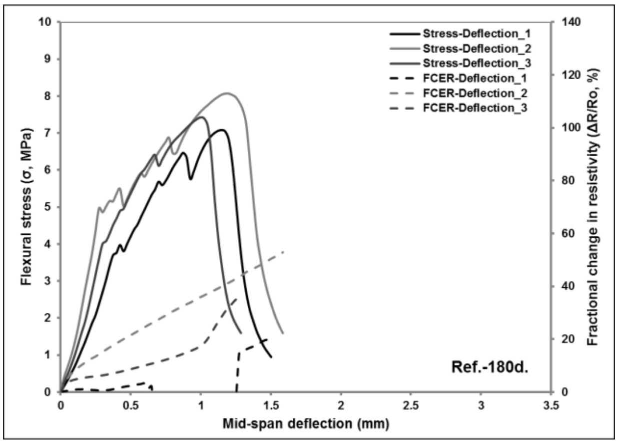


Fig. 2. Flexural stress and FCER versus mid-span deflection of the plain mixtures at different curing ages.

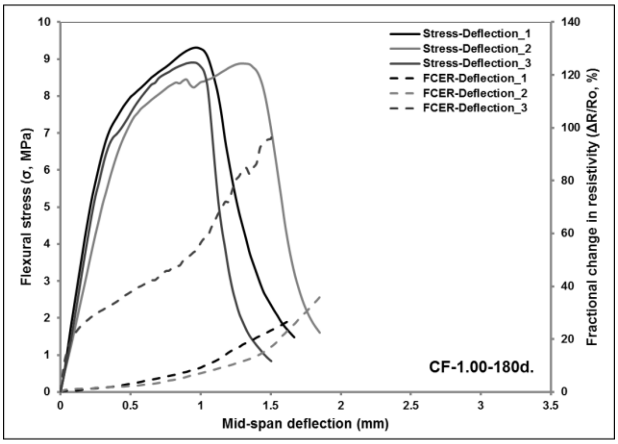
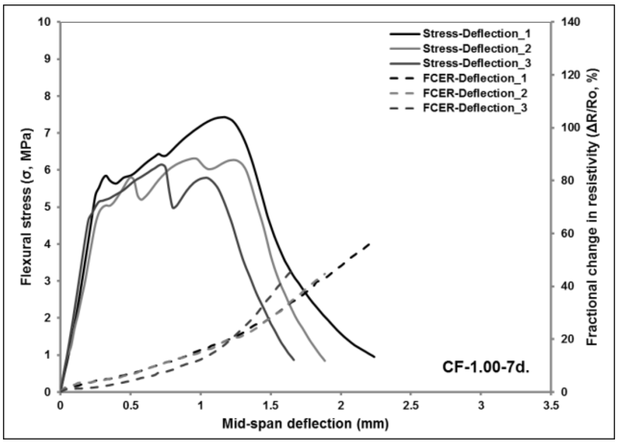
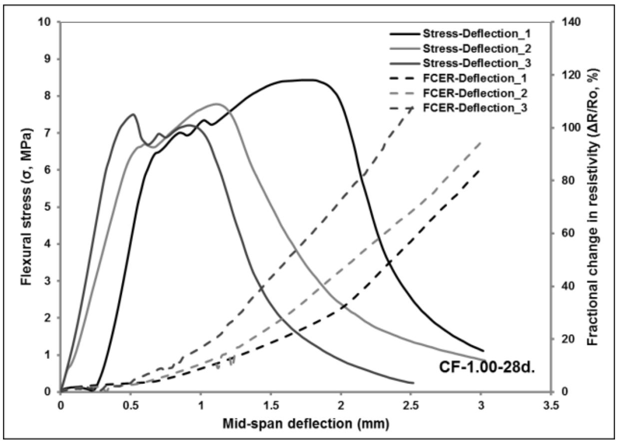
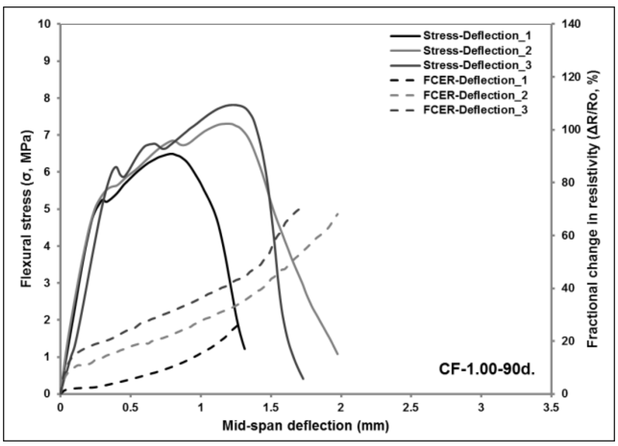


Fig. 3. Flexural stress and FCER versus mid-span deflection of the CF-based mixtures at different curing ages.







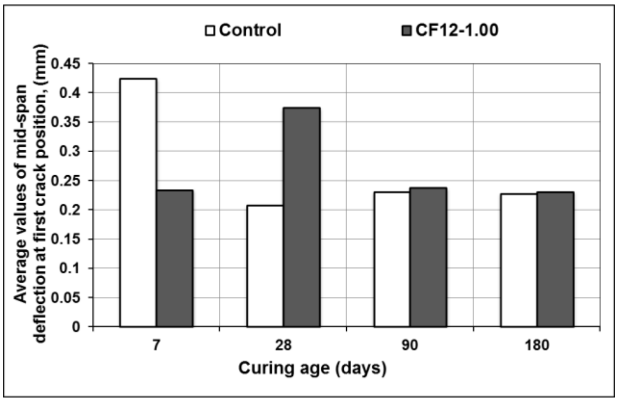


Fig. 4. Mid-span deflection (average values) at first crack position for the adopted curing ages.

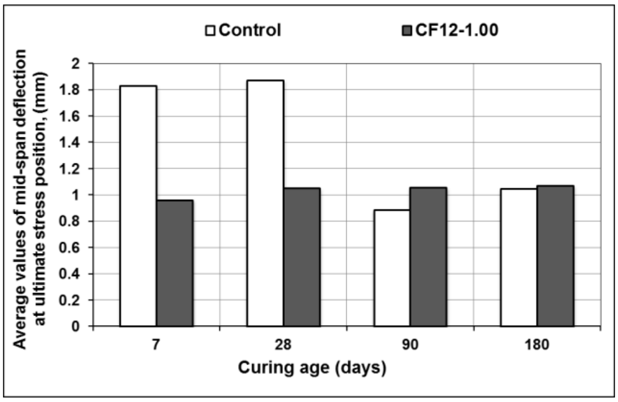


Fig. 5. Mid-span deflection (average values) at the ultimate stress position for the adopted curing ages.

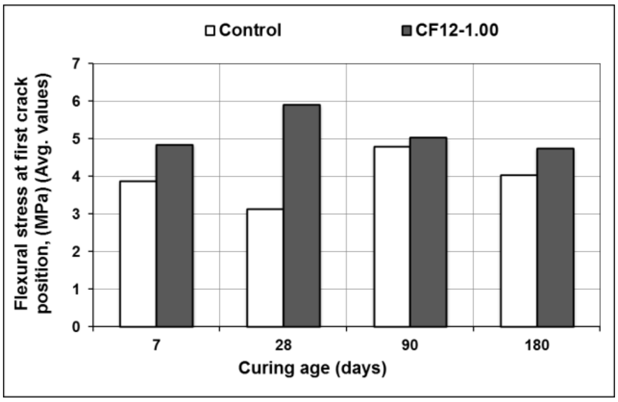


Fig. 6. Flexural stress (average values) at the first crack position for the adopted curing ages.

the CF-based MCC. Moreover, at the end of 180 days, the improvement in the FCER was 73% for the carbon-based cementitious composite. As a general behavior, it can also be concluded from this figure that the self-sensing ability in the beginning of the loading process of the developed MCC was magnified with the progressive curing ages.

The changes in the resultant values of FCER due to the same applied flexural stress but with different ages of curing are due to the development of hydration process with the age progression and the effects of the hydration products on the electrically conductive networks within the cement-based matrices which lead to these

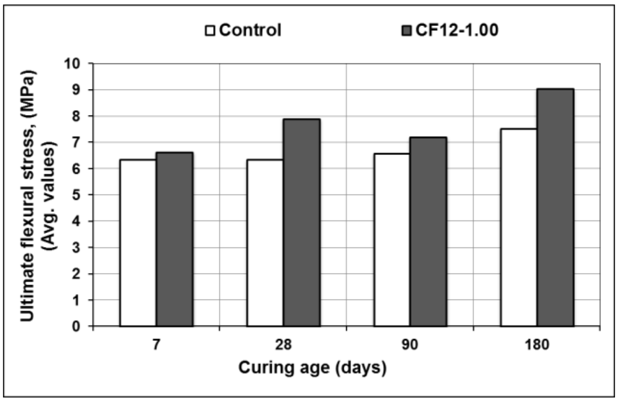


Fig. 7. Ultimate flexural stress (average values) for the adopted curing ages.

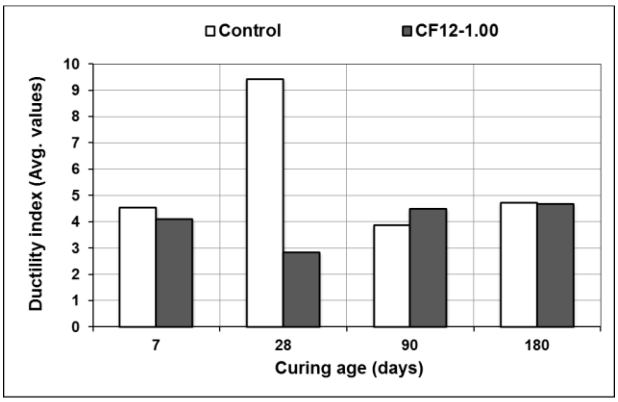


Fig. 8. Ductility index (DI) (average values) for the adopted curing ages.

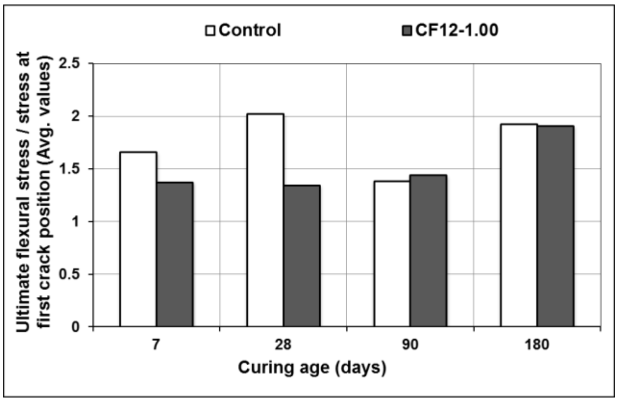


Fig. 9. Ratio of the ultimate flexural stress/stress at first crack position (average values) for the adopted curing ages.

deviations. This fact clears the big changes that have been occurred in the mixtures during hydration process between 7 and 28 days. After 28 days, no big differences can be seen in the FCER values which indicate the bounded activity of the hydration process at the advanced ages of curing after this age. The effect of adding the conductive materials within the cementitious composites on the self-sensing ability can also be noticed clearly. It can be pointed out that the self-sensing capability in terms of FCER improves with age, which is important in order to ensure the continuity of monitoring the structural behavior of the monitored structures/infrastructures. This gives an indication of the durability of the developed composites to use as a stress and damage sensor.

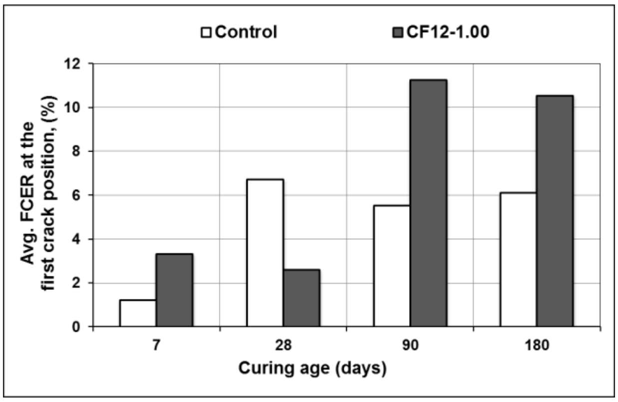


Fig. 10. FCER (average values) at different curing ages.

5 Conclusions

Mechanical and self-sensing behaviors of cementitious composites incorporating micro-scale CF were investigated under monotonic four-point bending. The following conclusions were drawn:

- The manufactured prismatic plain cementitious composite specimens and those reinforced with micro-scale CF exhibited a deflection hardening behavior under monotonic four-point flexural loading.
- The CF-based cementitious composite specimens enhanced the flexural strength characteristics at all the

adopted curing ages with 20% of enhancement compared with the plain specimens at the end of 180 days of curing.

- The incorporation of CF within the ECC matrix has an important role in improving the self-sensing ability of the monitored structures/infrastructures in terms of stress and damage diagnosis especially in later ages.
- The self-sensing ability of the developed MCC under monotonic four-point bending was magnified with the progress in curing ages.

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