

Piezoresistivity in self-compacting concrete mixes with hybrid additions of carbon fibers and nanofibers

Javier Puentes^{1*}, Irene Palomar¹, and Gonzalo Barluenga¹

¹Universidad de Alcalá, Architectural School, c/ Santa Úrsula, 8, 28801, Spain

Abstract. Self-sensing properties of concrete structures can be achieved through the incorporation of additions in carbon-based materials (CBM) that modify electrical properties and provide piezoresistive (PZR) properties to the cement paste. PZR in pastes and mortar mixes with different types of carbon-based materials such as fibers of different sizes has been extensively tested. However, very limited studies on self-sensing properties in concrete are available due to the lower content of paste volume that leads to a decrease of concrete PZR. However, self-compacting concrete (SCC) can be an ideal candidate to implement PZR, due to two fundamental traits: the larger amount of paste of SCC compared to a conventional concrete and larger volumetric fraction of fiber can be incorporated due to a reduced effect on SCC workability and consistency. The present study aims to assess PZR properties of SCC with carbon-based components of different sizes. Combining carbon nanofibers (NFC) and carbon fibers (CF), in hybrid systems, could lead to obtain SCC with self-sensing properties identifying their effectiveness thresholds. The self-detection performance of PZR-SCC samples under mechanical stress was verified by resistivity and PZR experimental tests.

1 Introduction

The incorporation of self-sensitive properties in cement-based materials, seeking to make the material itself part of the real-time monitoring systems of structural conditions - Structural health monitoring (SHM) [1-2], has significantly advanced thanks to the implementation of new properties using carbon-based materials (CBM). Establishing efficient monitoring systems, in the first place, aims to reduce the number of resources directed to inspection, within a protocol to increase the durability and extend the service life of concrete structures and infrastructures [3-4]. A second long-term aim is to incorporate the self-sensing property as a tool, as part of a stochastic structural system. The system should act according to the data acquired in real time against accidental actions and punctual environmental effects (earthquakes, snowstorms, wind, floods), to improve the structural response (resilience) of the structure [3]. It is mainly proposed initially at the study level to collect data and finally as an immediate response action. The response will be conditioned according to the strength of the subjected action and according to the implemented and self-controlled response system, either by energy dissipation or transfer, as part of components of a smart system [5].

The stress measures that the concrete structure is subjected to, must be able to be performed in any location of the structure, for this reason, the need to use the concrete itself as a sensor material allows the viability of the system compared to the independent

sensors inside/attached the structure that limits the area of action and the reliability of the data as a function of the service life of the sensor [3].

The following study presents the development of a concrete with addition of carbon nanofibers and carbon fibers to obtain the property of self-sensing. Through piezoresistivity, the variation of electrical resistivity depends on the applied stress [3]. The study was carried out by testing independently a mixture of two sizes of carbon fibers (6mm and 12mm) and Carbon nanofibers in a self-compacting concrete (SCC). In the mixes, the dispersion, workability of the mix, direct resistivity measurement and finally the variation of the electrical resistivity by subjecting the mix to compressive loads (piezoresistivity) were studied. With these data it is intended to establish the most effective fiber amount thresholds to be used as final reference values, combining both fibers, considering the synergistic effect of using different fiber sizes [6,7].

The use of SCC is presented as an advantage over ordinary Portland concrete (OPC) [8,9], on the one hand it allows a homogeneous distribution of the fibers and on the other hand it can allow a greater effectiveness of the self-sensing property due to the greater volume of paste compared to the volume of the material. The only limitation is to establish the dosage thresholds that compromise the self-compactability due to the use of fibers.

The present study is preliminary to the development of a hybrid mixture of different sizes of carbon materials, nanofibres and fibres. The aim is to develop a

* Corresponding author: javier.puente@uah.es

design technique for mixtures with CBM additions with much more efficient of self-sensing properties. The design is based on the efficiency and synergy effects at the nanostructural level in the paste and its potential application to different carbon-based additions.

2 Experimental program

2.1 Materials

For the present study, a SCC with the addition of limestone filler was used with a siliceous gravel with a maximum size of 12 mm. The dosage of self-compacting concrete and the compatibility and its effect with carbon fibers have been tested in previous projects and the results have been published [10,11,12]. On this dosage, the variation of the CBM was carried out. The properties of the fibers used in this study are shown in Table 1.

Fiber volume fraction percentages of 0.5%, 1%, 1.5% and 2% were used for both nanofibers and carbon fibers.

The carbon fiber was used as a mix of 50% of 6 mm and 50% of 12 mm size.

Table 1. CBM properties.

Material	Carbon Fiber	Carbon Nanofiber
Length (μm)	6 x 10 ³ 12 x 10 ³	30
Diameter (nm)	7x10 ³	20-80
T. Strength (GPa)	4.0	-
Resistivity (Ω m)	1.5x10 ⁻⁴	1.0x10 ⁻³
Density (g/cm ³)	1.80	1.97
Carbon content	95%	-

In the initial part of the project, the effect of each of the fiber sizes was studied individually, but with the objective of proposing a hybrid system of fiber size, looking for the tunnel effect between the different sizes and types of fibers to improve their efficiency within the paste [13].

2.2 Methodology and testing setup

Two types of SCC specimens were made, first a cubic specimen of 100 mm for resistivity measure and prismatic specimens of 40 x 40 x 160 mm for PZR. The second specimen was made by embedding 4 meshes of stainless steel in the whole section of the specimen as shown in figure 1. The mesh works as a connection terminal for the resistivity and piezoresistivity test. The figure 1 shows the distribution of the terminals.

2.2.1 Electrical properties

The electrical properties were done according to Ohm's law, Eq. (1).

$$R = V/I \tag{1}$$

Where R is the resistance (in Ω), V is the potential difference in volts, and I is the current in amperes. Resistivity ρ in (Ω.m) can be calculated as Eq. (2).

$$\rho = R*(S/L) I \tag{2}$$

Where R is the resistance in Ω, S is the cross section of the specimen in m² and L is the length in m.

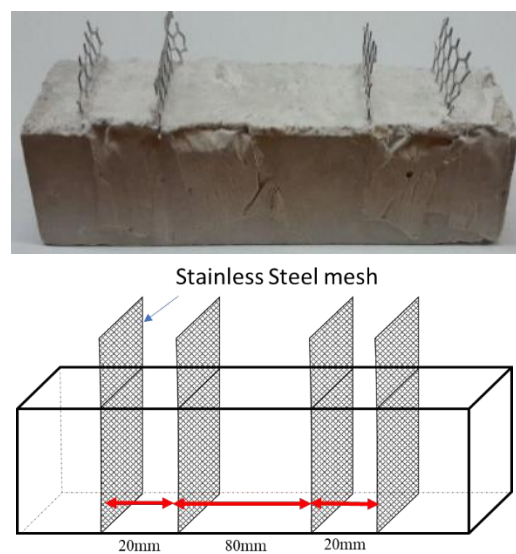


Fig. 1. Concrete specimen of 40 x 40 x 40 mm with stainless steel mesh terminal.

2.2.2 Resistivity measure

As a preliminary step, the initial resistance test was carried out, figure 2 shows the test setup.

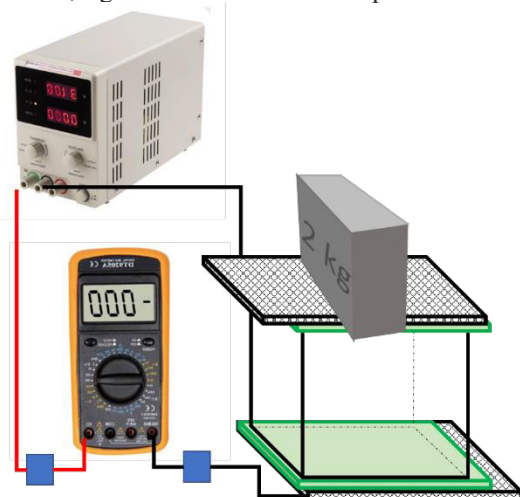


Fig. 2. Resistivity test setup by Standard UNE 83988-1 for 100 x 100 x 100 mm specimen.

The electrical resistance of the specimens was carried out with saturated specimens. To ensure the degree of saturation in all specimens tested, these were cured by immersion in water, the specimens were removed from the curing tank immediately before testing and the excess surface water was removed to perform the test. In this way it was checked the conductivity of the material against the flow of electric current and as an initial reference measure for the dosage quantity thresholds. The values obtained together with the workability of the concrete are the ones that will condition the amount of carbon fibers used in the concrete mix.

The electrical resistance of the material was evaluated with the Standard UNE 83988-1 at ages of 7, 28 and 60 days. The procedure described in the standard was followed and the measurements were performed according to the cubic specimen of 100 x 100 x 100 mm, using a wet sponge to guarantee a good contact with the surface of the specimen as shown in the figure 3. The sponge wetting and wringing process was performed before each individual data collection to ensure repeatability of test conditions for all specimens.

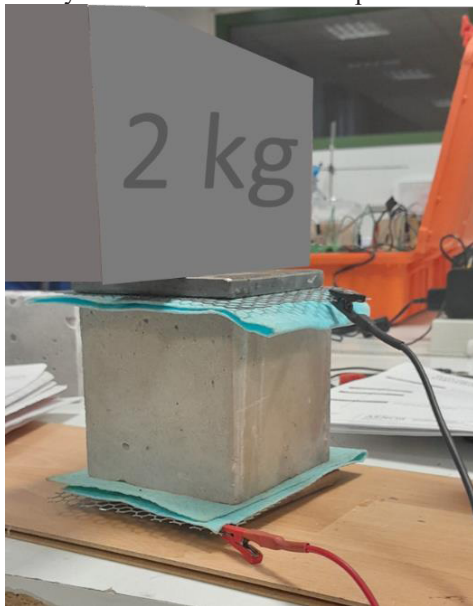


Fig. 3. Resistivity test setup by Standard UNE 83988-1 for 100 x 100 x 100 mm specimen.

In the procedure, 10V of current were supplied. The resistance total, $R(t)$ of the system is the result of the concrete resistance $R(c)$ + sponge resistance, $R(s)$, to obtain the value of $R(c)$, it is measured only the sponge $R(s)$ and subtracted from the final value obtained, in this way the value of $R(c)$ is obtained.

2.2.3 PZR Measure

The PZR at 60 days through the application of a test protocol. Designed to measure the variation of the electrical resistance as a function of the applied compressive load, as shown in figure 4.

A multi-test compression machine with a load capacity of 20 T was used for the PZR test. Data logging was performed using a Campbell model CR100X to

collect stress, strain, electrical resistance, and current data while testing.

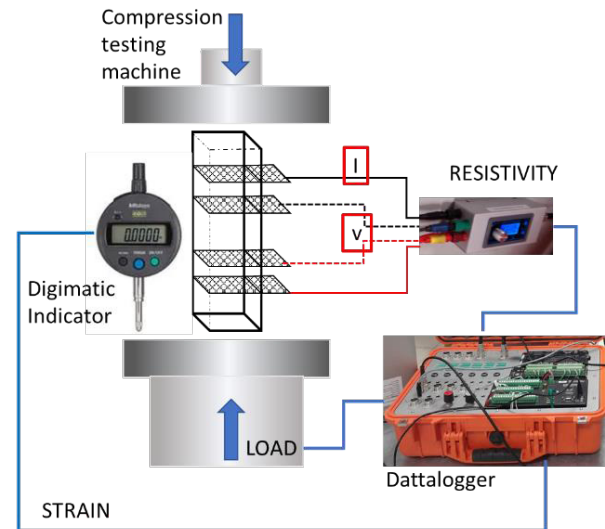


Fig. 4. PZR test setup for 40 x 40 x 160 mm specimen.

The tests were performed in continuous loading and unloading cycles between 0 and 20 kN and the rate of increase/decrease was 1kN/sec. The current supply used at the outer terminals of the specimen was 12V (CC).

Before testing, the system was supplied with current for 15 min to try to equilibrate the system and avoid polarization. Previous studies have described how the increase of the resistance during the measurements because of the dielectric properties associated with the capacitance and polarization of the CBM in cement composites [14]. The strain measurement was carried out with a Mitutoyo digimatic indicator, figure 5. The deformation was preliminarily measured in reference to the displacement of the compression machine plates as shown in fig.5, the next stage of the study involves the use of strain gauges to determine the unit deformation of the material itself.



Fig. 5. PZR test setup for 40 x 40 x 160 mm specimen.

The self-sensitive behavior is measured through the ratio of the fractional change of the resistivity ($FCR - \Delta\rho/\rho_0$) and the applied load [15]. The result and the resistivity variation were compared between the different dosages through the FCR.

3 Experimental results, analysis, and discussion

The electrical resistance and PZR tests were carried out in accordance with the recommendations of the standard, UNE 83988-1 (20⁺2C and 40%RH). Before to the electrical resistance test and PZR tests, reference measurements were taken to compare the two types of specimens at 7 days, following the UNE EN 83988-1 standard of 100x100x100mm and in the specimens of 40x40x160mm without the weight and connected directly to the external terminals. The results are shown in fig 6.

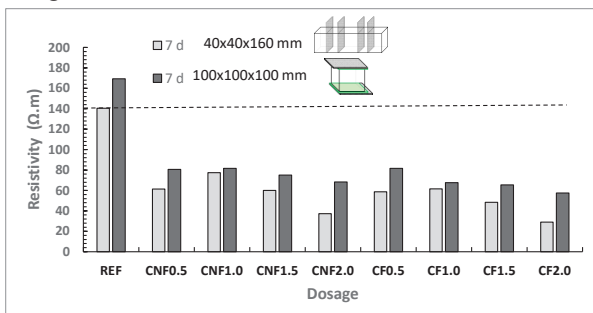


Fig. 6. Electrical resistance results comparison according to UNE-83988-1 for the two types of specimens at 7 days.

3.1 Resistivity Results

The results of the resistivity test according to the UNE-EN 83988-1 standard, have shown a reduction of resistivity in general, both with the use of CNF and CF.

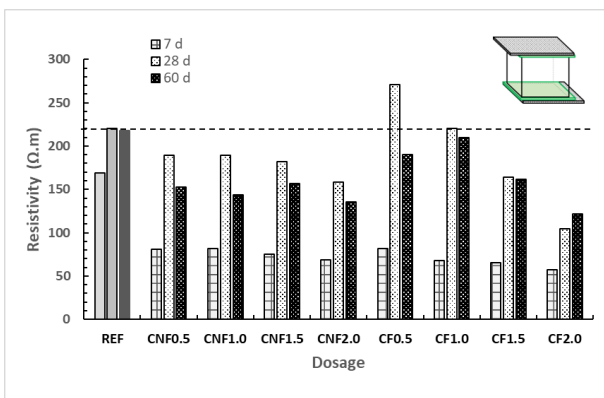


Fig. 7. Results for resistivity test of: Ref and the amount variation of CNF and CF.

The evidence of the reduction of the resistivity value is observed from the first measurement (7 days). In the case of the addition of CNF there is a reduction of the resistivity compared to the reference, but at 28 days there is no improvement according to the amount of CNF, figure 7. In the case of CF there is a clear reduction in resistivity depending on the amount of fiber used. Up to 50% is reached with respect to the reference dosage and especially at 28 days, figure 7.

3.2 PZR Results

In figure 8, it can be observed that the reference dosage does not show a variation of the electrical property of sensitive resistance against the applied load (20 kN).

The applied stress in the 40x40x160mm prismatic specimens is 12.5 N/mm². The average strength in the Ref concrete is 32.5 N/mm² and the average strength in the SCC with CNF is 29.5N/mm², while the average strength for the concrete with fibres is 30 N/mm². The applied stress in the cyclic loads of the PZR test is around 30% of the ultimate strength (12,5 N/mm²). In no case does it exceed 40% of the ultimate strength of the concrete, set as the stress limit for the elastic modulus. The specimen is not being affected or damaged while the PZR test is being performed.

For the dosage with variation of CNF in different proportions, it is observed that the variation of the FCR is more accentuated as the quantity of CNF in the mixture increases with respect to the reference dosage (REF). At 2% CNF volume fraction (VF), the greatest variation of the resistance is identified, figure 7.

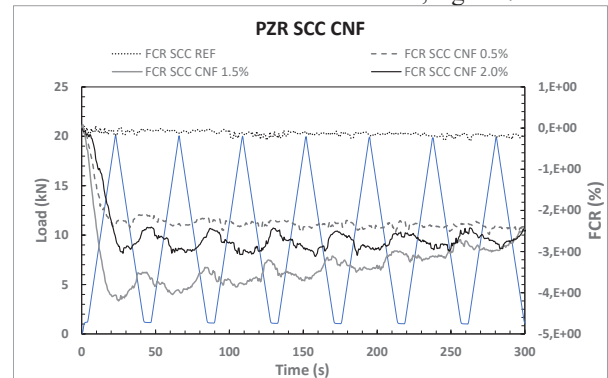


Fig. 8. PZR Results for SCC with CNF, Result test relative to Ref dosage.

In the dosage with variation of CF, figure 9, a variation in the resistivity becomes more evident as the percentage of fibre VF is increased. The limit of 2% has been established according to the workability of the mixture and its self-compacting consistency. In this figure

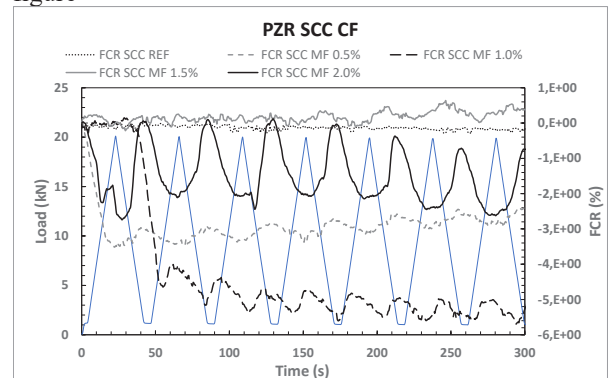


Fig. 9. PZR Results for SCC with CF, Result test relative to Ref dosage.

In the Fig 9., the SCC MF 1.5% mixture shows a difference in the sign variation of the results with respect to the others. This result may be related to the possible polarization of the sample. This effect is determined by the complex electrical behavior of the carbon

components that it have established in previous studies [14]. The literature recommends the use of AC instead of DC to avoid these phenomena.

Possible offset/delay of the values are caused in some cases by equipment resolution and data acquisition latency; however, this factor is taken into account in the next stage of the study with the improvement of the PZR test procedure. It is working on the incorporation of the gauge factors (GF) as a comparison reference using strain gauges as measuring components. Between the two additions, CF is more effective. the results show a greater variation of resistance between the maximum and minimum values of the load.

4 Conclusions

The results obtained in this preliminary study, have been able to verify the variation of the electrical properties of the material, due to the use of carbon-based additions.

The test to evaluate the response of the electrical resistance properties of the material, under cyclic stress loading has also been verified. Although with the limitations of the strain measurement equipment due to its resolution and reliability in data collection. However, it has been possible to establish a series of positive conclusions in the PZR properties, these serve as a preliminary reference and are part of the basis for a second stage of the study that incorporates: strain measurements with strain gauges to obtain comparative results of gauge factor (GF) and greater accuracy of results.

A piezoresistive property has been identified in self-compacting concrete with the individual addition of carbon-based materials, carbon nanofiber and carbon fiber. In both cases the self-sensitive property responds to loading/unloading cycles.

In all the tests performed, the polarization of the specimen has been limited or at least reduced in some cases, by simply stabilizing the system by supplying current to the specimen at least 15 minutes before performing the test. In other cases, the polarization has not been controlled and has affected the variation of the sign of the results. However, the use of FCR coefficient values with respect to the initial values, try to establish methods of comparison of results.

It was possible to identify a more efficient behavior with the use of CF and in the maximum amount studied (2% VF). In this case the FCR was more sensitivity to the applied load.

In the case of CNF, no variation in the workability of the mixture was established as the dosage of the nanofibers was varied, in the next stage it is proposed to continue increasing the amounts to establish the percolation threshold of the CNF and combined with CF.

In the case of CF, the superior limit of dosage should not be higher than 2% of VF as it compromises the workability and the property of self-compactibility of the concrete.

Once of the values for each of the additions have been established, a hybrid addition of the two sizes of fibers studied will be carried out. The amount of 2% of

CF will be used as a reference and varying the amounts of CNF addition. It is intended to exceed the reference quantity of the present study, since no limit of use was established that would compromise the workability of the mix with the CNF.

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References

1. S. Taheri. *Constr Build Mater*, **204**,492-509, (2019)
2. F. Azhari, *Cement-based sensors for structural health monitoring*, Master Thesis. UBC (2008)-
3. A. D'Alessandro, F. Ubertini, A. L. Materazzi, *Int., j. civ. environ. eng.*, **10** 5, 599-604. (2016)
4. M. Abedi, R. Fanguero, A. Gomes Correia, *Constr Build Mater*, **310**,125-139 (2021)
5. W. Gilewski, A. Al Sabouni-Zawadzka, *Arch. Civ. Mech. Eng.* **15**, Issue 2, 469-478 (2015)
6. F. Azhari, N. Banthia, *Cem Concr Compos.*, **34** 7, 866-873 (2012)
7. Alonso, M.C., Puentes J., *Materials* **13**, no. 5: 1106 (2020)
8. I. Gull, M.A. Tantray, *Mater. Today: Proc.*, **38**, Part 5, 3181-3189 (2021)
9. L. Ferrara, Y-D Park, S.P. Shah, *Cem Concr Res*, **37**, 6, 957-971, (2007)
10. J. Puentes, G. Barluenga, I. Palomar., *Proceedings of International Symposium on Nanotechnology in Construction, NICOM 5.* 24-26 May 2015, Chicago, USA (2015)
11. J. Puentes, G. Barluenga, I Palomar, *Constr Build Mater.*,**73**, 89-96, (2014)
12. Arvind Kumar Cholker, M.A. Tantray, *Mater. Today: Proc.*, **43**, Part 6,3774-3779 (2021)
13. B. Han, X. Yu, J. Ou, *J. Mater. Sci.*, **45** 14, 3714-3719. (2010).
14. A.O. Monteiro, A. Loredó, P.M.F.J. Costa, M. Oeser, P.B. Cachim, *Constr Build Mater.*, **154**, Pages 1079-1086, (2017)
15. B. Han, S. Ding, X. Yu, *Measurement*, **59**, 110-128 (2015).