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Electrical conductivity of self-monitoring CFRC

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Abstract

The present work is concerned with the analysis of the electrical conductivity in carbon fiber reinforced cement composites and of the main parameters influencing the phenomenon: fiber volume fraction, fiber length, hydration time and sand–cement ratio. AC measurements were carried out on CFRC (carbon fiber reinforced cement) and the experimental results were analyzed using the percolation theory. The present study could be useful for the adoption of CFRC as a smart material.

Keywords: Carbon fiber; Cement composites; Electrical resistivity; Percolation theory

1. Introduction

The main goal of much research in recent years, in the monitoring field, has been the development of "health monitoring systems", which generally involve the extensive use of sensors, embedded in structures during the production process, or externally bonded in critical positions.

A valid alternative to the use of sensors is the adoption of fiber-reinforced materials with a small percentage of conductive fibers, commonly called "self-monitoring" materials [1,2].

Previous studies [3,4] demonstrated that moist concrete has a resistivity of about $10^4\,\Omega$cm and this increases up to $10^{11}\,\Omega$cm for oven-dried concrete (105 °C). The plain concrete is an insulating material. The addition of fiber improves the mechanical performance and strongly modifies the electrical conductivity so producing a "self-monitoring" capability which is derived from the correlation of the damage level to the electrical behavior. Therefore the material itself becomes a sensor, is durable and allows the entire structure to be checked.

In this work, attention is focused on CFRC (carbon fiber reinforced cement composites), with the goal of characterizing the material from the electrical point of view. This is a prerequisite for successive developments which will allow the damage evolution to be evaluated by simple electrical measurement ERM (electrical resistivity monitoring).

In this initial research the principal objective is the evaluation of electrical conductivity phenomena, their modeling and the definition of the main parameters which affect the mechanical and electrical behavior, such as the effect of matrix microstructure, percentage of fibers, relative humidity, etc. The percolation theory is adopted to try to analytically model the observed phenomena because its statistical–mathematical nature appears capable of treating the multiphase system under analysis.

2. Experimental procedure

2.1. Materials and specimens

To make the mortar specimens, Portland cement (type I, 52.5 R) and standard sand, as prescribed by the UNI EN 196-1 (1996), were used. The geometrical

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and mechanical properties of the carbon fibers (PAN type) used in this analysis are listed in the following Tables 1 and 2.

To obtain a better fiber dispersion and mortar compactness, silica fume (15% by weight) was used. To recover the loss of workability produced by the silica fume a fluidifying agent was added. The specimens were made from a mix with the following ratios: water–cement ratio (w/c) = 0.45; sand–cement ratio (s/c) = 1; silica fume by 15% weight of cement; fluidifying-cement weight ratio = 0.4%. A standard mixing procedure was used. The fluid mortar was put into plexiglass moulds and was compacted using a shaking table. The specimens had the dimensions of 40 × 40 × 160 mm and were cured at constant temperature at 20 °C for 24 h. The tests were made in the laboratory atmosphere (temperature: +20 ± 2 °C; relative humidity: 65 ± 5%).

2.2. Electrical measurements

The electrical conductivity of the carbon fiber cement composites is fundamentally of two types: electrolytic and electronic. The first involves the matrix because it is connected to the ionic movement in the evaporable water of the cement composite [3].

The second one is connected with the movement of the free electrons present in the conductive phase, which are, in our case, the carbon fibers. In the present work a prevalence of the electronic part in the electrical conductivity is expected. Moreover, to eliminate the electrolytic effects, particularly the effect of the electrode polarization, an alternate current (AC) was adopted. In effect these polarization phenomena will not disappear but they can be modeled as resistance series or as a parallel capacitor.

For AC the Ohm law assumes the form $V = ZI$, where V, Z and I are phasors and they contain the information about the phase and the modulus (I is the current expressed in Ampere, V the potential in Volt and Z the impedance in Ohm). In the case of a parallel C–R arrangement, the impedance has the following expression:

$$Z = \frac{R}{(1 + \omega^2 C^2 R^2)^{1/2}}$$

$\omega = 2\pi f$, f is the frequency expressed in Hz, C is the capacity expressed in Faraday and R is the resistance expressed in Ohm.

The electrical measurements were conduced in a frequency range of 10–100 kHz and with a voltage of 10 mV. The frequency upper limit was chosen as a reference value for the electrical resistance, because at 100 kHz the practically constant value of the impedance indicates that it is close to its real part (pure Ohm resistance) [5,6].

The electrical resistance measurements were carried out using a Galvanostat/Potentiostat PARSTAT 2263-1 made by Princeton Applied Research (USA), with Power Suite acquisition processing data software (Fig. 1). The device has five electrodes: Ground (G), Working (W), Sense (S), Counter (C) and Reference (R) electrode. The two- and four-probe methods were used in the electrical measurements, as depicted in Fig. 2.

In the four-probe method, the current flows between C and W placed on external electrodes, while the potential is measured between R and S placed on the inner ones.

Table 1
Carbon fiber mechanical properties

Density (g/cm^3)	Tensile strength (GPa)	Young modulus (GPa)	Elongation at break (%)	Carbon content Wc (%)	Electrical resistivity (Ωcm)
1.75	2	180–240	1.20–1.30	>95	10^{-2}–10^{-3}

Table 2
Carbon fiber geometrical data

Diameter μ(m)	8	8	8
Length	4 μm	3 mm	6 mm

Fig. 1. Equipment used for electrical resistance measurements.

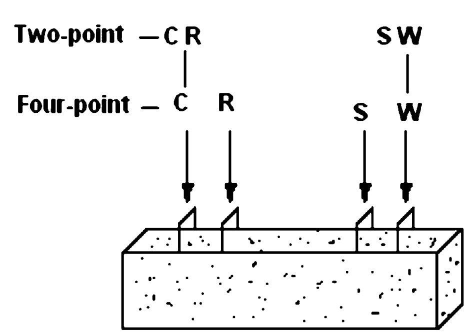

Fig. 2. Two- and four-probe electrical measurements: electrode set-up.

2.3. Electrical configuration analysis

Efficient electrical contacts and a suitable choice of the electrical configuration are very important to ensure reliable measurements and test repeatability. On the basis of some models available in literature [6–9], five different configurations were analyzed (S1, S2, S3, S4 and S5).

For the S1 configuration the electrical resistance measurements were made by two copper electrodes 0.3 mm thick and with 40 × 18 mm transverse dimensions, 8 mm set in the cement composite (Fig. 3). The electrodes are at a distance of 120 mm. In the S2 configuration the electrodes are made of lead plates, 1 mm thick and with 40 × 50 mm transverse dimensions, clamped on the specimen with a G-clamp. To guarantee uniform contact pressure between vice and electrodes, wood cubes were inserted. A paste of ethyl alcohol and carbon dust was spread on the specimen ends. The S3 configuration is a variation of the S1 model and makes four-probe measurements possible. The internal electrode distance is 80 mm, while the external ones are at a distance of 120 mm. The S4 set-up can be considered as a combination of S1 and S2, and makes four-probe measurements such as S3 possible. In the S5 configuration, similarly to the Chung et al. analysis [8], the electrodes are made of four copper wires with a diameter of 0.5 mm, wrapped on the specimen. The internal electrode distance is 80 mm, while the external one is fixed at 120 mm. The electrical contact between copper wire and specimen was made by a conductive silver paint.

All the electrical measurements were carried out after 5 days of curing. In Table 3, for all five configurations, the average electrical resistance measurements, made at a frequency of 100 kHz, are reported, with the standard deviation and the coefficient of variation (CV).

From the comparison of the results it is clear that the smaller scatter in the experimental data, so producing the most reliable system for electrical measurements, is obtained with the S3 configuration. The next best one appears to be the S4 set-up, which is also a four-probe model. The two-probe configurations produce greater coefficients of variation. This effect is probably due to the strong influence of the voids and of the aggregate concentration in proximity of the two electrodes. For the S5 configuration the dispersion in the experimental results is probably a consequence of a non-perfect electrical contact between copper and specimen, even though conductive paint is used. In the following, the S3 configuration will be adopted.

Table 3
Comparison between the results obtained using the proposed configurations

	$\overline{R}$ (Ω)	sd (Ω)	CV (%)
S1	230.5	51.4	22.3
S2	91.5	11.3	12.4
S3	37.1	2.5	6.70
S4	54.8	3.8	6.90
S5	80.9	61.6	76.0

2.4. Contact area influence

To analyze the influence of the contact area on the electrical resistance measurements, six series of specimens were analyzed. Three of them use the four-probe configuration and the remaining three the two-probe one. The electrode drowning depth was increased and, consequently, the conductive section, which passes from 3.2 cm^2 to 6.4 cm^2 and to 9.6 cm^2. Fig. 4 shows the electrical resistance relationship to the L/A ratio, where L is the electrode distance and A is the electrode contact area.

From the curves it is clear that in the two-probe configuration the electrical resistance values are strongly influenced by the electrode contact area, while in the four-probe set-up the electrical resistance is practically independent of L/A ratio. These results are confirmed in the Millard experimental work [9] on reinforced

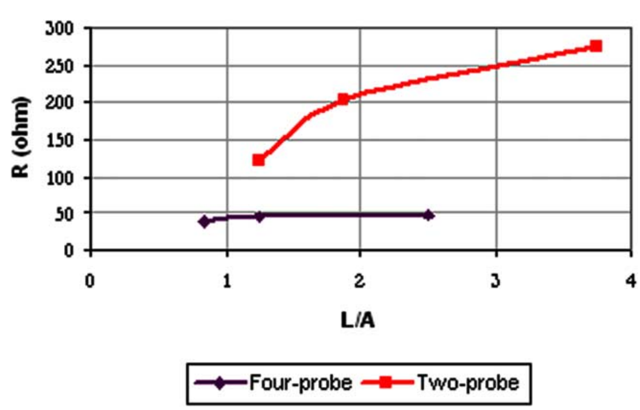

Fig. 4. Electrical resistance versus L/A ratio.

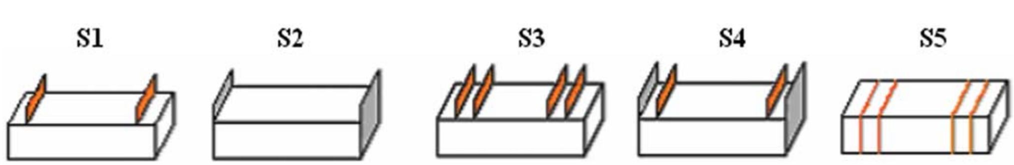

Fig. 3. Two- and four-probe electrical configurations.

concrete, where with other measurement devices, the four-probe method produces the best results.

3. Experimental results

Having defined the electrical configuration, the influence of the main physical and geometrical parameters on the electrical measurements and on the conductivity were evaluated. From the resistance data the electrical conductivity σ was obtained from the second Ohm-law:

$$\sigma = \frac{1}{\rho} = \frac{L}{RS}$$

where ρ is the electrical resistance, L is the internal electrode distance, S is the electrode conductive area and R is the measured resistance. The electrode ($40 \times 18 \times 2$ mm) is inserted into the cement composite (8 mm depth). The electric field is assumed constant and the end-effects considered negligible. This hypothesis will be analyzed more thoroughly in successive experimental work to check if it is completely correct or if some corrections are needed.

3.1. Fiber content

In Fig. 5 the experimental results obtained on CFRC specimen with fibers 6 mm long, w/c fixed to 0.45 and s/c equal to 1, after 1 day of curing, are reported.

As stated in the percolation theory [10–12], it is possible to observe that the magnitude in the electrical conductivity strongly increases with the increase the fiber content. Point (1) corresponds to a low system connection, the fiber contacts are rare and the composite conductivity is very close to that of the matrix; point (2) is representative of the value near the percolation threshold. Beyond this value-point (3) the composite conductivity increases slightly with the increase in fiber content, so further fiber addition will not produce significant variations in the system conductivity point (4). In the percolation theory, it is hypothesized that a critical value ϕ_c exists, corresponding to the strong change in the system conductivity. In effect this is not a point, but a zone, rather small, in which the conductivity value strongly increases, and after this zone the increase is more gradual. This zone is called "transition zone", and its boundaries are represented by the fiber contents ϕ_{c1} and ϕ_{c2}. With the available data it was impossible to determine the ϕ_{c1} value, which logically belongs to the 0–0.025% range, on the contrary was possible to determine the ϕ_{c2} value approximately. This will be around 0.15% of the fiber volume content in the case of the 6 mm fiber length and around 0.2% for the 3 mm ones.

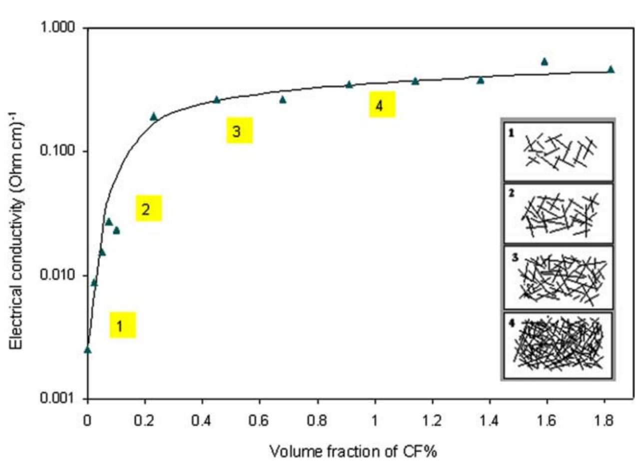

Fig. 5. Fiber content effect on CFRC specimen (fiber length = 6 mm) after 1 day of curing.

4. Effect of fiber length

The effect of CFRC fiber length on conductivity is shown in Fig. 6. As expected, the electrical conductivity increases with the increasing in the fiber length. In particular the threshold value increases when the fiber length decreases. In fact to form the conductive network a greater content of short than long fibers is necessary, because the latter are more likely to be connected themselves to form the percolation path.

From the graph it is clearly visible, for 6 mm and 3 mm fibers, the presence of a narrow zone where conductivity grows significantly; on the contrary for the fibers 4 μm long a gradual increase in conductivity has been found. This can be connected to the fact that the shorter fibers are not capable of forming a continuous conductive network that governs the system phase transition, from insulating to conducting. However the addition of fibers 4 μm long involves an increase on the conductivity degree of the system of about one order of magnitude (from 0.002519 $(\Omega\text{cm})^{-1}$ to 0.01575 $(\Omega\text{cm})^{-1}$). In the case of the fibers 6 mm and 3 mm long

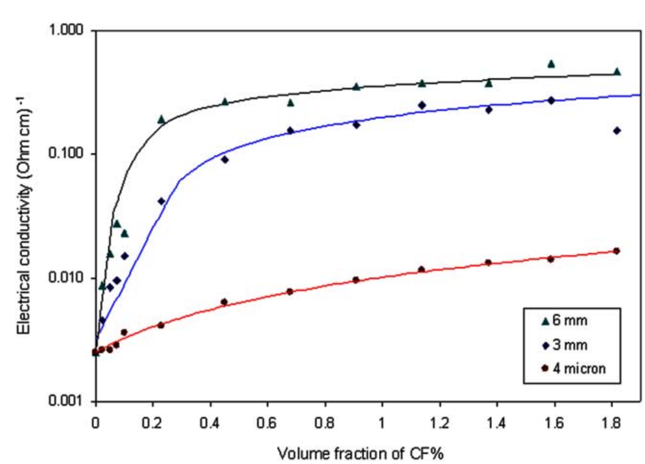

Fig. 6. Effect of the fiber length for sample in CFRC, 1 day hydration.

the increase is greater: the sample conductivity with 1.8% volume fraction of 6 mm carbon fiber is more than two orders of magnitude greater than that obtained in the specimens of mortar alone (0.002519 $(\Omega\,\mathrm{cm})^{-1}$ as against 0.455789 $(\Omega\,\mathrm{cm})^{-1}$).

It can be concluded that it is not necessary to exceed the threshold value for the fiber content and that the use of longer fibers allows the required value of conductivity to be obtained first.

5. Hydration time effect

The electrical conductivity process inside a CFRC is strictly connected with the microstructure of the material. It can be deduced that the electrical conductivity can change with the advance of the hydration process which happens in the cement matrix during the hardening phenomenon. In Figs. 7–9 the results, obtained with the three different fiber lengths adopted in the experimental program, are reported.

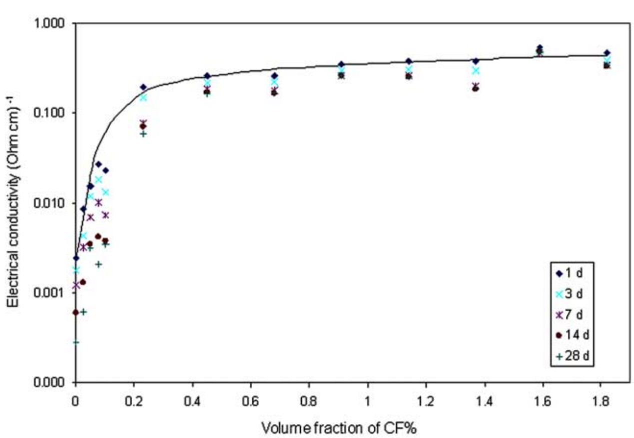

Fig. 7. Effect of the hydration time on CFRC specimens with 6 mm fiber length, s/c = 1, w/c = 0.45.

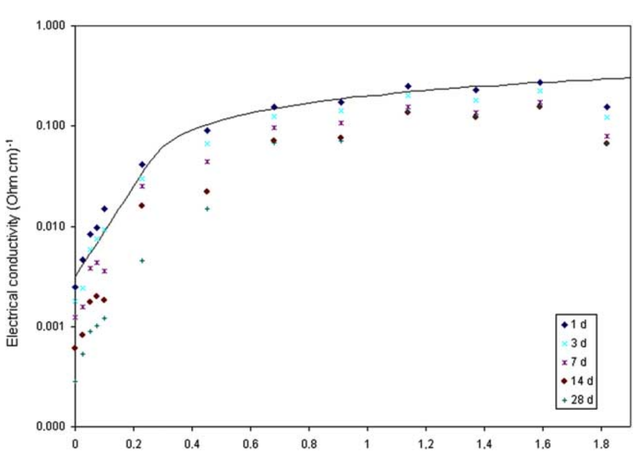

Fig. 8. Effect of the hydration time on CFRC specimens with 3 mm fiber length s/c = 1, w/c = 0.45.

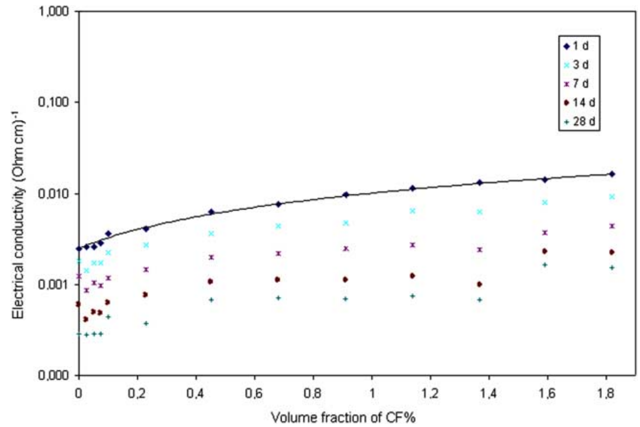

Fig. 9. Effect of the hydration time on CFRC specimens with 4 μm fiber length s/c = 1, w/c = 0.45.

From the analysis of the results it can be deduced that the hydration time has no significant effect on the conductivity of the CFRC composites, especially for the cases of 3 mm and 6 mm fiber lengths: the percolation threshold does not change with the hydration time, even if it is possible to observe that some electrical conductivity values decrease, especially in the case of low fiber content. This is much more true when the fiber length increases: the data scattering, for 6 mm fiber length, is lower if compared with the 3 mm and 4 μm cases. The influence of the hydration time on the electrical conductivity of the CFRC can be better observed in Fig. 10, where the changes in the electrical conductivity are reported for the three fiber volume fractions (point 1 is relative to the incipient percolation situation, point 2 is connected to the continuous network growth and point 3 is representative of a well developed network).

As expected the electrical conductivity decreases with the hydration time. It changes in a significant way for

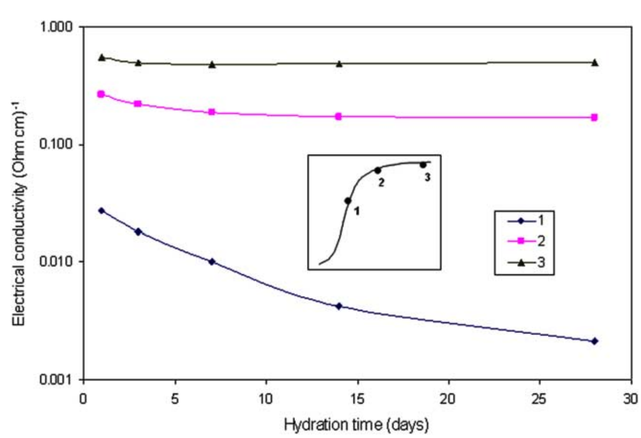

Fig. 10. Effect of the hydration time on 6 mm fiber length CFRC specimens for different volume fractions: (1) 0.075; (2) 0.45; (3) 1.6, 3 mm fiber length.

system 1, while few changes are observable in cases 2 and 3. These results confirm the theoretical analysis: when the fiber volume fraction surpasses the threshold value, the major part of the electrical conductivity of the CFRC is of the electronic type (connected to the free electron flow in the conductive network), and it is practically independent of the electrolytic conductivity of the concrete.

6. Sand/cement ratio effect

The effect of s/c on the electrical conductivity of the CFRC was analyzed for specimens with w/c equal to 0.45, for fiber length of 3 mm and 0.5% of fiber volume fraction (Fig. 11). The s/c ratios considered in the experimental program are: 0, 0.5, 1, 1.5, 2, 2.5, 3. The conductivity behavior for different s/c ratios is shown in Fig. 11. The measurements were made at 1 and 28 days of curing.

The hydration time, in this case, appears to have a strong effect on the electrical conductivity: at 1 day of curing and for low values of the s/c ratios, the conductivity value is quite high and decreases in an exponential way with the increase in sand, tending, for higher s/c ratios, to reach the conductivity values at 28 days of curing. In this particular case (fiber length = 3 mm), the system appears not completely percolated, making stronger the effect of s/c ratio.

The decrease in conductivity is due to the insulating nature of the sand, which between the fibers makes an obstacle or a cut in the electron flow through the conductive network, so decreasing the total conductivity of the system.

7. Analysis and discussion of the experimental data

From the observation of the experimental results it appears that the electrical conductivity of the CFRC can be analyzed using the percolation theory. With this theory the conductivity of the system can be expressed in the case $\phi > \phi_c$ as:

$$\sigma_f = c(\phi - \phi_c)^t$$

where c and t are two constants evaluated in a way as to minimize the deviation between the experimental and the theoretical values. To determine the conductivity of the system it is important to know the threshold value ϕ_c, defined as the fiber volume fraction where the clusters of the conductive fibers start to be in contact with one another, so forming the conductive network through the insulating matrix. In our experimental work the threshold value ϕ_c belongs to the range from ϕ_{c1} to ϕ_{c2}, in a way as to obtain the maximum correspondence between the theoretical curve and the experimental data. A measure of the effectiveness of the model can be the determination of the R^2 value. For the CFRC specimen series of the present work with fibers of 6 mm and 3 mm length, the application of the method allowed the determination, for the previous parameters, of the values shown in Table 4.

The correspondence between the theoretical curve and the experimental data is shown in Figs. 12 and 13. By analysing the correlation coefficient R^2, the good adherence of the examined model both for the 6 mm fiber length and the 3 mm ones is evident. Instead, the theoretical percolation law was less suitable for the correct interpretation of the experimental data in the case of 4 μm fiber length, probably because the basic assump-

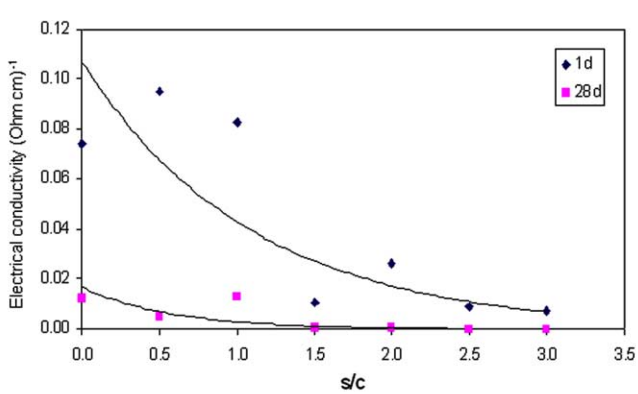

Fig. 11. Effect of the s/c ratio on the electrical conductivity of the CFRC specimens.

Table 4
Percolation threshold and parameters values for the system with different fiber lengths

Fiber length	c	t	ϕ_c	R^2
6 mm	0.375	0.312	0.15	0.981
3 mm	0.226	0.561	0.2	0.984

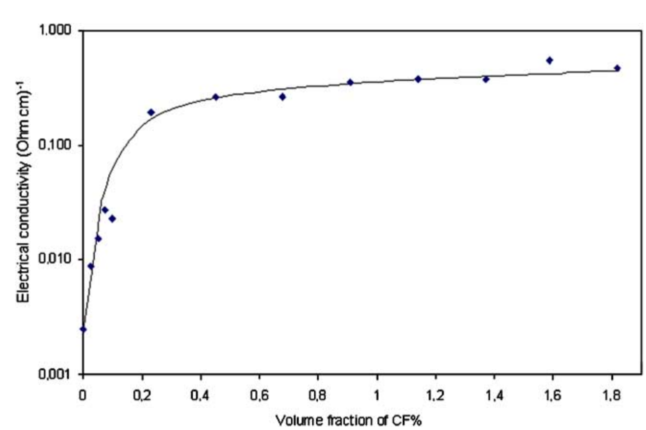

Fig. 12. Correspondence between the theoretical curve and the experimental data for the specimens with 6 mm fiber length, at 1 day of curing.

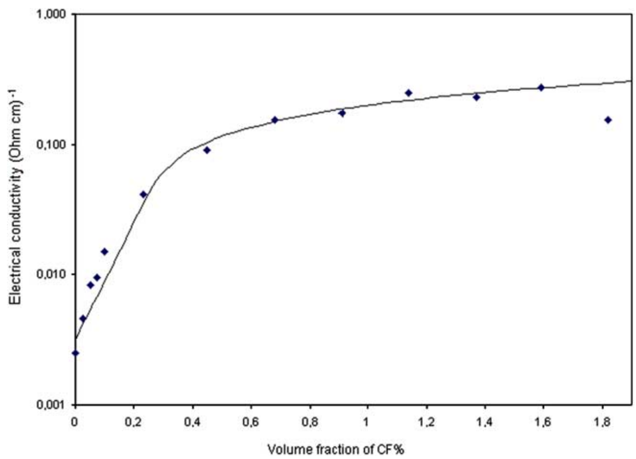

Fig. 13. Correspondence between the theoretical curve and the experimental data for the specimens with 3 mm fiber length, at 1 day of curing.

tion of the percolation network construction is no longer valid.

The values of the parameters c and t obtained in our analysis are lower than the analogous ones obtained experimentally by Xie et al. [7], while other researchers obtained values of t in the range 1.05–8.2 [13]. However, it is important to point out that in literature widely varying values for c and t are reported, both in the cases of the experimental interpolations and the theoretical modelling. The same can be said for the threshold values. Finally, it is important to underline that problems in the reproducibility and distortion of the results are often reported.

8. Conclusion

The experimental study carried out in this research showed that the addition of a small quantity of carbon fibers to cement mortar specimens can produce a significant increase in the electrical conductivity of material. The experimental results were analyzed of the percolation theory point of view. The fiber volume fraction which produces the transition from insulating to conductive material, called threshold value, is lower in the case of 6 mm fiber length than that in the case of 3 mm fiber lengths. Moreover it was possible to show that the addition of more fiber content, above the threshold value, does not produce any significant increase in the electrical conductivity of the material. The present study contributes to the use of non-destructive tests based on electrical measurements.

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