ORIGINAL ARTICLE



Electrical percolation and fluidity of conductive recycled mortar cement: graphite powder: recycled sand with addition of industrial waste carbon fiber

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Received: 19 March 2020 / Revised: 10 June 2020 / Accepted: 9 September 2020 © Korean Carbon Society 2020

Abstract

The use of recycled materials, such as the fine recycled aggregate made from concrete waste and carbon fiber (CF) product of industrial waste, for the manufacture of conductive recycled mortars (CRM), transforms the mortar base cement normally made with *cement:sand in a sustainable multifunctional material*, conferring satisfactory mechanical and electrical properties for non-structural uses. This action provides ecological benefits, reducing the use of natural fine aggregates from rivers and the amount of concrete waste deposited in landfills resulting from construction waste. In this investigation the effect of the addition of CF on electrical properties in hardened, wet and dry state, electric percolation in dry state and fluidity of the wet mixture of a *cement* based CRM was evaluated: *fine recycled aggregate: graphite powder*, CRM specimens with dimensions of $4 \times 4 \times 16$ cm. were manufactured for 3, 7 and 28 days of age and sand/cement ratios = 1.00, graphite/cement = 1.00, water/cement = 0.60 and CF = 0.1, 0.3, 0.5, 1.0, 1.5, 2.0, 2.5 and 3.0% compared to the weight of cement. The results demonstrated the effect of the addition of CF in CRM, reducing fluidity of the mixtures due to the opposition generated by its physical interaction of CF with recycled sand or recycled fine aggregate and graphite powder (GP), in its case, placing the electric percolation percolation at 0.30% and 0.45% of CF for CRM with and without GP, respectively. Increases in electrical conductivity (EC) without the presence of GP are defined by the contact between the CF and the conductive paths formed. In contrast, with the presence of GP, the EC is defined by the contact between the CF and the GP simultaneously, forming conductive routes with greater performance in its EC.

Keywords Multifunctional material · Sustainability · Electrical conductivity · Workability

1 Introduction

Cement-based mortars have poor performance in their EC, in dry state they are considered insulating materials, while in wet state they can be classified as semiconductor,

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situation due to the properties of ionic conductivity conferred by the pore solution of their cementitious matrix. To improve the EC of cement-based materials, the adition of conductive materials in the form of dust and/or fiber is used, among which are: CF, GP, carbon nanotubes and metallic fibers. These materials are considered suitable conductive additions, due to their excellent EC and mechanical properties, managing to transform cementbased mortars into multifunctional materials, by providing satisfactory mechanical and electrical properties not only for their structural use, but also for their electrical use and/or electrochemical [1-5]. In the specific case of GP and CF, the effect produced as an addition in mortars depends on their physical properties: length, particle size, average diameter, percentage of addition and dispersion in the cementitious matrix [6-9]. The dispersion and high CF content greatly affects the air content in the mortar mass, influencing the mechanical and electrical properties of the material [10–14]. Obviously, the non-homogeneous dispersion of the additions of conductive material in the cement matrix, either GP and/or CF, tends to produce a negative effect on the performance of various properties, both mechanical and electrical: compressive strength, tension, flexion, conductivity and electric percolation [15–21]. A homogeneous dispersion of the CF in the cementitious matrix, allows to reach limits of electric percolation with percentages much lower than those of GP additions, due to the high aspect ratio of the CF, obtaining multifunctional materials of high EC with low volumetric percentages of CF, complex situation in the GP use [22–25].

On the other hand, the fine aggregate or sand of cementbased mortars can be substituted or replaced by fine recycled concrete aggregates produced by mechanical crushing, achieving ecological benefits due to the reduction of the use of river sands and also reducing the amount of concrete waste, produced by the construction industry, which ends up deposited in landfills [26, 27]. Regarding its properties, the RFA is composed of natural aggregate coated with mortar or hardened paste, which affects the mechanical and physical properties of the mortar in fresh and hardened state, presenting less mechanical resistance, lower density, different setting time, increases in water absorption with respect to the natural aggregate, among others [28–32]. The use of RFA is an action that contributes to the sustainability of building materials [33–39].

It is evaluated, in this investigation, the effect produced by the addition of CF in electrical properties in hardened, wet and dry state, electric percolation in dry state, and in the fluidity of the wet mixture of a cement based CRM manufactured with RFA and GP in the same proportion.

2 Experimental part

2.1 Materials

2.1.1 Carbon fiber, graphite powder and cement

For the manufacture of CRM, it was used composite Portland cement (CPC) 30R type I, RFA, GP Loresco SC-3, CF with a length of 10 ± 1 mm and distilled water. The properties of the materials are shown in Table 1 and the distribution of the GP and CPC particle sizes is shown in Fig. 1.

The CF used to manufacture CRM specimens was from industrial waste products, previously making its selection and cutting for use in CRM mixtures. The diameter of the CF was constant, of continuous morphology, smooth and

Table 1 Material proper	ties	
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Material	Average diameter (µm)	Superficial area (m ² /g)	Specific density		
Carbon fiber	7	0.227	1.76		
Graphite powder 204		2.29	1.85		
CPC 30R 27.50		20.23	3.07		

free of defects, in Fig. 2 a photograph of raw CF and its micrograph is shown.

2.1.2 Recycled fine aggregate

The concrete used for the manufacture of RFA was quality control laboratory waste material, the crushing of the concrete was carried out by means of a jaw crusher.

Subsequently, the material retained between mesh No. 4 and No. 50 was selected, guaranteeing the absence of fine crushing products, cement powder, avoiding producing a mortar mixture with high water demands. The granulometry of RFA was performed according to ASTM C standards136 [40], ASTM C 33 [41] y ASTM C 125 [42]. Figure 3 shows the source material, left, and the material after the crushing process, right.

2.1.3 Specimens of conductive mortars

The EC was determined in specimens of CRM with 3, 7 and 28 days of curing in distilled water, prismatic specimens with dimensions of $4 \times 4 \times 16$ cm. with material ratios in mixtures of: sand/cement = 1.00, graphite/cement = 1.00, water/cement = 0.60 and CF = 0.1, 0.3, 0.5, 1.0, 1.5, 2.0, 2.5 and 3.0% with respect to the weight of the cement. Two types of specimens were manufactured, without and with GP, both with different percentages of CF, Table 2 shows the dosages used in the mixtures.

The manufacturing procedure of CRM with CF and GP according to Table 2, was carried out using the procedure described in ASTM C 305–14 [43] with the variants indicated below:

- 1. For the case of CRM type M-GP-CF, RFA was manually mixed with the GP until a homogeneous material was obtained in visual appearance, prior to the beginning of the manufacture of the mixture.
- CF was dispersed with the total water of the ultrasonic mixing for 30 min
- CF was placed with the total of water in a mixing vessel, adding the total cement and mixing at a slow speed of 140±5 r/min for 30 s







Fig. 2 Photography or raw CF and micrograph

- The total amount of recycled sand and GP was added, in its case, slowly for 30 s, while mixing at a slow speed of 140±5 r/min.
- 5. It was mixed for 30 s at an average speed of 285 ± 10 r/min.
- 6. The Mixer is stopped, remaining covered at rest for 90 s. In the first 15 s the walls of the vessel were quickly scraped off
- 7. It was mixed for 60 s at an average speed of 285 ± 10 r/min.

2.2 Methods

2.2.1 Determination of the fluidity of the mixture

The fluidity of the mixture was performed according to ASTM C 1437–13 [44] for all dosages indicated in Table 2, classifying the mixture according to its percentage of fluidity in dry, plastic or wet, as shown in Table 3.



Fig. 3 Crushing waste concrete for RFA manufacturing

Mortar	% of C	% of CF in relation to the weight of cement						Relation graphite/ cement	
M-CF	0.10	0.30	0.50	1.00	1.50	2.00	2.50	3.00	0.00
M-GP-CF	0.10	0.30	0.50	1.00	1.50	2.00	-		1.00
	Mortar M-CF M-GP-CF	Mortar % of C M-CF 0.10 M-GP-CF 0.10	Mortar % of CF in relation M-CF 0.10 0.30 M-GP-CF 0.10 0.30	Mortar % of CF in relation to the v M-CF 0.10 0.30 0.50 M-GP-CF 0.10 0.30 0.50	Mortar % of CF in relation to the weight of c M-CF 0.10 0.30 0.50 1.00 M-GP-CF 0.10 0.30 0.50 1.00	Mortar % of CF in relation to the weight of cement M-CF 0.10 0.30 0.50 1.00 1.50 M-GP-CF 0.10 0.30 0.50 1.00 1.50	Mortar % of CF in relation to the weight of cement M-CF 0.10 0.30 0.50 1.00 1.50 2.00 M-GP-CF 0.10 0.30 0.50 1.00 1.50 2.00	Mortar % of CF in relation to the weight of cement M-CF 0.10 0.30 0.50 1.00 1.50 2.00 2.50 M-GP-CF 0.10 0.30 0.50 1.00 1.50 2.00 -	Mortar % of CF in relation to the weight of cement M-CF 0.10 0.30 0.50 1.00 1.50 2.00 2.50 3.00 M-GP-CF 0.10 0.30 0.50 1.00 1.50 2.00 -

Table 3 Classification of the mixture according to fluidity

Mixture type	Fluidity rank (%)
Hard	80–100
Plastic	100-120
Dry	120–150

2.2.2 Determination of electrical conductivity

After checking the fluidity, and after the curing time has elapsed, the EC was determined for the specimens made with the mixtures according to dosages in Table 2 for ages of 3, 7 and 28 days. The electrical resistivity was measured



a 4PM-RM

b 4PM-DC

Fig. 4 Experimental arrangement for the determination of electrical resistivity

using 2 methods: Four-Point Method with resistivity meter Miller 400 (4 PM-RM) and Four-Point Method by Direct Current (4 PM-DC) according to [45, 46], the experimental arrangement is shown in Fig. 4, the resistivity was determined from the Eq. 1 and the EC with Eq. 2.

$$\rho = Fm * 2 * \pi * a * R \tag{1}$$

$$\sigma = \frac{1}{\rho} \tag{2}$$

where: $\rho = \text{Resistivity} (\Omega.\text{cm})$. $\sigma = \text{Conductivity} (S/\text{cm})$. Fm = Geometric factor that involves the length of the specimen (L) and the separation between the electrodes. a = Separation between electrodes (cm). $R = \text{Electric resistance} (\Omega)$.

Fm was determined based on the L/a relation and methodology proposed by Morris et al. 1996 [44] and Garzón et al. 2014 [45], as shown in Fig. 5. Fm corresponds to 0.1547 for the dimensions used of the CRM specimens and the separation between electrodes.

3 Results and discussion

3.1 Properties of the materials

3.1.1 Granulometry of the recycled fine aggregate

The mechanical properties of mortars are partly defined by the granulometry of fine aggregates [25, 26]. The

Fig. 5 Fm geometric factor

granulometric distribution of the RFA was uniform, outside the granulometric limits of ASTM as shown in Fig. 6. Being predominantly coarse recycled sand by classification with a minimum of fines, it guarantees lower demands for water in the mixture, making it possible not to use chemical additives to achieve workability and providing more paste to the cementitious matrix, area, where the CF is housed.

3.1.2 Carbon fiber

The characteristics of FC define various properties of the cementitious material due to its incorporation in the matrix and the synergy that they generate with the rest of the components [15, 16]. The chemical composition of the CF surface is shown in Fig. 7, with the presence of C, Cu, Zn and Si with different intensities in the signals. There were no impurities that could significantly affect the performance of the CR of CRM incorporated in the CF to the cement matrix.

3.1.3 Graphite powder

The GP added to cementitious matrices provides EC properties, making the material multifunctional [17, 19, 20]. Figure 8 shows the GP morphology and GP X-ray diffraction pattern used in the M-GP-CF mixtures, the main signal is 2Θ at 25.70 and a secondary signal at 43.30, corresponding to graphite according to the Inorganic Crystal Structure Database. There was no presence of impurities that could affect CD, after incorporating GP into the CRM.







3.1.4 Hardened conductive mortars

The addition of carbonaceous material in mortars and concrete provides the material with non-structural properties. [22–24]. Figure 9 shows the morphology of the CRM type M-GP-CF y M-CF. For the case of the 0% of CF, the interaction of GP with the cement matrix is appreciated, while in the case of the presence of CF, the contact between the CF is observed, which favors the EC in the CRM by adding CF in different proportions with respect to the weight of the cement. However, mixtures with percentages of CF have a tendency to vary their workability due to the physical interactions of the CF with the RFA of the mixture in wet state.

3.2 Fluidity and electric percolation of CRM

3.2.1 Fluidity in the CRM mixtures

Fluidity is a parameter of workability of mixtures, on which the proper placement of mortar in engineering works depends, this property is determined in fresh state according to ASTM C 1437-13 [43]. In the case of CRM with additions of different CF contents, it is shown in Fig. 10 the fluidity of both types of mixtures, M-CF and M-GP-CF. The maximum fluidity was of 150% for mixtures of 0% CF with and without GP content, where the high water content present due to the water/cement relation of 0.60, caused an initial equivalence in fluidity between M-CF and M-GP-CF.





b Morphology

Fig. 8 Properties of GP

However, from contents of 0.3% CF in M-GP-CF the fluidity decreased, due to the increase in internal friction between recycled sand and GP in interaction with CF, presenting a rapid decrease in fluidity with increasing percentage of CF, compared with mixtures without GP, where for CF percentages of up to 0.50% the fluidity continued to be constant, 150%, being the maximum value that can be reached with the used mix design parameters. For percentages above 1.0% of CF content, the M-GP-CF mixture is no longer manageable, showing a tendency to inhomogeneous CF dispersion and presence of high air contents, which comes to form discontinuous zones in the conductive routes of the carbonaceous material affecting the EC. In the case of M-CF mixtures, this situation occurs after 2.0% of CF content. In the M-CF mixtures there was a decrease in the fluidity with an increase in the percentage of CF, lower decreases compared to mixtures with GP. The addition of CF in CRM reduces the fluidity of the mixtures due to the opposition generated with the physical interaction and the increase between the friction of the RFA and the GP, if applicable.

3.2.2 Electric percolation of mortars type M-CF

EC of a cement based material with additions of CF depends largely on the dispersion of the carbonaceous material [10, 12]. Figure 11 shows the EC in wet state for different % of CF with respect to cement weight, the increase of the % CF produces increases of EC for all ages of curing at approximately the same rate of change, concluding that the EC does not depend on the age of the CRM. In addition, an electric percolation threshold is not clearly defined due to the contribution of the ionic conductivity of the pore solution to the EC of the CRM. Similarly, in Fig. 12 the EC in dry state is shown for different % of CF for the age of 28 days, the increase of % CF produces increases in the EC, being more notable the increase for the percentage of 0.45%, where there is an increase of more than one order of magnitude, this being considered as the *threshold of electric percolation*, since higher percentages of CF do not represent significant increases in EC. The percolation threshold was identified in a percentage lower than 2% of CF, considered the appropriate limit in the fluidity of a mixture type M-CF to be able to work in engineering works. On the other hand, in dry state there is no contribution of the pore solution in the EC, being the increases in EC governed by the contact between the CFs and the conductive routes that they form, as shown in the diagram in Fig. 12, allowing to be the percolation threshold limit easily located, compared to the wet state.

Figure 13 shows the relationship between the EC obtained by 4 PM-RM and 4 PM-DC, confirming an acceptable degree of approximation between the two different methods used to determine the EC.

3.2.3 Electric percolation of mortars type M-GP-CF

The simultaneous use of GP and CF produces a synergistic effect on the properties of EC when incorporated into pastes and mortars [25]. Figure 14 shows the EC in wet state for different % of CF with respect to the weight of cement, when increasing the % CF there are increases in EC for all ages of curing in approximately the same proportions. Therefore, it is confirmed that the EC does not depend on the age of the CRM, nor is an electric percolation threshold clearly defined due to the contribution of the ionic conductivity of the pore solution to the EC of the CRM, as in the case of M-CF mixtures. Similarly,



a M-GP-CF, No addition of CF



b M-GP-CF, With addition of CF



C M- CF, No addition of GP



Fig. 15 shows the EC in dry state for different % of CF for the age of 28 days, when increasing the % CF there are increases in the EC, being more notable the increase for the percentage of 0.30%, where it has an increase of more than one order of magnitude for EC, this being considered as the *threshold of electric percolation*, since higher percentages of CF does not represent significant

increases in EC. Figure 16 shows the interconnections of the GP with the CF in their different percentages, where for contents less than 0.5% FC they are presented with isolated contacts between the fibers without achieving conductive paths that can exceed the electrical percolation threshold, causing increases lower than those reached for higher percentages than mentioned. The percolation



Fig. 10 Mixture fluidity of CRM



Fig. 11 Conductivity in CRM type M-CF in wet state

threshold was identified in a percentage lower than 1% of CF, considered the appropriate limit in the fluidity of a mixture type M-GP-CF to be able to work in engineering works. Unlike the wet state, there is no contribution of the pore solution in the EC, being the increases in EC governed by the contact between the CFs and the GP, as shown in the scheme of Fig. 15, the which form the

conductive routes that contribute to the performance of this property, allowing to be easily located the percolation threshold limit and reducing the threshold with respect to the M-CF mixtures.



Fig. 12 Conductivity in CRM type M-CF in dry state, 28 days old





Fig. 14 Conductivity in CRM type M-GP-CF in wet state



Fig. 15 Conductivity in CRM type M-GP-CF in dry state, 28 days old



Fig. 16 CRM with different CF contents

4 Conclusions

Cement-based CRMs: *recycled fine aggregate: graphite powder* with CF addition are an alternative as multifunctional material, being also sustainable due to promoting the reuse of recycled materials. These CRMs show a rapid decrease in the fluidity with the increase in the percentage of CF due to the physical interaction between the CF and the RFA in wet state. For percentages above 1.0% of CF, the mixture with GP ceases to be manageable, with a tendency

to non-homogeneous dispersion of CF, high air contents and formation of discontinuous areas in the conductive routes of the carbonaceous material, in the case of CRM with the absence of GP, this situation occurs after 2.0% of CF. The addition of CF in CRM reduces the fluidity of the mixtures due to the opposition generated with its interaction with RFA and GP, in its case. The electrical percolation threshold for CRM with GP content was estimated at 0.30% of CF, below the case of absence of GP with 0.45% of CF. This is because the increases in EC without the presence of GP are governed by the contact between the CFs and the conductive routes that they form, while with the presence of GP the EC is defined by the contact between the CFs and the GP simultaneously, forming conductive routes with higher EC performance.

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