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MICROSTRUCTURE AND DURABILITY OF FLY ASH CEMENT GROUTS FOR MICROPILES

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ABSTRACT

This paper presents a study on the possibility of using fly ash cement as grouts for micropiles. This type of special geotechnical work is commonly used for many applications. Generally, micropiles grouts are prepared using Portland cement, although the standards do not restrict the cement type to use, as long as they achieve a strength requirement. In this research, fly ash cement grouts made with w:c ratios 0.40, 0.45, 0.50 and 0.55 were studied from 2 up to 90 days of age. Their microstructure was characterized using the non-destructive impedance spectroscopy technique, electrical resistivity, and mercury intrusion porosimetry. Their durability properties have been studied by determining the water penetration under pressure, and the chloride diffusion coefficient. The compressive strength was also measured and determined, and a maximum water:cement ratio, different for each cement type was obtained. All the results were compared to those obtained for Portland cement grouts. The results obtained confirm that the performance of micropiles made using fly ash cement grouts is adequate, and as it is well know the cements with mineral admixtures provide environmental benefits, so the use of cement including fly ash will contribute to the sustainability, with similar properties to those given by OPC.

Keywords: micropiles, special geotechnical works, fly ash, durability, microstructure, impedance spectroscopy, water:cement ratio.

1.-INTRODUCTION

In the field of geological engineering, the use of special geotechnical works has become very important. Some of the most commonly used special geotechnical works for civil engineering structures and for building foundations are piles, micropiles, soil anchors and jet grouting injections. There are great differences between those types of works and one of these differences is related to the material in which the steel reinforcement elements are embedded. In the case of the piles, concrete is usually used. However, for micropiles, soil anchors and jet grouting injections, the reinforcement elements are embedded in cement grouts, although mortars might also be used. This fact is very important, because the behaviour of the cement grouts and mortars shows many differences compared to concrete. For example, in general the porosity of hardened grouts is greater than the porosity of concretes [1], [2], and it could influence the durability and mechanical properties of the elements of each particular special geotechnical work. But on the other hand, a higher amount of cement might improve the durability of this type of elements. So, a different performance could be expected if the material used to protect the reinforcement steel elements is cement grout or concrete, as it is usual for the majority of civil engineering structures. Furthermore, the uncertainties can increase as a function of the cement type used, especially if it is used a sustainable cement, which incorporates some kind of active addition, instead of an ordinary Portland cement, as it is the usual practice.

Between the different types of grouted special geotechnical works, in the particular case of this research the micropiles have been studied. Micropiles are cylindrical members with diameters of under 300 mm, drilled and grouted with cement grout or mortar injected in one or two phases, reinforced with steel tubing and sometimes strengthened with one or several ribbed bars [3]. In Fig. 1.a and 1.b it is shown an example of micropiles use, and four different sections of a micropile, depending on the type of reinforcement employed [3–5].

Regarding the different standards about micropiles materials and implementation existing all over the world, it is important to highlight the Spanish / European Standard for micropile construction UNE-EN 14199 [4] and the US Department of Transportation, Federal Highway Administration's manual entitled Implementation manual for Micropile Design and Construction Guidelines FHWA-SA-97-070 [3]. Moreover, in Spain the Ministry of Internal Development has published a guide for designing and building micropiles in road works [5], which develops and supplements the contents of European micropiles standard [4].

Nowadays the global warming constitutes an important environmental problem, and one of the ways to solve it is reducing the CO_2 emission of the industries. In the particular case of cement industry, the use of active additions to improve their sustainability is an important field of study [6–10]. The most popular active additions are ground granulated blast-furnace slag, fly ash and silica fume. In general, these additions are wastes of other industrial processes, but their hydration reaction produces materials similar to those of clinker hydration. So, they can be reused to replace a percentage of this clinker in the cement final manufacture product.

As it has been abovementioned, one of the most popular active additions is fly ash, whose effects on the properties of cement-based materials are the object of considerable research [6, 11, 12]. One of the main property of this admixture is its capacity for reacting with portlandite, which is a product of the hydration of the calcium silicates of the clinker, through the pozzolanic reactions [11, 13, 14] . New hydrated phases are obtained as products of these reactions that improve the properties of cement-based materials. Fly ash performs very well particularly for structures in marine environments [6, 15–17].

Nevertheless, in spite of this good behavior for many uses, the cements containing active additions in general, and especially fly ash, are not commonly used for preparing cement grouts for micropiles. There are not strong reasons which talk out of its use for this purpose. Moreover, regarding other special geotechnical works, the situation is very similar and only there are few studies in this field. One of these researches has been recently published and it deals with the optimization of both the w:c ratio and the binder design, by using silica fume in order to modify the viscosity [18] and to improve the service behavior of cement grouts. With respect to fly ash, there are some studies that claim the feasibility of using fly ash in structural fills, and other geotechnical applications [19, 20]. In view of that, as it has been shown, up to our knowledge the performance of fly ash cements for micropiles grouts has not been studied, especially with regard to their microstructure and durability, despite the fact that there are many evidences that they could produce an improvement compared to ordinary Portland cement. Besides, regarding the compressive strength, fly ash grouts could also perform well, mainly in the long term [21, 22].

On this point, in the Spanish / European Standard for micropile construction UNE-EN 14199 [4] no cement type is explicitly specified. The only restriction on this aspect is reaching a minimum compressive strength. Similarly, the Ministry of Internal Development’s guide for designing and building micropiles in road works [5] and the US Manual FHWA-SA-97-070 [3] lay down the minimum compressive strength for micropiles, but not the type of cement to be used and it is acceptable the use of a wide range of water:cement ratios. Despite that, as it has been previously mentioned, at least in Spain cement grouts for micropiles are usually prepared with ordinary Portland cement (CEM I).

Then, this research aims to study of the possibility of using fly ash cement as an advantageous material for micropile preparation. To the purpose, the microstructure, durability and mechanical properties of cement grouts for micropiles have been studied. The grouts have been prepared using different dosages (w:c ratios), and using an ordinary Portland cement, and a fly ash-rich commercial cement, to study the viability of using this cement type.

The characterization of the microstructure of the grouts has a lot of interest, because it is directly related to the durability properties and the mechanical properties of these materials [23], [24]. In this work, it has also been used non-destructive techniques for studying the grouts porous network, such as impedance spectroscopy [1, 2, 25–27] and electrical resistivity by means of Wenner four-point test [28]. These techniques are nowadays an important research field because they have many advantages, for example the possibility of using the same samples for all the tests throughout the research. This fact permits a better monitoring of the microstructure evolution.

In relation to durability of fly ash cement grouts, its study is consequently highly pertinent, especially in the particular context of micropiles, where the reinforcement elements are embedded in the hardened cement grouts instead of concrete, as it has been abovementioned.

In this research, water penetration under pressure was the test used to assess durability, due to water is the main vehicle for the ingress of aggressive agents in cement-based materials [23, 29]. Grout resistance to chloride ingress was also analysed, inasmuch as these ions are among the primary inducers of steel corrosion, and they can be present in waters and soils in contact with micropiles. The mechanical property studied was compressive strength, since as noted above, this is the main and fundamental parameter specified for codes and standards for determining whether a cement is apt for this application.

Finally, because the grouts in these applications harden in contact with the surrounding terrain, exposing it to possible aggressive agents, its properties were characterised from very early ages (2 days) and up to 90 days.

2.- EXPERIMENTAL PROCEDURE

2.1.- Sample preparation

The tests were performed on cement grouts (pastes). These grouts were prepared using two types of commercial cements, a type CEM I 52.5 R/SR Portland cement, (CEM I hereafter), and a pozzolanic cement with a fly ash content from 36 to 55% of total binder, type CEM IV/B(V) 32.5 N (labelled CEM IV hereafter), according to Spanish / European standard UNE-EN 197-1 [30]. The reason for using these commercial cements instead of preparing mixes with ordinary Portland cement and fly ash, is that the accurate preparation of the mixes at the construction site would complicate the process of grouting the micropiles.

With regard to the dosage of the grouts, four different water to cement ratios were used: 0.4, 0.45, 0.5 and 0.55. As mentioned before, the Spanish guide for designing and building micropiles in road works [5] allows w:c ratios of from 0.4 to 0.55, while Spanish / European standard UNE-EN 14199 [4] specifies that the ratio must be lower than 0.55. Manual FHWA-SA-97-070 [3], in turn, stipulates that the w:c ratio in grout for micropiles must lie between

0.4 and 0.5. Then, the w:c ratios studied in this work permit to analyse the influence of this parameter, according to the abovementioned standards. However, it is important to emphasize that in the case of Spain, the grouts are usually prepared with w:c ratio 0.5, in spite of the abovementioned different dosages allowed by the standards and manuals.

Several types of specimens were prepared. All the samples were kept in a 95% RH chamber with a temperature of 20ºC for 24 hours immediately after setting up the grouts. On one hand, cylindrical specimens were prepared and cast in molds of 10 cm diameter and 15 cm height. After the 24-hours curing time, they were demolded and cut to obtain slices of approximately 1 cm thickness. Other cylindrical specimens were cast to diameters of 10 and 15 cm and a height of 30 cm. The 10-cm diameter samples were used to study the variations in electrical resistivity and the 15-cm specimens to determine compressive strength and the penetration of water under pressure. Finally, prismatic specimens with dimensions 4 cm x 4 cm x 16 cm were also prepared (UNE-EN 196-1:2005 [31]) to compare their compressive strength to the strength obtained for the 15-cm diameter x 30-cm high specimens.

When the 24-hours curing had finished, all the specimens were submerged in distilled water until the testing age. These curing conditions are intended to simulate the conditions of micropiles that are cast in situ and stay in contact with soil and water from the very first day. The only exception was the 15-cm diameter x 30-cm high specimens, which were stored in a humidity chamber at 20ºC and 95% RH as specified in Spanish /European standard UNE-EN 12390-2 [32], to which refers the Spanish guide for designing and building micropiles in road works [5] for those particular specimens.

2.2.- Mercury intrusion porosimetry

The grouts microstructure was characterized using mercury intrusion porosimetry, as well as the non-destructive techniques previously mentioned. This is a well-known and extensively used technique [33], although it has some drawbacks [34]. The porosimeter employed was an Autopore IV 9500 from Micromeritics. This porosimeter allows determining pore diameters between 5 nm and 0.9 mm. Before the test, samples were oven dried for 48 hours at 50ºC. Two measurements were made on each material. Total porosity and pore size distribution were studied through intrusion curves. The tests were performed at 2, 28 and 90 days of age.

2.3.- Impedance spectroscopy

The impedance measurements on the cement grouts were carried out using the impedance analyzer Agilent 4294A, which allows capacitance measurements in the range from 10^{-14} F to 0.1 F, with a maximum resolution of 10^{-15} F. Impedance spectra of samples were obtained in the frequency range from 100 Hz to 100 MHz, using two different methods. For both methods, the electrodes were circular (Ø = 8 cm) and made of flexible graphite, attached to a copper piece with the same diameter. First, impedance spectra were obtained with a contacting method, being the electrode in direct contact with the sample. Afterwards, the measurements were also performed using a non-contacting method. This method minimizes the possible contributions of the sample-electrode interface as shown elsewhere [35], and minimizes as well the runaway capacitance existing due to the border effect [36]. It consists of placing a polyester sheet (100 μm thick) between the sample and each electrode. The impedance of the polyester sheets is subtracted from the total impedance measurement, to get only the impedance response of the sample. As this setup gives an almost capacitive impedance spectrum, the answer of the sample is transformed to a spectrum in capacities using the Cole-Cole transformation [1].

For validating the obtained impedance spectra, the Kramers–Kronig (K–K) relations were used, to ensure causality, linearity and stability of the measurements [37]. As an example, Fig. 2 depicts the Cole-Cole plots at different ages for CEM IV grouts, while Fig. 3 shows the validation of the impedance spectrum of a CEM IV grout using the K-K relations, as mentioned before. The differential impedance analysis was developed by Stoynov et al. [38], and gave excellent results on cementitious materials [1]. It was applied to the spectra before assuming the equivalent circuit as valid for fly ash cement grouts. Fig. 4 shows the result of the analysis on one impedance spectrum. The result is valid for all the data obtained, and the two maxima that shows the plot of the time constant of the material, τ, at each frequency, versus number of points, indicate the presence of two time constants in the impedance spectrum. The number of time constants justifies the fitting of the obtained data to the equivalent circuits proposed by Cabeza et al. [1], which included two time constants. These circuits are shown in Fig. 5. Both circuits have been used for different types of materials [1, 9, 27]. The fitting of the measured data to the model proposed is made using a Simplex optimization method, which is described elsewhere [35]. Regarding the impedance parameters, it is important to emphasize that the resistance R_2 and the capacitances C_1 and C_2 can be obtained using both contacting and non-contacting methods. In this research, the evolution of those parameters has been studied from non-contacting measurements because of its higher accuracy. For each cement type and w:c ratio four different samples with approximately 1 cm thickness were tested. The evolution of impedance parameters has been followed until 90 days of hardening.

The main advantages of using this technique, in addition to being non-destructive, are that the measurement is global, over the whole area of the surface, and it does not give local information on the microstructure of the sample, as the mercury porosimetry does. The non-destructive character allows also to follow the evolution of the microstructure of the same sample over the time, and the rest of available techniques do not allow this follow up. It has to be pointed out here that this technique has been mainly used for OPC samples, where there is not a pozzolanic reaction as it happens in fly ash cements. The possibility of frequent measurement on samples allows to study the effect of this pozzolanic reaction more properly.

2.4.- Electrical resistivity

This parameter gives information about connectivity and pore size in a material. In this research the electrical resistivity was determined in cement grouts specimens using the Wenner four-point test described in Spanish standard UNE 83988-2 [39]. This very well-known method is widely used in cement-based materials [40-41]. Specimen electrical resistivity was measured directly on a Proceq analyser.

2.5.- Water penetration under pressure

The samples tested were cylinders of 15 cm diameter and 30 cm height according to the Spanish / European standard UNE-EN 12390-8 [42]. The test consists of applying water to the specimens at a pressure of 500±50 kPa for 72±2 hours. When the test had concluded, the samples were split axially and the depth of water penetration was measured in each half. Despite this test is designed for hardened concretes, it was applied here to the cement grout because the standards on micropiles [5] refer to the provisions of Spanish Structural Concrete Code EHE-08 for characterizing most grout properties [43].

Regarding the conditioning of the specimens before the test, the standard UNE-EN 12390-8 does not specify a certain procedure. Then, in this research the specimens were kept for 72 hours prior to the test at a temperature of 20±2 °C and relative humidity of 50%, as suggested the standard. Two samples were tested at 28 and 90 days of age, for each type of cement and w:c ratio. Finally, the results obtained were the mean and maximum depths of the water penetration front for each sample.

2.6.- Forced migration test

The study of the resistance against chloride ingress of the hardened cement grouts has a lot of interest. In this research, the forced chloride migration test was performed on water-saturated cement grouts, according to the standard UNE 83987 [44]. The main result obtained is the non-steady-state chloride diffusion coefficient D_{ns}, in m^2/s. Samples of approximately 1 cm thick were tested. The experimental procedure of the test [45] is based on monitoring the anolyte conductivity, which has been shown to be proportional to the chloride concentration of the anolyte.

The cement grouts were saturated for 24 hours before the migration tests, according to ASTM Standard C1202-97 [46]. The sample was placed in a cell between two electrolyte containers, whose capacity was 500 ml. The surface of the sample exposed to the migration test was circular of 6.5 cm diameter. The stainless steel electrodes, for establishing the driving electric field, were placed in the apertures of the cell and the distance between them was 25 cm. The catholyte and anolyte chambers were filled with a 1 M NaCl solution and with distilled water, respectively. The applied driving voltage was 12 V, although the effective potential drop between both sides of the cement grout disc was measured periodically. The conductivity measurements of the anolyte solution were performed every 12 hours since the beginning of the test. These measurements were performed with a Crison GLP31 conductimeter, with automatic compensation of the readings to 25ºC standard temperature. Temperature data of the electrolytes were also recorded.

For each cement type and w:c ratio three different samples were tested. The tests were performed at 2, 28 and 90 days of age. The reason for performing a first test at 2 days has to do with the real service conditions of micropiles. As it has been said before the micropiles stay in contact with soil and water from the moment they are cast. That means that they can be in contact with aggressive substances (in case there are in water or soil) from the very beginning. So, performing that test can give us important information on the real service conditions and the real degradative processes that could take place in a micropile in service, and study the viability of using fly ash cement to construct those elements.

2.7.- Determination of compressive strength

As it was stated in the introduction the standards do not restrict the cement type for micropiles, as long as they achieve a compressive strength requirement. The Implementation manual FHWA-SA-97-070 [3] suggests that the neat cement grouts should reach a compressive strength between 28 and 35 MPa at 28 days of age. In the case of standard UNE-EN 14199 [4], the minimum compressive strength required for the grouts at 28 days is 25 MPa.

The reference standards for micropiles [4, 5] establish that the compressive strength must be determined using cylindrical samples with double length than diameter. For that reason the compressive strength was determined in samples with 15 cm diameter and 30 cm height. The compressive strength was measured following the standard UNE-EN 12390-3:2009 [47].

For each condition (cement type and w:c ratio) two measurements were taken.

3.- EXPERIMENTAL RESULTS

3.1.- Mercury intrusion porosimetry results

As it was stated in the experimental section two samples were tested for each condition. Fig. 6 shows the intrusion curve obtained for CEM I samples tested at 28 days hardening. The results for the two samples are shown, one using continuous line and symbol, and the second measurement made with a dotted line. As it can be seen in Fig. 6 there may be minor differences among the two samples in some cases, but there is a good reproducibility. For the sake of simplicity only one measurement will be shown in the rest of the figures. The second result that could be extracted from this figure is that the increases in w:c ratio increases the total porosity of the samples. This result is general for every cement type and age.

A more interesting analysis can be extracted from Fig. 7, where the time evolution of the porosity is studied for samples with w:c ratio 0.5. It can be easily observed that the total porosity decreases with time, but some differences can be seen as a function of the cement type (Fig. 7.a for CEM I results and Fig. 7.b for CEM IV results).

First of all, for every studied age the total porosity of the samples prepared with CEM IV is higher than for the samples prepared with CEM I. This result could be also expected since the strength class of CEM IV is lower than the strength class of CEM I (see experimental procedure section). Samples prepared with CEM I show a very small evolution of the pore network between 28 and 90 days, whereas there is a greater evolution for CEM IV samples. This evolution produces a pore network with higher amount of small pore diameter (below 100 nm) at 90 days for the CEM IV as compared with CEM I pore network. This evolution and the final pore network are mainly due to the pozzolanic reactions of the fly ash.

3.2.- Impedance spectroscopy results

The resistances R_1 and R_2 are related to the pores of the sample which are filled with electrolyte [26]. Changes in the value of the resistance may come from the variation of the pore dimensions, or by the drying of the pores [1, 48, 49]. The evolution with time of resistance R_1 can be observed in Fig. 8 for both types of cement grouts. For CEM I samples, the resistance R_1 kept practically constant or hardly increased with time. At early ages, CEM IV grouts showed lower R_1 values than those observed for CEM I ones. Nevertheless, since approximately 20 days, the resistance R_1 started to increase for CEM IV samples. First, this rise of R_1 was slow and at 30 hardening days the values of this parameter for CEM IV grouts were still lower or similar to those observed for CEM I ones. Since then, the CEM IV R_1

values started to increase faster and at 90 hardening days their values were higher compared to CEM I ones.

The results of resistance R_2 are depicted in Fig. 9. In general, the evolution of this parameter was very similar to that previously described for resistance R_1.

The changes with hardening time of capacitance C_1 for CEM I and CEM IV specimens are shown in Fig. 10. This capacitance is related to the solid fraction in the samples [26]. For the majority of the samples studied, this parameter increased with time. At early ages, the capacitance C_1 was lower for CEM IV samples than for CEM I ones. At 90 days, this parameter was very similar for both types of cement, or even it was a little higher for CEM IV grouts.

The results of capacitance C_2 for both types of cement studied are depicted in Fig. 11. This parameter is related to the pore surface in contact with electrolyte present in the material [48, 50]. At early ages, the capacitance C_2 increased with age for CEM I samples and showed higher values than those observed for CEM IV ones. However, it kept practically constant or hardly increased since approximately 20 days for the majority of CEM I grouts. On the other hand, the capacitance C_2 for CEM IV samples showed low values at early ages, but this parameter continuously increased with age, and at 90 days the capacitance C_2 was similar or even higher for CEM IV samples than for those prepared using CEM I.

3.3.- Electrical resistivity results

The results of the electrical resistivity measured using the Wenner method are shown in Fig. 12. As it can be seen it is noticeable that the values of resistivity for the cement containing fly ash are much higher than for the ordinary Portland cement. Moreover, in the case of cement type IV clearly the higher is the w:c ratio the smaller is the resistivity. The resistivity for both cement types increases with the hardening time.

3.4.- Water penetration under pressure

The results of the water penetration under pressure (maximum and average penetration) are shown in Fig. 13. As it can be seen in the plots, the average penetration measured following the standard UNE-EN 12390-8, is always smaller for cement type IV, containing fly ash, than for ordinary Portland cement (CEM I). As could be expected, the increase in the w:c ratio also causes an increase of the average penetration of water in the samples. The values of average penetration show a decreasing tendency with the hardening age for both cement types. The results of the maximum penetration depth are very similar to these about the average penetration depth.

3.5.- Forced migration tests

The results of non-steady-state chloride diffusion coefficient (D_{ns}) for CEM I and CEM IV grouts are shown in Fig. 14. This coefficient decreased with age for the majority of CEM I and CEM IV grouts. At all ages, CEM IV grouts showed very low diffusion coefficients in comparison to those observed for CEM I ones.

3.6.- Compressive strength results

The results of the compressive strength measured in cylindrical specimens fulfilling the indications of the standard UNE-EN 14199 are shown in Fig. 15. It is clear there that the samples prepared with CEM I have a higher strength than the samples prepared with fly ash cements (CEM IV). This result is in coincidence with the different strength class of the cements used (see section 2.1). The compressive strength increases with time, regardless the cement type and the w:c ratio. As it was explained in the experimental section, the requirement of the standard is that the minimum compressive strength at 28 days should be

25 MPa for the grouts. Taking this into account it can be established that both cement types could be used. There is only a limitation in the w:c ratio. For ordinary Portland cement a maximum w:c of 0.5 should be used, while for the fly ash cement a maximum w:c ratio of about 0.45 should be selected.

4.- DISCUSSION OF RESULTS

The total porosities for CEM IV grouts were higher than those observed for CEM I ones at all hardening ages studied (Fig. 7). This result is consistent with findings reported by other investigations [6, 12, 51]. On the other hand, at early ages (2 and 28 days) CEM IV samples had a coarser porous network than CEM I ones. However, the microstructure of CEM IV grouts was more refined in the long-term, as showed their greater volume of finer pores at 90 days (see Fig. 7.b). It is well-known that the portlandite is necessary to start the pozzolanic reactions of fly ash [6, 11, 12, 14] , and it is formed during the clinker hydration. Then, it is needed more time to start the fly ash pozzolanic reactions and, as a consequence, to observe the effects of this addition in the microstructure of the grouts. This fact could explain the pore size distribution of CEM IV grouts in the short-term, especially at 2 hardening days, when it is probably that the degree of development of the pozzolanic reactions of fly ash was very low. Besides, the progressive pore refinement with age showed by CEM IV grouts, could be due to the formation of additional CSH phases [51] as products of fly ash pozzolanic reactions, which leads to a more compact porous structure of fly ash hardened grouts.

Regarding impedance spectroscopy results, the resistances R_1 and R_2 are associated with the electrolyte present in the pores of the sample. Since all the samples were kept under immersion, as stated in the experimental section, the changes in the value of the resistances can only come from changes in the pore dimensions [52]. In the short-term, the lower resistances observed for CEM IV grouts (see Fig. 8 and Fig. 9) could be related to the their coarse microstructure, due to the still limited formation of new hydrated products from fly ash pozzolanic reactions, as has been already explained. On the other hand, the important increase with time of the resistances R_1 and R_2 for CEM IV grouts would show a progressive closing of their pore structure, probably related to the development of pozzolanic reactions, as indicated the pore size distribution results. In view of that, the results of resistances R_1 and R_2 corroborate the important pore refinement of grouts microstructure produced by fly ash, previously observed by mercury intrusion porosimetry.

The dielectric capacitance C_1 is related to the solid fraction of the samples, then it is expected that this parameter increases as solid formation is produced due to the development of clinker hydration and pozzolanic reactions of fly ash. This parameter is independent of pore size distribution. In general, the capacitance C_1 increased with age for the majority of the samples studied. This would indicate a progressive formation of solid phases. This is in accordance with the abovementioned decrease with age of total porosity. The apparent disagreement among the values of total porosity and capacitance C_1 for samples prepared with different cement types (CEM I and CEM IV) come from the fact of the different chemical composition of the materials, fact that will change the dielectric properties and as a result, the value of the capacitance.

The capacitance C_2 is associated with the pore surface in contact with the electrolyte present in the material and it is related to the amount of wet pore surface. Since samples are kept submerged, it is expected that pores would be saturated. So changes in the capacitance C_2 would be mainly due to the formation of CSH gel layers on pore walls, which will occupy the pores [26]. These products are deposited on the pore surface and they form rough structures, which increase the specific surface of the pores and the tortuosity of the pore network. This rise of pore specific surface brings about an increase of the solid-electrolyte interface, which entails higher values of capacitance C_2. In general, the capacitance C_2 increased with age for both types of cement studied. At early ages, the lower values of this parameter were observed for CEM IV grouts. However, in the long-term the capacitance C_2 was similar for CEM I and CEM IV grouts, although it was a little higher for CEM IV ones, see Fig. 11.

In general terms, these results are in keeping with pore size distributions obtained using mercury intrusion porosimetry and with the results of resistances R_1 and R_2. The low capacitances C_2 for CEM IV grouts in the short-term could be due to the scarce development of fly ash pozzolanic reactions, as has been already explained. The important rise with hardening age of this parameter could be related to the formation of additional CSH phases, as products of pozzolanic reactions. These CSH phases would be formed over the existing pore surface, increasing the pore surface, the tortuosity of pore network and the solid-electrolyte interface, as suggest the capacitance C_2 results. Finally, the higher values of this parameter at later ages for CEM IV grouts than those observed for CEM I ones would indicate that their microstructure was more refined, which would corroborate the mercury intrusion porosimetry results.

The results of the Wenner resistivity test are coincident with the results of the resistances measured with impedance spectroscopy. This result is the expected, and in agreement with the rest of microstructural characterization. However, the impedance spectroscopy gives a more in deep information, due to the analysis of the capacitances. The resistivity for fly ash cement gives a better correlation of the resisitivity with the total porosity.

Regarding the results of microstructure characterization, it seems that the use of a fly ash cement for preparing cement grouts for micropiles could produce a more refined porous network of the hardened cement grout (cement paste) in the long-term (90 days), compared to ordinary Portland cement. The microstructure of cement-based materials is related to their service properties and especially to their durability [24]. As a consequence it could be expected an improvement of the micropiles durability if they are made using a fly ash cement.

Besides, the use of this type of cement would also bring about an increasing of the initiation period of steel corrosion, which would extend the expected service life of the micropiles.

With regard to w:c ratio, the results obtained indicate that this parameter does not seem to produce so much influence on the microstructure of cement grouts as the type of cement, except the expected increase of porosity when the w:c ratio is higher.

Finally, it is worth to emphasize that the results of the non-destructive technique of impedance spectroscopy are in agreement with those obtained using mercury intrusion porosimetry.

The results of water penetration under pressure show that there is a bigger influence of the smaller pore dimensions than of the total porosity. That is the reason why the penetration of water under pressure is lower for cement containing fly ash than for ordinary Portland cement. This result is essential for the use of CEM type IV to grout micropiles. The penetration of water is one of the main durability indicators [23], and this result confirms that the aggressive will have a smaller penetration in the micropiles made of CEM IV, and so will the aggressive substances, so these cement types, in addition to being more sustainable, will guarantee in a more efficient way the durability of the micropiles.

Chlorides can produce the corrosion of reinforcing steel bars and pipes, especially in micropiles in contact with waters with high contents of this aggressive. The non-steady-state chloride diffusion coefficient showed much lower values for CEM IV grouts at all ages than for CEM I ones, as it can be seen in Fig. 14. Many studies have demonstrated that the use of fly ash produces a substantial improvement in chloride ingress resistance [53, 54] . The low diffusion coefficients of CEM IV grouts in the short-term, even though the cement paste is more porous, and with bigger pores, can be explained as being a consequence of the higher binding capacity of fly ash cement, as compared to Portland cement. This binding capacity is due to the high content of calcium aluminates brought by the ash [53]. At later ages, the higher microstructure refinement could also contribute to the decrease of chloride diffusion coefficient observed for CEM IV grouts, besides the abovementioned binding capacity of fly ash.

The results of the chloride diffusion coefficient would confirm the fact that the use of fly ash cement for preparing cement grouts for micropiles would produce an improvement of their durability, not forgetting the economic and environmental benefits that bring the use of a waste such as the fly ash. Moreover, it is important to emphasize that at 90 hardening days, the non-steady-state chloride diffusion coefficient for CEM IV grouts were very similar for samples prepared with w:c ratios between 0.4 and 0.55.

The results of the compressive strength, as it was explained in the results section limit the maximum w:c ratio, that is not in compliance with the standard, so, from the point of view of the application of these cements for micropiles grouting this parameter should be controlled before using them.

In order to check the possibility of injecting the grouts to prepare micropiles, its fluidity was measured. The results of the fluidity of all the tested cement grouts, are shown in Table 1. As it can be seen in the table the fly ash cement shows a greater workability than the ordinary Portland cement, as it is reported in the literature [55-59]. As it can be seen in the table the lower is the w:c ratio the better is the fluidity of the fly ash cement compared with the ordinary Portland cement. This result proves that even though the fly ash cement requires a lower w:c ratio to achieve the minimum resistance, it could be pumped to prepare the micropiles in the same conditions as the CEM I.

5.- CONCLUSIONS

The main conclusions that can be drawn from the results previously discussed can be summarized as follows:

1. The cement grouts made using fly ash cement exhibited higher microstructure refinement in the long-term (90 hardening days) than those prepared using ordinary Portland cement.
2. The use of fly ash cement for micropiles grouts produced an important improvement of their resistance against chloride ingress.
3. The results of the non-destructive technique of impedance spectroscopy were in keeping with those obtained using mercury intrusion porosimetry. In view of that, the impedance spectroscopy can be used for studying the microstructure development of fly ash cement grouts. The resistivity gives only results about resistance, which are consistent with the results of impedance spectroscopy.
4. The penetration of water under pressure guarantees the lower penetration of water and/or aggressive substances in the micropiles prepared with fly ash cement, giving a more sustainable and durable structure.
5. The reduced porosity of the cement matrix due to the lowering of the w:c ratio has certainly a positive effect on the durability in general. In the case of the resistance to chloride penetration, the effect of w:c ratio on this resistance is less evident as this parameter is influenced by the ability of the matrix to bind chlorides. However, the w:c ratio is determinant from the point of view of the compressive strength, and has to be taken into account to fulfill the minimum values required by the standards.
6. In view of the results obtained in this research, and under these conditions, the performance of micropiles made using fly ash cement grouts is adequate compared to ordinary Portland cement grouts.

6.- ACKNOWLEDGMENTS

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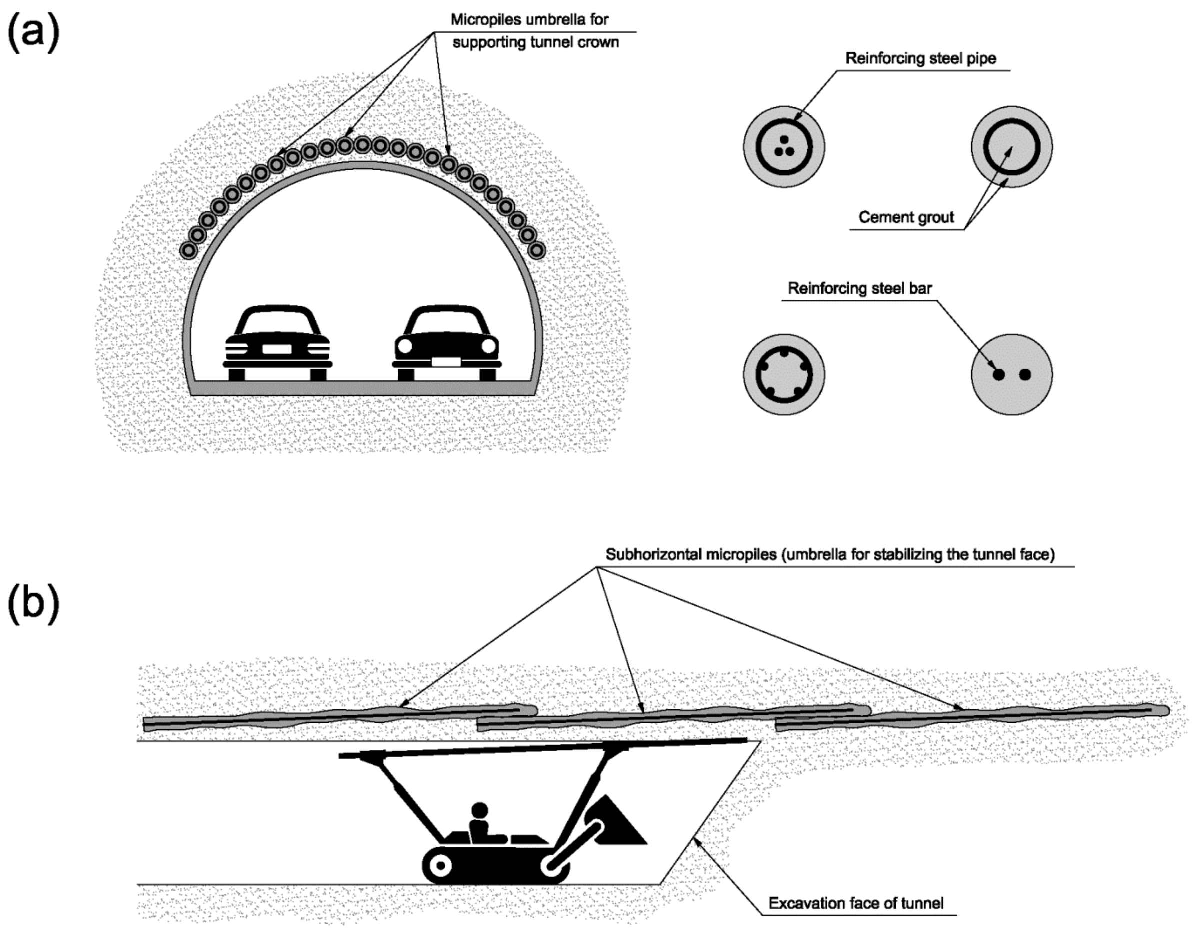

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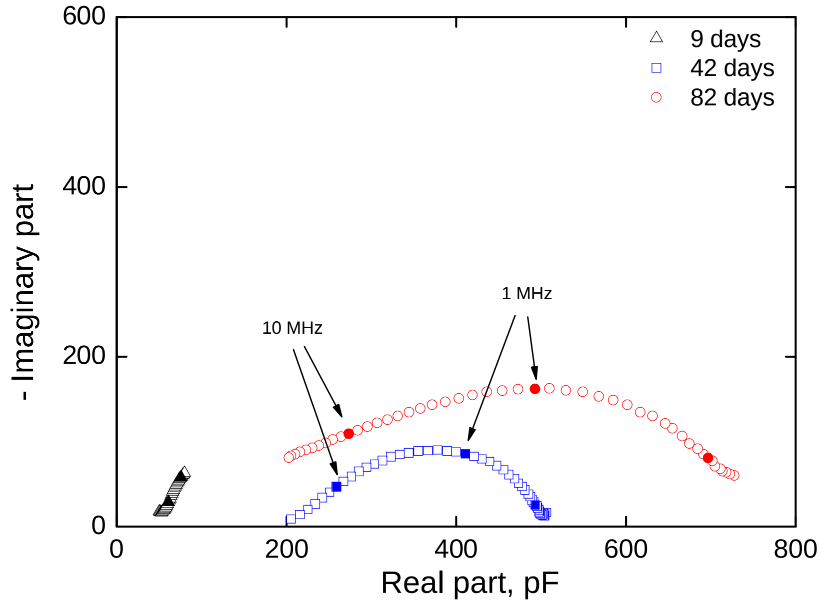

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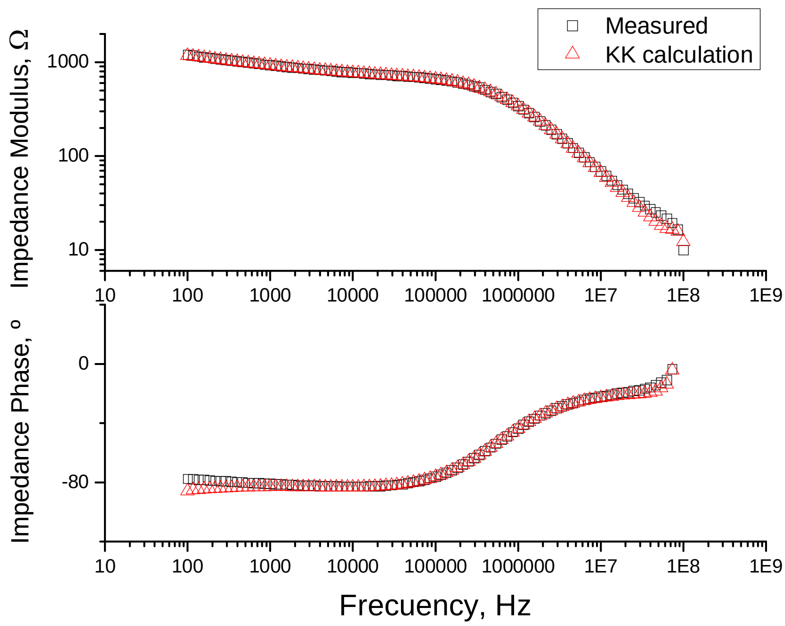

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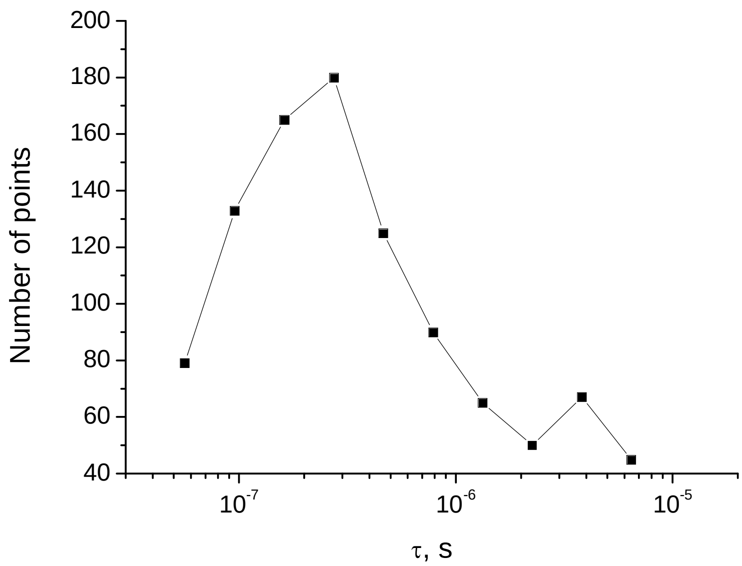

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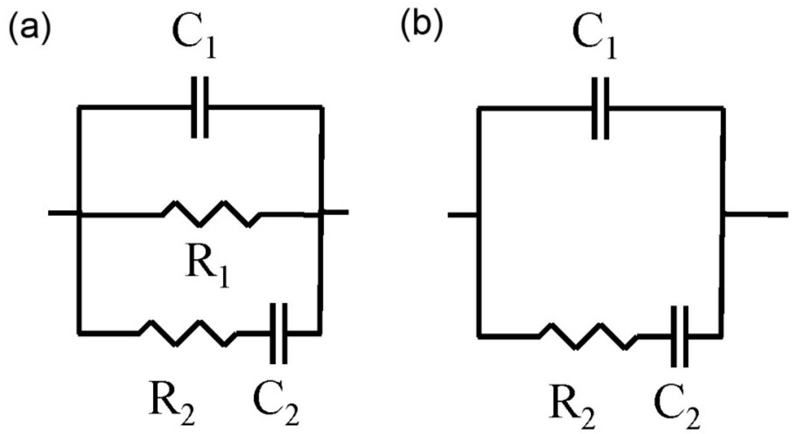

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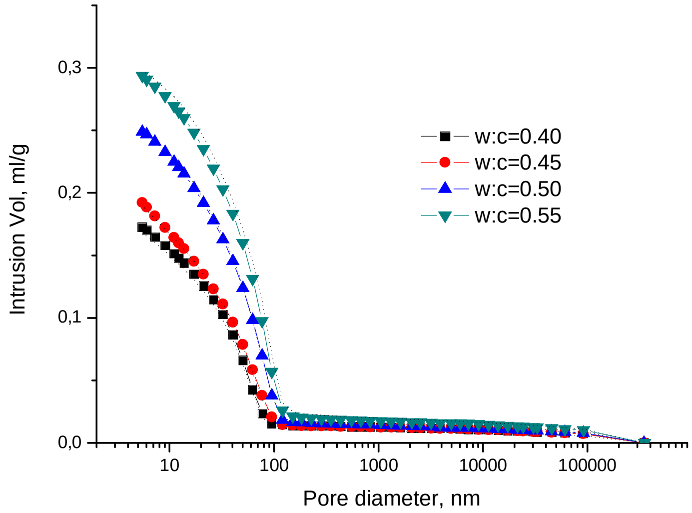

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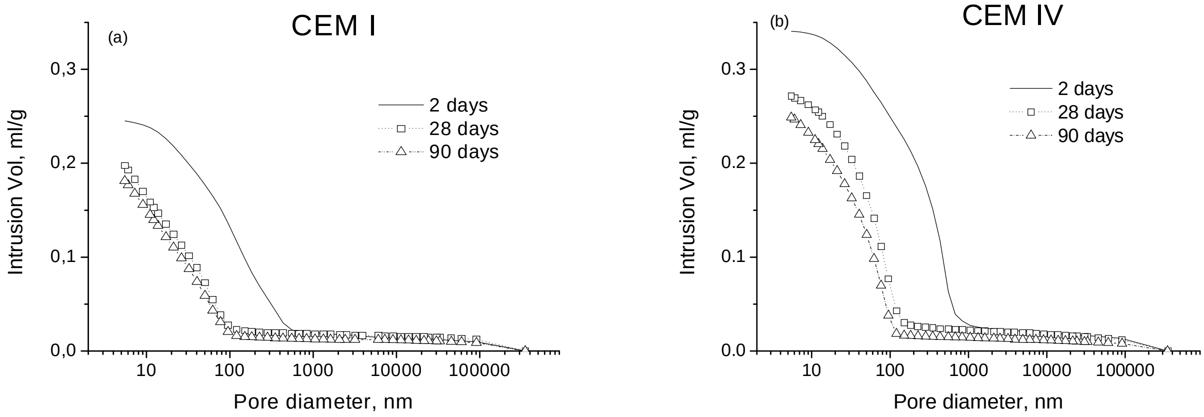

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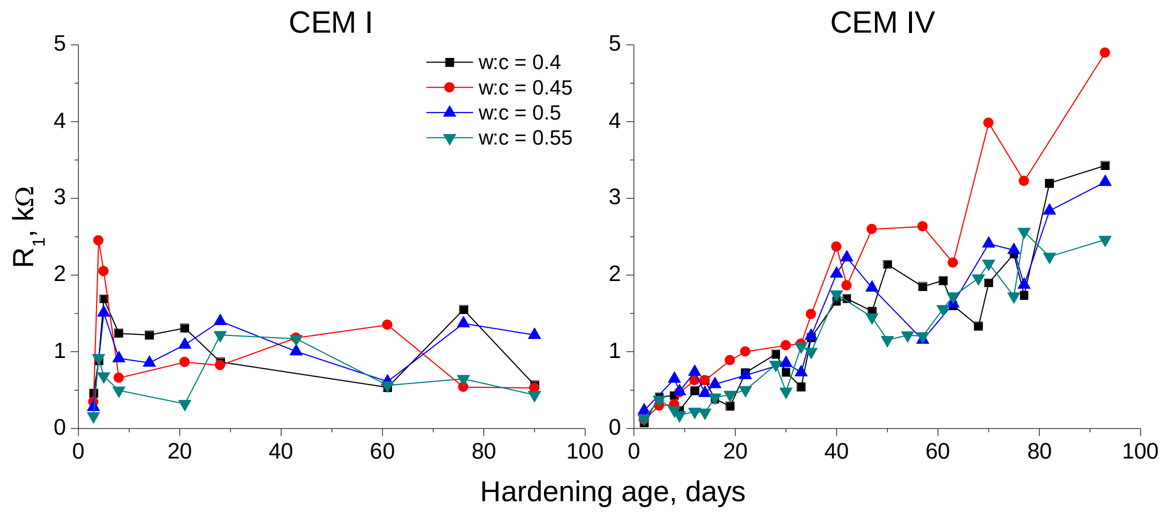

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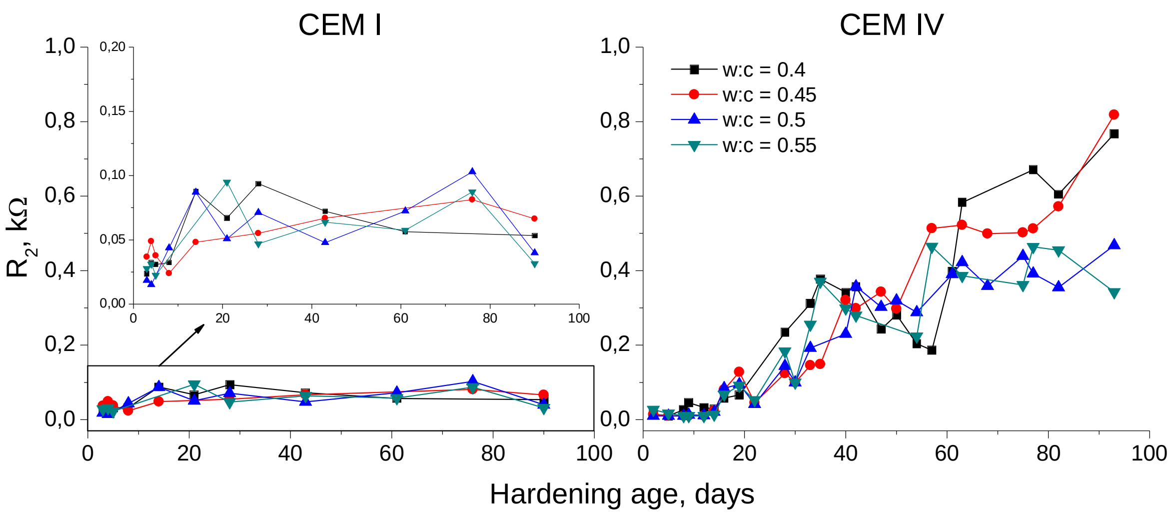

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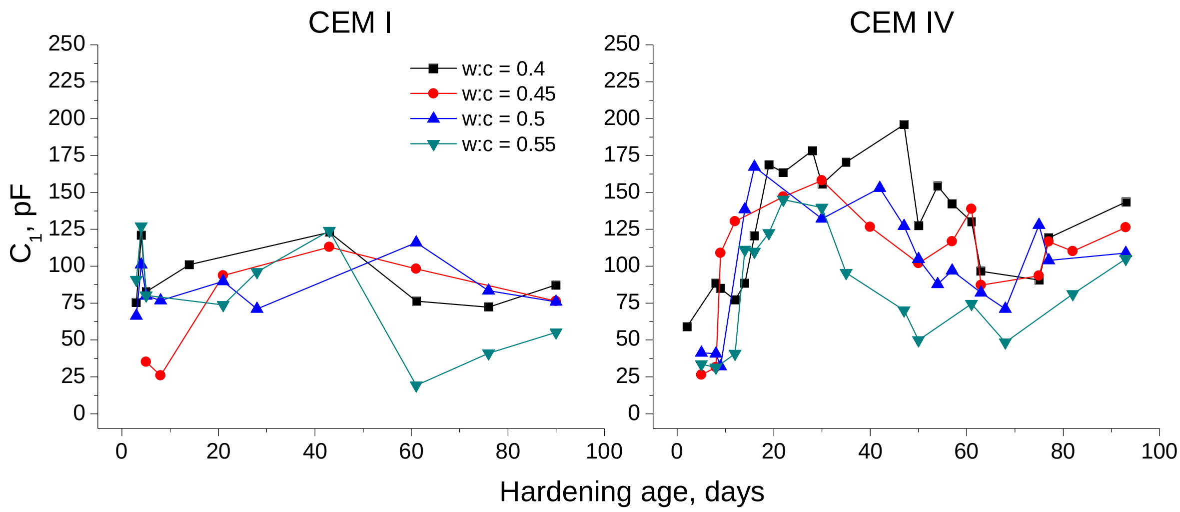

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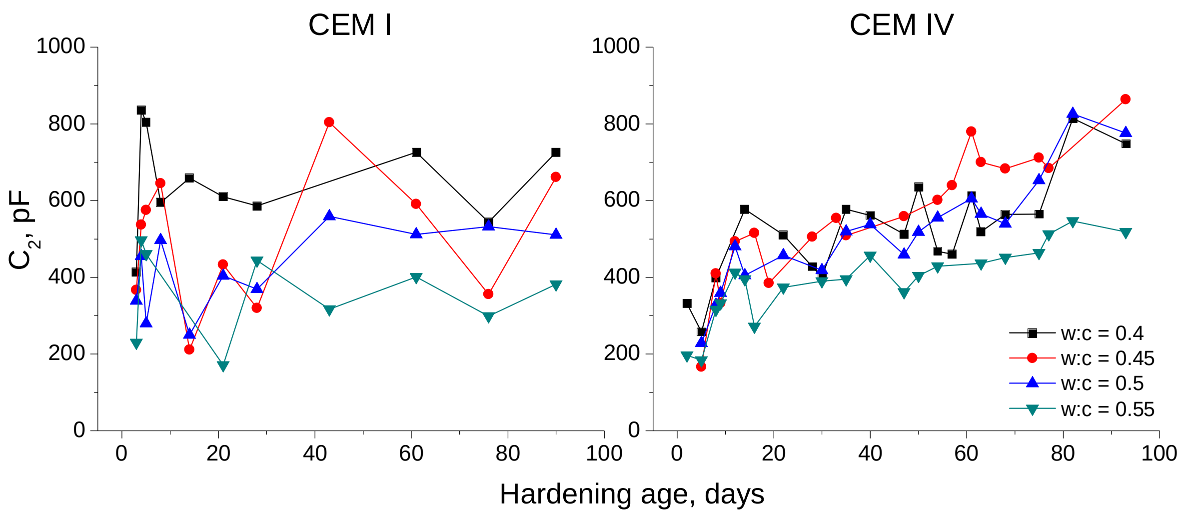

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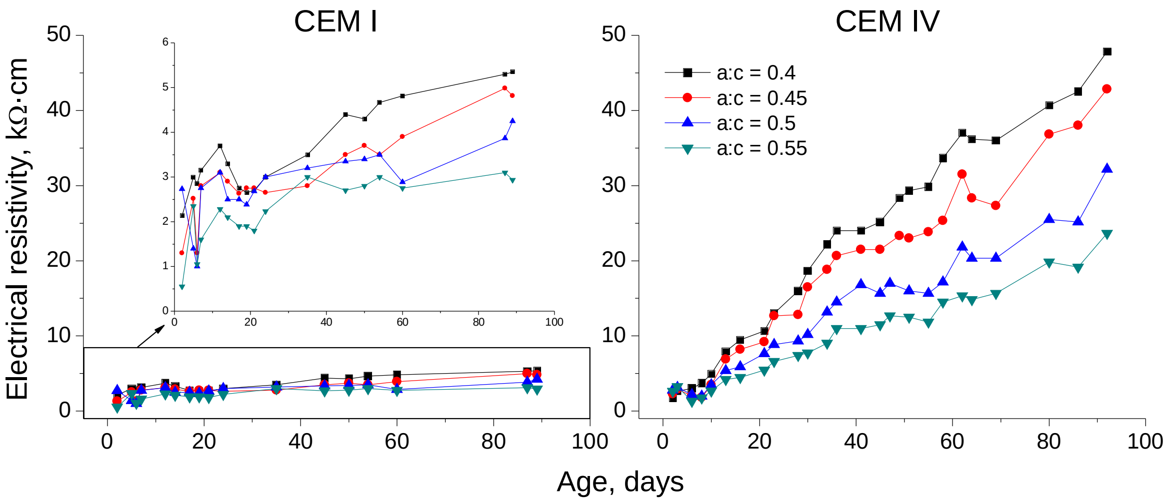

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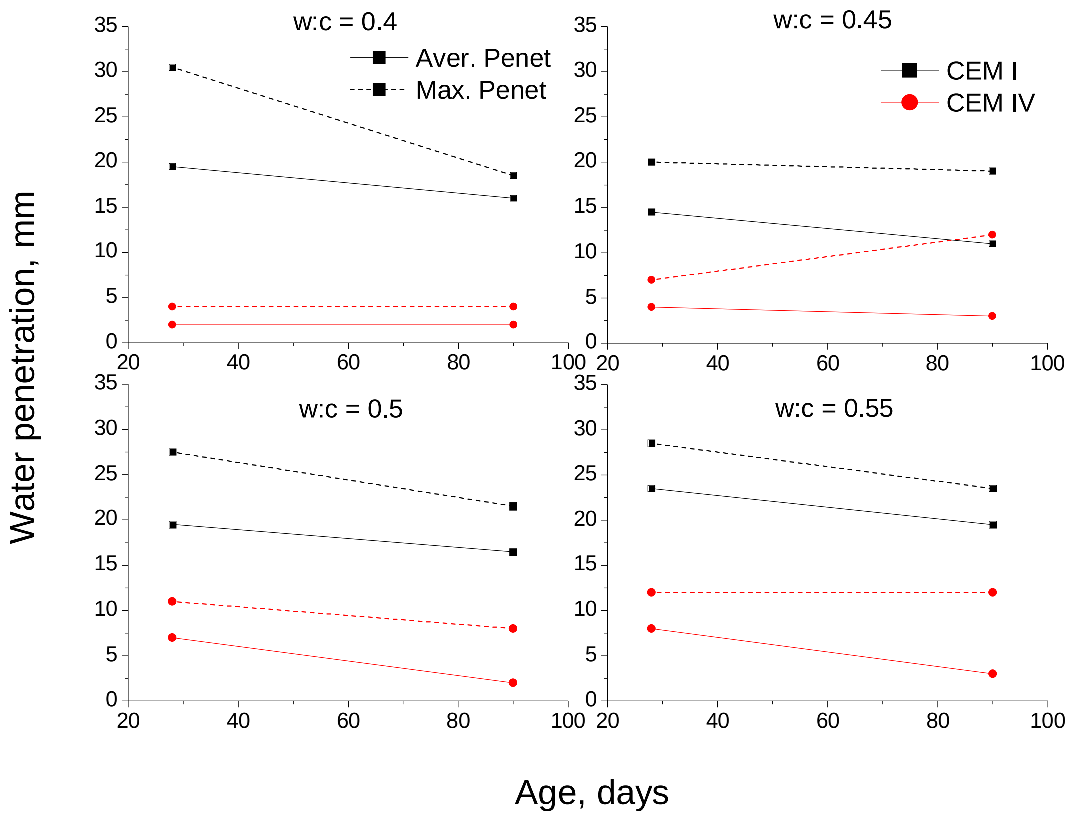

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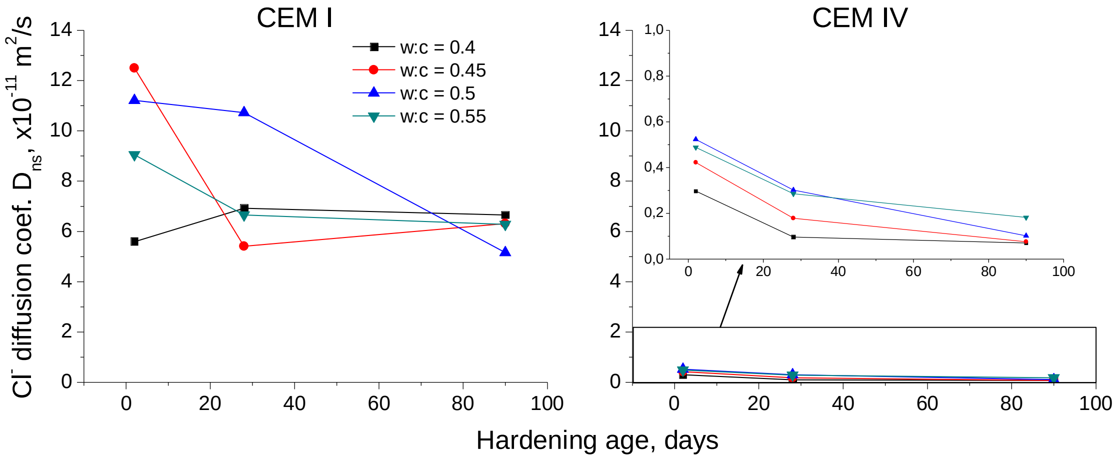

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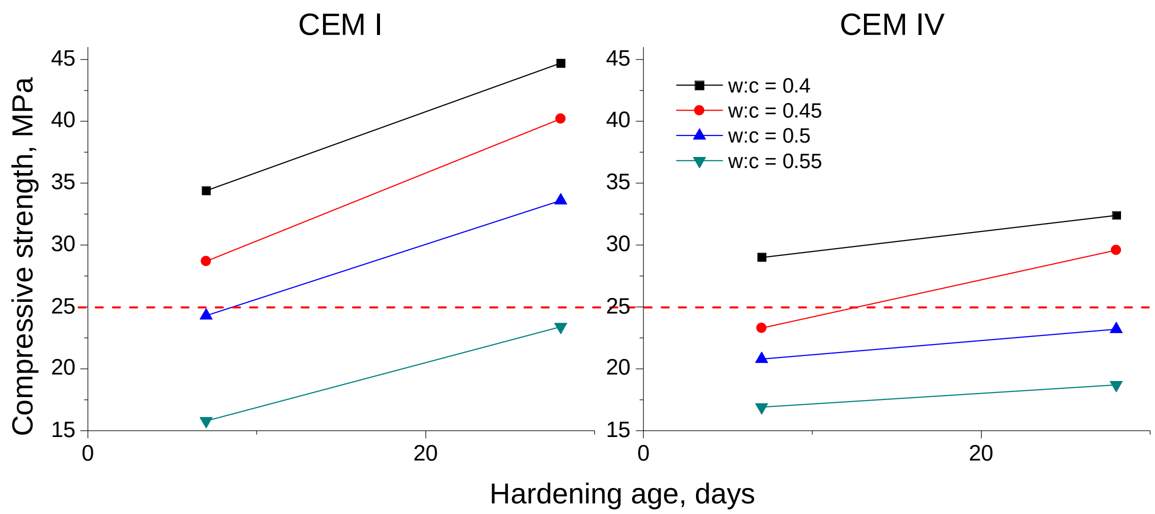

Fig. 15.- Results of compressive strength for CEM I and CEM IV grouts as a function of w:c ratio and age. Red line at 25 MPa denotes the minimum required strength at 28 days by the Spanish standard.

Table 1.- Results of fluidity of the samples made by letting flow the cement paste from the cone described in standard UNE-EN 1015-3

Cement type	CEM I				CEM IV			
w:c	0.4	0.45	0.5	0.55	0.4	0.45	0.5	0.55
Aver. diam., mm	13.75	16.65	21.25	24.15	16.35	20.4	22.9	24.4

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