A COMPARISON OF COLLOIDAL NANO-SiO$_2$ APPLICATION IN EARLY AGE HARDENED LIMESTONE MORTAR BY TWO DIFFERENT METHODS: ELECTROCHEMICAL MIGRATION AND CAPILLARY ABSORPTION

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The addition of nano-SiO$_2$ (NS) in the mix stage of cementitious materials (CM) based in Ordinary Portland Cement (OPC) has been demonstrated as a promising approach to lead the improvement of properties such as microstructure density, hydration kinetic and pozzolanic reaction rate, which are very important parameters concerning to the structure durability. However, in the case of hardened structures, the mechanism of how NS interacted with cement matrix is limited, because most of the involved reactions have already happened. In this work, the effect of the colloidal nano-SiO$_2$ application in the surface of an early age hardened limestone mortar is studied and a comparison between two different methods is presented: migration (by electrical field) and capillary absorption (by wet-dry cycles). Measurements of potential difference, current intensity and electrical resistance were carried out in migration cells and electrical resistance for capillary absorption cell. Optical microscopy (OM) and scanning electron microscopy (SEM) in backscattering electrons (BE) coupled with energy dispersive x-ray spectroscopy (EDS) were used to characterize the microstructure of the cement mortar samples. It was found that the nano-SiO$_2$ penetrate the cement mortar sample by both migration and capillary absorption from the surface to the interior and interact with the phases of the cement matrix. Ca/Si rates below the conventional values of the conventional phases were found (up to 0.5), which means that an increasing of cement matrix silicon content was promoted.

Keywords: *Limestone Cement Mortar, Nano-SiO$_2$, Surface Treatment.*

1. Introduction

Today more than ever, the construction industry is required to attend all social, economic and environmental problems concerning about its performance and processes, that means, take actions to mitigate all the negative effects in the three main sectors of our society (social, economic, environment) and look for the sustainable development of this industry. One of the most important issues affecting the environment is the process of building repair: up to 30% of the cement produced is used in repair and rehabilitation projects [1-4]. Traditional concrete structures repair methods are based on removing a big amount of the deteriorated material from the structure and its later replacement with new materials. Thus, it emerges the need of use more cement (with its respective CO$_2$ generation), water, aggregate, energy and labor hours; which lead to entailed extra costs, time out of service structures, solid waste generation and the risk of have compatibility problems between the damaged materials and the repair materials [5-7]. In response to this, the use on nano-materials has been studied as a non-conventional structure fabrication and repair alternative and it ensures a range of new properties for both fresh and hardened cementitious materials. Those properties would not have been possible to investigate without the development of new nano-technology knowledge [8-11].

One of the most investigated nano-materials in the building industry is the amorphous nano-SiO$_2$ (NS) because it is the major component of pozzolanic materials and it has high potential reactivity with portlandite (Ca(OH)$_2$), where NS acts as secondary C-S-H gel precursor [12-16]. The hydration kinetics of the pozzolanic reaction is proportional to the surface area of the involved particles in the process, whereby the nano-scale of the NS dramatically promote the reaction rate. Some studies have found that the addition of nano-SiO$_2$ with simultaneous substitutions of supplementary cementitious materials during the fabrication of concrete (mixing stage), can result in improvements of the early strength development and its the

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reaction rate. However, not much information is available about interaction of these nano-materials with already hardened structures [17-19].

This study is focused on the possibility of use NS in early age hardened limestone mortar by two different methods: electrochemical migration and capillary absorption, in order to evaluate the transport mechanism and microstructure interaction. The process of nano-particle migration is carried out here through the use of an electric field set by two electrodes around a mortar sample; which force charged particles to move in certain direction. The use of an electric field in building repairs has been used before in order to extract chloride ions from damaged concrete [20-23]. In the case of capillary absorption, this phenomenon is taken account because allows a considerable amount of transport mechanism in concrete microstructure and its respective effects related with the absorbed specie [24-27]. Limestone as the aggregate in the cement mortar was proposed because it can contrast with the used NS. In a silica sand cement mortar, it would be very difficult to detect migrated NS, because the microstructure is already enriched with Si. Both physical and chemical effects of the nano-particles in the cement matrix were analyzed.

2. Experimental

2.1. Material

Ordinary portland Cement (OPC: Compressive strength 42.5 Mpa at 28 days, clinker 95-100%, chlorides ≤0.1%, sulphate ≤4%, high resistance to saline environment and sulfates. Main calcium phases: C_3S 57.91%, C_2S 6.28%, C_3A 3.82%, C_4AF 15.44%)) was used to fabricate cylindrical limestone mortar samples with a diameter of 7.5 cm and a height of 15 cm. The standard used for sample preparation was UNE-EN 196-1 and ASTM C-109. Both of them with specifications about the fabrication of mortar for strength testing. Limestone aggregate ($CaCO_3$ 97.5-99.5%, SiO2 0.1-1%) was used because the silica aggregate can interfere with the analysis detection of nano-particles (silica sand mortar already has a silicon enrichment matrix) and distilled water was needed to avoid the intrusion of external ions. Granulometry analysis was carried out to compare particle size distribution (as it can be seen in Figure 1) with the standard of UNE-EN 196-1, as a basic requirement to the proper mortar fabrication.

The design of the mixtures was made according to the mentioned standards, with a cement content of 450 grams, 1350 grams of aggregate, and a water/cement ration of 0.5. Mortar samples were cured for 24 hours at room temperature and then unmolded and stored in a curing room with a temperature of 22 ± 2°C and a relative humidity of 95 ± 5 % during 7 days.

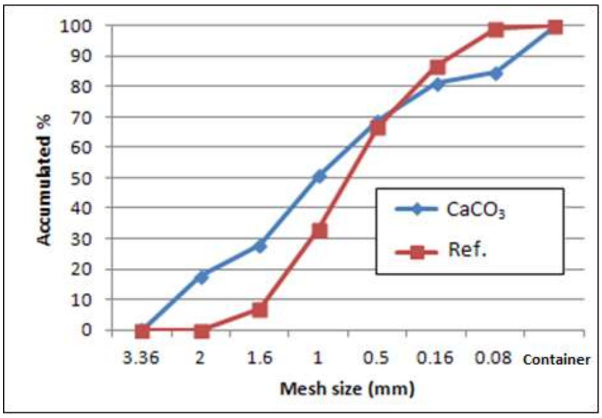

Fig. 1 - Granulometry analysis of $CaCO_3$ aggregate for the mortar fabrication.

A commercial colloidal nano-SiO_2 was used for migration and capillary absorption experiments, with a concentration of 30% wt. and a particle size of 7 nm. This product is normally used as a sealing layer or as a component in painting cover in structures. Figure 2 shows an x-ray diffraction analysis of the colloidal nano-SiO_2 after dried at 100°C and ground the obtain a proper sample. The amorphous nature of the material microstructure can be seen on the range 5 to 60 of the 2θ axis in the XRD analysis, which demonstrates the feasibility to be used as a pozzolanic component.

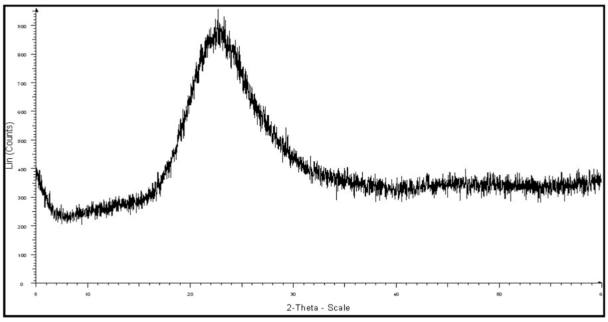

Fig. 2 - X-ray diffraction analysis of colloidal nano-SiO_2 after dried and ground.

2.2 Sample Preparation

Once the samples were cured for 7 days, they were cut as shown in **Figure 3**, where the top and bottom were removed to avoid sections with cement segregation. Samples of 1 and 5 cm of thickness were obtained from the remaining mortar sample to be used as migration and capillary absorption samples respectively. Samples were subjected to a water saturation process (standard UNE-EN1936/1999) on the following way: first samples were placed in a vacuum container with a reduced pressure of 133 Pa during 3 hours. After that, water was poured in the container for an hour while the vacuum pump was turned on. Finally, the mortar samples were kept in the water for 18 ± 2 hours at temperature of 20 ± 2 °C and then they were stored until the day of the experiment (7 days aged).

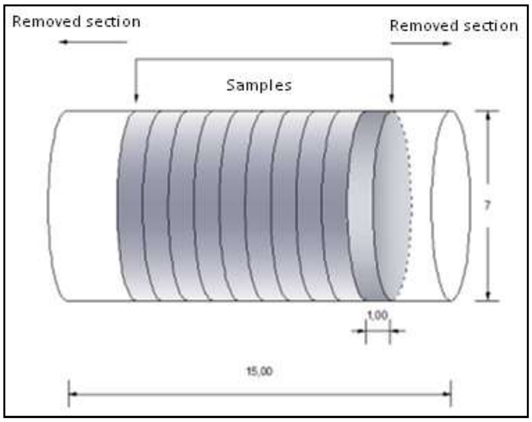

Fig. 3 - Scheme of preparation and cutting of mortar sample used for migration experiments.

2.3 Migration Cell Assembly

The assembly of the migration cells was carried out according to an electrical configuration, from which was possible to corroborate that an ionic transport from the surface to the interior of a mortar sample does exist. This electrical circuit was determined from the polarization of the nano-SiO_2 (slightly negative charged) and it did not consider other factors regarding oxidation-reduction reactions occurring in the electrodes, mainly due to water hydrolysis and which can affect the embedded steel in real practice (reinforcement concrete). The main aim of this arrangement is only to study ionic transport.

Cylindrical acrylic cells, with an intern diameter of 7.5 cm, were used where the mortar sample could be disposed. The cells had two accesses where the electrodes could be put and the electrical and electrochemical measurements could be performed. An IPS-405 programmable power supply from ISO TECH was used coupled with a multiplexor 34972A from Agilent Benchlink. These equipment allow the connection of multiple experiments at the same time, as well as the performing of continuous electrical measurements (difference potential and current intensity). The electrical circuit is shown in Figure 4.

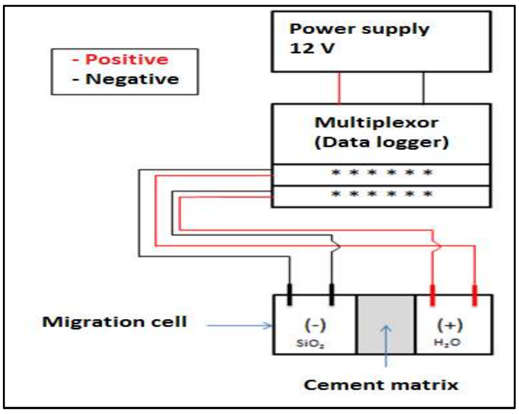

Fig. 4 - Schematic Configuration of the electrical circuit of migration cells.

2.4 Capillary Absorption Cells

Another arrangement was performed to carry out capillary absorption experiments to study the movement of nano-particle without the influence of an electrical field in mortar samples with a height of 5 cm. This time, the mortar sample was settled in a cylindrical container which acted as a pond. An absorbent material and electrodes were placed in the top and bottom sides of the sample to ensure the electrical conductivity and to perform electrical measurements at the end of each cycle. Finally, the cell was wrapped in a plastic cover to avoid evaporation of the nano-SiO_2 solution. The procedure consisted of 4 wet-dried cycles (1 week for cycle). The arrangement can be seen in Figure 5.

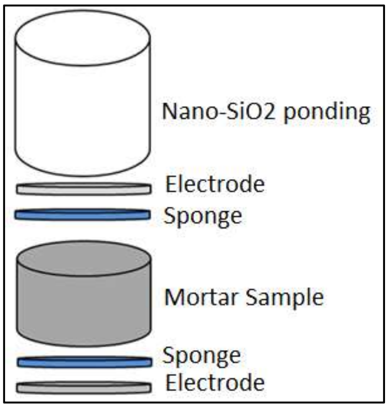

Fig. 5 - Arrangement of a capillary absorption cell for wet-dry cycles.

3. Experimental procedure

Once the mortar sample had been disposed inside the migration cell, one compartment (catholyte, negative electrode) was filled with colloidal nano-SiO_2 and the other was filled with distilled water (anolyte, positive electrode), then a 12V electrical potential was applied and an electrical current was impressed through the cell. Electrical potential and current intensity were continuously measured. Electrical resistance was calculated from those values. Samples of 5 ml of solution from both compartments were taken to measure pH and conductivity, and they were replaced with the same quantity to keep the cell filled. A general scheme of the migration cell is presented in Figure 6. When the experiment ended, the mortar sample was removed from the cell to be characterized later by different techniques.

In the case of capillary absorption cells, electrical resistance was measured when nano-SiO_2 was applied (at beginning of wet stage) and when it was removed. Thus initial and final values of resistance were obtained to future analyses.

For characterization, samples were fractured and a cross section was obtained. Then, the positive and negative edge of the samples were identified and the sections were separately analyzed.

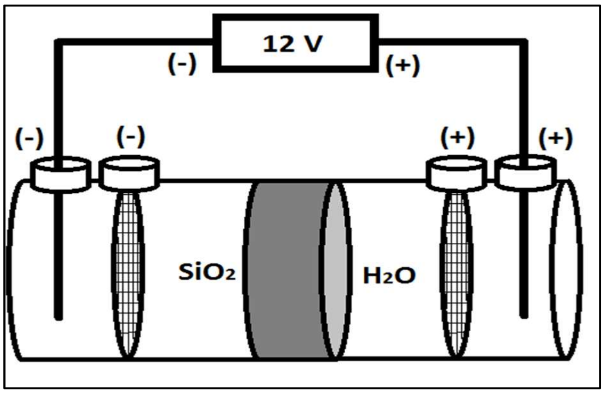

Fig. 6 - General scheme of the electrical circuit of a migration cell.

4. Results and Discussion

4.1 Electrical measurements

The electrical measurements obtained along the experiment in the migration cell are presented in Figure 7 (a) and (b), where the behavior of electrical potential and current intensity of the configured circuit in the migration cell can be observed. Potential difference, with an initial value of 6V, shows a tendency to oscillate during the experiment, but at the end, a final increment of 6.6V was presented after 8 days. Current intensity shows a variation in the first 50 hours of experiment and then a decrement from an initial value of 9.9 mA to 5.4 mA at the end of the experiment with the migration cell.

From the continuous measure of electrical potential and current intensity in the migration cell, it is possible to calculate the electrical resistance of the circuit through the ohm's law (R=V/I). Figure 7 (c) shows the behavior of this parameter and it can be observed that a variation related to current intensity exists, and it has a general tendency to increase, with an initial value of 606 Ω and a final value of 1215 Ω. Although these electrical resistance measurements were performed considering the cement mortar sample microstructure, the nano-SiO_2 solution, the distilled water and the electrochemical reactions (redox) in the electrodes as a whole electrical circuit, it is possible to expect an increase of electrical resistance due to two main phenomena [28]: a) the normal hydration process of the cement mortar sample; where the nucleation and growth of the C-S-H gel phase can reduce the available space to the electrolyte pore solution inside the matrix; and b) the physical effect of nano-SiO_2 of sealing the pore system by flocculation and coagulation after being migrated to the interior of the sample, as well as the possible pozzolanic reaction carried out in presence of portlandite to form new C-S-H compounds.

Figure 7 (d) shows the punctual measurements of electrical resistance in the case of the sample in the capillary absorption cells, taken from a potentiostat-*galvanostat equipment connected to the electrodes inside the cell and using a frequency of* 10000 Hz. It is possible to observe, in the first wet-dry cycle, that it did not present any significant variation of the electrical

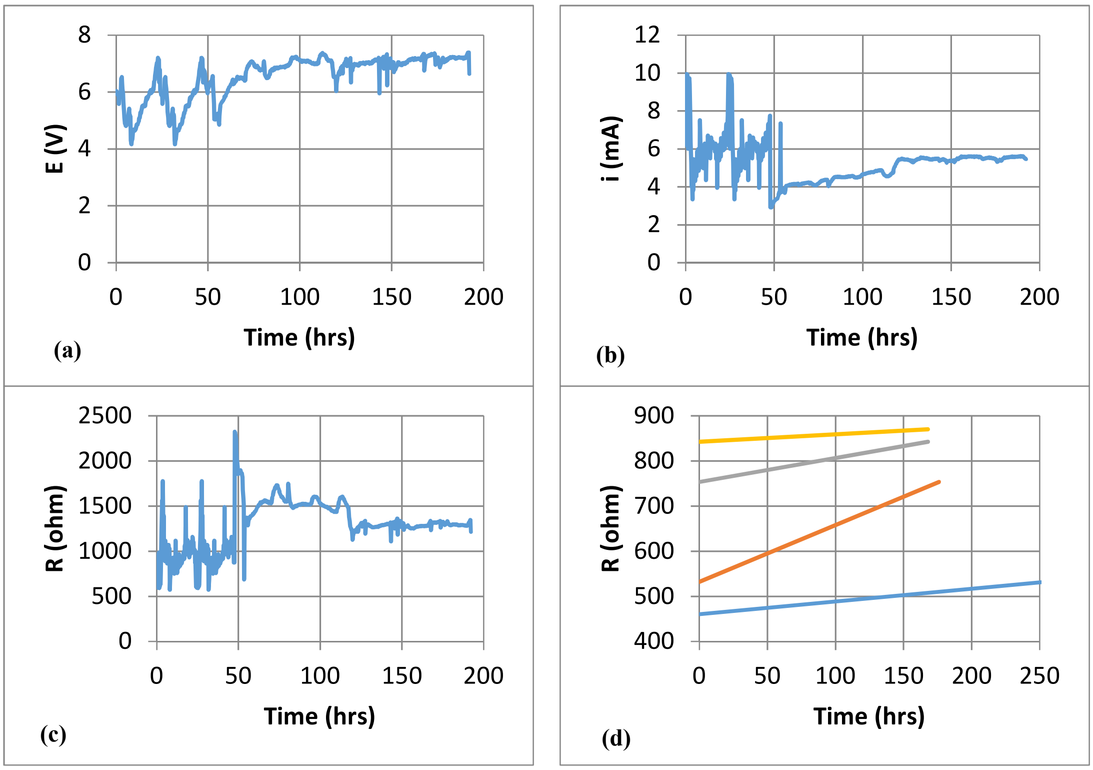

Fig. 7 - Electrical measurements of cells. (a) Electrical potential of migration cell, (b) Current intensity of migration cell, (c) calculated electrical resistance of migration cell and (d) 4 cycles of electrical resistance of capillary absorption cell.

resistance (as seen in Table 1). However, cycles 2 and 3 present a very pronounced slopes, which means a rapid increment of this parameter. Finally, in the last cycle, the value of the resistance stops to increase and it shows a behavior without any significant modifications. These values show the behavior of the mortar sample microstructure, where is probable that an interference between NS and the mortar is created [29-30], causing an electrical barrier phenomena which could affect the analysis of the electrical resistance. On the other hand, is it possible to attribute the increment of the electrical resistance to the same reasons of the migration cell, this means, due to the normal hydration process of the mortar and the physical and chemical effect of the nano-SiO_2.

Table 1
Electrical resistance measures of cycles in capillary absorption cell

Resistance (ohms)	Cycle 1	Cycle 2	Cycle 3	Cycle 4
R initial	460	532	753	842
R final	532	753	842	870

4.2 pH and conductivity measurements

Samples taken from both positive electrode and negative electrode compartments were analyzed. Figure 8 (left) outlines the behavior of the pH parameter in the migration cell for SiO_2 and distilled water compartments. The negative electrode compartment shows a tendency to increase probably due to the hydroxyl ion production by water hydrolysis, presenting an initial value of 6.6 and a final value of 11. Contrary to this, positive compartment shows a generally constant behavior along the experiment. There is just a small variation tending to increase the value from 9.5 to 10.4, which is not completely expected because of the tendency to corrode of the positive electrode. This is maybe the result of the lixiviation process of ionic species from the cement matrix to the solution.

Conductivity behavior in the negative compartment could not be measured because the nano-SiO_2 became slightly solid and the equipment to measure was not possible to operate after 4 days of experiment. However, the analysis obtained before, did not show any significant change and kept constant values between 3200 µS and 3300 µS. In the case of the positive compartment, the distilled water presented a tendency to increase its conductivity, from an initial value of 40.3 µS to 173.8 µS at the end of the experiment. This increment can be seen as an evidence of actual ionic transport through the electrical circuit configured in the migration cell (Figure 8 (right)The nature of the increment of this conductivity can be due to a couple of factors as leaching from mortar sample or redox reaction species produced during the experiment, nevertheless a future chemical analysis of this water solutions should needs to be carried out.

4.3. Optical Microscopy

A polished surface of a mortar sample cross section after migrated nano-SiO_2 treatment was analyzed by optical microscopy, where the positive and negative edges were identified. The images left side corresponds to the surface in contact with the colloidal nano-SiO_2 and the right side corresponds to the surface in contact with distilled water. The analyzed samples were polished using silicon carbide sandpaper in order to obtain a more detailed surface and to be able to identify the visual effect of the nano-SiO_2 [31]. In Figure 9 it can be observed that the sample presented a distribution of the aggregate particle size with wide dispersion due to the granulometry characteristics of the material. But the most significant aspect is the presence of a region clearly affected in the surface in contact with the nano-particles, which presented a different color with an approximately depth distance of 1.5 to 2 mm.

After capillary absorption treatment (see Figure 10) the mortar sample presented an affected area (top) which was not as significant as that in the migration cell. The following analysis (SEM-EDS) were needed to determine the nano-particles real effect.

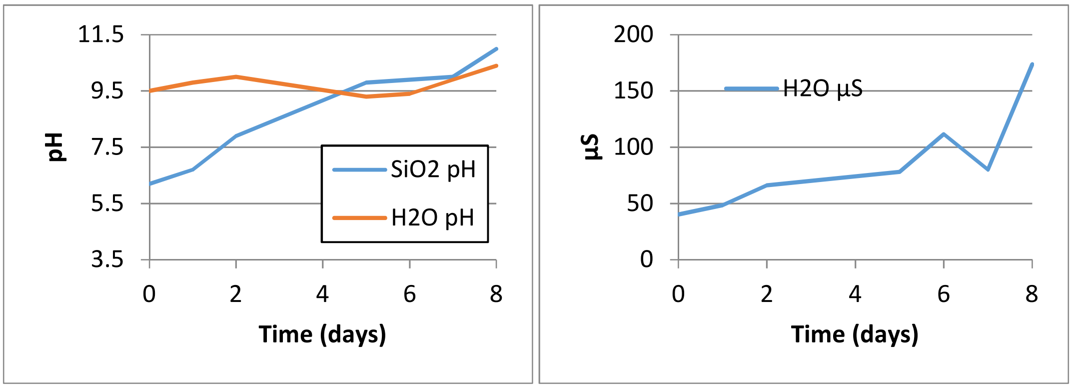

Fig 8 - pH behavior of catholyte (SiO_2) and anolyte (H_2O) compartment (Left) and conductivity of anolyte (H_2O) (Right).

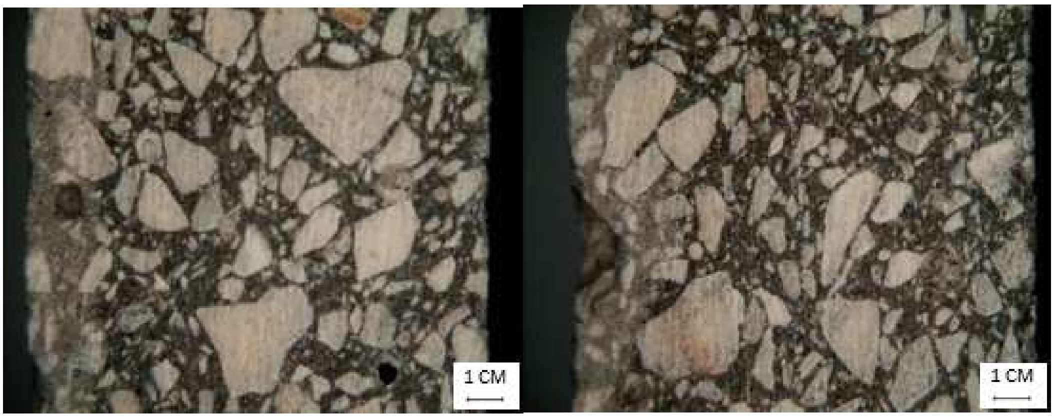

Fig. 9 - Mortar sample cross sections after migration treatment. Left edge corresponding to the surface in contact with nano-SiO_2 and right edge corresponding to the distilled water compartment.

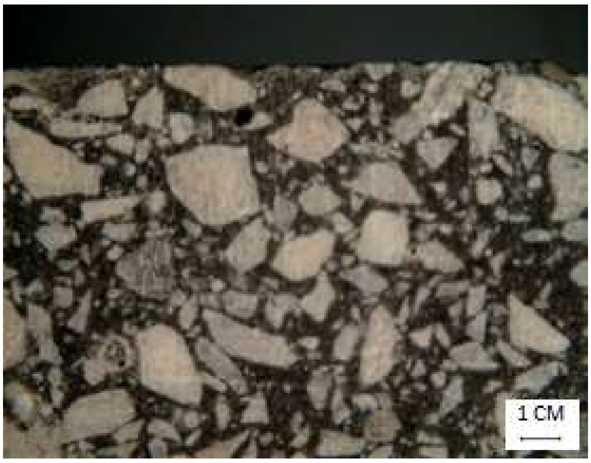

Fig. 10 - Optical microscopy image of the mortar sample after capillary absorption treatment with left side exposed to nano-SiO_2.

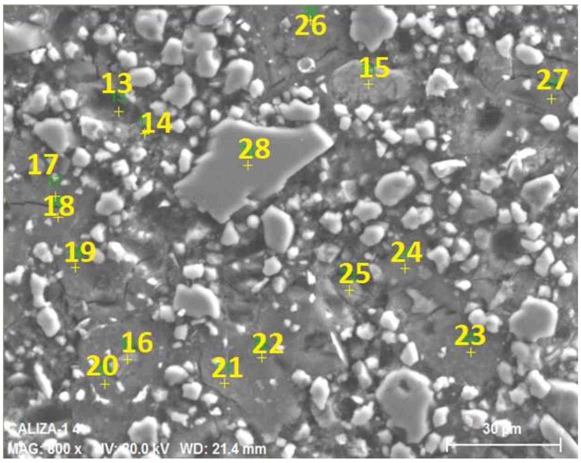

Fig. 11 - SEM image with BES of a mortar sample without migration treatment (control sample).

4.4. SEM/BE-EDS migration cell

Analysis by scanning electron microscopy in backscattering mode coupled with energy dispersive x-ray spectroscopy detector was carried out to determine the effect in the microstructure of the nano-particles at a micrometric scale and an elemental chemical analysis was performed as well. Samples were put in resin and they were polished in order to obtain adequate samples for measurements. Results in Figure 11 show a surface without nano-SiO_2 application (control sample) where can be seen (at 2 mm from the surface) a microstructure very similar to a conventional cement matrix; composed of C-S-H gel, anhydrous phases and aggregate particles. It can be observed in Figure 12 that the values of calcium and silicon content of this sample represent a Ca/Si ratio similar to that conventional reported values for the anhydrous phases ($C_3S=3$, $C_2S=2$) and C-S-H gel (1.5 – 2). Also, values of calcium

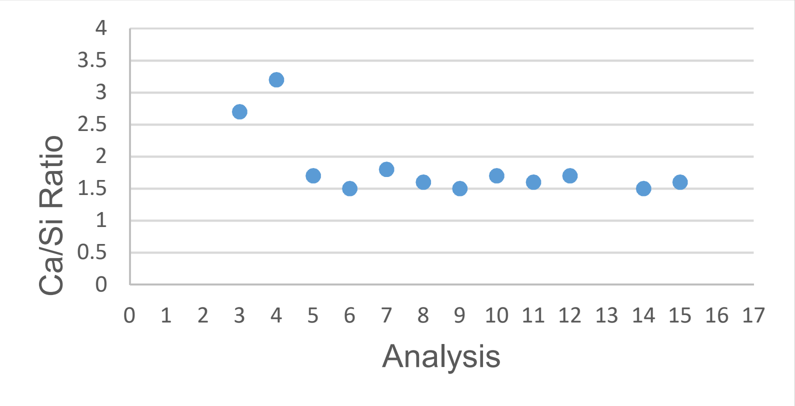

Fig. 12 - SEM-EDS analysis for chemical composition of the Ca/Si ratio in a control sample.

content are present up to 99%, which clearly means the presence of the $CaCO_3$ aggregate, but they went out of the chart (max value pointed was 4).

SEM image in Figure 13 presents a mortar sample after electrochemical migration treatment where a type of morphology has been identified, which resulted from the interaction between nano-SiO_2 and the cement mortar matrix. Figure 14 presents the elemental chemical analysis in some points of the polished surface. There are some points in the center of that particular morphology with Ca/Si ratios with values between 0.8 and 1 and analysis around those points shows values corresponding to a conventional C-S-H gel and others of anhydrous phase farther from the center. These Ca/Si ratios below of conventional values (1.5-2) indicate an enrichment of silicon, which can be an evidence of the effective transport to the interior of the mortar sample. [32].

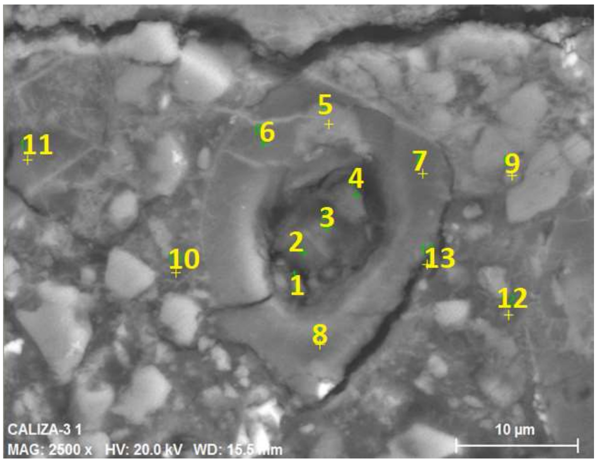

Fig. 13 - SEM image of a mortar sample after nano-SiO2 migration treatment.

Figure 15 shows other SEM image of the mortar sample after migration treatment, where a spherical particle was identified. Elementary analysis indicates that this particle is composed by a high content of Si (see Table 2), which suggest that the particle is related to the process of external nano-SiO_2 application. Surrounded areas show a modification in their content of silicon and present values similar to those of C-S-H gel.

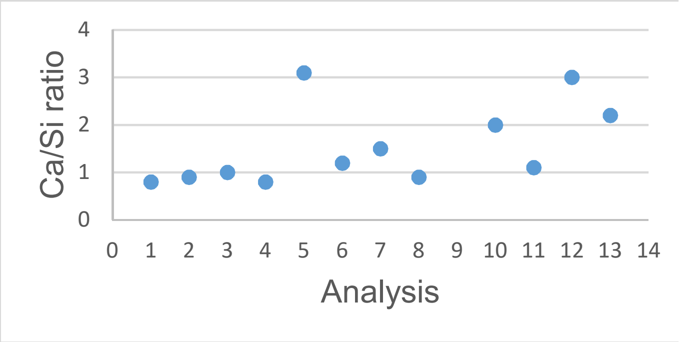

Fig. 14 - SEM-EDS analysis for chemical composition of the Ca/Si ratio in a migrated sample.

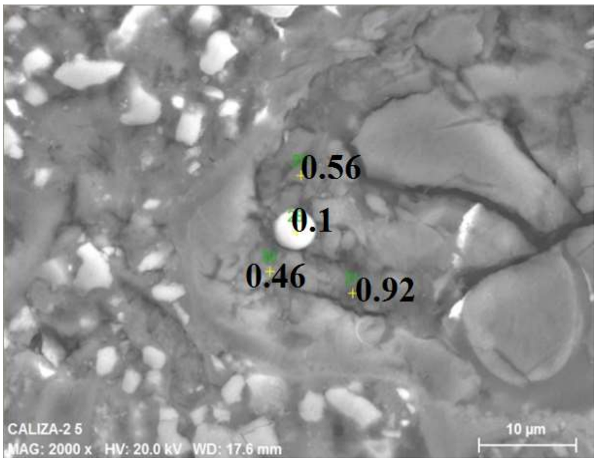

Fig. 15 - SEM image of a mortar sample after nano-SiO_2 migration treatment with the presence of a spherical particle related with the migration.

Table 2

Elementary chemical analysis of the mortar sample after nano-SiO_2 migration treatment (From to Figure 15).

Element	(%) / analysis			
Silicon (Si)	47.71	54.16	57.63	43.06
Calcium (Ca)	5	30.79	26.52	39.71
Ca/Si ratio	0.1	0.56	0.46	0.92

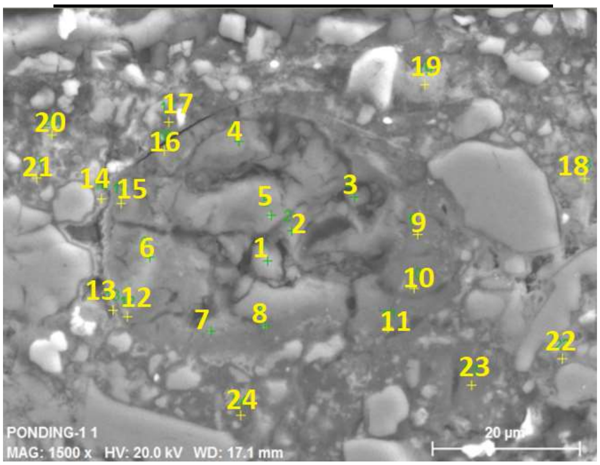

Fig. 16- SEM image of a mortar sample after nano-SiO_2 capillary absorption treatment.

4.5. SEM/BE-EDS capillary absorption cell

The mortar sample after capillary absorption treatment for SEM analysis was prepared in the same way of the migration samples, and the image is showed in the Figure 16. The zone is the edge at a distance of 20 µm from the surface which was in contact with the colloidal nano-SiO_2 and it shows a non-conventional morphology, where EDS analysis where performed. The Figure 17 indicate that the zone is composed by silicon up to 86.6%. These high contents of silicon do not correspond to any phase of a conventional cement matrix and they can just be an agglomeration of nano-SiO_2 on the pore system [33]. The above

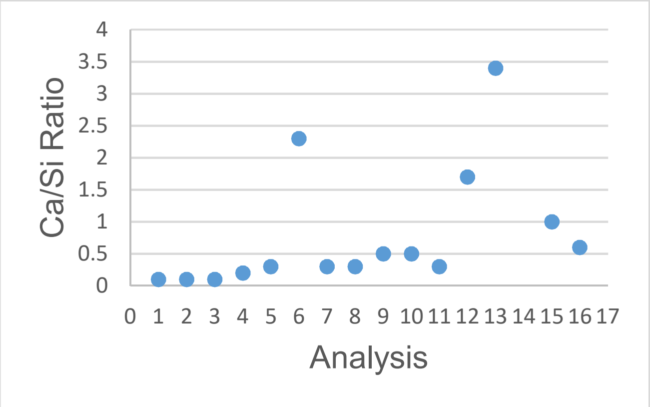

Fig. 17 - SEM-EDS analysis for chemical composition of the Ca/Si ratio in a capillary absorption sample.

mentioned is possible to infer because the absence of a silicon aggregate permits to identify the presence of external addition of SiO_2.

5. Conclusions

1. It is possible to obtain, with SEM-EDS analysis, significant evidence of the nano-SiO_2 transport into the limestone mortar sample. The cement mortar matrix presents an enrichment of Si and Ca/Si ratios values quite below of typical anhydrous and hydrated phases. Also, there are some indications which lead to conclude that the mechanisms consist in the agglomeration of nano-SiO_2 around anhydrous phase and then the subsequent incorporation of the silicon to present phases.

2. Electrical measurements carried out in a migration cell can contribute with important information about the microstructure of the mortar (porosity), where the value of the electrical resistance shows a tendency to increase, probably due to the combined effect of the hydration reactions progress and to the saturation of the pores systems by nano-SiO_2 as well as a possible pozzolanic reaction with portlandite. It is necessary future analysis to determine this possibility.

3. It can be observed by optical microscopy that an affected area appears in the mortar sample after migration treatment, with an approximate depth distance of 1.5 to 2 mm. In mortar samples after capillary absorption treatment, it could not be appreciated the affected region in the same way as migration treatment. Although, a penetration of the nano-particles is presented and analyzed by SEM-EDS. The difference between transport of the nano-particles by migration and capillary absorption seems to be the feasibility to react to the phases on the mortar sample, being this more promoted when an electrical field is applied and aslo, the penetration distance is higher in the electrochemical method.

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MANIFESTĂRI ȘTIINȚIFICE / SCIENTIFIC EVENTS

SynerCrete18: Interdisciplinary Approaches for Cement-based Materials and Structural Concrete: Synergizing Expertise and Bridging Scales of Space and Time, Funchal (Madeira Island), 24 – 26.10.2018, Portugal

The focus of the conference is set to communicating, discussing and arousing progresses in research, development and application of Cement-Based Materials and Structural Concrete, which have been attained through combination of expertise from distinct fields of knowledge. Exciting fields of research such as performance-based design, 3D modelling for analysis/design, Building Information Modelling and even robotics (e.g. digital fabrication or robotics design) are included.

Topics:

The conference is established as a final discussion around COST Action TU1404, which is centred in early age behaviour and its interaction with service life of cement-based materials and structures.

A strong focus is given to interdisciplinary approaches and towards new exciting fields of research that inherently require collaborative work among scientific disciplines. Therefore, in view of this global reasoning the following set of sub-topics is envisaged:

- Concrete technology and advanced material testing
- Multi-scale in time and space modelling and experiments
- Multi-physics simulation and structural design
- BIM and structural concrete
- Robotics and cement-based materials
- Digital fabrication
- On-site monitoring and structural condition assessment
- Scientific insights vs. standardization
- New materials : bio-based, geopolymers and alkali-activated materials
- Fibre-reinforced concrete and non-metallic reinforcement

https://synercrete.com/