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Investigación Básica

Use of nano-SiO_2 as a preventive maintenance surface treatment in concrete aged by carbonation

Uso de nano-SiO_2 como tratamiento superficial de mantenimiento preventivo en concreto envejecido por carbonatación

Uso de nano-SiO_2 como tratamento de superfície como manutenção preventiva em concreto envelhecido por carbonatação

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Abstract:

This study aims to evaluate the use of silicon base (NS) and functionalized (NF) nanoparticles as emerging preventive surface treatment (ST) in reinforced concrete specimens. The specimens were fabricated with a water/cement (w/c) of 0.65 and subjected to a previous aging period through exposure to CO2. Subsequently, two different variants of the treatment were applied by spraying (using a 0.1% dispersion of nanoparticles in water) and then re-applied to carbonation. The carbonation depth and contact angle results indicate that there is an influence between the degree of aging and the efficiency of each treatment.

Keywords: surface treatment, prevention, Nano SiO_2 , concrete, carbonation.

Resumen:

Este estudio, tiene como objetivo evaluar el uso de nanopartículas base silicio (NS) y funcionalizadas (NF) como tratamiento superficial (ST) preventivo emergente en especímenes de concreto reforzado. Los especímenes fueron fabricados con una relación agua/cemento (a/c) de 0.65 y sometidos a un periodo de envejecimiento previo mediante la exposición a CO2. Posteriormente, dos diferentes variantes del tratamiento fueron aplicadas mediante aspersión (usando una dispersión de 0.1% de nanopartículas

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en agua) y después fueron sometidas nuevamente a carbonatación. Los resultados de profundidad de carbonatación y ángulo de contacto indican que existe una influencia entre el grado de envejecimiento y la eficiencia de cada tratamiento.
Palabras clave: tratamiento superficial, prevención, Nano SiO_2 , concreto, carbonatación.

Resumo:

Este estudo tem como objetivo avaliar a utilização de nanopartículas à base de silício (NS) e funcionalizadas (NF) como tratamento preventivo de superfície (ST) emergente em corpos de prova de concreto armado. As amostras foram fabricadas com uma relação água/cimento (a/c) de 0,65 e submetidas a um período de envelhecimento prévio por exposição ao CO2. Posteriormente, duas variantes diferentes do tratamento foram aplicadas por pulverização (utilizando uma dispersão de 0,1% de nanopartículas em água) e, em seguida, submetidas à carbonatação novamente. Os resultados de profundidade de carbonatação e ângulo de contato indicam que existe uma influência entre o grau de envelhecimento e a eficiência de cada tratamento.
Palavras-chave: tratamento da superfície, prevenção, Nano SiO_2 , concreto, carbonatação.

1. INTRODUCTION

In the construction industry, reinforced concrete has become the most used material worldwide, due to its low cost, ease and speed of manufacture, not to mention the combination of the high compressive strengths provided by concrete and the mechanical properties of steel that make it the ideal composite material for structural applications (Aguirre and Mejía de Gutiérrez, 2013).

Deterioration in reinforced concrete structures (RCS) caused by corrosion of reinforcing steel has been the subject of study in recent decades. Some specialists (Hernández-Castañeda and Mendoza-Escobedo, 2006; Polder, Peelen and Courage, 2012; Angst, 2018) consider it the greatest challenge facing science and technology worldwide in the construction industry. The importance falls from the technical, economic and social concerns that causes the durability of the RCS, mainly when the reinforcing steel is exposed to aggressive environments. This is the case of exposure to chlorides, either from the raw material of concrete or by penetration from the surrounding environment or due to the carbonation of the concrete, which is another cause of corrosion of the reinforcement of these structures.

More than 50% of the RCS in service have deterioration problems due to the high permeability or low concrete quality. Direct losses due to costly maintenance work, and mainly repair work on structures worldwide, have been economically higher, especially in first world countries (Alhozaimy *et al.*, 2012). Currently, annual expenses are generated between 18 to 21 billion USD in rehabilitations or repairs to RCS due to corrosion of reinforcing steel (Beushausen and Bester, 2016). Reinforcing steel is protected against the corrosion process by the alkalinity of the concrete, which is the result of the cement hydration process. The pH decreases when the physical-chemical carbonation process occurs. Carbonation occurs by the reaction between CO_2 and $Ca\ (OH)_2$ producing $CaCO_3$ (1). As a result, $CaCO_3$ precipitates by reducing the pH in concrete, significantly decreasing the durability of reinforced concrete and its useful life.

$$Ca\ (OH)_2 + CO_2 \rightarrow CaCO_3 + H_2O \tag{1}$$

The application of surface treatments (ST) in particular has been extensively investigated in the last decades (Pigino *et al.*, 2012; Pan *et al.*, 2017a; Vivar *et al.*, 2017; Hou *et al.*, 2018). They are economical and effective methods to improve the concrete quality of the surface area and to protect RCS if compared with other methods (i.e.: decreasing the w/c ratio and using pozzolan additions, increasing coating thickness, etc.)

Most ST can reduce water permeability in concrete, specifically hydrophobic impregnation, which has been achieved with the use of silanes and siloxanes, that prevents the ingress of water without hydrostatic pressure. To retard the advance of carbonation, silicate-based treatment has a more effective protection than silane and siloxane; these hardly prevent the entry of CO_2 (Pan et al., 2017b).

Nowadays, the use of nanomaterials as products for indirect protection (on concrete) of steel has been extensively studied, demonstrating in some cases that they are able to improve the performance of construction materials. Particularly in concrete, the development of smart properties that have the ability to self-clean, antimicrobial, hydrophobic, super hydrophobic, as well as the increase in mechanical properties. (Sobolev *et al.*, 2008; Jalal *et al.*, 2012; Kupwade-patil and Cardenas, 2013; Fajardo *et al.*, 2015)

Use of silicon based nanoparticles at early ages has promoted an increase in the electrical resistivity of the cementitious matrix, a decrease in the degree of CO. penetration and a decrease in permeability. (Cruz-Moreno *et al.*, 2017). While the use of functionalized nanoparticles, such as functionalized silica nanoparticles, have allowed the development of surfaces with super hydrophobic, self-cleaning and bactericidal properties (Zhi *et al.*, 2017; Cruz-Moreno, 2019)

The influence of the use of ST to protect the degradation of aged concretes has been studied, providing hydrophobic and consolidating properties (Shen *et al.*, 2019). For long-term results, accelerated aging tests were performed. A decrease in carbonation progression and stable hydrophobicity was found and despite a progressive decrease in its performance over time, the residual effect usually provides a better service life for concrete (Christodoulou *et al.*, 2013; Creasey *et al.*, 2017).

The objective of this research is to analyze the effect and performance that causes the application of functionalized (NF) and non-functionalized (NS) silica nanoparticles on concrete surfaces with a certain degree of aging. On one hand, NF will provide a hydrophobic effect and, on the other, NS will provide a pore-blocking effect. The application of NS and NF is superficially by means of low-pressure spraying. This work focused on evaluating the use of silica-based nanoparticles (NS) and functionalized silica-based nanoparticles (NF) as a surface treatment for preventive maintenance methods in the deterioration of RCS in environments rich in CO_2.

2. PROCEDURE

The following paragraphs will explain the experimental development of the design, manufacture of specimens, obtaining the nanoparticles and ending with the aging by CO_2, in order to study the effect generated by the NS and NF nanoparticles as external agents in a treatment for superficial preventive maintenance in concrete. This study was divided into different stages, in order to gain a better understanding of the behavior and effects of nanoparticles in aged concrete.

2.1 Production of silicon-based nanoparticles (NS y NF).

Silicon nanoparticles (NS and NF) were obtained through sol-gel synthesis, following the procedure detailed in (Cruz-Moreno, 2015, 2019), a summary of its synthesis process is described below.

TEOS tetraethyl orthosilicate was used as a precursor of silica, ethyl alcohol as a solvent, deionized water, ammonium hydroxide as a catalyst and for the functionalization of the NS, 1,1,3,3-tetramethyldisiloxane was used as a surface modifier and nitric acid as dehydrating agent.

The procedure for the synthesis consisted of placing the ethyl alcohol with vigorous stirring at 70°C. Once this temperature was reached, the TEOS was incorporated and the stirring and temperature were maintained for 30 min. Then, the ammonium hydroxide was added, allowing it to react for an additional 30 min. Subsequently, the water was slowly added and allowed to react for an additional 60 min. Then, dropwise, excess ammonium hydroxide was added until a clear gel was formed. After 24 hours, it was placed in an oven at 110 °C for an additional 24 hours, in order to evaporate the largest amount of excess solvent and water.

To obtain the NF, it was carried out during the NS sol-gel synthesis process, where the functionalization was followed through from the addition of the distilled water and until the end of the reaction time of 60 min. Thereafter, 1,1,3,3-tetramethyldisiloxane was slowly incorporated and allowed to react for 120 min for its subsequent dehydration and surface modification when nitric acid was incorporated into the reaction. Afterwards, the ammonium hydroxide was carefully added dropwise, leaving to react for 24 h, at the end of the time it was placed in the oven at 110 °C for 24 h.

2.2 Fabrication of the specimens

Specimens were made with Ordinary Portland Cement (known as CPO 40, in accordance with NMX-C-414-ONNCCE), the proportion was designed according to the ACI 211 standard, using a water/cement ratio of 0.65, which is usual in the construction industry, see Table 1.

TABLE 1
Proposed concrete mixture ACI 211 PCA

w/c	Cement kg/m^3	Gravel kg/m^3	Sand kg/m^3	Water kg/m^3	Compressive Strength MPa	Porosity accessible to water (%)
0.65	330	756	918	215	32	11.25

For tests of compressive strength and porosity accessible to water, cylindrical specimens of lengths of 10 cm in diameter by 20 cm in length were produced. Concrete specimens were made in accordance with ASTM C39/C39M. Subsequently they followed a standard curing period (as indicated in ASTM C-231) to be later tested.

According to the Portland Cement Association (known as PCA), one of the minimum requirements for compressive strength recommended to provide protection to the concrete element in different exposure environments is 25 MPa, this indicates that the mixture complies with the protection recommendation, while the porosity accessible to water is 11.25%, indicating that the Cl. ingress will be high.

The carbonation monitoring was carried out on concrete prisms with dimensions of 10 cm x 10 cm x 30 cm. Curing was carried out with continuous water spray at 20 ° C and 100% relative humidity, where they remained for 28 days.

2.3 Aging by exposure to CO2

At the end of the curing of the specimens, they were exposed to a CO_2-rich environment until carbonation depths that represent different aging conditions were obtained, prior to the application of the NS and NF treatment. For this, three different carbonation depths were selected as initial aging. Taking into account that the average RCS coverage is 20 mm, 0%, 25% and 50% carbonation were considered on the specimens. In other words, the first series is one that has a carbonation depth of 0 mm, the second series 5 mm and the third series 10 mm. The exposure conditions were: an atmosphere of 8% CO_2 in air, 60% ± 10% RH, at 30 °C

2.4 Application of treatments with NS and NF

A dispersion was prepared with a dose of 0.1% of nanoparticles with respect to the volume of water. Magnetic stirring was used at 60 ° C for a period of 1 h, to facilitate the dispersion of the NS, avoiding precipitation and the crush. For the use of the NF, only magnetic stirring was necessary without the use of temperature.

Two surface treatments were applied to each aging stage: a) dispersion with NS and b) dispersion with NF. All treatments are referenced with a control series without treatment (as named here, REF). Before application, all specimens had a preparation, which consisted of cleaning the surface to remove dust, grease or stains. Dispersions were prepared for each treatment, and by means of an atomizer, the solution was sprayed homogeneously over the entire surface of the specimen, leaving a rest of 30 min for the application of a second layer. At the end of the application, the specimens were kept for 14 days under laboratory conditions to promote the reaction between the NS and the cementitious matrix. (Fajardo et al., 2015; Cruz-Moreno, 2019) . After this time, all the specimens were re-exposed to CO_2-using the conditions described in section 2.3- to promote the advancement of carbonation.

3. METHODS

3.1 Compressive Strength

The compressive strength test was performed on 100 x 200 mm cylindrical specimens as indicated by ASTM C 39 using a hydraulic press. The mix was designed according to the ACI 211 standard for a strength of 30 MPa.

3.2 Contact angle (AC).

With the objective of evaluating the hydrophobic effect that NF confers on accelerated aging exposures, the hydrophobicity generated on the concrete surface was determined. This was done by measuring the contact angle at different aging ages following the ASTM D 5725 standard in a KRÜSS Drop Shape Analyzer model DSA25 at 23 °C. Table 2 shows the classification of the surfaces according to the angle θ obtained.

TABLE 2
Classification of the surfaces according to the observable angles θ maximum and minimum

Hydrophilic	Hydrophobic	Super hydrophobic
$\theta_{min}^{max} < 90°$	$\theta_{min}^{max} \frac{< 150°}{> 90°}$	$\theta_{min}^{max} < 150°$

Measurements of five drops of water for injection (2µl) were taken per specimen and an image was immediately taken. Then, it was analyzed with the help of the ADVANCE V 1.9.2.3 software and the contact angle between the liquid and the surface was determined. The same specimens were used to determine the carbonation advance after making the phenolphthalein measurements. The evaluated part was on the surface where the treatments were applied, and it was measured at the same re-exposure ages, as can be seen in Figure 1.

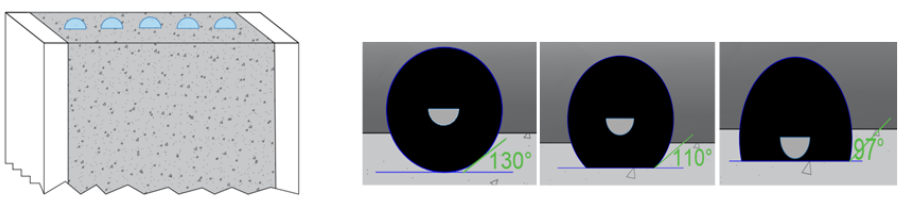

FIGURE 1
Interpretation of the way in which AC measurements were performed on the re-exposed specimens

3.3 Measurement of depth of carbonation

For the measurement of the depth of carbonation, phenolphthalein was used as a conventional indicator. The pink color it gives is an indicator of a concrete in good condition, that is, it still does not have carbonation problems. Figure 2 illustrates the carbonation advance for the concrete sample with and without treatments. For the measurements, the Image J program and a graduated ruler were used as a reference scale, 10 measurements were made per carbonate side of the specimen, having 3 specimens per treatment.

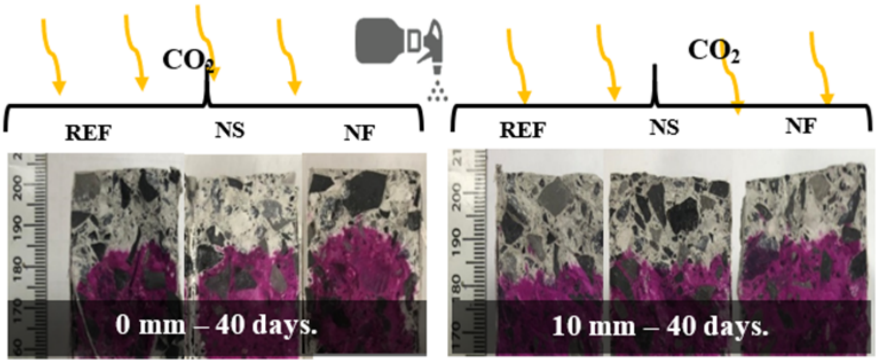

FIGURE 2
Advance of carbonation in samples of 0 and 10 mm of initial carbonation at 40 days of re-exposure, measured with Phenolphthalein.

4. RESULTS

4.1 Contact angle

In the Figure 3, the results of the REF and NF specimens are shown, in which the contact angle was monitored in order to know the behavior of the development of hydrophobicity on the concrete surface. Measurements were made on specimens without prior aging for more than 1000 h after application of the treatment. It can be seen that the REF sample had a practically constant behavior, with an average contact angle of 26.9°. On the other hand, the samples with NF treatment had a uniform behavior from 96 h after application, reaching a maximum angle of 123.7°. According to these results, after 24 h the surface changed from being hydrophilic to hydrophobic; and around 96 h, the NF has a stable AC in the concrete.

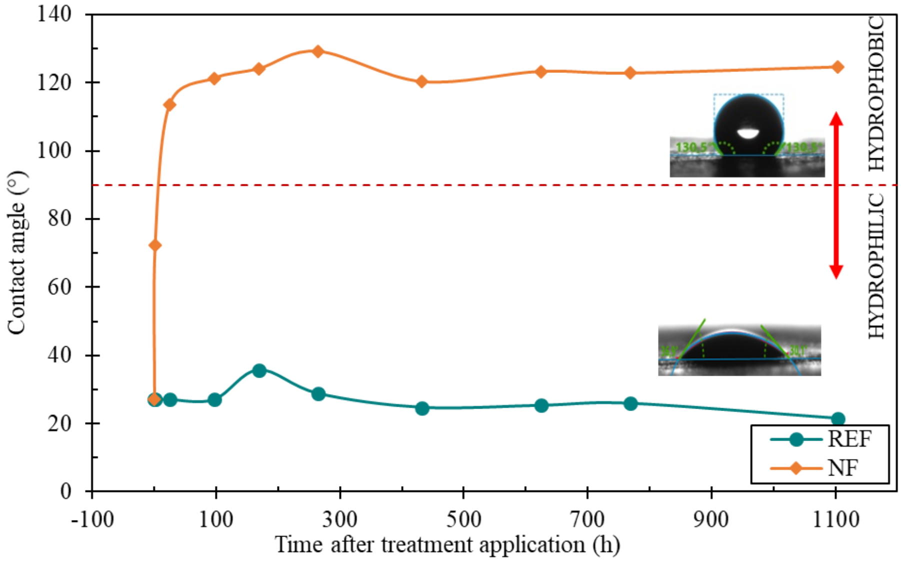

FIGURE 3
Contact angle behavior in samples with NF treatments hours after application.

In the Figure 4, the follow-up of the CA is presented, in the treated specimens and with the preselected initial ages, at different re-exposure ages to CO_2. As expected, the NS specimens had a similar behavior than the reference, since this treatment of silicon-based nanoparticles did not provide a hydrophobic effect.

The AC in the NF-treated specimens is practically constant, indicating that hydrophobicity is maintained during the exposure time, regardless of the degree of initial aging. In Figure 4.A, the NF presents the highest AC compared to the other series, being in ranges between 120-130°. This preservation of the angle can be attributed to the fact that the surface modification of the concrete due to the carbonation process did not affect the behavior of the CH. radicals, mainly responsible for the hydrophobicity of the surface.

In Figure 4.B and 4.C, the results obtained from specimens with an aging of 5 mm and 10 mm carbonation are presented. In both cases, it can be verified that the AC remained in a range between 115 and 120°, but without showing a clear trend. Based on the above, it can be concluded that aging caused by exposure to a CO. environment does not affect contact angle behavior, and therefore the development of hydrophobicity.

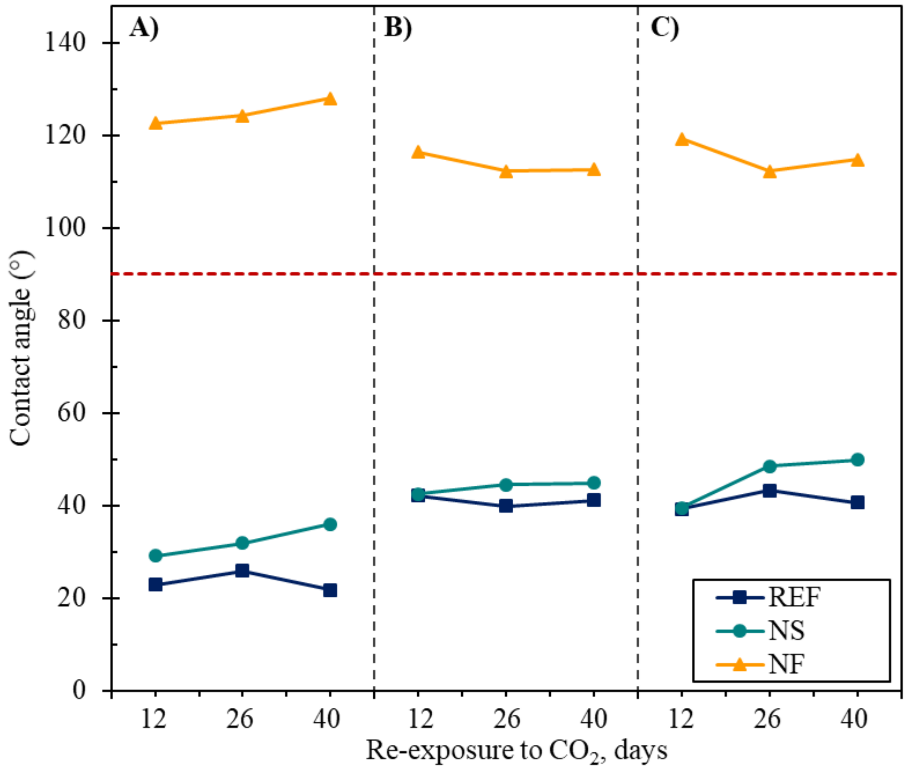

FIGURE 4
Variation of the contact angle of specimens with an initial aging of A) 0mm, B) 5mm and C) 10 mm of carbonation, after re-exposure to carbonation

4.2 Carbonation depth

In Figure 5, the results of the carbonation progress of the NS and NF surface treatments are presented, including the series without treatment (REF). It can be observed how the untreated series tends to have a greater carbonation depth compared to the treated samples, regardless of the degree of aging.

Specifically, in Figure 5.A, towards the end of the test period, a 36% decrease in the carbonation depth is observed in the NS-treated specimens, compared to the REF samples. While the treatment with NF obtained a 22% reduction. This is attributed to the formation of hydrated compounds as a result of the reaction of the nanoparticles with Ca(OH)2, obtaining a greater amount of gels that leads to reduced permeability. These results are in agreement with the literature, where the use of NS generates a barrier effect that improves the resistance of aggressive agents[4] [6]. The treatment with NF produces a hydrophobic effect on the surface of the concrete, which gives it the ability to yield the passage of gases such as CO2 insider, but stopping the entry of water through the repulsion of molecules of OH, for example, which are necessary to generate the reactions of the carbonation process.

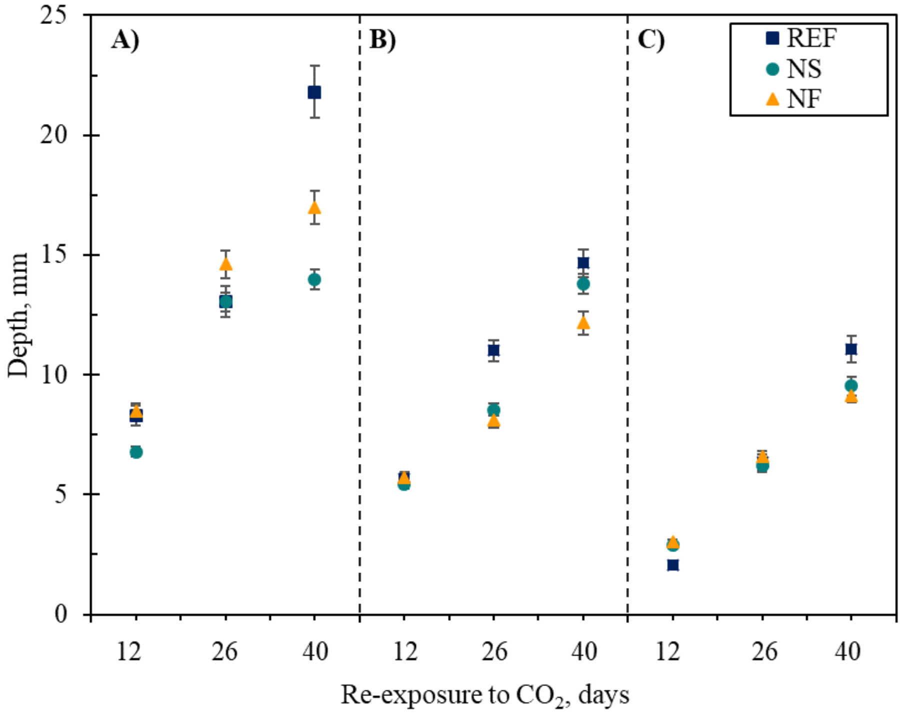

FIGURE 5
Depth of carbonation in specimens with A) 0 mm B) 5 mm and C) 10 mm carbonation prior to application of the treatment.

In Table 1, the values of the reduction of the carbonation advance of the specimens with the different treatments, obtained at the end of the re-exposure period, are presented. The reduction percentage (% Red) was obtained using the equation (2) established by Fajardo et al. (Fajardo et al., 2015).

$$\%Red = (1 - (X_{TRAT}/X_{REF})) * 100 \tag{2}$$

Where:

%Red= reduction in carbonation depth (vs. REF)
X_{TRAT}= carbonation depth of the treated samples (mm)
X_{REF}= carbonation depth of the untreated samples (mm)

TABLE 3
Percentage of reduction of carbonation vs REF of specimens treated with NS and NF; exposed to an aggressive environment with 8% CO2 at 40 days.

Initial carbonation	%RED	
	NS	NF
0 mm	36	22
5 mm	4	13
10 mm	7	7

According to the results obtained in Table 3, all the treatments used presented a greater reduction percentage in the concrete with an initial aging of 0 mm, compared to the 5 and 10 mm aging series. It can be clearly observed that the degree of aging had an effect on the performance of the treatments. These results are similar to those found in the literature, with the difference that the application of surface treatments was carried out at early ages, even during the first days after manufacturing. For example, Franzoni (Franzoni, Pigino and Pistolesi, 2013) found that the use of nano silica has a carbonation penetration reduction effect of around 14-47%. For his part, Ibrahim (Ibrahim et al., 1999) found that surface treatments based on silanes/siloxanes showed a reduction of around 20% in carbonation penetration.

The results obtained here allow to conclude that the degree of aging of the concrete reduces the performance of the treatments in the face of re-exposed to CO_2. In this way, the importance of performing an evaluation and diagnosis of the structure to be able to make a treatment selection is noticed. For structures subjected to industrial or industrial urban environments, treatment with NS and NF nanoparticles could be a feasible option when applied at early ages.

5. CONCLUSIONS

- NF treatment achieved greater hydrophobicity and stability after 96 hours of application.
- In no aging specimens (i.e., 0-mm of carbonation), the loss of AC can be attributed to a possible modification that generates carbonation between the CH3 radicals, managing to lose hydrophobicity. The effect of the initial aging of 5 and 10 mm did not affect the hydrophobicity on the surface by using NF treatment.
- As for CO_2 accelerated aging, it is observed that NS treatment did not show expected results, since the NS have the $Ca(OH)_2$ limiting that is consumed in the carbonation process.

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Additional information

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