Carbonation study in a cement matrix with carbon nanotubes

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Abstract. Carbon nanotubes as additions in Portland cement based matrices have a great potential to improve the mechanical properties of mortars and concretes; however, little work has been done regarding the effect that they have on the chemical durability of the cement matrix and reinforcing steel. In this work, carbon nanotubes are dispersed in water solution with Triton X 100 surfactant and blended in Portland cement mortars. A sharp decrease in compression resistance was observed at all curing ages (7, 14, 21 and 28 days) while a depth increase in the carbonation profile was found, which is associated with a decrease in the matrix durability.

1. Introduction

It is now a consensus in the literature that carbon nanotubes (CNT) are able to improve the mechanical properties of concretes and mortars [1-4]. Additions of CNT up to 0.2% by mass of cement have been found to improve the flexural strength of pastes, mortars and concretes, while additions greater than 0.2% are prone to agglomerations, hindering their reinforcing effect [5]. Adequate dispersion results of CNT in water have been obtained using an ultrasonic tip at 40 kHz frequency and 100 W potential for one hour [6]. Regarding the use of surfactants as chemical dispersing aids, it has been found that Triton X 100 is more efficient than sodium dodecyl sulfate (SDS) and cetylpyridinium chloride (CPC) [7-9]. Regardless of the potential of CNT as reinforcing agents, little work has been done to understand the effect of this type of nanoparticle on the durability of cement-based matrices. This issue becomes of high relevance when taking into account that CNT are a highly conductive material [10, 11] and may accelerate the electrochemical corrosion of steel rebars used as traditional reinforcement in concrete structures. This work presents a systematic study on the effect of carbon nanotubes in the carbonation penetration depth of Portland cement mortars.

2. Set up

Triton TX 100 surfactant of 98% purity produced by Sigma Aldrich was used as chemical dispersing aid for CNT in water. Firstly, surfactant and water were combined using a magnetic stirrer for 5 min and then CNT were added to this mixture in the proportions presented in Table 1 and then dispersed using an 500 W ultrasonic tip processor set to 40 % amplitude and 20 s on/off cycles. “NSR-10” standard was used dose three types of
mortars composed of Portland cement, natural sand, water, surfactant and CNT (S1: conventional mortar, S2: conventional mortar + TX100 and S3: conventional mortar + TX100 + CNT). These mortars were molded in 5 mm side cubes, cured for 7, 14, 21 and 28 days in a humid chamber and tested for compressive strength using the NTC 220 standard. Carbonation tests were performed by cubic mortar samples after 28 days of curing to 4% CO₂ concentration (100 times the atmospheric concentration) with 7, 14 and 28 days as exposure periods with a Relative humidity of 60% and 23 °C. Three samples of each mortar were exposed to CO₂. Phenolphthalein was used as test to carbonation profile. Details about the carbonation chamber could be found elsewhere [12]. Each carbonation test was made by triplicate. After carbonation test, samples were cut in half, so, for each mortar mixture there was 6 carbonation surfaces. Additional details about carbonation test could be found elsewhere [13, 14].

Table 1. Dates to dispersion (g: grams and J: Joule)

| Material       | Date (g)  |
|----------------|-----------|
| Water (g)      | 400.4337  |
| Triton TX-100 (g) | 3.2067    |
| CNT (g)        | 1.3258    |
| Energy (kJ)    | 156.89    |

3. Results and Discussion

Compressive strength results of the S1, S2 and S3 mortars after 7, 14, 21 and 28 days of curing are presented in Figure 1.

![Figure 1](image)

**Figure 1.** Compressive strength $F'_c$ (MPa) of mortars after (a) 7 days, (b) 14 days, (c) 21 days and (d) 28 days of curing.

It was found that the S1 mortar (conventional) showed higher strength than S2 (conventional mortar + TX100) and S3 (conventional mortar + TX100 + CNT) mortars, even though the mortars complied with the NTC 111 flow table criteria (115-125%). It was observed an atypical value in the S2 mortar at 14 days, which was attributed to material density, which was higher than the other mortars. There was also observed a clear strength difference between the S2 and S3 mortars with respect to the S1, this was attributed to a lower density of the S2 and S3 mortars (see figure 2) induced by presence of TX100 surfactant in the mixture. Literature reports have shown that the presence of
surfactant (in this case TX100) increases the porosity of cement matrices by stabilizing air bubbles and decreasing the surface tension of the mixing water [15, 16].

Figure 2. Density of S1, S2 and S3 mortars

The carbonation profile results from the studied mortars after 7, 14 and 28 days of exposure to CO₂ are presented in Figure 3. Of the 6 surfaces obtained after the carbonation test, only 3 are presented in Figure 3. It was possible to appreciate that the change in density of the mortars has a strong influence on the penetration depth of the carbonation profile. The carbonation profile of the S1 mortar, which is considered the control sample, presented an homogenous carbonation profile which increased in depth with the exposure time due to the penetration of CO₂ though the interconnected pore network.

Figure 3. Carbonation profile of S1, S2 and S3 mortars after a) 7 days, b) 14 days and c) 28 days of curing

For the S2 mortar, at all ages it was found that the carbonation depth was lower than that found on mortar S1. The S2 mortar has a lower density with respect to the S1 mortar, this is expected to increase the carbonation depth due to a higher porosity; however, the
results showed the opposite. This behavior can be associated with the presence surfactant in the mixing water, which stabilizes non-connected air bubbles in the matrix, decreasing its apparent density but decreasing its permeability to as, which makes it more difficult for CO₂ to penetrate into the mortar. In the S3 mortar it was evident that the carbonation depth is much higher than the ones found in the S1 and S2 mortars. This can be explained by the presence of CNT, which adsorb a portion of surfactant onto their surface, this portion of surfactant will not be available to stabilizer air bubbles and might help to generate a more connected pore network [7] consistent with a lineal relation between porosity and carbonation depth [16].

4. CONCLUSIONS

CNT increase the compressive strength by 38 % of mortars cured 28 days with respect to mortar blended only with surfactant, i.e, of similar density and porosity. The presence of surfactant helped positively to reduce the carbonation depth, while the presence of CNT increased considerably this depth probably due to a more connected pore network.

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