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# PERFORMANCE OF SILICA FUME MORTAR UNDER MARINE ENVIRONMENT.

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## ABSTRACT

The present work intended to gather data about the performance of silica fume as a chemical admixture to mortars under the marine atmosphere in the tidal zone, and also evaluate if the environment has any major role in the development of the mortar's properties. To achieve the objectives of the study two groups of mortar were made, one used silica in its composition, and the other didn't. The mortar was cast in cylindrical and prismatic molds to, later, be subjected to natural drying and wetting cycles in the sea coast of Maceió. The cylindrical specimens were 10 x 5 cm and the prismatic ones were 50 x 15 x 4 cm and had reinforcement inside. The tests were conducted in the ages of 14, 28, 91, 136 and 180 days and concerned measuring the compressive, tensile and elastic modulus of the samples and also determine the corrosion potential of the reinforced mortar using the ASTM C 876-91 method. As an addition, X-Ray diffraction tests were conducted to verify if the compounds within the mortars had change to help explain the results from the earlier experiments.

**KEYWORDS:** Silica fume, durability, mortar, marine environment.

## INTRODUCTION

The durability of reinforced concrete structures (RCS) has been the aim of several studies all over the world. The interactions that both men and nature have over these structures are of great concern since they're the ones that slowly, but continually, degradates its functionality and stability. If correctly understood, these interactive factors, along with the knowledge of concrete chemistry, can help enhance the performance of the structure and maximize the exploitation of its materials.

The engineer's role – in a construction or reparation of a structure – involves knowing and selecting the appropriate materials that will help satisfy the needs of safety, usability and durability in such scenarios. These tasks are even more difficult because of their case dependent nature: in a cold environment, where freeze and thaw are common, and air entraining additive can be prescribed; sewer conducts may require special blended sulfate resistant cements; in elements with very dense and entangled reinforcing steel, a more fluid mixture may be designed. To attend some special needs, like the ones above, the silica fume has been incorporated to the concrete.

The silica is a known by-product of the metal silicon and ferrosilicon alloys. It's a very thin particle powder (around a hundred times smaller than an ordinary cement particle) composed basically by amorphous silicon dioxide ($SiO_2$). The fact it's amorphous allows it to react with a certain compound dispersed in the hydrated cement paste – the calcium hydroxide ($Ca(OH)_2$) – to form hydrated calcium silicate (C-S-H), which is a structural phase of the hardened cement [1].

The use of silica fume is known to result in improved early strength resistance and impermeability, both because its chemical reactions and physical properties [2], commonly referred as pozzolanic and filler effect. The promise of these enhancements are the primary reasons why silica fume has been so widely accepted as a supplementary cementitious addition in the construction of very large/tall buildings and repair of damaged structures.

Being a relative new material, the effects of the silica fume in the concrete and mortars, and the influence of the environment on the above are still to be completely understood. It's not unusual to find studies showing contradictory information about the consequences of using silica fume, putting in question the filler effect [3], the permeability reduction [4] and even the quality of the C-S-H formed by its pozzolanic reaction [5].

Another point of inconsistency regards the performance of silica fume concrete and mortars when exposed to sulfate rich areas, in particular when the cation associated with the sulfur is the magnesium. Some say the use of silica makes the cementitious system weaker to such substance [6] while others claim that the addition or substitution of part of the cement by silica does not interfere in the performance of mortars and concretes [7].

The present work intends to enlighten some aspects the performance of silica fume mortars when exposed to a sulfate rich atmosphere, like the marine one. To achieve so, several mortar cylinders were molded with and without the substitution of a part of the cement by silica fume. These cylinders were positioned in the tidal zone, under drying and wetting cycles. After pre-determined periods of 14, 28, 91, 135 and 180 days, the mortar cylinders were tested for compressive strength, tensile strength (Brazilian Test), elastic modulus and X-ray diffraction. Parallel to this, some steel armed mortar plates were cast and subjected to the same conditions of the cylinders. The reinforcement was important to help evaluate the corrosion potential of the plates (according to the procedure showed at ASTM C 876-91).

## EXPERIMENTAL PROCEDURE

Since the objective is evaluate the influence of the silica fume on mortars, two mixtures were proposed – one containing the said chemical admixture and another without it, as shown in table 01. Both of them were exposed to the exact same environment simultaneously, which happened to be the breakwater that protects the docks of a chemical company (Fig. 01).

**FIGURE 01 - MARINE BREAKWATER.**

Although the chemicals manipulated in such industry could compromise the results, the docks where the experiments took place are pretty far from the company facilities, allowing one to neglect the effects the factory's chemical compounds could have over the mixtures. Also, the seawater quality is monitored by a third-party company, which stated that the water conditions are on par with other samples taken several kilometers away from the docks.

**TABLE 01 – MORTAR MIXTURES.**

| Mix # | Mortar Type | Cement | Sand | Silica Percentage | Water | Superplasticizer |
|---|---|---|---|---|---|---|
| 1 | With Silica | 9.4 kg | 25 kg | 6% [600g] | 0.45 [4.5 liters] | 0.5% (ml/g) [50ml] |
| 2 | W/O Silica | 10 kg | 25 kg | 0% | 0.45 [4.5 liters] | 0.1% (ml/g) [10ml] |

By previous experiences and with the help of the literature, it was decided that the amount of silica used should not exceed the average interval of 3-10% [1], and so the value was fixed in 6%. This was not a random choice – many of the structures repaired in the city (Maceió), where the experiments took place, were reported to use percentages not higher than 5.5% [18]. Also, the literature demonstrated that the use of silica beyond 5% shows greater compressive strength gains [1, 8, 9, 10]. The 6% value reflected the search of a medium-to-high strength mortar allied with some economy.

All the mixtures required the use of superplasticizers. The silica fume ones achieved good workability using the minimum dosage (0.5% in ml/g) recommended by its manufacturer, while the cement only mortars used 1/5 (one-fifth, or 0.1%) of the lowest quantity proposed by the producer to reach the same plastic aspect observed in the silica fume mortar.

Both type of mortars were cast in 10 cm x 5 cm cylindrical molds, following the guidance of NBR 5738:1994 [11] and stood there for 24 hours, when they were demolded and accommodated inside a plastic box primarily used to carry soft drinks. The top of the box was covered and sewed with a plastic net (Fig. 02) and it was taken to the selected breakwater to start the exposition period.

**FIGURE 02 - MORTAR CYLINDERS.**

**FIGURE 03 - MORTAR PLATES.**

The mortar was also cast inside 50 cm x 15 cm x 4 cm rectangular molds to shape the armed plates. The steel was a mesh with 5 cm x 5 cm openings and one salient bar covered with rubber tape to, latter, permit the execution of the corrosion potential tests. Five specimens at a time were placed inside another plastic box and a hose was used to help separate the plates, avoiding contact damages (Fig. 03).

All the specimens were placed and tied together in the tide variation area of the breakwater where they constantly experienced wetting and drying cycles. This procedure lasted for 14, 28, 91, 135 and 180 days. In the end of each time period, a group of 18 mortar cylinders (9 based in mix #1 and 9 made with mix #2) and 6 mortar plates (3 from mix #1 and 3 made with mix #2) were taken to perform compressive, tensile and elastic modulus tests, as well as measure the corrosion potential of the plates and observe possible changes in the hydrated cement phases with the aid of X-ray diffraction.

## RESULTS AND DISCUSSIONS

After the cylinders and plates were recovered the laboratory tests took place. A minimum of three samples were used for each experiment and the results are shown in the following sections.

### *Compressive strength*

The compressive tests followed the recommendations of the NBR 5739:1994 [12]. The cylinders were capped with a sulfur/cement mixture to ensure a plain and hard contact surface and the tests were performed using the EMIC DL 3000 machine.

The table 02 shows the strength development of both types of mortars. As one can see, the silica fume mortars quickly achieved high strengths (above 53 MPa) in the first 28 days, while the mortar from mix #2 reached around 47 MPa in the same time span. The strength gain between 14 and 28 days is also higher for the mix #1 (almost 7 MPa) when compared to the mix without silica fume (which showed less than 1 MPa gain).

Until the ninety first day, all mortars exhibit increase in this property. However, this trend chances when they reach the day 135. The silica fume mortar losses more than 10 MPa and this loss continues to take place until the last day of measurements, when this mortar shows its lowest value of compressive strength, 39.83 MPa.

**TABLE 02 – COMPRESSIVE STRENGTH OF MORTARS.**
**The values in bold represent the average of three specimens.**

| Mortar Type | Compressive Strength (MPa) | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | 14 Days | | | 28 Days | | | 91 Days | | | 135 Days | | | 180 Days | | |
| | CP1 | CP2 | CP3 | CP1 | CP2 | CP3 | CP1 | CP2 | CP3 | CP1 | CP2 | CP3 | CP1 | CP2 | CP3 |
| With Silica | 47.1 | 47.3 | 46.4 | 54.3 | 54.3 | 52.7 | 55.3 | 52.5 | 54.9 | 45.2 | 39.2 | 46.7 | 42.0 | 38.3 | 39.2 |
| | | **46.93** | | | **53.77** | | | **54.23** | | | **43.70** | | | **39.83** | |
| W/O Silica | 46.7 | 46.5 | 45.8 | 48.4 | 45.2 | 47.4 | 47.4 | 48.6 | 47.9 | 48.6 | 45.4 | 47.8 | 52.6 | 49.8 | 52.5 |
| | | **46.33** | | | **47.00** | | | **47.97** | | | **47.27** | | | **51.63** | |

The mortar without silica fume also appears to suffer strength loss in the day 135, although it is almost ignorable by realistic means. Furthermore, in the 180$^{th}$ day, this mortar even shows signs of compressive strength gain, achieving a peak of 51.63 MPa, its highest value observed within the period of experiments.

### *Tensile strength*

For the tensile strength tests, the guidelines present in the NBR 7222:1994 [13] were obeyed. This time the cylinders didn't need capping but just two wooden sticks to help centralize the specimens in the same machine used for the compressive strength tests.

**TABLE 03 – TENSILE STRENGTH OF MORTARS.**
**The values in bold represent the average of three specimens.**

| Mortar Type | Tensile Strength (MPa) | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | 14 Days | | | 28 Days | | | 91 Days | | | 135 Days | | | 180 Days | | |
| | CP1 | CP2 | CP3 | CP1 | CP2 | CP3 | CP1 | CP2 | CP3 | CP1 | CP2 | CP3 | CP1 | CP2 | CP3 |
| With Silica | 5.3 | 5.4 | 4.9 | 4.7 | 5.2 | 5.0 | 5.5 | 5.1 | 5.1 | 5.2 | 4.9 | 5.5 | 4.9 | 4.8 | 5.4 |
| | | **5.20** | | | **4.97** | | | **5.23** | | | **5.20** | | | **5.03** | |
| W/O Silica | 4.3 | 4.1 | 4.8 | 4.9 | 4.7 | 5.4 | 4.3 | 4.4 | 4.6 | 3.6 | 3.2 | 3.4 | 4.8 | 5.2 | 5.1 |
| | | **4.40** | | | **5.00** | | | **4.43** | | | **3.40** | | | **5.04** | |

The values obtained for the tensile strength are presented in table 03. The results obtained show some variation, but now, contrary to what was observed in the compressive strength tests, they're more substantial in the mortars without silica fume. Nonetheless, in the last days, the mortars appear to converge to similar tensile strength values, no matter if the mixture has silica or not.

In the silica fume mortar is possible to see that the tensile strength fluctuates between 5 MPa throughout all the ages of testing, and ends showing a tensile strength of 5.03 MPa. Standard deviation analysis demonstrates that the small variations observed are statistically insignificant.

In the cement-only mortar the fluctuation seems to reach higher amplitudes. There are tensile strength values ranging from as low as 3.4 MPa to a high of 5.04 MPa. The 3.4 MPa value may be an anomaly always possible to happen when dealing with experimental procedures and real exposure environments. If not, this low shows that the tensile strength of the cement only mortar is more sensitive to environment.

Comparing the tensile and the compressive tests, keeping in mind the definition that the first is supposed to be about 10% of the second, it can be assumed that whatever is pushing down the compressive strength of the silica fume mortar does not affect its tensile strength the same way. Looking to the mortar without silica, the same applies, but in an inverse manner, since the tensile strength appears to be more affected than the compressive one.

***Elastic modulus***

The elastic modulus test was carried out following the text present in the NBR 8522:2003 [14]. It is worth mentioning that the said norm is designed for concretes, and, since a pertinent test doesn't exist for mortars it was decided to consider the experiment applicable, as the research is a comparative one between mortars with and without silica fume.

The table 04 displays the elastic modulus obtained in the laboratory. The values above 91 days for the silica mortar and 135 days for the ordinary mortar could not be acquired due to technical problems and that's why they don't appear at Table 04. To get around this problem it was decided to get the other values using mathematical methods proposed by the NBR – 6118:2003 [15] through the following equation.

$$Eci = 5600.\sqrt{fck} \quad (2)$$

**TABLE 04 – ELASTIC MODULUS OF MORTARS.**
**The values in bold represent the average of three specimens.**

| Mortar Type | Elastic Modulus (GPa) | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| | 14 Days | | | 28 Days | | | 91 Days | | |
| | CP1 | CP2 | CP3 | CP1 | CP2 | CP3 | CP1 | CP2 | CP3 |
| With Silica | 35.3 | 36.3 | 36.8 | 40.4 | 35.9 | 36.3 | - | - | - |
| | | **36.14** | | | **37.54** | | | - | |
| W/O Silica | 35.8 | 34.5 | 34.9 | 36.4 | 32.1 | 37.1 | 36.6 | 36.4 | 40.0 |
| | | **34.94** | | | **35.17** | | | **37.64** | |

Where Eci is the elastic modulus and fck is the compressive strength of the sample. Before using this formula, it is wise to consider possible deviations it may input in its results when judged against the findings in the laboratory tests. By comparing the values, it was observed that the calculated elastic modulus always presented results greater than the ones obtained with the experimental procedure. Sometimes this difference was small, such as 3%, but could also be quite significant as 9% higher.

To bring the mathematical model a little more close to the laboratory numbers, a compensation of 7.2% down (a median of the divergences observed) was applied to the calculated values, resulting in the Table 05. This table shows smaller disparities between the compensated values and the ones founded with the experiments.

Analyzing the results shown by Table 05, it can be seen that the mortar with silica fume yet again suffers more loss than the mortar without silica. In a real structure, it could be possible to assume that silica fume mortar wouldn't deform (as result of equal stress) as much as the mortar without this pozzolanic addition.

**TABLE 05 – COMPARISON OF THE EXPERIMENTAL, CALCULATED AND COMPESATED ELASTIC MODULUS.**

| **Mortar Type** | **Age (Days)** | **Experimental Elastic Modulus (EEM - GPa)** | **Compression Strength (MPa)** | **Calculated Elastic Modulus (CEM - GPa)** | **Compensated Elastic Modulus (COEM - GPa)** | **Difference** $\frac{COEM}{EMM}-1$ |
|---|---|---|---|---|---|---|
| **With Silica** | 14 | 36.14 | 46.93 | 38.36 | **35.60** | -1.5% |
| | 28 | 37.57 | 53.77 | 41.06 | **38.10** | +1.4% |
| | 91 | - | 54.23 | 41.24 | **38.27** | - |
| | 135 | - | 43.70 | - | **34.35** | - |
| | 180 | - | 39.83 | - | **32.80** | - |
| **W/O Silica** | 14 | 34.94 | 46.33 | 38.12 | **35.38** | +1.2% |
| | 28 | 35.17 | 47.00 | 38.39 | **35.63** | +1.3% |
| | 91 | 37.64 | 47.97 | 38.79 | **36.00** | -4.4% |
| | 135 | - | 47.27 | - | **35.73** | - |
| | 180 | - | 51.63 | - | **37.34** | - |

## *Corrosion potential*

The corrosion potential tests were executed following the orientation of the ASTM C 876-91 norm. The mortar plates were taken to the laboratory and the tests were performed using a digital multimeter model MD 6200 from ICEL and copper-copper sulfate electrodes. There were four points of measurement (named P1, P2, P3 and P4) per specimen, shown at Fig. 04, and the results are presented at Table 06.

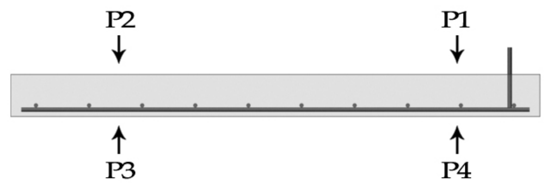


**FIGURE 05 - POINTS OF MEASUREMENTS FOR THE CORROSION POTENTIAL TESTS.**

Although the ASTM C 876-91 says that beyond – 350 mv there's more than 90% probability that the reinforced steel, no mortar specimen showed signs of corrosion after they were opened. All the steel inside them was in prefect conditions, with very small, if any, orangish stains. Maybe these are proof that corrosion was starting to develop, particularly in the case of silica-free mortars, but the mortars with silica also exhibited little stains on their steel armor. Nevertheless, more research should be done to better understand the influence of silica on the corrosion phenomenon.

**TABLE 08 – CORROSION POTENTIALS OF MORTARS.**

| Mortar Type | Potential ( – mV) | | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | 28 Days | | | | 91 Days | | | | 135 Days | | | | 180 Days | | | |
| | P1 | P2 | P3 | P4 | P1 | P2 | P3 | P4 | P1 | P2 | P3 | P4 | P1 | P2 | P3 | P4 |
| With Silica | 252.3 | 248.0 | 261.6 | 270.3 | 281.4 | 296.7 | 237.1 | 242.6 | 345.0 | 358.5 | 303.3 | 256.7 | 248.4 | 245.6 | 268.8 | 279.0 |
| W/O Silica | 248.5 | 258.5 | 285.5 | 289.0 | 301.8 | 305.4 | 218.5 | 234.1 | 345.5 | 339.6 | 239.3 | 324.0 | 467.7 | 465.5 | 369.3 | 313.3 |

### *X-Ray Diffraction*

Some of the X-ray diffraction results are shown in Fig. 06 and 07. The samples for this experiment were taken from the tensile strength specimens, and two kinds of sampling were made – one was composed from the mortar in the outer surface of the ruptured cylinders (around 1 cm deep) and the other was obtained from the inner part of the same cylinders (about 1 to 2.5 cm deep). The complete graphics can be found at ALMEIDA [19].

The figures show some variations within the compounds of the hydrated cement paste. At first, it's worth noting that the $Ca(OH)_2$ levels are significant lower in the silica fume mortars at the $28^{th}$ day, when compared to the mortars made from mix #2 at the same age. This can be explained as the result of the pozzolanic reaction of the silica fume – which consumes the calcium hydroxide present in the ionic pore solution of the cement paste.

Other phases that exhibited variations were the ettringite and silicon sulfide ones. If it's made a comparison between the mortar types, these compounds showed fairly higher levels in the specimens taken from the surface of the cement only mortar than the ones obtained from the silica fume.

In the 180[th] day, the test reveals some changes in the X-ray peaks. Regarding the mix #2 mortar, it can be noted that, while the $Ca(OH)_2$ higher values may have come down a little, they're still greater than the ones seen in the 180 days old silica fume mortar. This difference is even more pronounced if the specimens from inside the mortars are contemplated. Alternatively, the ettringite and silicon sulfide values remain almost the same for both mortar types.

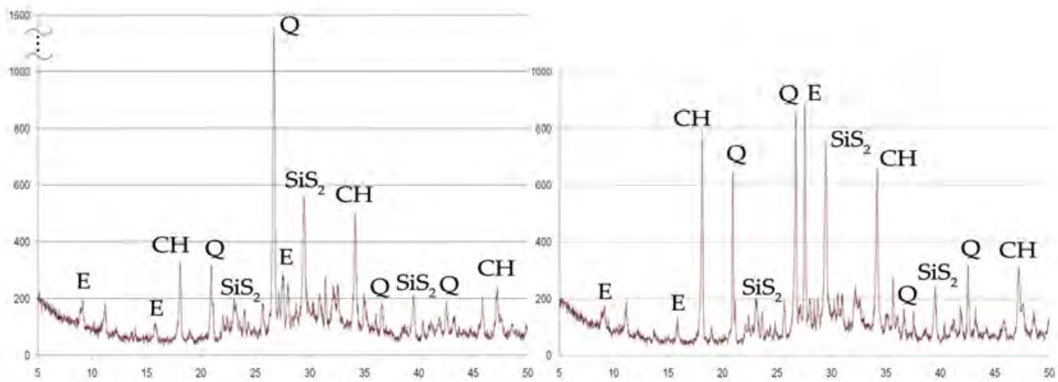


**FIGURE 06 - X-RAY DIFFRACTION OF 28 DAYS OLD SURFACE SAMPLES FROM SILICA FUME (RIGHT) AND CEMENT ONLY (LEFT) MORTARS.**
Q = Quartz; CH = Calcium Hydroxide; E = Ettringite; $SiS_2$ = Silicon Sulfide.

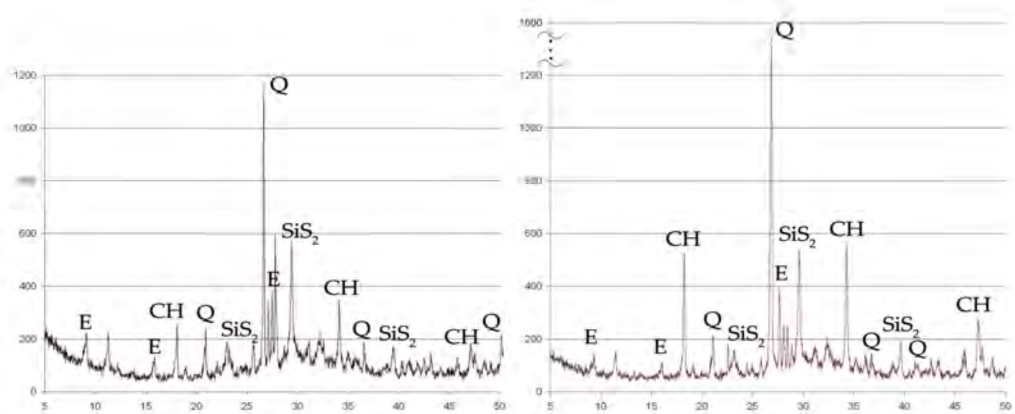


**FIGURE 07 - X-RAY DIFFRACTION OF 180 DAYS OLD SURFACE SAMPLES FROM SILICA FUME (RIGHT) AND CEMENT ONLY (LEFT) MORTARS.**
Q = Quartz; CH = Calcium Hydroxide; E = Ettringite; $SiS_2$ = Silicon Sulfide.

## CONCLUSION

By the present work, it can be said that the use of silica in marine environments is, at least, controversial. The compressive strength showed signs of lowering in the latter ages, reaching less than 75% of the strength it showed at 28 days – and that's the date used (in Brazil) to specify the concrete or mortar strength to a construction. The mortar without silica, on the other hand, continued to develop its compressive strength, even after a small bump in the 135[th] day.

Little can be say about the tensile strength. This property showed some variations through the experiment, but still ended up achieving substantial (and almost the same) strength in both silica and non-silica mortars. This time the oddness came from the ordinary mortar, with a result dropping to 3.4 MPa. There are no apparent reasons for this; maybe it's a turning point in its strength cycle to maturity in such environment, or the samples had been damaged in the ambient of exposure. To be sure, the entire experiment would have to be repeated.

The elastic modulus followed the trend of the compressive strength results. This was expected since the lattes values of the modulus were determined using the compressive numbers. According to this, the silica fume mortar showed greater decrease in the elastic modulus than the cement-only mortar.

Measuring the corrosion potentials gave signs that the steel rebars might have been under corrosion attack, specially the ordinary mortar ones. Investigating the rebar with the help of magnifying lens revealed that there were almost no signs of corrosion – only small and sparse little orange dots could be identified on both mortars. The corrosion potential values pointed that there was more than 90% of chance that corrosion was occurring on ordinary mortars at the age of 180 days, while at that same time, the conditions of the silica fume mortars were uncertain, accordingly to the ASTM standard. Also, this experiment should have used more points of measurement to ensure more reliability to the test. Unfortunately, the nature of the exposing environment combined with logistical issues prohibited this from happening.

The X-ray diffraction helped see some phase changes that took place in the mortars. It showed that the silica fume mortar $Ca(OH)_2$ levels vary greatly, and the lowest values for this phase were observed in the samples after almost 180 days of exposure. This wasn't true for the cement only mortars – they managed to keep their calcium hydroxide above 500 A.U. (aleatory units).

It is known that the $Ca(OH)_2$ is one of the first compounds to be attacked by deleterious chemical substances, and that its presence is important to make stable the C-S-H of the hydrated cement. Its lower values in the silica fume mortars may indicate that some of it could've been leached away, exposing the C-S-H to harmful compounds, such as the magnesium sulfate. More tests – and more exposing time – are essential to shed some light in phenomenons observed.

The use of silica on mortars subjected to the marine environment to improve the mechanical properties and ensure a longer durability is still a topic of great discussions. These pondering points are important to spread the knowledge about such subject and help the development of the engineer science.

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