

Aging of recycled aggregates mortars by drying-wetting cycles

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Abstract: Due to the large amount of CO₂ emissions produced nowadays by the construction sector, the scientific community is looking for measures that will bring constructions to be as environmental-friendly as possible. One of the most popular lines of research is the use of construction waste as recycled aggregates for the production of recycled aggregates concrete. Regarding the use of coarse recycled aggregates, there are a large number of studies that validate their use at both a mechanical and durability levels. Regarding the use of fine particles, which are inherent to the existence of coarse particles, it is necessary to increase the existing knowledge before they can be widely used. In this paper, the authors have sought to isolate the weakest part of those concrete mixes with exclusively recycled aggregate, i.e. a mortar made exclusively with fine recycled aggregates, in order to analyse the effect of drying-wetting cycles on mixes with recycled aggregates only. Specifically, three types of fine aggregates were used, one natural silica aggregate, one aggregate from crushed ballast and one from crushed sleepers. The mortars made with these aggregates were subjected to 50 durability cycles in distilled water, seawater and water with sulphates. Mechanical, visual and microstructural tests were carried out to analyse the effect of each of these environments on each of the mortars.

Keywords: Recycled aggregate; mortars; fine recycled aggregates; railways; durability; fly ash.

1. Introduction

It is estimated that ~10% of man-made CO₂ emissions come from concrete production and transportation [1,2]. For this reason, Society demands actions that allow maintaining the benefits derived from the construction and industry activities, while minimising greenhouse gases emissions. One of the most studied actions due to its great potential is the use of construction and demolition waste (C&DW) as recycled aggregates (RA) [3–5]. Nowadays, it is generally assumed that the replacement of natural aggregate with recycled aggregate leads to a reduction of the properties of concrete. However, it is also proven that, especially in the case of using only coarse recycled aggregate (coarse-RA), if this replacement is done in a controlled way, it is possible that the loss of properties of concrete is still compatible with its use in structures. Many researches have proven that concrete manufactured with this RA (RC), depending on how concrete was produced and the properties of RA, may have good mechanical [6–10] and durability [7,11–13] properties. López Gayarre et al. [14] found a correlation between physical properties (density) and mechanical properties (compressive strength and modulus of elasticity) of RC. Other authors have analysed the effect of RA on the permeability and durability of RAC. Ariyachandra E. et al. [15] analysed the influence of RA on chloride ion diffusion and chloride binding capacity of concrete. Salman and Ahmad [16] analysed the effect of biomineralization technique over RAC on the durability properties of RC.

As a starting point, the possibility of valorising C&DW as coarse aggregates for the manufacture of concrete is a great achievement, although not enough from an environmental point of view. By generating coarse-RA, smaller particles are also generated that can be valorised. For this reason, several authors have conducted studies analysing the effect of incorporating fine recycled aggregates (FRA) in

concrete, obtaining satisfactory results both in terms of mechanical [8,17–19] and durability [9,20] properties in recycled concrete manufactured with FRA. Although it is well known that the properties of recycled aggregates can be highly variable depending on a large number of variables, such as the origin, the process of obtaining them, the age of the crushed concrete or the presence of contaminants [21].

Generally, the durability of concrete is affected by the possibility of external agents circulating inside it, damaging it. For this reason, the permeability of concrete is a key parameter to determine its durability, since the lower the capacity of the different aggressive agents to penetrate into concrete, the more durable it will be. Generally, RA are more permeable than natural aggregates, especially in the case of FRA [22,23]. For this reason, generally, it could be said that a recycled aggregates concrete will have less durability than conventional concrete with equivalent cement content. On many occasions, when the recycled aggregate comes from a structural concrete of medium-high strength, the durability of the recycled aggregates concrete will largely depend on the permeability of the cement paste. If concrete is designed with compacted paste (low water/cement ratio) and adequate aggregates, concrete with good behaviour in the long-term may be obtained, even using RA. For this reason, the use of FRA has great influence on concrete durability [25].

In this research, the weakest part of concrete made with 100% RA has been isolated, i.e. a mortar made with fine recycled aggregates only. Specifically, three « types of mortar have been designed; one with silica sand as a reference, a second with recycled sand from crushed concrete sleepers and, finally, a third with recycled sand from crushed ballast. These mortars have been tested for their durability, more specifically for wet-dry cycles in different fluids that can damage concrete: distilled water, seawater and water with sulphates. To maximize the effect of these cycles of wet-dry cycles, it was decided to design samples that were denominated mini mortar samples that are cylinders of 20 mm diameter and 40 mm height that allow having a high ratio between surface and volume. In order to analyse the effect of these durability cycles on these mini-cylinders, the following parameters were taken into account: evolution of compressive strength as a function of the number of durability cycles suffered; external aspect of the samples as a function of the number of durability cycles and finally, after 50 durability cycles, the microstructure of these mortars.

2. Materials and methods

2.1. Materials

Two types of specimens were manufactured; standardized mortar specimen (SMS) of 160x40x40 mm³ and cylindrical specimens (MMS) of 40 mm height and 20 mm diameter, Figure 1. The geometry of the MMS was chosen to maximize its area/volume ratio, which will increase the damage suffered.

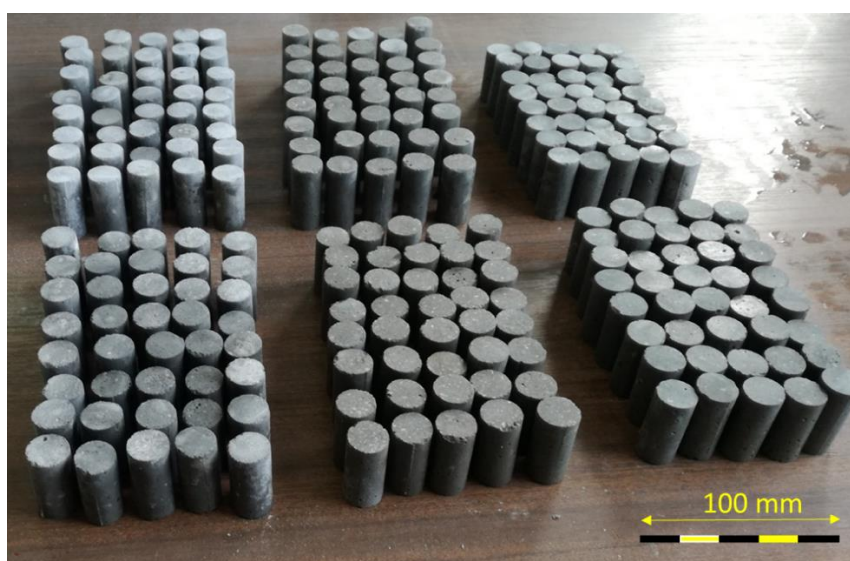


Figure 1. MMS used for the durability tests.

To manufacture both kind of samples, three mix proportions were designed: the first, a reference mortar with natural sand; the second with fine recycled aggregates from crushed railway sleepers; and, finally, the third with fine recycled aggregates from crushed railway ballast. The two recycled aggregates were obtained by crushing the out-of-service railways elements [25]. The reference mortars (NS-Si) were designed based on EN 196-1 [26] using standardized siliceous aggregate (NS-Si). In order to compare the three types of mortars, the same grading curve, volume of aggregate, cement content and effective water/cement ratio were used. In order to obtain these comparable mix proportions, the process described in [27] was used. This procedure intends to achieve mortars with the same effective w/c ratio and the same grading curves for each of the designed mixes. First, the aggregates' physical properties were determined, i.e. density and absorption (Table 1), after which it was possible to determine the mix proportion of each of the mortars (Table 2).

Table 1. Aggregates' physical properties.

Material	Density [g/cm ³]	Absorption [%wt.]
NS-Si	2.53	0.26 ± 0.025
RS-S	2.50	3.45 ± 0.025
RS-B	2.75	0.11 ± 0.025

Table 2. Mortar mix proportions.

Matrix	Cement [kg/m ³]	Sand [kg/m ³]	Effective water [kg/m ³]	Total water [kg/m ³]
NS-Si	494	1466	247	251
RS-S	494	1449	247	297
RS-B	494	1594	247	249

The cement used in the manufacture of the mortars is a CEM IV (V) 32.5 N type according to EN 197-1 [28]. It has a high replacement of clinker with fly ash. Its density according to UNE 80103 [29] is 2.85 g/cm³ and its Blaine specific surface is 3885 cm²/g according to EN 196-6 [30]. The mortars were manufactured following the procedure described in EN 197-1 [28] and were cured by immersion in water until the beginning of the tests.

2.2. Quantification of the adhered mortar

Due to the high absorption coefficient detected in RS-S, it was decided to perform a test to analyse whether this high value is due to the nature of the aggregates or the particles' size. In this work, the procedure used by Thomas et al. [31,32] to identify the natural aggregate and the bonded mortar has been followed. This procedure consists of performing a computerized axial microtomography (μCT) test on a sample of aggregate. From this test, a 3D digital model of the scanned particles is obtained, in which it is possible to distinguish, in grey scale, the materials according to their density, being the whitest the most dense material and the blackest the least dense one, as per Figure 3 (b). To carry out this test, a PVC cylinder of 50 mm in diameter and 60 mm in height was filled with compacted material (A: 5-12 mm; B: 2-5 mm; C: 0-2 mm), as per Figure 3 (a). Subsequently, a μCT test was performed on each of these PVC cylinders. Each of these tests consists of three phases: scanning, reconstruction and quantitative analysis. To ensure that all results are comparable, the same process has been used and the same parameters have been used at all stages.

In the scanning phase, a Skyscan 1172 μCT was used, similar to the one used in previous studies [31]. During this phase, the source received 80 kV of tension and 100 μA of current and a filter of Al+Cu was used. In this phase, the sample is rotated inside the μCT, making five radiographs for each degree, with a resolution of 6 μm as pixel size.

In the reconstruction phase, it is possible to determine, by means of an algorithm, what the cross sections of the sample look like from the X-rays generated during the scanning phase and build a 3D

model using them, as per Figure 4. In this phase, it was necessary to make some corrections. Ring Artifact correction was applied (11), also a Beam Hardening correction was applied (31%) and, finally, a Smoothing of 1 was also applied.

In Figure 2, an example of a cross-section of one of the scanned samples can be seen. First, and predominantly, air is represented in black. A circular crown of small thickness, quite diffused, can also be seen, which corresponds to the PVC mould in which the sample is introduced. The rest of the elements that appear in the transversal section identify either natural aggregates or mortar.

To ensure that all results are comparable, the same process has been used and the same parameters have been used at all stages. The range of greys, following the guidelines commented on in the previous paragraph, means that the particles in a dark grey correspond to the presence of mortar. Similarly, the cuts in a light grey detect the natural aggregates separated from the mortar during the crushing process. Finally, figures that combine both types of greys, see the detail on the right side of Figure 2, indicate the natural aggregate particles that contain adhering pieces of mortar. The quantitative analysis intends to determine the percentage of recycled aggregate in the mortar. Once the natural aggregate and the adhered mortar are discretized, it is possible to quantify the volume of each one of them and, later, to estimate the percentage in volume of adhered mortar present in the aggregate sample.

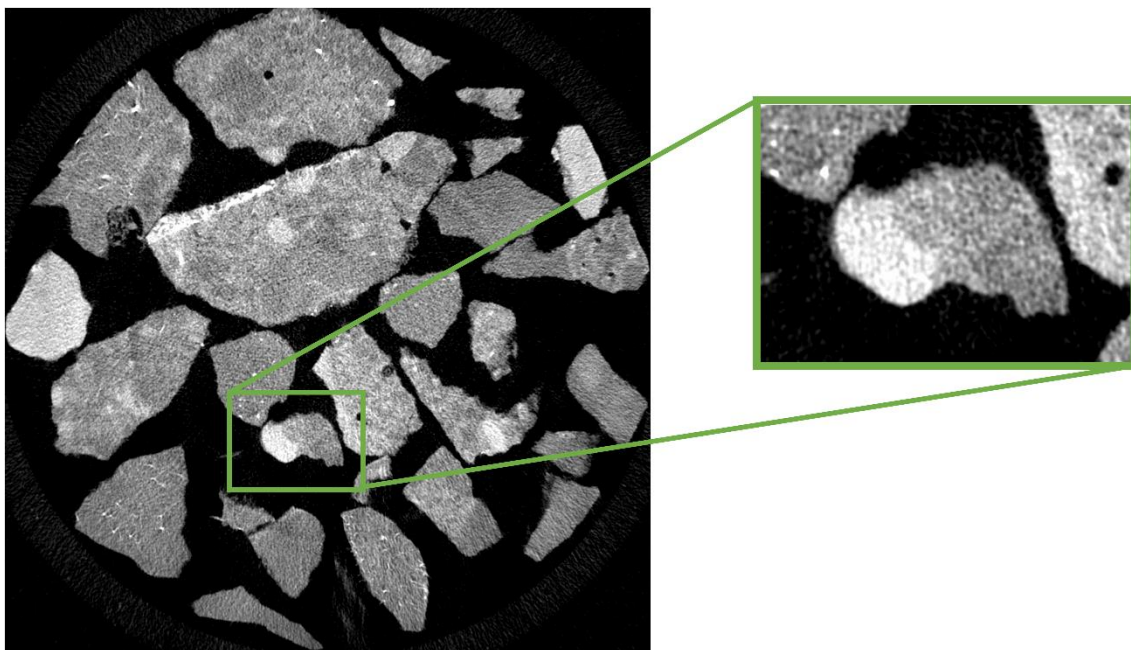


Figure 2. Identification of adhered mortar and natural aggregate from micro-CT image.

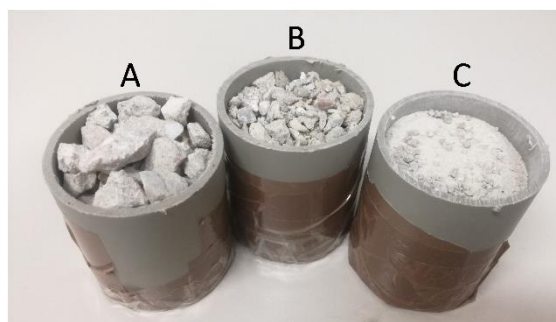


Figure 3. Samples for micro-CT tests. (A) Sample of recycled aggregate of size 5-12. (A) Sample of recycled aggregate of size 5-2. (A) Sample of recycled aggregate of size 0-2.

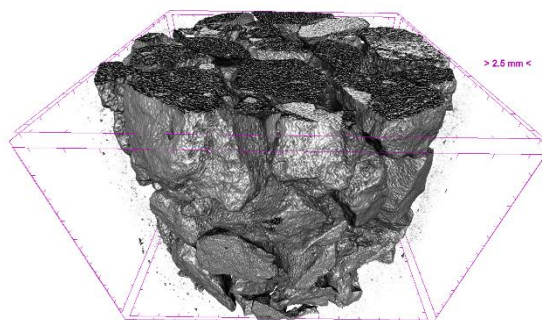


Figure 4. Samples of the reconstruction of a micro-CT test.

2.3. Mechanical properties

The SMS were tested according to UNE-EN 1015-11 [33]. First, the 40x40x160 mm³ prisms are broken by bending, obtaining two halves that are then used to determine the compressive strength.

For testing in uniaxial compression, a servo-hydraulic machine of ± 100 kN capacity was used. In these tests, the compression tool of standardized mortars was coupled, equipped with a ball joint in the top head that guarantees the axial load. The load application speed was 0.05 mm/s, ensuring that there were no accelerations, and that the test times were, in any case, less than 30 s and no more than 90 s. The procedure for testing the MMS was identical to that of the SMS after its bending test. The test speed of the mini mortars is the same as per standard UNE-EN 1015-11 [33] for testing standardized mortars, since they have the same height.

2.4. Durability

In order to perform the most aggressive durability tests, it was decided to use a type of specimen with a high surface/volume ratio, so they will be more susceptible to damage by the penetration of external agents. The behaviour of the MMS against aggressive external agents was analysed, when submitted to dry-wet cycles in different aggressive fluids, specifically distilled water, seawater and water with a 5% potassium sulphate solution.

As there is no specific regulation for wet-dry cycles to be applied on mortars, standard UNE-EN 14066 [34] was adapted. The tests began when the test specimens were 28 days old. To analyse the effect of these cycles, part of the test specimens was kept immersed in water, with no cycles, and used as control.

The procedure followed to analyse the effect of the humidity-dry cycles consisted in assigning, from each one of the mortars, ten samples as control (1-10), ten samples damaged by cycles in distilled water (11-20), ten samples damaged by cycles in seawater (21-31), and ten samples damaged by cycles in water with sulphates (31-40). 50 cycles were performed, which consisted in keeping the samples in an oven for 18 h at 70 °C and 6 h submerged in the corresponding waster solution. In Figure 5, it is possible to see the three types of mortars in the drying phase of the first cycle.

To analyse how these wet-dry cycles affect the mechanical properties in different aggressive environments, the evolution of the compressive strength was controlled according to the number of cycles. For this purpose, of the ten specimens available for each material-environment binomial, the compressive strength was determined by means of the average of 3 tests after 15 cycles, 3 tests after 30 cycles and 4 tests after 50 cycles. The compression tests were carried out according to the procedure described above.

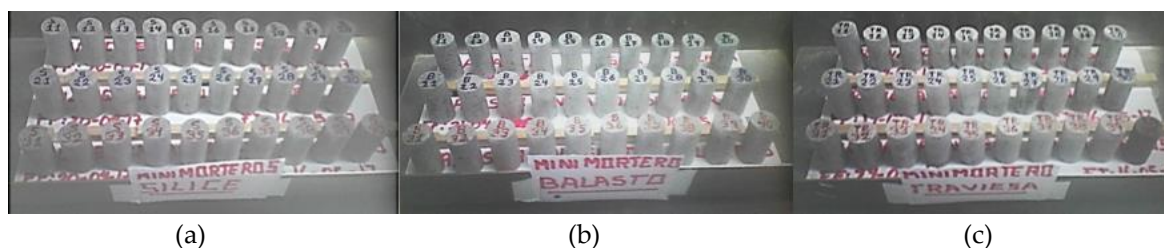


Figure 5. Drying phase of the durability tests; (a) NS-Si, (b) NS-B and (c) NS-S.

2.5. Scanning electron microscopy

Samples were selected from the compression-tested specimens and coated with gold in order to obtain a higher definition at high magnification. These samples were analysed using a scanning electron microscope (SEM) under high vacuum conditions. The analysis was carried out with both secondary and backscattered imaging in order to identify the effect of the ageing cycles on the different kinds of mortars. In addition, EDX was used for chemical analysis.

3. Results and discussion

3.1. Comparison between standardized samples and reduced size samples

From each of the three size fractions analysed, the volume of mortar and the volume of natural aggregate have been quantified, obtaining the volume percentage of RA that is adhered mortar.

For particles of size 5-12 mm, 70.2% is adhered mortar. In the case of the particles between 2 and 5 mm, the quantity of adhered mortar increases to 76.1%. Finally, in the case of particles smaller than 2 mm, the quantity of adhered mortar topped at 85.2%.

Comparing the results with those by Thomas et al. [31], the same conclusion is reached, i.e. re-crushing recycled aggregate increases the amount of mortar present in the new particle size fraction. This increase in the amount of adhered mortar is the reason why such high absorption values have been obtained.

3.2. Mechanical properties

Figure 6 shows the compressive strength of SMS and MMS specimens at the age of 28 days.

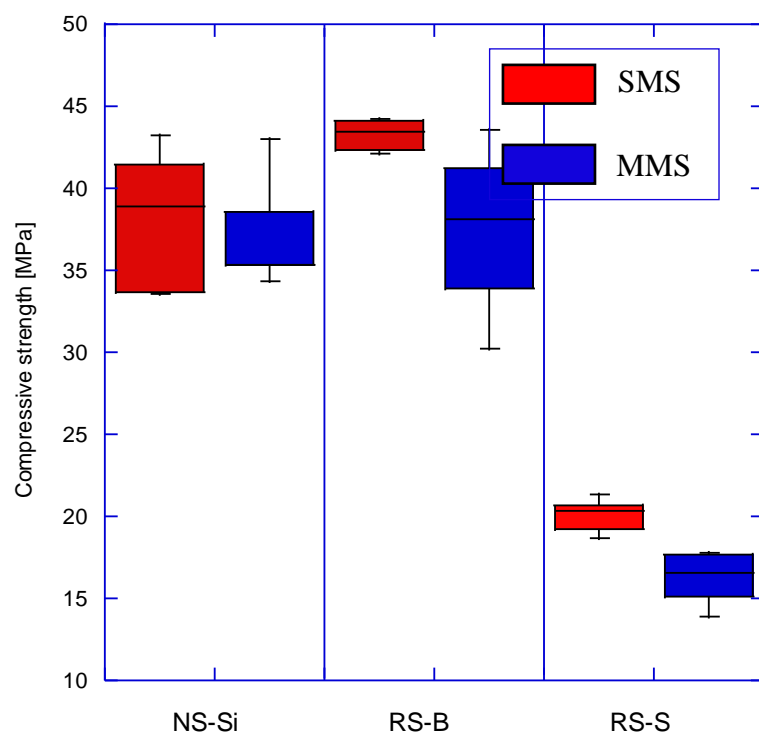


Figure 6. Compressive strength of SMS vs. MMS for the three materials.

When comparing the results at 28 days of SM and MMS, it is concluded that the values reached are similar, which validates the use of the MMS test specimens for mechanical characterisation. Comparing the mortar with natural aggregate (NS-Si) with the ones made with recycled sands, it is observed that the mortar with RS-B achieves a compressive strength similar to that of the NS-Si. It may seem surprising that mortars NS-Si and RS-B show similar results when, in general, the siliceous aggregate shows fewer

mechanical properties than the diabase aggregate. The explanation is in Figure 7, which shows a SEM image of these two types of sand, where it can be seen that they have a clearly different geometry, i.e. the silica particles (Figure 7 (a)) are much more rounded than those from crushed ballast (Figure 7 (b)), which have a flaky geometry. This flaky geometry can work as a crack initiation point, which explains why, in spite of an aggregate with better mechanical properties, a more resistant mortar is not obtained [27]. On the other hand, the mortar that incorporates RS-S has suffered an important loss of strength capacity, almost 40%, due to the worse quality of the recycled sand coming from crushed sleepers.

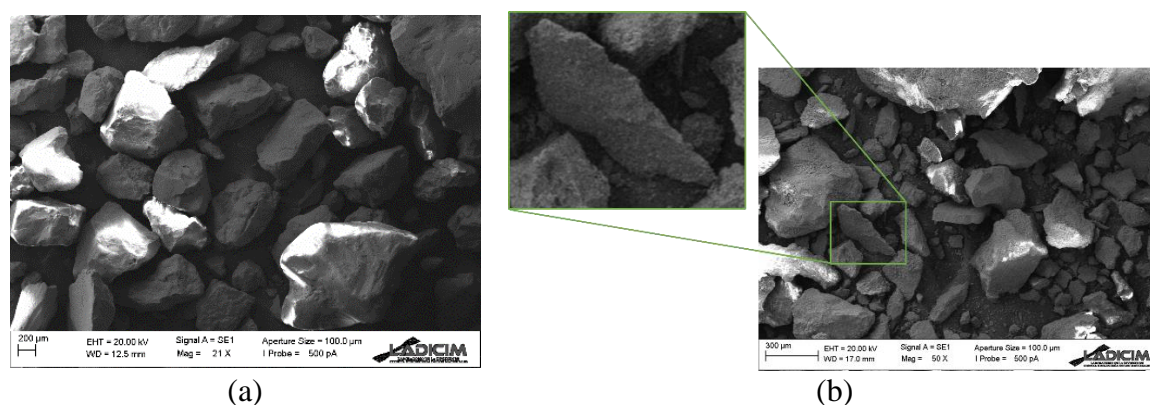


Figure 7. (a) Natural sand particles' geometry; (b) recycled ballast sand particles' geometry.

3.3. Compressive strength evolution

The results registered are shown in the four graphs in Figure 8. In the upper left part, the evolution of the compressive strength of the specimens that have not been submitted to the wet-dry cycles, tested at the same ages as those exposed to the different environments, is represented.

The remaining three graphs show the evolution of the compressive strength of the MMS studied in the four environments considered: specimens continuously immersed in water, or subjected to wet-dry cycles in distilled water, water with sulphates and seawater. The one located on the upper right corresponds to MMS-NS-Si, the lower left to MMS-RS-B and the lower right to MMS-RS-S.

The evolution of the compressive strength of the different mortars over time when they are immersed in tap water, Figure 8 (a), is similar, with a progressive increase attributable to the gradual collaboration of the fly ash contained in cement, manifesting its pozzolanic character.

After wet-dry cycles in distilled water, Figure 8 (b), the evolution of compressive strength is similar to the results obtained when the specimens are immersed in tap water, except in the case of RS-S. In the case of RS-S, the compressive strength of the samples has dramatically decreased after cycle 15. In the case of performing the cycles in seawater, the compressive strength of NS-Si has significantly increased, but in the case of RS-B and RS-S there has not been an appreciable variation. Finally, in the case of performing the cycles in water with sulphates, the compressive strength of NS-Si and RS-B has suffered a significant increase, while the compressive strength of RS-S is similar to that of the reference samples.

After wet-dry cycles in seawater or in water containing sulphates, the mortars do not show any damage. From a mechanical point of view, they do not suffer any reduction of compressive strength. On the contrary, an increase in compressive strength can be observed, which is remarkable in the higher quality mortars (MMS-NS-Si and MMS-RS-B). This is quite unexpected, but other authors have found similar phenomena. Fiol et al. [35] also found an increase in compressive strength of mortars after drying-wetting cycles in seawater. Kampla et al. [36] concluded that the presence of fly ash produces a strength improvement against wet-dry cycles, since it significantly improves the resistance to water absorption. Ghassa Fahim et al. [37] concluded that fly ash strongly influenced the mechanical and durability properties of alkali-activated mortars, and after exposing the mortars to sulphate attack of 6 and 12 months they observed an increase in compressive strength, which they explained due to a high Na content in their aggregates, which affected the formulation of CaSO_4 and stable behaviour against a sulphate environment.

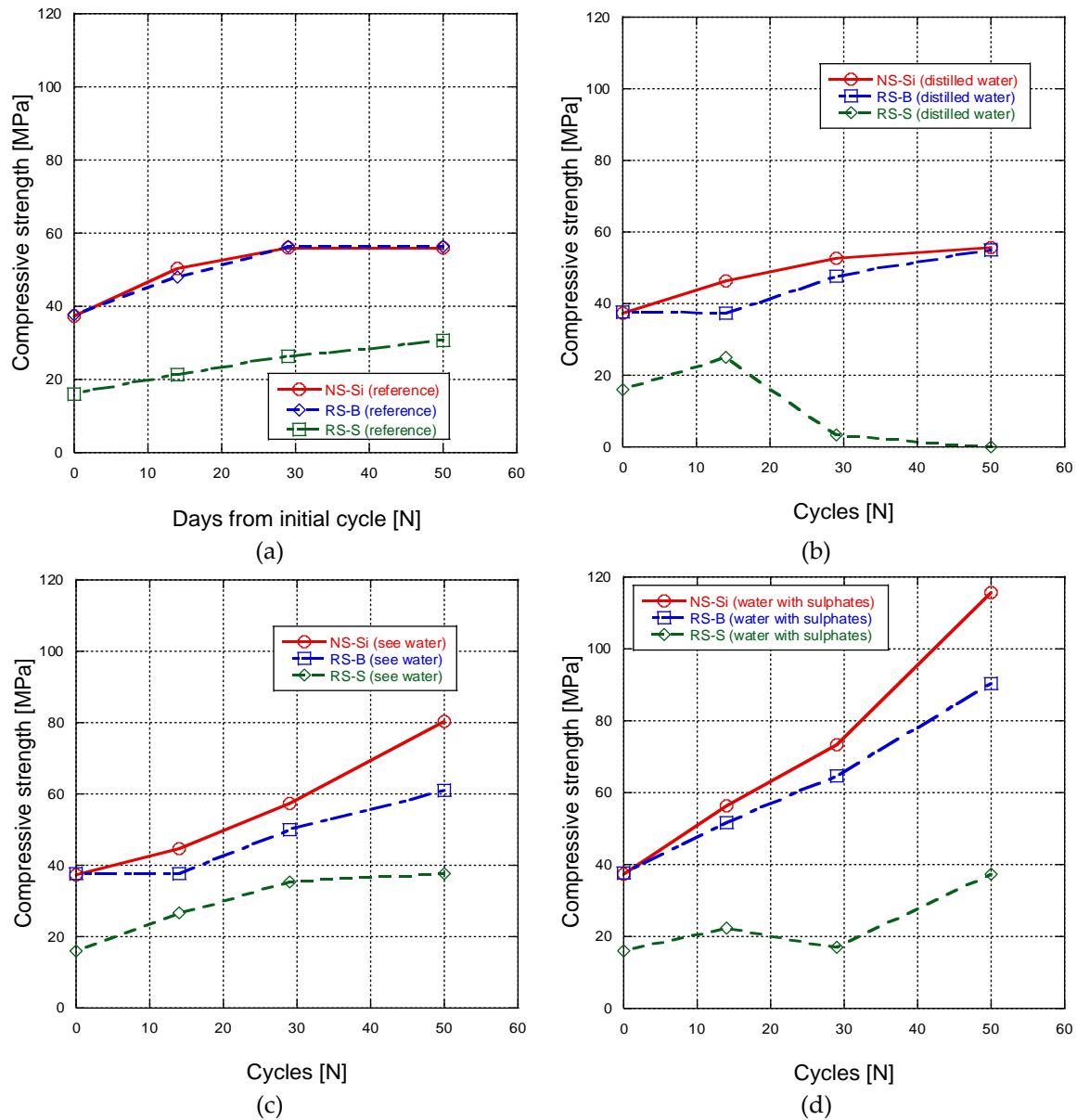


Figure 8. (a) Evolution of the compressive strength of mini-mortar samples without durability cycles; (b) evolution of the compressive strength when mortars are subjected to cycles in distilled water; (c) evolution of the compressive strength when mortars are subjected to cycles in seawater; (d) evolution of the compressive strength when mortars are subjected to cycles in water with sulphates.

It seems that these results are largely due to the type of cement used (containing fly ash), which not only controls the generation of expansive reactions, harmful to the structural integrity of the material. It is thought that the conditions of continuous moisture and heat promote and increase the pozzolanic effect of the fly ash. Following these improvements in the mortars that produce fly ash activation, each of the fluids for the wet-dry cycle tests have damaged the material to a certain degree. The most aggressive environment has turned out to be that in which distilled water is used, which has a high purity. Because of this exposure, the MMS-RS-S suffers a progressive deterioration that leads to its disintegration without reaching 50 cycles. The other two mixes show better behaviour, which translates into a minimum loss of compressive strength.

3.4. Morphological analysis of fractures

Periodically, in parallel to compressive strength tests, photographs were taken of the external

appearance of the specimens. In Figure 9 to Figure 11, it is possible to appreciate the changes manifested as the exposure time for the three types of mortar increased. The changes are observed when comparing the appearance of the specimens before starting the moisture-drying cycles, after passing 25 and 50 cycles in the above-mentioned environments.

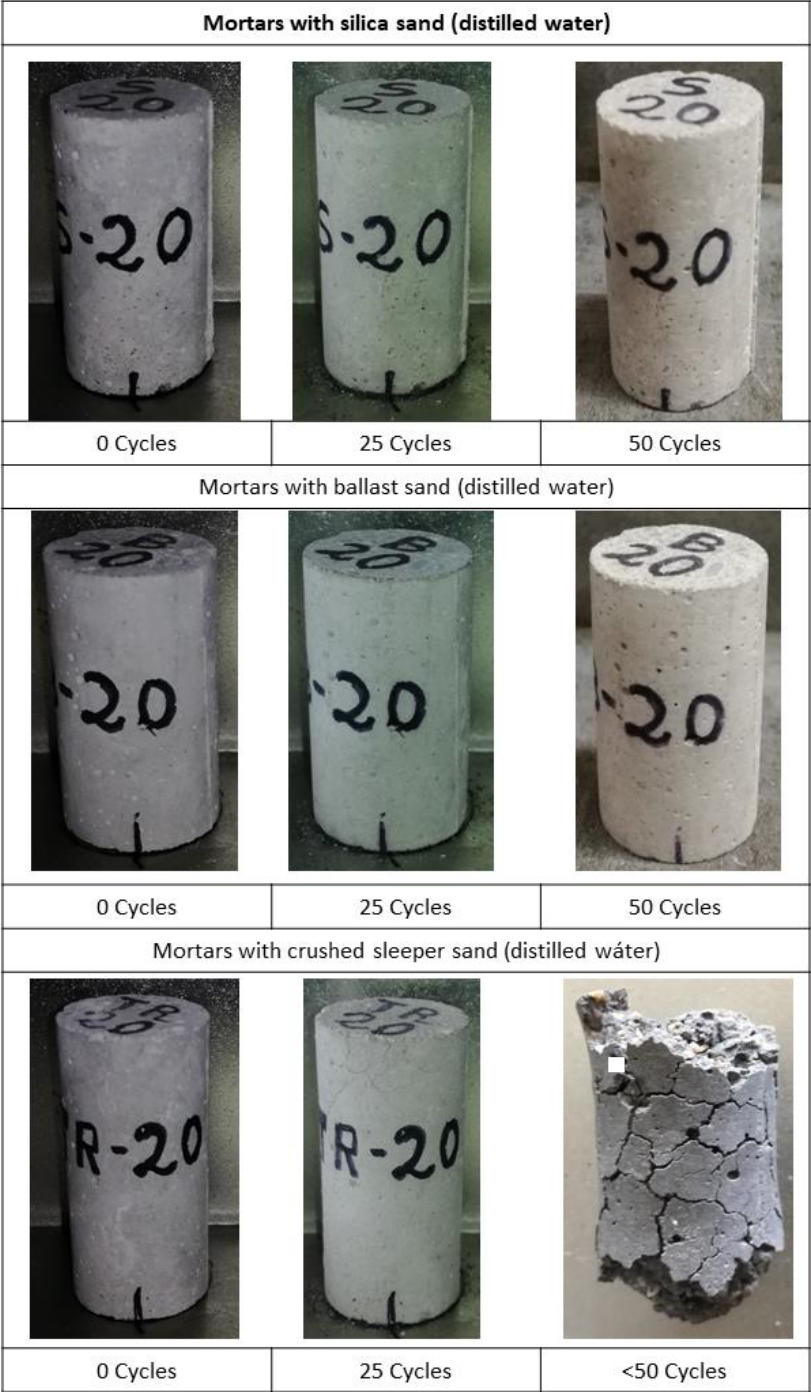


Figure 9. Evolution of the external surface during the dry-wet cycles in distilled water of the three types of mortar for different numbers of cycles.

In the case of the mortars subjected to dry-wet cycles in distilled water, in the case of MMS-NS-Si and MMS-RS-B, the samples have not suffered any type of damage at surface level. In the case of MMS-RS-S, the samples disintegrated before reaching 50 cycles, which is why the cycles on these mortars were stopped.

In the case of the mortars subjected to durability cycles in seawater, in the same way as in the

previous case, MMS-NS-Si and MMS-RS-B have a similar behaviour, although in this case some surface scaling can be seen. In the case of MMS-RS-S, this time the samples have not suffered a damage so severe as in the case of distilled water. MMS-RS-S suffered a type of damage similar to MMS-NS-Si and MMS-RS-B but to a greater degree.

Finally, in the case of the mortars submitted to cycles of humidity dryness in water with sulphates, a similar behaviour is also seen between MMS-NS-Si and MMS-RS-B, but this time what is seen is some discoloration of the specimens. In the case of MMS-RS-S on the other hand, they have suffered some damage, especially at the edges, which are the zones more susceptible to be damaged since the external agents can penetrate through different fronts and, therefore, more quickly.

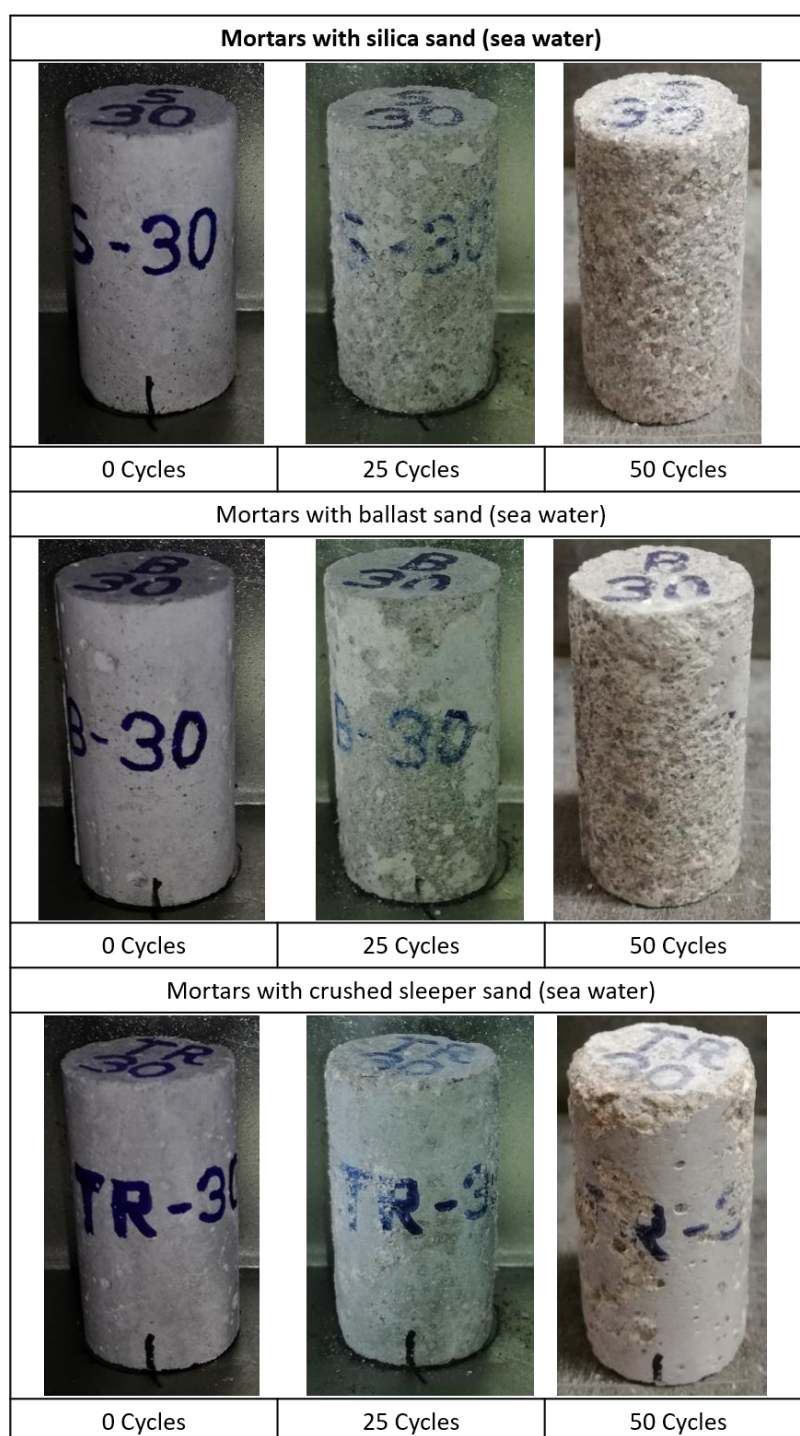


Figure 10. Evolution of the external surface during the dry-wet cycles in seawater of the three types of mortar for different numbers of cycles.

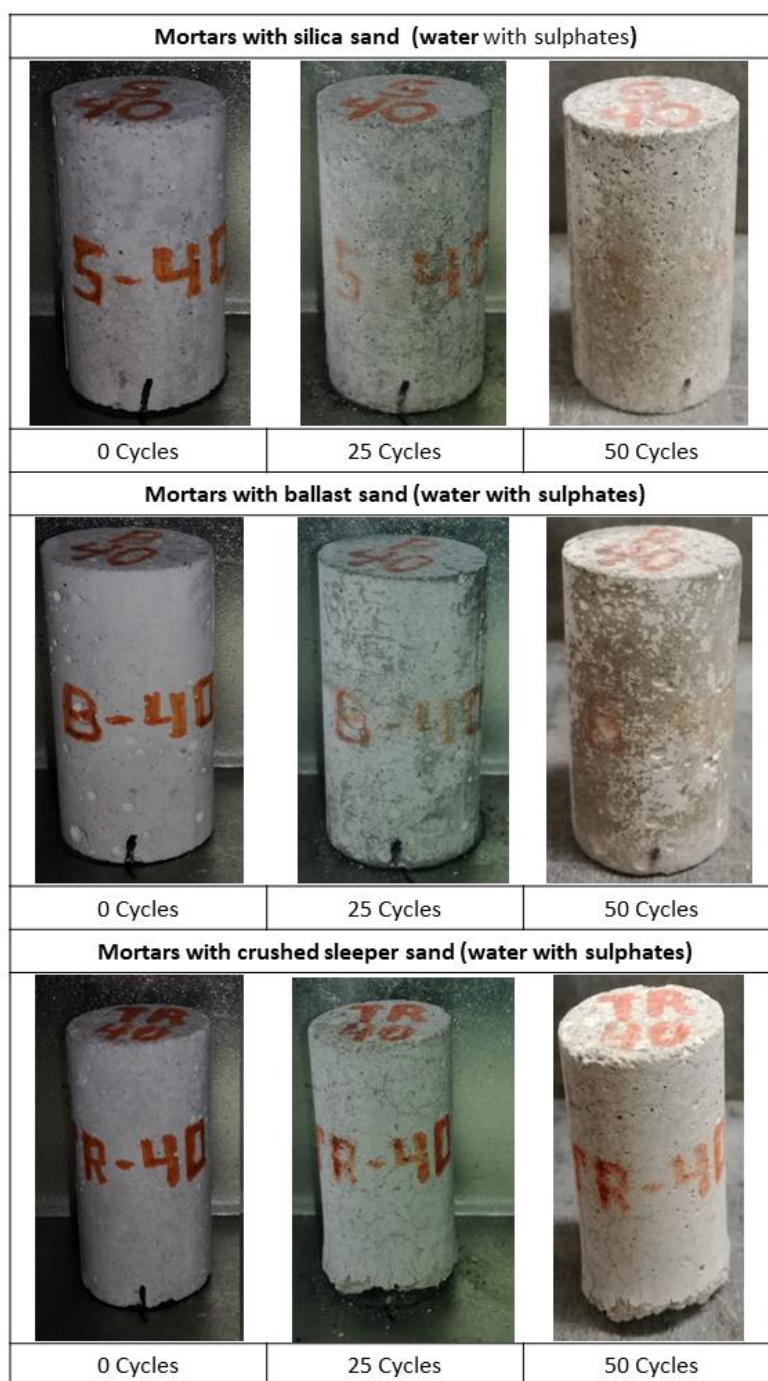


Figure 11. Evolution of the external surface during the dry-wet cycles in water with sulphates of the three types of mortar for different numbers of cycles.

3.5. Scanning electron microscopy

At the end of the durability cycles, a scanning electron microscopy (SEM) study was carried out to analyse how the dry-wet cycles had influenced the products that could be found in the mortars. These SEM tests were also carried out on specimens that had not been subjected to cycles, i.e. standard specimens. In Figure 12 to Figure 15, a series of micrographs of the three types of specimens in the four specified environments are included. Figure 16 shows the chemical composition of different microstructures found in the different mortars.

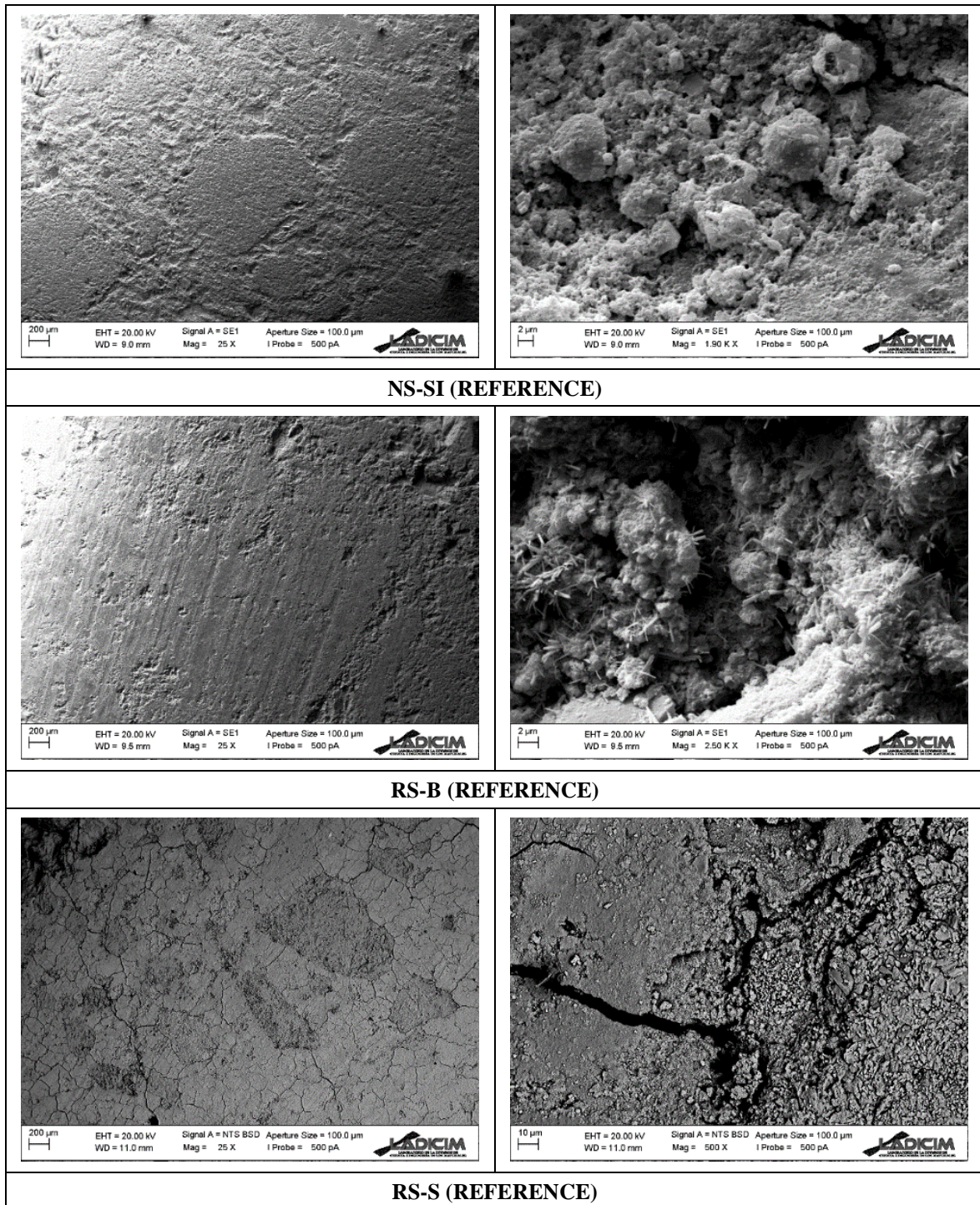


Figure 12. Microstructure of the three types of mortar not subjected to any durability cycles.

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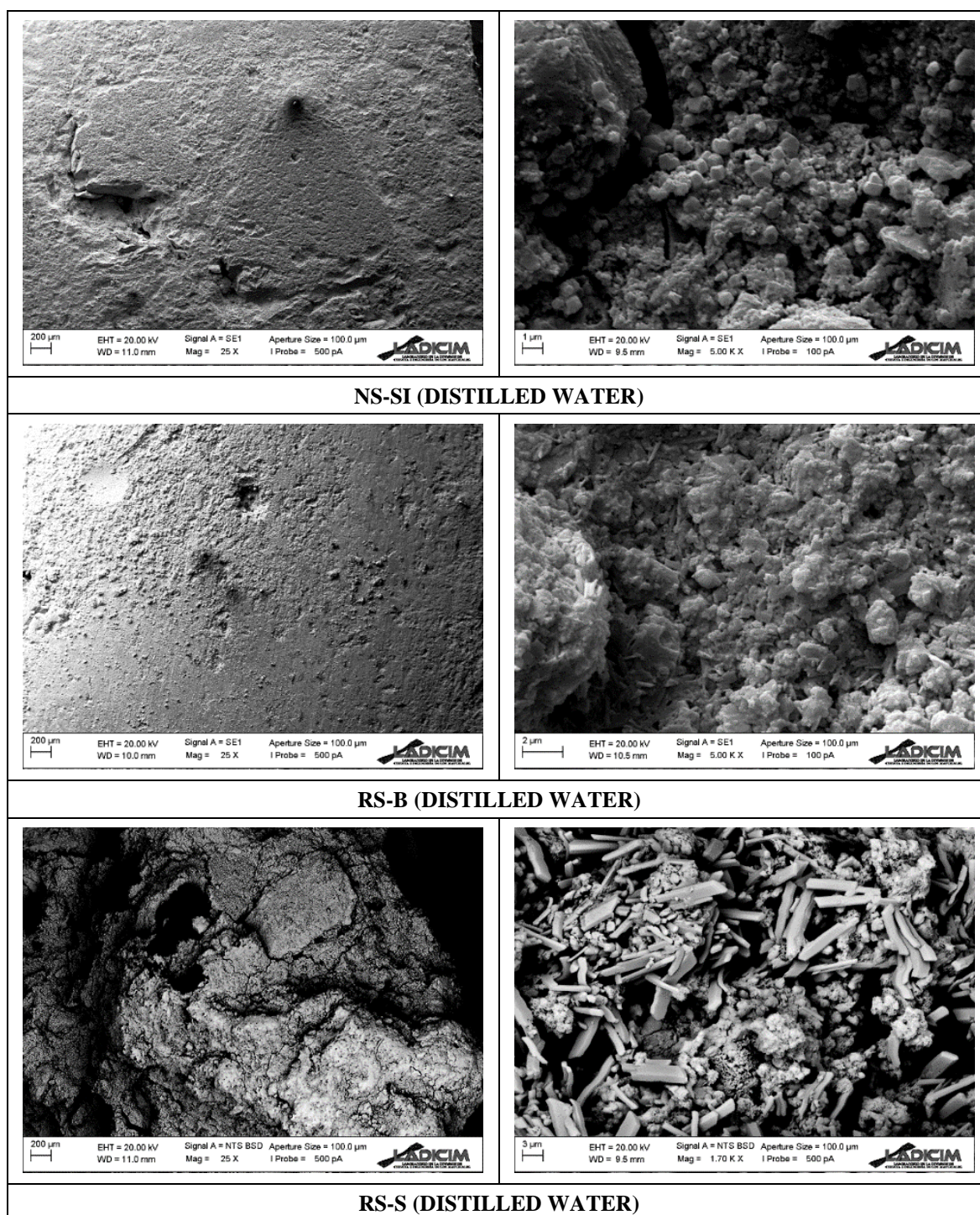


Figure 13. Microstructure of the three types of mortar after the durability tests performed in distilled water.

In the case of specimens subjected to wet-dry cycles in distilled water (Figure 13), NS-Si and RS-B microstructures similar to those found in the case of the standard samples can be seen. This may explain why no modifications have occurred in the mechanical behaviour of these samples. In the case of the RS-S specimens, the structures were concluded to be ettringite after the chemical study in Figure 16 (c). Due to the expansive character of the ettringite, the samples suffered self-destruction. The reason for the bad results shown by the mortar with recycled sand coming from sleepers in contact with distilled water in their high permeability. This facilitates the processes of dissolution and repeated recrystallization cycle after cycle of the hydrated products of cement, especially of portlandite, a fact that is corroborated after the microscopic observation that was carried out.

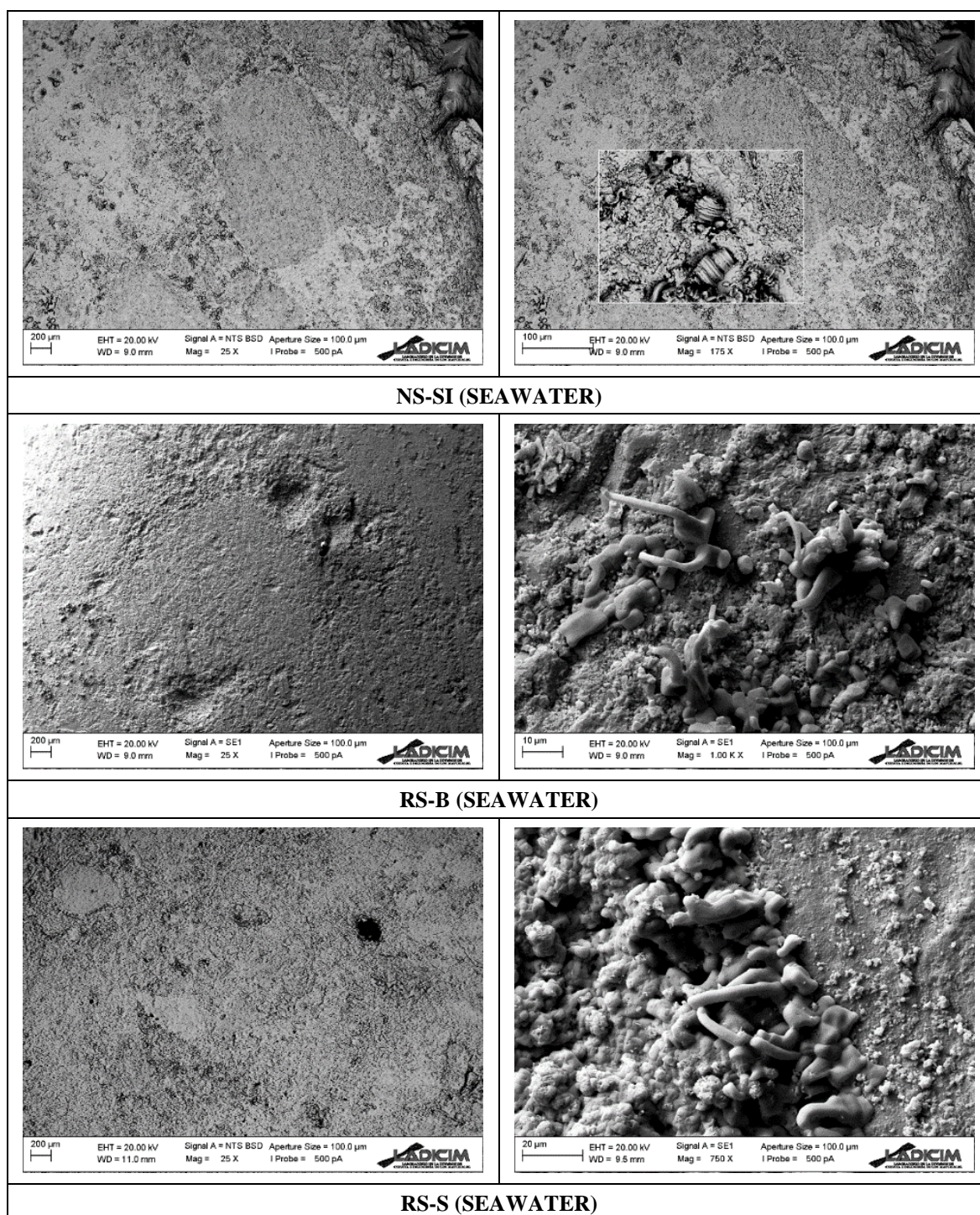


Figure 14. Microstructure of the three types of mortar after the durability tests performed in seawater.

In the case of the samples subjected to durability cycles in seawater (Figure 14), similar structures were found in all three types of mortar. After chemical analysis, as per Figure 16 (d), it was concluded that these were salt deposits (NaCl), which is a less common marine salt formation [38].

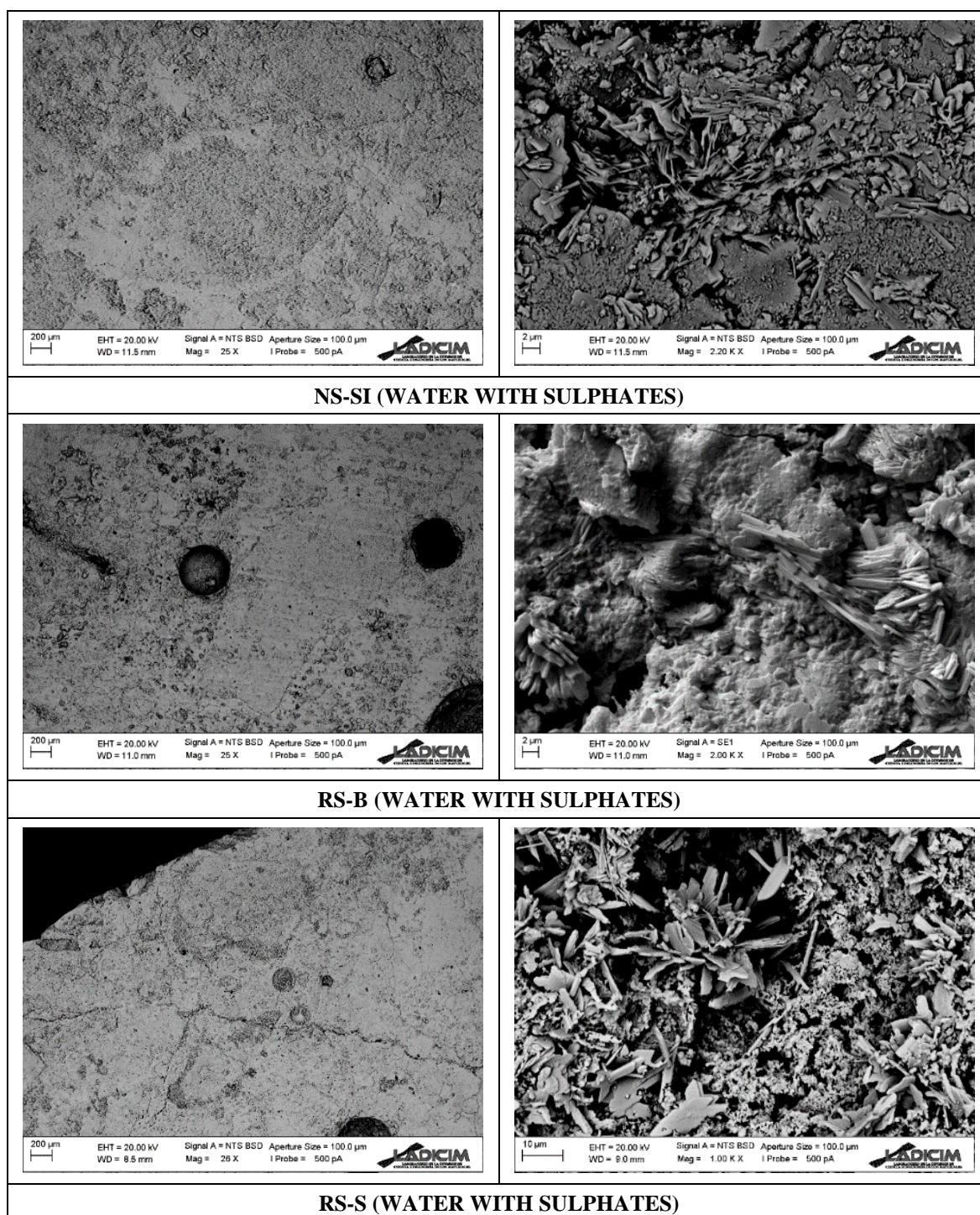
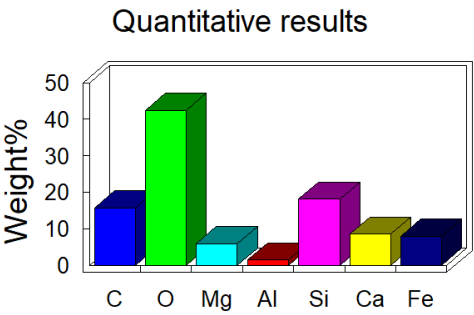
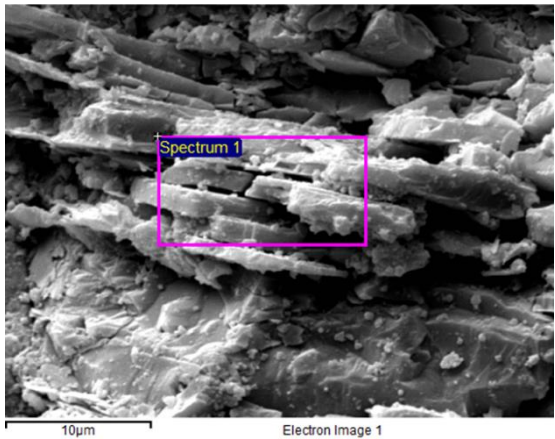


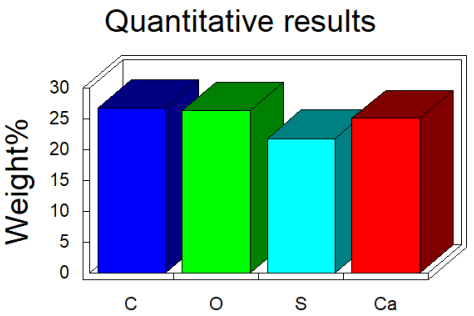
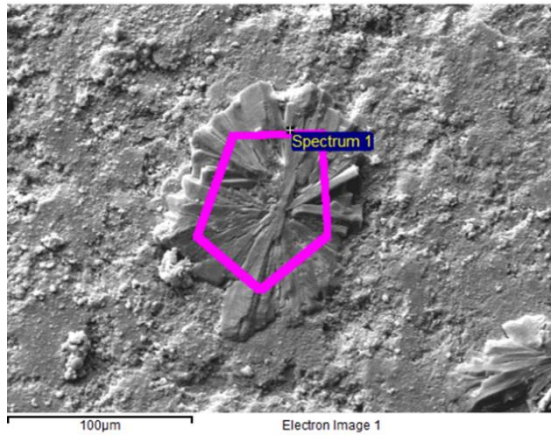
Figure 15. Microstructure of the three types of mortar after the durability tests performed in water with sulphates.

Finally, in the case of the samples subjected to durability cycles in water with sulphates (Figure 15), the micrographic study reveals the deposit of crystallizations of the salts dissolved in the surrounding water with their recognized morphologies (hexagonal plates in the case of sulphates). The chemical analysis of Figure 16 (e, f and g) shows that these are potassium sulphate deposits. These deposits seem to contribute to the densification of the matrix and, also, to the gain of mechanical performance [35,37].

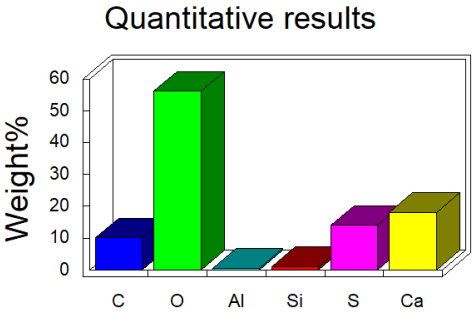
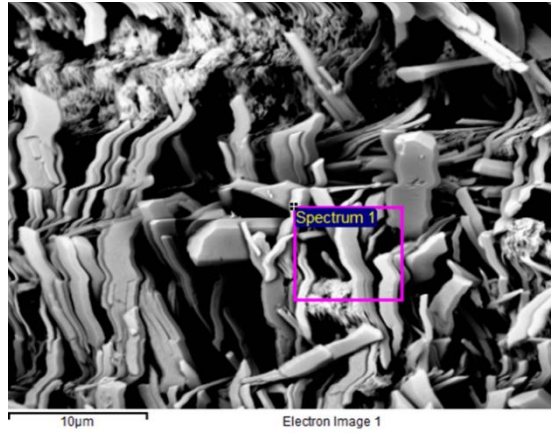
It is expected that increasing the number of cycles, when the pores and fissures are saturated, will lead to the deterioration of the material because the pressure that these compounds will apply to the mortar will continue to grow.



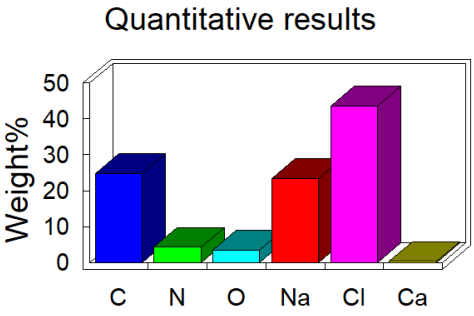
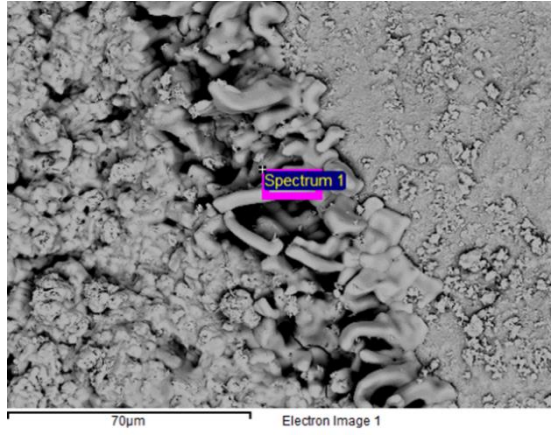
(a) RS-B (distilled water)



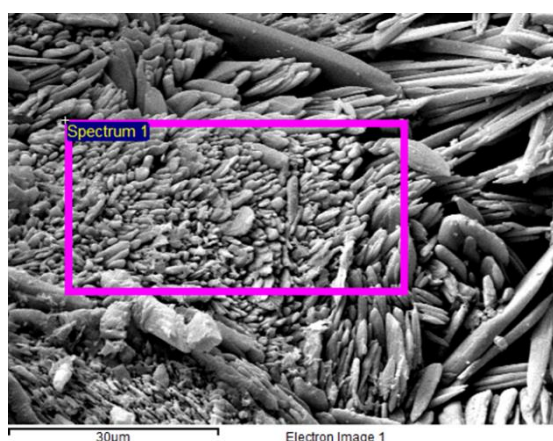
(b) RS-B (distilled water)



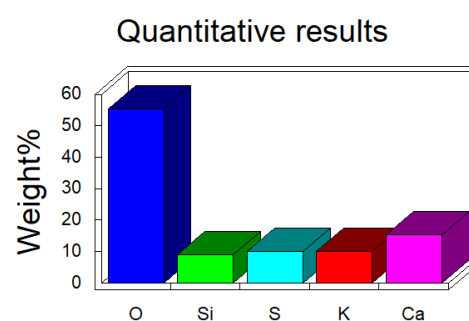
(c) RS-S (distilled water)



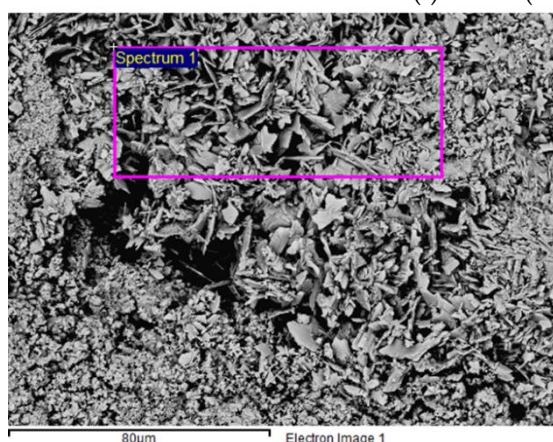
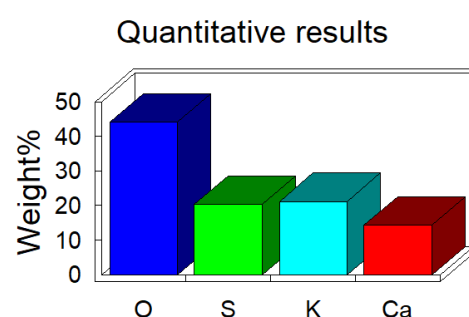
(d) RS-S (seawater)



(e) NS-SI (water with sulphates)



(f) RS-B (water with sulphates)



(g) RS-S (water with sulphates)

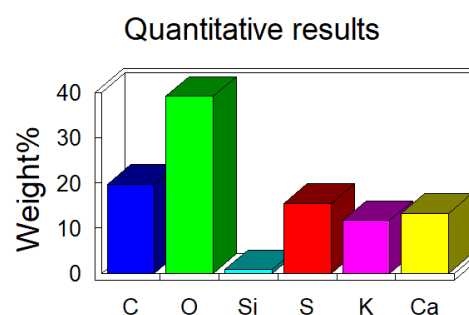


Figure 16. Chemical composition of the different microstructures found in the mortars.

4. Conclusions

After analysing the mechanical, visual and microstructural behaviour of mortars subjected to different accelerated ageing processes, the following conclusions were drawn:

- Mortars made with recycled sand from crushed ballast have a very similar behaviour to the reference mortars containing natural silica sand. They withstand 50 cycles to the three types of exposure analysed, showing, in the worst case, a slight surface peeling;
- Mortars made from recycled sand obtained after crushing out-of-use sleepers are affected to a greater extent by exposure cycles. This deterioration is particularly noticeable when the liquid used is distilled water, which results in almost total disintegration of the sample. Microstructural analysis of this mortar shows an abundant presence of ettringite that,

given its strong expansive character, could be the origin of the massive deterioration observed in the samples after being exposed to 25 wet-dry cycles;

- The increase in mechanical properties seen in the mortar samples after some of the durability tests carried out are due to these cycles allowing the mortars to be exposed to high humidity and temperatures of 70 °C. These boundary conditions promote the hydration of the compounds still existing in the mortar. As a cement with a high content of fly ash has been used, after 28 days there is still an important capacity for evolution of the compounds;
- The increase seen is expected to be a combination of the increase explained above and a reduction due to durability cycles. However, until the 50th cycle, the value of the increase is greater than the loss in most of the conditions analysed.

As a future work to validate the conclusions here exposed, another experimental campaign is planned divided in two stages, one similar at 28 days, but carrying out a fifth group, dry-wet cycles in tap water, and the same set of tests at 90 days. *In addition, due to the great importance of the origin and the crushing procedure of the aggregates, as a future work, it is also proposed to contrast the results presented in this work with those from other recycled aggregates.*

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