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Performance of mortars based on recycled glass as aggregate by accelerated decay tests (ADT)

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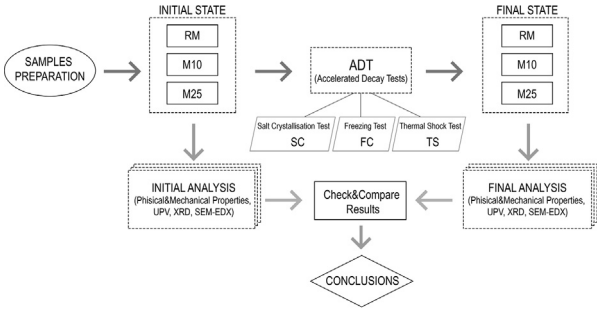
HIGHLIGHTS

- Crushed glass aggregates show good performance in accelerated decay tests.
- Mortars with glass improves significantly against the crystallisation of salts.
- Glass means a clear improvement in the compression resistance in the decay tests.
- The ITZs of the glass aggregates develop a lower degree of microcracking.

GRAPHICAL ABSTRACT



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ABSTRACT

The incorporation of crushed glass as a substitute for natural aggregates in mortar and concrete represents a positive impact on the manufacturing processes. The use of glass residues can lead to the improvement of certain properties of sustainable construction products. In these cases, it is important to analyse in detail the behaviour of the material to verify possible applications at an industrial level and contribute to the development of new construction products and solutions. This work studies the behaviour of mortars with recycled glass as a partial replacement of aggregate against common accelerated decay tests, such as salt crystallization, freezing and thermal shock. The influence on the basic physical and mechanical properties has been evaluated by statistical analysis of the results, observing a particularly significant improvement in the resistance of mortars with recycled glass against the expansive action of salt crystallization (>20%). The influence of alteration processes on the internal structure has been analysed by ultrasound transmission tests and its correlation with Young's modulus. The values obtained are in the range of similar materials, having the samples subjected to freezing test a major degree of deterioration. The internal structures have been studied by scanning electron microscopy, observing in samples subjected to salt crystallization tests a greater homogeneity and a lower degree of microcracking in the aggregate/paste interfaces of the glass grains.

1. Introduction

The reuse of household waste is an objective of public administrations to reduce the consumption of natural resources and the deposits of useless material in landfills. This objective requires political and technological measures to develop technology capable of recovering certain wastes for production processes and to

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promote actions from public authorities whose aim is to minimize environmental impacts. These public policies must take into account the impact of products throughout their life cycle, including the waste hierarchy and, when appropriate, the potential for multiple recycling (Directive (EU) 2018/852; ASTM D636-17) [1]. The construction sector, due to the high consumption of raw materials, should play a relevant role in this field to provide an environmentally friendly and economical solution by eliminating this waste and reducing the cost of concrete/mortar (Directive (EU) 2018/851; ASTM E3073-17) [2].

There is a high volume of glass waste generation and a great recovery capacity in domestic production. The volume of waste from selective collection of glass has experienced a sustained increase since 2000. The total production of glass containers in the European countries associated to the European Container Glass Federation (FEVE) is currently 21.7 MT and 78.662 million units, representing an increase of 10% and 9.8% respectively in relation to the 2012 production levels [3].

The European Union (EU) objectives established that a 70% of all domestic glass generated by citizens from member countries should be recycled by 2017 [4]. Currently, the projection of the volume of urban glass waste obtained by selective sorting approaches 12 MT / year. According to the data of the FEVE published in 2019, in the past 15 years, EU consumption of products packed in glass increased by 39%, while glass recycling increased by 139% and the average rate of recycling glass packaging in the EU was stabilized at 76% [3].

Waste glass could be effectively used as a construction material. The ground glass powder could be used as supplementary cementitious material and the remaining glass cullet could be used as an alternative to standard sand [5]. Although, the use of recycled glass in mortar is still limited, there are numerous studies about the behaviour of building mortars with partial replacements of the aggregate by crushed glass residue [6]. Previous researches confirm that the replacement of sand by crushed glass allows modifying certain properties of the mortar, without decisively affecting the basic physical and mechanical properties (porosity, density, resistance to flexural and compression) [7,8]. The work done by Mohajerani *et al.* (2017) reveals the disparity of results that can be obtained with this type of materials (crushed glass) depending on the characteristics of the components and the dosages used in glass replacements [9].

The use of glass as an amendment to Portland cement concrete serves as a potential alternative to traditionally used supplementary cementitious materials. So, considering that, one can find studies related to the behaviour of mortars with crushed glass (CGM) in special aggressive environmental situations, where the improvement of the resistance to the alkali-aggregate reaction was observed [10] and how CGM achieve a decrease in expansion caused by alkali-silica reaction when waste glass is used as a pozzolan [11].

Other property that is significantly affected is the thermal conductivity. The influence of glass aggregate on the thermal conductivity of CGM and its favourable influence on resistance to high temperatures are well known [12,13]; developing a mechanical resistance better than conventional mortars [14]. Regarding the study of the accelerated decay phenomenon, there are studies that show positive results in the behaviour of CGM at low temperatures. Mardani-Aghabaglou *et al.* (2019) have shown that an improvement of frost resistance of CGM was attributed to the presence of better interfacial transition zone (ITZ) between matrix and coarse aggregate [15]. This fact is more evident by increasing the percentage of crushed glass replacement used in the mixture. The addition of glass aggregate in mortar/concrete contribute to the material durability and sustainability and could lead to enhance the properties of these materials under the combined conditions of freezing and thawing cycles with expansive salts [16,17].

The resistance to the action of expansive salts is a well-studied process for conventional mortars and concretes, mainly sulphates attack. Previous research have shown a better behaviour in the mechanical strength of CGM after the sulphates attack in comparison with reference samples, particularly for flexural strength [18]. These results confirm the investigations of Chen et al. (2006) who presented a lower reduction in resistance in the material with a higher percentage of crushed glass [17]. Regarding to their possible applications in construction and engineering, CGM may be suitable for application in cases of aggressive sulphate conditions that may occur in soil and groundwater, in industrial effluents and wastes from chemical and mining industries, as well as in seawater [19].

ADT are experiments that provide information from random samples that are tested under severe conditions to know their level of deterioration. ADT allows establishing a qualitative prediction of the behaviour of materials under evaluation by comparing obtained results from modified samples and reference ones [20].

The objective of this work was to evaluate the behaviour of CGM subjected to ADT by crystallization of expansive salts (SC), freezing (FC) and thermal shock (TS) cycles, analysing its behaviour against reference mortars. The study has been carried out through destructive and non-destructive tests that allowed analysing the physical and mechanical properties of the materials. Likewise, the internal structures have been analysed to verify how deterioration has manifested itself at the microscopic level, the appearance of microcracks and the effect of SC. This work evaluated CGM results in comparison with mortar manufactured only with natural siliceous sand as aggregate.

2. Materials

Three mortars with a 1:3 ratio (cement:aggregate) have been manufactured (Table 1). The first one is the Reference Mortar (RM) which contains exclusively sand and the remaining two contains a 10% and a 25% of crushed glass as partial replacement of the sand (M10 and M25 respectively). These percentages have been established based on previous studies that showed optimal results around 25% replacement of aggregate by glass [9,13].

The materials used in mortar manufacture and their dosages are shown in Table 1: CEBASA CEM I 42.5R cement, (http://www.cebasa.com); natural siliceous sand provided by the Eduardo Torroja Institute (UNE-EN 196–1:2018) [21], with a maximum compactness of 98% and a maximum diameter of 2 mm; glass from domestic waste, mostly common packaging, was crushed in jaw crusher and sieved to a particle size of less than 2 mm with similar granulometric fractions to the standard sand. The grain size distribution was established by fractions according to Fuller's maximum compactness curves [13,22]. Its chemical composition was determined by XRF (Table 2) and it was shown as a conventional soda-lime glass from domestic recycling [23,24].

Samples have been validated according to their docility and the requirements of workability of the standard UNE-EN 13395-1 [25].

The granulometry of the crushed glass was the same as that of the sand, this denotes that the compactness of the aggregate will be similar, although it must be borne in mind that the specific surface area may differ due to the different shape of the grains, being those of sand rounder than crushed glass shards This issue has

Table 1
Dosages of components for each batch.

	Cement (gr)	Sand (gr)	Glass (gr)	Water (cm^3)
RM	450	1350	–	300
M10	450	1215	135	300
M25	450	1012.5	337.5	300

Table 2
Chemical composition (XRF) of the used glass.

SiO_2	Al_2O_3	Fe_2O_3	MnO	MgO	CaO	Na_2O	K_2O	TiO_2	P_2O_5	SO_3	LOI
72,05	1,50	0,19	–	0,76	11,38	13,19	0,72	–	–	–	0,10

influence in the ITZ behaviour during the ADT and mechanical results.

3. Methodology

There are no specific standards for CGM, so the experiments were carried out by adapting applicable standards according to their development. However, those standards that suppose a major degree of aggressiveness were selected. Non-standard tests can provide a good evaluation of the physical and mechanical properties of the CGM, being able to reach significant conclusions according to the proposed experimental methodology [26].

Series of 40x40x160 mm specimens were prepared for each dosage (Fig. 1), manufactured in accordance with UNE-EN 196–1:2018 [21] standard specifications and cured under optimum humidity (95%) and temperature (20 °C ± 2) conditions for 28 days. Each specimen was sectioned to obtain three 40x40x40mm samples and they were prepared in three group to undergo different ADT:

- Salt Crystallisation Test (SC): 15 salt crystallization cycles by immersion for 2 h in a 14% solution of $Na_2SO_4{\cdot}10H_2O$ and subsequent drying in an oven at 105 °C ± 5 (UNE-EN 12370:1999) [27].
- Freezing Test (FC): 25 freezing cycles consisting of 18 h at −15 °C ± 5 and 6 h in a de-icing tank at 15 °C ± 5 (UNE 67028:1997) [28].
- Thermal Shock Test (TS): 20 thermal shock cycles consisting of 18 h at 70 °C ± 5 and 6 h in a water tank at 20 °C ± 5 (UNE-EN 14066:2014) [29].

Fig. 1. RM, M10 and M25 specimens before being sectioned.

The apparent density and the porosity under vacuum have been calculated in accordance with the UNE-EN 1936: 2007 standard. The mechanical resistance to compression at 28 days according to the UNE-EN 196–1: 2018 standard, with a load speed of 50 N/s until failure. To determine if there are significant differences in the results of the physical and mechanical properties, the Student's T-test has been used, establishing a significance level of $\alpha = 0.01$. This statistical model is valid for normal distributions with small sample sizes [30].

The ultrasonic pulse velocity (UPV) determinations were carried out on groups of 9 specimens (40x40x40 mm) for each of the ADT, considering the corresponding RM, M10 and M25 subgroups. Measurements were taken in the three directions of space (X, Y, Z), being Z axis the compaction direction of the specimens. An UltraTest GmbH Bp-5, equipped with 50 kHz cylindrical transducers, was used to determine UPV. These data allowed the calculation of Young's modulus "E" (GPa) according to the equation 1, depending on UPV "v" (m/s) and density "ρ" (Kg/m^3):

$$E = v^2 \cdot \rho$$

The mineralogical evolution of the materials has been analysed by X-ray diffraction in a Bruker-AXS diffractometer, model D8I-A25, equipped with a Cu Kα copper filament (λ = 1.5405 Á), with a Bragg-Brentano θ-θ configuration, nickel filter and Lynxeye linear detector, using the powder technique.

To evaluate inner changes in the structure of the CGM, the study by scanning electron microscopy (SEM) was carried out in a FEI-TENEO microscope equipped with Energy Dispersive X-ray Spectroscopy (EDS) for microanalysis. The samples were previously metallised with a thin layer of Au by sputtering, to avoid charge saturation during the measurement. EDS microanalysis of these samples was performed under the SEM equipment using a beryllium ATW2 window at 15 kV and specific software (Oxford INCA) for semiquantitative chemical analyses. Their calibration was done using a Cu sample.

All the tests described were performed on the samples in their initial state and after undergoing the ADT. In this way, its behaviour against degradation processes and the influence of partial replacement of aggregate by glass on the durability of the CGM can be evaluated.

4. Results and discussion

4.1. Physical properties and statistical analysis of the results

Table 3 shows the average values of the physical properties and highlights how the incorporation of glass does not imply important differences in the density of the material and how a slight decrease in porosity from RM to M25 occurs in the initial state. This decrease is significant for M25 [t(34) = 3.142, p = .003].

The accumulation of Na_2SO_4 crystals in the porous system, as a result of the SC test, causes an increase in density that reaches significance in the RM, M10 and M25 samples respectively: RM[t(22) = -6.893, p = .000]; M10[t(22) = -19.591, p = .000] and M25 [t(22) = -9.142, p = .003] and, consequently, a decrease in porosity that is also significant is observed: RM[t(22) = 4.870, p = .000]; M10 [t(22) = 9.522, p = .000] and M25[t(22) = 6.105, p = .000]. These results indicate that there is no relevant microcracking effect due to the process derived from the expansive crystallization of salts.

Table 3
Average values of bulk density ρ (g/cm^3) and vacuum porosity P (%).

SAMPLES	INICIAL STATE		FINAL STATE					
			SC		FC		TS	
	ρ	P	ρ	P	ρ	P	ρ	P
RM	1.93	25.83	2.13	24.09	1.94	26.47	1.95	24.95
M10	1.95	25.49	2.23	23.14	1.92	26.68	1.93	25.53
M25	1.94	25.07	2.20	22.66	1.93	25.98	1.93	25.01

As it could be appreciated in Table 3, RM sample subjected to the FC test did not suffer noticeable variation. In contrast, a significant increase in porosity was observed in M10 and M25: M10[t(22) = -4.819, p = .000] and M25[t(22) = -2.979, p = .007], motivated by microcracking caused by cycles of freezing.

Moreover, it is important to mention that the TS test have not caused significant modifications in the evaluated physical properties.

4.2. Compressive strength and statistical analysis of the results

The values obtained from the compression test resulted more interesting than expected, mainly when observing the differences in the behaviour depending on the type of alteration to which the materials have been subjected (Table 4). In all cases, the specimens M10 [t(14) = -4.397, p = .001] and M25 [t(14) = -5.427, p = .000] presented a significantly better mechanical behaviour than RM, whose resistance improved due to the replacement of aggregate by crushed glass.

Regarding the data obtained for the initial/final state, it has been observed that the specimens subjected to the SC test have a statistically little significant drop in resistance: RM [t(14) = 2.025, p = .062]; M10[t(16) = -0.314, p = .758]; M25[t(16) = 0.454, p = .656.

The specimens subjected to FC test presented a major loss of resistance than those subjected to SC: RM[t(14) = 6.509, p = .000]; M10[t(16) = 6.771, p = .000]; M25[t(16) = 9.301, p = .000] and a similar behaviour was manifested due to the substitution of aggregate by glass.

In the TS test, no significant influence of the alteration process was observed on the RM specimens RM[t(14) = 1.112, p = .285]. However, the incorporation of glass did not show any positive effect on samples M10[t(16) = 8.849, p = .000] and M25[t(16) = 4.736, p = .000].

Considering that the interfacial transition zone (ITZ) is a very heterogeneous gradual transition region, the better mechanical behaviour of M10 and M25, in comparison with RM, may be due to the less irregular surfaces of the glass aggregate that allow a continuous and homogeneous adhesion of the paste to the aggregate [31]. Thus, the crystallization of salts and the effects of freezing water in the interstices are hindered, so that the weakening of these interfaces is reduced.

Table 4
Average compression strength values (N/mm^2).

SAMPLES	INITIAL STATE	FINAL STATE		
		SC	FC	TS
RM	32.0	29.7	24.3	30.8
M10	36.8	37.1	27.5	29.1
M25	37.9	37.4	28.6	33.3

4.3. Ultrasonic pulse velocity analysis

The analysis of the results obtained enables the characterization of mortars for their deformability and not only for their mechanical resistance [32]. The obtained results from the UPV may be less accurate than those obtained from vibration excitation by impact [33], although the trends obtained showed significant data (Table 5).

An influence was observed in the initial state due to the replacement of aggregate by crushed glass, increasing the UPV proportionally to the amount of glass. As indicated above, the less irregular surfaces of the glass aggregate allow a continuous and homogeneous adhesion of the paste to the aggregate and contribute to a higher UPV.

There is a more pronounced trend in UPV for the specimens subjected to SC and significant decrease in those subjected to FC, although in this case there is no difference between the three groups of samples. In the specimens subjected to SC, there is an opposite effect that has its origin in the microcracking caused by the stresses due to the crystallization of the salts and the crystallizations themselves. There is a severe drop in the UPV which could be due to the cracks produced in the specimens subjected to FC.

Samples after SC test showed a clear positive result due to the addition of glass, reaching a difference of 17.7% in the UPV in sample M10 and 44.2% in sample M25. The values of UPV obtained after FC are similar and is higher on M25.

The values obtained for the YM[34] [35] are, in all cases, in common ranges linked to similar materials [35,36]. CGM samples subjected to SC test show a slight increase in YM and samples subjected to FC test present relevant decrease according to the drop in mechanical resistance.

4.4. Mineralogical analysis by X-Ray diffraction

Considering that the FC and TS tests do not generate any type of modification in the mineralogical phases present in the material, the mineralogical analysis by XRD was only carried out on the samples subjected to SC. The diffractograms reveal the usual phases of

Table 5
UPV Average (m/s), YM: Young's modulus (GPa), YMr: Δ Young's modulus relative to the initial value (%).

SAMPLES	INICIAL STATE		FINAL STATE								
			SC			FC			TS		
	UPV	YM	UPV	YM	YMr	UPV	YM	YMr	UPV	YM	YMr
RM	4131	32.9	3820	31.1	94	2439	11.6	35	3373	22.2	67
M10	4584	40.6	4483	44.8	110	2510	12.1	30	3536	24.1	59
M25	4219	34.4	4033	35.8	104	2344	10.6	31	3655	25.8	75

the mortar and do not indicate any phase of alteration due to degradative reactions. A small peak has been identified corresponding to traces of thenardite (Na_2SO_4) (Fig. 2).

4.5. Scanning electron microscopy (SEM-EDS)

The main purpose of the analysis by SEM analysis has been to study the microcracking produced in the mortar matrix due to the different ADT [37], the morphology of the cracks [38], the salt crystallization processes in the porous system produced in the SC test and the study of the possible modifications produced in the ITZ [39,40,41].

In samples subjected to SC, crystallization cores have been observed and identified by EDS as sodium sulphate (Na_2SO_4). The analysis of the images allows us to verify how changes are produced in the texture of the material, in the interfaces added to the paste, such as a major presence of microcracks in the environment of the grains. (Fig. 3)

Typical acicular crystals, commonly found in sulphates, have been detected in the perimeter of the aggregate grains, causing stresses, and generating fractures. (Fig. 4). According to other authors, it should be noted that the attack mechanisms of sulphates through immersion cycles are mainly due to physical attack of sulphates and not to reactions with the cement components and

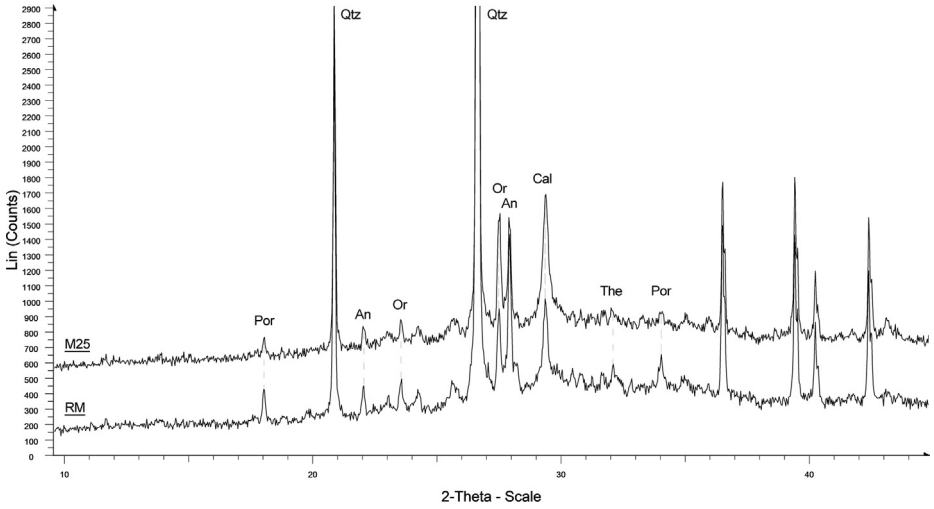

Fig. 2. X-Ray diffractograms of RM and M25 samples subjected to SC (Qz: quartz, Cal: calcite, Por: portlandite, An: Anorthite, Or: orthose, The: thenardite).

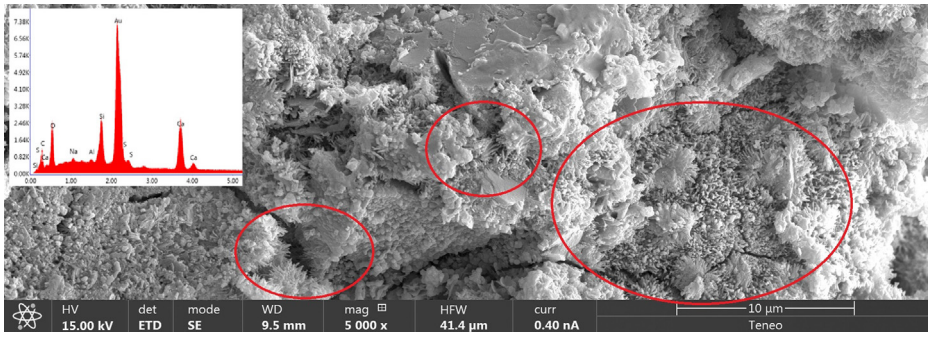

Fig. 3. M25 sample: Formation of sodium sulphate crystallizations and microcracks.

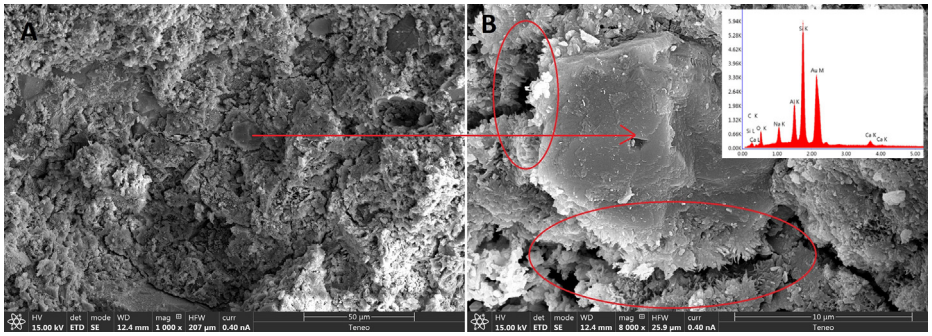

Fig. 4. Sample RM after a SC process: Generalized microcracking (4A) and acicular crystals (4B).

ettringite formation [42,43]. In any case, the attack of both could further accelerate the damage induced by volume expansion and microcracks [43].

The difference in morphology between the natural siliceous aggregates and the glass ones allowed to verify how the higher irregularity of the natural sand grains has favoured the degradation and consequent appearance of microcracks after the SC process (Fig. 5A). However, in the CGM specimens it has promoted a higher homogeneity in the ITZ with a better material behaviour (Fig. 5B).

Samples subjected to FC tests did not show appreciable differences between the reference samples (RM) and those containing glass (M10 and M25). In both cases, evident cracks have been observed in the matrix due to the expansive action of frozen water (Fig. 6A), also appreciating damage in the glass grains (Fig. 6B).

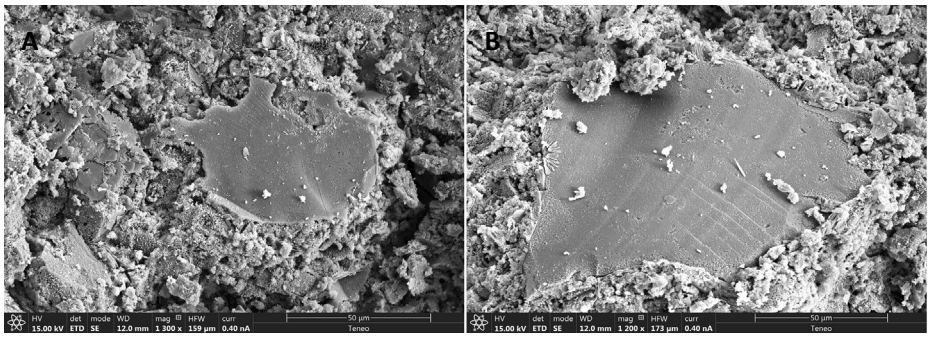

Fig. 5. M10 sample after a SC process: natural aggregate grain (5A) and glass aggregate (5B).

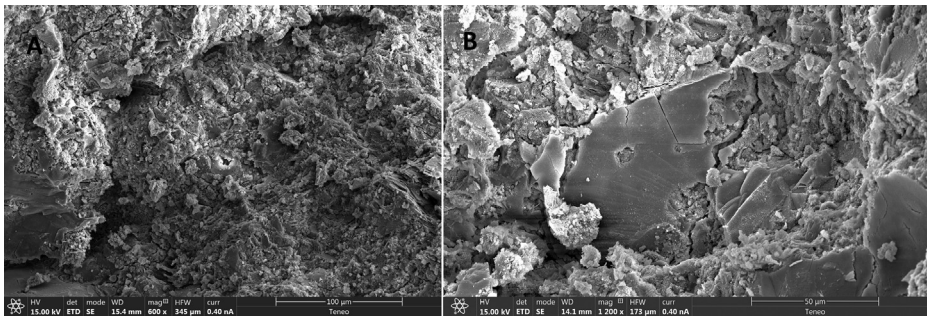

Fig. 6. RM and M25 samples after a FC process: Fissures in the matrix (6A) and glass grain (6B).

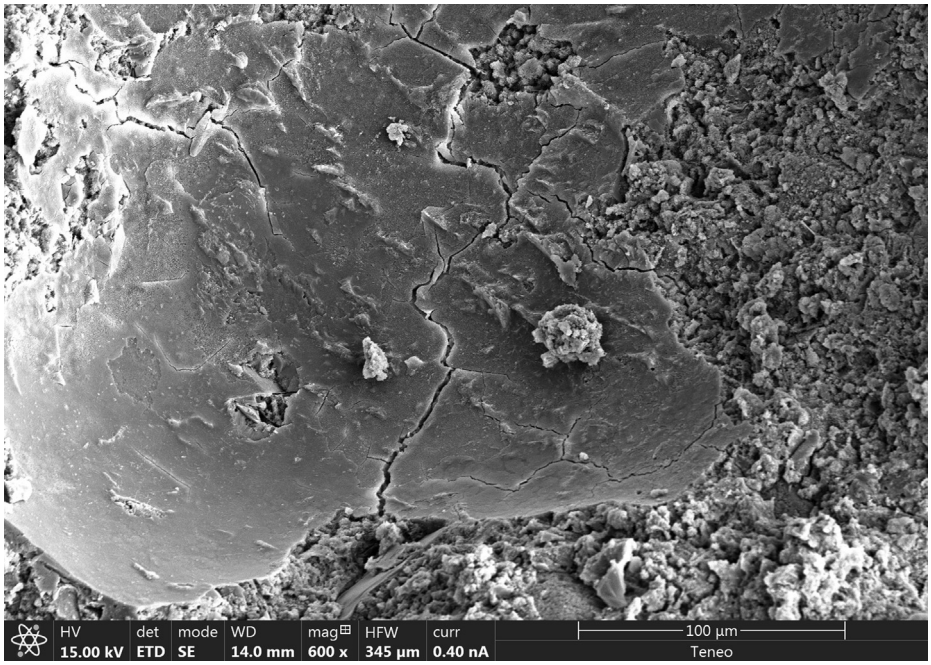

Fig. 7. RM sample after a TS process.

TS tests, in general, have produced a less noticeable effect on all samples. Only slight microcracks have been observed in the aggregates, with no apparent propagation to the matrix through the ITZ (Fig. 7).

5. Conclusions

The results obtained from the studied samples before and after performing ADT, showed a better behaviour of the specimens with glass aggregate, particularly in the case of SC test. Presenting an improvement to a lesser extent against the effect of the freezing and thawing cycles but being irrelevant when the specimens are subjected to thermal shock cycles. In no case a negative effect has been observed due to the addition of glass.

The compressive strength was clearly affected, and the incorporation of glass represents an evident improvement in the behaviour of the materials against the SC and FC tests. This effect can be associated with the greater regularity of the surfaces of the glass aggregates, which hinders the degradative action of the salts and the freezing of the water at the aggregate/paste interfaces. This positive effect occurs to a greater extent in samples subjected to salt crystallization as confirmed by the UPV test. Statistical analysis using the Student's *t*-test has allowed us to discriminate the significant phenomena from those that are not relevant in the analysis of physical and mechanical properties.

The observation by SEM of the interfaces of the aggregates allowed to verify a lower degree of microcracking in the ITZ of the glass aggregates. After the salt crystallization tests, a generalized and interconnected microcracking phenomenon was verified in the reference samples.

For all the above, it can be deduced that the incorporation of crushed glass can entail an effective modification in the composition of mortars subjected to aggressive environmental conditions, particularly in those cases in which they may be affected by the action of expansive salts.

CRediT authorship contribution statement

J.M. Alducin-Ochoa: Conceptualization, Investigation, Formal analysis. **J.J. Martín-del-Río:** Investigation, Methodology, Formal analysis. **M. Torres-González:** Investigation, Writing - review & editing. **V. Flores-Alés:** Conceptualization, Investigation, Writing - review & editing, Supervision. **D. Hernández-Cruz:** Methodology, Writing - review & editing, Supervision.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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