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Mussel shell mortars durability: Study of aggregate replacement limit

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ABSTRACT

The knowledge acquired through previous experimental phases with coating mortars with mussel shell aggregates led to conclude that irregular, flaky and hydrophobic particles of the mussel shell and the organic matter content introduce entrapped air and entrained air in the mixes. This phenomenon causes different and opposite effects on the main properties of mortars, which are in some way positive and negative (for durability), consequently, their durability cannot be easily predicted. The present work pretends to analyse the results of different durability tests, such as water vapour permeability, adhesive strength, and weathering cycles to recommend the maximum percentage of mussel shell sand used in coating mortars that guarantee the required lifespan.

After an in-depth literature review, it can be said that it is not easy to predict the durability of mortars using mussel shell aggregates. This question has been hardly analysed in the existing literature and the maximum substitution percentage of conventional aggregate that can be replaced is not clear. This work aims to answer this issue by analysing different properties: water vapour permeability, adhesive strength, and weathering cycles. Mussel shell content improves the water vapour permeability of both air lime and cement mortars but worsens the adhesive strength and weathering cycle behaviour. For most applications, 25 % of mussel shell aggregate can be employed, but for some applications, 50 % or even 75 % of mussel shell aggregate is feasible and will avoid the undesirable landfilling of this waste.

1. Introduction

Seashells are a waste of marine farming known to be produced in large volumes worldwide [1], there is a general interest in finding applications that would consume this waste and -if possible-give back value to the process. Mussel shells are one of the most produced types of seashells alongside oyster and cockle. Many studies show the applicability of these seashells as aggregate in concretes and mortars [2–6]. These seashells give some unexpected advantages to the mixtures for these applications: low capillarity and permeability, high porosity, and linked to this, low density and thermal insulation capacity [7–13], and also, a very aesthetic finish with seen fragments of nacre seashells [14,15]. Since, there are some drawbacks, due to the increase in porosity and the lack of adherence between paste-aggregate, it is needed to limit the percentage of seashells. Mortars and concretes with excessive quantities of seashells

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suffer severe mechanical strength. In this scenario, there is one application with high potential success: the coating mortars for building interiors. These products need aesthetic finishing and can benefit from the thermal insulation capacities that the seashell aggregates produce, in addition, they have low mechanical resistance requirements. This application could consume this waste and convert it into a valuable by-product in renovation works for renders and plasters in every type of building.

Seashells have been investigated for their use in mortars and concretes. Many authors focused on the use of the shells as substitution of aggregates, and others focused on the possibility of partial replacement of cement with shell fine powder. The durability of these materials is not analysed in deep and, on the contrary, many of these research works only introduce preliminary tests related to durability, such as adhesive strength, sulphates attack, chloride diffusion and shrinkage.

An exhaustive analysis of different studies on the use of seashell in mortars is presented in Table 1. Studies using seashells as aggregate in concretes were not included in this analysis. Only one of the 17 studies uses hydraulic lime as binder, the rest of them use cement. Eight of them use the shells as aggregate replacement in size below to 5 mm, and the rest of them use the milled shells as filler for the binder replacement, normally an Ordinary Portland Cement. Some of the studies included properties related with water effects, such as water permeability, capillarity and water absorption, which are indirectly related to durability and could be valuable indicators.

On the one hand, the studies that use seashells as filler for cement replacement indicated an increment in drying shrinkage with the increase of seashell powder content in mortars. The work of Liao et al. [16] reports that the water absorption coefficient, water permeability coefficient and chloride ion diffusion coefficient decreased with the increase of calcined oyster powder content. However, the results observed by Abdelouahed et al. [5] show that a high content of oyster powder as cement replacement promotes higher porosity and water absorption; this conclusion should be taken cautiously because the differences were very low

On the other hand, the works that use seashells as aggregate replacement, show a decrease in drying shrinkage in mortars [17]. Other authors [18] report an increase in water absorption of seashell mortars from their baseline. However, the study of Her-Yung Wang et al. [17] indicated a decrease in water absorption despite the use oyster shell as sand replacement. In this last case, the addition of fly ash with the oyster shell sand can explain this different behaviour, in contrast with the expectable increase of porosity

Table 1
Use of seashell aggregates in mortars.

Seashell species		Grain size	Recycling treatment	Ref.	Country	Binder	Studied properties
Sand (agg. replacement)	Filler (cem. replacement)						
Oyster		–	–	[20]	Korea	Cement	CS, ST
Oyster		–	105 °C	[21]	Korea	Cement	CS
	Mussel	<63 µm	Various (*)	[22]	Spain	Cement	CS, FS, consistency, adhesion
	Mussel, clam, cockle, oyster	0.5–40 µm	110 °C 24h	[23]	Thailand	Cement	Consistency, ST, CS, shrinkage, thermal conductivity
	Oyster		105 °C 24h	[24]	China	Cement	CS
	Periwinkle, oyster, snail	<63 µm	Washed, sundried, 800 °C for 4 h and ground	[25]	Nigeria	Cement	Consistency, CS, ST
Oyster		Fine sand	Washed, crushed, and ground	[17]	China	Cement, fly ash	Consistency, ST, CS, absorption, shrinkage
Oyster		0–5 mm	105 °C	[18]	Algeria	Cement	Consistency, density, absorption, porosity, CS, FS, modulus, SEM
	Oyster and mussel	Powder	18h oven dried	[26]	Philippines	Cement	Consistency, CS
Mussel		1.0 mm	Washed and dried	[19]	Morocco	Cement	Thermal behaviour, CS, chloride diffusion,
	Cockle	Powder	Clean, dried, crushed and ground	[27]	Algeria	Cement	Consistency, density, CS, FS, absorption, chloride penetration, weight loss
Oyster		0–5 mm	Crushed, sieved, washed and dried (100 °C 24h)	[28]	China	Cement, FA, GGBS	FS, CS, absorption, water permeability, chloride ion permeability, SEM
Sururú (mussel)		0–2.4 mm	Washed (water + liquid soap), sun dried, crushed, sieved	[29]	Brazil	Cement	Consistency, capillarity, CS, SEM,
Shell lime (oyster)		0–5 mm	Burnt and crushed	[30]	India	Hydraulic lime	CS
	Cockle	0–3 mm	Washed and crushed	[31]	Italy	Cement	CS, FS
	Oyster	ash	Calcined and ground (use as additive)	[32]	China	Clay-lime	CS, shear strength
	Oyster	<60 µm	Washed, dry, crushed and calcined (950 °C 2h), milled	[16]	China	Cement, metakaolin	Consistency, CS, FS, porosity, absorption, permeability, chloride ion diff.,shrinkage,

CS = compressive strength, FS = flexural strength, ST = setting time, SEM = microstructure analysis, UV = ultrasounds.
(*) Wash to remove salts + heat treatment to remove water and organic matter + ground.

due to oyster shell aggregate presence. In the work of Safi et al. [18], the authors report an increase -for self-compacting mortar-in porosity and water absorption with the increase in oyster shell aggregate replacement; with a limit, as these values decrease when 100 % of the seashell aggregate was used. The authors attribute this change in the trend to the irregular and flaky shape of seashells that break the fluidity of the mortar, and therefore, with different fresh-state properties the resulting pore structure is severely altered.

Regarding the evaluation of chloride ion penetration in seashells mortars, in general can be said that seashells could improve the mortar behaviour against the chloride penetration, but it depends on the exposure time, the age of the samples and the substitution percentage used. Ez-zaki et al. [19] report that mussel shell sand shows a lower apparent chloride diffusion coefficient than reference at 10 days of exposure, but higher when exposed for 30 days. So, there could be an initial beneficial effect that is attenuated with exposure time.

The last important effect in the durability are the physical properties of the shells: the fineness, the recycling treatment, and also the quantity of seashells replacement. The use as filler for cement replacement, when there are no variations in aggregate content, and replacement percentages are low, could promote higher compactness in the mortar mixture, and so improve some durability properties. The use as an aggregate replacement, with higher grain size, seems to increase mortars porosity and could negatively affect some durability properties. One additional research line of interest is the burning of seashells to obtain lime as a binder for mortars. This was an extended ancient technique used across the world [32–35], typically in areas where ground mineralogy did not offer an extractive alternative from the quarry. The process of calcination follows the same traditional limestone techniques with vertical discontinuous ovens. Some authors have analysed the effect of this type of limestone, looking for differences with conventional quarried limestone [36,37]. In general, the differences are minimal, however, some authors refer to the presence of unburned shell fragments after the processing. As these works are not fully comparable, they are not included in the comparative table.

The NW Spain and its shores shelter the largest European canning industry of mussel products. A few alimentary factories discard vast amounts of mussel shells every year (>20000 t/y) [38]. This waste is produced in a specific location, making possible an effective gathering and processing of discarded mussel shells. The authors of this work have worked previously with this type of shell by-product. In their work, they have analysed the use of these mussel shells as aggregate in concretes [8,11], in cement mortars [7], in lime mortars [9,10,12], and as loose-fill for acoustic and thermal insulation [14,15]. Derived from these studies various tests were carried out in coating mortars with partial replacement of mussel shell aggregates. These tests proved the feasibility of using crushed seashells as aggregates for elaborating plasters with cement and air lime. One opportunity was detected after these initial tests, the aesthetic of the mortar was rather appealing, so there could be a place in the market for a mortar with mussel shells. In addition, some properties of the resulting mortars were beneficial, water regulation and insulation capacities. The extension of the tests was limited and looking for an initial assessment of possibilities.

All this scientific work allowed us to conclude that the irregular, flaky and hydrophobic particles of the mussel shell and the organic matter content (polysaccharides) introduce entrapped air and entrained air in the mixes damaging the interfacial transition zone (Fig. 1). This causes a significant increase in porosity leading to a decrease in fresh and hardened densities [7,9,12] but also in the compressive and flexural strength of the mortars. Besides, mussel shell particles can act as a barrier to water, creating tortuosity paths showing a significant reduction in the capillary uptake of mortars [7,10]. Organic matter of mussel shell particles also reacts with some clinker components promoting a delay in cement hydration and causing an increase in the workable life of the mixes with mussel shell

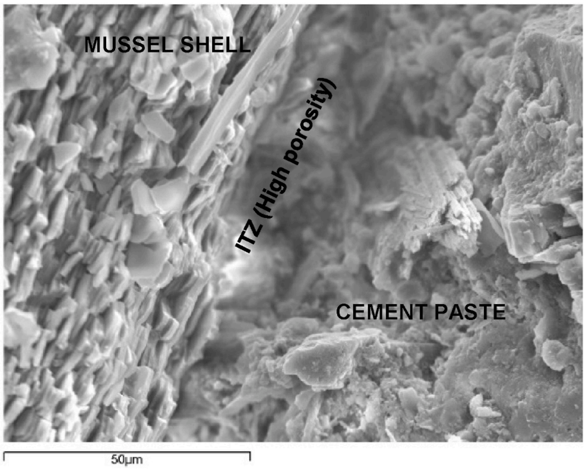

Fig. 1. Weak ITZ of mussel shell mortars.

aggregate [7]. In the particular case of mortars with non-hydraulic binders, this is, air lime, the effect of the organic matter is lesser than that observed in mixes with cement. Moreover, in this case, the mussel shell can act as a moisture retainer affecting the carbonation process of lime mortars, delaying the rate of carbonation at early ages but improving it after 180 days [9]. The mechanical strength values of lime mortars with mussel shells are not too far from the reference mortar with conventional aggregates [9,10,12]. According to these results, the authors decided to recommend using cement and lime mortars with a replacement ratio of up to 25 % –as a conservative recommendation–, because these mortars present similar performance to that of their baseline ones.

The results of the previous works with mussel shell aggregates are the foundation of this research, and some of the results regarding general properties are published separately [7–14]. However, it is considered useful to summarize them in this work so that the trends can be better understood and also make easier the discussion. Therefore Table 2, includes the most relevant information, including hardened density, porosity, water absorption, capillary uptake and mechanical strength. For air lime mortars, the carbonation state is also detailed.

Regarding the initial conclusions about durability, it was found that mussel shell aggregate content causes different and, to some extent, contradictory effects in mortars. On the one hand, it increases the porosity of the mixtures, making them more fragile, which, joined to the low paste-aggregate adherence, can be interpreted as expectable worsened durability [39]. However, the hydrophobic behaviour and irregular flaky shape of mussel shell particles promote low water absorption by capillarity in mortars and also low water penetration in mussel shell concretes [11], and this, on the contrary, could affect positively the durability aspect.

Therefore, the main objective of this present work is to assess the durability of mortars that include mussel shell aggregates. The parameters to be analysed would be water vapour permeability, adhesive strength, and weathering cycles. These experimental tests increase the knowledge of seashell mortars' behaviour and allow them to ensure their resistance over time. These mortars exhibit potential for application due to some beneficial properties such as aesthetic qualities and insulation capabilities. This combination could impact the markets and the industry making possible the commercialization –and consequent valorisation into by-products– of the large volumes of mussel shell wastes that are produced in the NW of Spain. This solution for this waste could be extended to other concentration points of seashells around the world. Finally, another aspect that needs to be reassessed is the maximum percentage of mussel shell sand used for coating mortars.

2. Materials and methods

2.1. Binders

In this work, three types of binders were used: masonry cement and two types of air lime. The cement used was a masonry cement MC12.5-X (without air entraining agent). The cement composition is OPC (clinker 41.3 %) and inorganic compounds (limestone 33.5 %, calcined natural pozzolana 19.8 %, gypsum 5.4 %).

The air limes used were a non-aged hydrated commercial lime powder (EN 459-1 CL90-S) and a slaked lime putty (EN 459-1 CL90-PL). Both were CL 90, with a 90 % minimum content of calcium and magnesium oxides. The lime putty was a commercial lime obtained by slaking quicklime with water and storing it for 6 months. The water content of the lime putty was 64 %. It is estimated that the lime putty used in this study was 10 months old. The non-aged lime powder was received, stored immediately and kept sealed until its use to avoid contact with atmospheric CO_2.

Table 2
Previous works' results of all the mortars studied.

	Age (days)	Density (kg/l)	Air content (%)	Water absorption (%) (*)	Porosity (%) (**)	Capillary coefficient (kg/m^2.min$^{0.5}$)	Compressive strength (MPa)	Flexural strength (MPa)	Carbonated area (%)
BC0	28	1.84	5.85	11.79	23.97	1.28	6.71	1.78	–
BC25		1.67	17.00	12.34	26.17	0.79	3.30	1.23	–
BC50		1.46	26.52	25.60	30.81	0.60	2.19	0.87	–
BC75		1.36	34.88	39.28	39.02	0.41	1.50	0.71	–
S0	90	1.77	4.00	14.59	28.65	1.71	1.94	1.23	63.30
S25		1.73	5.40	14.78	29.98	1.52	1.80	0.95	54.60
S50		1.66	8.50	19.01	32.27	1.34	1.69	1.04	54.10
S75		1.60	12.50	20.08	32.09	1.12	1.47	0.74	54.90
PL0	90	1.74	3.85	15.98	30.22	1.65	2.22	1.15	55.10
PL25		1.69	5.00	16.59	33.03	1.50	1.85	1.04	45.30
PL50		1.60	9.40	18.52	34.65	1.28	1.79	0.84	45.70
PL75		1.55	15.00	22.92	37.86	1.15	1.53	0.75	44.80

(*) after immersion and boiling.
(**) by mercury intrusion.

2.2. Aggregates

The limestone sand used comes from crushed limestone and it has a maximum size of 4 mm. Since its particle size distribution was excessively coarse for producing coating mortars, a size separation by sieving was performed into four different fractions: 0–0.063 mm, 0.063–0.25 mm, 0.25–1 mm, 1–4 mm. Then the size fractions were combined, resulting in suitable sand with a maximum size of 4 mm (LS). The sieve modulus of the resulting LS was 2.23, the particle density was 2.67 kg/l and the water absorption was 2.22 %. The main properties of this sand have been already published in previous works [7,12].

The mussel shell sand used was obtained from heat treatment (135 °C for 32 min [40]). Then it was crushed and sieved. This resulted in two different size fractions: coarse sand (CMS 0–4 mm) and fine sand (FMS 0–1 mm), with 2.65 kg/l and 2.73 kg/l as particle density respectively. These two fractions were combined to obtain a mussel shell sand (MS) with an equivalent particle size distribution to the limestone sand (LS), with a fineness modulus of 2.21. Mussel shell water absorption and organic matter content were 3.9 % and 2.1 % respectively.

2.3. Mortar mixes

Three series of mortars, one with cement, one with lime putty and the other with lime powder, were designed as a reference (Table 3). These mortars were designed to be used in a base layer. Each series was modified replacing, by volume, the limestone sand with mussel shell sand. The substitution rates used were 25 %, 50 % and 75 %. The mortar dosage of the base-layer cement mortar (BC) was designed with a cement to aggregate ratio of 1:5 (by volume) and a water to cement ratio of 1 (by weight).

In this work, the binder to aggregate ratio used for both non-aged lime mortars and lime putty mortars, was in the range of 1:2.5 to 1:2.3 (by volume). Thus, a water to lime ratio of 1.73 and 1.77, by weight, were used to design the mortars with hydrated lime and lime putty, respectively. As a result, twelve types of mortars were obtained, four with cement: BC0, BC25, BC50 and BC 75, four with non-aged lime powder: SL0, SL25, SL50 and SL75 and another four with lime putty: PL0, PL25, PL50 and PL75.

After chemical and physical characterization of binders and aggregates, the raw materials were mixed to obtain the various mortars. The mixing and moulding procedures were developed according to UNE-EN 196-1 [41] and UNE-EN 1015-11 [42] respectively, both detailed in previous works [7,12]. To assess the durability performance of the mortars different tests were carried out. The cement mortars were tested 28 days after the mixing process was done, and in the case of air lime mortars, durability tests were carried out at the age of 90 days.

2.4. Water vapour permeability

The water vapour permeability test was carried out according to UNE EN 1015-19. Testing samples were made in a cylindrical PVC mould of 150 mm diameter and 18 mm height based on a cellular concrete base covered with gauze. The samples were maintained in the climatic chamber (20 °C ± 2 °C and 65 % ± 5 %) until the testing age. Just before the test, mortar samples were introduced in a circular stainless-steel plate of 150 mm diameter. A fixed volume of a potassium nitrate solution was placed at the bottom of the plate to provide a high indoor humidity (>90 %). The volume was fixed so that the samples were separated from the solution by approximately 1 cm. The union between the steel mould and the specimen was sealed with petroleum jelly. The sealed samples were placed in the climatic chamber at 20 °C ± 2 °C and 65 % ± 5 % of relative humidity during the testing measurements. Samples were weighed every day until water vapour transfer is stable (that is when three consecutive measurements are on a straight line) (Fig. 2).

The water vapour resistance factor is a parameter that, considering $1.94\ 0.10^{-10}$ [kg/(s.m.Pa)] as the diffusion coefficient for water vapour in air at atmospheric pressure and 20 °C, can be calculated as follows:

$$\mu = \frac{1.94\ .\ 10^{-10}}{\delta_p} \tag{1}$$

Where δp [kg/(s.m.Pa)] is the water vapour permeability that can be assessed as:

$$\delta p = \bigwedge . \mathrm{d} \tag{2}$$

Being "d" the specimen thickness and "$\bigwedge$" the vapour permeance that is calculated as:

$$\bigwedge = \mathrm{m}/(A.\Delta p) \tag{3}$$

Where "Δp" is the vapour pressure gradient between the climatic chamber environment and the steel plate (Pa), A is the surface area (m^2), and m is the slope value of linear regression which reflects the water vapour flow (Kg/s).

Table 3
Baseline mortars dosages by weight (g) for 1 L.

	Cement	Hydrated lime/lime putty	Added water	Conventional aggregate	Water to binder	Paste to aggregate
BC0	312.0	–	312.0	1560.1	1	0.40
S0	–	207.4	358.0	1468.2	1.73	0.39
PL0	–	629.1	0	1270.3	1.50	0.50

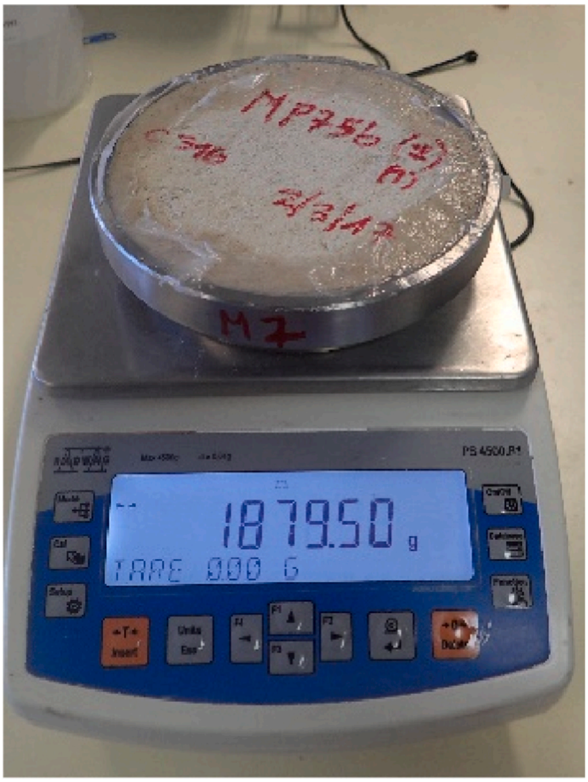

Fig. 2. Water vapour permeability test.

2.5. Adhesive strength and ultrasonic wave propagation

The adhesion of mortars to the background wall was determined according to the Standard UNE EN 1015-12. Mortars were applied on a ceramic substrate and the test was carried out with a pull-off tester Matest E142-01. It was determined the force needed to pull off a circular area of the rendering and then the tension that causes the rupture was calculated. At least 15 circles of each mortar type, with a diameter of 50 mm and a height of 10 mm, were tested as the scatter of this test is high.

As the pull-off test produces scattered results, an ultrasonic wave propagation test was performed on the same adhesion specimens just before the destructive test. Ultrasonic measurement equipment, Pundit Lab from Proceq, was used to perform the ultrasonic test in transmission reception mode. The frequency of both transducer couples was 150 Hz. Several measurements were made on each mortar sample applied on the ceramic substrate, on different parts of the sample. At least eight speeds for each type of mortar were registered (see Fig. 3).

2.6. Weathering cycles

This test was performed based on UNE EN 1015-21. For each type of mortar 3 prismatic specimens (160 × 40 × 40 mm) were subjected to two climatic cycles, whose effects are evaluated through mechanical strength. Both cycles included four steps. The first cycle is a Hot-Cold cycle where the Hot and Cold steps were separated by steps where samples were kept in a climatic chamber (CC) at ambient conditions for 30 min in each case. In the hot step, samples are heated with an infrared radiation lamp, and a temperature of 60 °C ± 2 °C is maintained on the surface for 8h ± 15min. In the cold step, samples are kept in a fridge at an air temperature of −15 °C for 15h. The second cycle is a Water-Cold cycle, and also in this case, the Water and Cold conditions were separated by CC ambient

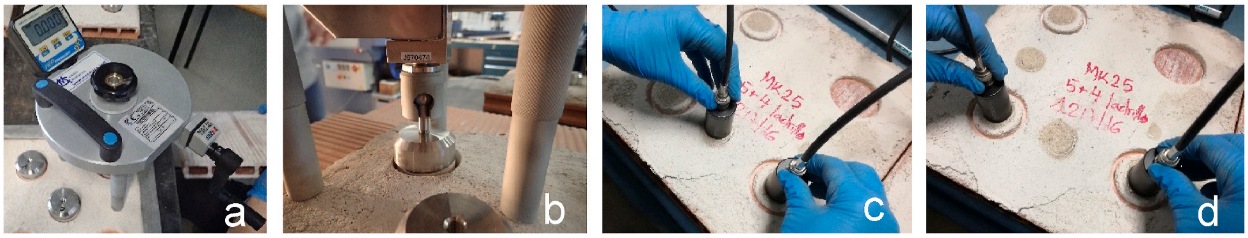

Fig. 3. Pull-off test (a, b) and ultrasonic wave propagation test (c, d) in mortars applied on the brick surface.

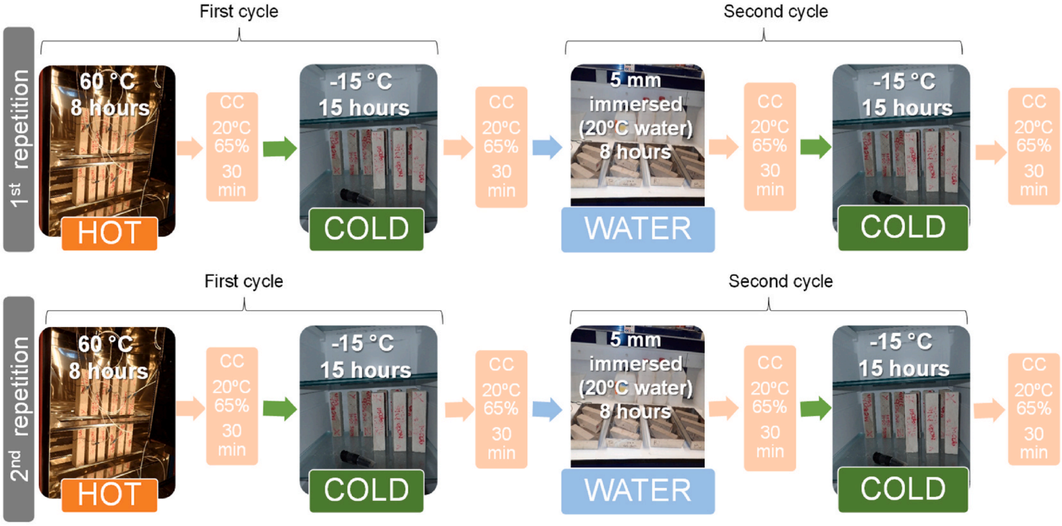

Fig. 4. Scheme of weathering cycles.

steps. In the water step, samples are partially immersed in water at 20 °C ± 2 °C to a depth of approximately 5 mm for 8 h ±15 min. The cold step is the same explained before. The timing was controlled with a data logger device. The cycles were repeated twice as shown in Fig. 4. The time between the two repetitions were 24 h.

Besides, the compressive strength test was carried out on prismatic specimens 160 × 40 × 40 mm using a universal multi-purpose compression/flexural machine, MAETEST S205 N Unitronic 50 Kn with Cyber-Plus evolution control. Specimens were tested according to UNE EN 1015-11. Firstly, the flexural test was performed by breaking the specimen into two halves, and afterwards, over each half, the compressive strength test was developed. The loading rate selected in the flexural test was 0.02 kN/s and in the compressive strength test was 0.15 kN/s.

3. Results and discussion

3.1. Water vapour permeability

Fig. 5 shows the results of the water vapour permeability test of all mortars. The use of mussel shell aggregate significantly increases the water vapour permeability of all coating mortars, and so, decreases the water vapour resistant factor (Figs. 6 and 7).

The baseline cement mortar shows a value of 14.7 [ng/(m.s.Pa)], which is similar to that shown by other authors [43]. Air lime, hydrated and putty, reference mortars show higher values than cement ones, in a range of 21–29 [ng/(m.s.Pa)], which is also similar to values found in the literature [44].

Mussel shell content in cement mortars leads to values in a range of 40–60 [ng/(m.s.Pa)], which are similar to mortars with lightweight aggregates [45] that are considered thermal correctors and hygric regulators [46].

Hydrated lime mortars show the highest values of water vapour permeability for all substitution percentages. However, the variation shown in S75 respect S0 (300 %) is nearly the same as that shown by BC75 respect BC0 (311 %). Air lime mortars variations are more noticeable from the 25 % of substitution. Accordingly, the lowest water vapour resistant factors (Figs. 6 and 7) are presented

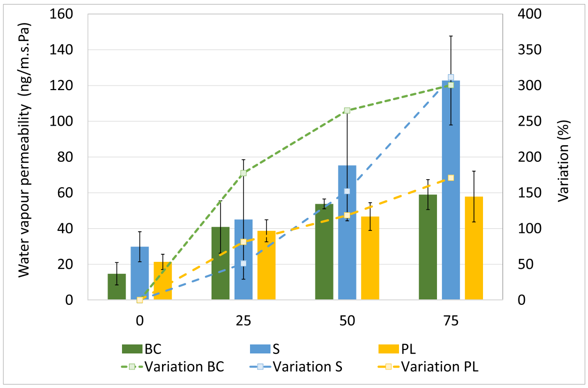

Fig. 5. Water vapour permeability.

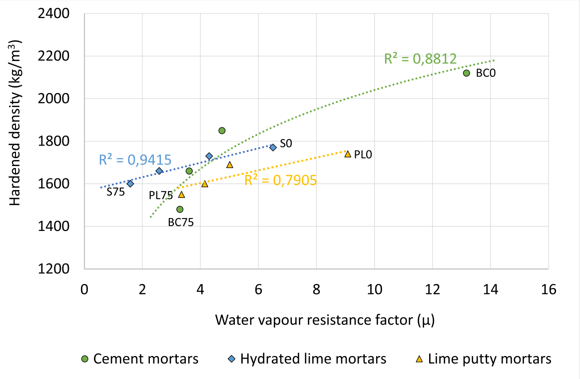

Fig. 6. Water vapour resistance factor vs hardened density.

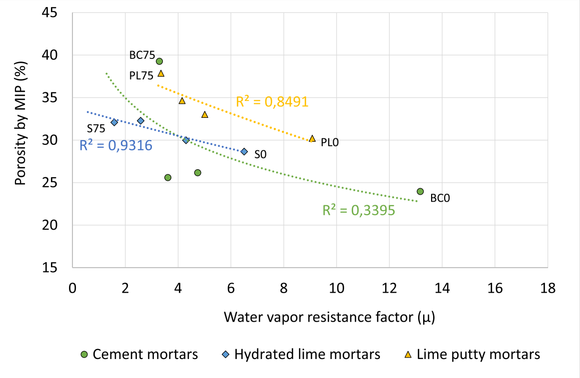

Fig. 7. Total porosity by MIP vs water vapour resistance factor.

by S50 and S75, with values of 2.6 and 1.6 respectively. These values are considered very low and are similar to those presented by natural and recycled insulation materials [47] such as kenaf, wood fibre, hemp shiv composite, textile waste, etc.

Water vapour permeability governs the movement of water vapour in porous materials which allows the material to “breathe”, and in this sense, the mortar helps to dry out the substrate, where the liquid water has evaporated [48]. As it was stated by Thomson et al. [49], moisture in pores occurs as adsorbed vapour on the surface and as condensed liquid, partially or completely filling pores. Transport of moisture within the pores of mortar occurs only within interconnected pores. This is permeability. In the water vapour permeability test, relative humidity higher than 90 % is provided, so moisture transfer through vapour diffusion is governed by a difference in vapour pressure, once the steady state is reached.

Mussel shell aggregate promotes an increase in the total porosity of all mortars and so a decrease in their hardened density with both types of binders: cement and air lime. This increase is caused by both flaky shell particles and their organic matter content

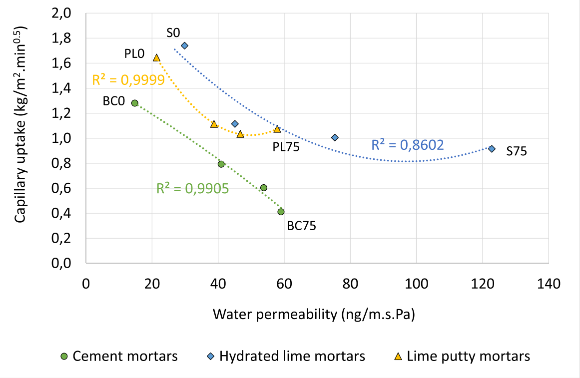

Fig. 8. Capillary uptake vs water vapour permeability.

introducing entrapped and entrained air in the mixes, respectively. This increase in porosity can explain the increase in the water vapour permeability, or the decrease in the water vapour resistance factor (Figs. 6 and 7).

The water vapour permeability phenomenon is also linked to water absorption by capillary uptake. Both properties usually show the same trend, water vapour permeability increases or decreases as the capillary uptake increases or decreases, respectively, as the latter is closely related to the pore volume. However, mussel shell incorporation causes a decrease in capillary uptake despite an increase in the pore volume takes place. Hence, in mortars with significant mussel shell aggregate content, the water vapour permeability and the capillary uptake follow opposite trends (Fig. 8). When the mussel shell aggregate is used, the mortar microstructure changes. Less small capillary pores (0.1–1 μm) are created and, on the contrary, the volume of coarse pores (>1 μm) increases [12]. This is more noticeable in hydrated lime [12] mortars than in lime putty or cement mortars and explains the high values of water vapour permeability obtained with these mortars. In addition, flaky and hydrophobic mussel shell particles create tortuosity paths for water, preventing water migration through the capillary pores [7,9–11], which explains the reduction in the capillary uptake that these mortars show.

The mussel shell creates a more porous system in the cement and lime mortar matrix that facilitates the water vapour diffusion process through the structure. The porous system of mussel shell coatings and the existence of channels facilitates the water vapour diffusion process through the structure [50]. This singular porous matrix, promoted by mussel shell content, gives cement and air lime mortars an interesting behaviour against moisture for coating mortar application.

3.2. Adhesive strength and ultrasonic wave propagation

All mortars were tested to determine adhesive strength to a ceramic substrate. Besides the numerical result obtained, the failure mode (Fig. 9) was also registered. According to the standard (UNE EN 1015-12), when adhesive failure type occurs (failure at adhesion interfaces between the mortar and the ceramic base), the result obtained corresponds to the adhesive strength. In case a cohesive failure mode happens (failure in the substrate material or in the mortar itself), the adhesive strength is higher than the stress calculated with the failure load obtained in the test. Finally, a mixed failure mode can occur when, in the same test, both previous failure modes take place. In this case the mortar partially losses the bond to the substrate and partially breaks itself or breaks the ceramic substrate. Mixed failure modes are not considered in the standard, so in this study, this failure type is assumed as a cohesive failure. Fig. 10 shows the possible failure modes when the pull-off test is developed.

The occurrence of each failure mode in each case was calculated and the results are shown in Fig. 11. In general, all cement mortars show a greater number of adhesive failure modes, and lime mortars show a greater occurrence of cohesive or mixed failure types. Regarding the influence of the mussel shell content, in the case of cement mortars, it can be seen that the presence of a high mussel shell content, as is the case of BC75, changes the trend from adhesive failure to cohesive failure mode. In all the rest of the mortars (cement or lime-based mortars) the incorporation of mussel shell does not affect the failure type.

Fig. 12 shows the average value of the pull-off test for each mortar. The average value was calculated using the results that provided adhesive failure mode for the cement mortars, and cohesive failure mode for all the air lime mortars, even so, the scatter in this test is high. Thus, the results obtained in all cement mortars show the adhesive strength values, while in the case of lime mortars, the pull-off strength represents the minimum threshold of their adhesive strength.

In air lime mortars, both hydrated and putty, the values are in a range between 0.05 and 0.10 MPa. In the case of cement mortars, as expected, the values are higher, being in a range between 0,18 and 0,097 MPa. Despite the scatter shown by this test, in all cases, an increase in the mussel shell aggregate content leads to a decrease in the adhesive strength. In the case of lime mortars, the effect of using mussel aggregate is slight and shows the same trend as the flexural strength [9,12], related to the tensile strength that, in the case of cohesive failure mode, is controlling this failure type. On the contrary, in the case of cement mortars, the incorporation of mussel shell is significant, especially from the 25 % replacement percentage.

The worse bond between the paste and the aggregate observed in mussel shell mortars (Fig. 1), joint to the increase in the porosity caused by entrapped air, might be promoting the lack of adhesion to the substrate. In cement mortars, both these issues joint to the increase in the porosity caused by entrained air, due to the organic matter of mussel shell particles (that affects more in cement matrix than in air-lime matrix) rise the influence. These results are consistent with the mechanical strength of mortars in previous results [7].

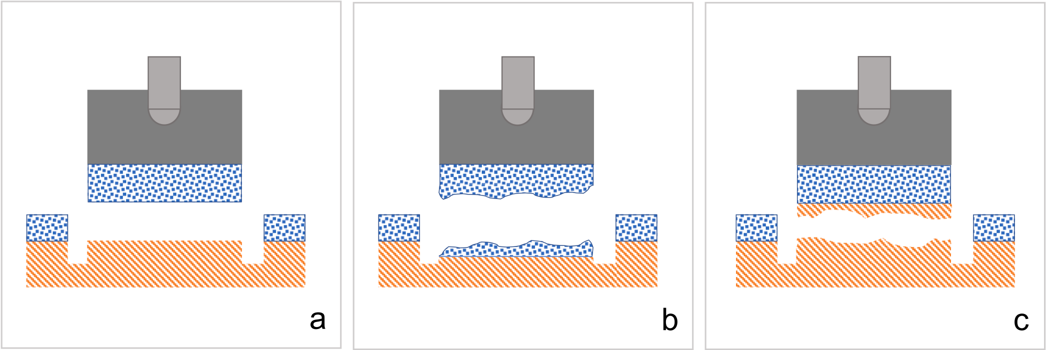

Fig. 9. Type of fracture (UNE EN 1015-12): a) Adhesive between the mortar and the substrate, b) Cohesive within the mortar; c) Cohesive in the substrate.

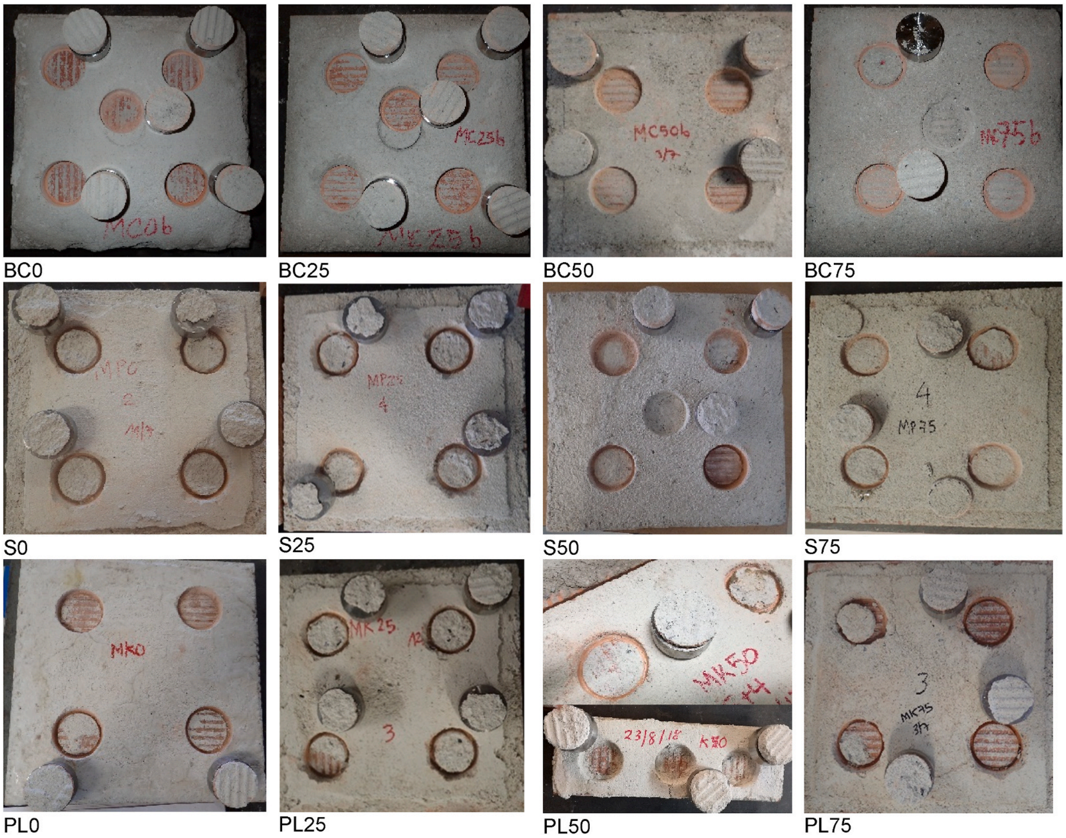

Fig. 10. Pull-out test images of each type of mortar.

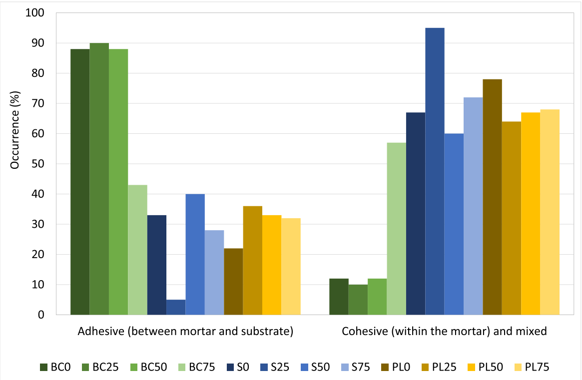

Fig. 11. Type of fracture in pull-out test, percentage of occurrence in each type of mortar.

The results obtained in this study from the pull-off test in cement mortars are not easily comparable to other published studies. The works that study the adhesive strength of cement mortar with seashells as aggregate are scarce.

Ballester et al. [22] develop pull-off tests to measure adhesion strength, but the mixture design is very different from the mortar mix used in this study, so the results cannot be compared. They employ mussel shell as a powder material and find higher values of adhesive strength (0.43, 0.44 and 0.47 MPa) and also a trend that is opposite to the tendency found in this study, the more mussel shell content the higher the adhesive strength. The use of a high strength cement (CEM II AV 42,5R), a fine size fraction of calcined and ground mussel shell (lower than 40 μm), and also a low substitution percentage of the conventional aggregate (4, 8 and 14 %) can explain these differences.

Other works that measure adhesive strength in masonry or coating mortars partially replacing the conventional aggregate with other recycled aggregates have been found [43]. In all these cases the use of a high strength cement (instead of masonry cement, as is

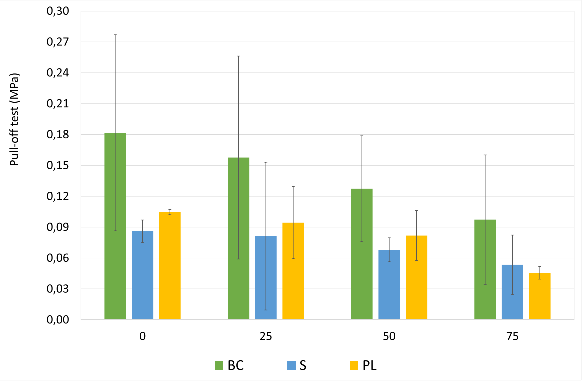

Fig. 12. Pull-off test results of mortars.

the case of the present study) outcomes in higher values of adhesive strength than those obtained in this study.

Regarding air lime mortars, there are no works that employ seashells as aggregates. Branco et al. [51], using conventional aggregates, obtain values of adhesive strength in air lime and lime putty mortars between 0,07 and 0,14 MPa at 90 days of age, detecting, in all cases, a cohesive failure mode. Other works that analyse the adhesive strength of lime mortars with different pozzolanic additions [52] show values ranging between 0.01 and 0.13 MPa (similar to the ones obtained in this work). Again, in both studies, the cohesive failure mode was identified as the main type of failure. In the work of Veiga et al. [53] it is stated that an adhesive strength at 90 days-old in the range of 0.1 and 0.3, or cohesive failure mode must be got (it is required) in rendering and repointing substitution mortars for ancient buildings.

As the pull-off test provides a high scatter, in this work, it was decided to combine it with another testing method that could reduce the uncertainty and confirm the trend shown when mussel shell aggregate is employed. It was decided to carry out a non-destructive test such as the ultrasound wave propagation test. The results obtained are shown in Fig. 13. It is seen that the trend in all mortars is similar to that observed with the pull-off test. The use of mussel shell aggregate in cement and air lime mortars produces a decrease in ultrasound pulse velocity. As in the adhesion strength, the effect of mussel shell content is much more noticeable in cement mortars than in air limes mortars. The lower density due to the high porosity caused by mussel shell aggregate can explain the results. Again, an increase in the porosity of air lime mortars due to mussel shell content can explain the decrease in ultrasound pulse velocity.

Fig. 14 shows the relationship between the adhesive strength and the ultrasound pulse velocity of all mortars. All series show a very clear correlation between both results, and a straight line can be drawn in all cases. The adhesive strength is directly related to the pulse velocity obtained in the ultrasound wave propagation test. Therefore, although the ultrasound test also introduces some scatter, as it can be developed easily on site and as many times as required, it is a good complement to the pull-off test.

The ultrasound analysis in mortars with seashells as aggregates were also developed by a few researchers. Etuk et al. [54] employ oyster, periwinkle and snail shells calcined and ground as cement replacement powder in concretes. Therefore, due to the use of coarse aggregate, the ultrasound velocity values obtained were higher than those got in this study. However, they also observed a decrease in the ultrasound pulse velocity when the content in seashells increased, which was explained by the low density and highly porous matrix of the samples with seashells. Kumar et al. [55] analyse the performance of cement mortars with granite powder. The results they got in the ultrasound pulse velocity test are similar to the ones shown by the reference mortars of the present study. However, mussel shell mortars show lower values than those that contain granite powder.

Regarding lime mortars, again, no studies have been found that incorporate seashells in this type of mortars. Branco et al. [51]

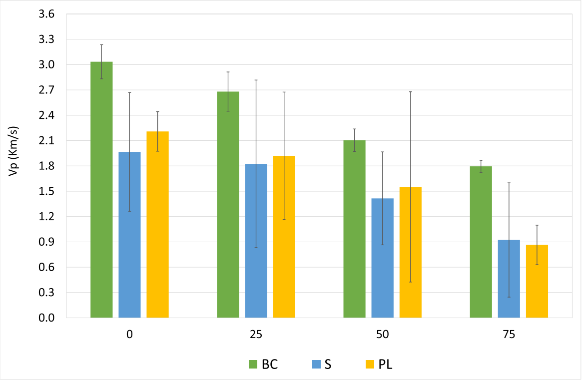

Fig. 13. Ultrasonic wave propagation of mortars.

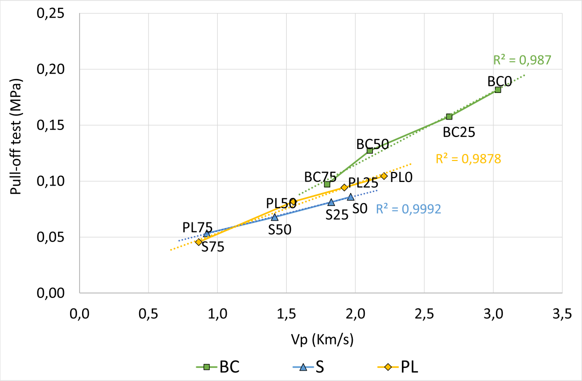

Fig. 14. Pull-off test results vs. ultrasonic velocity.

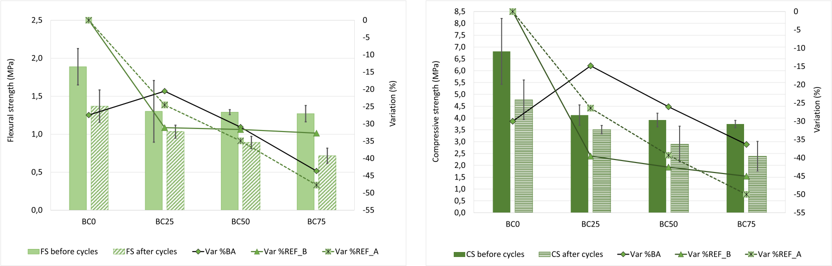

Fig. 15. Flexural and compressive strength of cement mortars before and after cycles.

obtain values of ultrasound pulse velocity of different air lime mortars with conventional aggregate in the range of 1700–2300 m/s, which agrees with the values of S0 and PL0.

3.3. *Weathering cycles results*

The flexural and compressive strength of cement and lime mortars after weathering cycles are shown in Fig. 15, Figs. 16 and 17. The values of the mechanical strength at the same age before the cycles are also represented to analyse variations. The analysis has been

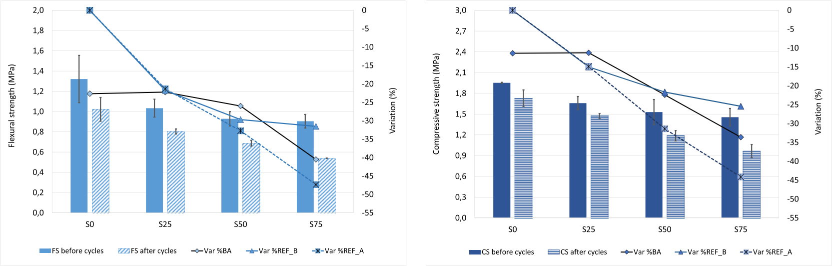

Fig. 16. Flexural and compressive strength of hydrated lime mortars before and after cycles.

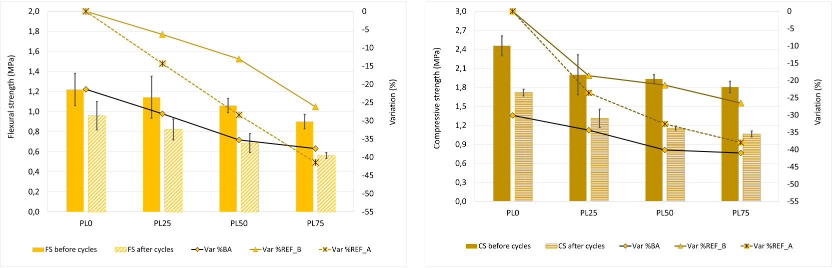

Fig. 17. Flexural and compressive strength of lime putty mortars before and after cycles.

developed in two different manners:

i) The percentage of variation (%Δ) in the strength before (B) and after (A) weathering cycles of each mortar (%ΔBA).
ii) The percentage of variations in the strength regarding the reference mortar (without mussel shells). This has been calculated with the strength before the weathering cycles (%ΔREF_B) and after the weathering cycles (%ΔREF_A).

As it is observed, the effect of weathering cycles in both flexural and compressive strength is negative in all mortars, promoting a decrease in the mechanical resistance values.

Analysing the variation due to the weathering cycles (%ΔBA) in cement-based mortars, it is observed that BC25 shows lower decreases in both flexural and compressive strength than the reference mortar (BC0). BC50 and BC75 mortars exhibit, however, higher variations than BC0, being the values of 30 % and 45 % respectively. When differences in the strength values regarding the reference mortar are studied (before, %ΔREF_B, and after, %ΔREF_A, cycles), it is seen that BC25 strengths after cycles have declined lower than before cycles. However, from 50 % substitution onwards, the mechanical strength loss is higher after than before the weathering cycles.

Regarding hydrated lime mortars, the variation due to the weathering cycles (%ΔBA) are similar in S0 and S25. S50 and S75 mortars display, nonetheless, higher variations (around 30 and 50 %). The comparison of the strengths shown by the mussel mortars regarding the ones displayed by the reference mortar before and after cycles shows that S25 presents the same percentage of variation, no matter if it has been subject to cycles or not. On the contrary, S50 and S75 show higher declines when weathering cycles have been applied than before the cycles.

The results obtained with lime putty mortars show that all mussel mortars present higher variations than the reference one, and this variation is higher as the mussel aggregate content rises. Moreover, in this case, all mussel mortars exhibit higher declines in strengths (when compared to the strengths of the reference mortar) after weathering cycles than before them.

Many studies have confirmed that porosity is highly related to mortars' durability; Lanas et al. [56] conclude that the deterioration due to freeze–thaw cycles is more noticeable in mortars with high porosity and low strength. According to Török et al. [57] there is a strong relationship between porosity and frost behaviour. Other authors [58] classified the pores accordingly to their effects on the freeze/thaw resistance: harmless (up to 0.02 m), less harmful (0.02–0.05 m), harmful (0.05–0.2 m), and more harmful (larger than 0.2 m). This classification agrees with other studies [49,58] where is stated that capillary pores are responsible for the mortar damage when subjected to wet and dry cycles. Netinger et al. [58] concluded that the most important factor affecting durability is the pore connectivity among macropores. It is also well known [58,59] that the addition of an air-entraining agent improves both cement and lime-based mortar's resistance to freeze-thaw cycles.

Reviewing the literature, it is seen that the research works that deal with the study of the effect of weathering cycles or freeze-thaw cycles on mortars with seashells as aggregate are scarce. Nguyen et al. [60,61] measured the number of freeze-thaw cycles that have to be applied before failure in a pervious concrete with seashells. They conclude that the seashell concrete presents a weaker performance against freeze/thaw attacks than the baseline concrete. The cycles introduce tensile stresses in the specimens, and the seashell concrete behaves worse under this kind of stress than the baseline concrete. It was also suggested, that the chloride and organic matter content of the seashells might generate a negative interaction with the cement matrix causing a rapid harmful effect.

The behaviour of the mussel shell mortars after weathering cycles can be explained by the paste-aggregate bond and the pore size distribution.

Regarding paste-aggregate bond, the mussel shell particles show a weak ITZ with both air lime and cement matrix. The flaky shape and the smooth surface of mussel particles joined to the high porosity introduced in the ITZ by the activity of the organic matter (especially in cement-based mortars) are the main issues that justify this weak interface ([7,12]).

Regarding porosity, it has been previously proved that mussel shell incorporation reduces capillary pores (<5 μm) in cement mortars [7] and in air-lime mortars [12]. However, the porosity, in the range of 5–200 μm, shown in Fig. 18 and measured by MIP, is higher in mussel mortars (both cement-based and lime-based ones) than in the baseline coatings. To analyse the pores larger than 200 μm, SEM images are used. Fig. 19 shows that large pores, with a diameter larger than 400 μm, can be clearly seen in the mussel shell

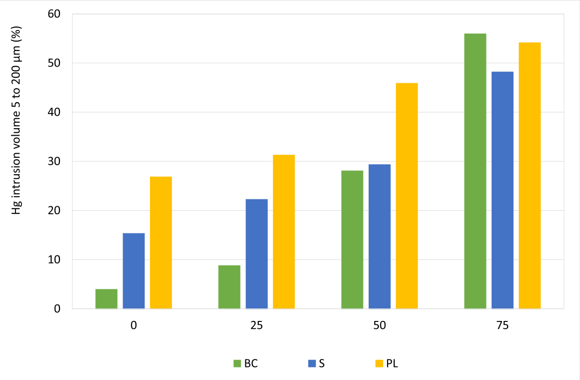

Fig. 18. Volume of pores in the range of 5–200 μm measured by MIP (at 28 days for cement mortars and 365 days for air-lime mortars).

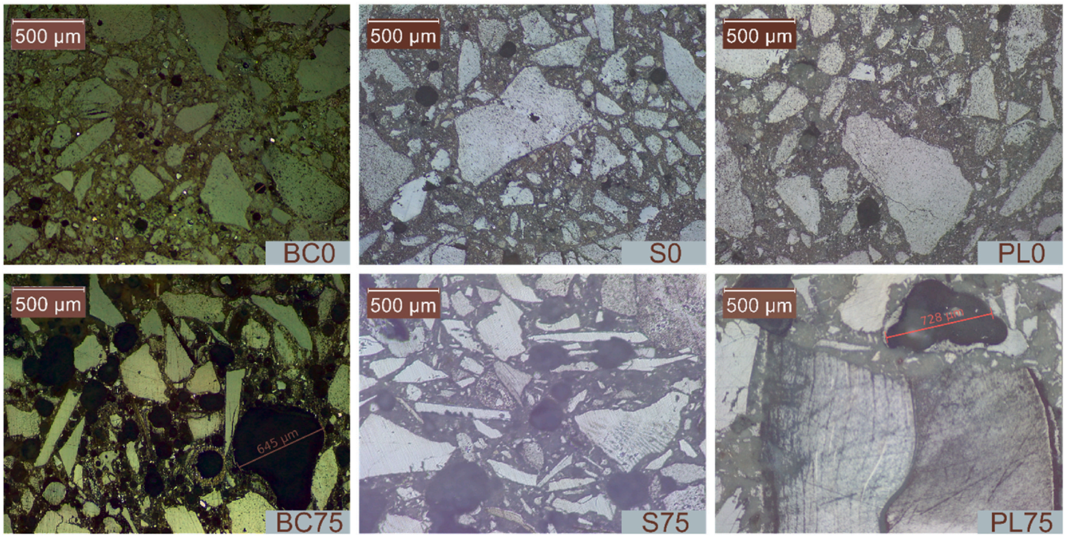

Fig. 19. SEM images of each baseline mortars (first row), and of mortars with 75 % of mussel (second row).

samples with 75 % of mussel shell aggregate. These large pores are not observed in the reference mortars.

In previous works, it was also seen that mussel shell aggregate incorporation reduces water absorption by capillarity, and also promotes water retention in mortars [7,9,10,12]. These facts lead to consider that the flaky hydrophobic mussel shell particles produce a macropore structure poorly interconnected. Therefore, it is likely that during the weathering cycles, the water remains trapped in these pores generating tensions that promote cracking, weakening the mortars.

It can be then concluded the highly porous structure (with a high volume of large pores poorly interconnected), the fragility of the shells and the weak paste-aggregate bond facilitates the development of microcracking in mussel mortars under the stresses caused by changing the weathering conditions (temperature and humidity) during cycles. In general, when mussel shell content increases the behaviour of mussel mortars under weathering cycles worsens. However, when low replacement percentages of mussel shells are used, the porosity introduced by the mussel particles may be beneficial, especially in cement-based mortars where the organic matter content (chitin) highly acts as an air-entraining agent [7,9,10,12]. This can explain why the BC25 shows less strength reduction than the baseline mortar after cycles and the S25 exhibits similar reductions to the S0.

4. Regulatory framework of coating mortars: classification and recommendations

The assessment of the seashell mortars durability should include the key parameters that need to be measured for conventional coating mortars. In the end, the intention is that when the scientific background is sound enough, the market will start to offer this type of seashell mortar. In a preliminary scientific study, it is not necessary to ensure the full extent of the tests, however some of the tests are a reliable reference that is accepted by the market agents, so it is important to exhibit results according to the mortar requirements.

Concerning the specifications for rendering and plastering mortars they are collected in the standard (UNE-EN 998-1 [62]). According to this standard, coating mortars are classified into different groups depending on their applications:

- GP, mortars for ordinary use.
- LW, lightweight mortars
- CR: coloured mortars (with pigments or coloured aggregates).
- OC: mortars for one-coat application
- R: renovation mortars.

Table 4
Coating mortars classification according to their properties (UNE-EN 998-1).

	Class	Value
Compressive strength range at 28 days	CS I	0.4–2.5 MPa
	CS II	1.5–5.0 MPa
	CS III	3.5–7.5 MPa
	CS IV	≥6 MPa
Capillary coefficient	W 0	Do not specified
	W 1	$c \leq 0.4$ kg/m^2.min$^{0.5}$
	W 2	$c \leq 0.2$ kg/m^2.min$^{0.5}$
Thermal conductivity	T 1	≤0.1 W/m.K
	T 2	≤0.2 W/m.K

Table 5
Mussel shell mortars classification according to UNE EN 988-1.

Mortar type	Compressive strength (MPa)	Thermal conductivity (*) (W/m.K)	Capillary coefficient ($kg/m^2.min^{0.5}$)	Water vapour resistance factor (μ)
BC25	CS II	>0.89	W0	4.7
BC50	CS II	>0.66		3.6
BC75	CS II	>0.49	W1	3.3
S25	CS I or CS II	0.66–0.82	W0	4.3
S50	CS I or CS II	>0.66		2.6
S75	CS I	0.61–0.66		1.6
PL25	CS I or CS II	0.66–0.82	W0	5.0
PL50	CS I or CS II	0.61–0.66		4.2
PL75	CS I	0.49–0.61		3.4

(*) Estimated values according to the density (UNE EN 1745, Table A.12).

- T: mortars for thermal insulation.

The standard establishes different requirements for mortars properties according to each of these applications. The main ones are used to define different mortar classes (Table 4).

With the results obtained in this study and in the previous works developed by the authors, mussel shell mortars can be classified as it is shown in Table 5. This classification enables the mussel shell mortars to fulfil the requirements for different applications (according to UNE EN 988-1). Thus, all mussel shell mortars, with cement or air lime as a binder, can be used as a mortar for ordinary use GP and as coloured mortars, if pigments are added to the mixture. Despite none of them can be used as a lightweight or thermal mortar, it can be considered that all air lime mortars with mussel shells could be applied as renovation mortars, as they do not release soluble salts, present high porosity and water vapour permeability, and also low capillarity.

On the other hand, the European Assessment Document for the External Thermal Insulation Composite System (ETICS) with renderings [63] states that a base coating for thermal insulation purposes has to achieve a bond strength value of at least 80 kPa (0.08 MPa) showing cohesive or adhesive failure mode. According to this, it can be stated that all mussel shell mortars with cement as a binder, and air lime and lime putty mortars with 25 % and 50 % of mussel shell as aggregate, can be used as mortar coating for ETICS.

5. Conclusions

In this work, water vapour permeability, adhesive strength, ultrasound pulse velocity and weathering cycles were carried out to analyse durability behaviour. The main objective is to provide recommendations about the maximum percentage of mussel shell sand that could be used in coating mortars to ensure their lifespan. The results obtained can be summarized as follows:

- The use of mussel shell aggregate significantly increases the water vapour permeability of all coating mortars, and so, decreases the water vapour resistance factor. This is beneficial as it promotes surface condensation control.
- An increase in the content of mussel shell aggregate means a loss in the pull-off strength (related to adhesive strength) and a decrease in the ultrasound pulse velocity. Despite this fact, all mussel shell mortars with cement, and mussel mortars with air lime and lime putty that incorporate 25 % mussel shell fulfil the requirements of adhesive strength established by ETICS (higher than 0.08 MPa).
- The effect of weathering cycles on both flexural and compressive strength is negative in all baseline and mussel shell mortars. The incorporation of a high content of mussel shell decreases the mechanical resistance after cycles in a higher extent than in the baseline mortars. However, when a low mussel shell percentage is used (25 %) the effect of the weathering cycles is similar or even less negative than in the baseline mortars.

The employment of a 25 % of mussel shell replacing conventional aggregates, provides coating mortars with properties that fulfil the requirements stipulated in the standard for most of the applications (but for thermal or lightweight mortars). If most of the commercial ready-mixed mortars incorporate this percentage, the environmental problem associated with landfilled mussel shells could be reduced.

The results obtained in this work also point out that the use of mussel shells in other specific applications as the design of plasters (interior walls) can be a good option, especially as in this application the technical requirements are not so demanding, which probably enables for higher replacement percentages (50 % in lime base mortars an even 75 % in cement-based mortars). The incorporation of high percentages of mussel shells in finish coatings (plaster and renders) could also be possible when some type of fine finish treatment (burnished, polished, honed …) is applied. Finally, the use of mussel shells in heritage is another further research that is opened with this work.

Author statement

Martínez-García, Carolina: Methodology, Validation, Formal analysis, Investigation, Writing – original draft, Writing – review & editing. **González-Fonteboa, Belén**: Validation, Formal analysis, Investigation, Supervision. **Carro-López, Diego**: Validation, Formal analysis, Investigation, Supervision. **Martínez-Abella, Fernando**: Validation, Formal analysis, Investigation, Supervision.

Declaration of competing interest

The authors declare that they have no conflict of interest.

Data availability

Data will be made available on request.

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