Development of Artificial Ageing Tests for Renders – Application to Conservation Mortars

by

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ABSTRACT
Renders are an extremely vulnerable part of constructions as they are constantly subject to atmospheric aggression. Although they act as sacrificial layers, over underlying walls and must have maintenance procedures, they must satisfy minimum durability requirements. In order to ensure that these requirements are fulfilled, artificial ageing tests are usually performed, simulating extreme conditions, both climatic and others. Inserted in a study of conservation mortars with pozzolanic additives, artificial ageing tests were developed on the basis of flexibility and adaptation to the national climate. A rain/heat cycle and a freeze/thaw cycle were performed and the state of mortars was assessed after each cycle by both visual evaluation and weighing. Water permeability and pull-off tests were performed on specimens with no severe degradation. Additional specimens were prepared and placed alternatively in a sodium chloride solution and in a conditioned atmosphere and mass alterations were registered.

1. INTRODUCTION

Rendered walls belonging to old buildings frequently need intervention due to more or less extensive degradation of renders. For this purpose, mortars must be developed that satisfy both compatibility requirements with walls and possible remaining mortars and minimum durability requisites. Mortars based on aerial lime and certain pozzolans usually have characteristics that satisfy mechanical and physical compatibility [1-2] and are therefore a possible recommendation for use in conservation situations. Furthermore, there is possibility for an easier workmanship, due to the fact that thicker renders can be used as they suffer both carbonation and pozzolanic reaction, allowing for complete hardening, even in the innermost layers. This avoids the execution of thin layers that must carbonate before the placement of a further layer, a process that takes longer and demands specialized working abilities.

However, the increasing need for durable solutions, implying less maintenance, demands that rendering mortars withstand external actions without severe degradation. These actions can be divided into climatic actions, atmospheric pollution and the action of soluble salts, either by capillary action or by direct exposure (marine environment, direct contact with water, direct contact with salt-containing materials). For the purpose of evaluating the impact caused by these destructive agents, artificial ageing tests are usually performed. A series of standards and recommendations [3-6] have been published for theses tests, mainly relating to concrete or single-coat rendering mortars. Moreover, other testing methods have been developed throughout the world, sometimes taking into account specific climatic conditions that occur in several countries [7-9]. It is on the basis of a flexible test that must be performed taking into account application conditions, that a further artificial ageing test was developed, as extreme conditions (rain, heat, freeze/thaw) are felt throughout the world but seldom coincide in terms of major severity, implying that mortars do not need to fulfil all maximum durability requirements, but those to which they will actually be exposed.

2. BASIS FOR THE DEVELOPMENT OF ARTIFICIAL AGEING TESTS

Artificial ageing tests were developed taking into account Portugal's climatic conditions and other situations that could cause severe damage on rendering mortars.

2.1 Climatic conditions

In Portugal, moderate climatic conditions are felt throughout the country although some regions, mainly in the interior or at greater altitudes, experience temperatures below freezing point a few times a year. Data from the National Institute of Meteorology shows that the minimum temperature ever registered was of –13.3ºC at *Penhas Douradas* (the highest national point, where minimum temperatures usually occur) in the year of 1889 and the maximum temperature was of 48ºC in the southern interior, during the Summer of 2003. The usual minimum temperatures are around –7ºC to –9ºC in the coldest regions, whilst the seaside areas seldom register negative temperatures and when this happens they are in the range of –1ºC to –3ºC. As for usual maximum temperatures, an uppermost limit of 40ºC can be established for the whole territory.

In terms of rainfall, there is an extreme variability from year to year, although the northern part of the country usually receives more rainfall and 42% of total rainfall occurs during the winter months.

Using this data, a heat-rain cycle and a freeze/thaw cycle, which is particularly destructive to mortars due to tensions produced during freezing, seem to be the most adequate for testing mortars to be used under the overstated climatic conditions.

2.2 Action of salts

Apart from climatic conditions that produce degradation especially by freezing and thawing actions, the other elements that play an important role in deterioration of external renders are soluble salts. These are mainly in the form of chlorides, sulphates or nitrates and their action can be simultaneous, depending on exposition factors. Exposure to pollution usually results in chemical reactions with sulphates present in the atmosphere; data from Portugal's Environment Institute gives values of 40μm/m^3 of sulphate emission in the most polluted urban areas of Oporto and Lisbon. Water absorption y capillary action can produce an intake of salts; in this case the solubility of calcium sulphate is low, of 2g/l, so soils with a higher percentage of sulphates also contain potassium, magnesium or sodium sulphates. Direct exposure to sea-water, responsible for ingress of chlorides and sulphates into mortar and substrate, produces contact with a concentration of 27g of sodium chloride (NaCl) per litre of water and usually has an additional sulphate concentration of 2g/l. On the other hand, building materials may also have a considerable amount of soluble salts: sulphates are usually present in cement mortars and bricks, whilst unwashed sea sand can have variable amount of chlorides.

Reactions that take place with soluble salts produce damage in varied ways and some salts are more hazardous than others. The soluble salts that are usually found in masonry are sodium chloride and sodium sulphate. The action of sodium chloride is thought of as being mainly physical, created by its crystallization at a relative humidity below 75%, filling pores and creating an inherent pressure on pore walls. This phenomenon would only affect smaller pores [10]. However, recent studies suggest that sodium chloride deposits around pore walls, causing pressure and dilation of materials due to crystallization expansion combined with adherence to pore walls[11]. Chlorides can produce chemical reactions, forming Friedel's salt ($Ca_2Al(OH)_6(Cl,OH).2H_2O$) with in the presence of calcium aluminate phases [12].

The action of sulphates takes place by chemical interaction between sulphates and mortar components. In lime mortars, this occurs by reaction of the sulphate ions with calcium hydroxide, forming gypsum, as shown in Equation (1).

$$Ca(OH)_2 + Na_2SO_4 + 2H_2O \rightarrow CaSO_4.2H_2O + 2NaOH \quad (1)$$

In the cases of hydraulic mortars or mortars containing hydraulic components in the form of calcium aluminate phases, a further reaction takes place, between gypsum and calcium aluminate, forming ettringite (Equation(2)).

$$3[CaSO_4.2H_2O] + 3CaO.Al_2O_3 + 26H_2O \rightarrow 3CaO.Al_2O_3.3CaSO_4.32H_2O \quad (2)$$

Both compounds are destructive, creating cracking and degradation of mortars. However, besides sodium sulphate, potassium sulphate or magnesium sulphate often cause these reactions. In this last case, and when dealing with mortars with hydraulic characteristics, the complete disintegration of mortar can take place. This occurs due to the fact that, in presence of the magnesium ion, the formation of brucite ($Mg(OH)_2$) takes place by replacement of Ca^{2+} by Mg^{2+} in $Ca(OH)_2$. Brucite is extremely insoluble and its creation removes hydroxide ions and eventually, there is a replacement of C-S-H by M-S-H, that greatly weakens the mortar by removing a key strength-creating product [12]. This accounts for the fact that magnesium sulphate is the most destructive of sulphates for hydraulic or pozzolanic mortars.

Thaumasite ($CaSiO_3.CaCO_3.CaSO_4.15H_2O$) is another possible reaction product, that takes place when the sulphate ion reacts with carbonates and calcium silicate hydrates, but its formation conditions imply temperatures lower than 15ºC and relative humidities over 90% and its occurrence on a significant level is usually linked to the presence of tricalcium aluminate (C_3A) [13-16].

The transition of sodium sulphate between its hydrated phase (Mirabilite) and its anhydrous phase (Thenardite) is also an important cause of tension development in mortars and consequent decay. Mirabilite is a stable phase in temperatures up to 32,4ºC, above which thenardite is formed. Below 32.4ºC mirabilite dehydrates to thenardite at relative humidity below 71%. Recent studies suggest that thenardite crystallization is mainly responsible for damage by sodium sulphate in porous materials [17-18].

Combined presence of sulphates and chlorides, as is the case of the action of sea-water, can create especially severe damage. Formation of gypsum usually comes to a halt when it precipitates around calcite, inhibiting further reaction; however, the presence of NaCl can act as a catalyst, dissolving gypsum and keeping a free surface around calcite, thus allowing for the reaction to proceed [11].

Taking these facts into account and the additional knowledge that bricks may simultaneously contain sodium, potassium and magnesium sulphates [15], a test was devised so as to reproduce particularly adverse conditions, using mortar executed on a brick specimen and partially immersed in water with an NaCl content simulating sea-water.

3. TESTING PROCEDURES

3.1 Mortars

Five different mortars were tested. A lime mortar with a 1:3 (air lime: sand) volumetric ratio (L) was used as a comparison mortar and mortars with aerial lime and natural and artificial pozzolanic additives were prepared, with a 1:1:4 (aerial lime: pozzolanic additive: sand). The employed pozzolans were Cape Verde Pozzolans (mortar CVP1 and CVP2), Azores Pozzolans (mortar AP) and Fly Ash (mortar FA). Fly Ash is mainly composed by a vitreous phase, quartz, mulite and hematite whilst the natural pozzolans both have feldspars as main components; Cape Verde Pozzolans also contain large amounts of zeolites. Two different volumetric ratios were used for the Cape Verde Pozzolanas : 1:0,5:2,5 (CVP1) and 1:1:4 (CVP2).

3.2 Specimens and equipment

Mortars were executed on common industrial fired brick specimens, on a single layer of 20mm, as shown in Figure 1. This simulates what is generally done on site, as a unique thick layer is easier to accomplish and is therefore often executed. The specimens were then stored in a conditioned room with a temperature of 20ºC ± 2ºC and a relative humidity of 65% ± 5%, for 90 days. Specimens L and CVP1 were cured for 2 years.

3.3 Testing procedure

Two parallel tests were performed: an artificial ageing test comprising rain/heat and freeze/thaw cycles and a salt resistance test based on the placement of specimens in water with sodium chloride.

Artificial ageing tests were designed on the basis of national climatic conditions and were composed of the following cycles:

- rain-heat cycle, with a temperature of 40ºC (4h), followed by a rain period (4h) with an intensity of 1l/min and a drying period of 16h
- freeze/thaw cycle initiated by a rain period (4h) followed by a temperature of –10ºC during 4h and a waiting time of another 4h

These cycles were performed in a climatic chamber (Aralab Fitoclima 700 EDTU), with specimens placed as shown in Figure 2 and each cycle was repeated 10 times. Between cycles and at the end of the test, specimens were weighed and degradation patterns were registered (Figure 3). Water permeability and pull-off testing were performed following EN 1015-21[6].

Resistance to salts was monitored by placing specimens in the same conditioned atmosphere (20ºC, 65%RH) in a tray with water containing 27g/l of NaCl (simulating sea water) and reaching a height correspondent to half of the specimen (Figure 6). In this way specimens are at the same time in direct contact with water and there is a capillary absorption phenomenon taking place. On the other hand, soluble salts from the brick can migrate to the mortar. Specimens were kept during one week in these conditions and then dried at ambient temperature until they reached constant weight. Cycling was performed until degradation was evidenced by a weight loss.

Figure 1 Preparation of specimens

Figure 2 Specimens after cycles

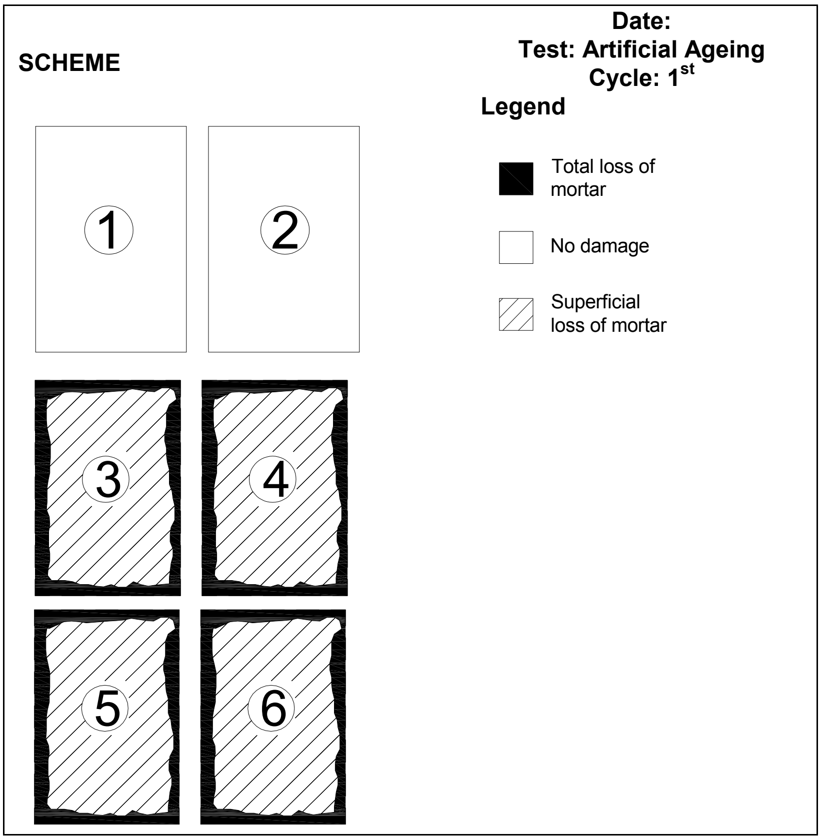

Figure 3 Generic scheme of specimen degradation after first cycle

4. RESULTS

Results of the artificial ageing test are represented in Figure 4, with the percentage weight loss of mortar after each cycle (medium and standard deviation). Hardly any weight losses are observed after the first rain-heat cycle and the behaviour of mortars to this type of weathering is generally good. Freeze/thaw cycle, much more aggressive to mortars, produced greater differences in results. Although most mortars also had small mass losses after freeze/thaw was performed, one of the Lime mortars lost its superficial layer and the borders were eroded and one of the Cape Verde pozzolan mortars (1:0,5:2,5 ratio) detached itself completely from the substrate. This is depicted by the great standard deviations observed in Figure 4. The failure observed in the mortar/brick interface may be due to tensions caused by the freeze/thaw action or/and to lack of adhesion between the mortar and the brick surface. Water permeability tests gave similar results for CVP2, AP and FA mortars, with CVP specimens showing slightly lower permeability (Figure 5). Testing for adhesive strength using the pull-off test gave low results, with an average of 0.03MPa and a maximum of 0.05MPa. The overall evaluation of these results shows that they may not be representative of real applications on ancient masonry.

Salt resistance testing produced a common pattern of salt precipitation, as seen on Figures 6, 7a and 7b. Salt crystallized on the uppermost brick surface, where it was brought to by capillary action. Most mortar degradation occurred on mortar borders contiguous to this surface, but the brick itself suffered severe degradation, especially innermost and on the zone that was never immersed.

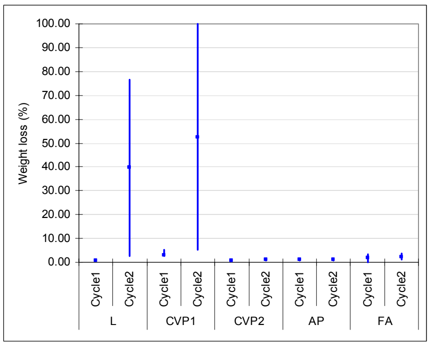

Figure 4 Weight loss of mortar (%) after rain-heat cycles (Cycle 1) and freeze-thaw cycles (Cycle 2)

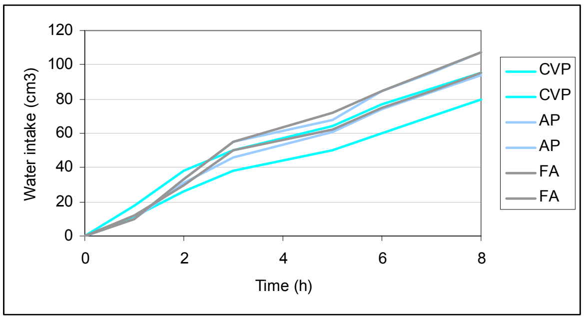

Figure 5 Water permeability

Figure 6 Degradation pattern of specimens after first cycle

Figures 7 a) and b) Specimens after salt resistance test

Analysing the mass variations (Figure 8), it is evident that specimens with greater curing times (L and CVP1) have a slower initial mass gain, in comparison with other specimens, cured for 90 days. On the long term, aerial lime mortar specimens (L) have a more erratic behaviour and the highest mass gains, due to salt crystallization. Mortars CVP2, AP and FA show similarities, with an especially big mass gain after the sixth cycle was performed, related to salt crystallization, followed by a mass loss due to degradation of the specimen (mortar and brick). The behaviour of CVP1 is particular, due both to its age and composition. This specimen shows the smallest variations during the salt cycling. One of the mortars with Fly Ash detached itself from the brick during the seventh cycle; this may be due to salt formation on the brick/ mortar interface and/or lack of adhesion between mortar and brick.

An XRD was performed on the salt that precipitated on the upper brick surface of a specimen with Azores pozzolan mortar, revealing mainly halite (NaCl), and traces of gypsum ($CaSO_4.2H_2O$), as shown in Figure 9.

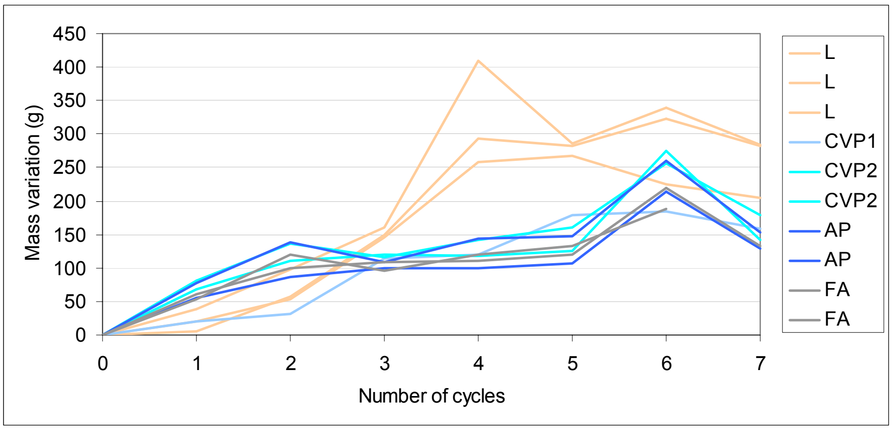

Figure 8 Mass variation (%) during salt cycles

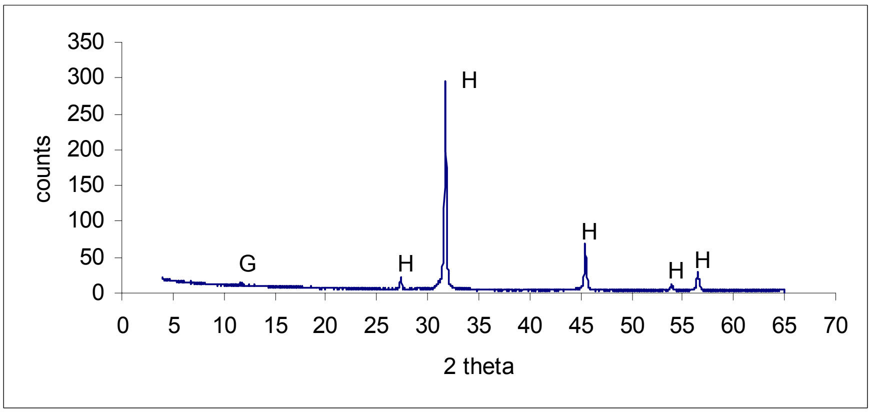

Figure 9 XRD of salt precipitation on brick on specimen with mortar containing Azores pozzolans

5. CONCLUSION

Generally, mortars containing pozzolans showed a good behaviour both in the artificial ageing test and in the salt crystallization test, especially when compared to pure lime mortars All mortars resisted without visible damage to heat rain cycles and it seems that failure may occur due to freeze/thaw action. . However, the use of brick as a substrate and execution procedures may account for some of these failures, as adhesion between brick and mortar seems to be low. This may not be a problem in old walls due to their higher roughness; however, a better test must be developed to better simulate real application conditions.

he action of salts, with a few wetting/drying cycles also produces damage. This damage has two main causes: salt crystallization and loss of adhesion in the brick/mortar interface. In terms of salt formation, although halite and gypsum were found on the salts that crystallized over the brick, a further analysis to both mortar and brick with evident decay is necessary to determine decay factors.

he developed tests are considered adequate as their results allow for a differentiation among the tested mortars.

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