# **Durability of Clay Roofing Tiles under Salt Mist Atmosphere**

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# ABSTRACT

Clay tiles are largely used in traditional construction in Portugal. In coastal zones some cases of accelerated decay have been affecting the roofing tiles durability, apparently due to the exposition to a salt mist atmosphere. To evaluate the performance of the clay roofing tiles subjected to a sea environment, an experimental study is being carried out based on artificial accelerated weathering tests. The study includes the roofing tiles characterization, the development of accelerated weathering tests, the evaluation of the weathering effect through observation and physical and chemical tests and also the comparison with roofing tiles that suffered natural weathering in coastal zones of Portugal. The study aims at reproducing the pathology mechanisms observed in real conditions, and identifying the parameters - of both composition and manufacture - having a higher contribution to the decay of roofing tiles to provide industry with relevant data to improve the manufacturing process.

This paper presents tests performed on several kinds of tiles and the results obtained regarding soluble salt effects in the specimens' microstructure. Mercury Intrusion Porosimetry (MIP) was used to assess differences in the porous structure. Soluble salt content profiles after weathering were also performed, aiming to determine the depth of salt crystallization.

Accelerated weathering tests can cause different levels of alteration and degradation to ceramic roofing tiles. Hydrophobic tiles may resist longer to salty mist but they can suffer high damage if there is some penetration of salty solution. Tiles present rather diverse porosimetric structures. The concentration of salt crystallization on the outer layers may induce the destruction of thin superficial layers.

# **KEYWORDS**

Durability, Clay roofing tiles, Accelerated weathering tests, Salt mist.

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### **1 INTRODUCTION**

The crystallization of soluble salts is considered one of the main causes for the degradation of porous construction materials [Rodrigues J. D. and Gonçalves T. D. 2006]. To simulate, in the laboratory, the weathering defects due to salty mist, specimens from different types of tiles have been subjected, in a climatic chamber, to artificially accelerated weathering test sets of 10, 20 and 30 cycles of wetting with a salty mist and subsequent drying. The effects of weathering cycles were studied through visual assessment, weight variations and water absorption tests performed before and after weathering. The changes in microstructure were assessed through determination, before and after weathering, of the pore size distribution by Mercury Intrusion Porosimetry (MIP). After weathering, soluble salt content profiles were performed, to determine the depth of salt crystallization.

### 2 MATERIALS AND METHODS

### 2.1 Test Specimens

5 specimens from each tile were prepared through transversal cuts. In these tests the specimens cut from the mid transversal area of the roofing tiles were preferentially used, having the width of the tile and circa 9 cm length (Fig. 1). The cut faces of the specimens were sealed with epoxy resin. Roofing tiles from two manufacturers (F1 and F2) were used:

F1 NH - manufacturer 1, model 1, non-hydrophobic red tiles F1 B - manufacturer 1, model 1, non-hydrophobic white tiles F1 H - manufacturer 1, model 1, hydrophobic red tiles F2 NH - manufacturer 2, model 1, non-hydrophobic red tiles F2 B - manufacturer 2, model 1, non-hydrophobic white tiles F2 H - manufacturer 2, model 1, hydrophobic red tiles F2 T2 NH - manufacturer 2, model 2, non-hydrophobic red tiles

F2 T3 NH - manufacturer 2, model 3, non-hydrophobic red tiles

### 2.2 Artificial Accelerated Weathering Tests

Tests were designed based on the European Norm EN 14147:2003 and adapted to what was thought to be realistic conditions for this product [Cruz et al, 2007]. These tests were carried out in a climatic chamber where the specimens are subjected to sets of cycles of wetting under salt mist atmosphere and subsequent drying, the temperature in the chamber maintained at 35°C. Salt mist is produced by spraying a salt solution into the chamber with a concentration of 110g of sodium chloride per litre of de-ionised water. The results of two preliminary tests with cycles of 48 h (10 h of salt fog + 38 h of drying) and 24 h (8 h + 16 h) allowed the selection of the 24 h cycles for the final tests. For each type of tiles, one or two specimens were subjected to 10 cycles, another one or two to 20 cycles and three or four to 30 cycles.



Figure 1. Specimens before and during tests.

### **2.3 Characterization Tests**

Before the cycles, the specimens were dried at a temperature of 60°C to constant mass, as a basis for the weight variations tests. They were also subjected to a water immersion test based on Annex B of EN 539-2:2006, which measures the specimen's weight increase after 48 h of water immersion. For some types of tiles, 'new condition' specimens taken from the same tile as those used for weathering tests, where subjected to Mercury Intrusion Porosimetry (MIP) tests, based on ASTM D 4404 [ASTM 2004], to determine their microstructure.

After weathering and desalinization, specimens of all but one type were subjected to water immersion under vacuum, for characterization purposes. This test, based on EN 1936:1999, measures the weight increase after 72 h of immersion (24 h under vacuum followed by 24 h under water + vacuum and more 24 h under water at atmospheric pressure). Finally, the specimens are weighed twice: once submerged, and after being wiped. This test permits the determination of apparent density, open porosity, and water absorption under vacuum.

### **2.4 Weathering Effect Evaluation Tests**

During the weathering tests, the specimens were visually inspected. After desalinization, they were submitted to water absorption, and samples of some specimens were subjected to Mercury Intrusion Porosimetry. These tests were carried out for comparison of results and evaluation of their sensitivity to the degradation suffered by the specimens. To determine the depth of salt crystallization, salt content profiles were obtained on weathered and non-desalinised samples of two different kinds of specimens, using the hygroscopic moisture content (HMC) method [Gonçalves T. D. and Rodrigues J. D. 2006]. After being submitted to 30 cycles of salt mist wet/drying, one specimen of F2T3NH and another of F2B4 were dry cut and, in this way, two C1 samples were obtained (Fig. 2). In each of the C1 samples 5 layers were marked and from them 5 samples were obtained using sandpaper (from the upper face to the under face: C1 A, C1 B, C1 C, C1 D and C1 E), control samples with sodium chloride by itself and with new tile powder were also used (Fig.3). All the samples were dried at 60°C and then inserted in a climatic chamber at 20°C 85%HR, their weights were measured to constant mass and HMC values were calculated.



Figure 2. C1 samples.



Figure 3. Grounded layers and control samples.

# **3 RESULTS**

# 3.1 Visual Assessment, Weight Variations and Water Absorption

Visual inspection results (Table 1) demonstrate different types and levels of degradation caused on the specimens by the weathering tests.

Specimens	Degradation	Examples				
F1 NH	Pitting Granular disaggregation					
F1 B	Pitting Granular disaggregation Peeling					
F1 H	None Bowing Plaques	F1H7C				
F2 NH	Pitting Granular disaggregation	ELMINC ELMINC				
F2 B	Pitting Granular disaggregation Peeling	F2 B 5 C				
F2 H	None					
F2 T2 NH and	Pitting Granular disaggregation					
1°2 13 INFI	Peeling	FZTZIHHOC				

Table 1. Alteration and degradation forms with some examples.

Hydrophobic specimens didn't show immediate defects but some of them suffered huge degradation following the access of the salty solution to the material under the protected surface. Hydrophobic tiles from manufacturer 2 seem to be more resistant to weathering than those from manufacturer 1. Non-hydrophobic tiles, both red and white, showed low to medium degradation levels characterized mainly by pitting, some disaggregation and peeling, with a slightly higher level in the white ones.

In general, weight determinations before and after weathering didn't show significant differences, thus they do not quantify the visual degradation at the present level. However, for white tiles of both manufacturers and some hydrophobic red tiles of manufacturer 1, where degradation is higher, weight loss is more significant.

The results of water absorption under vacuum obtained (Table 2) show that the white tiles present higher porosity and higher water absorption than the red tiles, for both manufacturers. Concerning the red tiles, the water absorption and porosity are higher for those from manufacturer 1. The lowest values concern the hydrophobic tiles from F2 and the model T3 also from F2. There is a significant increase of water absorption after weathering (Table 3) in the hydrophobic red tiles of manufacturer 1 (F1H). For the other specimens, so far the water absorption does not quantify the visual degradation.

Manufacturers 1 and 2				Manufacturer 2			
Specimens	Apparent density [Kg/m3]	Open porosity [% V]	Water absorption under vacuum [% W]	Specimens	Apparent density [Kg/m3]	Open porosity [%]	Water absorption under vacuum [% W]
F1NH	2070	23.0	11.1	F2NH	2200	18.4	8.4
F1B	2020	24.3	12.0	F2B	2060	21.8	10.6
F1H				F2H	2200	17.9	8.1
F2T2NH	2130	21.3	10.0	F2T3NH	2210	17.9	8.1

Table 2. Water immersion under vacuum test results (characterization tests).

Table 3.	Water	absorption.
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	Manufacturers 1 and 2			Manufacturer 2		
Number of		Before	After		Before	After
cycles	Specimens	weathering	weathering	Specimens	weathering	weathering
		[%]	[%]		[%]	[%]
10		9.34	9.21		5.83	5.83
20	EINH	9.37	9.26	F2NH	5.81	5.86
30	ГІЛП	9.22; 9.22;	9.20; 9.08;		5.89; 5.90;	5.86; 5.90;
(3 spec)		10.15	10.03		6.46	6.49
10 (1 or 2)	F1B	9.89	9.78	F2B	7.26; 7.26	7.26; 7.25
20 (1 or 2)		9.04	9.00		7.30; 6.80	7.31; 6.83
30		9.12; 9.59;	9.07; 9.57;		7.24; 6.83;	7.25; 6.84;
(3 spec)		9.20	9.06		6.79	6.76
10	F1H	3.89	3.03	F2H	0.58	0.24
20		8.75	10.68		0.57	0.37
30		4.24; 6.16;	9.15; 10.00;		0.61; 1.19;	0.53; 0.56;
(3 spec)		4.31	8.92		0.42	0.28
10 (1 or 2)	F2T2NH	8.24	8.17	F2T3NH	5.97; 5.95	5.80; 5.74
20 (1 or 2)		8.31	8.31		6.08; 5.85	5.88; 5.66
30		8.30; 8.23;	8.30; 8.22;		5.68; 5.71;	5.55; 5.56;
(3 spec)		8.20	8.15		6.13	6.00

### **3.2 Mercury Intrusion Porosimetry**

The pore size distributions obtained (Fig. 4) show some differences among the various tiles: the red non-hydrophobic model 1 tiles of both manufacturers F1 and F2 have the largest pores within a very thin range of values (around 0,2-0,5  $\mu$ m); the white tiles from manufacturer F1 show a large range of pore dimensions, including the finest pores around 0,05-0,09  $\mu$ m and some larger pores of the range 0,2-0,4  $\mu$ m; the non-hydrophobic tiles of the model T2 from manufacturer F2 have finer pores than the other non-hydrophobic red tiles (mainly in the range 0,1-0,3  $\mu$ m).

The curves also show that the deterioration didn't provoke significant changes on the pores dimensions. Possibly, the type of degradation obtained is still rather superficial and only longer tests will produce the rupture of pores due to salt crystallisation.



Figure 4. Pore size distribution.

# **3.3 Salt Content Profiles**

Figure 5 shows the Hygroscopic Moisture Content over time of the five grounded layers (Fig. 3) of each of two C1 samples (Fig. 2) obtained from weathered non-desalinised specimens F2T3NH7C and F2B4C.



Figure 5. HMC graphics of grounded layers A, B, C, D and E.

Figure 6 shows the same HMC over time of the grounded layers, but now together with the HMC of sodium chloride control samples (Fig. 3). The last chart shows the HMC over time of control samples with powder from new tiles (Fig. 3).



Figure 6. HMC graphics of grounded layers and of control samples.

From HMC values on day 7 the NaCl content of each layer was obtained (Table 3). In both types of samples, NaCl content is much higher in the outer layers. In the inner layers of the white sample the content of NaCl is higher than in the inner layers of the model 3 red tiles.

Louora	NaCl content (%)			
Layers	F2T3NH	F2B		
C1 A	4.0	3.1		
C1 B	0.4	1.2		
C1 C	0.4	1.1		
C1 D	0.4	1.1		
C1 E	2.6	2.7		

### Table 3. NaCl contents

### **4 DISCUSSION OF RESULTS**

Visual inspection results demonstrate different types and levels of degradation caused by the weathering tests on the specimens. Hydrophobic specimens don't show immediate defects but they can suffer huge degradation if the solution can pass through the protected surface. Hydrophobic tiles from manufacturer 2 seem to be more resistant to weathering than those from manufacturer 1. Non-hydrophobic tiles, both red and white, show low to medium degradation levels, with a slightly increased level in the white ones. Characterization tests demonstrate different physical characteristics of the various types of tiles. Naturally, hydrophobic tiles show much lower water absorption than non-hydrophobic tiles, especially in manufacturer 2 tiles. This difference is less evident in vacuum conditions. For both absorption conditions, non-hydrophobic tiles from manufacturer 1 have higher absorption than model 1 tiles from manufacturer 2, whose model T2 is nevertheless the most

absorbent. White tiles show slightly higher water absorption and higher porosity than nonhydrophobic model 1 red tiles. These characteristics can explain the difference also detected in visual degradation. In general, weight variations and water absorption tests do not quantify the visual degradation at the present level. The MIP results show some differences in the porous structure of the four models tested. Tiles F1 B and F2 T2 present larger ranges of pore sizes and they are also those where higher degradation was observed. The finest and more homogeneous range of pores is of nonhydrophobic model 1 red tiles from both manufacturers, which showed intermediate levels of degradation. The salt content profiles demonstrate higher concentration of sodium chloride in the outer layers, in the upper and lower faces, especially in the case of the red tile tested. These crystals may be harmless if they are upon the surface but they may also provoke peeling if they concentrate under a very thin layer of material, which can be easily detached. The concentration in outer layers may thus indicate a potential for quick deterioration.

### **5** CONCLUSION

Cycles of wetting with a salty mist and subsequent drying can be used to cause different levels of artificial weathering alteration and degradation to ceramic roofing tiles. Hydrophobic tiles may resist longer to salty mist but they can suffer high damage if there is some penetration of salty solution. White tiles showed higher levels of degradation than the non-hydrophobic red ones. The tiles present rather diverse porous structures, which could be the cause of different behaviour under the testing cycles. The white and the non-hydrophobic model T2 red tiles, which show higher degradation, have larger ranges of pore sizes. The concentration of salt crystallization on the outer layers may induce the destruction of thin superficial layers.

To simulate real adverse conditions it is necessary to get higher degradation levels. The intention is to pursue the study and achieve these results by increasing the number of cycles or changing some of the parameters of the weathering tests.

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