

## **Use of a Silicone-based Hydrophobic Treatment in Ceramic Floor Tiles: a Case Study in Portugal**

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### **Abstract**

Hydrophobic treatments are often used in porous ceramic floor tiles to prevent the occurrence of stains during the use phase.

Premature failure of the ceramic pavement of several apartments and townhouses of a golf resort in Portugal occurred between 2006 and 2007. The problem took place three to four years after application. It consisted of tile detachment, which resulted in markedly irregular pavement surfaces.

The anomaly affected only those tiles treated with a silicone-based hydrophobization product, regardless of them being located in a ground or upper floor. Similar, but non-hydrophobized tiles, which had been used before in some dwellings, showed no anomalies.

This article describes the problem in detail and presents the work carried out to identify its causes. It included site-inspections with collection of samples, as well as thermal (EN ISO 10545-8) and moisture (EN ISO 10545-10) expansion tests.

It is concluded that the anomaly was probably due to the confluence of four main factors: (i) presence of moisture in the buildings; (ii) high moisture expansion of the ceramic tiles, (iii) poor application of the tiles; (iv) use of the hydrophobic treatment in these tiles.

**Keywords:** water repellent, hydrophobic treatment, ceramic tiles, moisture expansion

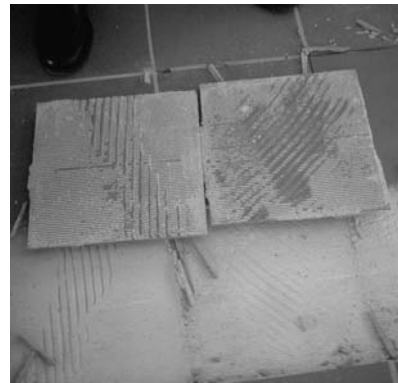
## **1 Introduction**

In 2008, a private real estate development company asked an opinion on the causes of the extensive detachment of the red ceramic floor tiles used in several dwellings (apartments and townhouses) of a golf resort, in Portugal. This article describes the work carried out in that framework and the conclusions it allowed achieving. On request, the names of the company and resort are not disclosed.

The inspection of an apartment indicated that the anomaly was probably due to expansion of the tiles, which eventually led to their detachment (Figs. 1 and 2).



**Figure 1:** Damaged pavement of the upper floor apartment visited



**Figure 2:** Back of detached tiles

The tiles are red clay extruded tiles (group Alla-2 [1]) manufactured by the Portuguese company Soladrilho. Prior to delivery, silicone-based water repellent surface treatment SOLIMPER HIDRO was applied on the tiles at the factory.

Similar tiles had been used, in the first buildings of the resort, but with no water repellent treatment. However, due to their high tendency to stain, they were eventually replaced by the present hydrophobated tiles. These previous non-hydrophobated tiles showed no detachment problems.

In order to assess the contribution of the ceramic tiles for the expansion of the floor coating, the coefficients of thermal and moisture expansion of the tiles were determined. This article presents the experimental results obtained and discusses them, together with other information on the buildings, the materials and their circumstances, in order to identify the causes of the problems encountered.

## **2 Inspection data**

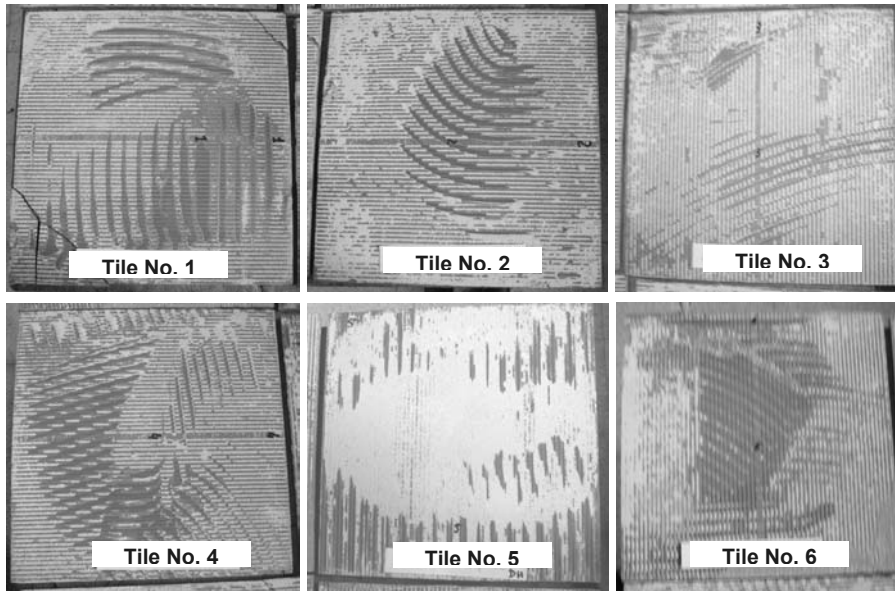
The inspection of an upper floor apartment (Figs. 1 and 2) provided the following information, which was obtained, either from direct observation of the construction, from the contactor or from the real estate company:

- The tiles have facial dimensions of 300 mm by 300 mm and a thickness of approximately 11.5 mm. The width of the joints is around 7 to 10 mm.
- The detachment problems affected tiles applied three to four years before. Occurrences were observed in different months of the year (January, March, August, September, October, December) in numerous apartments and townhouses.
- Tile detachment happened in general suddenly (and often producing noise). In the case of the visited apartment, where the floor coating had been completed four years ago, the detachments had occurred suddenly, three weeks earlier, in October.
- A dehumidifier was installed in one of the compartments of the apartment. We were informed that the purpose was to decrease the relative humidity of the air, which was often very high in the interior.
- The apartments and townhouses of the resort, including the visited apartment, are used mostly during weekends and holidays in spring and summer. They are generally unoccupied during a significant part of the year, especially in the cold months.

## **3 Testing**

### **3.1 Materials**

The tests were carried out on specimens extracted from six tiles that had suffered detachment. The remains of adhesive mortar present on the back of the tiles (Fig. 3) were, as much as possible, removed by brushing.



**Figure 3:** Back of the tiles used in the tests

## **3.2 Test methods**

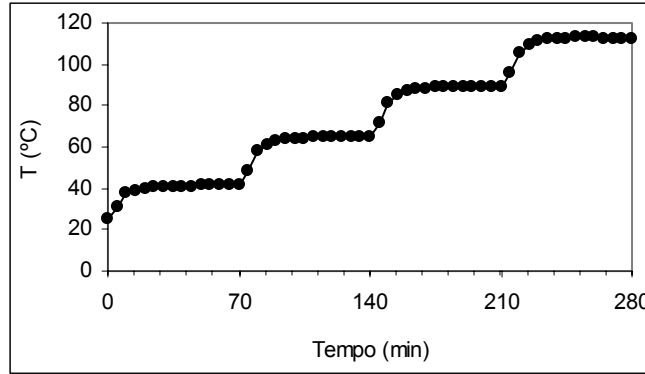
### **3.2.1 Thermal expansion**

The test for determining the coefficient of linear thermal expansion was carried out using the procedure of EN ISO 10545-8 [2].

The test was performed on two specimens with facial dimensions of 100 mm x 30 mm. The specimens were respectively extracted from tile No. 6 (Fig. 3), either perpendicularly or in parallel to the grooves on the back of the tiles (which indicate the extrusion direction).

A DILAMATIC® dilatometer from Theta Industries was used. It provides a resolution of 1 $\mu$ m for length variations and of 0.1 °C for surface temperature.

The temperature program consisted of 20 °C increments over 10 min, followed by 60 min at constant temperature. The surface temperature of the specimen was measured at the end of each period at constant temperature.



**Figure 4:** Temperature program used in the thermal dilation test

The coefficient of linear thermal expansion  $\alpha_l$  is given by expression (1):

$$\alpha_l = \frac{\Delta L}{L_0 \cdot \Delta T} \quad (1)$$

where  $L_0$  is the length of the specimen at room temperature,  $\Delta L$  the increase in length between room temperature and 100°C and  $\Delta T$  the corresponding increase in temperature.

### 3.2.2 Moisture expansion

The test for determining the moisture expansion was carried out according to the procedure of EN ISO 10545-10 [3]. It consists of immersing a reheated tile into boiling water and then measuring the proportional change in length.

The test was carried out on six specimens extracted, respectively, from each of the tiles shown in Fig. 3. The specimens had flat dimensions of 100 mm x 35 mm. Their length was oriented along the extrusion direction (direction of the grooves on the back of the tile).

The length difference of the specimens in relation to a standard Invar steel bar of 100 mm length is measured before and after the treatment with boiling water. This treatment consists of maintaining the specimens immersed in boiling water over a period of 24 hours. The length measurements were performed using a Mitutoyo digital deflectometer providing a resolution of 0.01 mm.

In order that different measurements of the same specimen were always done between the same points of the specimen, small holes were drilled in the two extreme faces of the specimens. As in the case of the steel bar,

which has a small hole at each end, the measurement of the specimen length was done by inserting the top and bottom tips of the measuring instrument in these holes. The results correspond, therefore, to an effective length, equal to the total length of the specimen minus the depth of the two small holes.

The length measurements were always repeated once, with a three hour interval. The ones subsequent to the treatment with boiling water were carried out only after cooling of the specimens to room temperature.

Moisture expansion (mm/m) is given by expression (2):

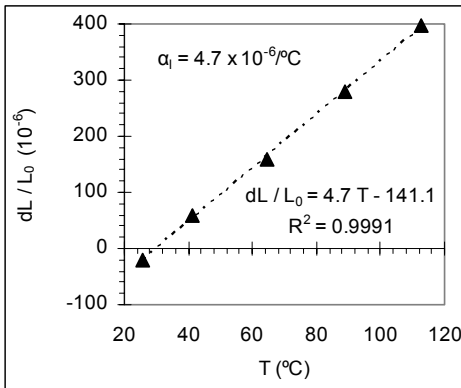
$$1000 \Delta l / L_0 \quad (2)$$

where  $L_0$  (mm) is the average initial length of the specimen and  $\Delta l$  (mm) the average difference between the final and initial lengths of the specimen.

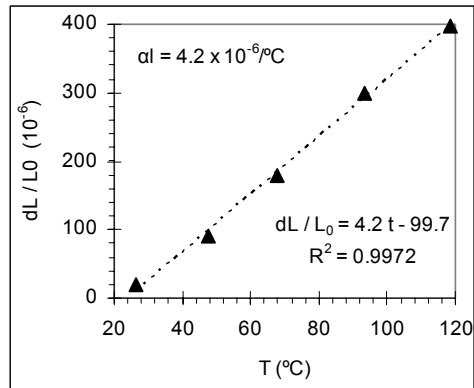
### 3.3 Results

The results obtained in the thermal and moisture expansion tests are presented in Figs. 5 to 7.

Before being cut to extract the specimens, the tiles were subjected to quick tests carried out by immersing them in water at ambient temperature. These preliminary tests showed that the tiles do retain some water absorption capability, despite the indication that a water repellent treatment had been applied on their surface.



**Figure 5:** Thermal expansion parallel to the extrusion direction



**Figure 6:** Thermal expansion perpendicular to the extrusion direction

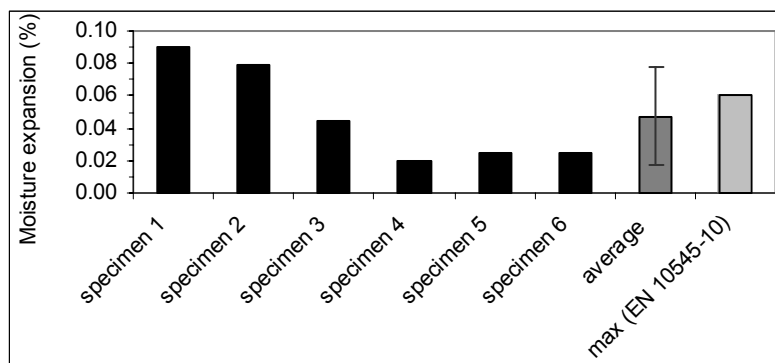


Figure 7: Moisture expansion

## 4 Discussion

### 4.1 General

Ceramic tiles tend to dilate differentially to the substrate, due to the action of temperature or moisture, thus, giving rise to tensions in the coating system. Detachment occurs when the adhesion of the tiles to the substrate is not strong enough and able to limit their expansion to values compatible with the width of the joints and deformability of the bonding and joint mortars. The complex system of compressive and tensile stresses that arises in the coating may also cause rupture of the ceramic tiles.

### 4.2 Thermal expansion

The thermal expansion test is used to evaluate the linear dimensional variation that the tiles experience when subjected to a temperature increase.

As seen in Figs. 5 and 6, the values obtained for the coefficient of linear thermal expansion are  $4.7 \times 10^{-6} / ^\circ\text{C}$  (extrusion direction) and  $4.2 \times 10^{-6} / ^\circ\text{C}$  (direction perpendicular to the extrusion direction).

No thresholds have been established for this coefficient, in particular in standard EN 14411 [1], which sets the requirements to be met by the different types of ceramic tiles. It is, however, possible to conclude that these values do not differ much from, and are even slightly below, the ones currently obtained for ceramic materials, which are in the order of  $5$  to  $6 \times 10^{-6} / ^\circ\text{C}$  [4].

It is, therefore, unlikely that thermal expansion is, in itself, a decisive factor in the observed detachment problems.

### **4.3 Moisture expansion**

The moisture expansion test aims at reproducing, in an accelerated way, the total expansion that ceramic tiles can reach over time due to the action of moisture, in current conditions of temperature and pressure [4, for example]. As it happens with ceramic materials in general, much of this expansion is irreversible. This irreversible expansion is due to a slow capture of water and its further chemical reaction with the minerals in the ceramic, occurring in a very slow process that starts the moment the material leaves the kiln and lasts for years.

As for thermal expansion, there are no established thresholds for the moisture expansion, in particular in standard EN 14411 [1], which sets the requirements to be met by the different types of ceramic tiles. In this case, however, the CEN test standard [3] recommends a value of 0.06% for the maximum moisture expansion of ceramic tiles. The establishment of this value takes into account that moisture expansion, although generally not contributing to the emergence of problems when the tiles are properly applied, can do so when that application is poor or when the coating is subjected to particularly harsh climatic conditions.

As shown in Table II, which presents the results of the test, the average moisture expansion of the tiles is 0.05%, a value that approaches the maximum recommended value of 0.06%. Furthermore, that threshold value is clearly exceeded by two of the specimens.

The dispersion of the experimental values obtained for the six tested tiles is very significant. The maximum amplitude (0.07%) has the same order of magnitude of the individual moisture expansion values, which means that the behaviour of the tiles may differ significantly.

These results indicate that the studied tiles can achieve a high moisture expansion and, therefore, this is probably one of the factors behind their detachment.

### **4.4 Use of the water repellent treatment in the tiles**

The studied tiles were treated at the factory with a water repellent surface product.

This type of treatment acts by giving hydrophobic properties to the pore walls (hydrophobic effect), thereby neutralizing the capillary suction of the material. In practice, some products also block part of the material porosity (sealing effect), in which case there is a simultaneous reduction in vapour permeability.

The actual working principle of the water repellent treatment used in the studied tiles is not known. The information conveyed by the data sheet of the product indicates that it should act essentially as a water repellent. However, there is no evidence that allows in fact dismissing the risk of a parallel vapour permeability reduction.



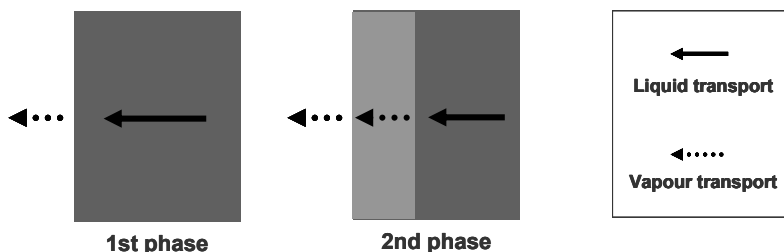
But, regardless of the effective working principle of the product, there is circumstantial evidence indicating that the use of the hydrophobic treatment is associated with the occurrence of the problems observed:

- (i) all the detached tiles have been hydrophobated;
- (ii) detachment of the initial non-hydrophobated tiles was never observed.

There is no information on the method used to apply the treatment on the tiles, in particular, on whether it was applied on both faces and all sides. However, the moisture expansion tests and also the preliminary absorption tests carried out at ambient temperature showed that the tiles do absorb some water. Therefore, regardless of the application method, the fundamental fact is that the treatment does not totally prevent water from penetrating the tiles.

It is not known whether the use of the water repellent treatment produces any change in the moisture expansion properties of the tiles, although that seems rather unlikely. But the use of such treatment can drastically alter the behaviour of the tile in relation to the penetration and permanence of moisture in its pores, not only as regards the penetration of liquid water but also as it concerns drying.

Indeed, it is commonly accepted that sealing treatments hinder porous material drying because they block the pores. Differently, water repellent treatments without a parallel sealing effect are often argued to not alter drying because they do not block those pores. But this is not necessarily true, as will be explained.



**Figure 8:** Drying of a porous material

Drying of porous materials (ceramics, stone, mortar, etc.) has two main phases [5, for example], which are shown in Figure 8:

- 1) In the first phase, the water content is high and there is continuity of the liquid phase throughout the material. The transport of moisture takes place mainly by capillarity towards the evaporation front, which is located at the surface. The drying rate is constant and assumes its higher value.
- 2) With the decrease in the water content of the material, at a certain moment, the liquid flow towards the surface becomes insufficient to compensate for the evaporative demand. As a consequence, the evaporation front recedes into the material and, therefore, the second drying phase begins. During this phase, moisture migrates by liquid capillary transport to the evaporation front and, then, by vapour diffusion through the pores up to the surface of the material. The drying rate decreases gradually as the evaporation front recedes progressively further into the material.

The presence of a water repellent treatment in the surface layers of a drying material implies that the transport of moisture through the treated layer can only happen by vapour diffusion. Therefore, even if the drying conditions are such that an untreated material would be in the first phase of drying, in a treated material, the evaporation front is always located inside the material. This means that, for sufficiently high moisture contents or slow evaporation conditions, the drying rate of a treated material tends to be lower than that of a similar but untreated material.

Vapour transport itself may also be slower through the water repellent layer if the hypothesis of vapour diffusion being accelerated by the presence of water in the pores is true. This mechanism is the so-called “liquid assisted vapour transport” which was attributed to a shortening of the effective path length for vapour diffusion arising from the presence of liquid [6,7]. This implies that vapour transport is faster through a non-hydrophobic material where residual pockets of liquid water remain in the pores.

There are also moisture transport mechanisms that occur only in hydrophilic materials. It is the case of surface diffusion, which happens at low moisture contents and consists in the diffusive migration of the water adsorbed at the pore surfaces, due to the existence of relative humidity gradients.

The considerations above indicate that the water repellent treatment will tend to hinder drying, thereby prolonging the presence of water in the tiles (experimental evidence of the fact that water repellent treatments hinder the drying of porous materials is reported in another article presented at this conference). And a longer permanence of water will aggravate the effective moisture expansion of ceramic materials.

This conclusion is consistent with the fact that detachment of the non-treated tiles was never observed.

#### 4.5 Presence of moisture

A longer presence of moisture in their pore system is probably, as discussed in the previous sections, the factor behind the detachment of the treated tiles.

A detailed assessment of the possible origins of this moisture and of the exact way it can penetrate into the body of the tiles is not possible only on the basis of the available data. There are several hypotheses: penetration of condensed water through the upper surface of the tiles (if the water repellent is not 100% effective or has not been properly applied), internal condensations in the mass of the ceramic material, penetration through the joints, etc.

But it is known that the apartments and townhouses of the resort, which is located near the Atlantic coast, are closed and uninhabited most of the year and have frequently a high RH, as reported by the technicians from the involved companies and corroborated by the presence of a dehumidifier in the visited apartment. And, in general, a high RH eventually results in the occurrence of dew point condensations on the surface or in the mass of the construction materials.

Another factor that may give rise to or aggravate the presence of moisture in these materials is the presence of soluble salts. Indeed, soluble salts slow down the drying of porous materials [5] and, due to their hygroscopic characteristics, may attract large amounts of moisture from the air. In the present resort, which is located near the Atlantic coast, salt transport (in particular, sodium chloride) due to fog and wind is a solid hypothesis.

#### 4.6 Application of the tiles

Though a comprehensive analysis of the technique used to applying the tiles is not possible based on the available data, the simple observation of the detached tiles provides already relevant information on this respect.

Tile bond failure was predominantly adhesive: as seen in Fig. 3, rupture of the bonding mortar happens, in most cases, at the interface mortar/tile. Furthermore, the bonding mortar rows are often visible in the back of the tiles, which suggests that they were not sufficiently pressed during the application. These two facts indicate that the adhesion of the tiles to the substrate was not probably very high.

## **5 Conclusions**

The coefficient of linear thermal expansion of the studied tiles is within the current values for ceramic materials. The moisture expansion of some of the tested tiles, however, is higher than the threshold of 0.06% recommended [3] when: (i) the application technique is not totally in agreement with the "rules of the art"; (ii) the tiles are subjected to particular climatic conditions.

The weak adherence of the tiles to the substrate, evidenced by the adhesive rupture of the bonding mortar, and the fact that the mortar rows were not conveniently compressed indicate that the tiles were not properly applied.

On the other hand, the presence of moisture in the apartments and townhouses of the resort is probably significant. Furthermore, the water repellent tends to hinder drying of the tiles and, therefore, to prolong the presence of the moisture that eventually succeeds in penetrating the ceramic body. These two factors configure particular climatic conditions that tend to aggravate the effective moisture expansion of the tiles.

It is therefore likely that the observed detachment problems are due to the confluence of four factors:

- presence of moisture in the apartments and townhouses of the resort
- significant moisture expansion of the tiles
- poor application of the tiles
- use of a water repellent treatment on the tiles

The fact that detachment occurred only with the hydrophobated tiles and never with the non-hydrophobated tiles (which are in all the rest similar) indicates that the water repellent treatment had a relevant contribution for the problem.

## **Acknowledgements**

The experimental work was carried out by LNEC technicians Luís Nunes and João Ribeiro.

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