# Susceptibility to degradation of marbles present in Portuguese monuments

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## Abstract

The present article aims to evaluate the degradation susceptibility of marbles with a relevant presence in Portuguese monuments using various artificial aging procedures.

Consolidation treatments are used in conservation and restoration when stones show loss of cohesion and mass. The action of these treatments is usually studied in laboratory and often carried out on sound stones that do not truly represent naturally weathered stones. In this way, the work also intends to contribute to the study of aging procedures for the production of artificially decayed marble samples with characteristics closer to reality.

Different varieties of marbles were selected taking into account their availability and their presence in Portuguese monuments. The selected varieties were artificially aged by various laboratory procedures based on thermal action using a kiln and an heated plate to produce a homogeneous and gradual decay in-depth, respectively. The evaluation of their degradation susceptibility was based on the textural, physical and mechanical characterization of sound and artificially aged samples.

The results showed different degrees of alteration in the properties of the marbles, which were related to their initial intrinsic characteristics, especially to their textures. The different procedures of artificial aging by thermal action proved to be adequate and effective to produce aged samples. All types of marbles showed an increased open porosity and water absorption and a reduced integrity.

***Keywords**:* Marble, texture, artificial aging, thermal action, durability, Portuguese heritage.

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## 1. Introduction

High cultural, architectural and economic values are associated to build heritage. The progressive interest on its preservation led to the development of new technical capabilities, required for an effective and safe conservation of the heritage.

The exploitation of marbles in Portugal dates to Roman times. The abundance and quality of marble, as a building material, led it to be used in many Portuguese historic buildings that still prevail today. However, its exposure to severe environmental conditions and multiple aggressive agents over time trigger degradation processes, causing loss of cohesion, chemical changes and, consequently, a decrease in physical and mechanical properties.

“Sugaring” is one of the most common degradation patterns in marbles due to thermal actions. It consists of granular disintegration, i.e., the detachment of isolated grains, with appearance similar to the one showed by loose sugar grains on a stone surface (ICOMOS- ISCS, 2008). This phenomenon is caused because calcite and dolomite are anisotropic in terms of their thermal expansion coefficients. Thermal variations (daily and seasonal) lead to differential expansion/contraction of marble grains, which causes internal stresses along grains interfaces, causing micro-cracks and granular disintegration (Siegesmund et al. 2000).

Consolidation treatments are applied in order to restore cohesion between those grains and to restore the mechanical properties of decayed marbles, nevertheless, consolidation is a risky action due to its irreversible nature and the possibility of causing harmful effects to stones (Sena de Fonseca et al, 2017). The evaluation of the efficacy and harmfulness of consolidation actions is usually done in a laboratory, using sound stones. As the efficacy of these actions depend on the weathering level of the stone (Sassoni et al, 2011), the results obtained in laboratory do not correlate well with the ones obtained in-situ. To avoid these situations, the efficacy of consolidants should be evaluated on decayed stones samples. However, the reduced availability of naturally decayed samples with relatively homogeneous characteristics and the not viability of using stone elements from historic constructions justifies the need to investigate procedures for the artificial aging of marbles. Such procedures should allow to obtain relatively homogeneous samples and similar, as much as possible, to the decayed marbles found in historic buildings.

Several authors have developed different procedures to artificially age sound marbles by using ice-thaw cycles, salt crystallization cycles, acid attack, mechanical deformation and thermal shock (singular or cyclical) (Sassoni & Franzoni, 2014). The most effective and simple methodologies for marbles use thermal action (Ban et al, 2016). Recently proposed methodologies are based on heating marble samples using a kiln at high temperatures (300ºC - 400ºC) (Franzoni, Sassoni et al., 2013). An alternative method was also presented, in which the heating of the samples is done in direct contact with a heating plate in order to induce a temperature gradient in the sample, causing a gradual degradation throughout the sample (Delgado Rodrigues et al., 2007). On the other hand, many experimental studies seek to simulate the environmental temperature variations so the temperature in several studies do not exceed 100ºC (Fig. 1) (Franzoni and Sassoni, 2013).

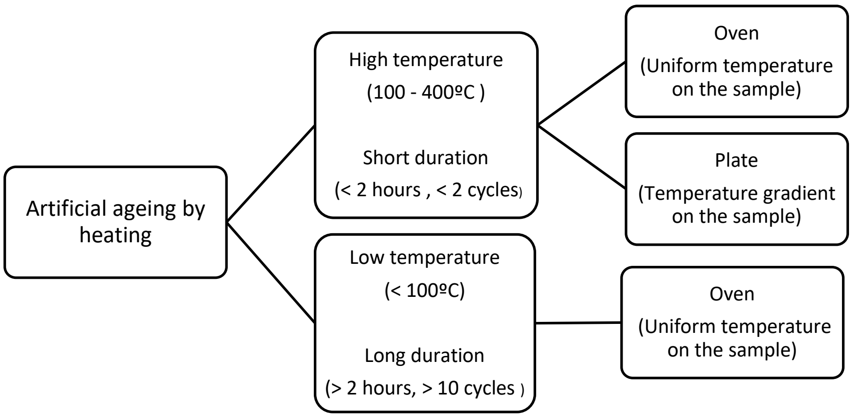


*Figure 1 – Artificial ageing by heating methodologies summary.*

The present dissertation aims to evaluate the influence of intrinsic characteristics in the susceptibility to degradation by thermal action of Portuguese marbles. For this purpose, several methodologies to trigger artificial aging were tested in different marble varieties. It also intends to contribute for the study of methodologies that produce artificially aged samples of marble in order to obtain decayed samples with characteristics suitable for the study of consolidation treatments in laboratory.

## 2. Materials and methods

### 2.1. Marbles

The marble used in this study came from four different areas, three of them located on national territory (Estremoz, Trigaches e Ficalho) and one located in Greece (Thassos):

1. Estremoz white marble ("E")
2. Trigaches light grey marble ("T1 to T9")
3. Ficalho white marble ("F")
4. Thassos white marble ("G")

Ficalho, Estremoz and Trigaches marbles are calcitic marbles, have an important presence in Portuguese built heritage and a vast set of different intrinsic characteristics, especially different textures. The Greece marble was selected because it is a dolomitic marble (Zeisig et al., 2002).

The experimental program used 20 samples (4x4x3cm) of Estremoz (E) and Trigaches (T4, T6/T7, T8 e T9); 5 samples (4x4x3cm) of Trigaches (T1 e T2), Ficalho (F) and Thassos (G) marble. One small slab of each type of marble (9x9x3 cm) was also used for textural analysis.

### 2.2. Marbles characterization

The intrinsic characteristics, texture and physical properties, of marbles were determined. Specifically:

- Fabric parameters (texture): Grain size, size distribution of grains, grain boundary configuration, preferred orientation of grains and intragranular microcracking;
- Open porosity, bulk density, real density and maximum water content;
- Water content after 48 hours of immersion;
- Capillarity water absorption coefficient.

Additionally, ultrasonic pulse velocity (UPV) was determined in dry and saturated 4x4x3 (cm) samples. Marbles ultrasonic speed characterization was made in two ways: i) Direct method with flat transducers (150 kHz), positioning the transducers in the center of opposite faces, according to direction 1 and 2, performing three measurements in each position; ii) Direct method with exponential transductors (54 kHz) and in different positions previously marked on the faces of the specimens (0.3 cm, 0.8 cm, 1.3 cm, 1.8 cm, 2.3 cm and 2.8 cm from the upperface). The maximum and minimum velocity of each marble were also used to calculate the anisotropy indices.

### 2.3. Aging procedures

The study of degradation susceptibility was carried out in 5 specimens (4x4x3 cm) of each variety of marble. Four experimental procedures named A, B, C and D were defined. Thermal action was based on the heating of the specimens in a heating plate and an oven. The procedures differ from each other in terms of temperature, time of exposure to heating and water saturation.

The four artificial aging procedures performed were as follows:

1. Procedure A - Heating dry samples using a heating plate at 300ºC for 5 minutes;
2. Procedure B - Heating dry samples using a heating plate at 300ºC for 2 minutes;
3. Procedure C - Heating dry samples in an oven at a temperature of 300ºC for 1 hour;
4. Procedure D – 1$^{st}$ phase: Specimens previously immersed in water for 48 hours heated in an oven at 200ºC for 1 hour; 2$^{nd}$ phase: Cooling of the specimens at room temperature; 3$^{rd}$ phase: Heating the specimens in an oven at a temperature of 400ºC for 1 hour.

The evolution of the marbles characteristics was made by comparing their properties before and after the thermal actions.

## 3. Results and Discussion

### 3.1 Sound marbles

#### 3.1.1. Fabric parameters (texture)

Table 1 shows the summary of the main fabric parameters analysed for marbles texture characterization.

Ficalho marble [F] has a very fine-grained texture, in which the crystals are not visible at macroscopic observation. Estremoz [E] and Thassos [G] marbles have both a fine-grained texture and the grains have an equigranular distribution, although they are different in the grain boundary configuration, polygonal and interbolar boundary, respectively. Estremoz marble [E] also has a slight preferential orientation of the grains. Trigaches marbles show a seriate to equigranular size distribution, characterized by the higher frequency of a determined grain size. Trigaches marbles are part of three texture categories: medium-grained, coarse-grained and very coarse-grained. The Trigaches varieties with smaller grains have a lobulated boundary configuration (T1, T2 and T4) and the other show lobulated to polygonal grains (T6/T7, T8 and T9).

*Table 1 - Summary of the main fabric parameters analysed for marbles texture characterization.*

| Marble | Texture | Average Grain size(mm) | Granular interface configuration | Size distribution of grains | Preferential grain orientation | Intragranular microcracking |
|---|---|---|---|---|---|---|
| **Ficalho [F]** | Very Fine | < 0,1 | Lobulated | Equigranular | No | No visible cracking |
| **Estremoz [E]** | Fine | 0,7 ± 0,3 | Polygonal | | Yes | |
| **Thassos [G]** | | 1,4 ± 0,3 | Lobulated | | | |
| **Trigaches [T1]** | Medium | 2,2 ± 1,1 | | Seriate to equigranular | No | Moderate |
| **Trigaches [T2]** | | 2,3 ± 0,8 | | | | Reduced |
| **Trigaches [T4]** | Coarse | 3,2 ± 1,5 | | | | |
| **Trigaches [T6/T7]** | | 5,0 ± 1,3 | Lobulated to polygonal | | | Moderate |
| **Trigaches [T8]** | Very Coarse | 6,1 ± 1,7 | | | | |
| **Trigaches [T9]** | | 7,0 ± 2,0 | | | | Reduced |

### 3.1.2. Physical properties

In general, open porosity of the studied marble samples are quite low, between 0.49 % and 0.86% (Trigaches marble [T9] and Thassos [G], respectively). Thassos marble [G] has the highest real and bulk density, while Ficalho marble [F] presents the most expressive standard deviation, which indicates that is the most heterogeneous variety (Table 2).

In terms of water absorption by capillarity, it was possible to verify that Thassos marble [G] has the highest value of water absorption coefficient by capillarity (CC). Estremoz marble [E] has the second highest value of CC revealing that fine-grained textures tend to have higher water absorption. Ficalho marble [F] reveals a significant variability of characteristics since it is the most heterogeneous. From Trigaches marbles, [T8] resulted a higher capillary absorption coefficient (CC) value may be due to its significant intragranular microcracking, although the remaining textural parameters are similar with other Trigaches marbles, particularly [T9]. In terms of asymptotic values (VA), the results follow the same trend as capillarity coefficients (Table 2).

### 3.1.3. Ultrasonic pulse velocity (UPV)

The propagation velocity obtained with flat transducers in the dry samples showed that Trigaches ultrasonic velocities was on 4000 and 5000 m/s range. Outside this velocity range were the results of the Estremoz [E], Thassos [G] and Ficalho [F] marble samples. In the case of Estremoz marble, it was verified that the values obtained vary according to the direction tested, showing maximum values around 4200 m/s and minimum values around 3200 m/s. This difference reveals an anisotropic behavior, and an average anisotropy index of 27% was determined. This behavior could be explained by the fact that Estremoz marble shows texture with a preferential orientation of its grains. Thassos marble is similar, presenting an anisotropic behavior with an average anisotropy index of 25%.

It is also verified that the ultrasonic velocity is higher in saturated than in dry samples, this is explain by the fact that ultrasonic velocity propagation is higher in water than in the air, when samples are saturated the pore system is filled with water instead of air, so ultrasonic waves travel faster across the marble samples resulting in higher UPV measurements (Table 2).

The uniformity of the data obtained with exponential transducers along the samples of all marble varieties reveals that there are no large voids or other significant heterogeneities along the Z axis of the samples (Figure 2).

*Table 2 - Summary of the main properties analyzed in the characterization tests.*

| Marbles | Average grain size (mm) | Grain boundary configuration | Porosity P[%] | Water content max. $W_{máx}$ [%] | CC [$g/m^2.min^{0.5}$] | VA [g/m2] | Average ultrasonic velocity (m/s) | |
|---|---|---|---|---|---|---|---|---|
| | | | | | | | Dry | Saturated |
| | | | Average | Average | Average | Average | D1 | D1 |
| Ficalho [F] | < 0,1 | Interlobate | 0,70 ± 0,17 | 0,26 ± 0,06 | 5,40 ± 2,63 | 92,90 ± 25,23 | 5622 ± 979 | 6170 ± 274 |
| Estremoz [E] | 0,7 ± 0,3 | Polygonal | 0,63 ± 0,07 | 0,23 ± 0,03 | 7,72 ± 1,11 | 72,84 ± 3,54 | 3768 ± 566 | 5939 ± 66 |
| Thassos [G] | 1,4 ± 0,3 | Interlobate | 0,86 ± 0,05 | 0,30 ± 0,02 | 9,71 ± 1,04 | 96,76 ± 5,94 | 4119 ± 553 | 6111 ± 347 |
| Trigaches [T1] | 2,2 ± 1,1 | Interlobate | 0,55 ± 0,03 | 0,20 ± 0,01 | 6,88 ± 0,88 | 66,16 ± 8,78 | 4132 ± 81 | 6041 ± 241 |
| Trigaches [T2] | 2,3 ± 0,8 | Interlobate | 0,57 ± 0,06 | 0,21 ± 0,02 | 6,37 ± 0,88 | 69,78 ± 4,47 | 4223 ± 231 | 5968 ± 244 |
| Trigaches [T4] | 3,2 ± 1,5 | Interlobate | 0,52 ± 0,10 | 0,19 ± 0,04 | 4,98 ± 0,64 | 54,90 ± 6,11 | 4553 ± 175 | 6309 ± 189 |
| Trigaches [T6/T7] | 5,0 ± 1,3 | Interlobate to polygonal | 0,55 ± 0,09 | 0,20 ± 0,03 | 4,84 ± 0,57 | 50,70 ± 2,84 | 4538 ± 196 | 6163 ± 177 |
| Trigaches [T8] | 6,1 ± 1,7 | Interlobate to polygonal | 0,62 ± 0,08 | 0,23 ± 0,03 | 6,51 ± 0,91 | 51,07 ± 4,08 | 4359 ± 268 | 6167 ± 364 |
| Trigaches [T9] | 7,0 ± 2,0 | Interlobate to polygonal | 0,49 ± 0,07 | 0,18 ± 0,02 | 5,40 ± 0,51 | 61,14 ± 1,41 | 3892 ± 695 | 5931 ± 995 |

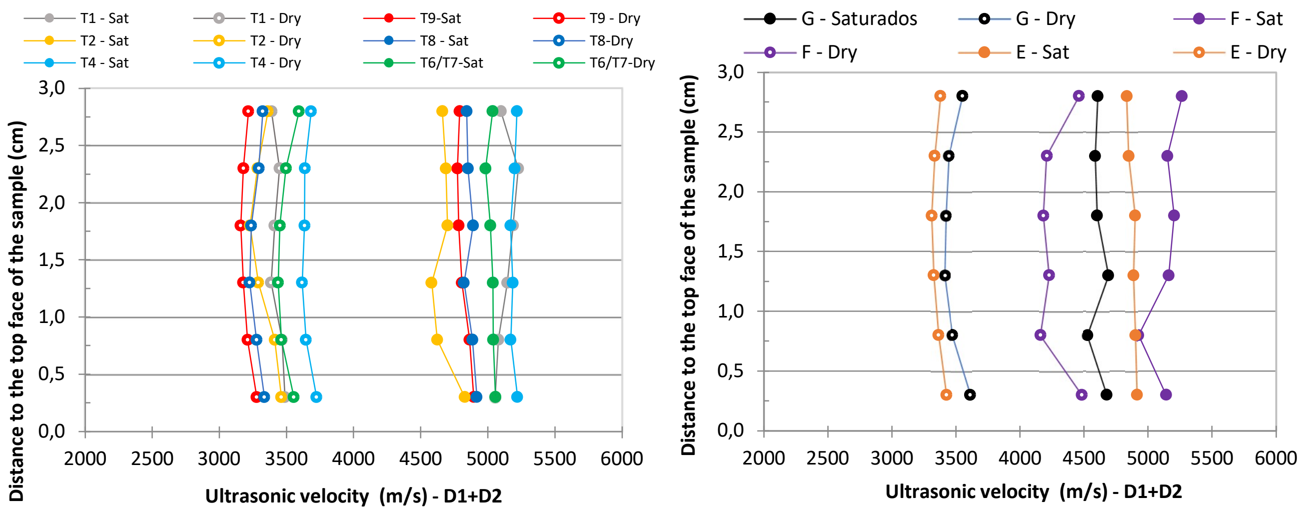


*Figure 2 – Ultrasonic velocity obtained with exponential transducers along the samples*

## 4. Alterations induced by the aging procedures

### 4.1. Marble selection for aging procedures

From the characterization of the tested marbles, a selection of marble varieties was made for the use on artificial aging procedures. The selection was made with the goal of identifying susceptibilities in marbles associated with different textural characteristics. The varieties selected for subsequent artificial aging were the varieties Estremoz [E] and Trigaches [T4], [T6/T7] and [T9]. The main properties considered in the selection of the marble varieties was the average grain size and its respective texture.

### 4.2. Alterations on Physical properties

In general, thermal action has increased open porosity and water absorption in all marbles.

Surface heating with procedures A and B promoted a change in open porosity of less than 30% and 10%, respectively. On the other hand, procedures C and D caused major changes in the open porosity of marble samples with increases of 100% and 300%, respectively (Table 3).

Estremoz samples [E] revealed less porosity variation than Trigaches. Procedure D caused a higher average open porosity variation for coarser-grain textured marble varieties (ΔP (E) < ΔP (T4) < ΔP (T6/T7) < ΔP (T9)). This indicates a greater tendency for coarse-grained textures to be more susceptible to change when exposed to thermal variations (Table 3).

In general, it was found that marbles, after aging, present water absorption curves by capillarity with initial sections with higher slope, revealing a higher speed of water absorption than the sound samples. The absorption curves of aged samples also have a higher capillarity water uptake. Marbles are characterized by pores with fissure form, causing a high fluid transmission capacity but low storage capacity (Delgado Rodrigues, 2007), so the enlargement and appearance of new microcracks due to thermal action, makes this phenomenon more evident, revealing a greater increase in transmission capacity (CC) comparing to water storage capacity (VA).

Procedure A was the one that caused more tenuous changes in capillary water absorption, followed by procedure B. The results obtained in the aged samples on procedure C revealed a significant increase in the initial water absorption rate and capillarity water uptake. In relation to sound samples, aged samples showed 10 times higher capillarity coefficients (CC). Procedure D was again the procedure that caused the most expressive modifications in marble samples.

Estremoz marble showed lower increase in capillarity water absorption when compared to the Trigaches samples. Although the asymptotic values (VA) of Trigaches marble after ageing were similar, there was a tendency for the varieties of fine-grained texture to present lower variations of CC (Table 3).

*Table 3 - Schematic representation of the level of alteration of different varieties by procedure.*

| Procedure | Marble | P | CC | VA | $V_{US\ Dry}$ | $V_{US\ Sat}$ | Color |
|---|---|---|---|---|---|---|---|
| C | E | Yellow | Red | Orange | Yellow | Green | Orange |
| | T4 | Yellow | Red | Orange | Yellow | Green | Orange |
| | T6/T7 | Yellow | Red | Red | Yellow | Green | Yellow |
| | T9 | Orange | Red | Orange | Yellow | Green | Green |
| D | E | Orange | Red | Red | Yellow | Green | Orange |
| | T4 | Orange | Red | Red | Orange | Yellow | Yellow |
| | T6/T7 | Red | Red | Red | Orange | Yellow | Yellow |
| | T9 | Red | Red | Red | Orange | Yellow | Green |

| Procedure | Marble | CC | ΔE* |
|---|---|---|---|
| A | E | Yellow | Yellow |
| | T4 | Orange | Green |
| | T6/T7 | Orange | Green |
| | T9 | Orange | Green |
| B | E | Yellow | Yellow |
| | T4 | Yellow | Green |
| | T6/T7 | Yellow | Green |
| | T9 | Green | Green |

- *P- Open porosity; CC - Capillarity coefficient; VA - Asymptotic value; $V_{US\ DRY}$ - Average ultrasonic propagation velocity obtain with flat transducers in dry samples; $V_{US\ SATURATED}$ - Average ultrasonic propagation velocity with flat transducers in Saturated samples;*
- *Green – No alteration; Yellow – Some alteration; Orange - High alteration; Red: Very high alteration.*

## 4.3 Alterations on ultrasonic pulse velocity (UPV)

Overall, a reduction in UPV was caused by the aging procedures for all marble varieties. Besides, the anisotropy of Estremoz marble suffered an increase of 41% and 66% when exposed to procedures C and D, respectively. When exposed to procedure D, marble Trigaches T9 showed a significant modification as well (anisotropy index after thermal action = 22%).

The UPV in the saturated aged samples was similar to the registered in the sound samples, excepting the samples aged by procedure D, where the variation was around 25% in Trigaches marbles and 10% in Estremoz. This further emphasizes the impact of procedure D on the microstructure and indicate that in addition to microcracks widened, new microcracks may have also been created (Table 3).

Taking this into consideration, the UPV in dry samples proved to be more sensitive to increased cracking and consequent degradation of marbles. In dry samples, the air in long and continuous microcracks delays significantly the propagation velocity of ultrasonic pulses.

Ultrasonic velocity tests using exponential transducers enabled an evaluation of the decay in-depth. This test was especially relevant for samples subject to ageing procedures A and B. The results showed that the difference in velocities before and after the thermal actions is greater in the side of the samples closer to the heating plate and this difference tends to decrease in-depth (Table 4).

Procedure A caused a change in the characteristic UPV values throughout T4 and T6/T7 samples thickness, while in E and T9 marbles obvious sings of decay were only detected up to 2,3 cm (Table 3). Procedure B caused similar results, however the overall difference between sound and aged marbles was more tenuous, which was expected due to the shorter time of exposure to the heat source (Table 4). A shorter exposure time caused a reduced degradation depth, it reached 1,8 cm in E, 1,3 cm in T4 and T6/T7 and 0,3 cm in T9 (Table 4).

*Table 3 - Schematic representation of the level of alteration over the marble thickness, based on the ultrasonic velocity obtained with exponential transducers in dry samples.*

| Marble | Dist. top (cm) | Ultrasonic velocity - Exponencial trans. | | Marble | Dist. top (cm) | Ultrasonic velocity - Exponencial trans. | |
|---|---|---|---|---|---|---|---|
| | | Procedure A | Procedure B | | | Procedure A | Procedure B |
| E | 2,8 | | | T6/T7 | 2,8 | | |
| | 2,3 | | | | 2,3 | | |
| | 1,8 | | | | 1,8 | | |
| | 1,3 | | | | 1,3 | | |
| | 0,8 | | | | 0,8 | | |
| | 0,3 | | | | 0,3 | | |
| T4 | 2,8 | | | T9 | 2,8 | | |
| | 2,3 | | | | 2,3 | | |
| | 1,8 | | | | 1,8 | | |
| | 1,3 | | | | 1,3 | | |
| | 0,8 | | | | 0,8 | | |
| | 0,3 | | | | 0,3 | | |

- *Green – No alteration; Yellow – Some alteration; Orange - High alteration; Red: Very high alteration.*

## 4.4 Colorimetric changes

In general, the artificial aging procedures C and D showed positive ΔL* values, which means that thermal actions caused a whitening of the samples surfaces. Whitening was superior in procedure D, indicating that a longer thermal exposure can cause a more significant whitening. Regarding procedure A and B, there was a darkening of the lower face of all samples, being more pronounced in procedure A. Δa* results were close to zero, showing no color change in the red-green axis. In terms of Δb*, results were positive, which means marble surfaces became slightly more yellow. Regarding the chroma difference (ΔC*) a general pattern is not identified, although is verified that Estremoz marble samples present positive values in all procedures, which reveals a higher saturation (intensity) of color.

## 5. Conclusions

The present article evaluated the influence of the intrinsic characteristics of Portuguese marbles on its susceptibility to degradation by thermal action and contributed to the study of testing procedures that allow the production of aged marble samples with characteristics similar to naturally weathered marbles, found in constructions.

The texture characterization using fabric parameters analysis allowed to understand their influence on marble degradation caused by thermal expansion.

All marbles studied presented reduced porosity (0.5% to 0.9%) and water absorption capacity, as well as high ultrasonic pulse velocity, characteristics of sound marbles. Dolomitic marble (G) revealed the highest real volume mass value (2800 kg/m$^3$), open porosity (0.9%) and water absorption capacity.

The ultrasonic velocity tests revealed to be a very sensitive tool to evaluate changes in marble characteristics, even when marbles are in a premature state of degradation.

Procedures A and B, that consist on the heating of the samples in a heating plate, have proved to be efficient in creating a gradual decay along samples, whose decay level and depth depends on the marbles characteristics.

Procedure C, that consists on the heating of dry specimens in an oven at 300ºC for one hour, proved to be a fast and effective method to obtain aged samples that cause modifications in the entire volume of samples, with relatively homogeneous characteristics. On the other hand, procedure D that heats samples previously immersed in water for 48 hours, proved to be adequate to reproduce severe cases of marble degradation.

The results revealed that marbles with coarser textures and polygon intergranular interfaces have greater susceptibility to degradation by thermal action. Consequently, the ageing procedures should be selected and adapted to the intrinsic characteristics of the marbles used, for example by carrying out preliminary tests, to assess the necessary levels and degradation patterns that suit better each study.

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