CHARACTERIZATION OF A 19TH CENTURY DECORATED GYPSUM PLASTER PIECE: THE ROLE OF MICROSCOPY

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Abstract

In spite of the lack of raw material of good quality, some scientific communities think that the use of gypsum plasters for interior plastering of walls and ceilings in Portugal could date from the roman period, its presence being more expressive from the end of the 18th century till the first half of the 20th. The quality and variety of gypsum plaster decorations in the Portuguese monumental and domestic architecture contrast with the rare studies on this subject. The lack of information on these materials and solutions for their restoration and conservation are being responsible for a fast loss of important patrimony.

The present research occurs in this context, where the characterization of the Portuguese historical gypsum plasters is the first step to reach the main purpose: the development of new repair gypsum plasters compatible with the old materials.

In this paper the results of the characterization of a particular gypsum plaster piece from the end of the 19th Century, simulating a *marmorino*, are presented and discussed. The sample studied (named PE4) belonged to a 19th century palace, situated in the Algarve, whose decorative program is considered to have the most valuable plaster works of the south of Portugal.

The mineralogical and chemical properties were determined using the analytical methodology developed by the authors Santos Silva *et al.* (2005).

The information's obtained by the visual observation of the sample PE4 are summarized in Table 1.

Table 1 – Visual observation of sample PE4.

Approximate dimensions	No. of layers	Layer identification	Visual observation	Aggregates presence
h = 110 cm w = 13 cm t = 4-8 cm	3	PE4/1(A)	Beige thick plaster layer, reinforced with sisal fibres and iron wire. This wire was rusted and its expansion caused a fracture in the piece, which, together with some humidity, led to its detachment from the wall.	Perceptible
		PE4/1(B)	Layer applied over PE4/1(A) and very similar to it although a little bit whiter, finer and less porous.	Perceptible
		PE4/2	Finishing purple-brown decorated plaster layer, simulating a *marmorino*, with what seemed to be aggregates of different colours (orange, light purple-brown). Very stiff.	Perceptible

The observation of the polished surface of the sample with the stereo-zoom microscope showed that PE4/1 was probably produced in two phases, one immediately after the other, as the interface between them is almost imperceptible. Although carefully observed with magnifications up to 60X, this suspicion couldn't be confirmed and the solution was to look directly into the sample and do some fracture tests with the aid of a chisel and a hammer. In fact, PE4/1 was broken in two parts, clearly showing a preferential fracture pattern through a smoother surface (the joint surface).

The perfection of the union between the layers PE4/1(B) and PE4/2 was also set off with the aid of stratigraphic analysis as well as the presence of two types of "aggregates": some with a light brown effect (like transparent quartz) and others with an orange colour. These last ones seemed to be pieces

of clay from bricks, used as aggregate to give the decoration effect required. These hypotheses were not confirmed afterwards, namely by XRD and TGA/DTA analyses.

In fact, XRD analysis showed that the mineralogical composition of each layer is very similar in what concerns the main mineral constituent (gypsum); TGA/DTA results also confirmed that. PE4/1 presents a small quantity of calcite and PE4/2 presents anhydrite and hematite. Surprisingly, in this layer there were no quartz aggregates or clay minerals, as expected from the stereo-zoom observation. This means that the "aggregates" were, in fact, simulated, and made of different gypsum plaster mixtures previously prepared.

The scanning electron microscopic (SEM) observations of the fractured surface of PE4 not only confirmed some of the facts already determined by other techniques, like the difference of porosity between the internal layers that constitute PE4/1 and the PE4/2 layer, but raised also new questions. The most intriguing and difficult to explain is the difference of morphology between these two parts (PE4/1 and PE4/2). In fact, through the observation at higher magnifications, one could think that they correspond to completely different compounds.

Besides porosity and morphology, other physical properties have been analysed, namely density, dynamic modulus of elasticity, capillary water absorption and compressive strength and they confirm the existence of significant differences between PE4/1 and PE4/2.

However, as said before, XRD and TGA/DTA analyses have shown that the main differences between their compositions are the presence of a small quantity of calcite in PE4/1, and of anhydrite and hematite in PE4/2. Could one, or both, of these factors be responsible for such big differences between them? A plausible explanation is that anhydrite was the main constituent of the calcinated gypsum used for preparing the PE4/2 paste.

It is not easy to prove this theory and many different experiments were and are still being made; the hydration of synthesized pure anhydrite is one of them. This experiment was performed in order to study the morphology of the resulting crystals. The mixture was prepared with an anhydrite water ratio of 1:1 (so that the crystals could have a free development) and the SEM images of the paste obtained present a similar morphology to that observed in some pores in the paste of sample PE4/2. This does not prove that anhydrite was present in high quantities from the beginning but at least puts forward the question of the crystals morphology in PE4/2 when they have space enough to grow. A new anhydrite paste, with a higher plaster water ratio, will be soon prepared in order to see if the morphology of the crystals formed changed and if it is more similar to PE4/2 paste.

The observation of thin sections by polarised light microscopy (PLM) was used to clarify the existence of anhydrite and the distribution of the red pigment (hematite) in the matrix of PE4/2, as well as the possible presence of carbonated calcite nodules in PE4/1.

In what concerns the presence of crystals of anhydrite in PE4/2, this could not be confirmed. However, the thin section observations were very valuable, namely in the clarification on how hematite was used to produce the different reddish effects present in the decorated layer.

This characterization work is still not complete but the results obtained are already enough to conclude that the manufacture of the decorated plaster piece studied was a very elaborated and complex work, which implied a deep knowledge of the materials used and a complete control of the techniques of execution.

The use of different microscopical techniques, like optical and scanning electron microscopy, was very helpful in the characterization performed. However, further investigations will still be carried on in a near future in order to try to clarify if anhydrite was really used on purpose as binder or only as a final setting time retarder.

References

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