



SEM and microanalysis in the study of historical technology, materials and conservation (SEM 2010)

Thursday 9 – Friday 10 September 2010
BP Lecture Theatre, British Museum

FIRST CIRCULAR: CALL FOR PAPERS

The deadline for submission of abstract is 19 March 2010

Title of paper:

Multi-analytical study of the pigments in 17th Century Portuguese Azulejos

The presentation is for an oral or poster presentation

Summary of the paper (in no more than 500 words):

Historic glazed tiles (azulejos) are an important part of the cultural heritage of Portugal. Although they are often associated with the 18th century blue-and-white period, the previous glazed tile production was characterized by a much richer palette, including, besides the blue, yellows, oranges, greens, purple, browns and a quasi-black colour used for contours. The aim of the study is to understand how the Portuguese tile painters combined the pigments and, with a limited number of oxides, achieved a wide palette, and how they used these colours. SEM observation and EDS analysis were very useful to determine the distribution and composition of the pigments in the glaze.

17th century Portuguese azulejos were manufactured by the majolica process, in which the colours were applied over a white tin-glaze. The distribution of colours in the glaze depends on the pigment used. Lead-yellow and all colours obtained from it, such as lemon yellow, fern-green and orange remain mainly at the surface of the glaze, whereas cobalt blue and copper green sink and often disperse in the glaze. The layering behaviour in the glaze ensures that in cases of superposition the colours do not mix.

We analysed the morphology and chemical composition of such colours using SEM/EDS analysis complemented by Raman spectroscopy and optical microscopy.

For SEM/EDS analysis polished cross-sections were prepared, being the observations made in backscattering image mode so as to enhance the lead content of the glaze, allowing a sharp visual separation from the biscuit, and also the lead content of some of the pigments, to allow a clear definition of their distribution. X-ray mapping was particularly useful and illustrative for obtaining the distribution of the pigments in the glaze. The colours that are dispersed in the glaze, however, are difficult or impossible to tackle with X-ray mapping because of their low content at any section.

The white glaze in 17th century Portuguese azulejos is not homogeneous and there are often crystals in the glaze that X-ray mapping helped to identify in all likelihood as quartz and feldspar. There are also gas bubbles trapped in the glaze and, in some cases, concentrated under the reddish iron colours. These bubbles come out clearly in the backscattering images. They often constitute a nest for biological activity.

We identified the chemical composition of all the colours used by Portuguese tile-makers of

the 17th century. The orange palette is obtained with yellow and iron red. The browns are obtained with manganese purple and iron red. The quasi-black colour is also a mixture of manganese and iron oxides.

There are doubts on whether the layering of the yellow, that is so evident in backscattered images, represents the result of painting over a first glaze before re-firing. The SEM images have not shown any layering in the glaze proper. We shall now proceed to reproduce the 17th century techniques to verify whether the pigment morphologies seen can, as we believe, be successfully duplicated with a single firing of the glaze. The reproduced tiles will again be subjected to SEM/EDS analysis.

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