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Analysis of reaction compounds in 15th century polychromes by micro-XRD

A set of 15th painting samples containing submillimetric layers and mixtures of compounds samples were analysed with SR-XRD at BM16 (Altarpiece by Antigó and Borrasà painters (MNAC museum), altarpiece by Bernat Martorell, altarpiece of Mare de Déu (Vinseum, Vilafranca del Penedès), wood carved from Sant Joan d'Arties –Val d'Aran). Data analysis has already been performed and we are currently working on the publication of the data.

The samples, $<1\text{mm}^2$, are made up of structures of submillimetric layers, and with the help of a microscope small fragments of these samples were selected. These fragments were placed on an adhesive support and diffraction measurements were collected in transmission geometry using an X-ray footprint of 30x30 microns. Also, for some samples, a map of compounds was obtained from thin cross-sections (200 microns in thickness) of the layered paints, in which case a footprint of about 20x50 microns.

The results proved to be very interesting. Relevant information for pigments was obtained from the different layers, some of the pigments characterized were found to be mixtures of compounds, also reaction products were determined such as metal carboxylates that are the product of interaction among the binding media and pigments. Moreover alteration products such as calcium and copper oxalates were also determined. Even impurities related to raw materials in the making of the pigments were discovered to be present.

Noteworthy is the result related to the samples from silver foils applied to altarpieces and wooden carved sculpture. The most frequently corrosion product found and present in larger amounts is silver chloride in its form chlorargyrite. The presence of sulphides is also observed although they are formed in lower quantities or missing if the silver foil is properly protected from the atmosphere. Silver sulphides appear as the monoclinic form of Ag_2S , acanthite in most of the cases studied. However, the development of sulphides seems to be related to the formation of some precursor, Ag_8S , evolving into the cubic form of Ag_2S , before the most stable monoclinic acanthite is formed. The alteration products present on the surface are silver chlorides only, which are driven to the surface by efflorescence through cracks or pores. This silver chlorides show crystalline growths through the cracks and pores causing tensions and flaking of the layers. The samples analysed correspond to artworks preserved under very different atmospheric conditions, in various geographic areas with different climates. However, the alteration compounds formed are similar indicating that the corrosion process follows similar mechanisms. The figure below shows some of the results obtained.

“SR-XRD and SR-FTIR study of the alteration of Silver foils in Medieval paintings “
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