

**Experiment title:**

Characterization of archaeological blue pigments by High Resolution Powder Diffraction.

Experiment number:

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Beamline: BM25A	Date of experiment: from: 28-6-2006 to: 4-7-2006	Date of report: 26-2-2007
Shifts: 9	Local contact(s): Germán CASTRO	<i>Received at ESRF:</i>

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Report:

Maya blue is an organo-clay (palygorskite-indigo) complex used as a pigment. It is the most commonly used blue pigment in Mesoamerica (VII-XVI centuries) in prehispanic times, and it is very different to any other pigment used in other parts of the world. Perhaps its most striking property is its resistance to acids: it is not destroyed (decolored) even if boiled in nitric acid. Although it is agreed by most researches that Maya blue is made with palygorskite, the use of other mineral phases, like sepiolite, instead of palygorskite for making blue pigments is not excluded. This proposal aims at the identification of the palygorskite or other mineral phases in archaeological micro-samples containing blue pigment. The samples are powder from rests of paintings which come from different sites.

Experimental method

X-ray powder diffraction patterns were collected at beamline BM25A with a fixed wavelength about 15 keV (0.8 Å) at room temperature. Powdered samples were placed inside a 0.5 mm diameter capillary, which were rotated during exposure. Data were collected in a continuous 2θ-scan mode from 3° to 40°, but in many cases only a scan of several palygorskite peaks was done.

Results obtained

Two main results were obtained from the collection of pigments characterized in this experiment. First, a set of Aztec blue pigments showed different composition in the mineral component, all sharing the same organic colorant (indigo). This has led to a publication in Applied Physics A. Secondly, it was shown that a blue

pigment contains palygorskite that could be different than those from known sources of palygorskite in Mexico, all of them in the Peninsula of Yucatan [1]. This has led to a new experiment: BM25-01-634.

The abstract of the paper accepted for publication in Applied Physics A follows:

SYNCHROTRON POWDER DIFFRACTION ON AZTEC BLUE PIGMENTS

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Some samples of raw blue pigments coming from an archaeological rescue mission downtown Mexico City have been characterized using different techniques. The samples, some recovered as a part of a ritual offering, could be assigned to the late Aztec period (XVth century). The striking characteristic of these samples is that it seems to be raw pigments prior to any use in artworks, and it was possible to collect a few μg of pigment after manual grain selection under a microscopy monitoring. All pigments are made by indigo, an organic colorant locally known as añil o xiuquilitl. The colorant is always found in combination with an inorganic matrix, studied by powder diffraction. In one case the mineral base is palygorskite, a rare clay mineral featuring micro-channels in its structure, well known as the main ingredient of the Maya blue pigment. However, other samples present minerals as sepiolite (a clay mineral of the palygorskite's family) and calcite. Another sample contains barite, a mineral never reported in prehispanic paints. We present the results of characterization using high resolution powder diffraction (HRPD) recorded at the European Synchrotron Radiation Facility (BM25A, SpLine Beamline) complemented with other techniques. All of them drove to consistent results on the composition. A chemical test on resistance to acids was done, showing a high resistance for the palygorskite and eventually sepiolite compounds, in good agreement to the excellent resistance of the Maya blue.

Reference:

[1] M. Sanchez del Rio, M. Suarez, E. Garcia Romero, "Palygorskita en la península de Yucatán. Contexto etno-histórico y arqueológico". Abstracts of the meeting of the Sociedad Española de Arcillas, Oviedo, Septiembre 2006