Laboratoire M.S.M.A.P. SARL

Microanalyse Sciences des Matériaux Anciens et du Patrimoine - Etude des objets d'art

STUDY OF A MAYAN COMMEMORATIVE STELAE (H.: 202 cm, W.: 72 cm) Assumed provenance and period: Mexico or Guatemala, Usumacinta River basin, Maya, 795 A.D.



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NOTICE

The purpose of this study, performed following established norms of scientific integrity, is to carry out scientific investigations to provide analytical data concerning the manufacturing mode of the studied cultural property, the possible weathering of its constitutive material, either natural or artificial and to characterize the deposits or surface treatments on the object.

The investigations based on optical examination and physicochemical analyses of samplings of the object; follow the methods briefly described in the report, which are long-standing standards and protocols employed by the scientific community.

Comparison of the results obtained with the data actually available in the scientific community allows concluding if the physical evidences of the object are consistent or not with its supposed origin and period of time.

These scientific investigations are carried out not taking into consideration historical research, iconography and stylistics statements about the object. Information about provenance, period or attribution of the cultural property are under the responsibility of the owner or its authorized agent and written in the report only as indication. However, this given information is used in the discussion for final statement.

OBJECTIVES

Study of a stone sculpture: *Commemorative Stelae with a Lord Figure* (H.: 202 cm, W.: 72 cm) Assumed provenance and period: Mexico-Guatemala, Usumacinta River basin, Maya, 795 A.D.

The material from which the object is made, tool traces, weathering, polychromy and any surface deposits were analysed to determine whether it was subjected to long-term natural weathering after it was carved, compatible with its assumed age.

SYSTEMS USED

Stereomicroscope; Scanning electron microscope (SEM) with back-scattered electron (BSE, composition contrast) and secondary electron (SE, topographical contrast) imaging coupled with energy-dispersive X-ray element analysis (EDX); Raman spectroscopy.

SAMPLES

The study was based on replicas of the sculpture surface and micro-sampling. They were taken from:

- the background of the relief, in the central part of the headdress on a brown deposit (R1);
- the inside of an incision between the vegetal motif and the top of the skull of the pedestal (R2);
- a blue-green colour preserved in the upper glyph of the left side column of glyphs (R3);
- black spots (pigment? crusting material?) on an ear ornament on the right side of the skull in the pedestal (R4);
- a red colour preserved in the carvings of the belt of the Figure (R5).

Five micro-sampling of the pigments were taken:

- the blue-green colour from replica R3 (P1);
- the green colour from third glyph of the left side column of glyphs (P2);
- the blue-green background of left side corner of the pedestal (P3);
- the red colour from the glyph at the bottom of the left side column of glyphs (P4);
- the red colour from the vegetal motif of the pedestal (P5).

The replicas surfaces were coated with carbon for the SEM examination. This operation is partly responsible for the carbon peak (C) observed on the elementary X-ray spectra.

STUDY RESULTS

The type of stone, weathering of the material, tool traces and deposits on the surface of the stone were examined in turn. Our observations and analyses, illustrated on the following pages, showed that:

- The *Stelae* is carved out of a *dolostone* rock combining *dolomite* and *calcite*. Dolostone is used in Mayan architecture and sculpture.
- The minerals (dolomite and calcite) on the surface of the object show specific phenomena dissolution, change in the chemical composition indicative of long-term natural weathering that occurred after the sculpture was carved.
- This weathering has caused the surface alteration erosion, cracks, chipping of the stone- visible on the stelae.
- Weathering took place in a humid environment with a high matter organic content, in the presence of micro-organisms.
- The brown deposit observed all over the object is of biomineral origin. Its mineralization (calcification) is evidence of a long-term contact between the stone and the biomineral deposit.
- These characteristics are consistent with the long-term, natural weathering of dolomitic limestone, in the presence of microorganisms.

- The blue-green pigment remaining on different part of the stelae is *Maya blue,* a synthetic pigment combining indigo dye with natural clay (*palygorskite*).
- The red pigment is a mixture of clay (ochre) with natural iron oxide micro particles.
- All these pigments are used in Maya painting techniques. Of special interest is the use of the synthetic Maya blue, which is characteristic of Mayan wall painting and polychromy.
- There is no evidence of the use of modern pigments.

These different characteristics support the object's assumed age and provenance.

1- PRELIMINARY EXAMINATION

The sculpture is made out of a beige microgranular stone with numerous millimetric to centimetric rounded fossiliferous inclusions (Fig. 1b, blue arrows).

Depending from the location on the stelae, the sculpture surface has various aspects: on the left side, the lower part of the stelae shows areas where the surface is very well preserved (glyphs, as in figure 1a, or the left side of the pedestal...).

The upper part of the stelae shows an overall erosion process more or less pronounced, which affects as well the sculpture as the background of the relief (Fig. 1b).



Figure 1: Detail views of the sculpture surface (**a**-, glyph of the left side, **b**-, belt area, **c**-, feathers on the right edge of the stelae).

The right edge of the stelae, from the top to the glyphs area shows the more pronounced alteration, with cracks, and some chipping of the stone (Fig. 1c).

The more eroded areas are the outer part of the knot of the belt, and some glyphs on the left side. In these areas, the sculpture is heavily eroded and the details of the carving are almost totally erased (Fig. 2a, E).

All over the object, a brown deposit is observed, well preserved in the recesses of the relief or the incisions of the designs (Fig. 2b, arrows).







Figure 2: Detail views of the sculpture surface (a- glyph at level of the knot of the belt, b- central part of the headdress).

Some traces of **polychromy** are still visible by places in the recesses of the stone, or in the best preserved areas of the sculpture, as the left side glyph column. Two colours are present: a blue-green (Fig. 3a and b arrows) and a red (Fig. 3c, orange arrow).



Figure 3: Detail views of the polychromy (**a**- and **b**- , upper glyph from the left side column of glyphs, **c**- fourth glyph from the left side). The box locates figure 3b.

In one of the glyphs, a recessed part which retains some red polychromy shows fine parallel grooves (Fig. 3c, blue arrows) which could be the only remaining tool traces on the object.





2 - TYPE AND WEATHERING OF THE MATERIAL USED TO MAKE THE OBJECT

In the absence of a petrographic analysis of a thin section of this stone, elemental EDX analysis of this material, combined with examination of the type and morphology of the mineral phases, show the association of crystalline phases of dolomite (Fig. 4, MgCa) and calcite (Fig. 4, Ca).

These characteristics identified it as a *dolomitic limestone (dolostone).*



Figure 4: Detail view (SEM, BSE, x500) of the mineral phases constituting the stone. Replica R2.

Dolostone is used in Mayan architecture, as for example in the funerary crypt of the *Temple of the Inscriptions* of Palenque (1). This type of stone is present in the Usumacinta River basin.

Examination of the replicas reveals weathering of the mineral phases on the object's surface:

- **Calcite** (Fig. 5a, Ca) exhibits preferential and extensive dissolution phenomena compared with dolomite (Fig. 5a, MgCa).



Figure 5: Detail views (SEM, BSE) of the mineral phases constituting the stone (a-, x250) and of a weathered calcite phase (b-, x1800) and EDX analysis spectrum (c-) of the weathered calcite phase. Replica R2. The box locates figure 5b.

Examination of weathered calcite phases clearly shows micromorphologies consisting of numerous hollow spheres (Fig. 5b, arrows) evocative of structures of organic origin, such as bacteria.

EDX analysis (Fig. 5c) shows the main presence of calcium, with traces of iron, silicon, magnesium, aluminium, phosphorus and potassium.



The presence of traces of phosphorus in association with iron and silicate suggests a possible biomineral origin weathering, as in bacterial and algal biofilms, or microbial accumulations (2, 3) where phosphorus and iron (depending of their availability in the immediate surrounding) are often combined.

- **Dolomite** (Fig. 6a, MgCa), more weathering resistant, exhibits less marked alteration phenomena than calcite (Fig. 6a, Ca) but also shows traces of biomineral origin weathering (Fig. 6a, arrows, 6b).



Figure 6: Detail view of the weathered stone (a-, SEM, BSE, x1500) and EDX analysis spectrum (b-) of a weathered dolomite phase. Replica R3.



Widespread weathering induces extensive microfragmentation of the stone (Fig. 7), which causes the surface erosion and the cracks and chipping of the stone when the phenomenon is more pronounced.

Figure 7: Detail view (SEM, BSE, x900) of the weathered stone. Replica R5.

These characteristics are consistent with long-term, natural weathering of dolomitic limestone, in the presence of microorganisms. The weathering of the surface of the stelae took place after it was carved.

3 - STUDY OF THE POLYCHROMY

The blue-green pigment corresponds to more or less rounded microcrystallines phases (Fig. 8a, B, 8b), in direct contact with the stone (Fig. 8a, S).





Figure 8: Detail views (SEM, BSE, **a**- x1300, **b**- x4500) and EDX analysis spectrum (**c**-) of the blue-green pigment material. The box locates figure 8b.

The detail view (Fig. 8b) shows a fibrous material with the main presence of silicon, magnesium and calcium, associated with aluminum, iron, potassium, titanium, sulfur and chlorine.

The fibrous morphology and the silicon, aluminum and magnesium content suggest the presence of *palygorskite*, a clayey mineral typically observed in *Maya blue*. Calcium and iron can also be associated to the Maya blue pigment.



Traces of sulphur, chlorine, potassium and titanium result from the immediate environment of the analyzed phase.

The blue-green pigment from samples P1, P2 and P3 (Fig. 9) was analyzed in Raman spectrometry. Figure 9b shows the blue-green pigment in direct contact with the stone.



Figure 9: Detail views of sample P2 (a- stereoscopic microscope, x40) and P3 (b-, optical microscope, x50).

Raman spectrometry analysis of the blue-green pigment of the sample P2 (Fig. 10, green spectrum) reveals the presence of *indigo* (Fig. 10, black spectrum). Common peaks are index-linked to the green spectrum.



Figure 10: Raman analysis spectrum of the blue-green pigment of sample P2 (green spectrum), compared with the reference spectrum of indigo (black spectrum).

Raman spectrometry analysis of the blue-green pigment in samples P1 and P3 also shows the presence of indigo.

The blue-green colour corresponds to the Maya blue pigment, a synthetic pigment and compound of organic and inorganic constituents, primarily indigo dye combined with palygorskite.

Identification of this type of blue pigment is characteristic of Maya polychromy (4).

The color change, from blue to blue-green depends on change in the proportions between indigo and clay or the oxidation state of indigo molecule during the pigment synthesis (4, 5, 6).

The red pigment was observed on surface replica R5 (Fig. 11a, arrows), in direct contact with the stone (Fig. 11b, S)





Figure 11: Detail view of the replica R5 (**a**-, stereoscopic microscope, x43), detail view (**b**-, SEM, BSE, x3000) and EDX analysis spectrum (**c**-) of the red coloration material.

It consists of the association of **iron oxides microparticles (hematite?)** (Fig. 11b, arrows) and a clayish silicate and iron-rich material, very probably **ochre** (Fig. 11b, O, 11c).

Iron oxides and ochre are red natural pigments usually used in Maya painting.



Raman spectroscopy was performed on samples P4 and P5. However, for both, the too low Raman signal doesn't allow the exploitation of the results.

4 - STUDY OF THE SURFACE DEPOSITS

The brown deposit is more abundant on the top of the sculpture. It was sampled on replica R3 (Fig. 12a, white arrows).



Figure 12: Detail view (**a**-, stereoscopic microscope, x10) of replica R1, detail view (**b**-, SEM, BSE, x1800) and EDX analysis spectrum (**c**-) of the brown deposit.

SEM-BSE examination (Fig. 12b) shows a microcrystalline material and micromorphologies consisting of lining-up hollow spheres (Fig. 12b, orange arrows) evocative of structures of organic origin, such as bacteria.

EDX analysis shows main presence of calcium, silicon, aluminium, iron and magnesium, with traces of potassium, titanium and phosphorus, which could come from a sedimentary (?) environment of the object.





This brown deposit is visible all over the sculpture and always consists on microbial accumulation associated with elements of the environment of the object (Fig. 13).



Figure 13: Detail views (SEM, BSE) (a-, x4000, Replica R3, b-, x1300, Replica R2) of the brown deposit.

The presence of microorganisms indicates that it originates from a humid environment, necessary for the development of bacteria.

Microorganisms (Fig. 13, 14, arrows), in direct contact with the weathered stone (Fig. 14, S), appear partially (Fig. 13b) or completely (Fig. 13a, 14) mineralised by the rock (calcification), explaining the very high calcium detected (Fig. 12c).

These characteristics are evidence of long-term contact between the stone and the biomineral deposit.

The lighter deposit (Fig. 12a, orange arrows) corresponds to the association of the brown deposit and fragments of completely weathered stone

Figure 14: Detail view (SEM, BSE, x3000) of the weathered stone. Replica R5.



The dark crust, sampled in replica R4 (Fig. 15a), correspond to the amorphous texture material which appears with low atomic contrast in SEM-BSE observation (Fig. 15b).



Figure 15: Detail view (**a**-, stereoscopic microscope, x17) of the replica R4, detail view (**b**-, SEM, BSE, x1000) and EDX analysis spectrum (**c**-) of the dark crust.

EDX analysis (Fig. 15c) reveals an organic material with high calcium, silicon, aluminum and sulfur content and iron, phosphorus, magnesium, titanium and zinc.

The high calcium content results of stone fragments (Fig. 15b, arrows) included in the dark material.

This material is not a pigment and could be a modern pollution, (soot?).



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