Scientific Analysis of Pigments in 20th Century Paintings for Selected Historical Churches of the Bohol, Philippines

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ABSTRACT Through a combination of scientific analytical methods, the coloring materials used in 20th century paintings in historical churches of Baclayon, Dauis and Loay, which are municipalities in Bohol, Philippines, were studied. Inorganic pigments were identified using SEM-EDS and XRD. Iron-based pigments were commonly found in the paintings, yielding dark yellow and brown colors. Zinc oxide was identified as the white pigment in the ceiling paintings of Dauis Church and Loay Church, while titanium dioxide was detected in the column painting in Baclayon Church. Organic analysis showed the presence of Pigment Yellow 3, a synthetic organic pigment. Paint layers, as well as other components of the samples such as grounds and metal leaves, were examined microscopically. It was observed that different types of grounds were applied on different types of surfaces. Moreover, organic pigments were found in combination with white extender materials. Microscopic examination also revealed alterations in the artworks, such as the overpaint layer found in the samples from Baclayon Church cornice and the imitation metal leaf layers applied over the original gilt surface in the Loay Church *retablo*.

Key Words: Pigment, Historical churches, Scientific analysis, Bohol, Philippines

1. Introduction

Massive coral stone churches have been built at different times in almost all 47 towns of the province of Bohol. This island province is known for maintaining many of its colonial churches constructed as early as the 18th century, during the time of Spanish colonization in the Philippines (Torralba and Esguerra, 2008).

The current coral stone building of the Our Lady of the Immaculate Conception Parish Church in Baclayon, Bohol was built in 1727 under the Jesuits, who sowed the seeds of Catholicism in the province. Decades later, after the expulsion of the Jesuits from their missions in the Philippines, the Augustinian Recollects took over their territories in Bohol and placed their headquarters in Baclayon Church for some time. The Parish of the Holy Trinity Church in Loay, built in 1822, was one of those

buildings erected during the time of the Recollects in Bohol. Although the Our Lady of the Assumption Parish Church complex in Dauis has already existed a century earlier, the construction of its current coral stone church started in 1863 but was left unfinished by the end of the Spanish colonial regime. The construction was resumed by the Diocese of Cebu, which, by the 19th century, was already consisted mostly of Filipinos, until 1923 (Jose, 2001). The ceilings and some interior walls had been decorated with trompe l'œil paintings. Most of these paintings were originally made by teams of Canuto Avila and Raymundo Francia, who were officially commissioned by the Bishop of Cebu in the early 1920s to paint biblical scenes and images of saints on the galvanized iron sheet ceilings of Bohol colonial churches (Torralba and Esguerra, 2008).

As witnesses to major events in the history of their respective communities and testament to both European

influence and Filipino creativity, these churches have been declared as Historical Markers and National Cultural Treasures by the cultural agencies of the Philippines.

At present, many of these interior paintings remain intact, albeit with some later retouching, like those in Loay Church (Figure 1) and in Dauis Church. In Baclayon Church, nothing remains of Avila-Francia ceiling paintings, which were replaced with monochromatic sheets. Later paintings made in the 1950s can be seen over its crossing (Jose, 2001; Torralba and Esguerra, 2008).

Spanish colonizers used art to propagate Catholicism. It was through this that Western painting techniques were introduced to the Philippines. For a long time during the colonial period, Philippine paintings were almost used exclusively for religion (Hernandez, 2015). Despite the expanse of religious artworks across the province, literature about these is limited. Moreover, scientific research on

the material components of paintings in the Philippines, in general, is still in its infancy.

Much attention has been given to these churches, among the other heritage structures in Bohol and nearby provinces, because of the damages caused by a 7.2-magnitude earthquake in October 2013. The artworks studied in this paper were selected because the stakeholders of these parishes have considered them as part of the original church structures. Since there is currently no previous record of analysis on these artworks, this study was done to describe their states before they are subjected to restoration. Therefore, this study was conducted to shed light on the material components and to provide better understanding of the techniques employed in making the religious paintings for heritage churches in the municipalities of Baclayon, Dauis and Loay.

Figure 1. View from the choir loft of the Parish of the Holy Trinity in Loay, showing the church altar and ceiling paintings.

2. Materials and Methods

2.1. Materials

The samples used in the study were carefully collected from the detached parts or loose edges of the paintings. Fragments or powdered pieces were obtained from three historical churches, particularly from: (1) the column painting in Baclayon Church near its altar; (2) the interior cornice in Baclayon Church; (3) the ceiling painting over

the left side altar of Dauis Church; (4) a retrieved portion of the ceiling painting in Loay Church; and (5) the *retablo* in Loay Church. Descriptions of the samples are summarized in Table 1 photographs of the artworks are shown in Figure 2~4.

2.2. Methods

The collected samples were separated by color, and then preliminary observations and photographic documentation

Table 1. Sampling summary for the paintings in three historical churches in Bohol

Provenance	Sample No.	Area of sampling	Sample description
Baclayon Church	BAC 1	Dark yellow ornament of the Corinthian column capital	· Mostly dark yellow, with occasional yellow and brown fragments. · White ground layer.
	BAC 2	Red pattern on the cornice	· Patches of green and yellow overpaint on the red surface. · Light brown ground layer with some fibrous materials.
	BAC 3	Light blue overpaint on the cornice	· Light blue paint on white ground.
	BAC 4	Blue pattern on the cornice	· Occasional white streaks on the blue surface.
	BAC 5	Red portion of the Corinthian column capital	· Thick layer of ground/plaster.
	BAC 6	Wooden peg attached to the cornice	· Flat side of the peg covered with paint (mostly green).
Dauis Church	DAU 01	White portion of the inner frame	· Light gray fragments with white ground layer. Cupped.
	DAU 02	Brown portion of the inner frame	· Yellowish light gray powder.
	DAU 03	Dark area near the mitered joint of the inner frame	· Gray powder.
	DAU 04	Shadow of the decorations on the inner frame	· Dark gray powder.
	DAU 05	Light portion of clouds	· Light gray fragments with white ground layer. Cupped.
	DAU 06	Shaded portion of clouds	· Brownish gray powder.
	DAU 07	Highlights on the outer frame	· Yellow fragments with white ground layer. Cupped.
	DAU 08	Main color of the outer frame	· Reddish-brown flakes without ground layer.
	DAU 09	Dark portion of the outer frame	· Brown powder.
	DAU 10	Shadow of the outer frame	· Dark gray powder.
Loay Church	LOA 1	<i>Retablo</i>	· Metal leaf on yellow base coat and light red ground with white patches.
	LOA 2	Ceiling painting - main color of the frame	· Yellowish brown powder.
	LOA 3	Ceiling painting - outer border of the frame	· Blue-green powder.
	LOA 4	Ceiling painting - shaded portion of the frame	· Brown powder.
	LOA 5	Ceiling painting - highlighted portion of the frame	· Light brown powder.

Figure 2. The interior cornice and top of a column near the altar in Baclayon Church.

Figure 3. Collection of samples from the Dauis Church ceiling painting.

Figure 4. Retrieved parts of the Loay Church ceiling painting.

were done using optical microscope (Axiotech 100, Carl Zeiss, Germany) with a camera (D5200, Nikon, Japan) accessory. Microscopic images were scaled and measured using microscope software (AxioVision LE, Carl Zeiss, Germany). Selected fragments were embedded in epoxy

resin and their cross-section images were obtained, observed and processed using the said equipment and software. Elemental composition data were obtained using SEM (Scanning Electron Microscope, JSM-IT300, JEOL, Japan) with EDS (Energy Dispersive Spectrometer, X-MAX^N, Oxford, England). The samples were sputter-coated with platinum prior to SEM-EDS analysis.

Crystallographic phase analysis was carried out using XRD (Multi Purpose X-ray diffraction, Empyrean, PANalytical, Netherlands). The diffractograms were analyzed using High Score Plus software and ICSD database.

For the analysis of organic pigments, the pyrolyzer (EGA/PY-303D, Frontier Lab, Japan) was used together with GC/MS (Gas chromatography-mass spectrometry, 7890A GC/5975C MSD, Agilent, USA). Pyrolysis was done using at 600°C for 0.20 min. The pyrolyzer-chromatograph interface temperature was 300°C. The helium carrier gas flowed at the rate of 0.5 ml/min with a 10:1 split through a 30 m × 0.25 mm × 0.10 μm dimethylpolysiloxane column. The oven was programmed to increase from 50°C to 300°C at the rate of 10°C/min. A final temperature of 300°C was held for 5 min. MS detection covered a range of m/z = 33 to m/z = 550.

3. Results and Discussion

Based on the results of the physico-chemical methodologies applied to the samples, pigments corresponding to the distinguishable colors in each painting were analyzed for their characteristics. These analyses yielded information not only on pigments but also on the other materials present in the samples, giving an insight on the techniques applied on the paintings, as well as possible alterations done on them.

3.1. Pigments

The pigments observed in the samples through analytical methods are summarized (Table 2, Figure 5).

First, it was observed that hematite and goethite were used commonly used in red and dark yellow, brown, and yellowish brown colors.

Table 2. Minerals identified in the samples of the three Churches by XRD analysis

Provenance	Color	Location	Pigment
Baclayon Church (cornice & wall painting)	Yellow	Wall - highlights on the column capital	Pigment Yellow 3
	Dark yellow	Wall - column capital	Goethite
	Reddish-brown	Wall - background	Hematite, Goethite
	Red	Wall - background Cornice - red leaf patterns	Fe-containing pigment
	Blue	Cornice - blue leaf patterns	Lazurite
	Green	Cornice - overpaint	Organic pigment
Dauis Church (left side altar ceiling painting)	White	Clouds and inner frame	Zincite
	Yellow	Highlights on the main frame	Organic pigment
	Dark yellow	Main frame	Goethite
	Brown	Inner frame and shaded portions of main frame	Hematite, Goethite
	Black	Shadows on inner frame	Fe-based or carbon-based pigment
Loay Church (ceiling painting)	Yellowish brown	Main color of the frame	Hematite, Goethite
	Brown	Shaded part of the frame	Hematite, Goethite
	Green	Outer border	Organic pigment

These materials were detected in the dark yellow paint layers of samples BAC 1B and BAC 1C, indicating the use of yellow ochre in Baclayon Church cornice and column paintings. These iron oxide minerals were also found in the red and reddish-brown layers in the latter sample. Based on their color on the surface of the fragment, the reddish-brown pigment was identified as burnt sienna. Ochres and siennas belong to a family of earth pigments whose major ingredient is iron oxide. When its anhydrous form, hematite (Fe_2O_3) is the most abundant, it exhibits red color. Brown or yellow are shown when the hydrated form, goethite [$\text{FeO}(\text{OH})$], is dominant. The colors of these pigments vary according to the degree of hydration of iron oxide, the relative amounts of these iron oxide species, as well as amounts of other compounds such as other oxides and aluminosilicates (Bikiaris *et al.*, 1999; Genestar and Pons, 2005). These earth pigments were also found in the ceiling paintings in Dauis Church and Loay Church, giving dark yellow, yellowish brown and brown colors.

Hematite and goethite were also observed in areas with mixed colors. For example, in the colored samples DAU 08 and DAU 09, which were taken from the main

Figure 5. Results of XRD analysis for pigments in cornice and wall paintings of Baclayon Church (1: calcite; 2: rutile; 3: hematite; 4: goethite; 5: quartz; 6: Zn-S phase; 7: zincite; 8: barite; 9: lazurite; 10: $\text{Ca}(\text{HCOO})_2$ phase; 11: gypsum).

color and shadow of the outer frame in the Daus Church left side altar painting, these minerals were detected, along with the white pigment, zinc oxide (ZnO). The detection of multiple pigments in these samples indicates the use of a few colors of paint to produce different shades. This was also observed in the comparing the midtone and shadow of the clouds. For the samples DAU 05 and DAU 06, which were taken from the light and dark portions of the clouds, their difference is the presence of other pigments in the darker sample, DAU 06. Fe found in this sample, as well as other dark-colored samples taken from other paintings, may indicate the use of Fe-based black pigment like mars black. Moreover, this does not exclude

Figure 6. Results of XRD analysis for pigments in left side altar ceiling painting of Daus Church (1: zincite; 2: quartz; 3: barite; 4: lazurite; 5: hematite; 6: goethite; 7: gypsum).

the possibility of using carbon-based black pigments. Nonetheless, observing hematite and goethite in these samples suggests the addition of these pigments to produce a brownish tint.

Next, white inorganic pigments were observed not only in background layers but also in pictorial layers, sometimes in combination with other pigments. Zinc oxide was found in the samples DAU 01 and DAU 05, which were taken from areas in the painting that appear white. It was also found in the samples from the retrieved portion of the Loay Church ceiling painting, similarly with the colored samples from the painting in Daus Church. Likewise, the final appearance of the colors of the painting could be the result of mixtures of pigments. On the other hand, titanium dioxide (TiO₂), a white pigment, was observed in samples from the Baclayon Church column painting. The presence of these pigments indicate that the ceiling paintings were made not earlier than 19th century, while the column painting in Baclayon Church not earlier than 20th century. Zinc oxide and titanium

Figure 7. Results of XRD analysis for pigments in ceiling painting of Loay Church (1: Cu; 2: Au-Ag; 3: crocoite; 4: zincite; 5: quartz; 6: feldspar; 7: hematite; 8: goethite; 9: barite; 10: mica group mineral; 11: Ag-alloyed material).

dioxide have been used extensively as white pigment in paint, especially after the discovery of the hazards of lead white and the imposed regulations on its use (Feller, 1986; Fitzhugh, 1997).

White pigments zinc oxide, barite and titanium dioxide were detected in the sample of the green overpaint from the Baclayon Church cornice. Barite (BaSO_4) is used as extender for white paint and base for the preparation of some colored paints. It is also an ingredient in the lithopone pigment, which is made by the co-precipitation of barium and zinc to form BaSO_4 and ZnS (Feller, 1986).

For the sample BAC 4 from the Baclayon Church interior cornice, which has a paint layer that is mostly blue, lazurite and barite were detected. Lazurite, a sulfur-containing sodium silicate mineral, is an essential component

of the blue pigment, ultramarine (Plesters, 1993). Traces of white were found on the blue paint fragments, most probably from the new ground layer, in which Ba peaks were observed from SEM-EDS.

In the green overpaint layer, coloring material except for barite, rutile and zincite was not detected. Since the colored pigment cannot be identified using SEM-EDS and XRD, it is possible that it is organic. Moreover, it is also possible that the green color came from a mixture of two or more pigments. Likewise, except for a small amount of Cu, no mineral pigments were also found in the green sample from the Loay Church ceiling painting. One possible explanation is that the green color comes from a Cu complex with an organic ligand. White pigments such as zinc oxide were also detected in the yellow paint

Figure 8. Pyrogram of the yellow portion of BAC 1B at 600°C (1: *o*-chloroaniline/*p*-chloroaniline, 9.1024 min; 2: 1-chloro-2-isocyanatobenzene; 3: 4-chloro-2-nitrobenzenamine).

Figure 9. Structure of PY3 and its pyrolysis products.

Table 3. Layer construction and major elements of samples collected in Baclayon Church column painting

Cross section image	Layer	Description	Major elements
Paint layer (yellow)	Ca, Ti, Si, Al, Mg
	Paint layer (dark yellow)	Fe, Ti, Ca, Si, Al, Mg
	Translucent interlayer	Ca, Ti, Si, Mg
	Ground layer (white)	Ti, Si, Ca, Mg
Paint layer (reddish brown)	Ca, Fe, Mg, Si
	Paint layer (brown)	Fe, Ti, Ca, Si, Al, Mg
	Paint layer (dark yellow)	Fe, Ti, Ca, Si, Al, Mg
	Translucent interlayer	Ca, Mg, Ti, Si
	Ground layer (white)	Ti, Si, Ca, Mg

fragment from the ceiling painting of Dausi Church, and other substances were not identified. It is possible that the yellow pigment is an organic compound.

The use of organic pigments is another feature observed in the paintings studied. Some pigments were not identified using SEM-EDS and XRD, including the yellow pigment in the Baclayon Church column painting. Assuming that the colors of the unidentified pigments come from organic chromophores, instead of metal-ligand complexes, they were labeled as organic pigments in Table 2.

With this assumption, the yellow portion of BAC 1B was subjected to py-GC/MS. The pyrogram of this sample is shown in Figure 8. The compounds corresponding to

the peaks marked in pyrogram are characteristic to the yellow pigment, PY3 (CI 11710). The structure of the monoazo synthetic organic pigment is shown in Figure 9 (Sonoda, 1999).

Additionally, analyses of the samples taken from the gilt surface of the Loay Church *retablo* revealed that they contain Au, Ag and Cu, which are elements typically used in the manufacture of gold leaf. The Al and Cu metal layers indicate the use of imitation silver and gold leaves, which are cheaper alternatives to the original metals. Aside from the metal leaves, some pigments were also found in the preparation layers, including crocoite in the yellow base coat layer under the imitation leaves.

Table 4. Layer construction and major elements of samples collected in Baclayon Church interior cornice

Cross section image	Layer	Description	Major elements
Overpaint (green)	Zn, S, Ti, Ca
	New ground layer (white)	Ba, Zn, S
	Original paint layer (red)	Si, Fe, Al, K, Mg, Ca
	Ground layer (light brown)	Si, Al
Overpaint (green)	Zn, S, Ti, Ca
	New ground layer (white)	Ba, Zn, S
	Original paint layer (blue)	Si, Al, Na, S, K
	Ground layer (light brown)	Si, Al

Table 5. Layer construction and major elements of samples collected in Daus Church left side altar ceiling painting

Cross section image	Layer	Description	Major elements
Paint layer (gray)	Zn
	Ground layer (white)	Zn

It can be observed that among the coloring materials used in the three church buildings, Fe-based pigments and white pigments are widely used in various colors, and some have used unique coloring materials for each painting of the church. Of course, it is difficult to make a direct comparison between these artworks because they are of different media.

3.2. Preparation layers

The layers of selected paint fragment samples were microscopically examined and elemental analysis was performed on each layer to identify different materials such as constituent pigments, as well as other materials such as grounds and metal leaves. Through these results,

Table 6. Layer construction and major elements of samples collected in Loay Church *retablo*

Cross section image	Layer	Description	Major elements
Metal leaf (yellow)	Cu, Zn
	Metal leaf (green)	Al
	Base coat (yellow)	Pb, Ca, Cr
	Ground (white)	Ti, Al, Si, Ca
Metal leaf (yellow)	Au, Ag, Cu
	Ground (red)	Si, Al, Fe
	Ground (translucent)	Si, Al
Overpaint (white)	Ti, Al, Si, Ca
	Metal leaf (gray)	Al
	Base coat (yellow)	Pb, Ca, Cr
	Ground (white)	Ti, Al, Si, Ca
Metal leaf (yellow)	Au, Ag, Cu
	Ground (red)	Si, Al, Fe
	Ground (translucent)	Si, Al

the overlaying of materials, as well as modifications to an original or older version of the painting, were observed.

First, to accurately analyze the status of the coloring layers, the cross-sectional states of the painting samples collected from the three church buildings were photographed, and each layer was illustrated based on these images (Table 3–6).

As a result of examining the ground layer, for the paintings in Baclayon Church, samples obtained from the column can be distinguished from those obtained from the cornice because of their white ground layers (Table 3). Elemental and mineral analysis showed that these layers contain calcite (CaCO_3) and titanium dioxide (TiO_2), which are white pigments that are typically used in formulations of primer paints and grounds. However, even though they are paintings in the same church building, it has been confirmed that the ground used for each artwork is different. In BAC 2 and BAC 6 (Table 4), which were taken from the cornice of Baclayon Church, emission peaks of Si and Al were observed on the ground layer, which is the light brown layer at the bottom. This suggests the application of clay-based ground on the wooden surface of the cornice. Several layers were found above the ground layer, including the original pictorial layer, a new white ground layer and an overpaint layer.

From the examination of fragments obtained from the ceiling painting in Daus Church, it was observed that the base layer consists of a white primer, which provided an initial white background for the painting (Table 5). The primer was found to be made of very fine particles. Aside from its different composition, its consistency is finer than that of the white primer used for the Baclayon Church column painting.

Comparing these samples, it can be observed that the choice of material may have depended on the type of surface the painting was painted on. The white ground layer containing calcite and TiO_2 was applied onto the coral stone wall of Baclayon Church, while clay-based ground was placed over the wooden cornice. Moreover, a different type of ground was observed in the samples from the Daus church ceiling painting, whose support material is galvanized iron.

From the cross-section images, it can also be observed that there had been no interventions done on the sampling areas of the ceiling painting. In contrast, the presence of a new ground layer and an overpaint layer suggests that there had been an attempt to replace the original design on the Baclayon Church cornice.

Interventions may have also been observed in the samples obtained from the gilt surface of the *retablo* in Loay Church. As shown in Table 6, the samples consist of several layers, including an older metal leaf layer, which was not seen through optical microscopy but was observed using SEM, and newer imitation metal leaf layers. Under the gold leaf layer, a red layer was found containing Si, Al and Fe, indicating the use of bole, an iron-rich aluminosilicate clay (Sansone *et al.*, 2010). Similar to the Baclayon Church cornice, a clay-based ground was applied onto the wooden surface of the *retablo*. Newer layers were found above the gold leaf, including the new metal leaf layers and their preparation layers which consist of a white ground and a yellow base coat. The presence of Pb and Cr in the base coat layer suggests the use of chrome yellow (PbCrO_4) pigment. The Al and Cu metal layers indicate the use of imitation silver and gold leaves, which are cheaper alternatives to the original metals.

4. Conclusions

A study of coloring materials used in the paintings of historic churches in Bohol, Philippines, using a microscope and various spectroscopic techniques, gave the following conclusions.

First, analysis of paint samples from the three buildings revealed similarities and differences between the pigments they contain. Iron oxide pigments were commonly observed in the paintings studied, producing dark yellow and brown colors. Zinc oxide was found to be the white pigment used in the ceiling paintings in Daus Church and Loay Church, while titanium dioxide was detected in the Baclayon Church column painting. The titanium pigment was also observed in the overpaint samples from the cornice.

Second, the analytical data provided an insight not

only on pigments but also on coloring technique and types of materials used. It was observed that the choice of grounds varied according to the material onto which the painting was painted. For example, it was found that a clay-based ground was applied on the wooden surface of the cornice in Baclayon Church on the other hand, a white ground layer containing Ca and Ti was used in the column painting. For the Loay Church *retablo*, bole, a red aluminosilicate clay was applied, providing a surface for gilding. Several layers of metal were found in samples from the *retablo*, including one containing Au and Ag. It was observed that a new ground, a chrome yellow base coat and imitation silver and gold leaves were applied over the earlier gold leaf.

Third, aside from the mineral pigments, a few synthetic organic compounds were found in these paintings. Among the peaks in the program of the yellow sample from the Baclayon Church column painting are three peaks corresponding to Cl-containing fragments from the monoazo pigment PY3. For such organic pigments, a white substance such as zinc oxide and titanium dioxide is used as extender filler and then colored. The organic pigment is a substance used singly or simultaneously with other pigments.

Ultimately, the knowledge of the identity and properties of the materials used in these paintings will contribute to the understanding and protection of Bohol's 20th century artworks. This shall provide a basis for authentication and guide the conservator in planning a reliable restoration procedure and selecting compatible materials for future conservation and restoration work. To do so, for a more detailed analysis of the other organic pigments encountered in this study, as well as the other organic components such as binders and varnishes, appropriate separation and spectroscopic methods must be done in the future.

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