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# ARCHAEOMETRICAL ANALYSIS STUDY OF THE WALL PAINTING – FRESCO OF A ROMAN TOMB AT ABILA (QWAILBEH) JORDAN

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## ABSTRACT

This study examines the wall painting in the Roman tomb in the city of Abila (Qwailbeh). Archeometrical study to know the actual composition of the pigments used in the composition of the mural paintings is very important to archaeologists, especially with regard to restoration and preservation processes. This study aspires to provide a qualitative addition to this field by knowing the characteristics and components of these wall drawings in the city of Abila the techniques used in their implementation and identification of these dyes collecting information about them and determining the identification of pigments and binders for making these wall paintings. This research is also useful in presenting the factors affecting these drawings, which in turn threaten their survival in order to know the correct methods of preservation and protection of these drawings. The methodology used in the analysis of pigments included the use of three spectroscopy techniques, FTIR, XRD, and XRF on a complementary manner. These techniques for testing samples showed that the main element of all samples is calcium, and this indicates that lime was used as a binder and white dye at the same time, it was also noted that organic materials are varied as egg-white, wheat paste and beeswax were used as binders by fresco-secco technique as detected by FTIR. Pigments identified were red iron and yellow ochre (with a high content of hydrated iron oxides such as goethite and limonite) Egyptian blue, white lead, carbon black soot, and malachite.

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**KEYWORDS:** Archaeometry, wall painting, Abila, Decapolis, spectral techniques, ATR- FTIR, XRD, XRF, Fresco, Secco, pigments, ochres, Egyptian blue

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## 1. INTRODUCTION

Abila of the Decapolis, with the modern name (Quailibah) located about 13 kilometres north-north-west of Irbid in northern Jordan (Fig. 1) a height of 440m. above sea level, and its vicinity to Yarmouk River; the entire site is about five km south of Wadi Yarmouk. Abila, mentioned in ancient sources and an ancient inscription speaking of a "well known Abila of the Decapolis" is placed by Eusebius 19 kilometres east of Gadara (Nassar *et al.* 2022: 823-840; Nassar 2015: 229-241; Negev 1979: 4; Mare 1981: 343; 1982: 57; 1992: 309; Cohen 1995: 277). For a time, it was also called Selevkia (Greek: Σελεύχεια, also transcribed as Seleucei or Seleuciaia, and Seleukheia) (Schumacher, 1889: 35).

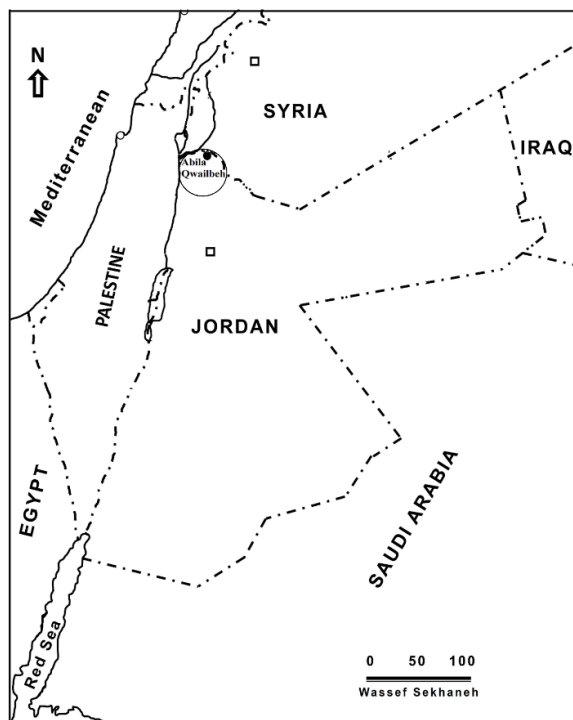


Figure 1. Map of Jordan, showing the site of Abila (Al-Sekhaneh *et al.* 2022: fig. 3)

New research methods applicable to diverse artifacts and the involvement of various disciplines have led to the ability to answer many questions about human existence, environmental dynamics, reconstruction of past environments, cultural knowledge, and intangible heritage. The field of archaeometry, or the archaeological sciences, is considered in many important specialized areas (Liritzis *et al.*, 2020: 49).

In 2003, an analysis was conducted on historic mortars from various architectural sites such as Islamic, Gothic, and local mansions in Palma de Mallorca. This examination aimed to explore the pigments in the samples (Genestar and Pons 2003: 291-298). Gen-

estar's study agrees with some of the methods and results used in the study of the examination of ancient mortars and colours, which include various styles such as Islamic style, Gothic and later iterations, derived from the palaces, monasteries and palaces of Palma de Mallorca. The analysis involved the use of scanning electron microscopy (SEM) in combination with energy dispersive X-ray spectroscopy (EDX), Fourier transform infrared spectroscopy (FTIR), and X-ray diffraction (XRD) techniques to characterize the structure and chemical composition of the studied samples (Genestar and Pons 2003:291-298; Ali *et al.* 2022: 63-80).

The presence of calcite in the samples analyzed by FTIR and XRF examination indicates the application of the fresco technique. The presence of some organic matter (possibly egg white). The presence of organic material detected by FTIR (e.g. egg yolk) could have been used as a binder in the paint layer, indicating the use of secco and also supporting the identification of gypsum in the samples. Finally, it was noted that calcite was often mixed with other pigments in the secco technique to achieve the desired color. desired aesthetic result. Based on the above, it is possible to apply the mixed technique in the plates studied (Konstanta *et al.* 2018: 251-256).

The present study aims to explore and display a city known for its extensive collections of fresco wall paintings, which consist of secco and have been identified through archaeological excavations. It is considered one of the important sites at local and global scale, with the primary goal of understanding their configuration and developing innovative conservation and protection strategies to encourage local authorities to protect these valuable archaeological treasures from decay and fading. To achieve and clarify these goals, the study employed advanced analytical techniques to examine the dyes used. We used state-of-the-art techniques such as X-ray and infrared spectroscopy and elemental analysis to gain a comprehensive understanding of the materials, pigments, and underlying structures of the mural painting. In addition, our research will focus on encouraging conservators to develop new restoration methods specifically tailored to the unique challenges of the city's murals. This innovative approach will involve the development of specific materials and conservation strategies that take into account local environmental conditions and the delicate nature of an important Romanesque work of art.

## 2. HISTORY AND EXCAVATIONS OF ABILA

Excavations started on the site in 1959 by the Jordan Department of Antiquities. However, excavations indicated that this site has been settled during

Hellenistic period (331-63 BC). This is proved Abila discovery of two tombs and some ceramic materials dating back to that period in different areas of Abila. Also, it's an important city among the prosperous Roman and Greek cities of Decapolis (Mare 1992: 309-313). Through the archaeological finds, it was found that the city of Abila is the same as Qwailbeh, and this was also confirmed by Schumacher, as he determined its location on two hills, one of which is called Tel Abila, which is the northern hill and the other is the southern one, and it is called Umm Al-Amad. The site was located on the course of a seasonal river, about a kilometre and a half to the south of Ain Abila. The city of Abila is surrounded by valleys except for the southern side, which made it a fortified and distinguished area (Beitenhard, 1963:26). In 1984, excavations of the mounds located at the site of Tel Abila and Tel Umm al-Amad were constructed, which shows evidence of Hellenistic settlement in the area (Mare et al. 1985:221-237; 1987: 38-51). The estimated number of artifacts found at the site is nearly 700 pieces in the Jordan Museum, which extends from the early Bronze Age to the Byzantine period (Darabseh, 2011:123). The exploration teams use the archaeological site temporarily for a few months during the year (Afanasyeva, 2004: 70, 71). The Jordanian Department of Antiquities conducted excavations for some of the tombs located along Wadi Qwailbeh, northeast of Tel Abila; those excavations were administered by Mare and others in 1980 AD. These excavations were followed by another excavation in 1982 AD.

The result of the mentioned excavations is evidence of settlement throughout different ages, such as the Bronze and Iron Ages, as an extension of the Hellenistic, Byzantine, and Roman periods (Mare, 1985: 6-16; 1992:57-77). 25 caves were found in Abila, each one of them has 15-25 tombs. Excavations revealed the presence of a mausoleum from the Umayyad period and a mosque dating back to the Islamic rule. It is believed that there are many other buildings at the site that have not yet been discovered (Al-Naddaf and Al-Saad 2007: 56; Shunnaq, 2005: 406-407). The tombs in Abila generally include three types of designs: the simple rectangular design, the rectangular design with an apse, or even two apses. It also includes the design with rectangular curves with arches (Nassar 1986: 44). There is a great similarity between the tombs located in the Al-Yasileh area in the north of Jordan and the tombs of Abila in terms of the nature of the rock used and the planning of the sepulchres in general and it contains many burial chambers, where this similarity was revealed in the planning of the burials for the two regions (Al-Muheisen 1988: 15, 16; 1989: 253-257; 1990: 617; Taqriier and Al-Muheisen 1992: 229-244).

## 2.1 The Research Question

The wall painting art conservation approach represented in this study consists of four different dimensions: A) it focuses on the systematic analysis and conservation of wall painting and its materials. The focus is on the original or first state of the significance of wall painting conservation and helps the deteriorated one to come back virtually to their life. The methodology is an important prerequisite for gaining as much knowledge and rich data material as possible. The criteria for a systematic survey of findings are, however, sustainable and refined within the discipline of archaeological restoration. Thus, a measure for determining the concreteness of hallmarks is introduced here which objectifies the comparison of wall paintings from different periods and various materials.

B) Determination of the layer-based age of the object has been proposed as an approach to supplement the archival historical data from the analysis of the structure of pigments and binding materials among the layers. This approach depends on the scientific analysis methods and techniques of the chemical composition of the various layers, with emphasis on the paint layer and the colorants which were used, and the colored layer of the wall paintings. These include, for example (a) the investigation of all layers by using the cross-sections technique procedures, (b) the characterization of original and ancient color layers, (c) testing of physical and chemical properties, taking into account the Conservation Approach Regulations and Ethics.

C) One of the most thoughtful problems that are faced by mankind is deterioration. Deterioration naturally impacts the wall pigments through chemical reactions that occur, between pigments and their environment. Wall painting should be prevented by the safest and lowest cost methods. The wall painting in the tombs has received little attention in art historical research and conservation over the years and has been regarded as less significant than the other great platforms of the time. Its poor state of conservation certainly contributed to this assessment not insignificantly. This neglect of the as well as of its close surroundings formed the occasion to place the altar of change at the center of art-technological investigations and thus to give new impulses to the debate on the part of art historians and archaeologists. The question that arises for this treaties is: How can research, with the help of the archaeometric and conservative approach benefit the expansion art re-foundation of the tomb wall painting and inscriptions virtually to the public.

D) The present article contributes to the question of the relationship between art analysis and - broadly to

be founded - technical history of the wall painting arts in the Roman era in Jordan. It is a question that the researcher first posed similarly almost on the occasion of the investigation of the Abila Tomb site of Abila. The historiography of this work of art history draws structural material from the extensive wall painting of historical source writings that the branch of the art technology and conservation of essential painting in the Abila Tomb. The work will be developed in the meantime of my work as a pioneer work in this field in the site as a first virtual reconstruction of the art in the Tomb. The works of art themselves form the second pillar of this field of research as source material and history of the archaeological site.

The present study proposes a systematic analysis of individual works of art by art technology that can make an independent contribution to the history of the arts and conservation, which represents the necessary prerequisite for this. It will supplement and update our point of view and perspective through the appropriate investigations. At the same time, the study raises the question of both the necessity and the sustainability of the integrated wall painting in the site as an art-conservation approach in the context of archaeological work related to the research, and that will continue to gain in the future of wall painting study in the Abila cultural heritage site, there are other Roman places have mural painting (Gutman *et al.* 2016: 193-206).

This study came to focus on this city rich in wall paintings to study it and know its composition and how to deal with it to preserve and protect it from de-

struction and fading, as these drawings are considered one of the most important archaeological treasures in the region and the world. Its importance lies in the fact that it contains samples of drawings of a variety of decorative designs, including human, plant, and geometric shapes, as well as birds and animals. The features of many of them are still not clear until now.

### 3.THE TOMB OF THE STUDY

The tomb is located in the southeast of Tel Abila, on a cliff cut off hillside in the rocks; it can be reached along a bumpy road which leads to three stairs. The gate leading to the tomb seems to be removed over time for a certain reason. The entrance wall of the tomb is 5.20 m long and 2.90 m high, and the opposite side is 5.20 m long and 2.90 m high. The facade contains a 2.70 cm wide and 2.65 m high curve, which contains six cavities, three upper and three lower (Fig. 2, 3). This tomb is characterized by the spread of paintings and decoration throughout the space, including the tomb ceiling, which is characterized by triangles, hexagons and rhombic shapes. Moreover, the cemetery includes human paintings represented by human heads and plant paintings, such as leaves and rose branches, as well as column paintings that decorate the space between cavities (Barbet and Vibert-Guigue 1988: Pl. 1, 78; Nassar ,1996 :44). The researcher, accompanied with Wassef Al Sekhaneh, made several visits to the site to document the study cemetery, take pictures and study the case of the tomb closely (figs. 2, 3).



*Figure 2. Tomb gate (Al Sekhaneh, 2022).*



Figure 3. Inside of the Tomb (Al Sekhaneh, 2022).

### 3.1. General condition of the tomb

It can be said that the tomb conditions are poor, as the erosion on the stones and murals is obvious. We can attribute this situation to several factors, the most important of which is the biological factors related to the growth of molds and some decomposing organisms. The accumulation of these organisms has led to the disappearance of some features of the tomb. The characteristics of the tomb, as well as the roots of some plants and herbs located above the tomb, have a great impact on the ceiling of the tomb.

Some factors are relevant to human theft and destruction, which appears obviously. Additionally, improper site restoration negatively affects the ceme-

tery. In addition, environmental factors have a significant impact on the quality of drawings and the overall tomb. The change of temperature, relative humidity and air pollution are some of the most important factors.

### 3.2. Wall painting in the tomb

- Tomb entrance wall: It contains a rectangular frame with a rose painting on the left.

-Tomb chest: The graph on this interface represents the image of two roosters facing each other inside a right triangle frame. Each one carries by its beak a branch of a plant. The roosters were distinguished by the color dark red, brown and the beak is green (figs. 4,5).



Figure 4. The right side of the apse (the chest of the tomb) motif (Al Sekhaneh, 2022).



Figure 5. The left side of the apse (Al Sekhaneh, 2022).

### 3.3. The apse (the curve)

A wavy decorative strip with small squares inside like chessboard, located at the front and upper right corner of the wall (Figs. 6,7).

There is a right triangle frame, in which there is an image of a woman's head. There is a similar frame on

the left side, and it is believed that there is a similar image on the right side (fig.8).

Geometric shapes with angles similar to the swastikas motif, symmetrical cubic shapes inside exists in the vaulting of the apse (fig.9).



Figure 6. Small squares inside apse like chessboard motif (Al Sekhaneh, 2022).



Figure 7. The apse (the chest of the tomb) (Al Sekhaneh, 2022).



Figure 8. Entrance wall of apse (Al Sekhaneh, 2022).



Figure 9. Swastika motif inside exists in the vaulting of the apse (Al Sekhaneh, 2022).

### 3.4. Tomb Ceiling

There are drawings of irregular hexagonal geometric shapes next to each other; they look like a frame

for smaller hexagonal geometric shapes. These hexagons are linked by rhombic geometric shapes colored in green and on both sides of the rhombuses are small triangles in yellow (fig. 10).



Figure 10. Wall painting of the tomb ceiling (Al Sekhaneh, 2022).

### 3.5. Condition of the Tomb Wall Painting

The Wall painting in this research are in Abila Tomb; the climate of the area and the interior microclimate seriously after opening the tomb affect the wall painting's condition. The temperature at the place inside is so high that makes the area an unsuitable place for the opened tomb unless proper care is taken. To this end, an environmental study is important during which, the microclimate of the tomb interior environment will be monitored (temperature, RH, volatiles, particulate matter) by the area seasonal climate changes 2. Site condition report

### 3.6. Wall Paintings colours

The thickness of the paint film in the tomb varies greatly in the thickness has been measured in the study, and it consists of one or more coloured substances, pigments or dyes, dispersed in, a medium in general that acts as a binder between the particles and the support. Pigments are fine, coloured powders of

varying particle size, insoluble in the medium, with which they form a more or less dense compound that reveals covering properties when applied. Dyes are transparent, soluble substances, capable of bringing their own colour to the transparent medium in which they are dispersed, the medium, by inclusion, absorption or by the formation of stable chemical bonds. The colours used in ancient wall painting are mostly based on finely ground metal oxides: their colours are generally determined by the nature of the metal atoms they contain. Strongly coloured pigments are mostly those containing the transition metals. copper ion, whose salts absorb the red part of the electromagnetic spectrum, returns a green-blue colour whose amount of green and blue depends on the chemical nature of the ions that surround it. In other compounds, such as those of iron, the location of electrons can limit the absorption of light: the red colour of iron ion is given by the movement of an electron from an oxygen near the iron ion.

#### 4. METHODS AND SCIENTIFIC INVESTIGATION

Visual examination of possible surface changes and alterations will allow us to recognize and locate deterioration spots for preservation state study. The starting point for all technical studies of works of art is typically a close examination by the eye, under proper artificial light or daylight. Cracks at or near the paint surface and delamination of layers that incorporate the image are the most common forms of visible damage to the wall painting inside. The fluctuation of the humidity, temperature, and direct contact with the wall painting may cause its deterioration after opening the tomb in the present time. In the light of the above, the scientific examination of the painting will consequently and most decisively contribute to our understanding of damage and deterioration impact on them for archaeological documentation, and help to provide a rational approximation of how the work might have looked originally and compatible with the virtual reconstruction and conservation ethical codes. This study would guide art historians and wall painters, to know how an artist worked, what pigments were used; for the conservator, to develop (with the help of the conservation scientist) and apply techniques necessary for (a) the conservation and protection of wall paintings, including virtual damage repair, and how to repaint missing parts virtually, and (b) for the conservation scientist, to draw information and build the knowledge of the used virtual construction methods, and the necessary understanding for the origin of the colors, materials were used by using some samples in the labs. The lack of proper care and in some cases, the neglect for wall painting within most of the archaeological sites in Jordan, demands immediate action that includes pioneering studies of Roman wall paintings and subsequent conservation actions in Jordan. The deterioration of the archaeological paintings, is in most cases, the result of many changes at the chemical and physical level, concerning structural components; for example, colors fade and sometimes disappear. These changes call for urgent action and should be taken care of as soon as possible to keep these masterpieces of wall painting in the Abila cultural heritage site safe for future generations in Jordan.

##### *Methods*

Fourier transform infrared spectroscopy (FTIR) to identify the functional groups that characterize the pigments used in the wall painting. Furthermore, it will be applied to identify the binding medium of the grounds and pigments. For Fourier Transform Infra-

red Spectrometry (FTIR) experiments, a small quantity of mortar, typically a few milligrams, was employed. The samples were carefully mixed and manually ground in an agate mortar and pestle. A residue of approximately 0.1 mg of the mortar sample was retained in the mortar, combined with roughly 0.5 mg of potassium bromide (KBr), and compacted into a pellet measuring 3 mm in diameter using a mechanical press. Each resulting pellet underwent 32 scans spanning the spectral range from 400 to 4000  $\text{cm}^{-1}$ . The identification of phases was conducted using the OMNIC Software. Pigment samples underwent X-ray diffraction (XRD) analysis at the Yarmouk University Archaeology Laboratory, using a Shimadzu LabX XRD-6000 X-ray diffractometer. This analysis involved directing an X-ray beam at the crystal planes of the samples, causing a change in its propagation direction and generating a spectrum that reflects the crystalline properties of the mortar sample. To characterize the samples, the instrument employed copper  $\text{KL}_{2,3}$  ( $\alpha_{1,2}$ ) radiation with a wavelength of 1.5418 Å, operating at 40 kV voltage and 30 mA current.

XRF analysis of corrosion products was made using Panalytical Phillips MiniPal 2 XRF Analyzer. The Minipal by Philips is a desktop XRF analyzer that can evaluate elements in powder samples. Without further sample preparation, samples are examined in 30 mm diameter sample cups. X-ray origin is 9 W Rh-K source (30 kV maximum; 1 mA maximum).

#### 5. RESULTS AND DISCUSSION

The IR is classified into three ranges: far infrared ( $< 25 \mu\text{m}$ ) or 400 - 10  $\text{cm}^{-1}$ , mid infrared (2.5-25  $\mu\text{m}$ ) or 4000 - 400  $\text{cm}^{-1}$ , and the near-infrared ( $< 2.5 \mu\text{m}$ ) or infrared regions 14285-4000  $\text{cm}^{-1}$ . The samples taken were analyzed in their original state and/or as a cross-section by integrated physicochemical measurement techniques, in particular by ATR-FTIR, also by X-ray powder diffraction, in order to determine the painting's mineralogical composition, the state of pigments of the frescoes, the decay caused in particular by chemical substances, especially by salts and detachments.

The ATR-FTIR spectrum is seen as a unique fingerprint of each molecule that can be obtained by passing infrared light by a compound at a given wavelength causing a decrease in percent transmittance (%T), manifested as a peak or band. Absorption in the spectrum corresponds to the vibrational frequencies of functional groups in the molecule. The absorption peaks (ATR-FTIR) were also measured on the different pigments (Fig. 12). The absorption peaks 3253  $\text{cm}^{-1}$  is assigned to the hydroxyl ions of the clay minerals, and 3175  $\text{cm}^{-1}$  to the hydrated iron oxide, which allows a delimitation between hematite and goethite.

Peaks 1445, 1541 and 1029  $\text{cm}^{-1}$  are assigned to Si-O stretching, while the weaker peaks at 672, 798, and 530, 455  $\text{cm}^{-1}$  are assigned to deformation modes involving the remaining Si-O and are attributable to quartz  $\text{SiO}_2$ . The weak absorption at 1625  $\text{cm}^{-1}$  corresponds to C=C, indicating the presence of organic binders as egg-white, wheat paste and beeswax. In addition, the C-N stretching at 1239  $\text{cm}^{-1}$  and the N-H stretching at 1705  $\text{cm}^{-1}$  are assigned. (Fig. 12), the infrared peaks and their corresponding respective assignments that corresponded to the green, grey, yellow, and red pigments.

In a macro-ATR-FTIR analysis of the samples, strong absorbing bands are observed at 2517,

1796, 1626, 1445, 1541, 873, and 715  $\text{cm}^{-1}$ , which are attributed to the stress and bending vibration of the  $\text{CO}_3$  carbonate ions of calcium carbonate  $\text{CaCO}_3$ . The peaks at 2971 and 2877  $\text{cm}^{-1}$  are also attributable to calcite. However, still in the 940-1210  $\text{cm}^{-1}$  range, peaks at 1141, 1082, and 1033  $\text{cm}^{-1}$  appear to be assigned to aluminosilicate materials and S-O vibrations. Finally, the peak at 1801  $\text{cm}^{-1}$ , which largely overlaps with the broad calcite band at 1445, 1541  $\text{cm}^{-1}$ , suggests the presence of some organic material. During the analysis of the colored layer of the sample, using the Raman technique (Fig. 11), strong luminescence was observed, which did not permit the appearance of peaks and the extraction of results.

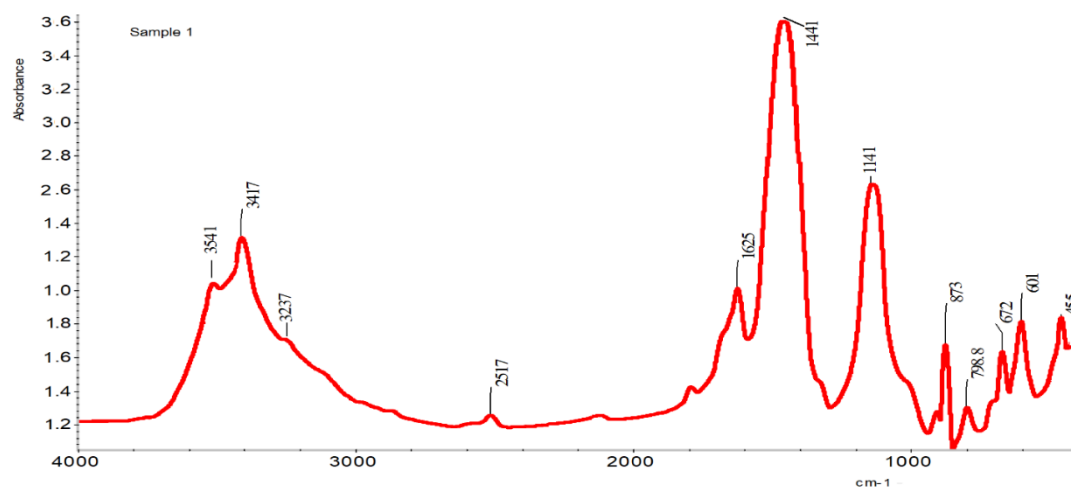


Figure 11. FTIR Spectrum of the mid-infrared region (4000-400  $\text{cm}^{-1}$ ) of the painting sample 1.

For this reason, the analysis of the sample was also performed by micro-ATR-FTIR spectroscopy in order to identify the dye more accurately. In the ATR-FTIR spectrum (fig. 12), characteristic peaks are observed mainly in the region between 1280  $\text{cm}^{-1}$  and 1001  $\text{cm}^{-1}$

<sup>1</sup>, attributed to the Si-O-Si vibrations. Except for the peaks at 1796, 1625, 1441 and 873  $\text{cm}^{-1}$  of the calcium carbonate, the sharp peaks at 1150 and 1033  $\text{cm}^{-1}$  and at 980  $\text{cm}^{-1}$  are all assigned to the Egyptian blue.

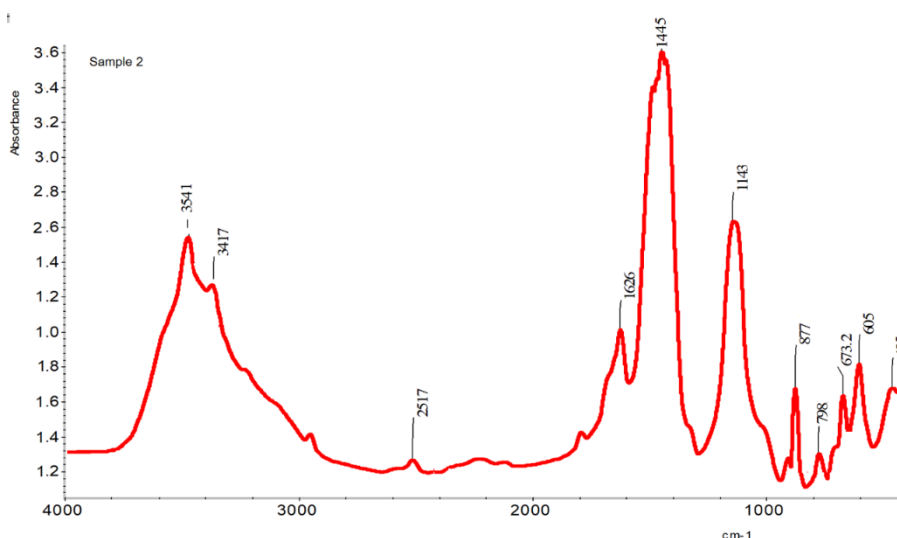


Figure 12. FTIR Spectrum of the mid-infrared region (4000-400  $\text{cm}^{-1}$ ) of the painting sample 2.

In the spectrum (figs. 13, 14, 15, 16), strong absorption bands are observed at 1443, 873 and 715 $\text{cm}^{-1}$  which are assigned to the stress and bending vibrations of the calcium carbonate ions  $\text{CO}_3$  of the calcium carbonate  $\text{CaCO}_3$ . The peak at 1796, 1626 $\text{cm}^{-1}$  is attributed to the vibrational modes 1 and 4 absorbing band originating from the calcite association. Thus, we conclude that calcium carbonate has a calcite crystal structure. The absorption bands appearing at 2981 and 2877 $\text{cm}^{-1}$  are also assigned to calcite. Still, in the

940-1210  $\text{cm}^{-1}$  region, peaks 1125, 1141, 1082 and 1033  $\text{cm}^{-1}$  appear, which correspond to Aluminosilicate are composed of aluminum, silicon, oxygen, materials, and S - O vibration mode.

The peak at 1801  $\text{cm}^{-1}$  indicates an organic substance, while the weaker peak at 1625  $\text{cm}^{-1}$ , also known as amide I, is the absorption band produced by proteins in their spectra.

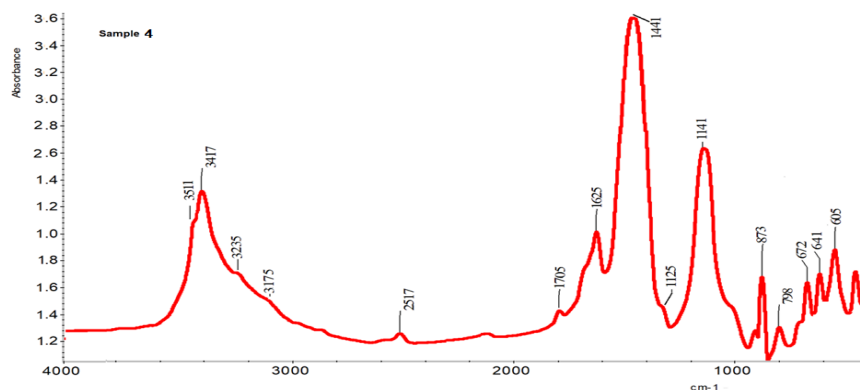


Figure 13. FTIR Spectrum of the mid-infrared region (4000-400  $\text{cm}^{-1}$ ) of the painting sample 3

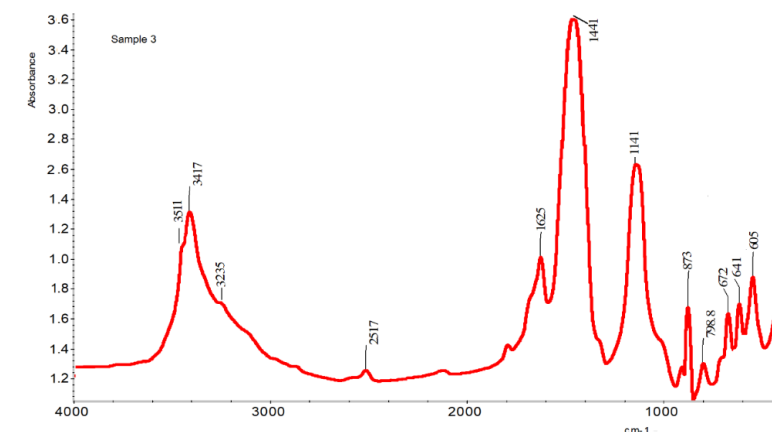


Figure 14. FTIR Spectrum of the mid-infrared region (4000-400  $\text{cm}^{-1}$ ) of the painting sample 4.

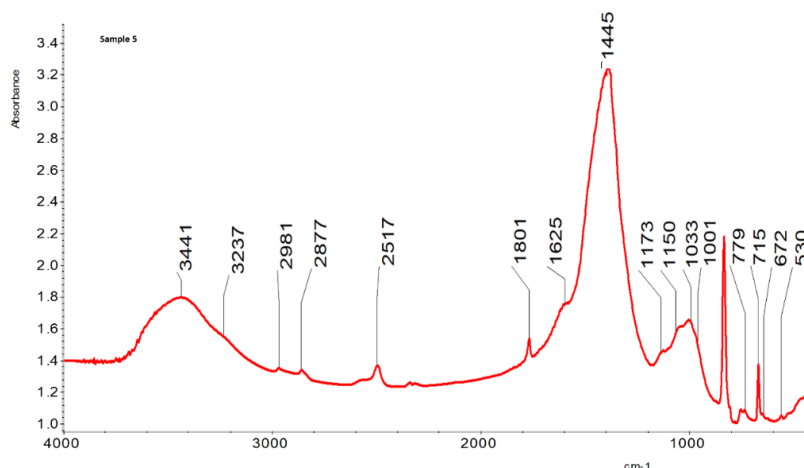


Figure 15. FTIR Spectrum of the mid-infrared region (4000-400  $\text{cm}^{-1}$ ) of the painting sample 5.

XRD mineral analysis of the red samples confirmed the presence of iron, as well as silicon and aluminum attributed to clay-silicate components. Based on the above, it is concluded that red ochre with hematite as the main component was used to yield the red hue, which is confirmed by the results of FTIR spectroscopic analyses, where the characteristic peaks of red ochre are identified.

As for the yellow samples, the significant concentrations of iron, but also of other silicate components detected by XRD mineral analysis, allow the conclusion that the yellow color in the frescoes of all three tombs is due to yellow iron ochre with a high content of hydrated iron oxides such as goethite and limonite. This conclusion is supported by FTIR spectroscopic analysis, which identified characteristic yellow ochre

peaks. The elemental analysis of the blue samples revealed the presence of copper (Cu), indicating the use of a pigment with copper as the predominant component. In the case of the blue shades, the spectra of the  $\mu$ Raman analyses were not able to provide complementary results due to the presence of the luminescence effect. Subsequently, FTIR analysis was performed, which identified the characteristic peaks of Egyptian Blue, confirming the use of the dye to produce the blue hues.

From the results of the XRF analyses of the black dye, which showed a high carbon C content, we conclude that the dye carbon black was used to produce the black hue. The FTIR spectra confirm our conclusion, as the characteristic peaks of carbon black can be seen.

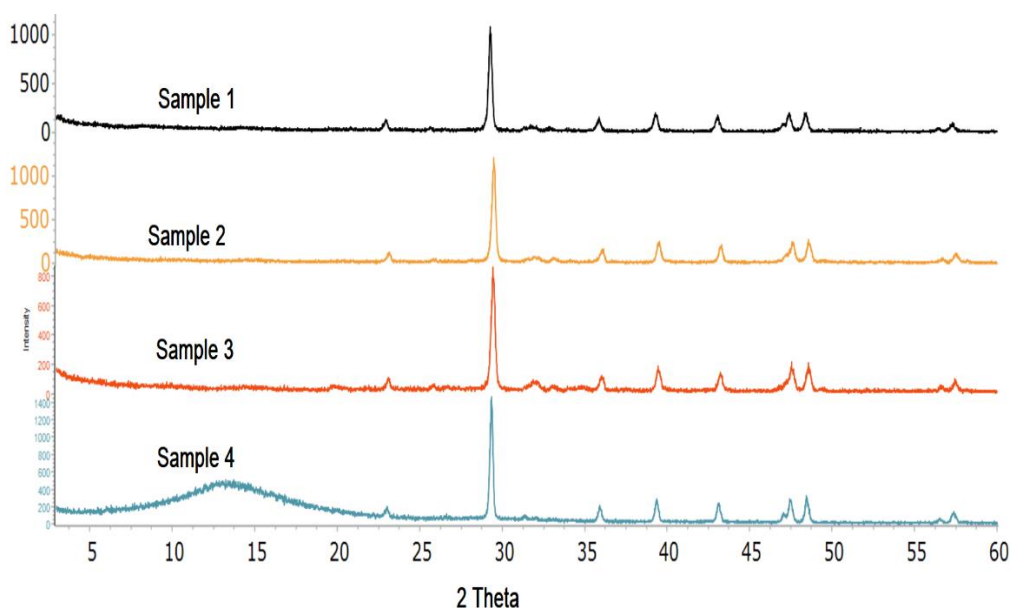


Figure 16. XRD patterns of the painted layers with preparation of Sample 1, 2, 3 and 4

In the green S1 sample from the Abila tomb, XRD mineral analysis detected the presence of copper oxide, indicating the use of a copper pigment, most likely cuprite. However, pure green levels are not often found in Abila's tomb. The pigments that were identified were calcite, red iron oxide, yellow ochre, Egyptian blue, and carbon black soot. They are found in all the mural paintings and form the basis for the colourfulness of the portrayals in the tomb. In contrast, the use of green in Roman wall painting is relatively rare and, the colors are often the result of a mixture of other coloring substances.

FTIR spectroscopy, (XRD) X-ray Diffractometer and (XRF) in (Fig. 16) and Table 1 are used for identification of the pigments of the murals paintings. ATR-FTIR infrared spectroscopy detected the presence of organic material. However, identifying the binder in the wall paintings is generally very complicated. The

problem is that the organic matter is very small compared to the rest of the material and the characteristic groups of substances degrade with time, making it difficult to reliably identify them. For this cause, but also to proceed with the investigation, it is suggested to apply other techniques, such as gas chromatography-mass spectrometry for future study. Finally, it is suggested to explore other wall paintings of other tombs in Abila from other sites or from another tomb in the same area, using the same techniques, in order to allow a comprehensive analysis.

Table 1 shows the results obtained from the XRF technique, along with the suggested pigments corresponding to each color. Crossways all measurements, notable quantities of Fe, Cu, Ca, P, and Pb were detected, potentially stemming from the mortar components. Particularly outstanding is the consistently high Ca content observed in all measurements. This

observation might be attributed to the possible existence of Calcite, potentially arising from the method employed during the creation of the mural painting under investigation. This process convoluted combination of pigments with water or limewater and subsequently applying the mixture onto freshly arranged plaster – a procedure akin to the fresco painting technique.

Table 1 shows the results obtained from the XRF with the suggested pigments corresponding to each color.

**Table 1. Qualitative XRF analytical results and identified pigments**

Color	Elemental Analysis -XRF	Pigments colors-FTIR
White	Ca, Pb	Lead White
Black	Ca, P	Black Bones
Red	Fe, Ca, Pb	Goethite
Brown	Fe, Ca, As	Iron Ochre
Green	Ca, Cu	Malachite

## 6. CONCLUSIONS

The study of the 8 samples taken from the wall paintings of the Abila tombs was carried out mainly by spectroscopic methods (ATR-FTIR, XRF, and XRD in the labs). These methods worked in a complementary manner providing a large amount of information capable of leading to firm conclusions on the identification of pigments, the mural technique, and the presence of organic binders. The results of the spectroscopic investigations show that the basic element of

all samples is calcium Ca, which indicates the mixing of pigments with lime. The lime acted as a binder and at the same time as a source of white pigment. In addition, X-ray powder diffraction (XRD) analyses identified calcium carbonate, with a calcite crystal structure ( $\text{CaCO}_3$ ). Therefore, the strong presence of calcite allows us to assume that the fresco technique was used to execute the murals, where the pigments were then mixed with lime water in order to make the required binding during application on the still-fresh mortar. At the same time, the ATR-FTIR spectroscopic results obtained indicatively from the preparation of the frescoes from samples and are confirmed the strong presence of calcite and at the same time indicated the presence of organic material. This fact leads us to hypothesize that the fresco technique was continued with drywall painting (secco), using organic material as a binder. It is logical that in monuments of this size the fresco technique cannot be fully traced, but possibly small details, corrections, or even whole sections of the painting were done with the Fresco-secco technique a mural painting technique in which paint is mixed with an organic binder then is applied to a dried plaster or lime. The results of the XRD analyses obtained from various points of the frescoes of the Abila tomb samples indicated the presence of high concentrations of iron oxide and copper oxide. Therefore, earthy and copper pigments were mainly used for the painting decoration of the frescoes.

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