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Disclosing Colors and Pigments on Archaeological Objects from the Aga Khan Necropolis (West Aswan Egypt) through On-Site Analytical Methods: Preliminary Results

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Abstract: The present study is aimed at the characterization of artifacts excavated in the necropolis surrounding the mausoleum of the Aga Khan in Aswan (Egypt), as part of the Mummies Investigations Anthropological & Scientific West Aswan Necropolis (MIASWAN) project. Four *cartonnages* and some pottery shards were investigated on-site by means of non-destructive and micro-destructive techniques, such as attenuated total reflection/Fourier transform infrared spectroscopy (ATR/FTIR) and visible reflectance spectroscopy Vis-RS). Thanks to the use of these techniques, several pigments employed in the creation of the artifacts were identified. Due to the impossibility of transporting the investigated objects out of Egypt, a first-ever on-site characterization of the artifacts from this important excavation was carried out through scientific methodologies. These extreme conditions made the use of analytical instrumentation very challenging. Nevertheless, several characteristic pigments and hues were successfully identified.

Keywords: pigments; ATR/FTIR; Vis-RS; cartonnages; West Aswan Necropolis; on-site analyses

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1. Introduction

A fundamental aspect of the study of artistically and historically significant painted objects is the identification of the compounds employed in their creation. Important information on the artifact can be obtained through the use of appropriate analytical methodologies, such as the painting technique, the availability of natural pigments, the ability of producing pigments from raw materials, and, consequently, evidence on the technological expertise in a particular historical period.

Furthermore, the physical and chemical characterization of materials can allow the detection of previous restoration or conservation work. In some cases, information on the authenticity and/or dating of the artwork can also be obtained. Indeed, research studies conducted in this field were able to identify the array of colors the ancient Egyptians had at their disposal [1–8].

Throughout millennia, the fascination for hues has always pushed mankind to search for different natural pigments to be used as colors or to find alternative ways to produce pigments synthetically. Evidence of the first synthetic pigment in history, Egyptian blue [9–11], dates to the Egyptian era. Indeed, thanks to the known technical skills of the Egyptians, it is thought that the pigment began to be produced around 3000 BC. It was the most widespread pigment in Egypt from the first dynasties to the end of the Roman era.

The symbolism of the colors also allows us to understand why Egyptian pictorial art is often associated with funerary art, where the purpose of the rich religious decorations was to make the afterlife more comfortable for the deceased.

Cartonnage is the term used for plastered layers of canvas or reused papyrus, where an extensive use of color has been made [12–14]. Cartonnage is flexible enough for molding while wet against the irregular surfaces of the body, making it the preferred choice to be used in funerary workshops to produce cases, funerary masks, or panels to cover all or part of the mummified and wrapped body [15].

In the present study, on-site spectroscopic analyses were carried out on some *cartonnages* excavated in the necropolis surrounding the mausoleum of the Aga Khan in Aswan (Egypt), as part of the Mummies Investigations Anthropological & Scientific West Aswan Necropolis (MIASWAN) Project. The excavations conducted since 2018 revealed an 800-year period covered by the necropolis, ranging from the 6th century BC to the 2nd AD. The analyzed artifacts were four different *cartonnages* and two pieces of pottery coming from tombs excavated in the necropolis.

To date, six campaigns of the Egyptian–Italian Mission at West Aswan (EIMAWA) mission, two of which in the framework of the MIASWAN project, were conducted by the scientific multi-disciplinary research group coordinated by Prof. Patrizia Piacentini of the University of Milan. The area of the excavation concession is approximately 100,000 square meters, of which, approximately 25,000 were mapped, uncovering the entrances to approximately 300 tombs (Piacentini, Pozzi Battaglia, Abd El-Moneim 2020).

The finds of the latter study come from two of the excavated tombs: AGH026 and AGH032. AGH026 revealed the presence of at least 46 individuals, including 14 complete mummies and commingled skeletonized and partially mummified remains, along with numerous other objects, including fragments of *cartonnages* of different sizes, such as foot covers, head covers, collars, and small fragments. All these artifacts still show vibrant and bright colors. In the second tomb (AGH032), 20 mummified bodies of adults and children were found wrapped in bandages with fragments of *cartonnage* still in place. The skeletonized human remains led to the identification of other 32 individuals. However, the study of the human remains of the Aga Khan necropolis attests to the presence of mummies composed of bones of different individuals. Similar findings are also attested in contemporary necropolises [16]. This makes it difficult to establish the exact number of deceased people who occupied the tomb, which could be around 45 individuals or less.

With regards to the artifacts in tombs, Egyptian law states that nowadays no item can be transported out of the country. Therefore, only on-site and non-destructive measurements are permitted using preferably portable instrumentation.

For this reason, the aim of the measurement campaign carried out from May to June 2021 was to evaluate the possibility of acquiring data through on-site analyses with instrumentation coming directly from Italy. Therefore, light and easy-to-use portable devices were employed, including a portable attenuated total reflection Fourier transform infrared spectroscopy (ATR/FTIR) instrument and a visible reflectance spectrophotometer, in order to obtain preliminary results on the investigated objects with the idea of conducting further investigations during subsequent campaigns.

2. Materials and Methods

2.1. Analyzed Samples

Four *cartonnages* and two pottery shards coming from tombs AGH026 (Figure 1) and AGH032 were analyzed (Table 1).These samples were chosen from finds that were being restored at the same time, encouraging interactions between the work of the restorers and the chemists.

Figure 1. Some of the researchers at work on the excavation (on the **left**) and the entrance of tomb AGH026 (on the **right**).

Table 1. List of the analyzed artifacts and the corresponding performed analysis.

Sample Name		Analysis
Cartonnage AGH026-B14		ATR/FTIR spectroscopy
Cartonnage AGH026-B13		ATR/FTIR Spectroscopy
Cartonnage AGH026- B31		ATR/FTIR Spectroscopy

Table 1. *Cont.*

Sample Name		Analysis
Cartonnage AGH026- B41		Visible Reflectance Spectroscopy
"Barbottina" pottery AGH032-14		Visible Reflectance Spectroscopy

The first artifact (AGH026-B14) was discovered in the 2019 excavation campaign. It is a *cartonnage* helmet, approximately 22 cm in diameter, from which the mask was removed by looters in ancient times. It is found in the south-west corner of the entrance to tomb AGH026 together with a group of other *cartonnages*, also removed from the mummies to which they belonged. The refined design on the head, characterized by a winged scarab holding a golden solar disk between its front legs and a red solar disk that recalls the *shen* ring, a symbol of eternity, between its back legs, exhibits the great value of this *cartonnage*. During the restoration, a fingerprint left by the painter was identified among the feathers of the beetle's wings. The rear of the helmet features a *djed* pillar surmounted by the head of the god Khnum and other geometric motifs. Particularly striking, however, is the rendering of the hair that framed the face. The hairstyle with rigid rows of black curls, rendered geometrically in a plastic way, is typical of female sculptural portraits of the Ptolemaic period, and is a typical dating element of the 1st century BC [17].

The second item (AGH026-B13), also excavated in 2019, is a foot (approximately 42 cm × 40 cm × 26 cm) coming from the same *cartonnage* nucleus as the previous one. The feet represented are wearing sandals. Traces of gilding are clearly visible both on the laces of the sandals and on the toenails. In front of them, a winged beetle is depicted. The side decoration is geometric except for a border of white rosettes. The soles of the sandals, decorated with geometric motifs, are separated by the stem of a lotus flower whose corolla opens at the front. From a stylistic point of view, the object could be dated between the 1st century BC and the 1st AD. However, as will be discussed further in the text, analyses revealed the use of madder, which suggests the latter date.

The third artifact (AGH026-B31) was also found in the 2019 mission in the south-east corner of the front part of room B of AGH026. It was removed in ancient times causing damage and overlapping with other parts of the same group of *cartonnages*. Only the lateral part, about 40 cm long, is well preserved. The deceased is here depicted praying before a god represented as a fantasy animal formed by a ram's head surmounted by a solar disk, four wings, and an elongated body in the shape of a bird that walks with four legs ending in the *maat* feather and that carries the mummified deceased on its tail, a symbolic viaticum towards eternal life. On a topological basis, also, in this case, the artifact can be dated between the 1st century BC and the 1st century AD [18].

The fourth *cartonnage* (AGH026-B41) was found in the third excavation campaign (May–June 2021). It is the upper, fragmentary part of the so-called "apron". It is depicted with a winged scarab and the winged goddess Nut holding the *maat* feather in her hands, both symbols of rebirth. Also, in this case, the dating on a typological basis is 1st century BC—1st century AD.

Lastly, two fragments (about 4 × 4 cm) of a so-called "barbottina" cup (AGH032-14) from the descent of tomb AGH032 excavated in 2021 were examined. Other fragments of this object were found at a later stage. This cup takes its name from the band decorated with vegetal elements painted in relief with ceramic liquid, followed by two phases: a second firing to obtain the glossy coating and the white overpainting of some elements. On a typological basis, the object can be dated back to the 1st century BC—1st century AD [19].

The *cartonnages*, after being found in the tomb, were transported to the warehouse on the eastern bank of Aswan where FTIR analyses were performed, except for *cartonnage* AGH0026-B41, which was investigated by visible reflectance spectroscopy directly on-site at the excavation. The measurements on the pottery were also conducted on-site. This instrumentation included an ATR/FTIR spectrophotometer and a visible reflectance spectrophotometer.

2.2. Instumental Techniques

The spectrophotometric analyses (FTIR measurements and visible reflectance analyses) were all performed with non-destructive/micro-destructive instrumentation on the artifacts (*cartonnages* and pottery, Table 1) and on fragments that had already fallen from the *cartonnages* and were present inside the plastic boxes in the Aswan warehouse. Scientific instrumentation was used and managed in extreme and challenging conditions, especially on the excavation, which included very high temperatures (often exceeding 40 °C) and the presence of desert sand, which is particularly dangerous for the instrumentation (Figure 1).

2.2.1. ATR/FTIR Spectroscopy

The investigations were carried out by attenuated total reflection/Fourier infrared (ATR/FTIR) spectroscopy to highlight the nature of the pigments present in the samples. These analyses were performed with a portable FTIR spectrometer (PerkinElmer Spectrum Two) coupled with an ATR accessory (UATR Two) equipped with a diamond crystal. The spectra were acquired on colored micro-fragments in the 4000–400 cm^{-1} range with a 4 cm^{-1} resolution. Each spectrum was acquired in transmittance mode with 64 scans. Moreover, the identification was performed by comparing sample spectra with reference materials or other literature spectra [20,21]. Although the instrument can be considered portable (or rather transportable), it allows only micro-destructive analysis since it requires the sample to be placed on the diamond crystal of the ATR accessory.

The instrument was used on-site in a temporary location where objects and mummies coming from the excavation were moved (Figure 2).

2.2.2. Vis-RS Spectroscopy

Visible reflectance spectroscopy (Vis-RS) analyses were performed by means of a Konica Minolta CM 2300d portable spectrophotometer. The instrument was calibrated using its 100% reflective white reference and a 0% reflective calibration box in the 400–700 nm range. All the measurements were carried out directly on the samples' surfaces.

The instrument in this case was employed directly on the artifacts at the excavation site.

Figure 2. FT-IR spectroscopy measurements at the warehouse where the objects were moved.

2.2.3. Optical Microscopy

A Dino-Lite USB microscope equipped with an includable/excludable polarizing filter and directly coupled to a portable PC was employed to analyze, in detail, the surfaces of the *cartonnages*.

3. Results and Discussion

Comprehensive archaeometric analyses in an archaeological Egyptian excavation are limited by the unfavorable conditions, such as high temperatures and the presence of sand.

Therefore, the analytical investigations were carried out by easily transportable instruments (FT-IR and Vis-RS) that do not need special customs permissions, such as those mandatory for diagnostic systems using X-ray fluorescence. Nevertheless, a laborious bureaucratic procedure was followed before shipping the instrumentation to Egypt.

3.1. CartonnageAGH026-B14

The first investigated artifact, AGH026-B14 (Table 1), was the headwear. ATR/FTIR analyses were carried out on small, detached fragments that were present in the storage box. It was possible to analyze a black and a red-colored fragment (Table 2).

Table 2. Results obtained by ATR/FTIR on *cartonnage* AGH026-B14.

Sample	Color	ATR/FTIR Peaks (cm^{-1})
1-B14	black	hydroxyl about 3700–3300 water 3300, 1622
		organic matter 2925, 2859
		calcite 1417, 872, 712
		Egyptian blue 1160, 1050–1055
		Silicate 1008, 853, 779, 672, 596, 530, 462
2-B14	red	hydroxyl about 3700–3300 water 3300, 1622
		organic matter 2926
		calcite 1406, 872, 711
		Silicate 1007, 780, 670
		hematite 529,463

3.1.1. Sample 1-B14 (Black)

Detecting black pigments solely by FTIR is typically complex. However, the technique was useful in identifying other species contained in the sample (Figure 3 and Table 2) such as calcium carbonate [20,22,23] and silicates [22,24].

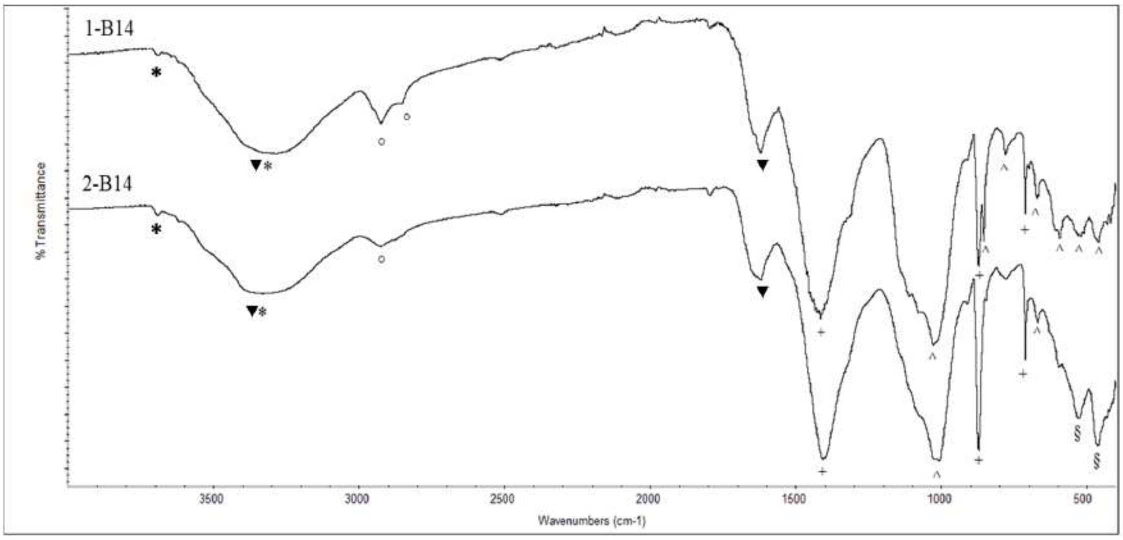


Figure 3. ATR/FTIR spectra of sample 1-B14 and 2-B14. Marker bands of calcite (+), hydroxyl group (*) organic matter (°), water (▼) silicates (^)and ochre (§) are highlighted.

Furthermore, the presence of Egyptian blue mixed with a black hue (possibly a coal-based pigment) could be hypothesized from the signals at 1160 and 1045 cm^{-1}, together with the band at 1008 cm^{-1}. Indeed, the presence of this black pigment can only be confirmed by Raman spectroscopy [7,25,26], which was not available on-site. Furthermore, observations by means of a USB microscope were carried out on this *cartonnage*, indicating blue grains mixed with a black pigment. This confirmed the use of Egyptian blue (Figure 4). It is interesting to note that in other cases the surfaces of the cartonnages were very homogeneous.

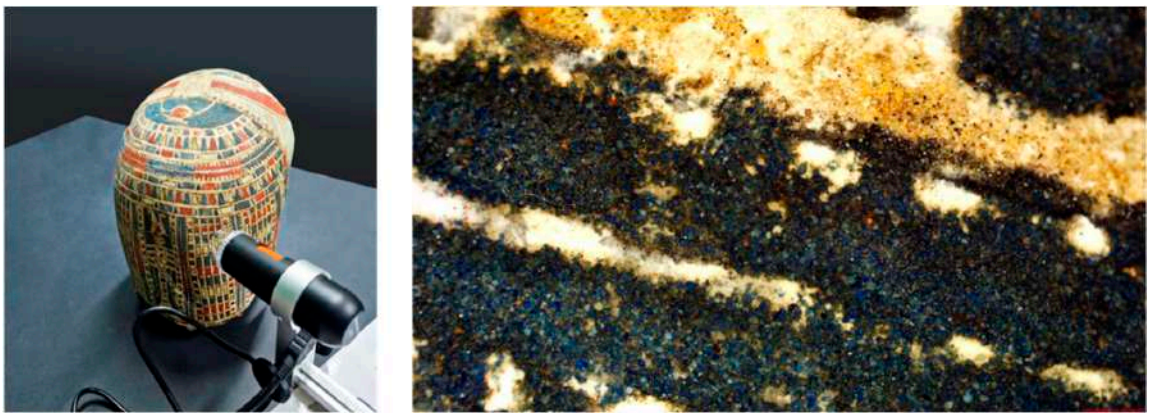


Figure 4. Observation of the surface of *cartonnage* AGH026-B14 carried out by means of a USB microscope (on the **left**) and an image acquired on a black area where blue grains mixed with the black pigment are evident (on the **right**).

It is very common to find silicates within pigments, especially earth pigments. Some bands attributed to silicates can be assigned to quartz or to clays, like those of the kaolin group [20–22,27,28]. Water signals at about 3300 and 1620 and some sharp signals in the range of 3700–3300 due to the hydroxyl group have also been highlighted. Kaolin was identified as a binder added to the preparation layer by other authors [29]. In our case, it is noteworthy that kaolin group minerals are present mainly within red-hued pigments while are absent, for example, in the blue-green ones, allowing to exclude their use in the preparatory layer.

In the spectrum, it is also possible to detect the presence of two bands typically attributable to organic matter, i.e., the bands of the symmetric and anti-symmetric C-H stretching mode at about 2925 cm^{-1} and 2859 cm^{-1} (further signals due to organic binders could be hidden by others bands). The presence of signals from organic substances deriving from the underlying *cartonnage* cannot be excluded. In fact, to achieve this technique in Egyptian funerary art, scraps of linen or papyrus were stuck together with plaster or resins and used to make sarcophagi and mummy masks. Once dried, the material could then be painted or gilded [15,30,31]. Another signal due to organic substances is the shoulder at about 1320 cm^{-1} that could be due to carboxylates (most probably calcium carboxylate) present after the degradation of the binder.

Calcium carbonate deriving from the substrate used to realize the *cartonnage* [7,31] was also highlighted.

3.1.2. Sample 2-B14 (Red)

Infrared spectroscopy is very useful to deduce the nature of red pigments. Indeed, it is possible to observe the characteristic bands of hematite at [26,28,32–34] (Figure 3 and Table 2). In addition, the specific signals of the red ochre pigment, hematite, and clays [24,28] are present. Also particularly evident are the signals due to the presence of some organic matter.

As for sample 1-B14, calcium carbonate from the substrate was present.

3.2. CartonnageAGH026-B13

Analyses were carried out, as for the first *cartonnage*, on small, detached fragments that were present in the storage box of the artifact. In this case, it was possible to analyze blue-green-, dark red-, pink-, gold-, black-, and yellow-colored fragments.

3.2.1. Sample 1-B13 (Blue-Green)

Sample 1-B13 appears blue-green. Egyptian blue was identified [7,26,35–37]. Instead, calcium carbonate is due to the substrate.

It would be interesting to understand whether this pigment is an altered blue or a hue (blue tending to green) obtained by mixing Egyptian blue with a yellow pigment [35], despite these bands seemingly not being present in the IR spectrum (see further on in the text how this pigment has been identified in sample 6-B13).

In addition, the color and spectrum of the sample could suggest another pigment that may have been applied to the artifact, namely the so-called green frit or Egyptian green, which is also synthetic and obtained using a preparation technique similar to that for Egyptian blue [38].

3.2.2. Sample 2-B13 (Dark Red)

In sample 2-B13, the typical and characteristic bands of hematite were observed (Figure 5) together with typical signals of silicates [28,33]. The presence of clay minerals is in accordance with hydroxyl group bands. Calcium carbonate present in the substrate of the artifact was evidenced. Furthermore, there is an evident signal around 1320 cm^{-1} that is attributable to a carboxylate (see sample 1-B14).

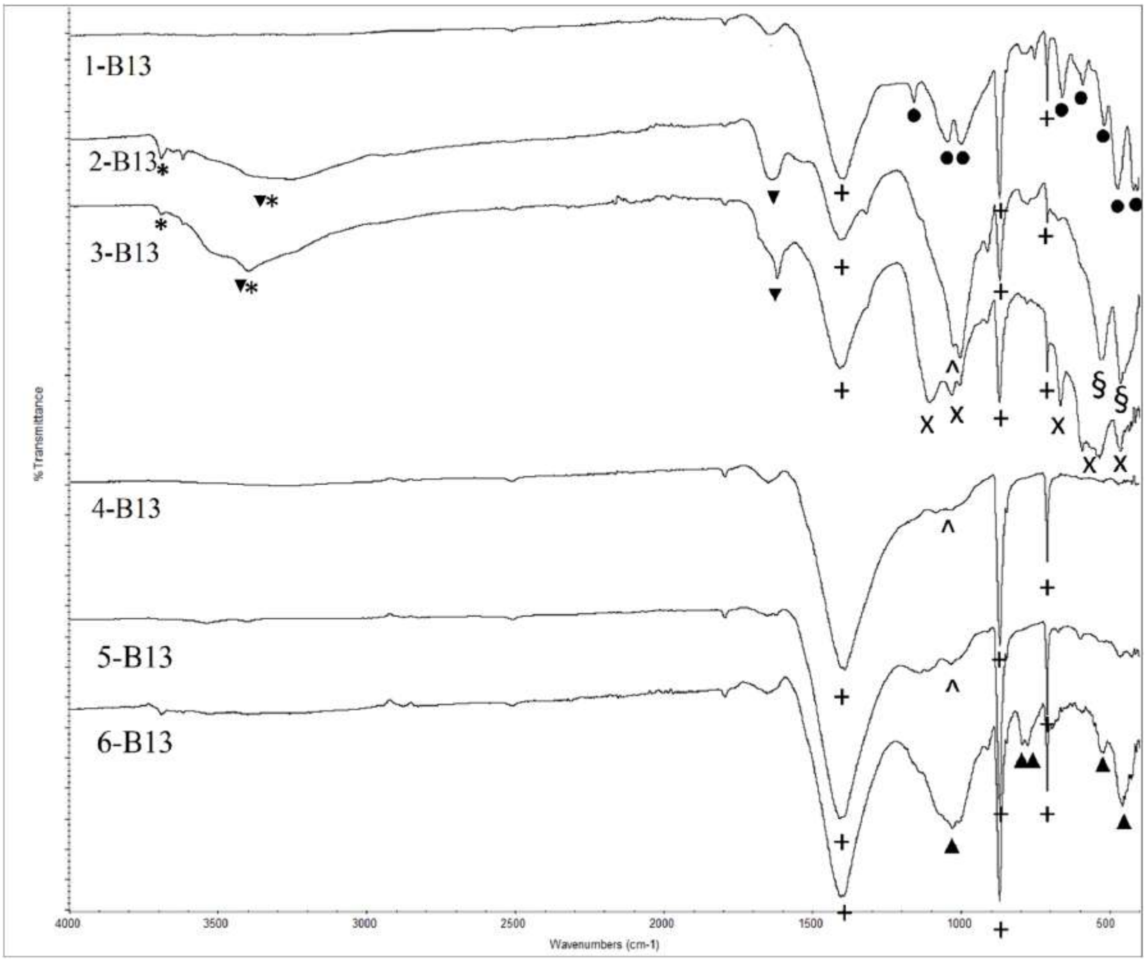


Figure 5. ATR/FTIR spectra ofAGH026-B13 sample. Marker bands of calcite (+), Egyptian blue (●), hydroxyl group (*), water (▼), silicates (^), red ochre (§), alizarine (x) (with the signal at 1100 cm^{-1} overlaid to gypsum), and yellow ochre (▲) are highlighted.

3.2.3. Sample 3-B13 (Pink)

The pink color represents a particular case (sample 3-B13, Figure 5), which shows a signal in the range of 600–1200 cm^{-1} that could be due to madder (alizarine) [39]. This is a natural organic pigment that has also been identified in other cases [40]. Alizarine is an organic compound and a historically important prominent dye. It is an anthraquinone originally derived from the root of the madder plant. Moreover, detailed analysis consisting of substance identification by chromatographic methods could not be performed in this case due to the impossibility of collecting the samples and bringing them back to the laboratory. With regards to pigment identification, it is important to mention that in the *cartonnage* sample having a similar pink hue, studied by multispectral analysis (MSI) in a follow-up campaign (results not presented in this paper), evident fluorescence induced by UV radiation was observed, suggesting the presence of madder as a dye.

For this sample, the presence of gypsum cannot be excluded because of the intense water signal around 3400 cm^{-1} (the signal due to sulphate around 1100 cm^{-1} could also be due to gypsum). Finally, the shoulder around 1320 cm^{-1} is probably ascribable to a carboxylate, as previously highlighted.

3.2.4. Sample 4-B13 (Gold)

The main signals observed for this sample are due to calcium carbonate (Figure 5). Since elemental analysis was not performed, the presence of gold cannot be confirmed. However, a signal at 1652 cm^{-1} due to a protein-type binder (attributable to amide (I)) that may have been used for gold leaf application can be observed. A very weak silicate signal at about 1000 cm^{-1} was also highlighted (Figure 5). As described in great detail in the literature [41], the gold leaf could have been applied to the *cartonnage* by means of lime plaster (based on calcium carbonate) and organic binders. The so-called bole [42], i.e., clays and thus silicate-based earths, was also used.

3.2.5. Sample 5-B13 (Black)

In the case of the black sample 5-B13, only the nature of the substrate consisting of calcium carbonate and silicates could be identified.

As already mentioned, it is difficult to identify black pigments by FTIR alone. However, it is known that black color in Egyptian art was typically obtained from carbon, using what is known as carbon black. This pigment could be easily recognizable and identifiable using the Raman technique [7,25,26], which was not employed in this study.

3.2.6. Sample 6-B13 (Yellow)

In sample 6-B13 the typical signals of yellow ochre were observed [43] (Figure 5 and Table 3) together with quartz, a mineral contained in yellow ochre [27,43]. Calcium carbonate was due to the substrate.

Table 3. Results obtained by ATR/FTIR on *cartonnage* AGH026-B13.

Sample	Color	ATR/FTIR Peaks (cm^{-1})
1-B13	blue-green	calcite 1406, 872, 712
		Egyptian blue 1160, 1050, 998, 872, 712, 661, 592, 473, 419
2-B13	dark red	hydroxyl about 3700–3260 water 3300, 1637
		calcite 1408, 871, 711
		silicates 1005
		hematite 527, 464
3-B13	pink	calcite 1393, 871, 712
		water 3399, 1620
		alizarine 1186, 1090
4-B13	gold	calcite 1407, 871, 712
		silicates~1000
5-B13	black	calcite 1393, 871, 712
		silicates~1000
6-B13	yellow	calcite 1406, 871, 712
		yellow ochre 1029, 796, 778, 529, 456

3.3. CartonnageAGH026-B31

In this case it was possible to analyze blue-green and red areas on fragments detached from the artifact and present in the storage box, as for the previous samples.

3.3.1. Sample 1-B31

The sample 1-B31 (Figure 6, Table 4), as already mentioned for the samples 1-B13, appears blue-green. Egyptian blue was identified [26,35–37] together with calcium carbonate attributed to the substrate.

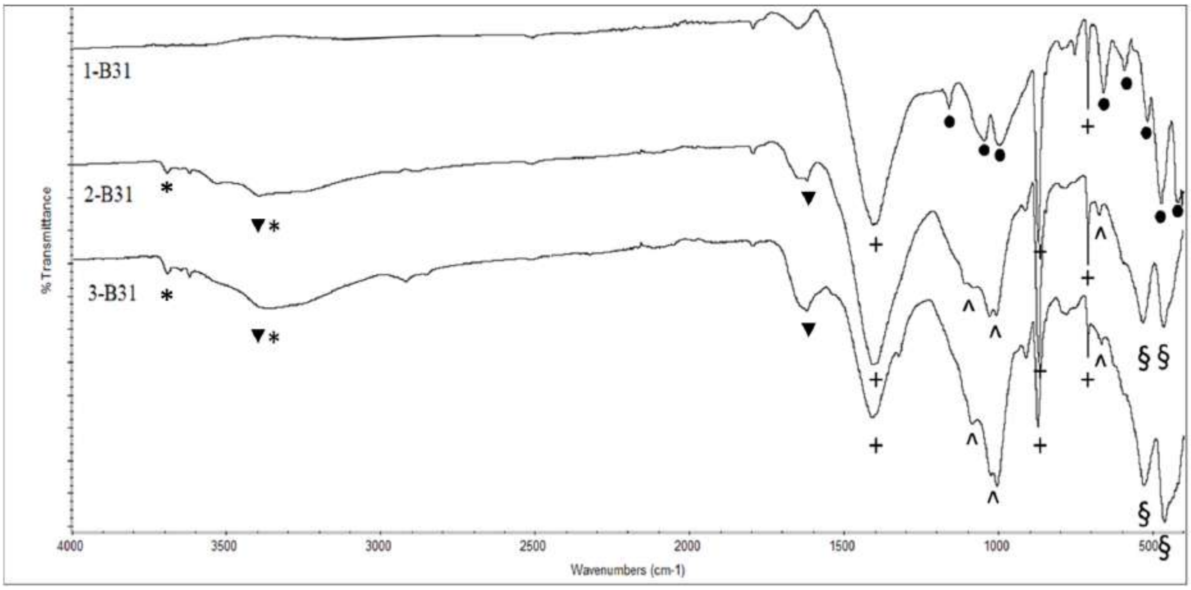


Figure 6. ATR/FTIR spectra ofAGH026-B31sample. Marker bands of calcite (+), Egyptian blue (•), hydroxyl group (*), water (▼), silicates (ˆ), and red ochre (§) are highlighted.

Table 4. Results obtained by ATR/FTIR on *cartonnage* AGH026-B31.

Sample	Color	ATR/FTIR Peaks (cm^{-1})
1-B31	blue-green	calcite 1401, 871, 712
		Egyptian blue 1160, 1046, 1000, 871, 712, 661, 592, 519, 474, 419
2-B31	red	hydroxyl about 3700–3300
		calcite 1407, 871, 711
		silicates 1030, 1008, 672
		hematite 533, 466
3-B31	red	hydroxyl about 3700–3300
		calcite 1409, 872, 711
		silicates 1087, 1005, 671
		hematite 530, 463

3.3.2. Sample 2-B31 and Sample 3-B31

On sample 2-B31 (Figure 4, Table 4), the characteristic bands of hematite were observed together with clays typical of the red ochre pigment [28,33]. Hydroxyl signals confirmed the presence of these minerals. Calcium carbonate is due to the substrate.

The characteristic bands of hematite were also detected in sample 3-B31, thus confirming the presence of red ochre [27,32,34]. As for the other red samples (1-B14, 2-B13 and 3-B13), a shoulder around 1320 cm^{-1}, probably ascribable to a carboxylate resulting from the binder degradation, is evident.

3.4. Cartonnage AGH026-B41

In the case of this *cartonnage*, measurements were carried out directly on-site at the excavation. Unfortunately, it was not possible to transport the fourth *cartonnage* to the warehouse (where the other cartonnages were stored) due to permission issues. Therefore, the only technique that was transportable on the excavation, i.e., Vis-RS, was used. The analyzed points are highlighted in Figure 7.

Sample	Colour
1c	Black
2c	Black
3c	Black
4c	Black
5c	Red/Yellow
6c	Red/Yellow
7c	Green/Blue
8c	Green/Blue
9c	Brown/Orange
10c	Brown/Orange
11c	White/Red
12c	White/Red

Figure 7. *Cartonnage* AGH026-B41 and the list of the points analyzed by portable visible reflectance spectroscopy.

Vis-RS measurements were carried out with the portable spectrophotometer that allowed identification of the color of the sample following comparison with reference spectra. Five groups of samples could be identified by observing the obtained spectra (Figure 8). Samples 1c, 2c, 3c, and 4c were black. In fact, the reflectance spectra show the curve close to the x-axis due to light being almost entirely absorbed. The spectra of samples 5c and 6c show the typical shape of red. Nevertheless, a very bright red has a well-marked inflection point [26], whereas the curve obtained is characterized by a smoother change indicating a more yellowish color [43].

Samples 7c and 8c, on the other hand, show the characteristic shape of a blue-green spectrum: in fact, the curve shows higher reflectance values around 540–620 nm [44].

Samples 9c and 10c resemble samples 5c and 6c but with less pronounced inflection points. The values tending to 700 nm are higher, thus indicating a less red and more brownish color.

Finally, spectra 11c and 12c allow us to deduce the presence of a color tending towards yellow, with low reflectance values at low wavelengths that increase as the wavelength increases in a gradual manner, differently than in the case with the red pigments [45,46].

To better highlight differences among the various hues, the reflectance spectra in the visible range were subjected to treatment by principal component analysis (PCA). In Figure 9, the obtained scatter plot is reported. This type of data processing has been widely used in scientific literature to treat spectral data coming from artifacts of historical and artistic interest [47].

From Figure 8 it can be observed that the black and blue-green colors have a similar behavior, while some variability can be observed for red, brown and pink samples. This might be due to the use of different recipes depending on the type of result, and thus color tone, desired.

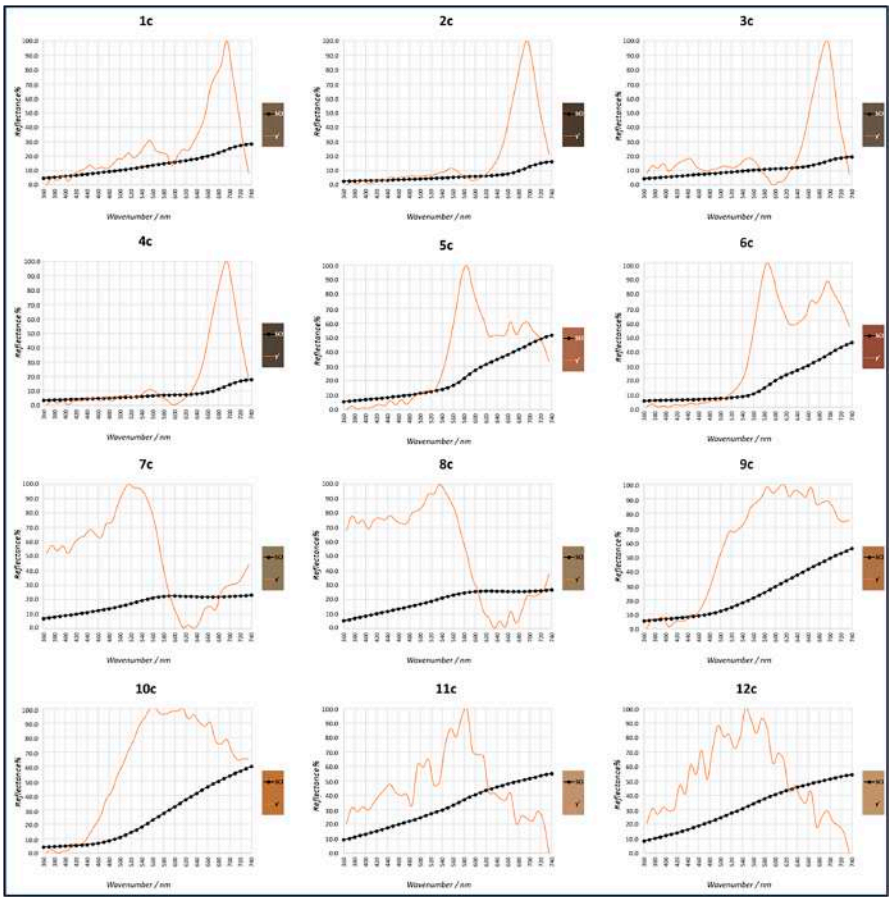


Figure 8. Visible reflectance spectra acquired on the points (**1**–**12c**) indicated in Figure 7 and respective first derivative of all samples of *cartonnage* AGH026-B41.

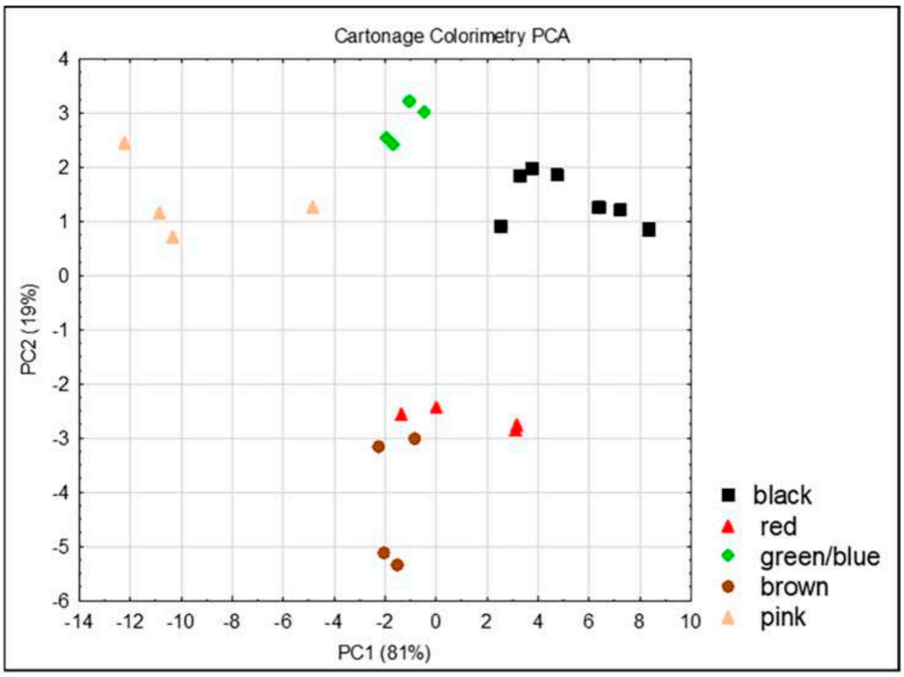
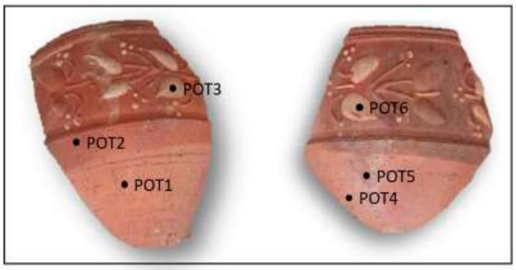


Figure 9. Scatter plot obtained after PCA analysis carried out on visible reflectance spectra acquired on different points on *cartonnage* AGH026-B41.

3.5. Pottery

Finally, two pottery shards coming from tomb AGH032 were analyzed by means of visible reflectance spectroscopy on-site at the excavation. The analyzed pottery is of interest to the archaeologists since it belongs to the same period as the examined *cartonnages*. Reflectance spectroscopy was used mainly to illustrate a methodological approach related to possible field analyses that could be performed in the future on other ceramic fragments excavated in the tombs. The different examined points are shown in Figure 10.



Sample	Colour
POT1	Light brown clay colour
POT2	Brown clay colour
POT3	White
POT4	Light brown clay colour
POT5	Dark brown clay colour
POT6	White

Figure 10. Pottery AGH032-14 and the list of the points analyzed by Vis-RS.

Preliminary information regarding the colors of the samples was collected from the reflectance spectra in the visible range (Figure 11) acquired on pottery.

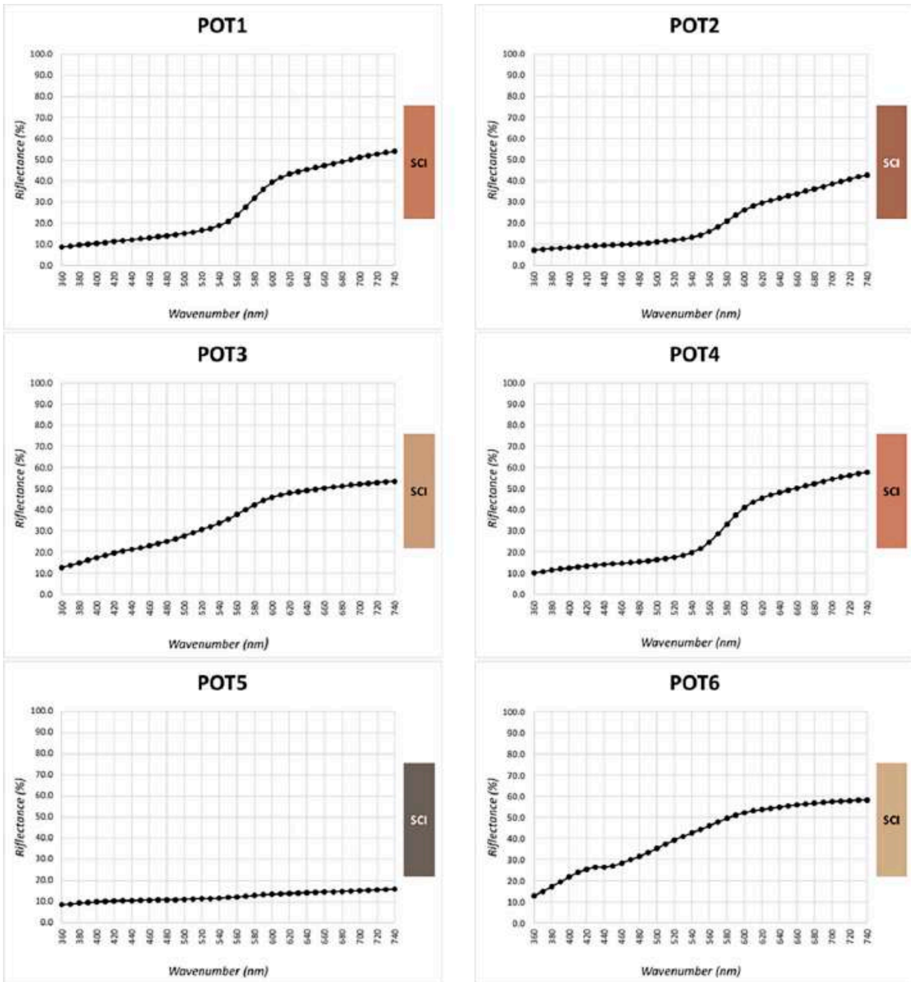


Figure 11. Visible reflectance spectra of all samples of pottery acquired on the points (**POT1–6**) indicated in Figure 10.

With regards to points POT1, POT2, and POT4, it was possible to identify an s-shaped spectrum typical of red tones [44]. In fact, an inflection point is evident around 580 nm in which the curve starts to rise, hence the sample no longer absorbs but reflects the light.

Points POT3 and POT6 appear to also be lighter to the naked eye, appearing almost white. They do not present a marked inflection point but show a smoother curve that rises towards longer wavelengths, indicating a more yellow color.

For point POT5, it can be observed that the spectrum absorbs at almost all wavelengths, corresponding to a darker hue.

4. Conclusions

On-site scientific investigations, including ATR/FTIR and Vis-RS measurements, have been carried out for the first time on artifacts coming from the necropolis surrounding the mausoleum of the Aga Khan in Aswan (Egypt). The instrumentation was used under very challenging conditions and, since no object could be taken outside of Egypt, a full analytical characterization could not be carried out. Despite these constraints, in some cases it was possible to disclose the palette of the *cartonnages*.

Three pigments abundantly used at the time (red ochre, yellow ochre, and Egyptian blue) were detected by means of ATR/FTIR investigations. The spectra of the blue-green samples (1-B13 and 1-B31) and those of the red samples (2-B14, 2-B13, 2-B31, 3-B31) are almost perfectly superimposable in all samples. Some differences were observed for the dark blue and black colors. In fact, a mixture of Egyptian blue and carbon black was employed to obtain a blue-black hue (1-B14).

Looking at other samples, such as the pink one, the use of an organic dye, i.e., madder, was hypothesized. This allowed the *cartonnage* to be dated probably to the first century AD.

It is also worth underlining that the same or similar pigments (except for pink) have been used in the different artifacts. This indicates that the palette remained the same along the manufacturing period considered (the *cartonnages* studied are all dated between the first century BC and the first century AD).

Reflectance spectroscopy applied on *cartonnage* AGH026-B41 revealed that the black and blue pigments applied on the artifact are quite similar, while some differences were observed for pink and red hues.

With regards to the two small ceramics, measurements were made exclusively on-site by Vis-RS in order to provide indications that could be useful if the measurements were to be carried out on other ceramic objects found on the excavation.

In the future campaigns, more accurate and detailed on-site investigations will be performed on the artifacts by other complementary instrumentation transportable on-site (i.e., XRF and Raman portable spectroscopy).

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References

1. Bonizzoni, L.; Bruni, S.; Guglielmi, V.; Milazzo, M.; Neri, O. Field and laboratory multi-technique analysis of pigments and organic painting media from an Egyptian coffin (26th dynasty). *Archaeometry* **2011**, *53*, 1212–1230. [CrossRef]
2. David, A.R.; Edwards, H.G.M.; Farwell, D.W.; De Faria, D.L.A. Raman spectroscopic analysis of ancient Egyptian pigments. *Archaeometry* **2001**, *43*, 461–473. [CrossRef]
3. Hallmann, A.; Rickerby, S.; Shekede, L. Blue and green in the decoration of a Kushite chapel in Karnak, Egypt: Technical evaluation using low-tech, non-invasive procedures. *J. Archaeol. Sci. Rep.* **2021**, *39*, 103190. [CrossRef]
4. Pagès-Camagna, S.; Raue, D. Coloured materials used in Elephantine: Evolution and continuity from the Old Kingdom to the Roman Period. *J. Archaeol. Sci. Rep.* **2016**, *7*, 662–667. [CrossRef]
5. Pozza, G.; Ajò, D.; Chiari, G.; De Zuane, F.; Favaro, M. Photoluminescence of the inorganic pigments Egyptian blue, Han blue and Han purple. *J. Cult. Herit.* **2000**, *1*, 393–398. [CrossRef]
6. Sakr, A.; Tawab, N.A.; Mahmoud, A.; Ghaly, M.F.; Edwards, H.G.M.; Elbashar, Y.H. New insights on plasters, pigments and binder in mural paintings of the Setka tomb (QH 110), Elephantine, Aswan, Upper Egypt. *Spectrochim. Acta Part A Mol. Biomol. Spectrosc.* **2021**, *263*, 120153. [CrossRef]
7. Lombardi, C.A.; Comite, V.; Fermo, P.; Bergomi, A.; Trombino, L.; Guglielmi, V. A Multi-Analytical Approach for the Characterisation of Pigments from an Egyptian Sarcophagus Cover of the Late Dynastic Period: A Case Study. *Sustainability* **2023**, *15*, 2002. [CrossRef]
8. Bonizzoni, L.; Bruni, S.; Galli, A.; Gargano, M.; Guglielmi, V.; Ludwig, N.; Lodi, L.; Martini, M. Non-invasive in situ analytical techniques working in synergy: The application on graduals held in the Certosa di Pavia. *Microchem. J.* **2016**, *126*, 172–180. [CrossRef]
9. Cartechini, L.; Miliani, C.; Nodari, L.; Rosi, F.; Tomasin, P. The chemistry of making color in art. *J. Cult. Herit.* **2021**, *50*, 188–210. [CrossRef]
10. Kovalev, I.; Rodler, A.S.; Brøns, C.; Rehren, T. Making and working Egyptian blue—A review of the archaeological evidence. *J. Archaeol. Sci.* **2023**, *153*, 105772. [CrossRef]
11. Sgamellotti, A.; Anselmi, C. An evergreen blue. Spectroscopic properties of Egyptian blue from pyramids to Raphael, and beyond. *Inorganica Chim. Acta* **2022**, *530*, 120699. [CrossRef]
12. Davies, W.V. *Colour and Painting in Ancient Egypt*; British Museum Press: London, UK, 2001.
13. Afifi, H.A.M.; Hassan, R.R.A.; Abd-EL-Fattah, M.A.; Menofy, S.M. Spectroscopic characterization of preperation, pigment and binding media of archaeological cartonnage from lisht, Egypt. *Sci. Cult.* **2020**, *6*, 9–23.
14. Hunkeler, C. New results on the manufacture and durability of cartonnage: Experimental archaeology as a method to understand the history of 22nd Dynasty cartonnage cases from KV40 (Thebes, Egypt). *J. Archaeol. Sci. Rep.* **2021**, *36*, 102836. [CrossRef]
15. D'Amicone, E.; Aceto, M.; Agostino, A.; Fenoglio, G. Cartonnages in tela e papiro stuccati e dipinti, e inchiostri: Due capitoli del progetto «colore». *Atti Dell'accademia Roveretana Degli Agiati* **2009**, *9*, 173–191.
16. Aufderheide, A.C.; Cartmell, L.; Zlonis, M.; Sheldrick, P. Mummification practices at Kellis site in Egypt's Dakleh Oasis. *J. Soc. Study Egypt. Antiq.* **2004**, *31*, 63–77.
17. Higgs, P. Resembling Cleopatra: Cleopatra VII's Portraits in the Context of Late Hellenistic Female Portraiture. In *Cleopatra Reassessed, The British Museum Occasional Paper*; Walker, S., Ashton, S.-A., Eds.; British Museum: London, UK, 2003; Volume 103, pp. 57–70.
18. Riggs, C. *The Beautiful Burial in Roman Egypt. Art, Identity, and Funerary Religion*; Oxford University Press: Oxford, UK, 2005.
19. Marabini Moevs, M.T. The Roman thin walled pottery from Cosa (1948–1954). In *Memoirs of the American Academy in Rome*; University of Michigan Press: Ann Arbor, MI, USA, 1973.
20. Chukanov, N.V. *Infrared Spectra of Mineral Species*, 3rd ed.; Springer: Berlin/Heidelberg, Germany, 2014.
21. Chukanov, N.V.; Chervonnyi, A.D. *Infrared Spectroscopy of Minerals and Related Compounds*; Springer: Berlin/Heidelberg, Germany, 2016.
22. Gadsden, J.A. *Infrared Spectra of Mineral and Related Inorganic Compounds*; Butterworths: London, UK, 1975.
23. Andersen, F.A.; Brecevic, L. Infrared spectra of amorphous and crystalline calcium carbonate. *Acta Chem. Scand.* **1991**, *45*, 1018–1024. [CrossRef]
24. Saikia, B.J.; Parthasarathy, G. Fourier Transform Infrared Spectroscopic Characterization of Kaolinite from Assam and Meghalaya, Northeastern India. *J. Mod. Phys.* **2010**, *1*, 206–210. [CrossRef]
25. Bell, I.M.; Clark, R.J.H.; Gibbs, P.J. Raman spectroscopic library of natural and synthetic pigments (pre-1850 AD). *Spectrochim. Acta Part A Mol. Biomol. Spectrosc.* **1997**, *53*, 2159–2179. [CrossRef]
26. Guglielmi, V.; Comite, V.; Andreoli, M.; Demartin, F.; Lombardi, C.A.; Fermo, P. Pigments on Roman Wall Painting and Stucco Fragments from the Monte d'Oro Area (Rome): A Multi-Technique Approach. *Appl. Sci.* **2020**, *10*, 7121. [CrossRef]
27. Mortimore, J.L.; Marshall, L.J.R.; Almond, M.J.; Hollins, P.; Matthews, W. Analysis of red and yellow ochre samples from Clearwell Caves and Çatalhöyük by vibrational spectroscopy and other techniques. *Spectrochim. Acta Part A Mol. Biomol. Spectrosc.* **2004**, *60*, 1179–1188. [CrossRef]
28. Bikiaris, D.; Daniilia, S.; Sotiropoulou, S.; Katsimbiri, O.; Pavlidou, E.; Moutsatsou, A.; Chryssoulakis, Y. Ochre-differentiation through micro-Raman and micro-FTIR spectroscopies: Application on wall paintings at Meteora and Mount Athos, Greece. *Spectrochim. Acta Part A Mol. Biomol. Spectrosc.* **1999**, *56*, 3–18. [CrossRef]

29. Afifi, H.A.M.; Etman, M.A.; Abdrabbo, H.A.M.; Kamal, H.M. Typological study and non-destructive analytical approaches used for dating a polychrome gilded wooden statuette at the grand egyptian museum. *Sci. Cult.* **2020**, *6*, 69–83.
30. Nicola, M.; Seymour, L.M.; Aceto, M.; Priola, E.; Gobetto, R.; Masic, A. Late production of Egyptian blue: Synthesis from brass and its characteristics. *Archaeol. Anthropol. Sci.* **2019**, *11*, 5377–5392. [CrossRef]
31. Scott, D.A.; Warmlander, S.; Mazurek, J.; Quirke, S. Examination of some pigments, grounds and media from Egyptian cartonnage fragments in the Petrie Museum, University College London. *J. Archaeol. Sci.* **2009**, *36*, 923–932. [CrossRef]
32. Stamboliyska, B.; Tapanov, S.; Velcheva, E.; Yancheva, D.; Rogozherov, M.; Glavcheva, Z.; Lalev, G.; Dimitrov, M. The altar wall paintings of the catholicon "The Nativity of the Virgin", Rila Monastery, Bulgaria: Identification of the painting materials by means of vibrational spectroscopic techniques complemented by EDX, XRD and TGA analysis. *Spectrochim. Acta Part A Mol. Biomol. Spectrosc.* **2012**, *247*, 119087. [CrossRef] [PubMed]
33. Helwig, K. The characterisation of iron earth pigments using infrared spectroscopy. *Irug2 V A* **1998**, 83–91.
34. Guglielmi, V.; Andreoli, M.; Comite, V.; Baroni, A.; Fermo, P. The combined use of SEM-EDX, Raman, ATR-FTIR and visible reflectance techniques for the characterisation of Roman wall painting pigments from Monte d'Oro area (Rome): An insight into red, yellow and pink shades. *Environ. Sci. Pollut. Res.* **2022**, *29*, 29419–29437. [CrossRef] [PubMed]
35. Edreira, M.C.; Feliu, M.J.; Fernández-Lorenzo, C.; Martín, J. Spectroscopic study of Egyptian blue mixed with other pigments. *Helv. Chim. Acta* **2003**, *86*, 29–49. [CrossRef]
36. Fermo, P.; Piazzalunga, A.; De Vos, M.; Andreoli, M. A multi-analytical approach for the study of the pigments used in the wall paintings from a building complex on the Caelian Hill (Rome). *Appl. Phys. A Mater. Sci. Process.* **2013**, *113*, 1109–1119. [CrossRef]
37. Vahur, S.; Teearu, A.; Leito, I. ATR-FT-IR spectroscopy in the region of 550-230 cm^{-1} for identification of inorganic pigments. *Spectrochim. Acta Part A Mol. Biomol. Spectrosc.* **2010**, *75*, 1061–1072. [CrossRef]
38. Hatton, G.D.; Shortland, A.J.; Tite, M.S. The production technology of Egyptian blue and green frits from second millennium BC Egypt and Mesopotamia. *J. Archaeol. Sci.* **2008**, *35*, 1591–1604. [CrossRef]
39. Pronti, L.; Mazzitelli, J.-B.; Bracciale, M.P.; Massini Rosati, L.; Vieillescazes, C.; Santarelli, M.L.; Felici, A.C. Multi-technique characterisation of commercial alizarin-based lakes. *Spectrochim. Acta Part A Mol. Biomol. Spectrosc.* **2018**, *200*, 10–19. [CrossRef]
40. Abdel-Ghani, M.; Afifi, H.A.M.; Mahmoud, R.; Osman, M. Graeco-romanegyptian cartonnage from the egyptian museum in Cairo, Egypt: Technical and analytical investigation. *J. Archaeol. Sci. Rep.* **2020**, *31*, 102360.
41. Hatchfield, P.; Newman, R. *Ancient Egyptian Gilding Methods*; Sound View Press: Madison, CT, USA, 1991.
42. Medhat, A.; Ali, M.; Abdel-Ghani, M. Analytical investigation on a coptic wooden icon from the 18th Century using SEM-EDX microscopy and FTIR spectroscopy. *Mediterr. Archaeol. Archaeom.* **2015**, *15*, 151.
43. Elias, M.; Chartier, C.; Prévot, G.; Garay, H.; Vignaud, C. The colour of ochres explained by their composition. *Mater. Sci. Eng. B* **2006**, *127*, 70–80. [CrossRef]
44. Cheilakou, E.; Troullinos, M.; Koui, M. Identification of pigments on Byzantine wall paintings from Crete (14th century AD) using non-invasive Fiber Optics Diffuse Reflectance Spectroscopy (FORS). *J. Archaeol. Sci.* **2014**, *41*, 541–555. [CrossRef]
45. Gueli, A.M.; Galvagno, R.; Incardona, A.; Pappalardo, E.; Politi, G.; Paladini, G.; Stella, G. Correlation of Visible Reflectance Spectrometry and Portable Raman Data for Red Pigment Identification. *Heritage* **2024**, *7*, 2161–2175. [CrossRef]
46. Marey Mahmoud, H.H. Colorimetric and spectral reflectance access to some ancient Egyptian pigments. *J. Int. Colour. Assoc.* **2019**, *24*, 35–45.
47. Riu, J.; Giussani, B. Analytical chemistry meets art: The transformative role of chemometrics in cultural heritage preservation. *Chemom. Intell. Lab. Syst.* **2024**, *247*, 105095. [CrossRef]