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MULTISPECTRAL ANALYSIS AND INVESTIGATION OF OVERLAPPING LAYER CARTONNAGE FRAGMENTS FROM EGYPTIAN MUSEUM, CAIRO

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ABSTRACT

This research presents the results of the study of unknown mummiform cartonnage from Egyptian museum stores that dates back to the late period (712 BC until 332 BC). It was decorated with blue-red-black-white and yellow pigments on the canvas support which prepared with thin ground layers. The cartonnage contain many parts that are missing due to poor storage in the basement of the Egyptian Museum and the spread of mice, in addition to stains resulting from their droppings. This has led to the obliteration of some colors, in addition to the separation and micro crack of the ground layer, paintings and layers of canvas supports.

The research aims to study the stratigraphic structure of the Cartonnage Fragments by Optical Microscopy (OM), Film analysis (UV-induced fluorescence imaging) and Multispectral Imaging technique that included (VIS) Visible light, (IR) Infrared CCD Reflected light, (IRF) Infrared Fluorescence, (IRFC) Infrared False Colors, (UVR) Ultraviolet Reflected light, (UVF) Ultraviolet Fluorescence, and (UVFC) Ultraviolet False Colors. The analytical techniques utilized in this study were Scanning Electron Microscopy (SEM) equipped with an energy dispersive X-ray detector (EDS), X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR), Raman and X-Ray Fluorescence (XRF) spectroscopy. The results indicated that canvas support consists of about one to three layers of linen, ground layer consists of calcium carbonate, pigments including Egyptian blue, red Hematite and yellow Orpiment were identified. The binding medium was identified as animal glue.

KEYWORDS: Multispectral Imaging, Raman, XRF, XRD, FTIR, SEM, Cartonnage Fragments.

1. INTRODUCTION

Ancient Egyptian cartonnage is a material made from layers of linen or papyrus, coated with gesso then painted. It was used to make 'masks' (head or upper body covers), foot cases, shaped symbolic plaques and even full body covers which were attached to the mummy-wrapped body prior to burial (D'Auria, 1988; Adams, 1984). During manufacture, moist layers of adhesive-soaked linen and gesso (either calcium carbonate- or calcium sulfate-based), were molded into a particular shape. After drying, the gessoed surface was smoothed before application of paint or gold leaf (Ali, et al 2016). A further layer of gesso was often applied with a brush to the underside of the casing to give it further strength and rigidity (David, 2009). The main periods in which cartonnage was used in Egypt were the Middle Kingdom (2025-1700 BC), the Third Intermediate Period (1069- 664 BC), the called late period (712-323 BC) and the Ptolemaic and Early Roman Periods (330 BC - 250 AD). Each period produced its own distinct style of cartonnage manufacture and decoration (Paul, 1995). The Late Period was the final phase of a vast unbroken, and inherently Egyptian, artistic and cultural tradition that dated back to the beginnings of human habitation in the region. By this time, ancient Egyptian culture had diminished, offering little more than a reflection of a once great culture. Even so, truly splendid works on a smaller, more personal, scale, and larger works echoing a nearly-dead tradition, abound during this period.

During the preservation of Egyptian museum stores, unknown destroyed cartonnage fragments from the late period were found. They were decorated with blue-red-black-white and yellow pigments after preparing the surface of canvas support with thin ground layers. The Cartonnage consists of two overlapping layers, perhaps due to the artist's attempt to use the remnants of an old cartonnage as the support layer, or as a result of manufacturing errors that the artist attempted to fix it by placing a second layer of linen which was prepared by the thine plaster layer and then decorated with different colors (Fig. 1). The fragments show signs of damage from rat and certain types of insects which led to lose a large part of it, in addition to separation and micro crack of the ground layer, paintings and layers of canvas support (Fig .2).

To study the stratigraphic structure of the Cartonnage fragments and identify the materials present on these fragments and how they have changed over time, a combination of digital documentation, non-destructive examination and analysis techniques were used.

Multi-spectral imaging is an extension of standard digital photography whereby an object is illuminated sequentially under different wavelengths from ultraviolet to near infrared and photographed (Fischer & Kakoulli 2006; Verri 2009). It has established as an important non-destructive tool for paintings investigation (Baronti et al., 1998; Bonifazzi, et al., 2007). The painting is irradiated by ultraviolet, visible and infrared rays and the reflected radiation is recorded in a camera sensitive in this region of the spectrum. The image can also be registered using the transmitter instead of reflected radiation. In special cases the painting can be irradiated by UV, VIS or IR rays and the fluorescence of pigments can be registered. Spectral reflectance characterization of painted surfaces can be used in several applications ranging from diagnostics (e.g. It provides information about which materials compose the painted surface) and to digital documentation and monitoring of the conservation state of an artwork in an absolutely non-destructive way (Bonifazzi, et al., 2007).

Film analysis using UV-induced fluorescence imaging is a consolidated technique for the rapid a priori analysis of an artwork since it gathers information on the presence of superficial organic or fluorescent materials, including dyes, binders, varnishes, protective and restoration treatments. Beside this rapid, but qualitative inspection imaging technique, more quantitative information on the properties of a fluorescence emission from an artistic surface can be gathered. With this aim, Fluorescence Lifetime Imaging allows the reconstruction of the emission lifetime in each point of the field of view. Optical fluorescence is the emission of photons from a molecule in an electronically excited state with main properties being its emission spectrum and its emission lifetime, which can be both used for discriminating and studying fluorescent materials. Whereas the emission spectrum gives an insight into the vibrational levels of the ground state of the fluorescent molecule, the emission lifetime is the average time a molecule remains in an excited state before returning to the ground state. It depends on fundamental quantum mechanical properties but also on the micro-environment of the fluorescent molecule. Depending on the nature of the excited state and on the properties of the emitting molecule, the emission lifetime can vary from picoseconds to milliseconds (Comelli et al., 2004) (Comelli et al., 2005).

The aim of this research is to focus on mapping the Egyptian blue pigment and to investigate the distribution of pigments on the surface of the cartonnage by using film analysis and multispectral imaging. Furthermore, non-destructive techniques were also used in this study: USB digital microscopy, Fluorescence Lifetime Imaging (FLIM), scanning

electron microscopy (SEM) equipped with an energy dispersive X-ray detector (EDS), X-ray diffraction (XRD), Fourier transform infrared spectroscopy

(FTIR), Raman and X-Ray Fluorescence (XRF) spectroscopy.



Figure 1: Overlapping layers of the cartonnage



Figure 2: Deteriorated of the cartonnage by rat and certain types of insects

2. MATERIALS AND METHODS

2.1 Samples

Micro samples of canvas, ground, black, greenish blue, red, yellow and white pigment were collected carefully from the destroyed edge, using a micro scalpel, to identify the constituents and degree of deterioration of the cartonnage layers, pigments, and binder.

2.2 USB optical microscopy

Surface morphology and stratigraphic structure of the painted layers fragments were studied using a handheld USB digital microscope (model PZ01 with image sensor 0.3 Mega pixels, its variable magnification ranging from 20 to 500X, photo capture resolution 640x480,320x240 and LED illumination light resource adjustable by the control wheel (Fig. 3).

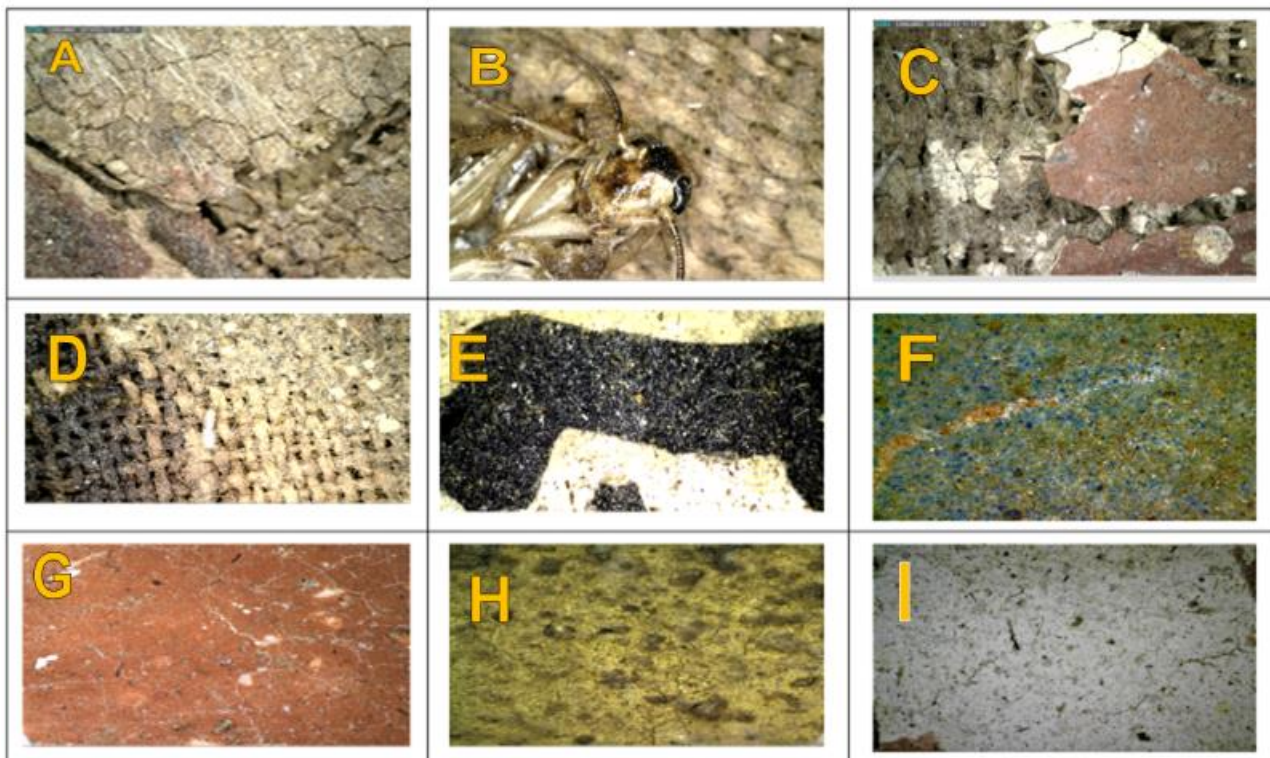


Figure 3: USB digital microscope of the cartonnage fragments shown: A sign of damage from rat, B types of insects, C lose of a large part of it in addition to separation and micro crack of the ground layer, D type of textile (linen canvas), E black pigment, F Greenish blue pigment, G red pigment, H yellow pigment, I white pigment

2.3 FLIM analysis

The portable imaging device developed at Politecnico di Milano is based on a UV pulsed laser source for exciting fluorescence of an area of interest was used (Nevin et al., 2014; Comelli et al., 2004; Comelli et al., 2005). Fluorescence, immediately following laser excitation, decays as an exponential law. By using a time-gated ICCD camera, capable of a minimum acquisition gate of 3 ns, and proper electronic synchronization, we record images at different delays with respect to laser pulses. Through linear algebra it is possible to reconstruct the map of the fluorescence lifetime, which allows one to distinguish the presence of different fluorescent compounds in the analyzed area.

FLIM analysis of the fragments suggests an intense emission from the white painted areas, with a peculiar lifetime of 3.5 ns when data are fitted considering the first 10 nanoseconds decay kinetic (an exemplifying area is highlighted by the red oval in Fig. 4).

This peculiar emission should be related to impurities in the calcite matrix of the white pigment, or eventually to a binder used. An interesting occurrence can be observed in Egyptian blue painted areas when performing UV-induced FLIM analysis. In the lifetime map retrieved considering the first 100 ns decay kinetic, Egyptian blue painted areas show a longer lifetime respect to surrounding areas (see white oval in Fig. 4), as expected remembering that this pigment is actually characterized by a microsecond lifetime when properly excited with red-light and analyzed in the infrared spectral region (Accorsi et al., 2009). Nevertheless, it has to be pointed out that the present set-up is not really suited for the analysis of the visible-induced luminescence emission of Egyptian blue. On the other hand, the lifetime map retrieved considering only the fast decay kinetic (first 10 ns), Egyptian blue painted areas show a very short emission lifetime, much shorter than surrounding areas (see black oval in Fig. 4). This occurrence should deserve further investigations, in order to understand which is the reason for this fast decay kinetic.

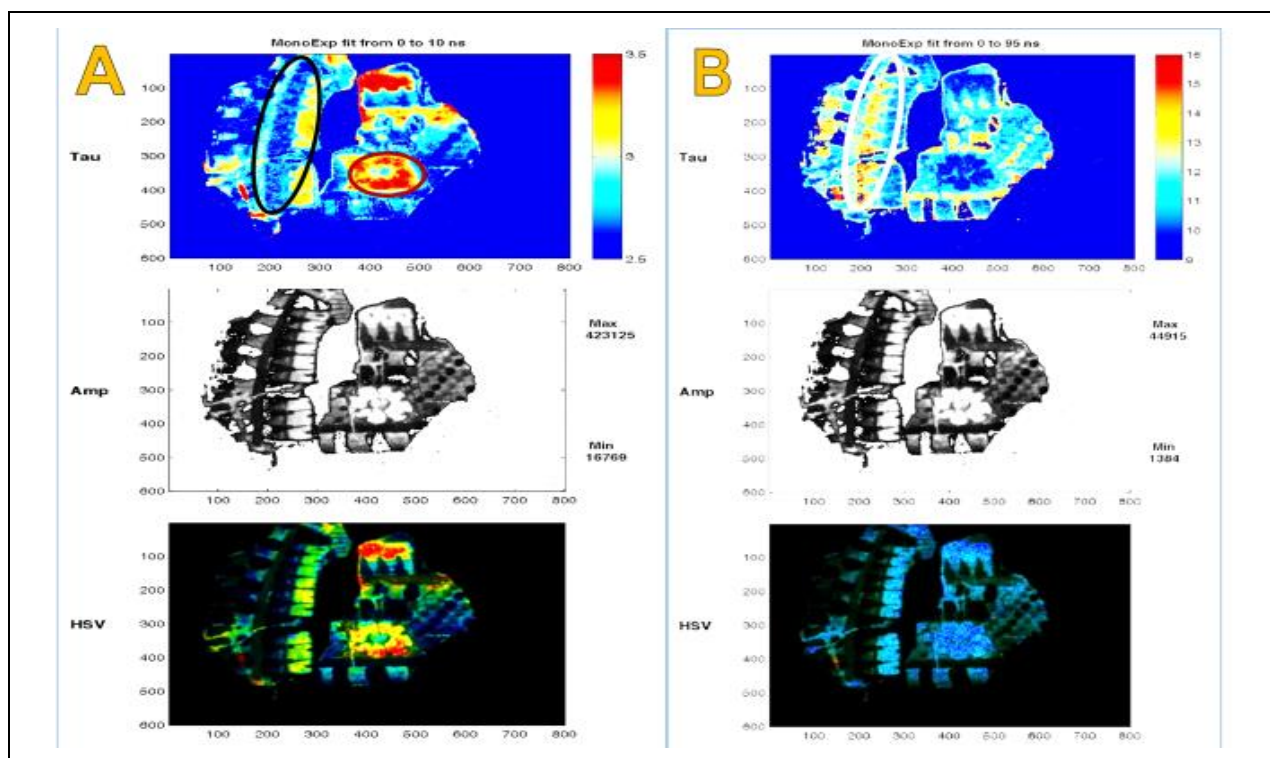


Figure 4. FLIM analysis of the cartonnage fragments. The amplitude of the fluorescence emission following UV and IR excitation two reconstructed lifetime maps, A the first 10 ns and B 100 ns decay kinetic.

Egyptian blue painted areas show a longer lifetime respect to surrounding areas (see white oval), and a very short emission lifetime, much shorter than surrounding areas (see black oval). The peculiar emission of white painted areas, (see the red oval) should be related to impurities in the calcite matrix of the white pigment, or eventually to a binder used.

2.4 MULTISPECTRAL IMAGING

The portable imaging device developed at Politecnico di Milano (Comelli et al., 2004; Comelli et al., 2005 and Nevin et al., 2014) is based on a UV pulsed laser source for exciting fluorescence of an area of interest. Fluorescence, immediately following laser excitation, decays as an exponential law. By using a time-gated ICCD camera, capable of a minimum acquisition gate of 3 ns, and proper electronic synchronization, we record images at different delays with respect to laser pulses. Through linear it is possible to reconstruct the map of the fluorescence lifetime, which allows one to distinguish the presence of different fluorescent compounds in the analyzed area. Multispectral imaging system, with sensitivity in the spectral range 380 - 1100 nm, was employed for recording multispectral images of the ancient the cartonnage fragments under analysis. With the aid of proper optical filters, images in the visible,

near ultraviolet (UV) and near infrared (IR) spectral range have been taken (El-Rifai et al., 2013).

The initial requirements were to focus on mapping the Egyptian blue pigment and to investigate the distribution of pigments on the surface of the cartonnage using multispectral imaging. The Cartonnage fragments measure about 16 x 13 cm and contain about six pigments were studied using the following techniques:- **VIS** - Visible, **IRF** - Infrared Fluorescence (visible induced luminescence (VILS)), **UVF** - Ultraviolet Fluorescence, **IR** - Infrared CCD Reflected, **UVR** - Ultra Violet Reflected, **IRFC** - Infrared False Colors, **UVFC** - Ultraviolet False Colors.

It was found that Egyptian blue can be seen with four different fluorescence intensities; The first appears on the blue-green triangular stripes, followed by the underplayed white-pale blue stripe, the dimmer dark green squares and finally a faint fluorescence of Egyptian blue on the line of black spots between the two lines of red rounded spots on the right fragment (Fig. 5).

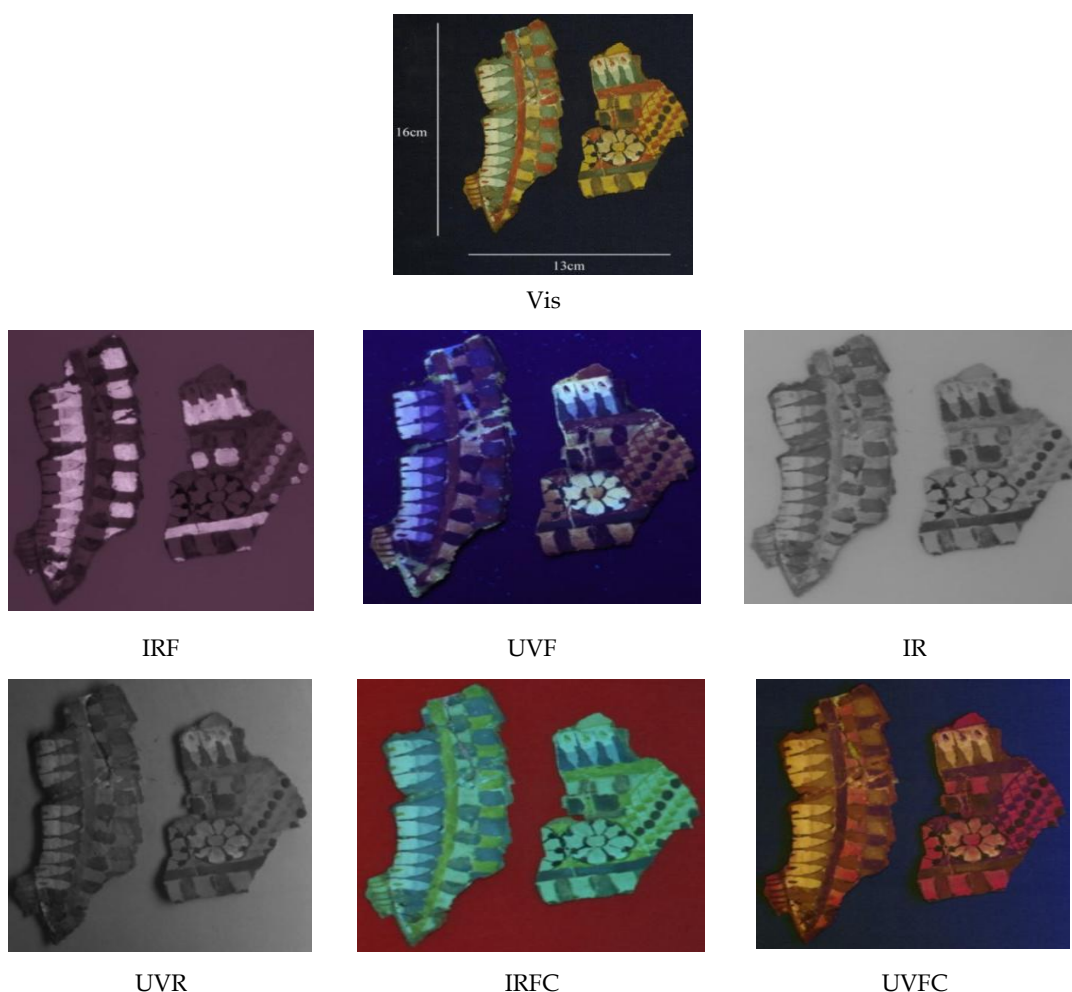


Figure 5: Multispectral imaging of Green-blue pigment, VIS - Visible light, IRF - Infrared, Fluorescence (visible induced luminescence (VILS)), UVF - Ultraviolet Fluorescence, IR - Infrared CCD Reflected, UVR - Ultra Violet Reflected, IRFC - Infrared False Colors, UVFC - Ultraviolet False Colors

2.5 RAMAN SPECTROSCOPY

Raman spectroscopy is one of the preferred methods for the non-invasive analysis of Cultural Heritage objects. The device developed at Politecnico di Milano is a portable scanning Raman spectrometer working in backscattering configuration (Brambilla et al., 2011). Excitation is provided by a 785 nm diode laser source coupled to an optical-fiber-connected to a remote probe. A CCD detector coupled with a spectrometer mounting two different dispersive gratings provides detection. Two different remote probes can be used depending on application and on the nature of the analyzed object: A remote scanning probe, which allows one to perform measurements at a long working distance (30 cm) from a point of interest within an area of a few square centimeters ($9 \times 9 \text{ cm}^2$); the probe is based on a fast optical system ($\text{NA}=0.25$) and includes a pair of galvanometric mirrors for deflecting the laser beam to the target point. A *micro-probe*, based on a 20x objective, may be used for the micrometric analysis of a single

point with a spatial resolution of about $100 \mu\text{m}$ and a working distance of 3 mm. Analysis of (red, yellow, black, Greenish blue) pigments from the cartonnage fragments has been performed mounting the micro-probe with the following working conditions:

Spatial resolution: $100 \mu\text{m}$, • Spectral resolution: 20 cm^{-1} , • Spectral range: $150\div 3000 \text{ cm}^{-1}$, • Integration time: $1\div 10 \text{ s}$, Density Power on sample: $200\div 300 \text{ W/cm}^2$.

The two analyzed red painted areas (RED 1 and RED 2) showed the same Raman signature, suggesting the presence of red earth (Hematite) with chemical composition Fe_2O_3 (fig. 3A). Raman spectrum on a yellow painted area allows the identification of Orpiment- with chemical composition As_2S_3 . For the black and blue pigment no Raman band was collected nevertheless on these painted layers the presence of a very strong luminescence emission peak at 890 nm confirmed the nature of the pigment as Egyptian blue (Table 1) (Fig. 6).

Table 1: Raman identified bands (s= strong, m= medium, w = weak, v = very, sh = shoulder, br= board)

Color of the analyzed point	Identified pigment	Raman bands (cm^{-1})
Yellow	Orpiment- As_2S_3	292m; 309s; 353vs; 381w
Red	Red ochre / hematite- Fe_2O_3	220vs; 286vs; 402m

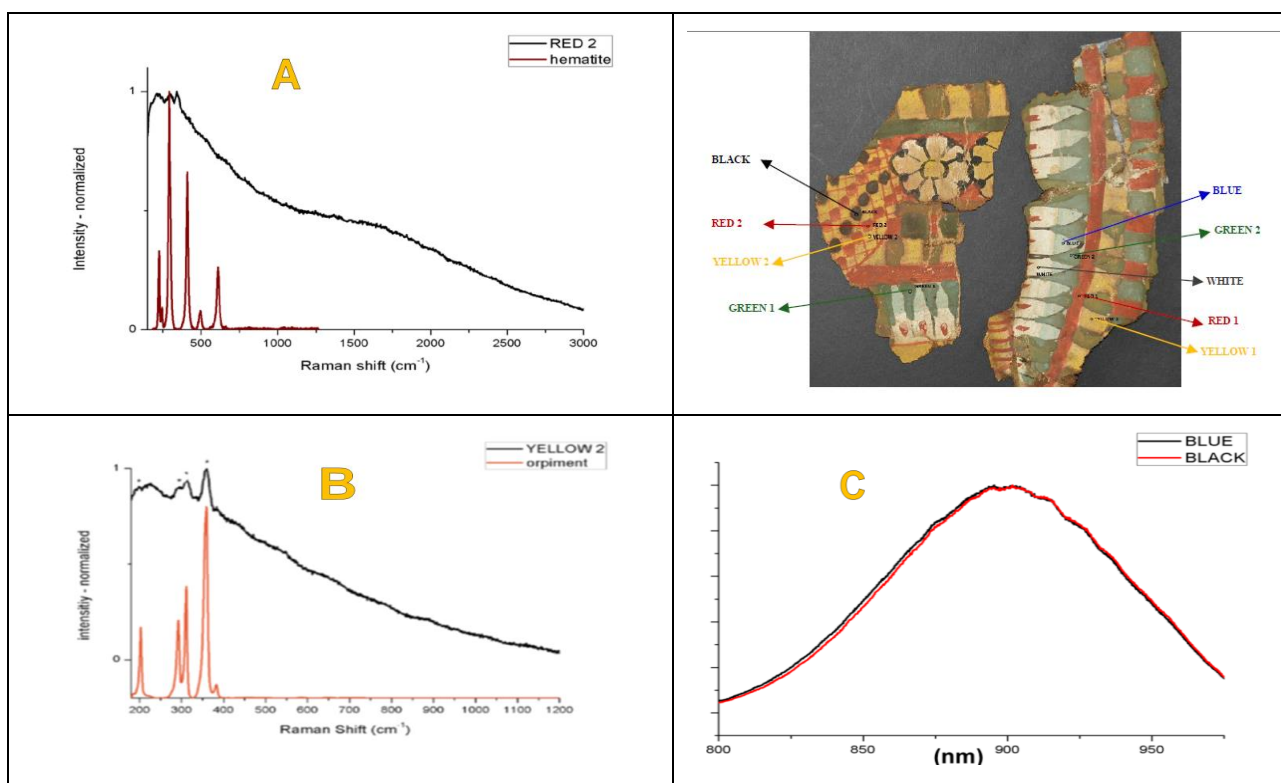


Figure 6: Points analyzed with Raman spectroscopy on cartonnage fragment, A Raman spectra of red painted areas with Hematite, B Raman spectra of yellow painted areas with Orpiment, C Spectra acquired on the black and Greenish blue painted areas, exhibiting a strong luminescence emission peaked at about 890 nm which suggest the presence of the Egyptian blue pigment.

2.6 XRF SPECTROSCOPY

X-ray fluorescence (XRF) analysis was performed with a portable EDXRF spectrometer (Elio Spectrometer, XGlab srl, Milan, Italy) specifically designed for in-situ analyses. The instrument permits the detection of elements from Na to U, with the field of analysis extending between 1 and 40 keV. X-ray radiation is generated using an Rh tube, with an electron accelerating voltage from 10 to 50 kV and a filament current from 5 mA to 200 mA. Beam focusing with a suitable collimator yield a spot size of the

sample of 1 mm in diameter. A large area Silicon Drift Detector (SDD), with an active area of 25 mm² and a 12 μm beryllium window, is used as the detection unit. The typical energy resolution is below 135 eV on the Mn-Kα fluorescence line and the typical Peak to background ratio are of the order of 10,000 XRF measurements of pigments confirmed the presence of Iron (Fe) in red pigment, Arsenic (As) in yellow pigment, copper (Cu) in greenish blue pigment, calcite (Ca) in white pigment and ground (Table 2) (Fig. 7).

Table 2: XRF results of the studied sample

Sample	Element							
	Si	Sn	Cl	K	Ca	Fe	Cu	As
Red	+				+	+		+
Yellow			+			+		+
Black			+		+	+		+
Greenish -blue	+	+			+		+	+
White	+			+		+		+
Ground			+		+	+		+

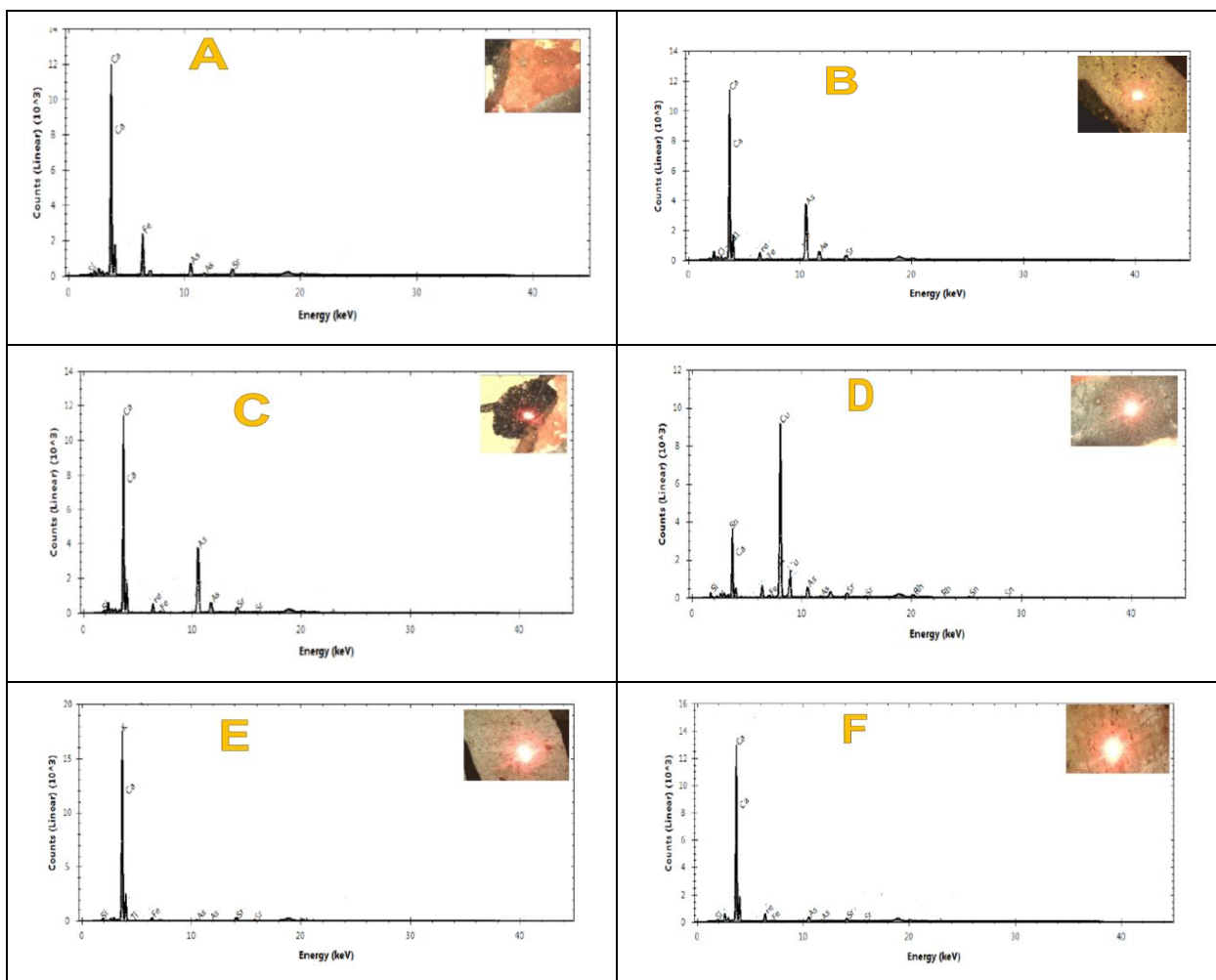


Figure 7: XRF results of the studied sample, A red pigments confirmed the presence of Iron (Fe) , B yellow pigment confirmed the presence of Arsenic (As) , C black pigment , copper (Cu) in greenish blue pigment , calcite (Ca) in white pigment and ground.

2.7 SEM with the energy dispersive X-ray spectrometry (EDAX).

Quanta 200 ESEM FEG from FEI Scanning Electron Microscope SEM, equipped with an EDX micro-analytical system, model FEI Quanta 200, with a field-emission source, offering a wide range of operating conditions, in which specimens can be exam-

ined with high chamber pressure environment. The X-ray analysis was carried out at 20 KV accelerating voltages. This examination was performed to detect the element's contents qualitatively and quantitatively in the samples. Images were acquired in backscattered mode (BSE) (Table 3) (Fig. 8).

Table 3: SEM-EDS data of the studied pigment

Sample	Element %													
	Na	Mg	Al	Si	P	S	Cl	K	Ca	Fe	Cu	Zn	As	Total
Red	-	-	9.46	15.01	1.44	4.88	11.20	1.95	35.78	18.66	1.19	0.43	-	100.0
Yellow	0.65	7.93	0.99	8.55	-	22.63	3.33	1.46	34.86	5.65	1.19	-	13.76	100.0
Black	0.63	1.22	3.09	12.72	-	3.84	8.53	3.43	54.83	10.01	0.46	0.07	-	100.0
Greenish-blue	1.79	1.02	0.12	39.46	-	3.71	3.01	2.04	31.46	9.10	7.68	0.62	-	100.0
White		0.37	3.42	8.54	0.93	1.64	4.03	1.23	66.25	13.58	0.38	-	-	100.0

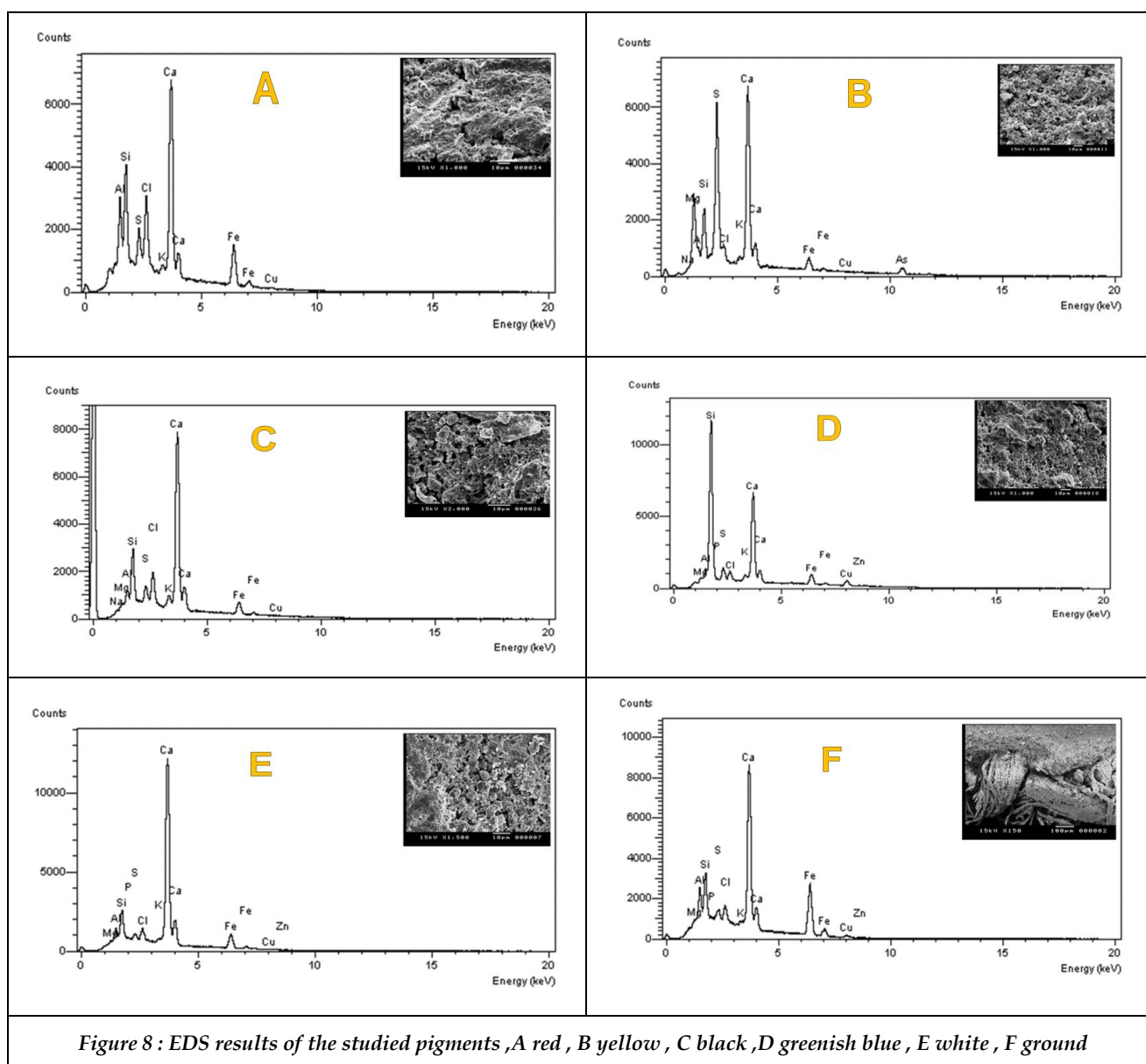


Figure 8 : EDS results of the studied pigments ,A red , B yellow , C black ,D greenish blue , E white , F ground

2.8 XRD SPECTROSCOPY

Cartonnage samples were analyzed using a Philips Analytical X-Ray B.V.; PC-APD diffraction software; diffractometer type: PW 1840, with a Cu tube anode; generator tension 40 KV and generator

current 25 MA. The Cu K α radiation consists of K α 1 (0.154056 nm) and K α 2 (0.154439 nm) components. X'Pert High score software was used for identifying the components of the painted layers (Table 4).

Table 4 : XRD results of the studied sample

Sample	The approximate mineralogical results							
	Calcite CaCO ₃	Quartz SiO ₂	Hematite Fe ₂ O ₃	Orpiment As ₂ S ₃	Cuprorivaite CaCuSi ₄ O ₁₀	Wollastonite CaSiO ₃	Atacamite Cu ₂ Cl(OH) ₃	Halite NaCl
Red	+	+	+++	-	-	-	-	-
Yellow	+	+	-	+++	-	-	-	-
Black	+	+	-	-	-	-	-	-
Greenish - blue	+	+	-	-	+++	+	+	+
White	+++	+	-	-	-	-	-	+
Ground	+++	+	-	-	-	-	-	+

+++ = major constituent ; ++ = minor constituent ; + = traces constituent ; - = not detected

2.9 FTIR SPECTROSCOPY

Fourier Transform Infrared (FTIR) Spectrophotometer, Shimadzu IR Prestige 21 was used to study the vibrational bands that provide information about the chemical functional groups of a sample. Sample fragments were analyzed with an FTIR spectrometer

Vertex 70 (Bruker) with ATR crystal accessory (Platinum diamond ATR) and standard MIR source, at 2 mm/Sec in the spectral region, ranging from 4000 to 500 cm⁻¹ to 4 cm⁻¹ resolution, to identify the organic binding media and some inorganic functional groups in the painted layer (Fig. 9).

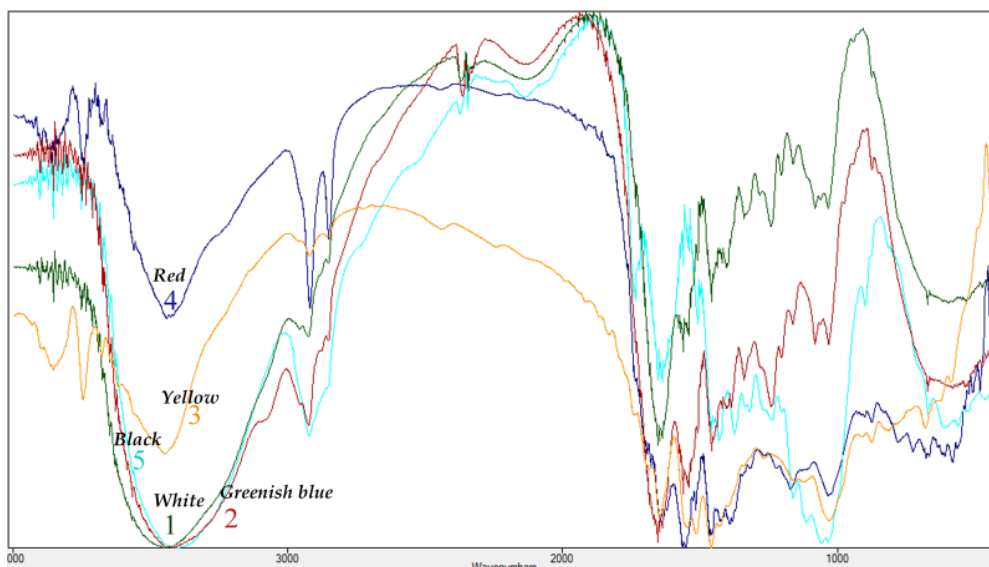


Figure 9 : FTIR spectrum of pigments shows that animal glue was used as a binding medium

3. RESULTS AND DISCUSSIONS

3.1. GROUND LAYER

The preliminary observation of the collected fragments revealed the presence of three main layers: a thin paint layer, a white preparation layer underneath and a textile support which consists of about one to three layers of linen. The preparation technique used seems to apply the white preparation layer directly on the canvas support followed by the painted layer. This structure was observed in all

samples and could also be visualized in the SEM results. Based on the results obtained, XRD analysis revealed that the preparation layer was composed of calcite CaCO₃ (Ali et al., 2016).

3.2. BINDING MEDIA

The spectroscopic study was essentially addressed to characterize the coloring medium used in the pigment samples. In all the analyzed samples, the use of animal glue was revealed in the FTIR spectrum by the presence of a band in the region

(1600- 1650) cm^{-1} , assignable to C=O stretching (amide I) and a band in the region (1500- 1550) cm^{-1} associated with C-N stretching and the deformation vibration of the N-H (amide II) (Derrick et al, 1999).

3.3. PIGMENTS IDENTIFICATION

3.3.1. RED PIGMENT

Red earth (Hematite) could be recognized from the comparison of the results of XRF and Raman methods. Raman spectroscopy, performed on different red points of the fragments, showed the same Raman signature suggesting the presence of hematite with chemical composition Fe_2O_3 (Afifi, 2012), with evidence of Raman bands at 220, 286 and 402 cm^{-1} . The crystal shape of this pigment is observed by using SEM, while XRF results confirm a consistent presence of Iron (Abdrabou et al., 2017).

3.3.2. YELLOW PIGMENT

Raman spectrum on a yellow painted area suggests the presence of Orpiment with chemical composition As_2S_3 with clear evidence of the bands at 230; 292; 309; 353 cm^{-1} . The presence of arsenic in the XRF data confirms this result. The XRD pattern shows the presence of orpiment and calcite, confirming also that the yellow color was composed of orpiment pigment. The presence of calcite is related to preparation layer. The crystal shape of this pigment is observed by using EDS. (Ali et al., 2018).

3.3.3. WHITE PIGMENT

XRF analysis reveals the presence of Ca which leads to characterize the white color as calcite. XRD analysis shows the presence of calcite confirming the result of XRF analysis. No Raman bands were detected due to the presence of a very strong luminescent background. The crystal shape of this pigment is observed by using SEM.

3.3.3. GREENISH BLUE PIGMENT

Greenish Blue pigment appears as dark and light colors in different parts of the fragments. XRF analysis reveals the presence of Cu, Ca, Si, Sn and Fe. XRD analysis shows the presence of cuprorivaite and quartz indicating that this green color was Egyptian blue {calcium copper silicate ($\text{CaCuSi}_4\text{O}_{10}$)}. No Raman band was detected due to the presence of a very

strong luminescent background. An emission spectrum peaked at 890 nm suggests the presence of the Egyptian blue pigment. Egyptian blue appears in greenish blue it will be the result of the manufacturing process and the percentage of raw materials used (XRD indicated the presence of Wollastonite) (Bianchetti et al., 2000; Newman et al 2002). This may be due also to the transformation of Egyptian blue as a result of deterioration by Sodium chloride salt (XRD indicated the presence of Atacamite) (Ali, 2003). The presence of atacamite in blue pigment gave rise to the suggestion that there was a copper chloride cancer in E.B, which was due to the reaction of copper ions included in the composition of the pigment with chlorine ions-resulting from the decomposition of Halite excited (Schiegl et al., 1989; Ali, 2002).

5. CONCLUSION

In our study, the use of multiple imaging methods with different rays helped in determining the actual tonal tones of the unknown mummiform cartonnage from Egyptian museum stores which dating back to the late period (664 BC until 332 BC,) that were hidden as a result of the damage. It was confirmed that the greenish blue color is one of the Egyptian blue tones. Painting technique, pigments and binding media used were characterized. The canvas support consists of about one to three layers of linen. The painting layers consists of: A fine preparing layer, made of calcite only and painted with red, yellow, black, blue, white pigment. This is a very typical cartonnage painting technique. The results indicated that the Egyptian blue transformed in some part to greenish blue, red hematite and yellow Orpiment were identified. These pigments were among the common pigments used in Egyptian cartonnage paintings. The binding medium in the painting was identified as animal glue, also found in other Egyptian cartonnage paintings. The obtained results will help the conservators to set up a scientific plan for restoration and preservation. In fact this cartonnage was not registered before in museum storage records and after the present work of restoration and conservation the cartonnage completed, the management of Egyptian museum made a new number in records (pv.2015.14).

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