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A Multi-Analytical Approach Applied to the Archaeometric Study of a Greco-Roman Decorated Wooden Panel from Egypt

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Abstract

The present manuscript describes a series of non-invasive analyses of a Greco-Roman Period polychrome consecration panel, found during excavation south of the pyramid of Djedkare at Saqqara (Haram el-Shawaf) and now located in the Atfeh repository (Cairo). Multiple techniques of examination and analysis (optical microscopy, fluorescent microscopy, scanning electron microscopy/energy dispersive X-ray spectroscopy, X-ray diffraction, and Fourier transform infrared spectroscopy) were undertaken to study the structure of the panel and chemical compositions of the base layer and pigments.

Introduction

Consecration panels were used in ancient Egypt for the purpose of worship during life, as well as for use in funerary contexts. The earliest examples of such panels were crafted in stone, with wooden ones being a later development. The artists mostly represented themes from ancient Egyptian mythology that continued to the Greco-Roman era. The rapid growth of modern analytical techniques developed by natural and physical sciences allows the derivation of a great deal of information about the techniques used to create these objects and about their state of preservation.[1] The use of such analytical methods plays an essential role in the characterization of the materials and techniques of ancient objects. Painted wood, for example, can be studied by multiple techniques: the optical microscope is a principal tool for studying wooden materials, as well as painted surfaces for identification of fibers in artifacts such as wood and textiles.[2] X-ray diffraction (XRD) is used to identify the crystallised pigments used on painted surfaces and the ground layer, and it can as well work with cellulosic materials.[3] The importance of using X-rays as a non-destructive method for analysis of colors is an important application for archaeology.

Examination and identification of textile fibers may be achieved with a scanning electron microscope (SEM).[4] SEM also aids in the identification of the structure of the wood, and the potential for advanced microscopes to visually enlarge a sample up to 400,000 times facilitates the study of changes in archaeological wood.[5] Fluorescence microscopy is an excellent method for the kind of substances that can be forced to emit fluorescence radiation by their own nature (primary fluorescence) or by staining with a specific fluorescent (secondary fluorescence).[6] Fourier transform infrared spectroscopy (FTIR) is used to identify molecular functional groups in the components; it is used also in the identification of organic and inorganic substances that have varying degrees of absorption in the infrared range.[7] The aim of the present paper is to characterize a painted consecration panel by multiple analytical methods that identify the materials of the wooden support, the painted textile, and their techniques of manufacture.[8]

Materials and Methods

Materials

The panel under consideration here dates to the Greco-Roman period, having been found in excavations. The excavations were carried out by Abd El Hamid Zayed at the Haram el-Shawaf which is south of Dejakare's pyramid in Saqqara in the 1954-1963 period. It measures 47 cm x 30 cm, with a thickness of 3 cm (Figure 1) and is currently stored in the Atfeh magazine. The structure of the panel consists of successive single or group of layers (Figure 2): a base of wood, multiple layers of textiles, a base (or ground) layer, and a painted layer. The painted layer depicts the goddess Hathor, who appears in the middle of the panel clutching an ankh in her right hand and lifting her left.

Methods

Optical Microscope

Observations of the coarse or prepared samples were performed using an Olympus BX51 stereomicroscope equipped with an Olympus DP10 digital camera. The optical images were captured in the reflected light, which helped to identify the structure of the paint layers and the color of certain individual pigment grains.

Fluorescent microscopy

Fluorescent microscopes use an illumination method that locates fluorescent materials by exciting the specimen with one wavelength of light in hopes that the fluorescence will appear by emitting a light at a different wavelength. The samples were studied with an Olympus BX60 microscope.

Environmental Scanning Electron Microscope (ESEM)

The morphology and chemical compositions of the panel were studied using SEM/EDS spectrometry, SEM–EDS, employing a Philips XL30 instrument, with an INCA Oxford spectrometer package, with an LaB6 source and an EDAX/DX4 detector, at a working distance of 10 mm, using an accelerating voltage of 20 kV, a spot size of 4.7 to 5.3 nm (INCA conventional units), and a process time of 5 seconds, corresponding to a detector dead time of 25–40%; and an acquisition time of 75 seconds. EDX data acquisition was obtained through GENESIS 6.x software.

X-ray Diffraction (XRD)

Non-destructive XRD analysis was carried out with a Buker Focus 8 with a Ni filter, Cu-Kα radiation 1.54056Å at 40kV, 25mA, 0.05°/sec. It had a high-resolution graphite monochromatic, rotating sample holder and a proportional detector. Measurements were carried out on the samples in the range $0° < 2\theta < 70°$ in steps of 0.02°.

Fourier Transform Infrared Spectroscopy (FTIR)

FTIR is a powerful tool for analyzing both organic and inorganic materials, including crystalline and amorphous minerals. FTIR was carried out by a Perkin-Elmer spectrometer. Samples were prepared and examined in transmission mode at a spectral resolution of 4 cm^{-1} over 32 scans. A background of the clean diamond cell was performed for each analysis undertaken; spectra were recorded in the 4000–650 cm^{-1} region.

Results and Discussion

The Support (Wood)

Optical microscope: The results of an investigation of a tangential section under optical microscopy confirmed that the wood in the panel is willow (*Salix* sp.), which shows the heterogeneous of parenchimatic rays under microscope, as well as irregular grain with a medium-to-fine uniform texture. The section shows a diffuse porous tissue with ring boundaries (Figure 3).

SEM: Results of SEM techniques applied to the wooden support agree with OM results. SEM shows cracks and deformation in the supportive cells of the wood (Figure 4).

Textile

Optical Microscope: The observation of the shapes of the fibers in cross sections under the microscope reveals hair-like flax fibers that have multi-sided cylindrical filaments with finely pointed ends (Figure 5).

Fluorescent Microscopy: The principle of fluorescence microscopy is to examine specimens containing fluorescent materials. The analysis shows the presence of animal glue that had penetrated between the linen fibers. It was used as a binder and over a long time caused the fibers to become hard and brittle. Under the fluorescent microscope, the cross section of pigment shows resin separated from the pigments layer, which indicates that the pigments have been exposed to a previous consolidation process. However, there is a report on a previous consolidation treatment of this artifact; the information was obtained from the head of conservation department in the repository (Figure 6).

SEM: The morphology of the surface of the fabrics was investigated using a scanning electron microscope. This revealed degradation as cracks. Fragility and weakness results from ageing and degradation occurring at high temperature in the manufacture or from the drying of cellulose and fibers. Twisting and warping are present in the fabric (Figure 7). The structure of the nodes is clearly observable in the fibers, and the weave pattern of the linen textile is plain weave (1x1).

FTIR: Residue on the textile was measured using the diamond anvil cell and compared to the reference animal glue. The spectra are similar; only small differences in the intensities could be found (Figure 8).

Paint

Optical Microscope: Investigation indicated that the thickness of the paint layer, including the ground layer and pigments, was between 200–400 μm and showed the heterogeneous thickness of the paint layer (Figure 9). The surface of the layer is likewise uneven. The cross section shows that the red pigments have penetrated into the ground layer.

Fluorescent Microscopy: Investigation of the paint surface indicates the presence of polymeric materials with specific fluorescence (Figure 10). According to the conservators, the panel was consolidated by using Klucel G (a hydroxylpropylcellulose compound) a few years ago.

SEM: Analysis of the ground layer and pigments by scanning microscopy shows a high proportion of calcium in the ground layer, with a small percentage of silica (Si) and some impurities (Al, Mg, K, Na) (Figure 11). These impurities indicate clay minerals present in the ground layer.

Black pigment: Data obtained by SEM/EDS showed that carbon was used as the source for black paint, silica (Si). In the same way, impurities (Al, Mg, K, Na) (Figure 12) indicate the presence of clay minerals in the ground layer, while the high percent of Ca refers to the ground layer. This resulted from cracks and thinness of the pigment layer, which allowed electrons to penetrate to the ground layer.

Red pigment: The principal red pigments according to SEM/EDS can be from two main types: red iron oxide (hematite) and red ochre (hydrated iron oxide, perhaps partially dehydrated goethite). Hematite is relatively abundant in sedimentary rocks among geological formations in Egypt, and impurities in the red pigments such as Na, Al, and Mg indicate low purification of raw pigments (Figure 13).

XRD

Red pigment: Data obtained by XRD showed that hematite and goethite were used in the decorated wooden panel. The presence of calcite refers to the ground layer, although the XRD pattern indicates clay minerals in the region before 10(2Θ) (Figure 14).

Conclusion

The multiple analytical methods that we have used provided information to identify the composition of the consecration panel. The support used is willow wood (*Salix* sp.). For the base or ground layer, multiple analytical methods confirmed the use of powdered limestone; finally, iron oxide and carbon were employed for the painted layer. Furthermore, it is noticed that craftsmen in Egypt during the Late Period, at least in this case, were not attentive to the purification of their raw materials. This much is clear from the clay minerals present in the pigments and base layer. It is hoped that the results presented here will help conservators determine appropriate conservation strategies for similar panels (e.g., water-based materials should be avoided).

Figure 1: Decorated panel.

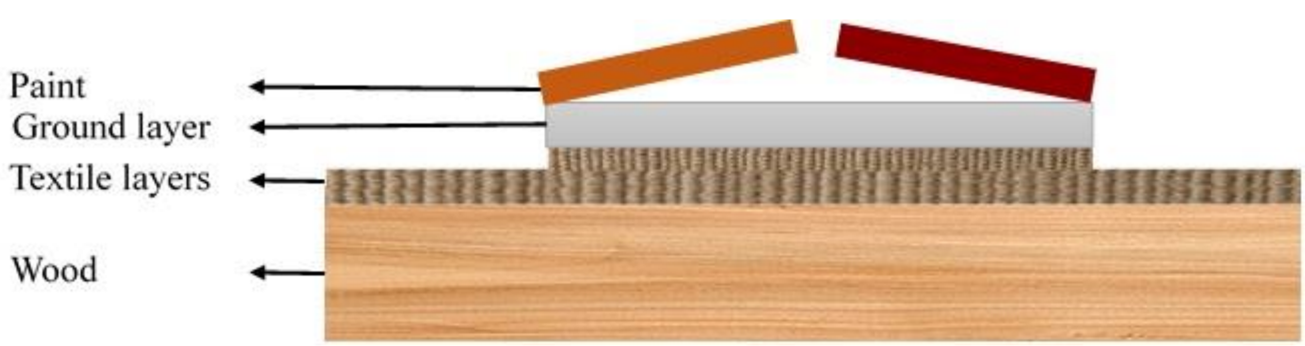

Figure 2: The structure of the consecration panel.

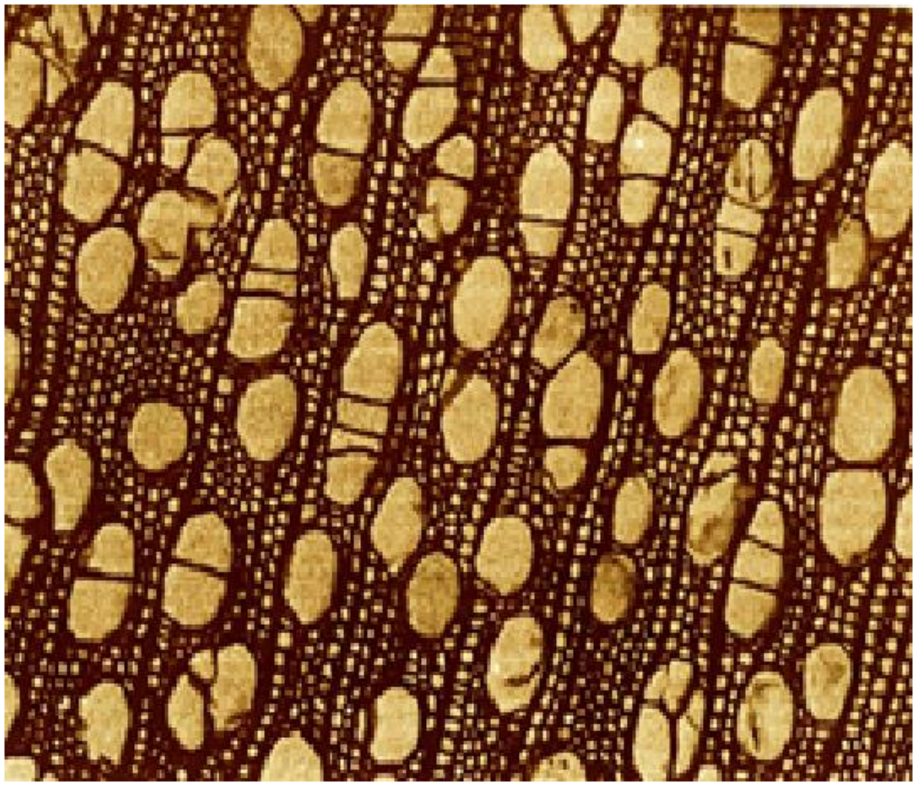

Figure 3: Optical microscope photo of section confirming that the type of the wood is *Salix* sp. (willow).

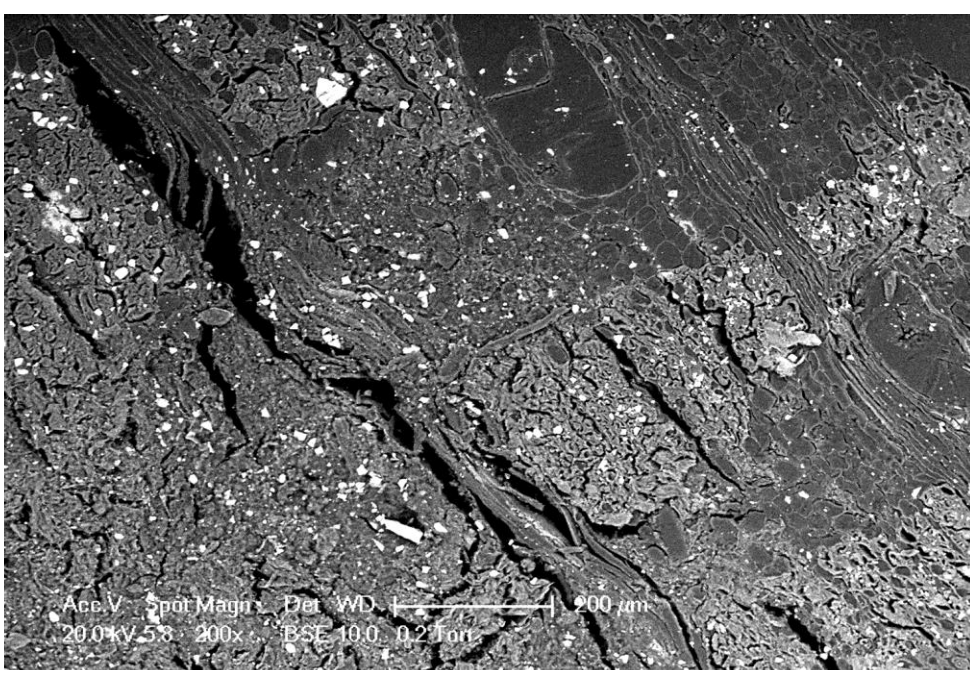

Figure 4: SEM micrographs showing the characteristic cell shape of sound vessels and fibers, as well the deformation of the wood as result of natural aging.

Figure 5: Linen fibers through the optical microscope, showing hardening of the linen fibers.

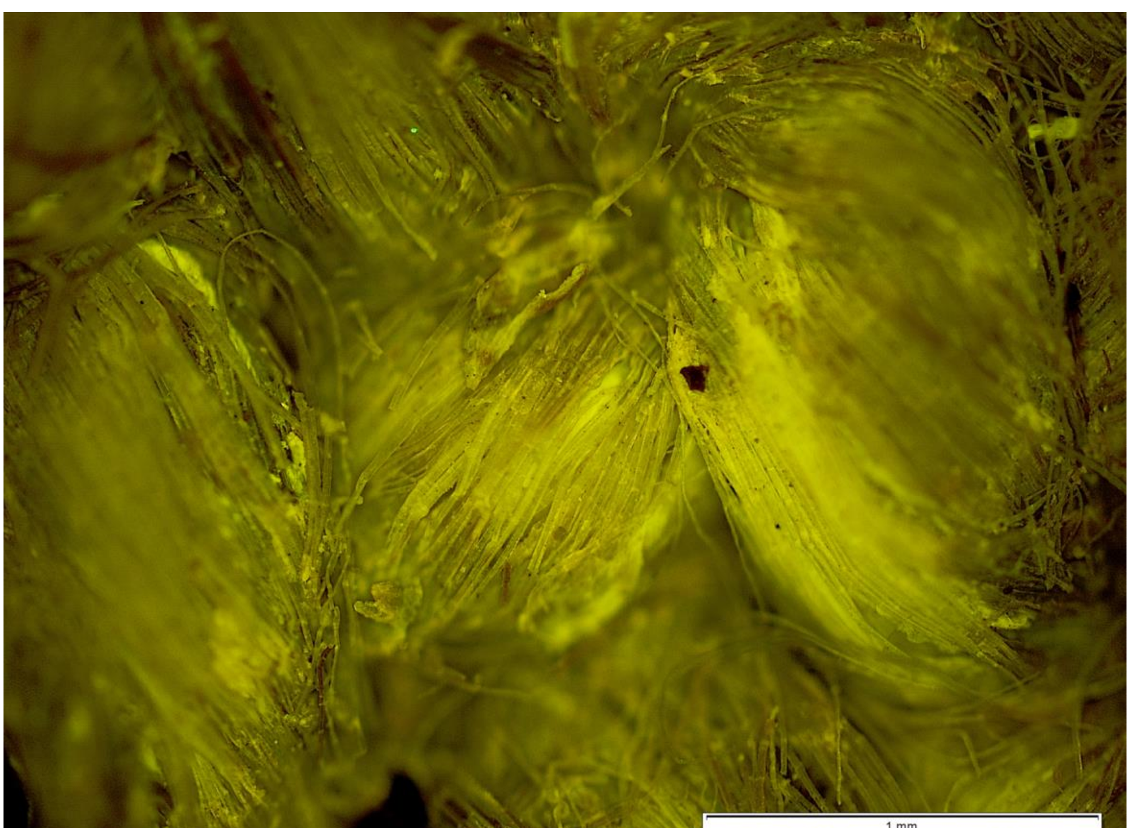

Figure 6: Fluorescent microscope image showing that animal glue has penetrated between brittle fibers.

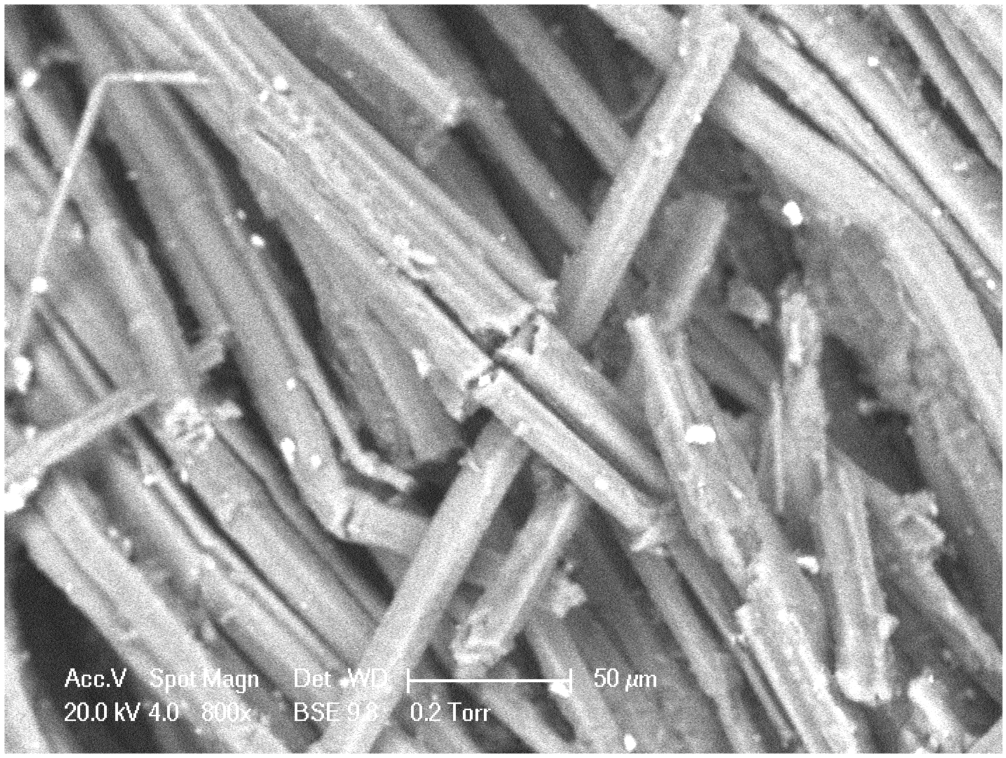

Figure 7: Cracks, twisting, and warping in fibers.

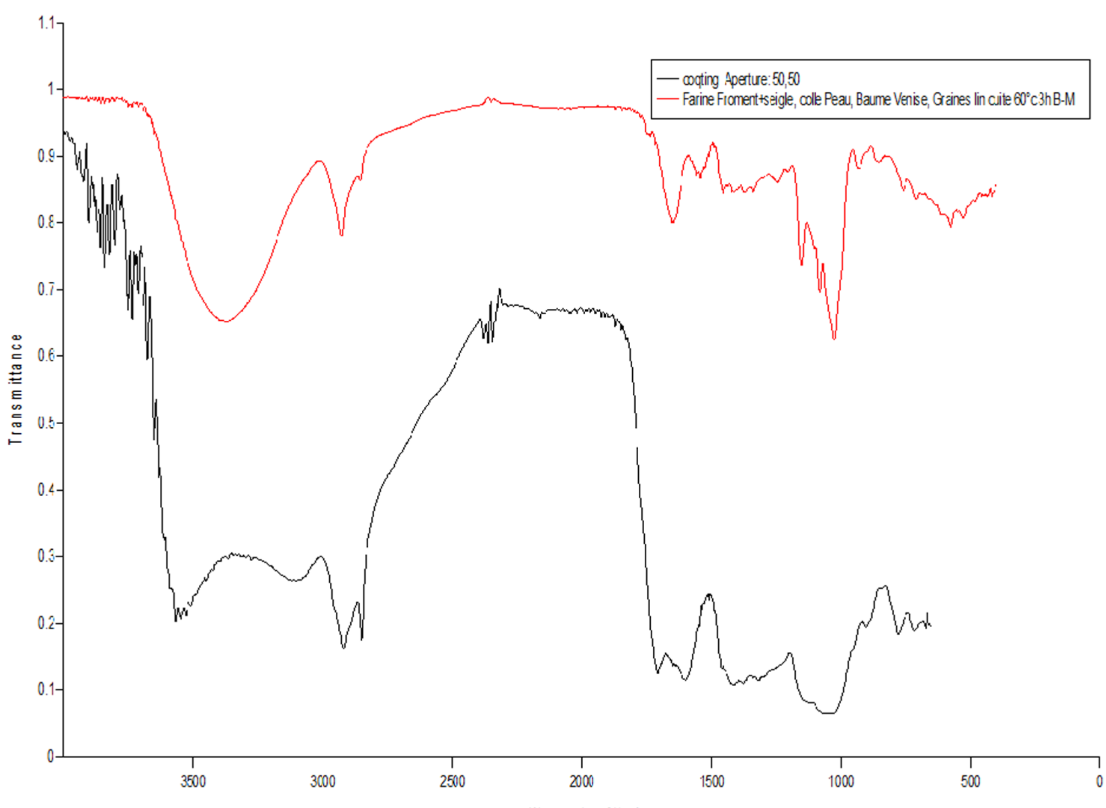

Figure 8: FTIR spectra of (upper) residue coating the textile of the consecration panel and (lower) the standard of animal glue.

Figure 9: Cross section of the ground layer and pigments.

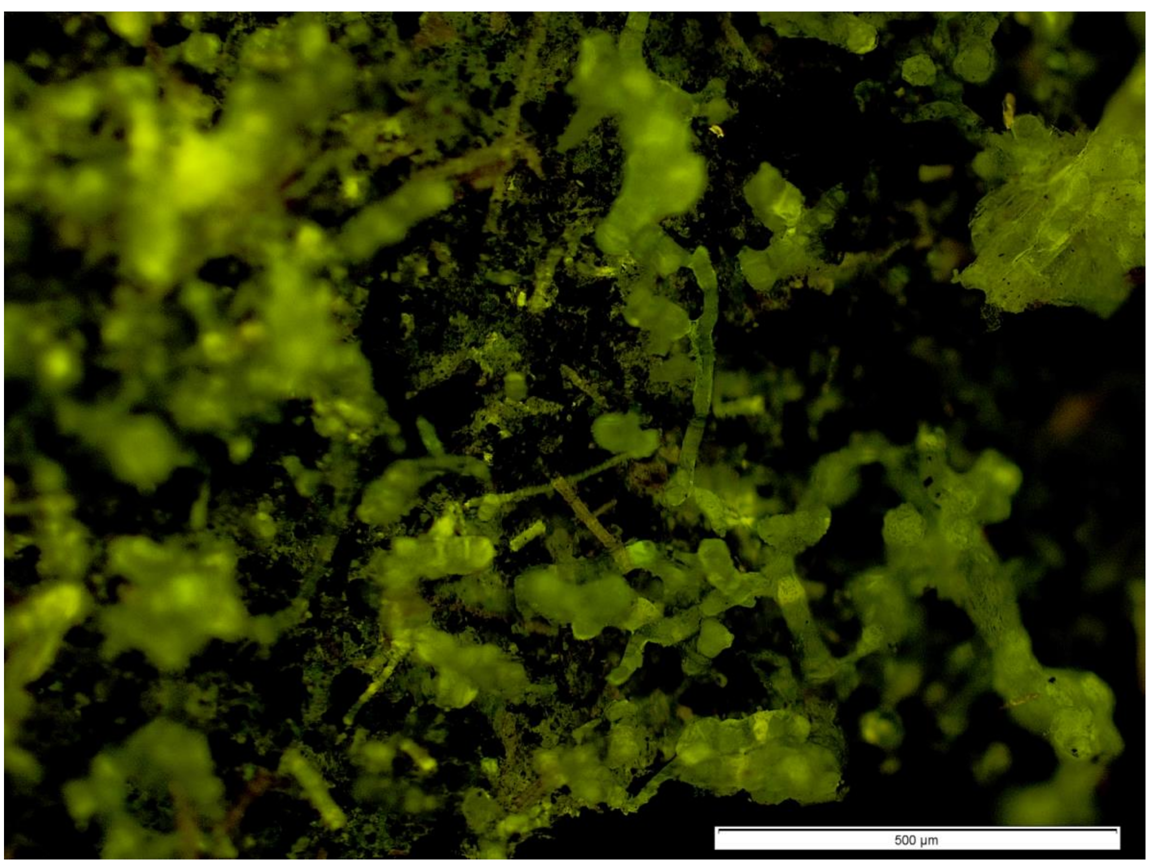

Figure 10: Previous conservation by Klucel G as revealed by fluorescence.

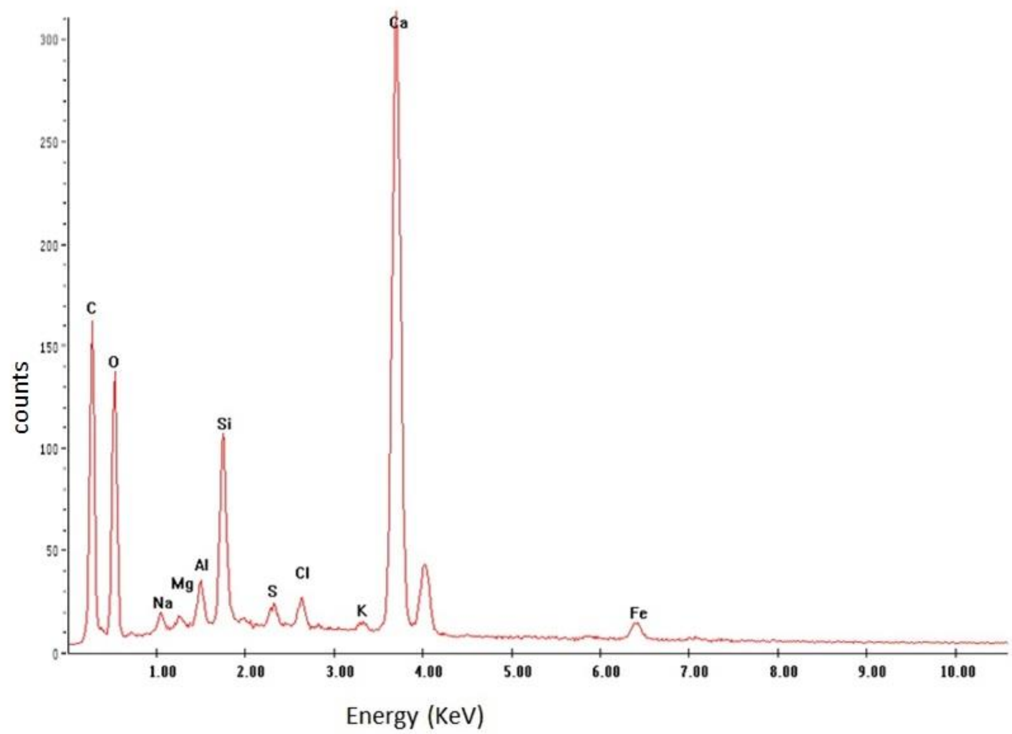

Figure 11: Pattern showing the analysis of the ground layer and the high proportion of calcium.

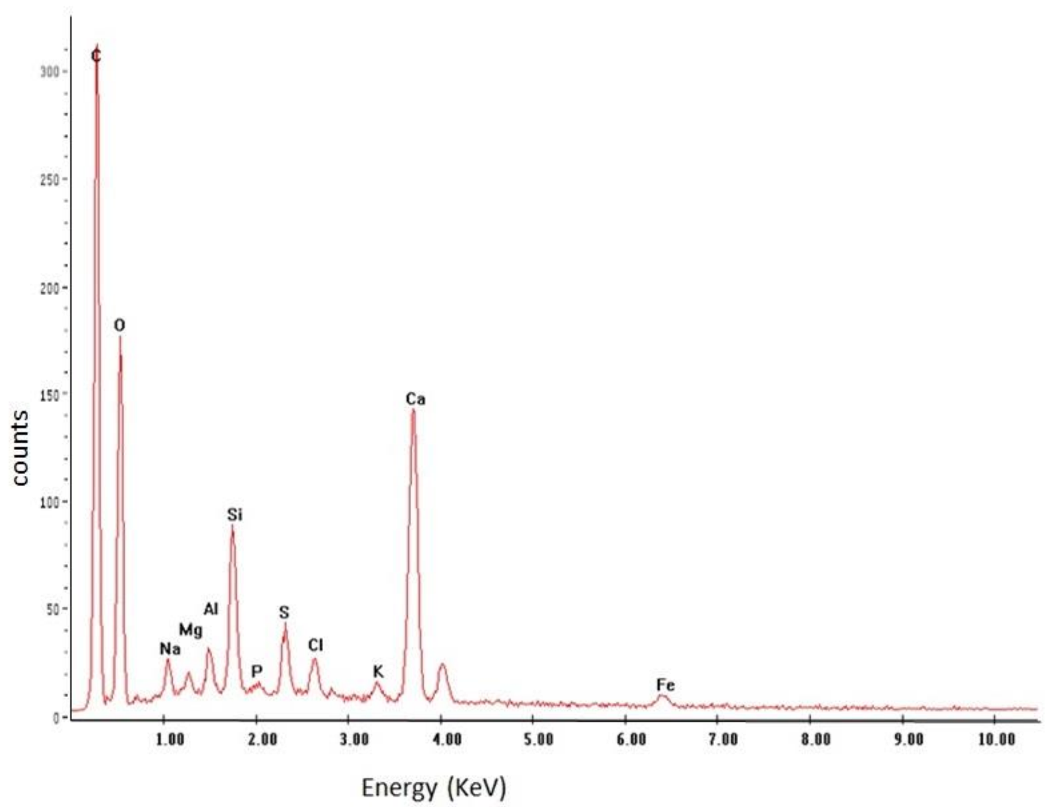

Figure 12: SEM/EDS pattern of black pigment.

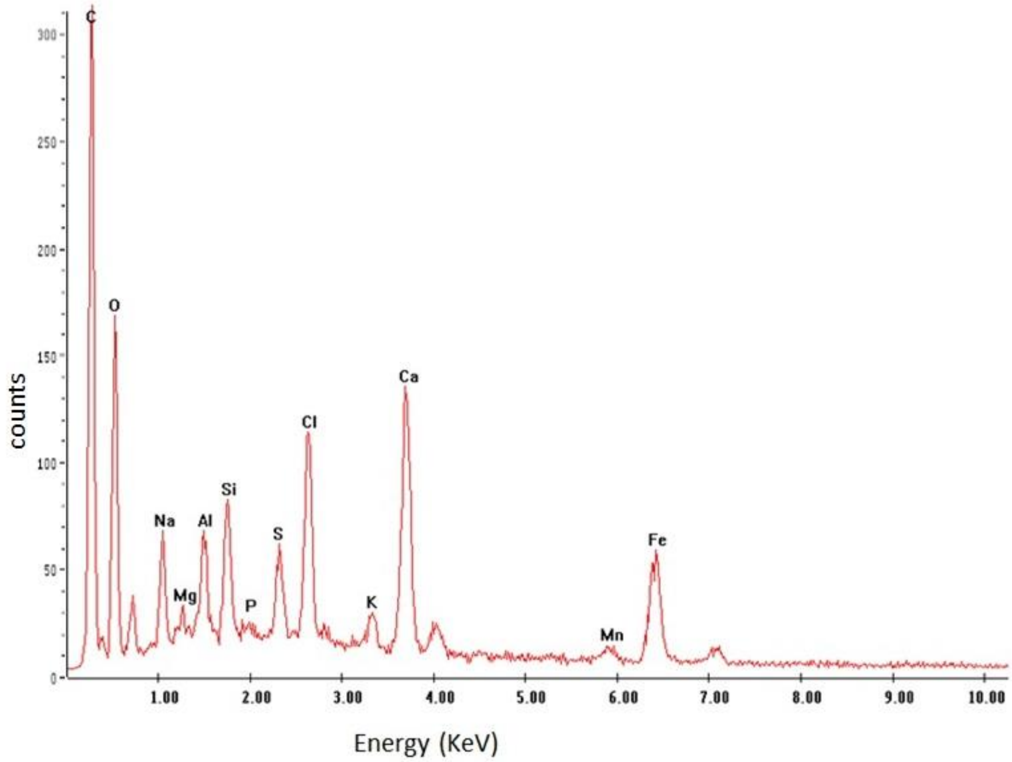

Figure 13: SEM/EDS pattern of red pigment.

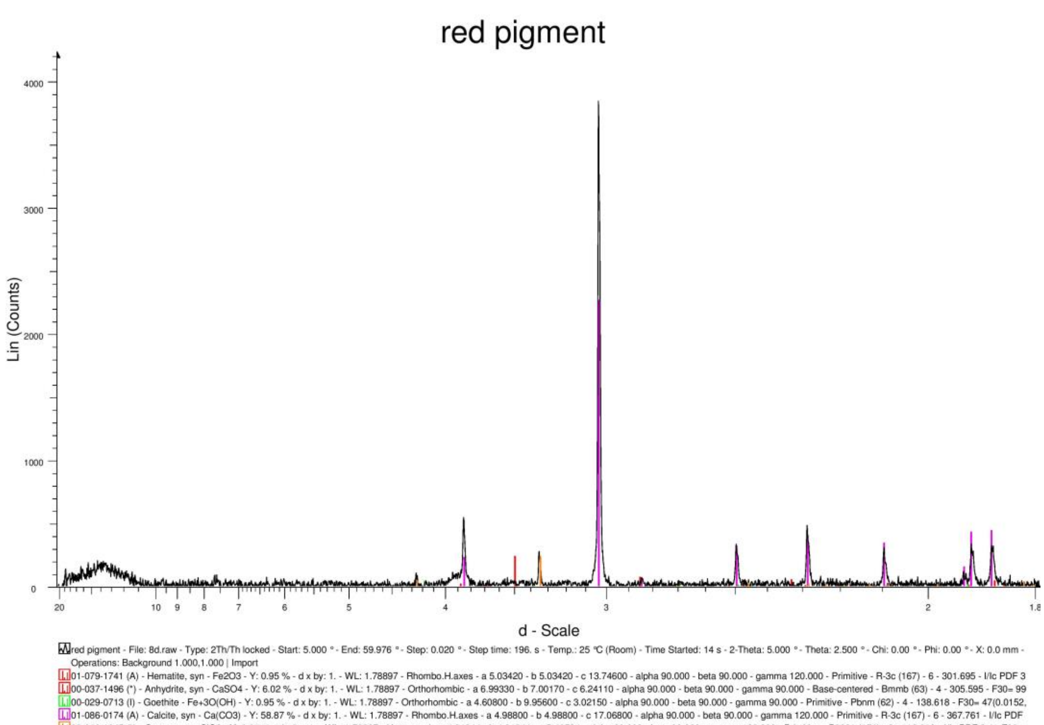

Figure 14: XRD pattern of red pigment.

NOTES

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