A non-destructive technological study of three fresco fragments from Iklaina, Pylos, Greece

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ABSTRACT

The objective of the present study is to conduct a non-destructive characterisation analysis of three fresco fragments from Iklaina, southern Peloponnese, Greece, in order to identify their manufacture techniques. Furthermore, this study aims at using the results of the scientific and analytical analyses to produce accurate replicas of these fragments (use of similar composition mortars, pigments, and manufacture techniques), which can be used for the restoration program of the site. These replicas will be exhibited alongside the originals in the new Pylos Archaeological Museum.

Due to the high archaeological value of the objects, the analytical approach we followed was completely non-destructive and was based on the parallel use of optical microscopy, p-XRF and SEM/EDS. The analyses suggest the use of Egyptian blue pigment for the backgrounds. The wavy black coil of hair was painted with an organic black pigment. The use of inorganic pigments has been revealed on the upper layers on the basis of the identification of minerals and rocks. The substrate of the wall paintings is made of aluminosilicate lime mortars. The results corroborate the suggestions of previous studies for the pigments comprising the Mycenaean artistic palette and provides further insight on the artistic and technological choices made by Mycenaean artists.

KEYWORDS

Fresco; Iklaina; Bronze Age; Mycenaean art; artist’s pallet; plaster

Introduction

Within the framework of an ongoing project related to the exhibits of the new Pylos Archaeological Museum a research collaboration between the Conservation Department of the Ephorate of Antiquities of Messenia and the Laboratory of Archaeometry of the University of the Peloponnese was established, aiming at conducting an analytical and technological examination of mortar fragments, with the purpose of preserving and enhancing Greece’s Cultural Heritage. The fragments analyzed are 3 unique Mycenaean fresco fragments from Iklaina, Greece. The analytical procedure applied was totally non-destructive as sampling was restricted in view of the value, the uniqueness, the size and the extremely fragile nature of the material (European Commission, 2003). As Adriaens (2005, p. 1504) emphatically states: ‘there is one essential difference between the analysis of ancient and modern materials—an art object or ancient artefact cannot be replaced, and the consumption or damaging of even a small part of it for analytical purposes must be undertaken only where vital data cannot otherwise be obtained’. The final aim of the project was the creation of accurate replicas which are now exhibited alongside the originals in the Pylos Archaeological Museum, using similar composition mortars, pigments, and manufacture techniques. The study and publication of all the Iklaina frescoes has been undertaken by Dr. Harikleia Brecoulaki.

Presentation of the items

Iklaina was a district capital of the Mycenaean state of Pylos, southern Peloponnese, which flourished between approximately 1600 and 1200 BC (Cosmopoulos 2016; for extensive bibliography see www.iklaina.org). Ongoing excavations conducted under the auspices of the Archaeological Society at Athens under the directorship of Michael Cosmopoulos have unearthed a group of monumental buildings surrounded by residential and industrial quarters (Figure 1). The dominant feature of the site is a large two- or three-story high building complex named the “Cyclopean Terrace Complex” because its north wing is constructed on a massive Cyclopean Terrace (Cosmopoulos 2012).

In the rooms to the south of the Terrace were found over 1,000 fragments of frescoes, three of which were selected for analysis in the present study: (1) Sample SF134 depicts a female face in profile (max. dim.
(2) Sample 2273 depicts a part of a ship (max. dim. 0.23×0.138 m); and (3) Sample SF211 depicts a female hand (max. dim. 0.067×0.045 m) (Figure 2). Initially thought to date to the Early Mycenaean period (Cosmopoulos 2015), these fragments are now dated to the LH IIIA-IIIA2 periods.

Methodology

The analysis of the wall painting fragments was carried out following a totally non-destructive, multi-technique approach, aiming at the examination of the preservation state of the samples and the identification of the materials and techniques used for their manufacture. The fragments were initially studied by optical microscopy, using a fibre optics system (FOM/iScope, Moritex). The microscopic observation enabled the documentation of the preservation state of the samples. Moreover, it provided important information on the presence of successive colour layers, thus facilitating the selection of the most suitable and representative areas for chemical analysis. Finally, taking into consideration that restoration works had been undertaken in the past, it was necessary to exclude the possibility of analyzing treated areas, which would affect the acquired data.

An initial chemical examination of the samples was conducted using non-destructive X-Ray Fluorescence (p-XRF), a technique extensively used for the non-destructive analysis of archaeological artifacts (Liritzis...
The analysis of both the mortars and the colour layer was carried out with a portable Bruker Tracer III SD set up, with a beam diameter of 3 mm. In order to optimise the analytical range, two settings were used for the mortars: (1) an unfiltered low-energy excitation mode (high voltage set at 15 kV and current of 24 μA, analyses carried out under vacuum) was used for the analysis of major and minor elements with an atomic number, Z, between 11 and 29; and (2) an Al/Ti filtered (0.012 inches Al plus 0.001 inches Ti) high-energy excitation mode (high voltage set at 40 kV and current of 12 μA) was used for the analysis of minor and trace elements with an atomic number Z>29. The collection time of each measurement was 120 sec and the data quantification was made using S1PXRF software.

For the identification of the pigments used for each colour, multiple areas of the colour layer of each samples were analysed; for the purpose of the present study, only the second type of settings was used, in order to focus primarily in the determination of the presence of metals commonly used as pigments (such as Fe, Mn, Cu, Co, Pb etc). The collection time of each measurement was 60 sec. It must be highlighted that the acquired data were only used qualitatively, due to the multi-layered structure of the samples. Care was taken to select areas where the paint layer was thick and the surface was clear, based on the results of the FOM examination.

Due to the relatively large beam size of the XRF tracer, it was not possible to analyse thin colour lines. In order to examine the chemical composition of selected areas in more detail, the colour layer of the fresco fragments was further analysed by Scanning Electron Microscopy coupled with an Energy Dispersive Spectrometer (SEM/EDS), using a SEM type JEOL JSM-6510LV coupled with an Oxford Instruments EDS. The analytical data were obtained by INKA software. The bulk analyses were conducted at low vacuum, 20 kV accelerating voltage and with a count time of 120 sec.

SEM/EDS can be a very useful technique for the analysis of multi-layered archaeological samples (Mastrotheodoros, et al., 2013). However, given that sampling was not possible, the fresco fragments were examined intact and without any prior treatment; the analyses were therefore again affected by the substrate composition. Additionally, sample 2273 was too large to be placed inside the SEM chamber, and was therefore not analysed by SEM/EDS.

Microscopic examination

In Figure 3, four LED-OM photos are presented, demonstrating representative paint layers of the artist’s pigments palette.

Chemical analysis

As has already been highlighted before, the examination of the intact fresco fragments by p-XRF and SEM/EDS allowed the qualitative characterization of the paint and the plaster. The horizontal orientation of the analyses introduced background information on the colored areas from the mortar substrates, which had to be taken into consideration when determining the pigments present. In addition, the fragments had already been conserved and restored so subsequent maintenance materials or other deposits must be considered while assessing the results.

The results show that the artist’s palette was simple and almost identical for all the examined fragments. The various colours were produced by the addition of common mineral pigments, carbon and a systematically produced synthetic blue. Table 1 shows the major and minor elements identified by p-XRF.

Blue Pigments

Blue pigments are of specific interest given that blue areas serve as the main background of the depicted scenes. The analyses of almost all selected blue areas resulted in similar spectra with strong copper bands. The combined presence of Cu, Ca and Si, in addition to the macroscopic and microscopic observation of blue areas, suggest the use of Egyptian Blue (CaCuSi4O10) (Figure 4).

Egyptian blue is the most important pigment in Bronze Age and it is a synthetic compound (Vlachopoulos & Sotiropoulou, 2013). In nature, the mineral cuprorivaite has exactly the same crystalline structure but it is very rare (Hughes, et al., 1997); therefore it was not used in big painted areas (Perdikatis 1993; Brecoulaki, et al., 2008). Synthetic Egyptian blue
invariably contains some calcite and quartz as impurities (Gettens & Stout, 1966). The pigment has been identified in Minoan and Mycenaean monumental paintings in Crete, Keros, Mycenae, Tiryns and Pylos, in Classical and Hellenistic paintings in Macedonia and other provinces of the empire such as Egypt, Israel and Cyprus (Kakoulli, 2009; Filippakis, et al., 1976).

In one examined point of sample's SF134 blue area, SEM/EDS analysis has revealed the additional presence of Na, Mg, Al, Si and Fe (Figure 5). These elements could

![Figure 4](image)

**Table 1.** Qualitative identification of major and minor elements by p-XRF present in each distinct colour area of the three wall painting fragments

| Sample | Area | Colour | Elements identified | Element responsible for colour |
|--------|------|--------|---------------------|-------------------------------|
| SF211  | 1    | White, red & blue | Ca, Mn, Fe, Cu | Cu (Fe?) |
|        | 2    | White, red & blue | Ca, Mn, Fe, Cu | Cu (Fe?) |
|        | 3    | Blue           | Ca, Mn, Fe, Cu | Cu |
|        | 4    | White          | Si, Ca, Mn, Fe, Cu | Ca |
|        | 5    | Red            | Ca, Mn, Fe, Cu | Fe |
| SF134  | 1    | Blue           | Ca, Mn, Fe, Cu | Cu |
|        | 2    | White          | Ca, Fe           | Ca |
|        | 3    | White-grey     | Ca, Fe           | Ca |
|        | 4    | Black          | Si, K, Ca, Ti, Mn, Fe | Organic |
|        | 5    | Black-brown    | Ca, Fe           | Organic |
|        | 6    | Red            | Ca, Mn, Fe, Cu, Zn | Fe |
| 2273   | 1    | Blue           | Si, K, Ca, Ti, Mn, Fe, Cu | Cu |
|        | 2    | Blue           | Si, K, Ca, Ti, Mn, Fe, Cu | Cu |
|        | 3    | Red            | Ca, Fe, Cu       | Fe |
|        | 4    | Red (oarsman)  | Ca, Fe, Cu       | Fe |
|        | 5    | White          | Cl, Ca, Fe       | Ca |
|        | 6    | Red yellow     | Cl, Ca, Mn, Fe, Cu, Zn | Fe |
|        | 7    | Yellow         | Cl, Ca, Fe, Cu, Zn | Fe |
|        | 8    | Red yellow     | Si, Cl, K, Ca, Ti, Fe, Cu | Fe, Si |
|        | 9    | White          | Cl, Ca, Fe       | Ca |
|        | 10   | Red            | Cl, Ca, Fe, Cu   | Fe |
|        | 11   | Blue           | Cl, Ca, Fe, Cu   | Cu |
|        | 12   | Blue           | Cl, Ca, Fe, Cu   | Cu |

![Figure 5](image)
suggest the use of glaucophane, a blue-purple mineral belonging to the amphibole group with the composition \( \text{Na}_2(\text{Mg}_3\text{Al}_2)\text{Si}_8\text{O}_{22}(\text{OH})_2 \). Riederer (1997) has noted the rare independent use of glaucophane as a pigment in the Greek islands. However, glaucophane, along with riebeckite, have also been found in association with Egyptian blue and even ferroglaucophane \((\text{Na}_2(\text{Mg},\text{Fe})_3\text{Al}_2\text{Si}_8\text{O}_{22}(\text{OH})_2)\) because of Fe as a mixture or a different layer (Katagas, 1974). The colour depends on the relative concentration of iron/magnesium. In fact, only magnesioriebeckite is blue, while pure Fe-riebeckite is black and glaucophane has a dark grey-bluish colour that becomes grey when ground (Perdikatsis, et al., 2000). At Akrotiri, Thera, Egyptian blue appears to have been used in most of the iconographic programme, in different ratios, mixed with riebeckite or as a second thin layer over an underlay of riebeckite (Vlachopoulos, et al., 2013).

**Red, Yellow and Orange Pigments**

In all red areas (Figures 6, 7) the pigments consist of a mixture of iron oxides, in the form of hematite \( \text{Fe}_2\text{O}_3 \). In Pharaonic Egypt, in the Old and Middle Kingdoms, this pigment was used specifically for the depiction of male human bodies (Goresy, 2000). The same way of painting the male bodies was also observed in Pylos (Lang, 1969; Brecoulaki, 2012).

In the yellow areas the presence of Fe and Si, suggests the use of yellow ochre, such as goethite \( \text{FeO(OH)} \) and limonite \( \text{FeO(OH)}_2\text{H}_2\text{O} \), in an aluminosilicate substrate \((\text{Al}_2\text{O}_3.2\text{SiO}_2.\text{H}_2\text{O})\).

A slightly darker orange colour, e.g. the oars of fragment No 2273 (Figure 6), is produced by the simultaneous presence of Mn and Fe, suggesting the use of a \( \text{Fe}_2\text{O}_3 + \text{MnO} \) oxide.

The red, yellow and orange colours that are known for Bronze Age Greece in general are natural earth

![Figure 6](image6.png)

**Figure 6.** Image of red, yellow and orange areas from sample 2273 (left) and representative XRF spectra of a red area (right, top), orange area (left, bottom) and yellow area (right, bottom).

![Figure 7](image7.png)

**Figure 7.** FOM image of red lines from sample SF211 (left) and representative SEM/EDS spectrum of the red area.
ochre based: hematite and/or iron hydroxides, either crystalline (goethite) or amorphous (limonite) as well as calcite, quartz and aluminosilicates (kaolinite, illite and other clay minerals) (Cameron, 1968; Profi et al., 1977; Cameron, et al., 1977; Brysbaert & Perdikatsis, 2008; Sotiropoulou, et al., 2012; Filippakis, 1983).

**Black Pigments**

The black pigments on fragment SF134 consist of carbon (Figure 8). No manganese compound (pyrolusite) or calcium phosphate (bone black) was identified in black areas. Therefore, the pigment belongs to ‘carbon based black group: Chars sub-group’ following the convention proposed by Winter (1983) and Eastaugh et al. (2004). Carbon black is made by partial burning or carbonizing of wood and other organic materials. Carbon makes a very stable pigment with excellent hiding power. It is unaffected by light and air and by hot concentrated acids and alkalis (Gettens & Stout, 1966).

The use of carbon black on Bronze Age wall paintings is well documented. In forty two samples from the Theran wall paintings of Xeste 3, analyzed by means of XRD, optical microscopy and SEM, no black pigments were found; therefore Perdikatsis et al. (2000) suggested the use of carbon black. The same suggestion was made by Profi et al. (1974) after analyzing Greek Bronze Age pigments from Mycenae by means of XRF, XRD and mineralogical microscopic examination.

**White Pigments**

The white areas consist mainly of calcite (CaCO₃) (Figure 9). It is interesting to note that on fragment

Figure 8. FOM image of a black and white-grey area (left) and representative SEM/EDS spectrum of a black area from sample SF134, suggesting the presence of carbon black.

Figure 9. FOM images of white and white grey areas (right, top) from sample SF134 and representative SEM/EDS spectra of a white-grey area (left, bottom) and white area (right, bottom), showing the presence of calcite.
SF134, on the whiter area of the face there is less Ca than on the white-gray areas. This difference is attributed to the fact that the whiter areas are slightly more damaged and the upper layer of the paint, consisted of pure calcite, has been flaked off (Figure 9). The identification of Al, Si (Figure 10) indicates the use of Kaolinite $\text{Al}_6\text{Si}_2\text{O}_{10}$(OH)$_8$.

Chalk also known as whiting (composed mainly of calcium carbonate), and gypsum (composed of calcium sulfate) were the most commonly used white pigments of antiquity (Gettens & Fitzhugh, 1993). Kaolin was used only in limited geographic regions where this primary clay was abundant or easily available (Goffer, 2007). Pigment samples with white color originated from Bronze Age Knossos consist mainly of calcite and sometimes of calcite and kaolinite (Profi, et al., 1976; Profi, et al., 1977; Perdikatis, 1993). Therefore, it appears that in Iklaina they followed a similar tradition in the selection of white pigments.

Plaster

Previous analyses on Bronze Age painted plaster from the Greek mainland analyzed by means of XRD analysis, gave a very homogenous picture. All plasters gave calcite (Profi, et al., 1974) as the main mineral with a small amount of quartz impurities. The impurities, organic and inorganic (quartzitic sand, crushed ceramics or even straw) are used as fillers (Brysbaert & Perdikatsis, 2008).

The samples analyzed in the present work gave an overall similar image. The substrate of the wall paintings is made of lime plaster (Figures 11, 12). The plaster of the three fragments consists of a single thick layer of very fine and homogeneous texture. The outer surface of the fragments on which the painted composition was drawn was carefully smoothed before paints were applied, assuring both a uniformly even ground and the stability of the paint. The mortar consists of

Figure 10. FOM image of a white area (right, top) from sample SF134 and representative SEM/EDS spectrum of the white area (right, bottom), indicating the use of kaolinite.

Figure 11. SEM/EDS spectrum of the plaster of sample SF211.
aluminosilicate lime plaster. In contrast, in the fragment 2273 the mortar shows a high concentration of chlorine (Cl). Chlorine is detected at lower concentrations in virtually all measurements of this sample. It is difficult to determine whether the presence of chlorine is due to surface deposition on the sample, or is a component of the mortar. However, due to the fact that the increased chlorine concentration always coincides with an increase in copper concentration, it could be suggested that the presence of chlorine is associated with copper corrosion mechanisms.

**Conclusions**

The present work focused on the examination of three Mycenaean wall painting fragments, recovered at the Cyclopean Terrace in Iklaina, Greece. The high archaeological value of the fragments rendered sampling impossible, thus complicating the analytical process. Despite these difficulties, the combination of three non-destructive examination techniques, FOM, p-XRF and SEM/EDS, allowed the determination of the pigments of almost the entire palette of the painters and the materials of the substrate of these fragments.

Blue areas were primarily coloured by Egyptian Blue. However, in one singular area, glaucophane was also identified, suggesting the parallel use of multiple blue pigments. In areas with red, yellow and orange hues the identified pigments consist of a mixture of iron oxides: hematite for red areas, yellow ochre for yellow areas and a combination of iron and manganese for orange areas. It is interesting to note that hematite was only used for the depiction of human bodies, an observation that has previously been made for wall paintings in Egypt and Crete. Black pigments were identified as carbon black. In white areas a combination of calcite and kaolinite was identified, which is again similar to known practices in Minoan Crete.

Moreover, the substrate of the wall paintings was determined as aluminosilicate lime plaster. The plaster of all three fragments consists of a single thick layer of very fine and homogeneous texture. However, in one of the samples (fragment 2273) significant concentrations of chlorine were identified. At this point it is not possible to determine whether the presence of chlorine is due to surface deposition on the sample or was a deliberately added component of the mortar.

Thus, the possibility of accurately extracting information using analytical techniques can be exploited when there are significant fragments, from which it is impossible to obtain a microsample. In this case, the results of the analyses will further make possible the preparation of replicas that will be exhibited alongside the originals in the Pylos Archaeological Museum using similar composition mortars, pigments, and manufacture techniques. There is, of course, the inability to adequately study the stratigraphy of a specimen, especially if our scientific interest is focused on points or areas not located on the periphery of the fragment, or the condition of the material around the fragment is not appropriate for examination.

Further research of parallels from various individual sites across the Mediterranean, including comparison with the aid of data base is required to reveal the creative technique followed by the artists to these specific wall paintings for a more thorough understanding of the artistic creation in the Bronze Age.

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