Polished Decorative Fields in Thracian Fresco Tombs from the Hellenistic period - Archaeometrical Research

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\section*{ABSTRACT}
In order to gather information on the technological characteristics of the monochrome decorative wall plasters in some Thracian fresco tombs (4\textsuperscript{th} - 3\textsuperscript{rd} centuries BC) found in South Bulgaria, several types of analyses have been carried out.

Samples of red, yellow and white large wall-painted fields in different monuments, have been investigated using a combination of Attenuated Total Reflectance Fourier Transform Infrared Spectroscopy (ATR-FTIR) and Differential Scanning Calorimetry (DSC) to identify the organic components of the paint layer. The nature of the pigments has been determined both by X-ray diffraction (XRD) and ATR-FTIR.

The obtained results point towards the recognition of final polishing processes performed on still wet plaster. This technique of creating polished wall painted surfaces in combination with organic coatings (known as stucco lustro) is traditionally associated with the Pompean frescoes and the work of roman painters. Based on the technical examination, it can be assumed that in pre-roman time, the Hellenistic paint-craftsmen in Thrace inland were also familiar to this technique.

\section*{KEYWORDS}
Wall paintings; Thracian tombs; polished plasters; organic coatings; beeswax

\section*{1. Introduction}
\subsection*{1.1. Thracian funeral tombs}
More than two thousand monumental tombs from the Hellenistic period (from the mid 4\textsuperscript{th} – 3\textsuperscript{rd} century BC) have been explored in Bulgaria, the modern state covering the main part of the historical and geographical region of Ancient Thrace (Theodossiev, 2011). All of the tombs are constructed under tumuli and often have a number of different chambers, related to specific Thracian funeral rituals and practices. A large diversity of ground plans and covering constructions is observed – the most popular being corbel vaults and domes, as well as wedged barrel vaults (Stoyanova, 2011: 335; Valeva, 1993). The Thracian graves of nobility usually contain gold, silver vessels and jewellery, various weapons, imported Greek pottery, bronze tableware, and a number of other funerary gifts.

During the second half of the 4\textsuperscript{th} century BC the mural painting practices in Thrace, as was common for the whole Mediterranean region (Kakoulli, 2002), flourished as never before. Nevertheless, this type of decoration, reserved for domestic or funerary monuments of the privileged individuals, was expensive and difficult to achieve. Fresco tombs in Bulgaria feature only about 10 per cent of all the discovered monuments from the period. At the same time, they are to be considered one of the main material sources to help understand the Hellenistic painting methods in Ancient Thrace.

\subsection*{1.2. Painted monuments – object of the study}
The object of the current study are five Thracian fresco monuments\textsuperscript{1} - four tombs and one residential building, considered to be the palace of a Thracian ruler. They have been dated back to the 4\textsuperscript{th} – 3\textsuperscript{rd} century BC. Located in South Bulgaria, they are known as Kazanlak tomb (Vasileva, 2005; Parvin, 2015), Alexandrovo tomb (Kitov, 2005a), Dolno Lukovo tomb (Nehrizov, 2006), Helvecia tomb (Kitov, 1999)
and Sevtopolis Palace (Dimitrov & Chichikova, 1978: 13). (Figure 1)

All of these monuments display Masonry-style decoration - their interior stone or brick walls are covered by plasters, thus, imitating the construction of a regular massive masonry. Marked by incisions carved in fresh mortar, the structure of the decor is often intensified by embossment and/or brightened by colour. Hence, the analyzed Thracian monumental interiors consist of multiple horizontal divisions, typical features of the Masonry style (Bruno, 1969; Brecoulaki, 2016: 681) and are rendered in different colours: a lower segment, including its components (pseudo-plinth, orthostates, string course) and a large monochromatic main area above. Some of the compositions end with a painted figurative frieze with funeral or mythological scenes on the upper part of the wall (Chichikova, 1999; Valeva, 1999).

Polychromy in Ancient Thrace still remains less studied and only a few cases are publicly available. The present scientific research was provoked by the high visual qualities of the polished monochromatic areas of the painted monuments described above. In all five cases, fresco technique and lime-based preparatory layers were applied. In order to gather information on the technological characteristics of these mirror-like surfaces, several types of analyses have been carried out. (Figure 2 and 3)

2. Experimental analyses

2.1. Sampling

Sampling points, labeled respectively as KZK, AL, DL, H, and SEV, are indicated in Table 1.

To determine the entire pictorial stratigraphy, the nature and distribution of the components, selected parts of the samples, including the lime-based preparatory layers, were embedded in polyester resin and then polished. The prepared cross-sections were inspected under a binocular microscope (Leica EZ4D). After a carbon-coating treatment they were examined and analyzed using SEM – EDX.

In order to study the inorganic mineral pigments, both a Fourier transform infrared spectroscopy (FTIR) and an X-ray diffraction (XRD) were carried out on the pictorial surfaces. For this purpose, small
amounts of the red/y/w pigments were collected by scratching the surfaces with a scalpel blade. In all the cases only one pigment layer was used.

The organic components of the paint layers were identified using both ATR-FTIR and Differential scanning calorimetry (DSC).

2.2. Laboratory examination: instruments and methods – analytical techniques

FTIR spectra were recorded on a Bruker Tensor 27 FT spectrometer at a resolution of 2 cm\(^{-1}\) and 64 scans, referencing to the air spectrum. The absorption FTIR spectra in the middle IR region (400–4000 cm\(^{-1}\)) were measured in solid state as KBr pellets (1 mg from painted layer in 300 mg KBr).

Bruker diffractometer D2 Phaser was used for the analysis of powdered and homogenized samples. The ATR-FTIR spectra of the samples were recorder in the region 600–4000 cm\(^{-1}\). The samples were directly deposited on top of the crystal and pressed in order to ensure a good contact with the ATR crystal. The experimental conditions were as follows: Ni Cu radiations at 10 mA and 30 kV. Spectra were collected from 5 to 60 2\(\theta\). The crystalline phases were identified by a comparison with JCPDS cards.

Differential scanning calorimetry (DSC) analysis was carried out using STA PT1600 TG-DTA/DSC (STA Simultaneous Thermal Analysis) apparatus (LINSEIS Messgeräte GmbH, Germany) under the following conditions: temperature interval: from room temperature to 200°C, temperature heating rate of 10° C/min and static air atmosphere. Individual samples (8.5 mg) were placed in platinum pans.

Scanning electron microscopy (SEM-EDX) analysis of the samples was performed with TESCAN SEM/FIB LYRA I XMU instrument with BRUKER Quantax 200 EDS detector in regimes of secondary electron image (SEI) and Backscattered Electron contrast (BEC).

**Figure 2.** Thracian wall painted monuments (4\(^{th}\)-3\(^{rd}\) century BC) - red samples. The arrows show sampling areas. 2.a. Graphical representation of the decorative scheme in the central burial chamber of Kazanlak tomb; photos illustrating the figural frieze in the domed chamber and the antechamber frescoes. 2.b. Graphical representation of the decorative scheme in the central burial chamber of Alexandrovo tomb; photos illustrating the figural frieze in the domed chamber (after Kitov, 2005b). 2.c. Sevtopolis Palace fragmented wall paintings.
3. Results

3.1. Colour palette

The analyses suggest that it was basic mineral pigments (red and yellow ochres) that were used to obtain pure colours of the paint layers. (Table 2)

Ochres are considered the major chromatic components of the ancient four-colour palette, largely used due to their high colouring capacity and stability (Perdikatsis & Brecoulaki, 2008: 559; Bikiaris et al., 1999: 3). They are natural mineral mixtures, which contain varying amounts of iron oxides and aluminosilicate as kaolinite or illite, quartz and calcium compounds as calcite, anhydrite, gypse or dolomite (Elias et al., 2006: 70).

**Red pigment**

The three red samples KZK, AL, SEV. have similar composition. XRD study of the red paint layers identifies hematite as the colouring agent. The obtained FTIR results demonstrate a good consistancy with the data collected from the XRD analysis. The representative infrared spectrum of the KZK sample illustrates the characteristic peaks of the red ochre (Figure 4). Peaks at 547 and 474 cm\(^{-1}\) are indicative of the presence of hematite mineral (after a comparison between the spectrum under examination and a referent one) (Pallecchi et al. 2009: 2639; Brecoulaki et al. 2006). Clay and alumino-silicates are also detected in high percentage in the sample. Kaolinite, which gives a pale white colour, having lumincence of red ochre is also present, based on the peaks at 3694, 3620, 1097, 1036, 1012 cm\(^{-1}\).

Elemental EDX analysis, performed on an area of the paint layer, detected predominant amount of Fe, together with Mg, Al, Si, K. The results are similar to

| Sample | Colour | Identified pigment | Analytical techniques used |
|--------|--------|-------------------|---------------------------|
| KZK.   | red    | Red ochre         | XRD, FTIR, SEM-EDX        |
| AL     | red    | Red ochre         | FTIR, SEM-EDX             |
| DL     | yellow | Yellow ochre      | XRD, FTIR, SEM-EDX        |
| H.     | white  | Calcite           | FTIR, SEM-EDX             |
| SEV.   | red    | Red ochre         | XRD, FTIR, SEM-EDX        |

Table 2. List of analyzed samples, identified pigments and determinative analytic techniques used.
other red samples – with small variations of the quantities of Mg, Al and Si. Ca was detected in all of the red layers.

Red ochre was detected by FTIR analysis in AL. samples (Todorov, 2011: 329; Glavcheva et al., 2016: 623, Figure 3).

Yellow pigment
The yellow colour of DL. sample is due to the yellow ochre, consisting mostly of goethite/limonite. The infrared spectrum of yellow ochre exhibits many similarities to the spectra of both red ochre and kaolinite – i.e. two distinct peaks at 547 and 474 cm⁻¹, characteristic of hematite (Figure 5). In contrast to KZK. spectrum, DL. samples have peaks of goethite/limonite appearing at 3120, 911 and 798 cm⁻¹.

Ochres often contain a natural mixture of two iron oxides – goethite (or limonite) and hematite, with their proportion in the pigment being one of the factors to determine its colour shade (Elias et al., 2006: 76–79; Bikiaris et al., 1999: 4; Perdikatis & Brecoulaki 2008: 564).

Finally, the conducted EDX elemental analysis verifies the high percentage of aluminium and silicon, similarly to the result of the red ochre reference sample. The absorption bands for alumino-silicates observed in the FTIR spectra of the red and yellow samples indicate that natural ochres were applied as pigments, rather than the pure powdered minerals hematite, goethite or limonite.

White pigment
The chemical elements presented in H. sample indicate that calcium carbonate was used for the pure-white plastered walls in the funeral chamber. In accordance with the Ca content found by SEM-EDX, the ATR-IR spectrum of the sample revealed bands for calcite 1797, 1432, 873, 713 cm⁻¹.

3.2. Glossy plasters – presence of beeswax within the paint layer

Analyses conducted on a painting in Alexandrovo tomb (Todorov, 2011: 331, fig. 8) detected beeswax within paint samples of the large red zone in the funeral chamber. The investigation was carried out via Gas Chromatography with mass spectrometer (GC-MS). The FTIR spectroscopy of AL. red sample also revealed considerable evidence for the presence of beeswax (strong C-H stretching bands in the region 3000–2800 cm⁻¹, stretching C=O band at 1738–1736 cm⁻¹, bending C-H bands at 1472–1463 cm⁻¹, stretching C-O band at 1219–1173 cm⁻¹ and torsion C-H bands at 729–719 cm⁻¹) (Glavcheva et al., 2016: 623–626, Figure 4).

Our DSC results confirm the above-mentioned observations and add new evidence on the usage of wax in the ancient process of creating polychromic polished plasters.

Beeswax is translucent solid substance - a complex mixture of hydrocarbons, long-chain esters and fatty acids, along with many other compounds in low concentration (Serpico & White, 2000: 489; Bucwald, 2008: 122). It is a bee secretion and is used for making the cells of the honeycomb. The wax can be produced by melting the honeycomb in boiling water and then straining to remove the impurities (Gettens & Stout, 1966: 5; Masschelein-Kleiner, 1995: 43). Due to its physical properties, beeswax was well known in the Ancient Mediterranean world and was applied in a variety of crafts – mostly as protective coating, adhesive or paint binder (Serpico & White, 2000; Newman & Serpico, 2000; White, 1978: 57).

Beeswax (similarly to other waxes) is chemically stable and its components undergo little changes over the centuries. It melts easily (at approximately 64°C) and this melting point remains fairly constant with ageing. Its heating properties can be evaluated objectively using DSC, which allows melting point
determination and thermal characterisation and is considered the qualitatively and quantitatively available method for wax identification (Ruguo et al., 2011; Buchwald, 2008; Glavcheva, 2016; Knuutinen & Normann, 2000; Burmester, 1992).

The melting properties of the paint samples and the referent beeswax sample were compared by using DSC (Figure 6). Thermography curves clearly indicate transitions with a very close start melting temperature for all the samples (including the referent). The onset of melting is observed at 31–48°C. Broadened curves of KZK., AL., DL., H. and SEV. illustrate comparable melting transition behaviour with predominant melting peaks within the interval of 49–59°C. (Table 3)

The contrast with the sharp melting/endothermic peak of the pure referent is an indication that there
are other inorganic components in addition to the beeswax – mainly mineral pigments responsible for the coloration (ochres and calcite). It could be argued that a small quantity of beeswax within the paint layer affects the thermal curve of the other components (Glavcheva et al., 2016; Knuutinen & Norrman, 2000).

Despite the limited amount of the analyzed material, the results suggest the presence of beeswax within the samples. In addition, the characteristic polydispersity of the components, the ageing and/or the presence of some other inorganic mixtures expand the melting transition area and have an impact on the observed fusion characteristics.

4. Discussion and conclusion

In ancient art painting, beeswax was traditionally associated with the encaustic technique. Numerous studies and scientific discussions were dedicated to this method of painting involving wax as a binder (Ward 1914: 155–164; Ramer, 1979). The present research reveals other less studied applications of waxes in the Hellenistic polychrome painting.

The identification of beeswax in various painted sections of the Thracian wall decoration presents a matter of particular interest. We suggest that this is a result of covering the painted surfaces with a protective coating (based on beeswax) during the final polishing processes – a technique which has resulted in a colour decoration with good visual and mechanical qualities. This hypothesis is based on the following:

- In situ examination, where it was possible to discover traces of polishing of a still-wet plaster. According to P. Mora’s comments on Kazanlak frescoes, the base of the wall decoration was burnished while the rendering was still fresh - an indication for this process being the slight contracting of the surface (Mora & Philippot, 1984: 86). This is valid for the lower part of the frescoes (where beeswax was detected!) in contrast to the coarsely-smoothed plaster surface of the central decorative freeze.

Other publications also report intentional polishing of the large red monochrome part of the wall in Alexander-Drovo tomb (Barov et al., 2006: 212; Todorov, 2011: 328).

Based on these arguments, a conclusion can be drawn that beeswax is presented only in distinct fresco-painted zones, where polishing processes were involved.

b. the preparatory lime-based layers – the craft of plastering.

The texture and appearance of fresco paintings are influenced both, by the number of the applied lime-based layers, and, by the selection of the aggregates and their ratio to binder. Applying a final layer which includes lime and crushed marble (or marble dust) is a well-known technique for improving the ability to polish the surface (Kakoulli, 2009: 29; Brecoulaki, 2006: 436; Weber et al., 2009).

The stratigraphy of the analyzed samples is presented in microphotographs of the prepared cross-sections show (Figure 7).

The basic way of plastering was found to be similar in all of the samples. An initial preparatory layer (also called arricio) was applied on the walls. Its thickness varies according to the structure of the masonry. The preparatory — arricco-plaster layers contain lime and aggregate from local sources, mainly unwashed river sands (this makes the arricio layers look slightly brownish in colour). It is clear that plasters of the finishing layers (also called intonaco) were prepared with carefully selected aggregates (smaller in size and with a light white colour). Crushed marble and lime are observed.

Figure 7. DL. presents the employment of pigmented mortar – a mixture of small amounts of yellow ochre added to the lime. Figure 7. AL. shows a pigment diffusion after polishing of the porous plaster.

One up to three layers of plaster were used in Ancient-Thrace fresco monuments, but marble-composed finishing layer is not common for the Thracian frescoes. The observable crushed marble in the uppermost plaster layer also suggests the finishing polishing processes.

c. statements in the ancient literature sources – secret varnishes.

Several statements presented in the ancient literature sources - comments by Pliny (Natural History) and Vitruvii (De Architectura), highlight the issue of final treatment of the wall with organic coatings (Reinach, 1985: 1–6).

Ancient paint-craftsmen were familiar with the so called “secret varnishes” and the working process of creating polished wall painted surfaces. To achieve the impressive effect of deep colour and mirror-like completion of the wall, a protective coating was required.
We suppose that waxing (using either pure wax or a mixture with other organic components) was applied as a final step of producing burnished plaster, in order to enhance the colour and the texture of the decoration and serve as a surface sealer that could be buffed from a soft sheen to a high gloss. This technique (commonly known today as stucco lustro) is traditionally associated with the Pompean frescoes and the work of the Roman painters (Bruno, 1992; Valeva, 1993; Mora & Philippot, 1984: 86–89). The results obtained for the present research have proved that it was practiced even earlier in Thrace, during the Hellenistic period.

The analytical examination of the wall paintings found in Hellenistic Thrace has revealed significant information on the use of complex painting/plastering techniques. At the same time, several issues concerning the exact recipe of the wax-based coating have emerged. It is to be considered that discovering the ancient painters’ savoir-faire requires further investigation.

Notes

1. When excavated, these monuments were in different states of preservation. While in Kazanlak, Alexandrovo and Helvecia tombs the wall paintings still remain in good condition, frescoes of the Sevtopolis palace and Dolno Lukovo were found detached from the walls and fragmented, thus providing an uncertain graphical representation of their entire decorative schemes.

2. In contrast to the rich painted Macedonian facades, Thracian funeral facades have only several details rendered in colour. Thracian tombs retain the decorative plasters entirely for the interior.

3. In his De Architecture, Vitruvius recommends the use of six plaster layers - the first three consisting of calcium hydroxide and sand and the other three consisting of calcium hydroxide and marble dust (Vitruvius, 1914). Even during the Roman period, the lack of coating displays a technique as elaborate as that described by Vitruvius. According to the analyzed plastered monuments from the Hellenistic period in the Mediterranean, representative number of lime-based preparatory layers vary from one to four (except private houses in Delos, where more sophisticated plaster application is presented) (Brecoulaki, 2006: 436; Kakoulli, 2002: 58-60; Kakoulli, 2009: 33-35).

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