The ancient Demetrias figurines: new insights on pigments and decoration techniques used on Hellenistic clay figurines

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ABSTRACT

The 1995–1996 rescue excavation at the northern cemetery of Ancient Demetrias (Volos, Greece) unearthed a large number of clay figurines of the Hellenistic period (3rd –2nd c. B.C.). The objects exhibit rich polychrome surfaces and represent well-known figurine types of women and children used as grave gifts. This paper aims to present the preliminary results of a new investigation on the study of the materials used to create the polychromy and decorate the figurines. Optical microscopy, portable X-ray fluorescence spectrometry (p-XRF) and ultraviolet illumination (UV) were employed. Among the pigments identified are red ochre, cinnabar, calcite, lead white, madder and copper-based blue. Of special interest is the detection of conicalchite and manganese black as pigments, as well as the identification of tin foil and the determination of its application as a decoration technique. Overall trends are presented on the application of each pigment on specific parts of the objects.

KEYWORDS
Demetrias; clay figurines; pigments; conicalchite; tin foil; p-XRF

Introduction

Ancient Demetrias, the city established by Demetrius Poliorcetes in the first decade of the third century B.C, was the strong political, economical and administrative centre south of Macedonia. Due to its concessive geographical position, it played an important role in the political and military matters of the third and second centuries B.C (Batziou-Efstathiou 1996, 11–43). With the support of the Macedonian kings, Demetrias developed into a strong international commercial port, where its permanent residents were drawn from citizens of various Greek cities, as well as Mediterranean and Middle Eastern countries (Batziou-Efstathiou 2001).

Outside the city walls, cemeteries developed during the Hellenistic era to the south, north and northeast of the inhabited area (Batziou-Efstathiou 1990, 199–201). Rescue excavations on the northern cemetery, sited northwest of the ancient town, at the “Bourboulithra” area (Figure 1), were performed by the 13th Ephorate of Prehistoric and Classical Antiquities (now Archaeological Ephorate of Magnesia) from 1995 until 1996 (Nikolaou 2000, 309–314). The head of the excavation was Ms E. Nikolaou. The cemetery, used as a burial ground from the fourth century B.C. up until the third century A.D., revealed a corpus of painted clay Hellenistic figurines dating to the third and second centuries B.C. The figurines served as offerings in order to accompany the deceased in their journey to the Underworld (Nikolaou 2012, 351–363).

The variety of the terracotta figurines of the northern cemetery, as well as the quality of their construction, indicate the existence of active workshops whose production reflects specific aspects of the economic and social life of Demetrias during the third and second centuries B.C. It is true that the figurines do not actually escape the common typology of the great coroplastic centres; however, there is a different approach to the common types, due to the inspiration of the local coroplasts surely adapted to the particular needs of the inhabitants of this important Hellenistic city (Nikolaou 2012, 351–363).

A preliminary investigation on the decoration of these figurines began in 2009. Permission for the analysis of 160 complete and partial figurines was granted in 2015 by the Greek Ministry of Culture, initiating a systematic analysis and investigation on pigment and painting techniques used. This paper will present the results of the analysis of the first 40 objects, chosen among the ones presenting the richest polychromy. Although in a very preliminary stage, the authors aim to demonstrate the initial results of this investigation, in the hope that it will provide further information regarding pigment and decorative techniques used on clay figurines in the Hellenistic era.

Methods of investigation, limitations and obstacles

All analysis/investigations were performed in the archaeological museum of Volos. All objects were
initially carefully examined under a Leica Wild M32 microscope to map the position of the pigments, which were then non-destructively analysed with a portable X-ray fluorescence (p-XRF) spectrometer, developed at the Institute of Nuclear Physics, NCSR “Demokritos” Greece, using two operational parameters (at 40 kV/50 mA for 300 s and 15 kV/100 mA for 1000 s, respectively). This particular instrument was readily available and provided immediate qualitative results without the need for object transportation or sample removal/preparation, both of which would have required special permission. Spectra from non-painted areas (plain clay) were collected to provide background reference and several spots, covering all pigments present, were analysed on each figurine. Due to the heterogeneity of the samples (both in form and quantity), multiple analysis of the same pigment in different areas (if available) was also performed for cross reference. In most cases, pigments survived in folds and incisions, limiting the capability of the equipment to retrieve securely identified spectra, if at all.

Additionally, all objects were investigated under ultraviolet (UV) illumination for organic lake pigments detection, particularly madder. This was achieved by using a hand-held UV light source emitting UV-A long wave at 366 nm.

The objects have been previously treated and consolidated with a polyvinyl acetate (PVA) co-polymer and, for their majority, gap-filled with plaster of Paris. While the former did not present signs of interference with the analysis, the latter was found to partially intervene. Therefore, results near gap-filled areas indicating small amounts of sulphur were not, at this point, taken into account, through fear of contamination from the conservation material. Furthermore, all objects had been mechanically scalpel cleaned and as a consequence a tremendous amount of pigment was lost and, in most cases, samples were scarce. A significant obstacle concerning p-XRF analysis in this study is the detection of Fe, Si and K on all samples analysed, deriving from the underlaying clay substrate, and thus complicating the secure identification of certain pigments.

Results and discussion

Since p-XRF analysis is unable to securely characterise pigments, especially on complicated multilayer surfaces such as the ones in question, a method of conclusive extrapolation was employed combining all techniques (a summary of the results is presented in Table 1). Carbon-based pigments and organic lakes (apart from madder) could not be detected. On certain samples, carbon black, as the black pigment, was assumed because of the absence of characteristic elements that would identify it as other.
| Find number | Colour       | Elements                                                                 |
|-------------|--------------|--------------------------------------------------------------------------|
| BE 16627    | White        | Ca                                                                       |
|             |              | Ca, Pb                                                                   |
|             | Light blue   | Cu                                                                       |
|             | Red          | Fe, Ca                                                                   |
|             | Salmon pink  | Fe, Ca                                                                   |
|             | Black        | Pb, Ca                                                                   |
| BE 17191    | White        | Ca                                                                       |
|             | Red          | Fe, Ca                                                                   |
|             | Intense      | Hg, S                                                                   |
| BE 16594    | White        | Ca, Fe                                                                   |
|             | Red          | Fe                                                                       |
|             | Intense      | Fe                                                                       |
|             | Pink         | Ca                                                                       |
| BE 46615    | White        | Ca                                                                       |
|             | Red          | Fe, Ca                                                                   |
|             | Pink         | Mn, Fe, Ca                                                               |
| BE 46609    | White        | Ca, Pb, Fe                                                               |
|             | Purple/pink  | –                                                                        |
| BE 46612    | White        | Ca                                                                        |
|             | Red          | Fe                                                                       |
|             | Intense      | Fe                                                                       |
|             | Pink         | –                                                                        |
| BE 16303    | White        | Ca                                                                       |
|             | Red          | Fe                                                                        |
|             | Intense      | Hg, S                                                                    |
| BE 46616    | White        | Ca                                                                        |
|             | Red          | Fe                                                                       |
|             | Intense      | Hg, S                                                                    |
|             | Pink         | Ca                                                                        |
| BE 46686 and 46635 | White | Ca                                                                 |
| BE 46645 and 16984 | Salmon pink | Ca                                                                      |
| BE 16731    | White        | Ca                                                                       |
|             | Light red    | Fe                                                                       |
|             | Black        | Mn, Fe, Ca                                                               |
| BE 16732    | White        | Ca                                                                       |
|             | Pink         | Mn                                                                        |
| BE 46683    | White        | Ca, Pb                                                                   |
|             | Light red    | Fe, Pb, Ca                                                               |
| BE 46659    | White        | Ca                                                                       |
|             | Salmon pink  | Ca                                                                        |
|             | Pink         | Ca                                                                       |
| BE 16498    | White        | Ca                                                                       |
|             | Red          | Fe                                                                       |
|             | Pink         | –                                                                        |
| BE 16146    | White        | Ca                                                                       |
|             | Pink         | –                                                                        |
| BE 46671    | White        | S                                                                        |
|             | Pink         | S                                                                        |
| BE 46636    | White        | Ca                                                                       |
|             | Pink         | –                                                                        |
| Eup. 1040   | White        | Ca                                                                       |
|             | Black        | Mn, Fe, Ca                                                               |
| BE 46650    | White        | Ca                                                                       |
|             | Black        | Mn, Fe, Ca                                                               |
| BE 46798    | White        | Ca, Mn, As                                                               |
|             | Pink         | –                                                                        |
| Eup. 1083   | White        | Ca                                                                       |
|             | Pink         | –                                                                        |
| BE 46655    | White        | Ca                                                                       |
|             | Black        | Mn, Fe, Ca                                                               |
| BE 16734    | White        | Ca, Pb, Mn                                                               |
| BE 17189    | White        | Ca                                                                       |
|             | Pink         | Mn, Fe, Ca                                                               |
| BE 46632    | White        | Ca                                                                       |
|             | Black        | Mn, Fe, Ca                                                               |
| Find number | Colour    | Major | Minor     | Pigment name                  |
|-------------|-----------|-------|-----------|-------------------------------|
| BE 46651 and 46631 | White | Ca     |           | Calcite (?)                   |
|             | Red      | Fe     |           | Red ochre                     |
| Salmon pink | Ca,Fe    | Ca,Fe |           | Mixture of calcite and red ochre (?) |
| BE 46617    | White    | Ca     | K, Mn     | Calcite (?)                   |
|             | Light blue | Cu     | As       | ?                             |
| BE 17217    | White    | Ca     |           | Calcite (?)                   |
|             | Pink     | Hg, S  | Mn        | Madder*                       |
|             | Red      | Cu, As | Si       | Egyptian blue (?)             |
|             | Blue     | Sn     |           | Tinning                       |
| BE 46778    | White    | Ca     | S        | Gypsum                        |
|             | Green    | Cu, As |          | Conichalcite                  |
|             | Light blue | Cu    |           | ?                             |
| BE 46695    | White    | Ca     |           | Calcite (?)                   |
| BE 46620    | White    | Ca     |           | Calcite (?)                   |
|             | Red      | Fe     | As       | Red ochre                     |
|             | Black    | Mn, Fe |          | Manganese black               |
| BE 46657    | White    | Ca     | Pb       | Calcite and lead white        |
|             | Red      | b      |          |                               |
|             | Blue     | b      |          |                               |
| BE 46649 a and b | White | Ca     |           | Calcite (?)                   |
|             | Red      | Fe     | Pb       | Red ochre                     |
| BE 46653 a and b | White | Ca     |           | Calcite (?)                   |
|             | Black    | Mn, Fe, Ca | Pb, Ir | Lead white                    |
| BE 46648    | White    | Ca     |           | Calcite (?)                   |
|             | Pink     | b      |          |                               |
| BE 46689    | White    | Ca     |           | Calcite (?)                   |
| BE 46691    | White    | Ca, S  |          | Gypsum                        |
| BE 46652    | White    | Ca, S  |          | Gypsum                        |
|             | Pink     | -      | -        | Madder*                       |
|             | Light blue | Cu, Ca |          |                               |
|             | Black    | Sn     |           | Tinning                       |
|             |          | Mn, Fe |           | Manganese black               |
|             | Red      | Fe, Hg |           | Red ochre and cinnabar        |

Note: The elements presented are the ones not detected on the control (clay) spectra or have a higher pick than the latter.  
*Identified by UV light.  
*Identified by UV light.  
Very small sample. Could not be analysed.  
p-XRF did not provide a characteristic for identification spectra.  
Microscopic observation.
The majority of the objects present a rich polychromy of more than one pigment, excluding the white substrate, with the creation of hues through pigment mixtures and multiple pigment layer applications. A characteristic example is BE 16627 (Figure 2), where copper-based blue was detected on the garment; a mixture of calcite and lead white below a mixture of red ochre and lead white, to create the salmon pink flesh colour, on the face; and red ochre to indicate the hair. Most of the pigments detected, such as lead white, red ochre, cinnabar, copper-based blues and madder, are often encountered on ancient painted surfaces, such as wall paintings and polychrome artefacts (Mau and Farrell 1993, 55–62;
Figure 5. XRF spectra of the green pigment on BE 46778 (40 kV/50 μA).

Figure 6. Side view of BE 46615 with manganese black as a substrate (magnified image ×10).
Kakoulli 1997, 130–140; Damiani et al. 2003, 341–354; Maravelaki-Kalaitzaki and Kallithrakas-Kontos 2003, 209–225; Brysbaert, Melessanaki, and Anglos 2006, 1095–1104; Perdikatsis and Brecoulaki 2008, 559–567; Kakoulli 2009; Brecoulaki, Kavvadias, and Verri 2014, 152–165; Fostiridou et al. 2016, 453–464; Kakoulli et al. 2017, 104–115).

Madder lake was identified, by its characteristic orange–pink fluorescence under UV illumination (Schweppe and Winter 1997, 124; Casentino 2013), on 12 out of the 40 objects analysed. In visible illumination all samples examined exhibited a pinkish purple colour, with the exception of BE 46612 and BE 46609 which exhibited a more purple tone (Figure 3).

Madder lake colour can range from different nuances and shades of red (light red, bright red, bluish red), to pink and purple (Kirby, Spring, and Higgitt 2005, 71–87; Brecoulaki 2014, 3–35; Fostiridou et al. 2016, 453–464). This is partly due to the different botanical species used, through their different principle colouring substance. For example, dyer’s madder (Rubia tinctorum L.) contains high amounts of alizarin resulting in red lakes with a bluer tint, while wild madders’ (Rubia peregrine L.) main colourants are pseudopurpurin and purpurin which form pink madder (Schwepe and Winter 1997, 112, 117–119; Cardon 2007, 112, 122).

It can also be due to the manufacturing process, and the inorganic substrates and precipitated compounds used to form the lake (mordants). It is known that different mordants are prone to produce different coloured hues with the same dye (Timár-Balázs and Eastop 1998, 74). For

Figure 7. BE 16731: manganese black applied to form the face features (magnified image ×6.5).
example, madder will provide a deep red colour with soda or potash, however, with iron (II) sulphate the result is black violet (Schweppes and Winter 1997, 112).

The most commonly used mordants of aluminium and calcium-based metal salts (Kirby 1977; Encyclopaedia Britannica 1998; Kirby 2011; Wongwad et al. 2012, 73–78; Kakoulli et al. 2017, 104–115) could potentially be identified by means of p-XRF via their characteristic elements. In the present study, aluminium was detected only on the madder sample of object BE 16732, while the XRF spectra of all other samples were no different to the background reference spectra, demonstrating the need for an alternative analytical method.

Apart from the preceding, particularly interesting observations include the following.

**Conicalchite**

The green pigment on object BE 46778, and the only green pigment currently detected in this study, appears to be conichalcite (calcium copper arsenate hydroxide – CaCu(AsO4)(OH)) due to the high peaks of arsenic and copper presented on the XRF spectra (Figures 4 and 5). The possibility of yellow and blue pigment mixture is excluded since only green pigment particles are visible under high magnification. Occurrences on the use of green pigments in general for the decoration of Hellenistic figurines are scarce (Pagès-Camagna, 2010, 250–251), conichalcite even more so. It has been used in association, or mixed, with malachite and yellow ochre on objects of the Macedonian era, and in pure form on the funeral couch of the tomb of Philip II at Vergina (Brecoulaki

\[\text{Figure 8. BE 46653a: manganese black applied on top of white background (magnified image ×15).}\]
and Perdikatsis 2002, 147–154; Perdikatsis and Brecoulaki 2008, 559–567). On clay Hellenistic figurines it has, thus far, been encountered only once on a figurine belonging to the collection of the Louvre Museum, where it was associated with the main malachite pigment (Pagès-Camagna 2010, 250–251).

Manganese-rich substrate/pigment

Manganese was detected on 10 out of the 40 objects analysed, mostly applied to provide the background layer. The possibility of it being a contaminant deriving from the burial soil is excluded, since either the brush-strokes are clearly visible or it is applied to indicate

Figure 9. BE 46652: tinning applied to enhance the throne (magnified image ×10).

Figure 10. BE 17217: upper part of the object with arrows indicating the detected tinning areas (magnified image ×10).
particular features. In this study, manganese-rich layers are presented in three forms: as a substrate directly onto the clay (Figure 6) which could imply that its application preceded the firing process; as a thin (Figure 7) and as a thicker pigment layer on top of a white substrate (Figure 8) which probably succeeded the firing process.

Its use has been recorded on murals since the Palaeolithic era (Profi, Perdikatsis, and Filippakis 1977, 107–115; Kakoulli 1997, 130–140; Chalmin et al. 2007, 220–222) and on various non-figural terracotta objects (Schweizer and Rinuy 1982, 118–123). It has been detected as the colour used to represent the hair and beard on a carved lime stone male head of the Upper Palaeolithic era (Powers 1994, 148), and as the black pigment, applied before firing, on terracotta figurines of the Cypro-Archaic I and II periods (Aloupi and MacArthur 1995, 145–155). Bibliographically, it has been only briefly mentioned on Hellenistic figurines from Pherai, an ancient town a few kilometres away from Demetrias (Asderaki-Tzoumerkioti and Doulgeri-Intzesiloglou 2010, 1–11; Asderaki-Tzoumerkioti et al. 2015, 311–320). Since, as it seems, the preferred black pigment of that period and of similar objects, according to current studies, was carbon black (Higgins 1970, 272–277; Mau and Farrell 1993, 55–62; Maravelaki-Kalaitzaki and Kallithrakas-Kontos 2003, 209–225; Fostiridou et al. 2016, 453–464), the use of manganese black in the region is of special interest.

**Tinning**

Another interesting aspect of this research is the presence of tinning. Although there are examples of gilding, particularly gold, as a decorative mode
**Table 2.** Position of each colour on finds.

| Colour | Value | Bk White | Quinac | Gore | Medium | Other | Garment | Face | Head | Body | Garment | Face | Head | Body | Garment | Face | Head | Body | Garment |
|--------|-------|----------|--------|------|--------|-------|---------|------|------|------|---------|------|------|------|---------|------|------|------|---------|
| Red 2  | 2     | 2        | 2      | 2    | 2      | 2     | 2       | 2    | 2    | 2    | 2       | 2    | 2    | 2    | 2       | 2    | 2    | 2    | 2       |
| Orange | 2     | 2        | 2      | 2    | 2      | 2     | 2       | 2    | 2    | 2    | 2       | 2    | 2    | 2    | 2       | 2    | 2    | 2    | 2       |
| Blue   | 2     | 2        | 2      | 2    | 2      | 2     | 2       | 2    | 2    | 2    | 2       | 2    | 2    | 2    | 2       | 2    | 2    | 2    | 2       |
| Green  | 2     | 2        | 2      | 2    | 2      | 2     | 2       | 2    | 2    | 2    | 2       | 2    | 2    | 2    | 2       | 2    | 2    | 2    | 2       |
| Purple | 2     | 2        | 2      | 2    | 2      | 2     | 2       | 2    | 2    | 2    | 2       | 2    | 2    | 2    | 2       | 2    | 2    | 2    | 2       |

*Applied on the whole sample
+ On the reverse
§ Timing is not included in the table
¶ BE 12117 is not included in the table since it only displays pigments on the feathers
on painting surfaces (Karydas et al. 2009, 811–829; Verhagen 2012, 5; Brecoulaki 2014, 3–35; Brecoulaki, Kavvadias, and Verri 2014, 152–165; Brecoulaki et al. 2014, 9–22), there are limited occurrences of tinning, once again solely from the town of Pherai (Asderaki-Tzoumerkioti and Doulgeri-Intzesiloglou 2010, 1–11; Asderaki-Tzoumerkioti et al. 2015, 311–320). Tinning appears on two of the studied finds. On find BE 46652 it was applied as a strip on the upper part of the throne, presumably to enhance it and to create a contrast with the manganese black background (Figure 9), while on find BE 17217 it was applied longitudinal to the feathers’ exterior area to enhance even more their already dazzling appearance (Figures 10 and 11). It is assumed that its use would provide a relatively equal visual effect to silver but with lower cost, thus adding to the objects beauty, appeal and value. At the moment it is not clear whether this

![Figure 13. Materials used for the white substrate.](image)

![Figure 14. BE 46612: application of lead white as a separate layer on the face (magnified image ×6.5).](image)
Figure 15. BE 46649 a and b: Red ochre on the hair area (magnified images ×10).

Figure 16. BE 46686 and 46635: salmon pink on the face area (magnified image ×15).
A tin layer was applied as a foil or by dipping the part into a tin melt, similar to pottery tinning (Holmberg 1983, 383–384).

However, on find BE 17217 the presence of a cinnabar layer, identified by its characteristic mercury peak on the XRF spectra (Figure 12), under the tin layer (visible on the detailed photograph of Figure 10) could indicate the use of the bole gilding technique, using cinnabar as the bole. This particular technique has been identified as the gilding technique on the chryselephantine couches from ancient Macedonia (Brecoulaki et al. 2014, 9–22) and on Tanagras figurines from the Louvre Museum’s collection (Pagès-Camagna 2010, 250–251). In both the aforementioned cases, gold leaf was applied above a yellow ochre bole. Perhaps the choice of the bole used would have depended on the nature of the metal overleaf and on the desired visual result of the end product. In the present study the use of cinnabar, an expensive and sparingly used pigment, as the bole instead of a cheaper pigment, such as red ochre, is particularly interesting.

**Figure 17.** Distribution of salmon pink pigment.

**Figure 18.** Distribution of manganese black.

**Figure 19.** BE 17191: cinnabar detection on the lips (magnified image ×16).
Application trends

Observing conclusively the results of this research, a trend is established concerning the areas of application of each pigment (Table 2). All objects analysed exhibit a white substrate used as the background layer. This layer provides the homogeneity needed for a smoother application of the following pigment layers, plus a neutral background which will not alter or interfere with the final chromatic result. As seen in Figure 13, calcium-based white (possibly calcite – CaCO₃) seems to be the preferred background layer. Lead white (basic lead carbonate – 2PbCO₃·Pb(OH)₂) was applied on top either as a separate layer (Figure 14), or as a mixture with calcite, in order to create an opaque, compact, smooth surface and to enhance the facial area with its subtle yellow-red undertone. In the few cases where gypsum (CaSO₄·2H₂O) was used as the background layer, no other white pigment was applied on top.

Red ochre, the predominant red pigment used, is mainly applied to represent the hair (Figure 15) or mixed with calcium-based white to create a salmon pink colour covering mainly the flesh parts (Figures 16 and 17). Manganese black is mainly applied at the backdrop, with the exception of BE 16731 where it has been used to underline the facial features (Figures 7 and 18). Cinnabar, a very stable and expensive pigment with special gloss, good covering characteristics and adhesive strength (Nöller 2015, 79–87), had a limited use, predominantly to represent the lips (Figure 19), while madder, copper-based blues and conichalcite were used on the drapery to enrich the colourfulness of the garments.

Conclusions – further research

Microscopic investigations aided in detecting various pigments and their combinations, while p-XRF analysis and UV illumination helped their characterisation. It is understood that the analytical techniques available at this stage can only provide certain, and sometimes inconclusive, information. Nevertheless, p-XRF provided a useful tool for the detection of pigments in areas which appeared pigment-free even under the microscope.

Successive pigment layers, colour alterations and tinning would certainly create a spectacular visual result. The fact that tinning and manganese black are solely encountered in this region could possibly demonstrate the flourishing of a particular clay figurine industry.

Apart from the observations, many additional questions arose. All pigments encountered at this stage should be securely identified, with emphasis on the application stage of the black pigment containing manganese, and on the nature of the blues containing copper. Further investigation is needed in the case of colour difference between the madder samples. Last but not least, tinning should be investigated in-depth as a newly discovered trend in Hellenistic clay figurines decoration.

To complete the research and to acquire a fuller understanding of the pigments, the layers of application and the techniques used, physical sampling and the use of alternative analytical investigating methods are required.

Notes

1. Commonly used mordant in antiquity was hydrated alumina, manufactured by using potash alum (KAl(SO₄)₂·12H₂O) as a substrate, and potash (KOH), soda ash (Na₂CO₃) or lime (Ca(OH)₂) to facilitate the reaction (Kakoulli et al., 2017: 104–115).
2. Aluminium can be identified by p-XRF if it is present in large quantities (Karydas et al., 2009).

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Disclosure statement

No potential conflict of interest was reported by the authors.

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