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pXRF and Polychromy: Identifying Pigments on Limestone Statuary from the Roman Limes, Preliminary Results

Louisa Campbell [1,*] and Charleen Hack [2]

[1] Archaeology, School of Humanities, College of Arts and Humanities, University of Glasgow, Glasgow G12 8QQ, UK
[2] Institute of Ancient Studies, Johannes Gutenberg University, Jakob-Welder-Weg 20, 55128 Mainz, Germany; chhack@uni-mainz.de
* Correspondence: louisa.campbell@glasgow.ac.uk

Abstract: This paper presents the preliminary results of an investigation on the unexplored topic of polychromy on provincial stone sculptures from the Roman provinces in Germania through the innovative application of heritage materials science techniques. A group of three life-sized statues dating to the 1st Century CE recovered from Ingelheim, near Mainz, retains remarkably well-preserved traces of pigments. These are ripe for emerging non-invasive technologies supplemented by micro-sampling to validate results and provide information relating to mixing and layering not available to the naked eye. The most strikingly visible areas of extant polychromy were retained on the sculpture of a young woman, reported on here as the first phase of this programme of research. The results suggest that the statue was originally covered in a gypsum layer before the application of complex and diverse recipes of pigment applied as mixtures and in layers to create required hues and shadowing on sculpted features. The palette includes ochres and green earth mixed with small amounts of minium (red lead), realgar and lapis lazuli (ultramarine blue) added to create skin tones, and a vibrant blue-green tunic created from Egyptian blue, bone black, ochres, cinnabar and green earth; the *palla* and *peplos* contained ochres, bone black, and orpiment, and mixes of these created the detail of coloured jewellery. Of great interest was the detection of bone black on many features, particularly as a shading agent to enhance sculpted features, such as folds in cloth, providing a more realistic and flowing articulation. This is a revolutionary observation that provides previously unexplored insights into artistic polychromic practice in Antiquity.

Keywords: polychromy; pigments; Roman sculpture; limestone sculpture; pXRF; microphotography; multi-spectral imaging; Roman frontiers; Germania

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1. Introduction

Three funerary statues from Ingelheim upon Rhine (Rhineland-Palatinate, Germany) are among the most outstanding sculptures from former Roman provinces in Germania. In addition to their artistic qualities, the life-sized sculptures of a man and woman as well as the torso and head of another woman are particularly significant due to traces of original polychromy. Despite the fragmentarily surviving character of the pigments, sufficient traces remain to reveal their original vibrant colour scheme which greatly enhances our understanding of contemporaneous garments and their socio-cultural context. They also provide insights into the statues' performance to contemporary audiences.

Since interest in provincial Roman polychromy [1–4] is increasing exponentially, these sculptures have been selected as the principal components of a digital reconstruction and colouration of their associated funerary monument [5]. Preliminary, as yet unpublished, micro-samples were extracted from a small number of locations on the two complete statues in 2022 [6]. Following on from that work, we have undertaken a comprehensive programme of research on the sculptures, including non-invasive portable X-Ray Flourescence (pXRF) and microphotography, supplemented by further micro-sampling from selected features for more detailed laboratory-based analysis.

The female sculpture retains the best-preserved pigments, so the results reported here focus exclusively on that statue. This work constitutes the first such investigation of polychromy on Roman statuary from the German Limes and is, therefore, transformational in understanding polychromy practice in the provinces.

2. The Woman's Statue: An Overview

In 1853, three Roman statues carved from Lorraine limestone (Figure 1) were discovered during archaeological fieldwork. They were positioned face-down in the ground at a shallow depth. Written-up many years later, the associated excavation report also mentions brick-built structures which were neither fully described, nor excavated [7] (pp. 325–326). This inadequate documentation relating to find contexts has, regrettably, resulted in the loss of critical information, including their precise location of discovery which can now only be estimated [8] (p. 10), [9] (pp. 30–31). What we can say, with certainty, is that the statues were found north of the former Roman vicus, today's city centre of Ingelheim (Figure 2).

Figure 1. Grave statues from Ingelheim upon Rhine (© Museum bei der Kaiserpfalz Ingelheim/baermedia GmbH Co. KG, used with permission).

The probable location lies close to the course of the Roman trunk road that connected nearby *Mogontiacum* (Mainz), later capital of the province *Germania superior*, with *Colonia Claudia Ara Agrippinensium* (Cologne) [10] (pp. 261–262, Figure 148). It is unclear whether the structures briefly referred to in the excavation report were built in Roman times or later. Initially, it was thought likely the sculptures did not reach their place of discovery until 8th Century CE and were subject to secondary post-Roman use in a medieval building [11] (p. 32).

Ingelheim became an important location during the 8th Century through Charlemagne's construction of an imperial palace [12], as the colourful interior design was clearly inspired by Roman traditions [13]. Additional buildings similar in style constructed close to the Rhine cannot be ruled out. Critically, a secondary period of use for the statues could have entailed an early medieval repainting of the tomb figures [14]. Repainting of stone monuments may also have been practiced in Roman times, potentially replicating original colours, while medieval artists may have applied different colours reflecting contemporaneous preferences hundreds of years later [4] (p. 5230) [15].

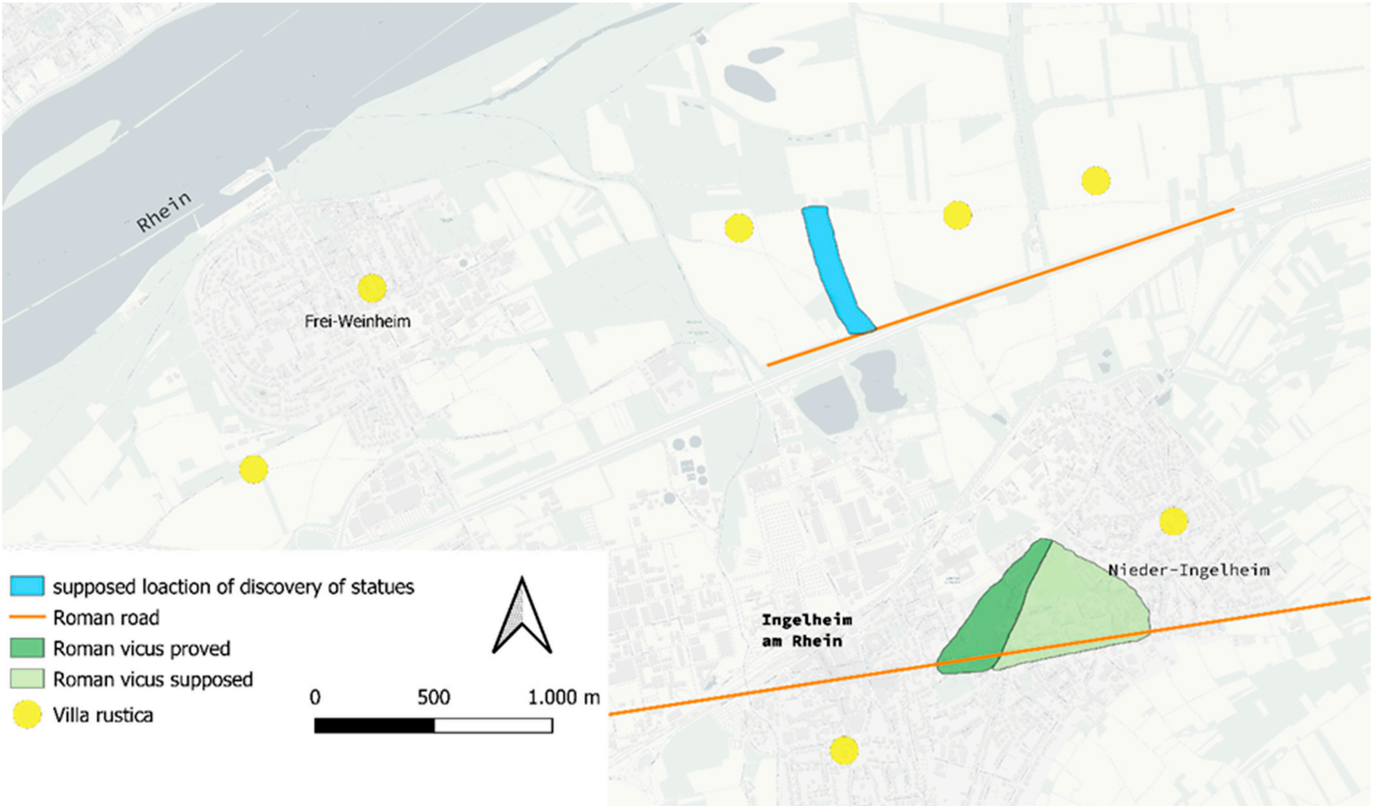


Figure 2. Map of Roman Ingelheim upon Rhine, based on map [16] (p. 23, Figure 3).

The presumed site lies within a protected nature reserve today, so it is not possible to perform any exploratory excavations to confirm exact locations or associated structures. We cannot be certain, therefore, whether the place of recovery corresponds with a Roman or post-Roman deposition of the statues. Nearby traces of a bigger *villa rustica* east of the proposed discovery location [9] (p. 54–55), [17] (p. 73 no. 261) may, however, suggest the statues were once components of a tomb at the burial site on this estate [9] (pp. 30–31), [18] (p. 402). Such a positioning would not be unusual for *Germania superior*, e.g., in Frankfurt-Zeilsheim [19], Avanches, Augst, and Langres [20] (p. 104). The discovery of only the statues could also be explained by the secondary use of Roman components. Although the ashlars and columns of an aedicule tomb might have been used for the construction of new monuments without much extra effort [21] (p. 679), the statues could have remained in place after the monument was dismantled.

Although it has been suggested the statues belonged to a sepulchral monument, the corresponding type remains debated. Because of their unfinished condition, with backs only roughly hewn, the statues are likely to have been carved with the intention of placement into a tomb niche [19] (p. 305), [22] (p. 113). The absence of visible pigments from Antiquity on the backs of all three sculptures appears to support that probability. Even if they were freestanding, perhaps only the fronts were visible from an original viewer's perspective, rendering the carving of rear features unnecessary [23] (pp. 203–204). In the museum setting today, at first glance, the sculptures appear to be complete in all directions, but freestanding statues of the aedicule monuments from Cologne [24] (p. 61) and Sarsina [25] have comparable sculpted features to the Ingelheim examples. Therefore, it is reasonable to infer that the structure of their associated tomb is comparable to this monument type [26] (pp. 59, 75).

Despite their surface damage, the exquisitely articulated features that remain are testament to the indisputable superior skill of the sculptor who created these magnificent monuments. The combination of hairstyles with the method of garment draping dates this group confidently to the Claudian period, joining a series of funerary monuments attributed to a known workshop from Mainz [27,28] (pp. 60–86).

Stylistically, the sculptures showcase the embracing of some aspects of Roman cultural traditions during the 1st Century CE by some members of society in the Germanic provinces [29] (pp. 170–174). The man is marked as a Roman citizen by wearing the toga. Paradoxically, the two women are not dressed in the Roman manner, but rather their attire is a hybrid Gallo-Roman style. This stylistic practice is known as *Menimane costume*, after the wife on the *Blussus gravestone* from Mainz-Weisenau [30–32] (pp. 199–207) and has been depicted on several gravestones dating to the 1st Century in the Treverian area around Mainz and Trier [33] (no. 2, 3, 12), [34,35].

Following this hybrid fashion, the female statues are depicted with tight, blouse-like tunics topped with additional scarves tucked into the neckline. Over the tunics lie fabric tubes resembling *peploi*. Unlike the Greek garment, however, the dresses in Ingelheim are not open at the long side, but rather, they are closed by a seam. Like the Greek *peplos*, this upper garment is held together with a *fibula* on each shoulder. An additional brooch attaches the skirt to the tunic underneath at the solar plexus. The resultant drapery below the breast is characteristic of this costume style.

Figure 3. (**Left**)—a nearly complete figure of woman statue from Ingelheim upon Rhine; (**right**)—surviving torso of second woman.

The clasps of both women are known as *thistle fibulae* due to their resemblance to the thistle flower. These *fibulae* are better preserved on the statue of the woman that survives as a torso (Figure 3, right). Despite the generally poorer preservation of the female bust, a similar garment style can be assumed for her as is depicted on the complete female figure that is the subject of this article. Several examples of these types of personal adornments have been recovered from archaeological contexts dating to the 1st Century, usually made with bronze, and their distribution across the Celtic-Germanic circle [36,37] (pp. 101–105) confirms cultural roots in these societies. Over these two garments of Celtic origin, each of the women wears a large, mantle-like piece of fabric that drapes around their bodies. This garment is most commonly interpreted as *palla*, a coat worn by Roman women in public [38] (pp. 426–428). The whole ensemble, therefore, is a manifestation of hybrid practice in the province created through the blending of traditional Celtic garments, artefacts, and style with emerging Roman fashions.

The deceased commemorated on these Ingelheim statues were very clearly of high status. The associated tomb monument was probably originally more than 10 m high. The purse held in the left hand of the woman (Figure 4, right) combined with various pieces of jewellery represents the wealth of the deceased. Most striking is probably the torc around her neck (Figure 4, left), which also originates in Celtic-Germanic tradition [27] (p. 272), [38] (428–429) [39] (p. 75 no. 733–734). It is carved in high profile and represented as a circular feature twisted from several wires. It is not possible to determine whether an opening is depicted on the front due to surface damage, though there do not appear to be any gaps in the raised profile of the piece. That said, it is possible that any such opening would be expected at the rear of the statue, which is uncarved in this instance. In fact, there do not currently appear to be any comparable twisted examples with stamped ends from the early imperial period from the north-western provinces. The representation on our Ingelheim statue does resemble examples depicted on tombstones from the Danubian provinces, which date to the second half of the 1st Century CE, e.g., [40] (p. 148 no. 106.4; p. 159 no. 148). Indeed, depictions of the Noric-Pannonian costume on such gravestones similarly strongly resemble the *Menimane costume* in their articulation of under and upper garments as well as the additional scarf and the positioning of fibulae, e.g., [40] (p. 152 no. 114.7).

Figure 4. Torc around neck, green pigment on tunic, and dark shaded areas of the scarf (**left**); pink between fingers and ring on left ring finger (**right**) (both © Museum bei der Kaiserpfalz Ingelheim/C. Hack, used with permission).

Although bracelets on the wrists of the female figure in Ingelheim have a simple appearance, the armlet on the right upper arm has a distinctive profile. In addition to the simple bronze arm rings, there are several known examples with a comparable design made of glass. Most of such bracelets from *Augusta Raurica* (Augst, Switzerland) were made of black glass [39] (pp. 245–246 no. 624–631). The last piece of jewellery to be mentioned is a ring on her left ring finger, which is difficult to identify due to the surface damage to her left hand caused primarily by degradation of the limestone (Figure 4, right). The style of this finger ring cannot, therefore, be accurately assigned, but it is clearly visible as a raised sculpted feature along with pink pigment that visibly survives in grooves between the fingers on either side of it.

The woman's hairstyle is more challenging to define (Figure 5, left). The very dark covering on her head is commonly interpreted as a bonnet or hairnet within which the hair is bound [32] (pp. 203). If it served as a net, however, no visible sculpted or coloured structures are visible and the covering pigmentation appears relatively consistent throughout, suggesting a more solid, flat, adornment. Variations in colours and painted structures help to interpret this feature. For example, on the left side of the woman's head, the original colouration is almost fully preserved. It presents as a faded greyish-blue visible on the headpiece, most likely a bonnet, both on the back of her head and on the bulge at the nape of her neck.

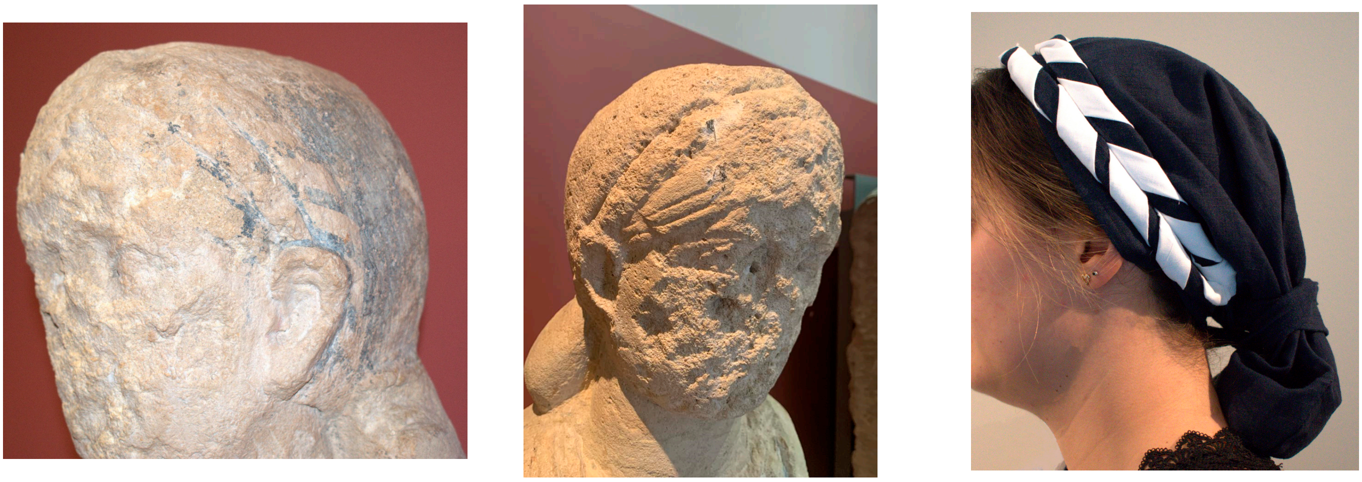


Figure 5. Left and right side views of a female statue with painted bonnet, stripes, and a lock of hair (**left**); experimental reconstruction of bonnet from linen and hair (**right**) (© Museum bei der Kaiserpfalz Ingelheim/C. Hack, used with permission).

Details are highlighted by stripes painted with the same pigment on what appears to be a raised double-ribbed headband. Although both strands showed visible stripes on a picture published in 1930 [27] (Table 26 Figure C), only approximately half of the painted stripes on the first band remain visible today. That said, the diagonal arrangement and clear separation of individual stripes interspersed with light stripes may represent two ribbons twisted in contrasting directions, creating a herringbone pattern [32] (p. 199, Figure 20.2). Since the stripes are only preserved at the left side of her head, it is unclear whether the ribbons ran parallel from one side to the other across the head, or if they were mirrored at the crown, but it is reasonable to assume they were emulated across the whole headwear.

Of particular interest is what appears to be a painted lock of hair that has escaped its covering and remains visible trailing down her left temple. Alternatively, this could be a strand of the ribbon woven across her headband as it is not possible to distinguish between possible dark hair colour and the dark proposed headpiece/bonnet. It is, therefore, possible that the painted stripes on her headband are indeed fabric ribbons woven together with strands of lighter coloured hair.

To test this hypothesis, and in an attempt to clarify details of the hairstyle and accessories, the museum at Ingelheim undertook experimental reconstruction to recreate the feature (Figure 5, right). A bonnet of linen emulating the headpiece was sewn and interwoven at the front with strands of the wearer's hair in the style articulated on the statue. When draped correctly, the twisting of bands into the hair ensured the headpiece was firmly affixed to the head.

Several areas of the woman's tunic retain traces of a vibrant green pigment (Figure 4, left), described by Ferdinand Kutsch as "olive green" [27] (p. 271). This pigment visibly survives on her left and right breast, right arm, under the right armpit, and on both sleeve cuffs. Although Helmut Schoppa's description of the colour as "light green" [41] was adopted in later descriptions [32] (p. 220), this does not adequately capture the vibrancy of this surviving colour.

The scarf folded into the woman's neck above the tunic retains a few barely visible patches of red beside dark colours [42], but the latter is only in the fabric folds (Figure 4, left). The same dark pigment is visible at different locations, always in shaded areas and between garment folds on the two complete statues (Figure 6).

Figure 6. Red on the upper garment, just above the palla, and greyish-black on top of the bulge, between the upper garment and palla (© Museum bei der Kaiserpfalz Ingelheim/C. Hack, used with permission).

The original polychromy applied to the upper garment offers more intrigue. Although traces of red are visible at the front, just above the bulge of the *palla* (Figure 6), light yellow-greenish colours were recorded 100 years ago [27] (p. 272), but those can no longer be seen with the naked eye. It is possible that these and other surviving pigments were unintentionally removed during cleaning of the statues in the 1950s [41]. The colour of the *palla* was always described as greyish-black, though this dark colour was only visible on top of the bulge and below it. Therefore, it probably served as a shading colour or painted demarcation of individual features, see below.

Aside from clothing, the woman's skin was also rendered with a natural flesh tone pigment. These have largely disappeared due to heavy surface damage to her face, but pink traces do remain visible between the fingers of her left hand (Figure 4, right) and inside the left ear.

In addition to ancient pigments, traces of more recent paint are also present on the statue, probably the result of contamination from storage conditions. Shortly after their recovery, all of the statues were transferred to the "Sammlung Nassauischer Altertümer" as gifts. This collection of antiquities is part of today's *Stadtmuseum am Markt* in Wiesbaden. Although the statues were part of an exhibition in Wiesbaden for around 120 years, the high number of monuments included in a "hall of stones" from Antiquity meant that the Ingelheim statues received very little attention during the 19th century [43] (p. 16, Figure 1; p. 17, Figures 2 and 3). For many years, the figures were placed against a wall [44] (pp. 155–156), where they likely received some surface scratches. Indeed, green and black brush strokes can be found on the right side and the back of the woman's statue (Figure 7). The colours are not degraded in the manner of original pigments, so it is reasonable to assume these are more modern contaminants. During WWI and WWII, the circumstances of storage are not clear and it is, therefore, possible that some of the surface scratches could have occurred during these recent episodes of the statues' itineraries. On a positive note, the statues were unlikely to have ever been installed outdoors for long periods of time and, therefore, not exposed to extreme weather or harsh environmental conditions.

Figure 7. Modern paint (green and black) on the right hip of the statue. (© Museum bei der Kaiserpfalz Ingelheim/C. Hack, used with permission).

In the late 20th century, the feet of both complete statues were reconstructed to provide a coherent representation. Fractures suffered as a result of post-depositional processes were also restored at that time. The colour of this conservation and these reconstructions is a perfect match for the unpainted limestone. Nevertheless, the smooth surface texture of the affected areas makes it easy to distinguish them from the timeworn original limestone. Thus, both recent colour and restoration areas could be reliably excluded from the investigation of Roman polychromy (Figure 8). Since 2021, the figures have been shown at the *Museum bei der Kaiserpfalz* in Ingelheim as loans from the *Stadtmuseum am Markt* in Wiesbaden. In addition to a resulted smartphone app, the colour scheme of the figures is discussed intensively in (self-)guided tours [5].

Figure 8. Complete female statue from Ingelheim, with restoration areas (marked in red) and locations of microphotography, indicated by numbers. Museum bei der Kaiserpfalz Ingelheim/baermedia GmbH Co. KG; edited by C. Hack.

3. Methods

Pigments are remarkably well-preserved on some sculpted features where traces remain clearly visible to the naked eye. Their survival is undoubtedly the result of minimal post-depositional interventions except for reconstruction of some areas mentioned above. These were carefully avoided during this analysis to minimise contamination from modern materials.

In July 2022, three microsamples were taken for Scanning Electron Microscope (SEM) and X-Ray Diffraction (XRD) analysis, one each from the red belt-like feature/lower *palla*; the black/grey bonnet; and the green/blue tunic cuff [6]. That analysis, planned for future publication, confirmed the red to be haematite (Fe_2O_3), a pigment commonly used in prehistoric times, and the desired hue was achieved by mixing different pigments through the presence of hydroxyapatite (calcium and phosphate found in bone, see below), calcium carbonate, and lime, the latter possibly derived from a dried binding agent. The black was identified as hydroxyapatite ($Ca_5(PO_4)_3(OH)$—bone black, previously considered less common than carbon-based blacks. However, emerging heritage materials science techniques have detected bone black in pigment mixtures on wall paintings, sarcophagi, and stele from Antiquity [45–48]. The previous SEM/XRD report suggested the green to be "Egyptian green" based on the presence of cuprorivaite, but it is herein confirmed as Egyptian blue ($CaCuSi_4O_{10}$), a calcium-copper-silicate with a crystalline structure in use since the 3rd Millennium BCE in Egypt.

Leading on from that initial research and to identify the broader palette of pigments present as well as their method of application, this current research subjected the sculpture to a suite of complementary analytical techniques. Portable non-invasive technologies, including portable X-Ray Fluorescence (pXRF), VIL imaging, and microphotography, were deployed to assess surface conditions and analyse the elemental composition of pigments detected on each sculpted feature. These techniques were supplemented with micro-sampling, where appropriate, for light microscopy and for embedding into resin for cross-section analysis to identify evidence for stratigraphic layers and/or mixing in the next phase of research. Since pXRF provides only summative information on elemental composition, a primary objective of this work was to determine whether the previous SEM/XRD results permit hypotheses based on the pXRF results to be supported.

3.1. Portable X-Ray Fluorescence (pXRF)

The pXRF instrument used was an Olympus Vanta M Series (VMR-CCC-G2-K) hand-held analyser with rhodium anode in a 4-W X-ray tube capable of voltage up to 50 kV. The instrument operates with two beams: one at 10 kV and one at 50 kV. Analyses were undertaken in the GeoChem (G2) mode where the X-ray tube operated at 40 kV and ~70 µA to measure heavier elements and at 10 kV and ~90 µA to measure lighter elements. The instrument was calibrated using the calibration spot provided by the manufacturer and samples were taken of the background stone to differentiate between the raw material and surface treatments. The measurement time was 30 s: 10 s for the heavier elements and 20 s for the lighter elements, and the area of analysis was 7 mm^2. Several of the forty elements from Mg to U that the instrument can detect were present below the limit of detection (LoD), and light elements with fluorescent peaks at low energies were poorly resolved at low concentrations.

A total of twenty-nine analysis spots were captured from this sculpture, as well as three additional ground unpainted locations. Sample spots are recorded in Table 1. Composition tables comprising the full dataset are provided in Supplementary Materials, while the concentrations of each main element associated with the pigments are provided in Table 2. The elements related to each painted feature are discussed in-text. Elemental concentrations are expressed in parts per million (ppm). Some elements, including Rb, Sr, and Zr, have been excluded from the broader discussion on analysis as probably occurring naturally in the limestone, confirmed by the ground analysis spots. The remaining 16 elements provided a level of quantification at various spots in concentrations sufficiently above background levels to confidently identify the pigments present, although some only at low trace levels. Samples were taken from as many features as possible to compare results, colours, and mixing/layering techniques.

Table 1. Locations of microsamples and pXRF analysis.

Spot No.	Location	Colour	Micro-Sample	pXRF
1	Right side head hair strand 1	Mix—yellow, brown, black		pXRF1
2	Right side head hair strand 2	Mix—black, some yellow		pXRF2
3	Right side of neck under jawline	Yellowish		pXRF4
4	Right side head band	Black		pXRF3
5	Right side head band	Black		pXRF5
6	Right side head bonnet and band	Mix—black, creamy-yellow	M1	pXRF19
7	Right side head base of bonnet knot	Mix—black, creamy-yellow		
8	Left cheek	Mix—cream, reddish orange		
9	Neck right side on torc/scarf shading	Mix—red, black, yellow	M9	pXRF6
10	Right ear inside	Pink	M2	pXRF22
11	Right side tunic bust	Mix—green, blue, red, yellow	M3	pXRF15&16
12	Right side cloak front	Reddish pink		pXRF7
13	Top right fold upper garment	Yellow		pXRF8
14	Top of fibula on bust	Reddish pink		pXRF9
15	Centre fibula on bust	Yellow		pXRF10
16	Centre of upper garment	Yellow		pXRF11
17	Fold of upper garment	Mix—grey with black inclusions	M4	pXRF12
18	Middle upper garment near cloak	Mix—red, orange, yellow		pXRF13
19	Right hand cuff of tunic	Mix—blue, red, green, purple		pXRF14
20	Centre of palla	Yellow		
21	Centre of top fold on palla	Yellow		pXRF18
22	Top of fold on left side palla	Mix—grey with black inclusions		pXRF27
23	Fold on front of palla	Yellow		pXRF26
24	Left hand index finger	Mix—cream, grey with black inclusions		pXRF20
25	Left hand, index finger shading	Pink with darker areas		
26	Left hand ring finger (possible ring)	Yellow		pXRF21
27	Left knuckle	Pink		
28	Right hand ring finger, inside ring	Red		
29	Right side on palla	Mix—yellow, cream (crustation on top with flecks of red, black)	M5	pXRF29
30	Left armpit of tunic	Mix—different shades of blue, greenish, and red inclusions	M8	pXRF17
31	Top of fibula left shoulder	Yellow		
32	Front of palla	Light greenish-yellow	M7	pXRF24
33	Front of palla	Light greenish-yellow	M6	pXRF25
34	Under crack on palla (shading)	Black		pXRF23
35	Inner ear	Pink	M2	pXRF22
36	Inner ear	Pink	M2	pXRF22

Table 2. Results of pXRF analysis (displayed as ppm).

Sample	Location	Colour	Mg	Al	Si	P	S	Ca	Mn	Fe	Co	Cu	As	Sn	Sb	W	Hg	Pb	Notes
Ground1	rear under right hand	none	<LOD	956	45,621	44,354	44,581	229,435	146	2212	<LOD	<LOD	26	182	114	27	<LOD	32	Possible error/contamination of surface
Ground2	rear under left hand	none	<LOD	10,110	96,215	<LOD	3412	89,451	41	2518	<LOD	26	11	206	<LOD	<LOD	<LOD	15	
Ground3	rear left bottom palla	none	<LOD	8056	75,236	<LOD	2105	86,416	148	2133	<LOD	<LOD	13	153	55	<LOD	<LOD	34	
AVE				6374	72,357	44,354	16,699	135,101	112	2288		26	17	180	85	27		27	
STD DEV				4803	25,420		24,155	81,710	61	203			8	27	42			10	
	HAIR																		
1	right hair lock	beige	<LOD	<LOD	29,864	62,885	55,801	218,027	492	2848	<LOD	<LOD	23	225	<LOD	57	<LOD	92	>P, S, Ca, Mn, W
2	right hair lock	blackish	<LOD	<LOD	64,396	58,248	38,384	187,432	405	2473	<LOD	57	48	219	<LOD	<LOD	<LOD	128	>P, S, Mn; Trace As, Pb
	BONNET/HEADBAND																		
5	bonnet right side	black	<LOD	<LOD	46,777	78,981	32,010	210,070	242	1873	63	34	24	323	<LOD	<LOD	13	125	>P, S, Ca, Mn, Co, Sn; Trace As, Hg, Pb
3	right headband	blackish	<LOD	<LOD	38,700	84,481	29,961	216,423	233	1472	28	15	36	169	<LOD	<LOD	<LOD	44	>P, S, Ca, Mn; Trace Co, As
	SKIN																		
4	right under jaw	none	<LOD	9033	75,971	13,827	25,584	185,764	301	3237	<LOD	<LOD	19	<LOD	<LOD	<LOD	<LOD	70	>Al, S, Ca, Mn, Fe; trace Pb
6	neck right of torc	brown	<LOD	<LOD	141,017	9759	7244	107,694	305	4572	102	<LOD	48	381	281	58	<LOD	61	>Si, Mn, Fe, Co, Sn, Sb; trace As, W
20	left hand knuckle	black	<LOD	6022	113,684	6645	10,311	185,224	402	8494	<LOD	34	43	267	<LOD	<LOD	<LOD	143	>Al, Si, Ca, Mn, Fe; +Pb; Trace As
22	inner ear	pink	<LOD	<LOD	23,856	40,626	34,604	164,308	428	2400	<LOD	35	<LOD	586	92	<LOD	21	91	>S, Mn, Sn, Hg; Trace Pb
	PALLA																		
7	palla right shoulder	brown	<LOD	2907	71,574	39,213	19,692	191,746	427	3995	<LOD	73	49	144	100	<LOD	<LOD	44	>Ca, Mn, Fe, Cu; Trace As
18	palla top near belt	crusty	<LOD	2609	69,016	14,283	4816	259,886	68	3116	38	<LOD	34	90	97	<LOD	<LOD	81	>Ca, Fe; Trace Co, Pb, As
19	palla waist	shaded	<LOD	<LOD	53,403	38,398	9559	160,044	511	3746	<LOD	22	<LOD	90	<LOD	<LOD	14	96	>Mn, Fe; Trace Hg, Pb
23	palla centre		<LOD	<LOD	46,722	24,169	26,612	166,085	107	3568	<LOD	59	25	261	<LOD	<LOD	<LOD	64	>S, Fe; Trace Co, Trace As
24	palla bottom right	orange	<LOD	<LOD	77,972	31,901	10,132	172,840	167	2911	<LOD	19	25	102	<LOD	<LOD	13	60	
25	palla bottom right	green/ yellow	<LOD	2883	77,963	51,584	8325	193,884	320	3032	<LOD	19	44	213	<LOD	<LOD	<LOD	52	>P, Ca, Mn, Fe; Trace As
26	palla left fold	yellow	<LOD	<LOD	35,039	53,464	14,998	228,275	524	3767	<LOD	<LOD	27	292	144	<LOD	10	31	>P, Ca, Mn, Fe; Trace As, Hg
27	palla top of left fold	shaded	<LOD	5222	44,405	61,641	17,608	190,817	927	10,684	<LOD	65	70	289	<LOD	40	<LOD	58	>P, Mn, Fe, Cu; Trace As, W
	PEPLOS																		
8	peplos fold top right	yellow	<LOD	<LOD	43,962	19,649	6710	250,237	159	2872	<LOD	25	25	138	<LOD	29	<LOD	52	>Ca
11	peplos shaded fold	yellow	<LOD	2126	51,461	55,329	17,128	177,668	259	9992	99	<LOD	34	377	<LOD	<LOD	<LOD	54	>P, Mn, Fe, Co, Sn; Trace As
12	peplos left fold		<LOD	<LOD	49,020	40,737	16,245	224,429	450	4211	<LOD	<LOD	19	165	<LOD	24	<LOD	37	>Ca, Mn, Fe
	TUNIC																		
14	tunic cuff	green	10,190	<LOD	95,243	57,132	25,446	182,985	379	12,963	<LOD	8093	46	<LOD	86	<LOD	<LOD	352	>Mg, Si, P, S, Ca, Mn, Fe, Cu, Pb; Trace As
15	tunic right bust	green	<LOD	<LOD	88,961	62,174	20,402	165,439	312	9548	<LOD	4669	<LOD	315	<LOD	24	12	93	>Si, P, Mn, Fe, Cu, Sn; trace Hg
16	tunic right bust	green	<LOD	<LOD	53,503	86,809	22,414	200,705	307	8774	<LOD	5103	126	79	<LOD	<LOD	15	1009	>P, Ca, Mn, Fe, Cu, As, Pb; trace Hg, Pb
17	tunic under arm sleeve	green	<LOD	1221	88,292	53,723	28,984	171,607	229	10,005	<LOD	6392	24	326	<LOD	<LOD	<LOD	89	>Si, P, S, Mn, Fe, Cu, Sn; trace Pb
28	tunic left cuff	green	<LOD	3889	66,489	41,828	20,572	155,918	278	12,605	<LOD	9372	28	303	<LOD	<LOD	<LOD	122	>Mn, Fe, Cu, Sn, Pb
	FIBULA																		
9	left of central fibula	red	<LOD	3168	31,178	597	3256	244,870	101	5494	<LOD	<LOD	8	201	<LOD	<LOD	<LOD	55	>Ca, Fe
10	centre of central fibula	yellowish	<LOD	1120	35,788	50,927	23,480	241,036	90	3011	<LOD	37	14	385	82	<LOD	<LOD	69	>P, Ca, Fe, Sn; trace Pb
	RINGS																		
21	left hand ring?		8857	3324	43,066	53,577	7426	263,852	208	4600	<LOD	12	62	163	<LOD	<LOD	<LOD	188	>Mg, P, Ca, Mn, Fe, Pb
29	right hand ring		<LOD	<LOD	78,262	26,982	28,209	186,287	314	4347	<LOD	20	28	145	<LOD	<LOD	<LOD	46	>S, Ca, Mn, Fe
	BELT/LOWER PALLA																		
13	centre	red	<LOD	<LOD	79,194	56,318	19,817	203,871	463	3596	<LOD	26	76	86	77	<LOD	<LOD	108	>P, Ca, Mn, Fe, Pb; Trace As

3.2. Portable Optical Microscopy

Surface examination at the visible and microscopic level of surviving pigments is fundamental for providing a comprehensive review of their condition and for revealing similarities or differences between painted features. In situ digital microphotography of multiple features where pigments were identified was captured using a portable optical microscope—a Dino-Lite Edge Digital Microscope (AM4515ZT, Dino-Lite, New Taipei City, Taiwan) 20–220× magnification, flexible LED control (FLC), integrated adjustable polarizer, Automatic Magnification Reading (AMR), and a 1.3 Megapixel Edge sensor. This provided powerful high-resolution images and with colour depth of eight-bit using the DinoCapture 2.0 software, before being exported as JPEGs.

3.3. Multispectral Imaging

In the short timescales permissible for this work, it was not possible to undertake a comprehensive programme of multispectral imaging (MSI) [49–52] to detect pigments that may remain invisible to the naked eye. It was, however, possible to capture visible induced infrared luminescence (VIL) images using a specially customised Canon EOS 2000d camera with a 58 mm area CMOS sensor lacking an ultraviolet and infrared cutting filter sensitive to the spectral range of ~350–1000 nm wavelength with a 18–55 mm focal lens fitted with Midopt LP830 filter (Midopt, Pallatine, IL, USA) and Canon Speedlite 580EX Shoe Mount Xenon Flash (Canon, Tokyo, Japan) fitted with a non-fluorescent diffuser and Hoya UV/IR cut-off filter (Hoya, Tokyo, Japan). Post-processing of acquired images was conducted using IDCube Lite 1.0 following the protocol established by CHARISMA [49].

3.4. Microsamples

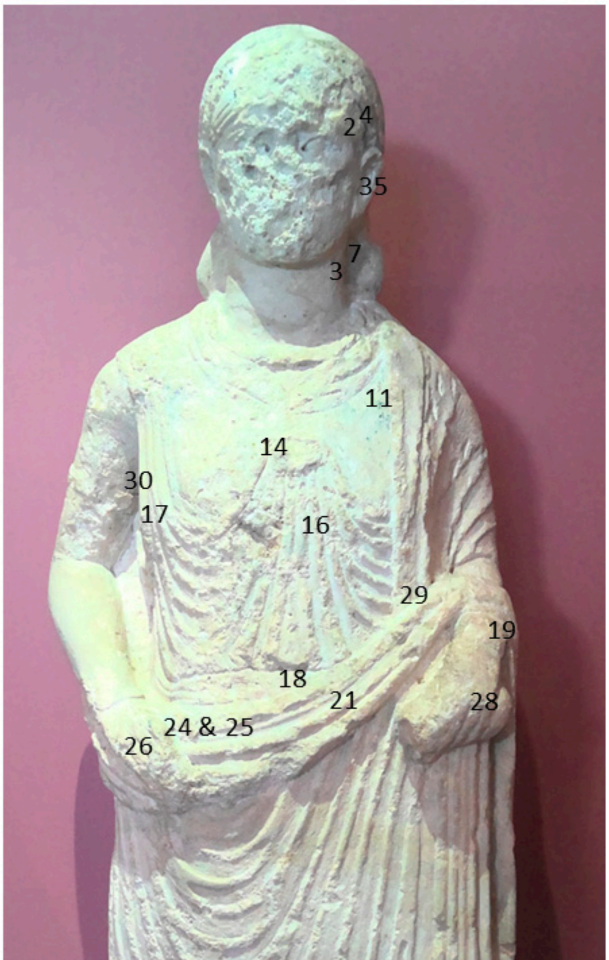
Microsamples were collected from nine areas where a reasonable amount of pigment was visibly extant by scraping with a scalpel and sealing the samples in labelled vials. The samples were later studied under an Olympus BX53 upright microscope (Olympus Corporation, Tokyo, Japan) with incident LED light and images were captured using an LM digital SLR adapter connected to a Canon EOS 2000D. These were supplemented by images of the microsamples captured on a Dino-Lite Edge for comparison to images taken in situ by the Dino-Lite (above).

4. Results

4.1. Non-Invasive Analysis Results

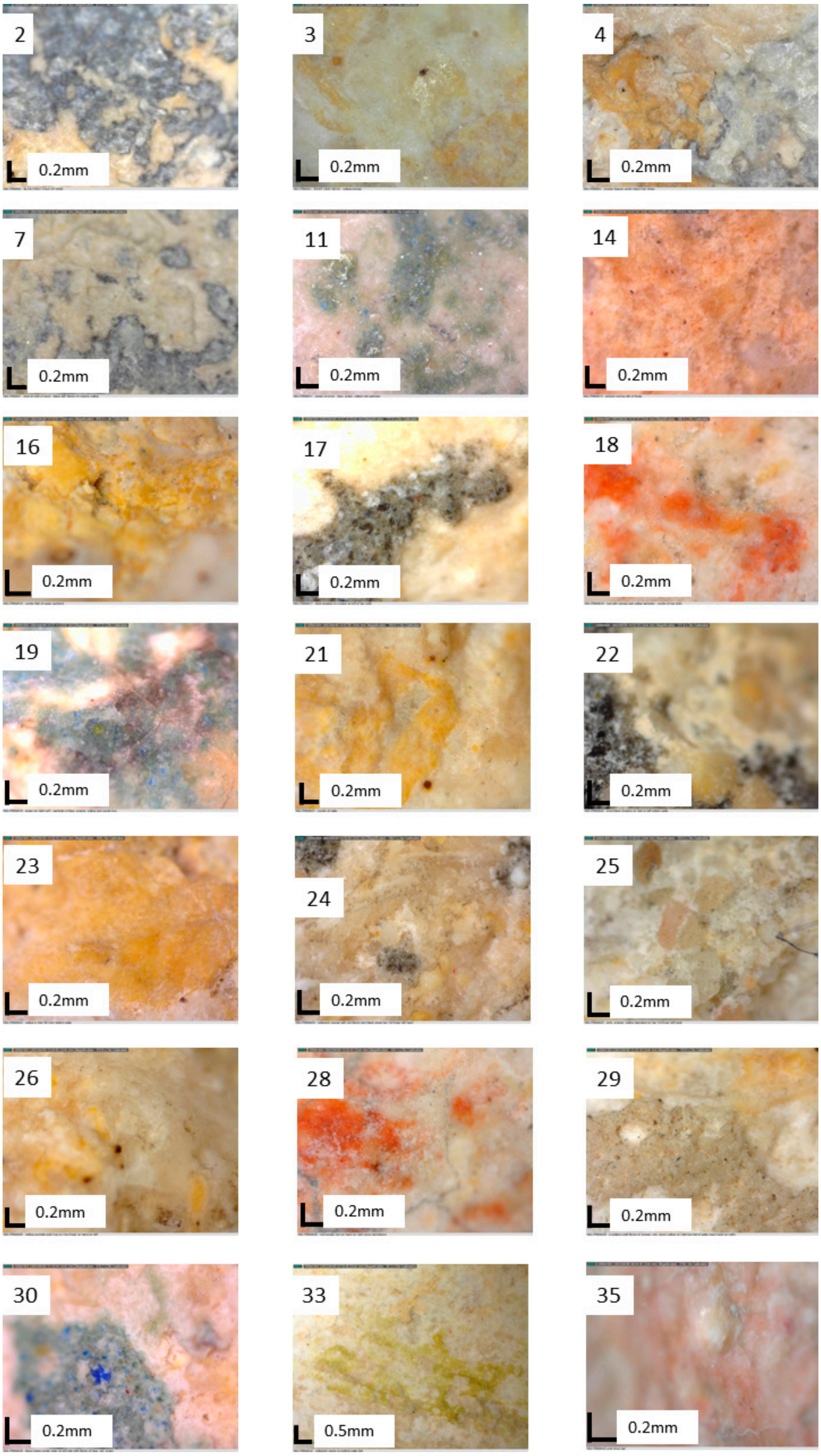
4.1.1. Microphotographs

Microphotographs were captured of features retaining extant pigment or surface treatments (Figure 8 and Table 1). Some traces survived extremely well (Figure 9), but many presented with a whitish crustation in the polychrome matrix. The source of this is likely salination products commonly found on limestone sculpture [53] or other natural products resulting from its calcareous character, but this could not be confirmed in situ since the Geochem2 mode of the Vanta instrument does not detect Na. Most research on this topic revolves around desalination by conservators rather than the process of salination or limestone by-products that can interfere with extant pigments and other surface treatments and, consequently, their detection by non-destructive analytical instruments. This is an aspect that will be addressed through ongoing research.



(**a**)

Figure 9. *Cont*.



(**b**)

Figure 9. (**a**) Locations of microphotograph of surviving pigments on various features. (**b**) Microphotographs.

4.1.2. pXRF

Of the 29 analysis spots (Figure 10), these could be categorised into eight sculpted and painted features for ease of comparison. These include hair, bonnet/hairband, skin, *palla*, *peplos*, tunic, fibula, ring, and belt-like feature/lower *palla* (Table 2). The results confirm, as anticipated from the microphotography, a complex mixture of pigments present across all features. They further support the results of earlier spot sampling through SEM [6]. The calcium carbonate may derive from the limestone raw material and high levels of S and Ca on many features suggests a gypsum preparatory layer applied in advance of pigment; alternatively, it could be derived from a secondary product of degradation. That cannot be confirmed in these preliminary results since the samples have not been cross-sectioned and processed through SEM/EDS in this first phase of the work; these are planned for the next phase of the research, but the initial SEM/XRD unpublished report suggests the presence of a mixture of bone ash, silicates, and gypsum in samples.

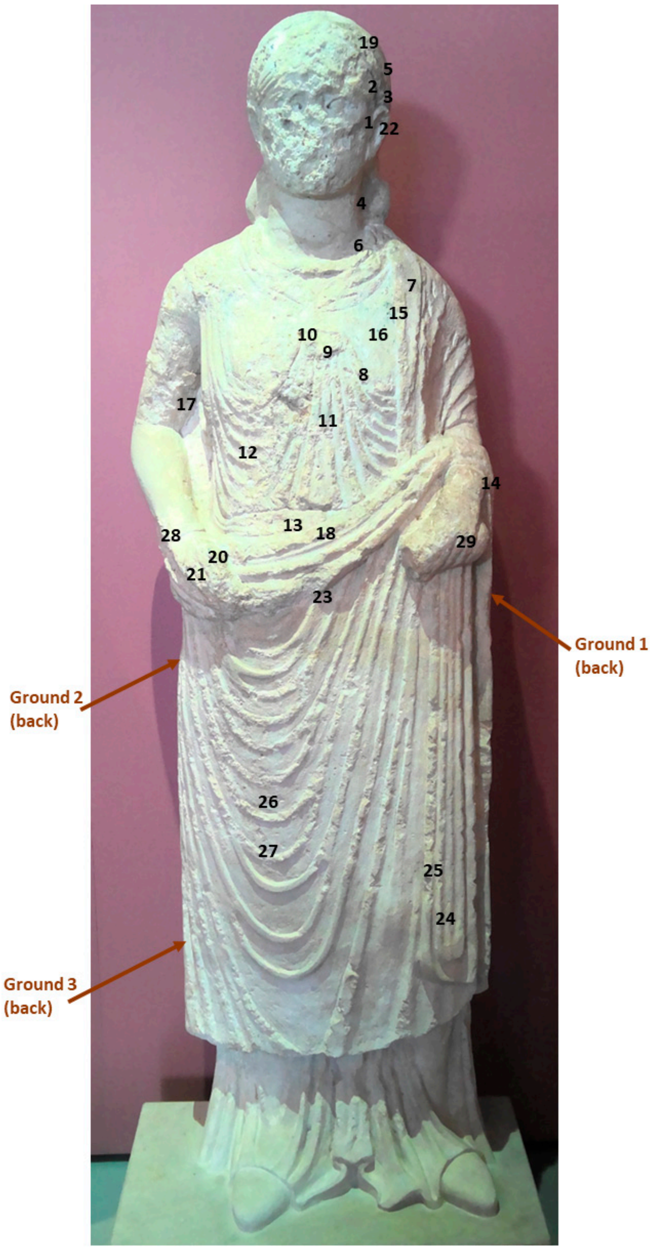


Figure 10. pXRF analysis locations indicated by numbers.

4.1.3. Multispectral Imaging (MSI)

In the limited timescales, it was not possible in this phase of the research to undertake a comprehensive programme of MSI. Given the reported presence of "Egyptian green", the decision was taken to prioritise visible induced infrared luminescence (VIL) imaging to verify that hypothesis since this technique is excellent for non-destructively detecting the presence of Egyptian blue, even in very low quantities. Images were captured from the four areas of the tunic where a vibrant blue/green colour is visibly extant and the results incontrovertibly confirm that Egyptian blue is, indeed, present in all three (the front left and right of chest, left cuff, and right underarm), Figure 11.

Figure 11. VIL images of areas where Egyptian blue survives.

4.2. Sample Analysis

Microsamples

Given the diversity of pigment mixing and layering present, the decision was taken to carefully extract microsamples from nine areas where polychromy visibly survives (Figure 12 and Table 1). These provide a clear visual representation of stratigraphic layers and mixtures present and are good comparanda to the in situ photographs (Figure 13). Micro-sampling has provided exceptional information on technological practices deployed for the polychromy, specifically that layering and mixing are present in most of the samples analysed. Although SEM/EDS and FTIR-ATR falls outside the remit of this paper, a future programme of work will undertake comprehensive cross-section analysis to ascertain the components of each stratigraphic layer for all three sculptures.

Figure 12. Microsample locations, numbered M1–M9.

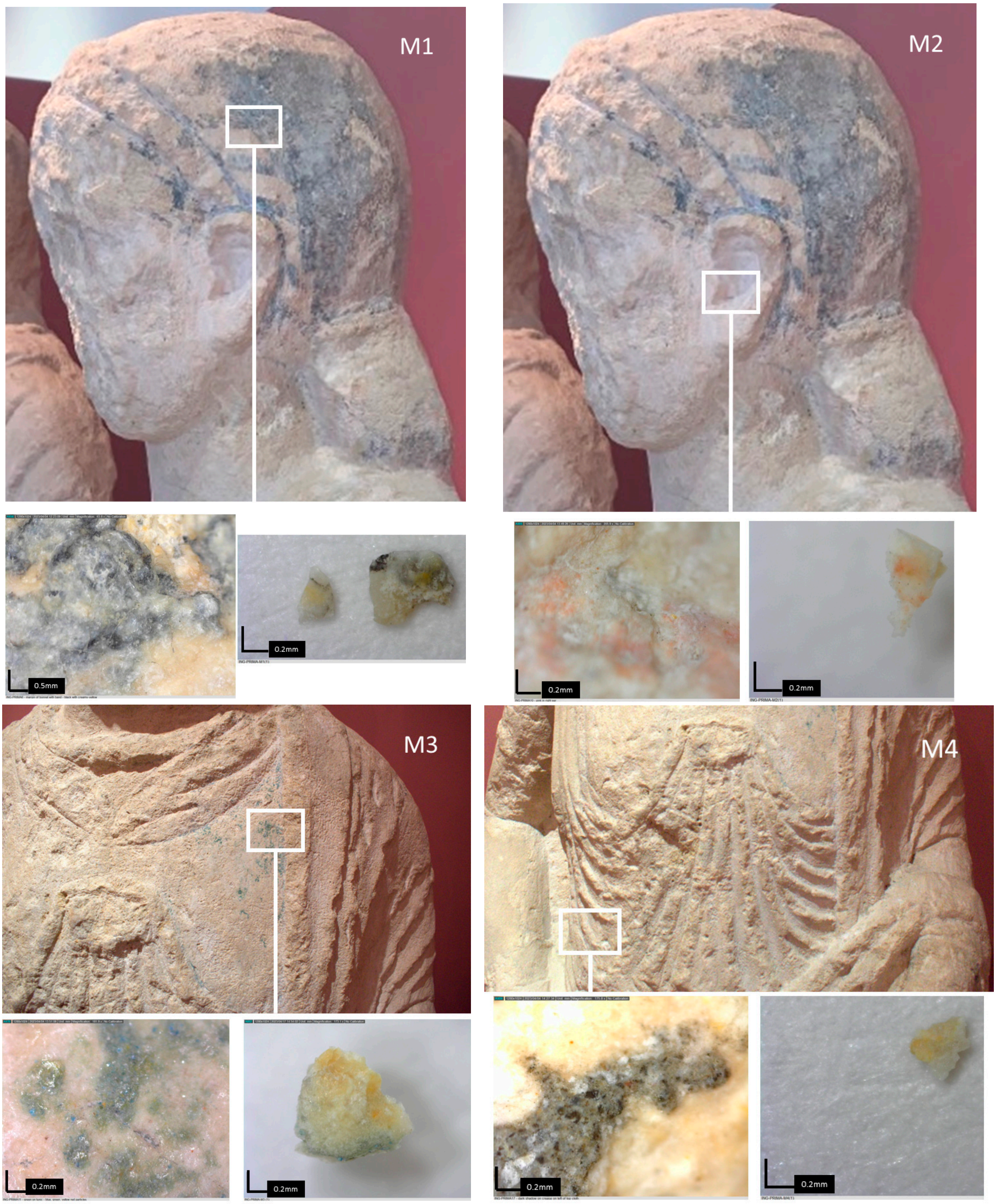


Figure 13. *Cont.*

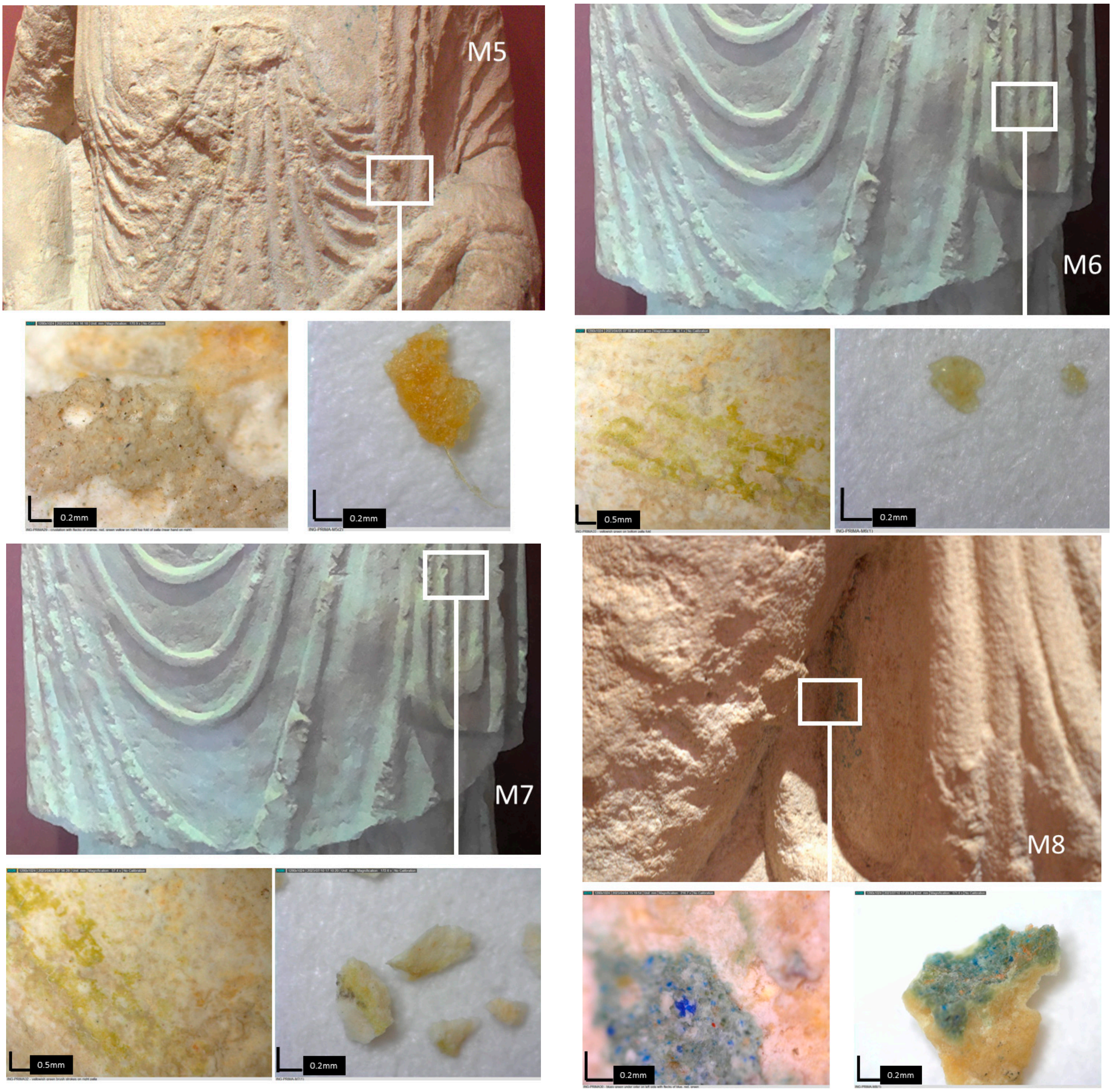


Figure 13. *Cont*.

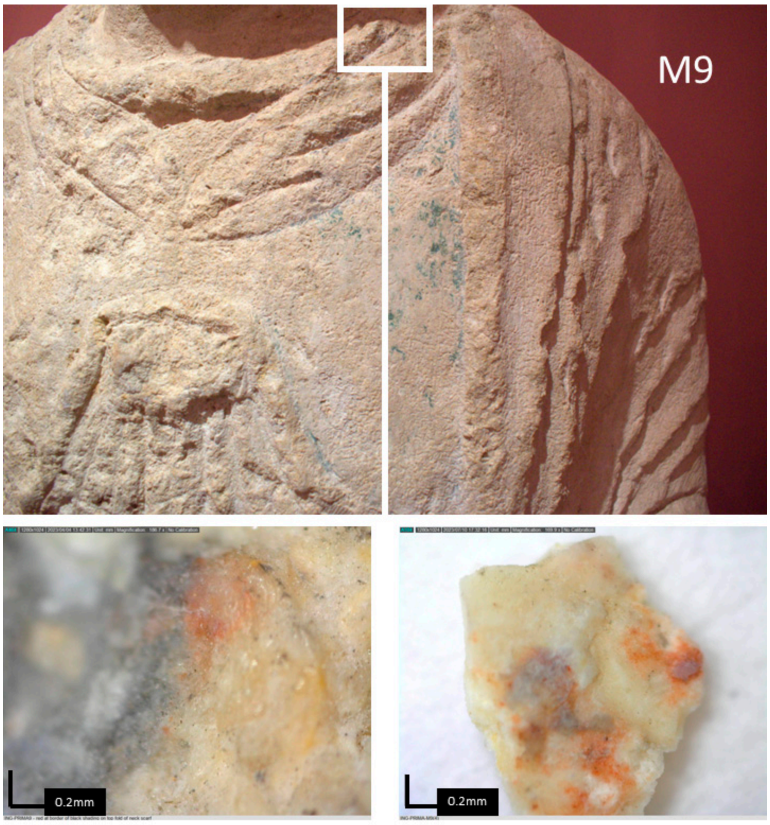


Figure 13. Comparative microphotographs of in situ polychromy and microsamples numbered M1–M9.

5. Discussion

As expected after the initial visual inspection, a complex mixture of pigments appears to be confirmed as having been used to create the diverse and vibrant polychromy on this sculpture and the surviving traces have provided a wealth of information from which it is possible to hypothesise a reconstructed palette of colour (Table 3). These should be taken as preliminary results based on summative elemental compositions using only pXRF; more comprehensive information will be available in a planned publication reporting on a second phase of research, including cross-sections, SEM/EDS, and FTIR.

It is beyond dispute that the complexities present in these mixtures and layers created subtle tones for different features. In other words, these were not blocks of primary colours applied in one episode of painting, but the work of a highly skilled artisan who was clearly intimately familiar with their raw materials. Their expertise is articulated through a combination of techniques, including mixing different amounts of selected pigments on the palette to achieve the hue required for their particular purpose combined with the layering of materials to create depth to these hues in the same way that contemporary artists continue to practice. These methods ingrained naturalism into the work and brought the sculpture to vibrant life as a 3D interactive monument that can be engaged with on a very personal level. It must surely have been a remarkable spectacle to audiences in Antiquity and this research permits the digital reconstruction of those pigments to recreate their vibrancy through various media for contemporary audiences in the museumscape.

Table 3. Palette of pigments.

Feature	Colour	Elements Detected by pXRF	Possible Compound/s	Suggested Pigment/s
Hair	Mid-dark brown	Elevated P, Ca	Hydroxyapatite: $Ca_5(PO_4)_3(OH)$	Bone black
		Elevated As	Arsenic(II) sulfide: As_4S_4/Arsenic(II) sulfide: As_2S_3	Realgar/Orpiment
		Elevated Pb	Dilead(II) lead(IV) oxide: Pb_3O_4 OR Basic lead carbonate $2PbCO_3{\cdot}Pb(OH)_2$	Minium OR Lead White
Bonnet	Black	Elevated P, Ca	Hydroxyapatite: $Ca_5(PO_4)_3(OH)$	Bone black
		Traces Hg	Mercuric Sulfide: HgS	Cinnabar
		Traces Pb	Dilead(II) lead(IV) oxide: Pb_3O_4 OR Basic lead carbonate $2PbCO_3{\cdot}Pb(OH)_2$	Minium (poss from hair below?) OR Lead White
		Traces Co, Sn	cobalt(II)stannate: $CoSnO_3$	Cerulean blue? (poss conservation product?)
Skin	Flesh tone	Elevated Fe	Iron Oxyhydroxide: FeO(OH)	Yellow ochre AND/OR
			Iron(III) oxide chromophore: Fe_2O_3 + clay + silica	Red ochre
		Elevated Al and Fe	Celadonite or Glauconite: $K[(Al,Fe^{III}), (Fe^{II},Mg)](AlSi_3,Si_4)\ O_{10}(OH)_2$	Green earth
		Elevated Si, Trace Co	sodium-aluminium-silicate-sulfate: $Na_7Al_6Si_6O_{24}S_3$	Lapis Lazuli/Ultramarine blue
		Traces As	Arsenic(II) sulfide: As_4S_4	Realgar/Orpiment
		Traces Pb	Dilead(II) lead(IV) oxide: Pb_3O_4 OR Basic lead carbonate $2PbCO_3{\cdot}Pb(OH)_2$	Minium OR Lead White
Palla	Yellowish-green	Elevated Fe, Mn	$Fe_2O_3\ MnO_2\ nH_2O$ + Si+ Al_2O_3	Brown ochre
		Elevated Fe	Iron Oxyhydroxide: FeO(OH)	Yellow ochre (shaded areas)
		Trace As	Arsenic(II) sulfide: As_2S_3	Realgar/Orpiment
		Elevated P, Ca	Hydroxyapatite: $Ca_5(PO_4)_3(OH)$	Bone black (shaded areas)
Peplos		Elevated Fe, Trace Mn	Iron Oxyhydroxide: FeO(OH)	Yellow ochre
		Elevated As	Arsenic(II) sulfide: As_2S_3	Realgar/Orpiment
		Elevated P	Hydroxyapatite: $Ca_5(PO_4)_3(OH)$	Bone Black (shaded fold)
Tunic	Green/Blue	Elevated Si, Cu, Ca	Copper calcium silicate: $CaCuSi_4O_{10}$	Egyptian blue
		Elevated P, Ca	Hydroxyapatite: $Ca_5(PO_4)_3(OH)$	Bone black
		Elevated Fe, Trace Mn	Iron Oxyhydroxide: FeO(OH)	Yellow ochre AND/OR
			Iron(III) oxide chromophore: Fe_2O_3 + clay + silica	Red ochre
		Elevated Pb	Dilead(II) lead(IV) oxide: Pb_3O_4 OR Basic lead carbonate $2PbCO_3{\cdot}Pb(OH)_2$	Minium OR Lead White
		Traces As	Arsenic(II) sulfide: As_2S_3	Realgar/Orpiment
		Traces Hg	Mercuric Sulfide: HgS	Cinnabar
		Elevated Mg	Celadonite or Glauconite: $K[(Al,Fe^{III}), (Fe^{II},Mg)](AlSi_3,Si_4)\ O_{10}(OH)_2$	Green earth (from skin at cuff)
Fibula	Red centre Yellow (right)	Elevated Fe	Iron(III) oxide chromophore: Fe_2O_3 + clay + silica	Red ochre
		Elevated P, Ca	Hydroxyapatite: $Ca_5(PO_4)_3(OH)$	Bone Black (shaded fold)
		Elevated Sn		Unidentified source
Rings	?	Elevated P, Ca	Hydroxyapatite: $Ca_5(PO_4)_3(OH)$	Bone black
		Elevated Pb	Dilead(II) lead(IV) oxide: Pb_3O_4 OR Basic lead carbonate $2PbCO_3{\cdot}Pb(OH)_2$	Minium? OR Lead White
		Elevated Mg	Celadonite or Glauconite: $K[(Al,Fe^{III}), (Fe^{II},Mg)](AlSi_3,Si_4)\ O_{10}(OH)_2$	Green earth (from skin)
Belt feature/lower palla	Red	Elevated Fe, Mn	Iron(III) oxide chromophore: Fe_2O_3 + clay + silica	Red ochre
		Elevated P, Ca	Hydroxyapatite: $Ca_5(PO_4)_3(OH)$	Bone black
		Elevated As	Arsenic(II) sulfide: As_4S_4	Realgar/Orpiment
		Elevated Pb	Dilead(II) lead(IV) oxide: Pb_3O_4 OR Basic lead carbonate $2PbCO_3{\cdot}Pb(OH)_2$	Minium OR Lead White

These inherent complexities have made it challenging to extrapolate the exact mixtures used to articulate each feature, but the combination of in situ microscopy, laboratory-based light microscopy of microsamples, pXRF, and MSI imaging have provided an unprecedented level of information to inform the digital reconstruction, for the first time, of polychromy on a Roman sculpture from the German *Limes* region. These preliminary findings serve as a platform for a more comprehensive programme of analysis of the microsamples, including cross-section analysis, SEM-EDS, FTIR-ATR, and Raman spectroscopy.

Although preliminary, some of the results are as expected insofar as the statue appears to have been covered in a gypsum preparatory layer and ochres used to create skin-tone along with green Earth [4]. Although not widespread across the sculpture, the results suggest small amounts of more expensive, and less easy to acquire in frontier contexts, pigments in some of the mixtures. These include minium (red lead) or lead white and realgar as well as possibly lapis lazuli used in small amounts added to the skin tones and in other features. Although uncommon in Roman contexts, lapis lazuli is a well attested pigment to the ancients and in use from the 7th–5th C BCE [37,38,50] and tests using this instrument on samples of lapis lazuli by the author confirm an elemental composition of high Al, Si, Ca, and Fe. It is, therefore, reasonable to conclude its presence in this palette based on the results, though more clarity will be provided in the next phase of research using a broader suite of analytical techniques.

Traces of cinnabar are also present in the bonnet which, although it presents as a flat black colour, is given tonal depth by the combination of bone black with small amounts of cinnabar, minium, or lead white and cerulean blue. The latter is the closest elemental match but as a more recently patented pigment after the 1860s, likely derives from modern intervention such as a conservation product or a contaminant from earlier storage conditions. There is no record of such intervention with which to verify this hypothesis, but a later period of repainting during, for example, the early medieval period cannot be discounted or confirmed. The mid-dark brown hair was also created with bone black mixed with realgar or orpiment, minium, or lead white and brown ochre to provide tone and depth.

Traces of cinnabar were also present in the tunic colourant which was created through the most complex and diverse mixture of all the polychromed features. This vibrant blue-green is articulated through a combination of Egyptian blue, bone black, red, and yellow ochres, lead white or minium, orpiment, cinnabar, and green Earth. Orpiment, ochres, and bone black created the different shades of yellow and yellow-green in the *peplos* and *palla*, respectively, and ochres depict different colours on the jewellery, probably defining jewels embedded into the metalwork (rings and fibula), as well as the red belt-like feature/lower *palla*, to which minium, realgar, and bone black was also added.

Perhaps the greatest surprise was the revelation that bone black is widely mixed across so much of the palette to darken and create different tonal effects and, most particularly, used as an agent for shading some of the carved features. For example, microphotography combined with pXRF results irrefutably confirm that sculpted folds of the *peplos* and *palla* are given additional depth through shading with a bone black mixture, which appears in some features to be mixed in a semi-transparent, possibly resinous, substance (see microphotographs 17 and 22). This is an extremely innovative practice that has recently been identified on the belt of a Minerva sculpture from Pannonia [4] where a thin layer of carbon black and ochre overlaid Egyptian blue enhanced the sculpted feature.

Only the application of these materials science techniques could detect such subtleties. Previous assumptions have inclined toward the carved features, creating sufficient depth to highlight them to the viewer. Pliny recorded that burnt yellow ochre, sandyx, quenched with vinegar or brown ochre, umber, was a technique employed by artists to create shadows in wall paintings [54] (pp. 20–23). To this, we can now confidently add the application of bone black mixtures to enhance the articulation of depth through shadows on carved features on statuary. FTIR will help identify the resinous matrix this pigment was mixed with to create the appropriate shading effect in the next phase of this research.

6. Conclusions

This exploratory research has broken new ground in significantly expanding the potential repertoire of vibrant polychromy practiced on Roman sculpture from the provinces as far as the very margins of the Empire on the German *Limes*. Having proposed a previously unrecorded palette of pigments applied to Roman sculptures, it is clear there is much scope for ongoing research into the polychrome phenomenon in these unique cultural contexts. It is our intention to continue our collaborative exploration into other, similarly underexplored sculptures, sculpted reliefs, and inscribed texts from across frontier contexts.

Supplementary Materials: The following supporting information can be downloaded at: https://www.mdpi.com/article/10.3390/heritage7030080/s1: File S1: Full pXRF results.

Author Contributions: C.H. provided the historical and archaeological content that contextualises the statues under discussion, wrote the original draft text relating to those aspects, and captured most of the photographs of sculptures and features. L.C. reviewed, edited, and supplemented original text, contributed the abstract, discussion, and conclusion, and undertook and reported on all the scientific analysis, including in situ microphotography, microsampling, pXRF analysis, and multispectral imaging. All authors have read and agreed to the published version of the manuscript.

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Data Availability Statement: The datasets used and analysed during the current study are available from the authors on reasonable request.

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