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Behind the light – **specificities of the materialities of a sixteenth century illuminated antiphonary housed in the Biblioteca Pública de Évora**[•]

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Abstract:

This work focuses on the material characterization of a sixteenth century illuminated antiphonary housed in the Biblioteca Pública de Évora (BPE) – the Manizola 116c. Based on several optical and spectroscopic techniques, namely optical microscopy (OM), scanning electron microscopy coupled with energy dispersive X-ray spectroscopy (SEM-EDS), micro-Fourier transform infrared spectroscopy (μ-FTIR), Fibre-Optic Reflectance Spectroscopy (FORS) in the ultraviolet and visible range (UV-Vis) and handheld-energy dispersive X-ray fluorescence (h-EDXRF), it was possible to identify the use of different azurite ground on different degrees of fineness to produce a range of blue hues throughout the manuscript as well as some differences in some of the paints' compositions suggesting the presence of two different *hands* for producing the illuminations. Digital images of the illuminations acquired with different angles for the light source revealed very interesting features concerning the painting technique used to produce these illuminations and with what could have been the intention of the illuminator to influence the relation between the reader and the manuscript.

Keywords: Antiphonary, manuscript, illuminations, gilding, FORS, μ-FTIR, SEM-EDS, h-EDXRF

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Resumo:

Este trabalho baseia-se na caracterização de algumas das especificidades materiais presentes num antifonário iluminado do século XVI à guarda da Biblioteca Pública de Évora (BPE) – o Códice Manizola 116c. Da análise com recurso a várias técnicas de microscopia e espectroscopia, nomeadamente a microscopia óptica (OM), microscopia eletrónica de varrimento com um sistema de microanálise por espectroscopia de raios-X por dispersão de energias acoplado (SEM-EDS), micro-espectroscopia de infravermelhos com transformada de Fourier (μ-FTIR), espectroscopia de reflectância (FORS) no ultravioleta-visível (UV-Vis) e fluorescência de raios-X por dispersão de energia portátil (h-EDXRF), foi possível identificar a utilização de diferentes granulometrias de azurite para produzir diferentes tons de azul ao longo do manuscrito, assim como diferenças nas composições de algumas tintas, sugerindo a presença de duas mãos diferentes para a produção das iluminuras. A aquisição de imagens digitais com diferentes ângulos de iluminação revelou características muito interessantes no que diz respeito à técnica de pintura utilizada para produzir as iluminuras e com o que poderia ter sido a intenção do iluminador de influenciar a relação entre o leitor e o manuscrito.

Palavras-chave: antifonário, manuscrito, iluminuras, douramento, FORS, m-FTIR, SEM-EDS, h-EDXRF

Introduction

An antiphonary is a type of book used during the liturgy, which contains the song portions of the Divine Office. As a choir book, it tends to be of a large size, as it should be read with ease from distance by all the elements of the choir (BROWN, 1994; HENRIQUES, 2014). The antiphonary of the Manizola collection held by the Biblioteca Pública de Évora (BPE) coted as Manizola 116c, measures ca. 550 mm x 390 mm and is bound in embossed leather-covered wooden boards, Figure 1.

Figure 1 - The Manizola 116c, *ff.*52v-53r (folium dimensions: 515 mm x 365 mm)

Images © BPE and HERCULES Lab.

Dated from the 16th century, the Manizola 116c was part of the inventory of the Portuguese female Cistercian monastery of São Bento de Cástris, located just outside Évora walls, at the time of its closure in 1890. At this time, the *armarium* of manuscripts comprised Books of Gospels and *Collationes*, collections of texts of private use of some of the nuns, and manuscripts of sacred music (CONDE and SILVA, 2015). From the set of manuscripts of sacred music, the Choir Books comprised seven antiphonaries, one antiphonary *Sanctorale*, one *Hymanrium*, two Graduals and two Books of *Invitatórios* (CONDE and SILVA, 2015; ORFEUS project, 2013). From the set of antiphonaries from São Bento de Cástris, the Manizola 116c stands out for the magnificence of its illuminations. For this reason, the Manizola 116c was selected from the set of manuscripts from the São Bento de Cástris *armarium* to be the first manuscript to be analysed following an analytical approach. There are no references for the place of production of this manuscript. However, its presence in the *corpus* of the library of the female Cistercian monastery of São Bento de Cástris, together with the fact that one of its miniatures represents Saint Bernard of Clairvaux (*f.*4r) – the founding abbot of the Cistercian Abbey of Clairvaux, suggest that it might have been produced for São Bento de Cástris (CONDE and SILVA, 2015), figure 2. Funded in 1274 by D. Urraca Ximenes, the São Bento de Cástris Monastery is the oldest female monastery in southern Portugal, which officially became part of the Cistercian Order in 1278.

Figure 2 - Isolate depictions in Manizola 116c: *left*, the representation of Infant Jesus in His Majesty (*f.*1r); *right*, representation of Saint Bernard of Clairvaux (*f.*4r)

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The Manizola 116c contains 62 folia, although the analysis of the full text and the quires of the book suggest that the manuscript might have been composed of 65 folia (JACOBS, 2016). From the 122 miniatures present in this antiphonary, only two are isolated depictions – the representation of Infant Jesus in His Majesty, related to Christmas day (*f.*1r), and the above-mentioned representation of Saint Bernard of Clairvaux (*f.*4r), figure 2. The remaining 121 miniatures are all illuminated capital letters. Besides the antiphons and responsories, with its related plainsong and other religious songs, the Manizola 116c contains an adding at the end related to the Office of the Dead (CONDE and SILVA, 2015).

This work presents some of the specificities found from the material characterization of the illuminations of Manizola 116c, namely those found for the use of different blue shades for the backgrounds of some of the capital letters present throughout the manuscript, figure 3; and the specificities behind one of the capital letters which stood out for the lack of movement, of three-dimensionality and of bright, so much present in the remain capital letters from Manizola 116c, figure 4.

Figure 3 - The use of different blue shades' backgrounds in the Capital letters from Manizola 116c: ***left, f.*****7r;** ***right, f.*****33r**

Figure 4 - Capital letters from Manizola 116c with gold paint backgrounds, reflecting the different painting technique used to produce the capital letters. ***Left, f.*****13v;** ***right, f.*****37v**

For this, a selection of microscopic and spectroscopic techniques was used, namely optical microscopy (OM), scanning electron microscopy coupled with energy dispersive X-ray spectroscopy (SEM-EDS), micro-Fourier transform infrared spectroscopy (μ-FTIR), Fibre-Optic Reflectance Spectroscopy (FORS) in the ultraviolet and visible range (UV-Vis) and handheld-energy dispersive X-ray fluorescence (h-EDXRF). The acquisition of digital images of the illuminations using different angles for the light source revealed interesting features concerning the painting technique used to produce these illuminations.

Materials and Methods

Digital images with two different light source angles (peripheral illumination and light source placed 45º to the surface of the miniatures) were acquired to infer on the effect of the gilding technique over the readability of the miniatures. OM was used to characterize the drawing and painting techniques used to produce the illuminations, as well as to micro-sampling some of the coloured paints. To infer on the similarities between paint colours with a same colour-hue, colorimetric measurements were made. SEM-EDS and h-EDXRF were used for the analysis of the gilding alloy. μ-FTIR and FORS to characterize the blue chromophores.

Digital photography: a Nikon DX300 digital camera was used for image acquisition and a commercial visible light source was used for illuminating, either by placing it peripherally to the illumination or 45º positioned to the surface of the illumination.

Optical microscopy: a Leica M205C stereomicroscope, with a zoom range of 7.8x to 160x, equipped with a Leica DFC295 camera and external illumination by optical fibres was used for magnified observation of the paint layers.

Colorimetry: colorimetric measurements were performed using a portable Datacolor Check II Plus spectrophotometer, with a diffuse illumination 8º viewing, a pulsed xenon light source, a spectral range of 360-700 nm, an effective bandwidth of 10 nm, and a bandwidth of 2 nm. The equipment was calibrated with white and black calibration surfaces used as standards. Analysis were made with measuring times <2.5 seconds with an aperture spot size of 3 mm. Each surface was analysed 3 times to create an average and identify variations within the painted surfaces.

Sampling: micro-sampling was performed in lacunas with a micro chisel from Ted Pella micro tools (micro-samples ranging between 20 μm and 50 μm) under a LEICA M205C stereomicroscope, with a zoom range of 7.8x to 160x, equipped with a Leica DFC295 camera and an external illumination by optical fibres.

SEM-EDS: analyses were performed with a scanning electron microscope HITACHI 3700N coupled to an energy dispersive X-ray spectrometer Brüker Xflash 5010. The analyses were made at 20 kV with a pressure of 40 Pa in the chamber.

μ-FTIR: an infrared spectrometer Brüker Hyperion 3000 equipped with a single point MCT detector cooled with liquid nitrogen and a 15x objective lens was used. The spectra were collected in transmission mode, in 50–100 μm areas, using a S.T. Japan diamond anvil compression cell. The infrared spectra were acquired with a spectral resolution of 4 cm^{-1}, 32 scans, in the 4000-650 cm^{-1} region.

FORS: analyses were made using an ASEQ Instruments LR1-T v.2 compact spectrometer, with a spectral range of 300-1000 nm and a spectral resolution of <1 nm (with 50 μm slit). Measurements were taken using the ASEQ CheckTR software. Calibration was made using Whatman filter paper. Samples were analysed at an exposure of 100-200 ms, 5 scans, and a BoxCar smoothing of 15. Each spot was measured three times for 5 seconds each.

h-EDXRF: a Tracer III-SD Fluorescence handheld EDXRF spectrometer (BRUKER) equipped with a 10 mm2 XFlash® SDD, a peltier cooled detector with a typical resolution of 145 eV at 100,000 cps, a Rh target and a maximum voltage of 40 kV was used. Analyses were made at 40 kV and 11 μA, without filter, an acquisition time of 30 seconds, and a spot size of 12 mm2 (3 mm x 4mm). The instrument was set up on a tripod and positioned approximately 2–3 mm away from the surface under analysis. EDXRF spectra were always acquired from three contiguous spots to evaluate the reproducibility of the results. All EDXRF spectra were acquired using the S1PXRF software and analysed using the ARTAX software. To evaluate the influence of the support over EDXRF results, the parchment of each folium was also analysed, and the results taken into account.

Results

From the 121 illuminated capital letters of Manizola 116c, besides the common vegetable and zoomorphic representations, those of anthropomorphic motifs stand out for their eloquence and movement, figure 5.

Figure 5 - Vegetable (a, *f.*9v), zoomorphic (b, *f.*13v) and anthropomorphic (c, *f.*13v) motifs in the Manizola 116c

Images © BPE and HERCULES Lab.

Besides this movement effect, the illuminator marked his illuminations with a three-dimensional effect mainly achieved by the use of a drawing technique produced with lighter hues for the drawings and on the effect of light on the gildings (section 3.1). Purposely, or not, different blue hues were found for the blue backgrounds of some capital letters, reflecting the use of different blue shades along Manizola 116c (section 3.2). On the contrary, at a first glance, gold paints presented similar hues along the manuscript (section 3.3).

Drawing techniques and its effect on the illuminations' readability

The images of Manizola 116c are highly sophisticated, as for every miniature (even for the simplest illuminated capital letter) there is a sense of three-dimensionality produced through two main techniques: the use of lighter hues (enhancing the three-dimensional effect of the drawing) and the use of gildings (emphasising the three-dimensional effect with the angle of the light source).

The use of lighter hues for three-dimensional effect on the drawings

The use of lighter hues for producing a three-dimensional effect on the drawing was a common painting technique at the time. The Manizola 116c presents in its illuminations the two most common techniques used for producing a three-dimensional drawing effect at the time: the use of lighter hues, produced by mixing lead white to the pure base paint, over the base paint (figure 6a); and the use of an intermediate hue for the background, over which the pure paint (with a darker hue) was applied for the contour, while lead white was applied, as a pure colour paint, for the lights (figure 6b).

Figure 6 - Magnified images of the use of lighter hues for the paint's lightening in *f.*13v (a) and *f.*9v (b) of Manizola 116c

Images © BPE and HERCULES Lab.

The effect of light on the gilding

The observation of the illuminations with different angles for the light source highlighted a very interesting feature of these miniatures: if with peripheral illumination the miniature shows a three-dimensional effect based mainly on the use of lighter hues as a drawing technique (figures 7a1, 7b1 and 7c1), with a source of illumination placed at 45º, the three-dimensional effect is enhanced due to the reflexion of light by the gilding details present in the illuminations (figures 7a2, 7b2 and 7c2).

Figure 7 - The effect of light on the gilding of *f.*13v (a), *f.*7r (b) and *f.*33r (c): peripheral lightening (a1, b1, and c1); 45º lightening (a2, b2 and c2)

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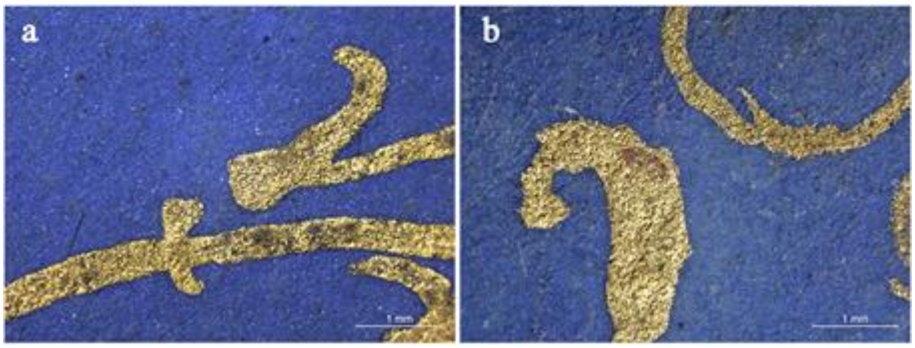
Considering that these books were commonly placed in a lectern positioned in the centre of the choir-stall to be visible to all singers, and that during daytime the light might enter the room from windows placed in lateral walls, the use of these drawing techniques strongly enhanced the magnificence of these miniatures, especially when the angle of the incoming light was such that it was reflected by the gilded surfaces. It is also possible that artificial light could also be positioned in such way to enhance the richness of the manuscript. In this sense, the Manizola 116c is, perhaps, one of the best examples of the term «illumination» – from the Latin *illuminare*, 'to enlighten or illuminate' (BROWN, 1994) – commonly used to describe the embellishment of a manuscript with luminous colours. In this case, the magnificence of colour was further enhanced by the application of gilding details, increasing the three-dimensional effect and shine of the miniatures.

Blue shades in Manizola 116c

Another interesting feature of Manizola 116c illuminations is the range of blue shadings from illumination to illumination. Figures 7b, 7c and 8 displays one of the interesting ranges of blue shades found in this manuscript.

Figure 8 - Magnified image of the blue paints' surfaces in *f*.7r (b) and *f*.33r (c) - full size images are presented in Figure 5



Images © BPE and HERCULES Lab.

CIELAB colour measurements of the blue paints from *ff*.7r and 33r were taken to infer on which colour parameter could be inducing this visual colour change. From the analysis of table 1, it is possible to verify that more than the change on the lightness of the colour (ΔL^*) and on the green-red hue (Δa^*), it is the yellow-blue hue (Δb^*) that differs more in these paints.

Table 1 - CIELAB colour coordinates[#] for blue paints of Manizola 116c (*ff*.7r and 33r)

	f.7r	f.33r	$\Delta_{L^*/a^*/b^*}$	Δ_E
L*	37.83 ± 2.06	33.61 ± 1.29	4,83	
a*	-7.30 ± 0.62	-9.33 ± 0.64	2,06	11,92
b*	-24.69 ± 2.68	-13.73 ±1.49	10,96	

[#] The L* coordinate represents the *lightness*; the a* coordinate the *green-red* component and b* the *blue-yellow* component.

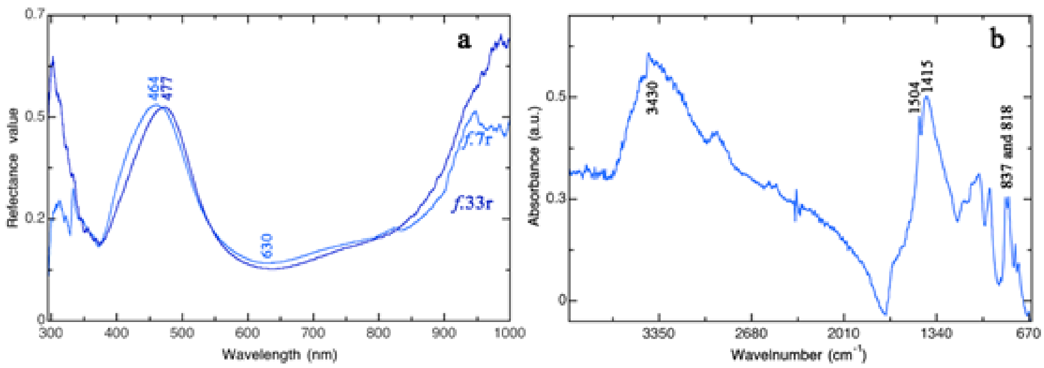
To evaluate the visual impact of the colour change between both blue colour-paints, colour difference, ΔE, was determined as:

$$\Delta E = \sqrt{(L^*_{f.7r} - L^*_{f.33r})^2 + (a^*_{f.7r} - a^*_{f.33r})^2 + (b^*_{f.7r} - b^*_{f.33r})^2}$$

By measuring the distance between both blue colour-paints in the colour space, it turns possible to evaluate the amount of colour change, although no indication of the direction in which colours differ is provided. Considering that the visual threshold allowing an observer to note the colour difference is at least 3 CIELAB units, the changes in colour between both blue paints is highly noticeable by eye ($\Delta_E=11.92>3$, table 1), (WITZEL, 1973; CEBALLOS et al., 2003). In this sense, it is possible to state that the visual colour differences found between both blue paints are strongly due to a yellowish of the blue paint (as the value of b* is the one that varies in a more pronounced way, which corresponds to a shift from the blue to the yellow component), when compared with the blue paint from *f*.33r which present a more deep-blue hue.

FORS analysis of both blue paints suggests the use of the same colour source: azurite, a blue basic copper carbonate ($2CuCO_3.Cu(OH)_2$), based on its characteristic reflectance band with a maximum at 464 nm for *f*.7r and 477 nm for *f*.33r, and a maximum absorption band at *ca.* 630 nm related to the d-d transitions of copper, figure 9, (ACETO et al., 2014).

Figure 9 - *Left*, UV-Vis spectra of blue paints in *ff*.7r and 33r; *right*, infrared spectrum of the blue paint in *f*.7r.



It is interesting to verify that for *f*.33r there is a shift of the reflectance maximum for higher values of wavelength (477 nm for *f*.33r while 464 nm was observed for *f*.7r). This shift of 13 nm is in agreement with the colorimetric results, which reflected a shift to the yellow region. This variation in the yellow component might be due to the different paint layer thickness, as the CIELAB colour measurements of thicker layers will be less influenced by the yellowing parchment, or to variations in how coarsely the pigment was ground, as a paint layer made with a coarser pigment might not cover so well the parchment and, thus, reflect more the yellowish of the parchment support.

μ-FTIR analysis of a blue paint micro-sample (samples size between 20 μm and 50 μm) collected from *f.*7r allowed to identify only the presence of azurite through its characteristic absorption bands ascribed to the bending modes of carbonate $\delta(CO_3^{2-})$ at 837 and 818 cm^{-1}, the two ν_3 asymmetric modes of the carbonate at 1504 and 1415 cm^{-1}, and the stretching vibration of the hydroxyl unit $\nu(OH)$ at 3430 cm^{-1}, corroborating the results of FORS analysis, figure 9, (FROST et al., 2007).

Gold alloys in Manizola 116c

Three different applications of gold paints were found in Manizola 116c: in light effects on capital letters, in backgrounds of capital letters and in full-painted capital letters, figure 10.

Figure 10 - Normalized size images of capital letters representing the three different applications of gold paints in Manizola 116c: light effects in capital letters (*ff*. 7r and 33r), backgrounds of capital letters (*ff*. 37v and 41v), and in full-painted capital letters (*ff*. 50r and 53v)

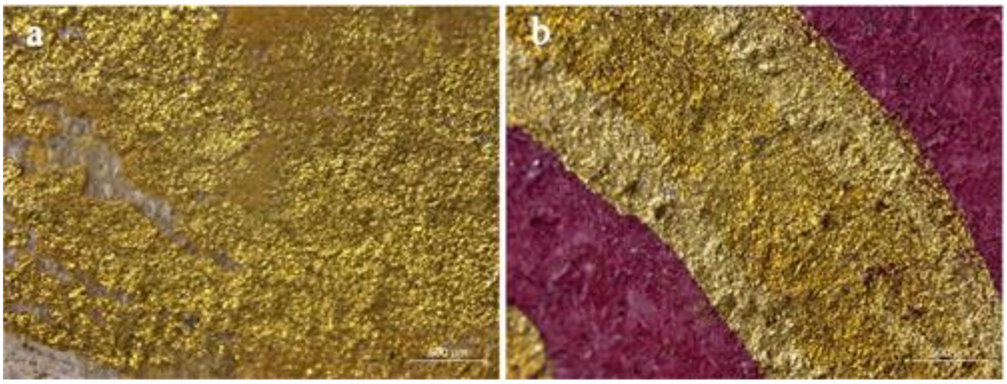
EDXRF results of the gold paints from *ff.*7r, 33r, 37v, 41v, 50r and 53v (see figure 8) revealed the presence of a gold (Au)-copper (Cu) alloy, with the exception to *f.*37v where, besides Au and Cu, silver (Ag) was also found in a small amount, table 2.

Table 2 - Chemical elements identified by h-EDXRF in some of the gold paints in Manizola 116c

Gold application	folium	Chemical elements
Light effect in a capital letter	7r	Au, Cu
	33r	Au, Cu
Background of a capital letter	37v	Au, Cu, Ag
	41v	Au, Cu
Capital letter	50r	Au, Cu
	53v	Au, Cu

When observed with or without magnification, gold paints present similar hues and similar surfaces' morphologies. Figure 11 presents two magnified images of the gold paints from *f*.37v - in which, from the six selected gold paints application, h-EDXRF identified the use of a different gold-alloy (table 2) – and from the gold paint used for painting the full capital letter present in *f*.50r, were this similarities between hues and similar surfaces' morphologies are evidenced.

Figure 11 - Magnified images of gold paints in Manizola 116c: a) applied as a background in *f*.37v; b) used for painting the full capital letter present in *f*.50r (b)



Images © BPE and HERCULES Lab.

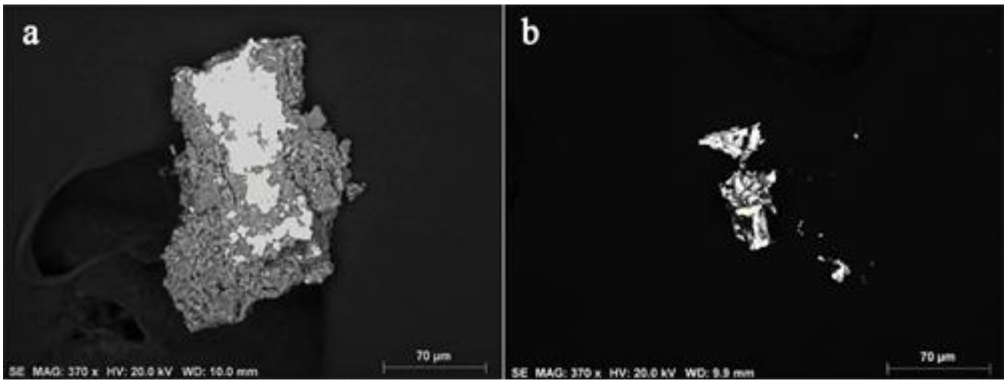
SEM-EDS analysis of two micro-samples from these gold paints (*ff*.37v and 50r) corroborated the use of two different gold alloys, table 3. Despite both samples present the same composition in gold (97%, %wt), it was interesting to verify that for f.37v, silver is the second element in the chemical composition (2%, %wt) with copper present in a residual amount.

Table 3 - Gold alloy composition (%wt) of gold paints in *ff*.37v and 50r, determined by SEM-EDS analysis

	f.37v	**f.50r**
Au	97	97
Cu	1	3
Ag	2	-

The use of gold alloys either with copper or with copper and silver was a common procedure for metalwork's' production, as a way to overpass the characteristic softness of gold. From these two elements, gold-copper alloys are the most frequently found, displaying characteristic reddish-gold hues according to the copper compositions (higher concentrations of copper leads to reddish alloys). For paler hues, silver was commonly added to the composition. Although related to metalwork's production, these gold alloys were also used to produce very thin leafs for gilding in paints, whereas gold pigment was produced by grinding these leafs (BIDARRA et al., 2009). In illuminations, gildings were commonly applied either as leafs or in powder, being the last the easiest to be applied (DONI et al., 2014; MERCURI et al., 2017). SEM backscattered images of two micro-samples of gilding paints from *f*.37v and *f*.50r evidenced the use of gold in powder to produce these gildings, figure 12.

Figure 12 - SEM backscattered images of micro-samples of gilding paints from a) *f*.37v and b) *f*.50r, evidencing the use of powder gold for the gildings



Despite these results correspond to a single micro-sample from each gold paint present in *ff*.37v and 50r, h-EDXRF analysis were performed in several spots of analysis and each spot analysed three times. The results reflected similar Au:Cu and Au:Cu:Ag intensity ratios, suggesting a similarity in the gold composition along the paints for both folia. In this sense, with such a small difference for gold-alloy compositions determined by SEM-EDS analysis, there is no clear explanation for the use of a different gold alloy to produce a single background of a capital letter in this Codex, as for one hand it does not correspond to a more economical solution (the content in gold remains the same), and for the other hand, there is no specific iconographic value that would justify the use of a different gold alloy or the intervention of a more or less skilled illuminator to produce the capital letter present in *f*.37v. Also, from the set of capital illuminations of Manizola 116c, the one present in *f*.37v stands out for its resemblance to the *pastel* technique, with less bright, less sense of three-dimensionality and with a drawing technique used for the figurative motif standing out for its naïveness, figure 4. It is likely that a different hand produced this capital letter, someone who tried to follow a similar iconographic programme using similar materials, but which could not withstand the scrutiny of a detailed observation and molecular analysis, standing out from the other capital letters of Manizola 116c.

Conclusions

The study of the materialities of Manizola 116c highlighted some very interesting features, allowing to infer more on the importance of this manuscript in the context of the Antiphonaries' production. The effect of light on the gilding and on the miniatures' readability became one of the most interesting findings of this research, providing the manuscript with an improved sense of three-dimensionality that would depend both the placement of the light source and the placement of the reader. Regarding the differences on the blue paints' hue, it was interesting to note how this difference is not attributed to differences in paint compositions, but to the ink thicknesses and how coarsely was the blue pigment azurite in these paints. On the other hand, the material analysis of gold paints present in Manizola 116c allowed to verify a difference on the gold alloy used to produce the gold background of the capital letter present in *f*.37v, which already stood out from all the capital letters of this manuscript for its painting technique. This difference might be attributed to the intervention of a second illuminator solely to produce this capital letter. Why? At his point, we may not provide any possible answer, only to encourage further iconographic studies concerning the capital letter of f.37v in the context of the entire manuscript, to infer on the possible reasons of a different hand for its production. Within these results, it becomes evident the importance of materials' characterization for better contextualize an illuminated manuscript in its history and in its relationship with the reader, reflecting that *behind the light* much can be known and understood.

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