Microchemical Journal 155 (2020) 104582

Contents lists available at ScienceDirect

Microchemical Journal

journal homepage: www.elsevier.com/locate/microc

An applied complementary use of macro X-ray fluorescence scanning and multi-light reflectance imaging to study Medieval Illuminated Manuscripts. The Rijmbijbel of Jacob van Maerlant

L. Watteeuw[a,*], M. Van Bos[b], T. Gersten[c], B. Vandermeulen[d], H. Hameeuw[d]

[a] *KU Leuven, Book Heritage Lab, Illuminare, Centre for the Study of Medieval Art, Charles Debériotstraat 26, 3000 Leuven, Belgium*
[b] *Royal Institute for Cultural Heritage, KIK-IRPA, Jubelpark 1, 1000 Brussels, Belgium*
[c] *Royal Library of Belgium, Conservation Department, Keizerslaan 4, 1000 Brussels, Belgium*
[d] *KU Leuven, Imaging Lab, KU Leuven Libraries, Ladeuzeplein 21, 3000 Leuven, Belgium*

ARTICLE INFO

Keywords:
MA-XRF
Photometric stereo
Multispectral microdome
Multi-light reflectance
Medieval manuscript illuminations
Rijmbijbel of Jacob van Maerlant

ABSTRACT

The colorful images in medieval manuscripts have striking characteristics that need meticulous approaches during art technical research in situ, as no sampling is permitted. Visual inspections and images with the microscope can reveal details about techniques and layers of the miniature. In this experimental research we applied two complementary techniques to reveal the pigments the artists used to create the folios of a famous Flemish illuminated manuscript, the Rijmbijbel of Jacob van Maerlant (13th century, Bruges). This essay emphasizes on the combined possibilities and advantages of two different approaches. It focuses on exploring the same illuminations with MA-XRF analyses in a lab environment and the multi-light reflectance technique (MLR) of the Multi-Spectral Microdome (MS MicroD) in situ. Through comparing and combining MA-XRF analysis with the MS MicroD datasets reference data, the ink and paint layer compositions could be characterized. The essay illustrates the capabilities of both techniques to obtain new insights in the material composition of the paint of a well-described and examined artefact. The genuine 13th century materials and the 19th century retouching could be visualized, differentiated and determined. Finally, the added value of the combination of the two examination methods is evaluated for art technical research on illuminated manuscripts.

1. Introduction

Since the introduction of high performant XRF scanning of panel paintings, excellent analytical results on pigment analyses are obtained on detailed and delicate artifacts such as medieval miniatures, giving far more results than the limited information of spot measurements [1–8]. The development and testing of the Multi-Spectral Microdome (MS MicroD, KU Leuven) with the multi-light reflectance (MLR) photometric stereo imaging in 2016 provided a supplementary approach to study the materials and techniques of manuscript illuminations [9,10,12–15,21]. Art technical research on high value illuminated manuscripts is time consuming, so the *modus operandi* is an important factor to take into consideration when selecting or combining research infrastructure and strategies. This essay explores the combined possibilities, advantages and limitations of those two different approaches. It explores the simultaneous MA-XRF analyses and reflectance imaging of the illuminations with the Multi-Spectral Microdome *in situ*. Through comparing and combining MA-XRF analysis with the MS MicroD datasets reference data, the ink and paint layer compositions could be characterized.

This setup was chosen for a unique and rare, never studied before illuminated manuscript: the Rijmbijbel of Jacob van Maerlant, a renowned illuminated 13th century manuscript, considered as the oldest illustrated manuscript in the Dutch language (Brussels, KBR, Ms 15001, *Rijmbijbel van Jacob van Maerlant*, parchment, Flanders, *c.* 1285) [16]. The remarkable bible in verse was created in the surroundings of Bruges, and was one of the earliest and richest illuminated copies of Maerlands poetic translation from the Latin to Middle-Dutch. Although famous to medievalists, art historians and philologist, the Rijmbijbel has never been examined in detail. The conservation campaign in 2018 in the Royal Library of Belgium was the stimulus to undertake this experimental research.

2. Instrumentation

2.1. The Multi-Spectral Microdome

The Multi-Spectral Microdome (MS MicroD an acquisition device of

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[*] Corresponding author.

https://doi.org/10.1016/j.microc.2019.104582
Received 29 August 2019; Received in revised form 26 December 2019; Accepted 26 December 2019

the Portable Light Dome system, PLD) of KU Leuven operates five different sets of narrowband LEDs (LED Engin) emitting in five different parts of the electromagnetic spectrum evenly spread on the inside of a hemisphere (Ø 30 cm): UV: LZ1-00UV00 (λ_D 365 nm), Blue: LZ1-30DB00 (λ_D 460 nm), Green: LZ1-30G100 (λ_D 523 nm), Red LZ1-30R100 (λ_D 623 nm) and NIR: LZ1-30R400 (λ_D 850 nm). The light sensor mounted on top is an Allied Vision Prosilica monochrome GX 6600, high-resolution 35 mm CCD (6576 × 4384pixels) with electronic shutter, spectral response between 350–1000 nm. The lens is a Jenoptik inc. CoastalOpt UV–VIS-IR 60 mm Apo Macro (i.e. calcium fluoride lens elements enabling true apochromatic performance: 315–1100 nm), this eliminates focus shift during the all-in-one recording sequence across all applied spectral bands assuring results in register and preventing hotspots [12].

After capturing 228 different images of one and the same object lit from 228 different angles (i.e. 44 angles for blue; 47 for green; 41 for NIR; 48 for red; 44 for UV), the processing calculates for each of these five sets a multi-layered image dataset, based on a robust *photometric stereo* algorithm. When uploaded in the stand-alone PLD viewing interface (v7.0.06) the imaged surface can be virtually relit interactively from different angles revealing minute details. In addition, on each of the five sets filters can be applied: color, shaded (without color), sketch (accentuating the indentations) and normal maps (2D visualization of the relief). By combining the results of the five sets standardized trichromatic false color images can be generated on the fly (R-G-B, NIR-R-G, NIR-G-B, R-G-UV, G-B-UV). Apart from that, the geometric and calculated reflective information can be combined and mixed within the PLD viewer interface, e.g. viewing the IR spectral data in combination with the texture information based on the UV normal image. For every point on the surface metric data (x-y-z measurements) can be derived and for every defined pixel the spectral albedo reflection intensities can be mapped. The latter give the potential to describe and classify materials with these same datasets: bidirectional reflectance distribution function (BRDF), i.e. future work.

Through the combination of these tools implemented in the PLD viewer interface, the dataset after recording and processing can virtually visualize the original as it appears to the human eye (visual observation), can visualize the original as it appears in the NIR/UV (beyond visual), and it permits the characterization of the documented materials. The datasets also allow spectral histograms and spectral reflection maps (SRM) to be generated for any selected area surface or individual pixel. Histograms plot the distribution of the calculated spectral albedo responses for each of the spectral bands used in the MS MicroD (NIR: dark red plot; R: orange plot; G: green plot; B: light blue plot; UV: dark blue plot): the x-axis gives the height of the spectral albedo response, the y-axis the probability distribution for each of the spectral components for the selected area; SRM generate and visualize the observed intensities of the spectral albedo reflectance per pixel by the emitted light for each LED as they are mounted on the inside of the MS PLD hemisphere. In the PLD viewer interface these can be compared to measurements calculated on a reference collection of known historical pigments (the Pigments Checker charts v.2.1, binder: Arabic gum and v5, binder: acrylic by CHSOS were used). When the recording settings are kept identical and the measurements can be made on pure samples these tools can be used to identify similar pigments and materials [12]. For previous preliminary results on (illuminated) manuscripts with the MS MicroD see [13–15]. All MS MicroD recordings were performed in the dark to eliminate pollution by ambient light.

2.2. MA-XRF maps

MA-XRF maps were registered using the M6 Jetstream (Bruker AXS, Berlin, Germany) with a Rh X-ray tube operated at 50 kV and 600 µA current. In the M6, the X-ray beam size is defined by poly-capillary optics and is determined by the distance between the measuring head and the object. Because of the undulation of the parchment folios, a spotsize of 150 µm was chosen, allowing to scan the illuminations in a save way. A step size of 125 µm and a dwell time per step of 15 ms was used. The spectra were collected, deconvoluted and examined with the Bruker M6 Jetstream software. Chemical elements were identified in the scan by examining the sum spectrum and maximum pixel spectra. Additional data treatment was done using Datamuncher and PyMca [17].

During MA-XRF analysis, the folio to be analyzed are installed in a parallel horizontal direction with the moving X-ray head. With the use of ‘book display cushions’ and flexible weights (so called ‘book snakes’), the manuscript is carefully positioned. In order to minimize as much as possible the movement of the examined parchment folio, part of the surface is covered by acid free paper, slightly pressed with a weight, as parchment is a highly hygroscopic organic material and could react with the environment. Moreover, a ‘buffer’ of ± 6 mm acid free board is used to partly block the X-rays coming from ink or paint on underlying folio's. This buffer blocks the low energy X-rays but is not efficient for blocking high energy X-rays. And the X-rays generated by ink or pigment on the verso side of the folio cannot be blocked at all and are both clearly visible on the image.

3. Results and discussion

3.1. Defining the 13th century illuminators pallet

Although there is limited reference in analytical research of 13th century manuscripts in the geographical area of Flanders, visual observations have established that the palette for manuscript painting in the Low Countries is limited. To contextualize the obtained research results with the MA-XRF and MS MicroD, the production of text and image on a parchment folio has to be taken in consideration. To understand in full the artefact and the obtained data, the sequences of production have to be determined. Four craftsmen are involved in the production of an illuminated codex: the scribe, the flourisher of the initials, the gilder and the illuminator, working in successive stages at the manuscript. The scribe was responsible for the ruled folio and writing the text (working with a metal point and a quill), the flourisher worked with the colors red and blue (with a quill), de gilder had gesso and gold foil (brush and knife). Finally, the illuminator had a set of brushes and circa nine colors made from different pigments on his table [18,19]. Below, we define the corpus of those genuine medieval materials for each actor, in the second section the later ‘modern’ materials by the 19th century restorer are pinpointed, using the same combination of research tools.

The data of the imaging and analyses for defining the 13th century pallet were brought together to obtain a comprehensive view of the materials of the genuine illuminations of the Rijmbijbel by analyzing the illumination on folio 9recto (Appendix A: slide 7), representing a chapter of Genesis (9: 20–23) with the story of the naked drunk Moses in the vineyard, being clothed by his two sons, the third haunting, is depicted on a golden ground in combination with a colored pen drawing of a goat eating branches.

Based on the combined results the following interpretations of the paint materials on the selected manuscript folio can be argued. We sum them up in the proper sequence of the production scheme.

The scribe - the dark ruling lines and the dark ink of the main text are executed using different materials. Ruling lines are drawn with a lead point (Fig. 1c, Pb-distribution). The visually dark brownish appearing ink of the main text has a very high reflectiveness in infrared and shows with the MS MicroD a similar spectral histogram as pure iron gall ink on a reference patch (Fig. 2a). MA-XRF analysis confirms the presence of iron in the ink.

The flourisher - For the blue penwork two compositions of paint are found alternating: a part of the blue lines is done using azurite ($Cu_3(CO_3)_2(OH)_2$) while other parts have a more greenish tint and are based on copper and iron (Fig. 1, Cu- and Fe-distribution and Appendix

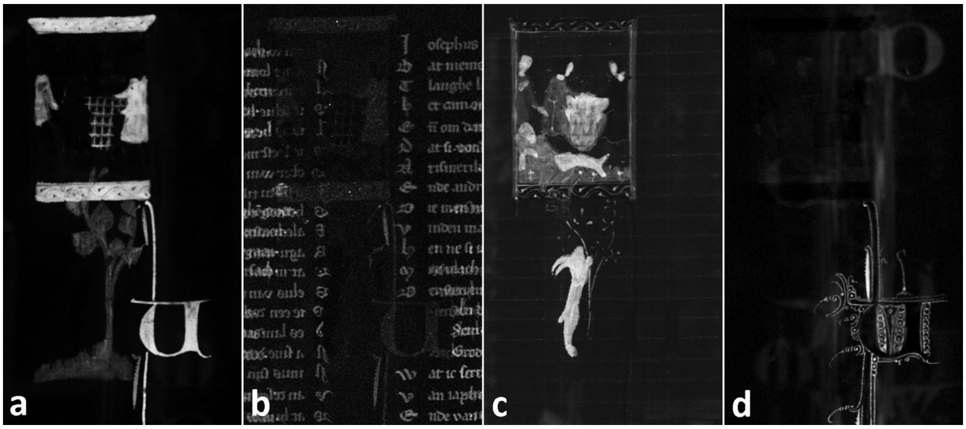

Fig. 1. Elemental distribution maps of the MA-XRF scanning and analysis results of a selected area on folio 9r: (a) copper (Cu-Kα), (b) iron (Fe-Kα), (c) lead (Pb-Lα), (d) mercury (Hg-Lα).

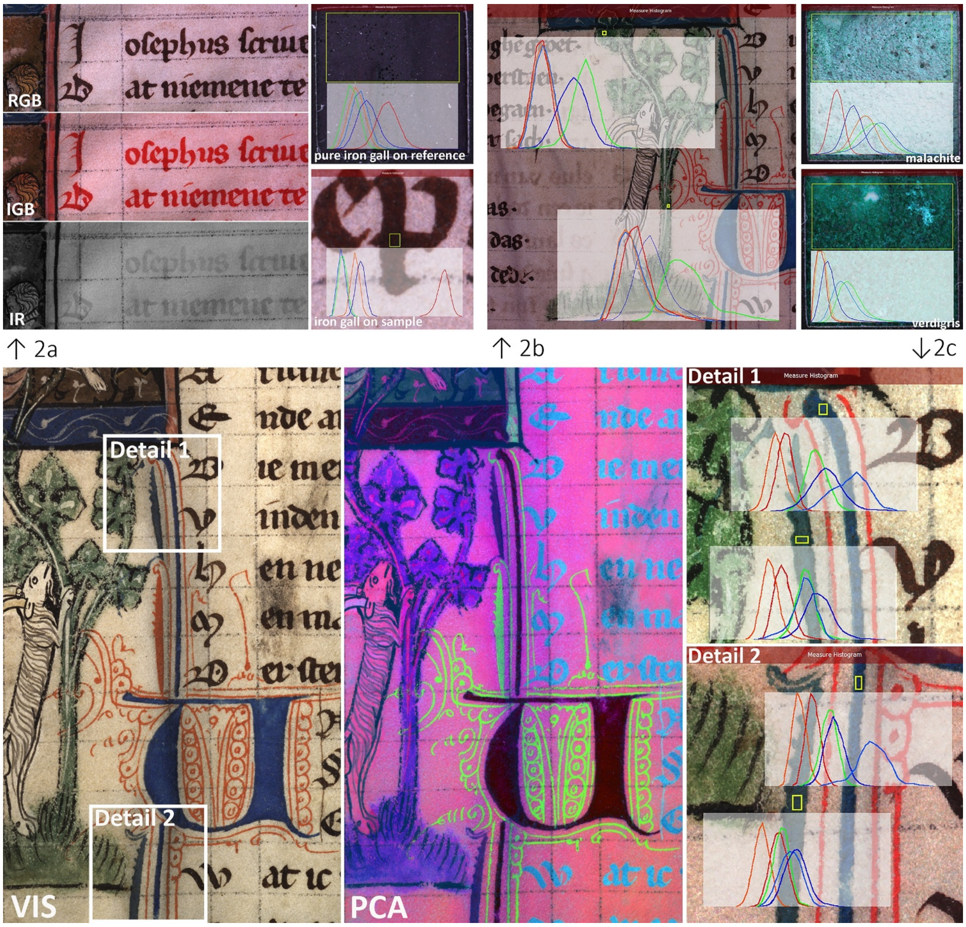

Fig. 2. Selection of the MS MD imaging, rendering and processing results. (a) Analysis on the ink, left: standard tri-chromatic red-green-blue (RGB) visual representation, tri-chromatic infrared-green-blue (IGB) false colour and mono-chromatic infrared (IR) image; right: five spectral band histogram plot on modern reference of pure iron gall ink patch (Pigments Checker chart v5) and five spectral band histogram plot on section of original ink of main text. (b) Analysis of green pigment, comparison with five spectral band histogram of original pigment with modern reference patches of pure malachite and verdigris. (c) Analysis of green-blue penwork, Left: standard tri-chromatic red-green-blue (RGB) visual representation, Middle: PCA based on MS MicroD image stack, Right: five spectral band histogram plots on two selected details, each time of the more pure azurite blue and the mixed greenish-blue.

A: slide 12). This insight is also observed in the MLR results: the most blue appearing penwork (to the right) resembles the MS MicroD spectral histogram responses known from azurite (Fig. 2c and Appendix A: slide 14), which reveals a higher (plotted more to the right) reflectiveness in blue compared to UV. That conclusion is based on the observation that the mutual ratio between the measured reflectiveness in blue and ultraviolet is the same as in the plot of the modern reference patch of azurite. This is in contrast to the more greenish blue penwork (to the left) which does not reveal this specific determinative reflectance behaviour of azurite. Both measure spots (Fig. 2c: yellow rectangulars) confirm this conclusion, for the more greenish mixture (left) it can be observed the mutual ratio of the blue and UV reflectance differs from the two measure spots of the azurite (right). Based on the MA-XRF results, azurite in combination with iron oxides could have been used for this more greenish mixture, a practice often applied in medieval manuscripts [20]. For both methods, MS MicroD and MA-XRF, the analytic data on the blue penwork can also be visualized with the help of combined false colors (tri-chromatic RGBs) and Principal Component Analysis (PCAs) (Fig. 1; 2c; Appendix A: slides 11–13); that allows one-on-one comparison investigations with standard true-color images. The PCA image transforms the multi-dimensional dataset into a new coordinate system, and by doing so, reduces the observed variations into a limited number of variables. The resulting component images show for each pixel which portions of its spectral curve are

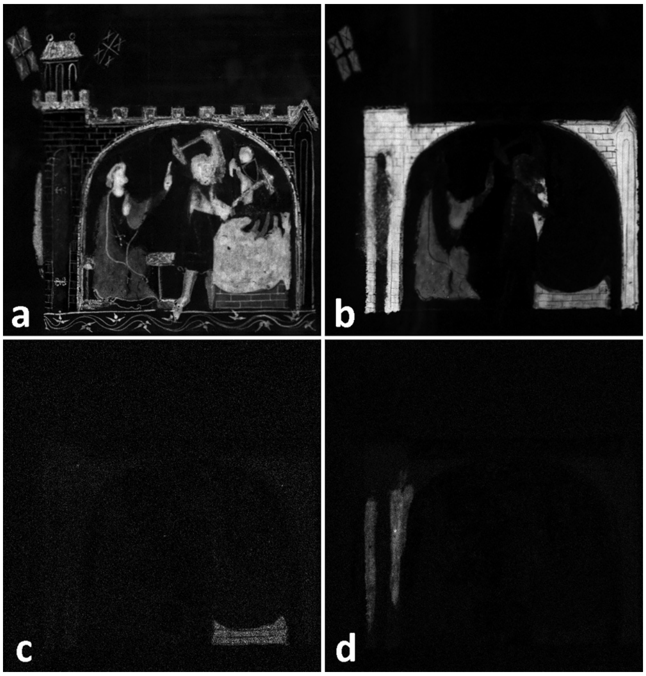

Fig. 3. Elemental distribution maps of the MA-XRF scanning and analysis results of a selected area on folio 86v: (a) lead (Pb-Lα), (b) copper (Cu-Kα), (c) barium (Ba-Lα), d – cobalt (Co-Kα).

contributed by the different basic spectral shapes common for all pixels [21]. For the blueish penwork the basic shapes identified of the spectral curves of both discussed pen strokes vary sufficiently which makes them contrast more strongly in the PCA image. The PCA result in Fig. 2c has been generated with the automated PCA plugin as implemented in the software package ImageJ (1.48v). In that case, each PCA result includes the principle components contributions of each of the input images (i.e. for the MS MicroD, five separate images derived from the UV, B, G, R, IR spectral albedo responses). To produce the false colour (trichromatic) image, the RGB channels have been populated with three of the generated PCA component images. These additional visual documentations confirms what the analytic data revealed. Remark good results can be obtained by including only the responses by five spectral bands. For a successful eight multispectral images solution for pigment identifications see [11].

- The intense red penwork in the initial has a reflectance similar to vermilion, nonetheless, based on the measured spectral albedo responses with the MS MicroD vermilion reveals only minor differences compared to the other historical reds, such as red ochre, realgar and madder lake (conclusions based on comparisons on reference patches on CHSOS Pigments Checker v.2.1 and v5). As MA-XRF shows clearly the presence of mercury, the use of vermilion as pigment in the red ink is confirmed. Contrary no inorganic red pigments are used in this illumination of the Rijmbijbel.

The gilder - A gold (leaf) is applied on a raised gesso layer. Based on the MA-XRF scan it can be defined as genuine gold (Au). In addition, with the MS MicroD dataset it reveals the expected measured reflectance patterns when the SRM patterns are examined and the multispectral responses in the various applied bands are used; a conclusion in line with [15].

The illuminator - The blue pigments (although they differ in density) all reveal the same five spectral band histogram layout; these parallel closely with the historic pigment azurite (see above). MA-XRF reveals the presence of Cu and Pb (lead white) in varying relative proportions (Fig. 1). Lead white was mixed with azurite to obtain different shades of lighter tones of blue.

- For the green pigment (with MA-XRF mapped as containing copper, Fig. 1), the five spectral band histogram of the MS MicroD resembles best responses obtained on verdigris ($Cu_2CO_3(OH)_2$) (i.e. with a low reflective response in red, closely identical to its response in IR; whereas red radiations on malachite (also $Cu_2CO_3(OH)_2$) has a higher reflective response, also higher compared to IR, see Fig. 2b). It must be stressed, probably due to the degree of purity and binder of the applied pigment, the similarities and variations among the reflective responses are limited and conclusions must be considered being preliminary.

3.2. *Defining the materials of the 19th century retouches in an illumination*

Some of the damaged Rijmbijbels' illuminations have post-production restoration treatments: in- and overpainting of damaged areas in the miniatures. The restoration of these damaged illuminations had a pure aesthetic approach and most probably can be dated mid-19th century. During the research a range of 'modern' paint materials of these interventions could be documented and visualized as they have different chemical compositions in comparison to the genuine 13th century illuminators paint box. In order to define the modern materials the retouched miniature (representing a soldier destroying an idol with a hammer on folio 86v) of the Rijmbijbel was examined with the two analysis methods: MA-XRF and MS MicroD (Appendix A: slide 15).

In Fig. 3 the MA-XRF distribution map of copper shows a blurry aspect at the left section of the architecture of the castle. The presence of cobalt in the same area indicates very likely a retouching using cobalt blue, painted on top of the original azurite layer. Lead seems to be present in an area that is not part of the illumination itself. Most probably lead white was used on the parchment to camouflage a carelessly executed overpainting. Also the presence of barium points to a retouching or overpaint: the synthetic barium sulfate is, just as cobalt blue, used since the beginning of the 19th century [22].

Another result was evidenced through the MLR imaging. Examining the infrared reflectance image in Fig. 4c, the zone of the face and lifted underarm appear blurry. They seem to have been damaged and later overpainted. There is only a hint of the original face (compared with the untouched faces of the other figures). The vanished lines have been repainted with different ink (no iron gall ink as these contour lines are not visible in the MA-XRF iron distribution map), hence the red coloration in the infrared-red-green rendering (Fig. 4b). The same effect is seen on other parts of the illumination, such as the hammer or next to the figure to the right. Generally – observed with the MS MicroD all over on the illuminations in the Rijmbijbel Jacob van Maerlant (for other examples see Appendix A: slide 19–20) – the black contour lines in the illuminations are carbon based, the iron gall ink is used for writing the text. In this case, based on the reflective responses of the pigments, it seems that alterations on the illumination figures have been made with carbon ink or indigo (both reveal very similar reflective responses), or a combination of these 2 media's.

Concerning the latter, this practice of adding indigo to carbon and iron gall ink is documented in recipes from the middle of the 18th century onwards [23–25]. In order to give colour to ink small proportions of indigo or aniline colours were added to make *blue-black* ink or *violet-black* ink [26]. The first reference to the use of indigo for coloring ink is that made by Eisler in 1770. It was first patented by the firm of Leonhardi of Dresden, in 1856, who claimed the use of both indigo and madder for the purpose and described his products as "alizarine inks”. The imaging with the MS MicroD registers and confirms this practice; the use of an ink containing indigo can be suggested.

4. Conclusions on the experimental joined set-up of MA-XRF and MS MicroD

The combination of MA-XRF mapping and MLR imaging can be considered as complementary identification methods to reveal material characteristics. MA-XRF mapping gives strong analytical ground truth data on a range of chemical elements, the MLR imaging of the MS

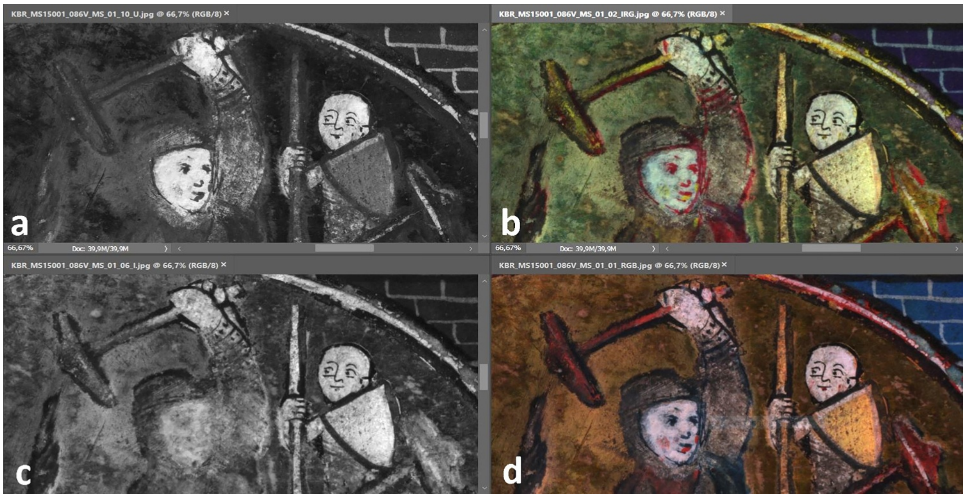

Fig. 4. A MS MicroD result on a detail on folio 86v: (a) UV reflectance, (b) tri-chromatic infrared-red-green (IRG) false colour rendering, (c) NIR reflectance, (d) standard tri-chromatic red-green-blue (RGB) visual representation.

MicroD yields strong visual information on the spectral characteristics of the materials and the surface geometry. In several cases, it allowed to refine the standard insights provided by the MA-XRF elemental distribution, i.e. the possible identification of verdigris and the indigo added to the black carbon paint used in the retouched illumination. Conversely, MA-XRF provides additional information compared to MS MicroD as for the vermilion or the mixture of azurite containing iron oxide.

The binders for preparing the tempera paint, such as egg yolk, egg white, whole egg or Arabic gum, used in the Rijmbijbel are until now not analyzed. Further research with ER-FTIR spectroscopy could shed more light on the composition of the used binding media for specific pigments and for concluding if finishing glazes were used [27]. The combination of the three techniques would certainly allow more advanced conclusions about the paint composition used by the illuminator. Identifying the applied binders would also allow fine-tuning the interpretation of the spectral results with the MS MicroD; as it has been established, binders might increase the fluoresces effect and might even shift particular distinctive spectral peaks along the reflectance curves of particular materials [28].

When we assess the *modus operandi*, the investigation time with the MS MicroD for the same surface on the artefact is shorter than a scan with the MA-XRF. The MS MicroD is portable, compact, lightweight and can therefore easily travel to the collection where the manuscript/artefact is kept. Thus, deploying the MS MicroD system for a ‘fast’ first imaging of an object gives a good dataset for a range of preliminary analyses in de form of visual information and the five spectral band histograms for the characterization of a number of materials. The same dataset can also be used for examination of the topography of the surface (photometric stereo), but this research field is not included in this essay (more on that see Appendix A: slide 20–21 and [13]. As stated above, the MA-XRF analyses deliver uninterpreted, accurate, strong true data on the distribution of detectable chemical elements. But, an important issue remains the time needed for the acquisition which for a scan with a high resolution, can take hours for only a few cm^2 (small spot size, small step size, high dwell time per step).

Based on the applied complementary use of the MA-XRF and MLR techniques on the selected sections on folios in the Rijmbijbel of Jacob van Maerlant, it is the research team estimation that the combination of the two approaches is beneficial and has great potential for the scientific documentation, assessment and study for this type of heritage objects. This approach will be further explored and fine-tuned in the future to refine the methodology presented here for *in situ* illumination analyses. This contribution acts as a first proof of concept.

Funding

This research was supported by the King Baudouin Foundation (KBR, Manoël de la Serna Fund, 2016) and FWO, Belgium (KU Leuven, RICH Project 2012–2016 / 3Pi Project 2018–2022/ Mid-scale research infrastructure).

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Supplementary materials

Supplementary material associated with this article can be found, in the online version, at doi:10.1016/j.microc.2019.104582.

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