### PERIODICO di MINERALOGIA

established in 1930



An International Journal of Mineralogy, Crystallography, Geochemistry, Ore Deposits, Petrology, Volcanology and applied topics on Environment, Archaeometry and Cultural Heritage

# The "Exultet 1" of Bari: multi-methodological approach for the study of a rare medieval parchment roll

Gioacchino Tempesta <sup>1,2,\*</sup>, Carlo Porfido <sup>3</sup>, Michele Bellino <sup>4</sup>, Alessandro Monno <sup>1,2</sup>

- <sup>1</sup> Department of Earth and Geo-environmental Sciences, University of Bari "Aldo Moro", Via Orabona, 4 - 70125 Bari, Italy
- <sup>2</sup> Interdepartmental Center "Research Laboratory for the Diagnostics of Cultural Heritage",
- University of Bari "Aldo Moro", 70125 Bari, Italy
- <sup>3</sup> Department of Soil, Plants and Food Sciences, University of Bari "Aldo Moro", Via Amendola 165/A - 70126 Bari, Italy
- <sup>4</sup> Diocesan Museum Bari Section, Bari-Bitonto Archdiocese, via dei Dottula 70122 Bari, Italy

## ARTICLE INFO ABSTRACT

Submitted: October 2017 Accepted: March 2018 Available on line: April 2018

\* Corresponding author: gioacchino.tempesta@uniba.it

DOI: 10.2451/2018PM753

How to cite this article: Tempesta G. et al. (2018) Period. Mineral. 87, 93-102

A rare medieval parchment roll, called Exultet 1 (first half of XI Century), preserved in the Diocesan Museum of Capitolo Metropolitano of Bari (Italy) has been studied. Certainly, it is one of the highest literary and artistic expressions ever produced in Bari, one of a kind. It is also the very first among medieval European liturgical rolls, in which miniatures are painted upside-down in respect to the text. To analyse inorganic pigments, a mobile laboratory has been set up at the museum. The techniques used, for a non-invasive and non-destructive in situ analysis, were: VIS-NIR spectrophotometry in reflectance mode with optic fibres (FORS), X-Ray fluorescence spectrometry (XRF) and finally µ-Raman spectroscopy. Such multi-methodological approach allowed to acquire many data without causing any stress to the artwork. The mineral pigments have been identified mainly by FORS and they are: red earth and minium, lapis lazuli and azurite, green earth and copper resinate, orpiment and yellow earth. In some areas, traces of a residual gold leaf have been identified by XRF. The presence of rare and precious pigments as lapis lazuli and gold leaf underlines the sacredness and the importance of the parchment. This is the first scientific investigation performed on the Exultet 1 of Bari.

Keywords: illuminated manuscript; pigments; in-situ analysis; fors; xrf; µ-raman.

#### INTRODUCTION

Around the 1000 AD, Greek monks moving from the Byzantine regions of Sicily and Calabria, during their stay at the Benedictine monasteries of Southern and Central Italy (e. g. Montecassino, Benevento, Salerno, Troia, Bari) they reintroduced the use of the scroll (Cavallo, 1973). Indeed, during Middle Age in Western Europe the codex (a book format similar to the modern one) was widespread, whereas in Byzantine areas the roll (which descends directly from the Egyptian papyrus) was still used, mainly for liturgical purposes (Weitzmann K., 1959). There still exist 31 parchment rolls produced in Italy during the XI century (Cavallo, 1973), and in particular the one known as "Exultet 1 di Bari" (dated 1025-1050 CE, Monastery of San Benedetto, Bari) is certainly one of the most renowned and beautiful. This 525 cm roll is composed by eight rectangular sheepskin (Van der Werf et al., 2017) folios (about 50X70 cm) sewn together from the short edge, with beautiful miniatures which frame and intersperse the *Praeconium* text. The name

Exultet originates from the first word of the Praeconium *Paschale*, the prayer sung before the blessing of the great candle during the Easter vigil ceremony. The greatest significance of the Bari scroll lies in the fact that it is the oldest in which the reversal text-image was utilised. Miniatures are depicted upside down relative to the text, thus when the deacon played the prayer from the ambo, the images appeared at the correct way to the faithful, helping them to understand the Latin text (like a "Biblia pauperum"). Such feature, that makes this parchment resembling a cinematographic film, had never appeared before in medieval scrolls. Furthermore, the Exultet 1 can be considered as a testimony of the dialog between West and East, since the beautiful style of its miniatures put together European and Byzantine artistic traditions. In fact, at the beginning of Eleventh Century (876-1071 CE) the city of Bari was the capital of the Italian territories under the Byzantine control and it became an important centre of cultural exchange (Cavallo, 1973). This scroll demonstrates how such dialogue can produce culture and beauty, with a great significance also in our troubling time.

The Exultet 1 is preserved and exposed at the Museo Diocesano of Bari, kept completely unrolled into an exhibitor built ad hoc. An extraordinary opening of the Exultet theca for cleaning purposes, gave us the opportunity to set, for a couple of days, a mobile laboratory in the Diocesan Museum to perform the characterization of mineral pigments.

All the analyses were performed with non-invasive and non-destructive techniques, in situ, using portable instruments (Figure 1).

Vis-NIR Fibre Optics diffuse Reflectance Spectrophotometry (FORS) X-Ray Fluorescence spectrometry (XRF) and  $\mu$ -Raman spectroscopy were applied for pigments and gildings qualitative characterization. Several authors in literature have successfully applied such non-destructive and non-invasive approach for pigments identification on parchment using the same complementary analytical techniques (Clark, 1995; Depuis and Menu, 2006; Lorusso et al., 2007; Milani et al., 2010; Bicchieri et al., 2011; Aceto et al., 2012; Colomban, 2012; Cavaleri et al., 2013). Furthermore, colorimetric measurements were performed in CIE Lab colour space for a quantitative description.

## **EXPERIMENTAL**

The knowledge of the ancient techniques of preparing parchments as well as preparing pigments is certainly the first step of the study, though considering that each art workshop or scriptorium had its own secrets and methodologies (Cennini, 1859; Weitzmann, 1947; Caffaro, 2003). However, the availability of very extensive and complete database of historical pigments with XRF, FORS and Raman spectra (Cosentino, 2014; Caggiani et al., 2016; Larsen et al., 2016), represents a very powerful instrument for pigment identification.

Due to the time constraints for our analysis, a preliminary selection of the points to be analysed by means of accurate optical observation was of great importance. Thus, maps were prepared in order to immediately referencing the acquired data on the parchment surface (Figure 2). The FORS technique has the advantage of being fast and accurate for pigment identification. Therefore, it was applied as first to all of the selected areas located by the map. Then, XRF analysis were performed, sampling at least 4 points for each colour and finally Raman was applied in few cases to confirm the pigment identification. After these spectroscopic acquisitions, a number of selected points were analysed for colorimetric characterization.



Figure 1. Photograph taken during the extraordinary opening of the Exultet's theca. The mobile laboratory has been set up at the Museum.



Figure 2. Distribution map, on the parchment roll, of all the points where the analyses have been performed.

#### Fibre Optics Reflectance Spectroscopy - FORS

When applied on pure colours, FORS allows a simply and rather fast characterization of the pigments, based on the comparison of the experimental spectra with the standards (Larsen et al., 2016). A FORS spectrophotometer AvaSpec-2048-USB2 was used, working from 350 to 1000 nm with a spectral resolution of about 1.4 nm. The diameter of the probe, about 200 µm, allows taking measurements even of very small painted areas depending form the surface distance. The capabilities to collect a reflectance spectrum from a small area was an important feature to avoid inhomogeneous coloured areas, especially when the surface was damaged or fractured with the tampering of the time. The FORS spectra of about 70 painted areas, with spot size about 2 mm in diameter, were acquired on the entire parchment roll. A calibrated tungsten halogen light source was used as illuminant. The spectra were collected and improved with Avasoft 8.0 and then exported in Excel for the processing, mathematical operations and the comparison with standards.

### X-Ray Fluorescence spectrometry - XRF

XRF was carried out not only to confirm the information obtained by FORS but also to obtain a chemical fingerprint of the analysed areas. About 20 points well spread on the roll were analysed with a Surface Monitor (Assing) equipped with a Mo X-Ray source (30 kV; 220 µA) and with a Silicon Drift Detector (SDD) with a resolution of 135eV FWHM at 6.3 keV at room temperature. Both source and detector were placed on two goniometers, managed by separate motors that allow acquiring XRF spectrum at different angles respect to the scanned surface. They were set at the same angle of 45° degrees respect to the surface. For each acquisition, the correct working distance (84 mm) between the instrument and the parchment surface was controlled by means of a laser interferometer. The spectra were collected and elaborated with the software Surface Monitor 7.0. One of the main strength of the Surface was its notably low weight (1.84 Kg), which makes it very manageable for in situ analysis. On the other and, performing the XRF analysis in the air introduce some physical limits, thus lighter elements were not detectable. In our case, with the previous described setup, only identification of elements with Z higher than 19 was possible.

## µ-Raman

Due to the difficulties connected to the shape and the dimensions of the scroll in using the Raman mobile instrumentation, these analyses were performed only on a few number of points, especially where FORS and XRF results were not univocally interpretable. A micro-Raman system (Horiba Jobin Yvon), equipped with a red He-Ne laser (632.8 nm) and optical fibres was used. Its low laser power on the sample, about 1 mW, guaranteed the safety of the parchment, while the frequency calibration was performed against the Raman peak of silicon. The instrument was equipped with an optic head with 60X objective, which has a spectral resolution of 10 cm<sup>-1</sup>.

Acquisitions were performed with LabSpec 4.18 software while the data elaboration and the identification of the analysed phases were performed with GRAMS/AI 8.0 suite (Thermo).

#### Colorimetry

The colorimetric analyses were performed with CM2600d (Minolta) on about 40 points, painted in red, blue, yellow, green, brown and black. This portable spectrophotometer-colorimeter (0.67Kg) is a compact system that may operate also with battery. Due to its functional characteristics (illumination angle/view 8°) the instrument is able to acquire simultaneously the visible spectrum (360nm to 740nm) with the specular component included (SCI) and excluded (SCE) adapting itself to the different surface conditions in question. The area of measurement may be selected using two masks with a diameter of 3mm or 8mm according to requirements. The instrument allowed to obtain information on colour in a large range of colour spaces (L\*a\*b, L\*C\*h, CMC (1:1), (2:1), CIE94, Hunter Lab, DIN99, Yxy, Munsell, XYZ, etc.).

#### **RESULTS AND DISCUSSIONS**

The characterization of the primary palette (red, blue, yellow and green) has been possible by analysing the reflectance spectra collected by FORS. Since pictorial binders play a key role in light reflectance (Cosentino 2014), thus influencing pigments reflectance spectra, their identification would be of primary importance while using FORS.

However, Van der Werf et al. (2017) did not obtain a certain characterization of Exultet 1 binders, probably due to the overall severe degradation conditions of the pictorial layer. In the same paper, Van der Werf et al. identified egg white as the binder used for the Benedizionale, a parchment similar to Exultet for its iconography. The Benedizionale, which is the second most important scroll of the Museo Diocesano, was at the beginning sewn at bottom of the Exultet 1 (Cavallo, 1973). Since the scrolls are coeval and both produced at the same scriptorium, it is reasonable thinking that also for the Exultet the pictorial binder used was egg white.

Therefore, Exultet pigments identification was carried out valuating the spectral features of egg tempera standards (Cosentino 2014) and those of the experimental The comparison showed only few small differences, which could be ascribed to the aging, to the presence of impurities in the pigments or in the binder (Van der Werf et al. found also traces of yolk egg on the parchment surface), pigments overlapping or also the use of dirty brushes.

Lapis lazuli and azurite (e.g. B23 and B34) were identified thanks to the characteristic reflectance maximum at about 450 nm and the completely different behaviour from 600 nm to NIR region, which allowed us to distinguish one from each other (Figure 3a). In fact, the two pigments have different mechanisms for the absorption of light: in lapis lazuli is due to charge transfer, whereas ligand field transitions determine the blue colour in azurite (Aceto et al., 2014).

Yellow pigments, which were classified as yellow earth and orpiment (e.g. Y22 and Y21), showed the typical S-shape spectral features with the inflection point around 500 nm. The yellow earth was identified by means of some different spectral features from 550 to 1000 nm (Figure 3b), which are characteristic for Fe - (hydro)-oxides that constitute this pigment. These spectral features are similar to red earth (Figure 3c) (Aceto et al., 2014).

The characteristic inflection point at about 550 nm allowed us to identifying red pigments. However, red earth and minium (e.g. R22 and R31) were distinguishable thanks to a maximum reflectance peak at about 750 nm and an absorbance band at 900 nm, which belong only to red earth, in particular to the mineral hematite (Torrent and Barron 2015) (Figure 3c).

Finally, green earth "Terra Verona" and copper resinate (e.g. G74 and G32) were identified. The broad absorption band at 700 nm was diagnostic for copper resinate (Figure 3d), whereas the reflectance maximum at 560 nm and a secondary broad maximum around 850 nm demonstrated the presence of green earth pigment, more specifically glauconite (Hradil et al., 2004; Cheilakou et al., 2009).

In general, minium and azurite were found only where the pictorial film is well preserved, while in contrast, all over the parchment the blue and the red pigments correspond to lapis lazuli and red earth, respectively. Among such well-preserved miniatures, there is the scene representing the "Ecclesia", which is present on the third sheet (Figure 4). The colours in this painting are perfectly preserved and bright, the blue of the vestments as well as the sparkly red of the dome of the ciborium. A similar condition is observable in the seventh sheet, where the spectroscopic results revealed the presence of the same materials identified in the "Ecclesia". The occurrence of specific pigments only in well preserved miniatures evidenced a restoration or a superimposed pictorial layer.

The darker blue pigment lapis lazuli, in the other



Figure 3. Comparison between standard and experimental pigments (red, blue, yellow and green) reflectance spectra obtained by FORS in the wavelength range of 350-1000 nm.

representative scenes results corrupted, with cracks and missing parts (Figure 5). The presence of this rare and expensive pigment underlines the importance and the preciousness of the Exultet 1.

The green classified as green earth is ubiquitous, and shows a pale appearance, but generally a fairly good conservation status; the copper resinate instead was detected only in the miniature representing the "Mother Earth" (G32) (Figure 4).

Despite the fact that pictorial layer is heavily corrupted and not detectable optically, XRF analysis allowed us to detect little traces of gold gildings in some miniatures, probably attached with an organic glue (Figure 5). Indeed, XRF did not revealed the

Fe characteristic peak of bolus armenus, which was generally used in that period for such purpose (Cennini, 1859; Caffaro, 2003). This finding is of great significance for historians, because not only the material was identified but also the technique used was unmasked.

Furthermore, XRF acquisitions gave also the chemical confirmation of FORS results, since the characteristic elements of the pigments were detected. For example, As in the case of orpiment (Y41), Pb for minium (R31), Cu for Azurite (B23) or for copper resinate (G42), Fe for red

earth (R23) or yellow earth has been confirmed (Figure 6).

The chemical analyses strengthened also the above discussions about the presence of later superimposed pictorial layer. In fact, the XRF spectra exhibited traces of elements that do not belong to the superficial pigments identified by FORS. For instance, Fe was detected together with Pb (R31), which could indicate the presence of minium and other pigments.

Another interesting XRF observation was that in every spectrum signals ascribable to Arsenic (As-K $\alpha$ : 10.5 keV) were observed, always quite intense. Such presence could be related to a diffused distribution of orpiment, from which the yellowish-golden aspect of the entire scroll originates. However, more likely it is due to the application of a protective layer, since preservatives varnishes often contain As as antibiotic. Such varnish may have been applied during the restoration of the '50<sup>s</sup> (Mascolo and Nardella, 2014).

XRF spectra also showed the ubiquitous presence of Ca. It could be related to the use of calcium hydroxide solution for parchment depilation (Poole and Reed, 1962), as well as to the powders and pastes of Ca, used to remove the grease in order to favour adsorption of inks. Moreover,



Figure 4. The third sheet of the parchment the one in better health situation of the entire roll. The upper miniature, the "Ecclesia", represents the deacon (on the left) singing the Praechonium, the bishop (at the right) and other religious in the celebration. The miniature at the bottom represents the "Mother Earth" and shows a fairly good conservation status.

presence of Ca is a distinctive of western manufacturing process, which used lime, whereas eastern used enzymatic treatment (Bicchieri et al., 2011).

Finally, the widespread presence of Pb could be connected to the stylus which was traditionally used in Middle Age to create the preparatory design, as Pb peaks have been observed analysing black streaks below the residues of blue lapis lazuli (B23) (Figure 6).

XRF analysis in R31 however, as discussed before, beside the Pb peaks revealed also the presence of Fe. The latter element, which does not belong to minium, could be referred to the presence of red earth under the minium layer (perhaps mixed with it). This finding, together with the fact that minium was used only for such miniature (sheet 3, Figure 4) and that the miniature itself is in much more healthy condition than the others are, suggests a repainting or a remaking intervention.

 $\mu$ -Raman analysis confirmed the identification only of lazurite (B72) and minium (R31) (Fig. 7), respectively above the right shoulder of the blue dressed Emperor (Fig. 5) and on the red dome of the ciborium (Fig. 4).

Many other painted points were analysed, but no spectra were readable due to fluorescence phenomena.



Figure 5. Details of two parchment sheets with several lacks of pigments: a) Jesus Christ on the throne (IV sheet). b) The miniatures called "Two Emperors" (VII sheet). Note that lapis lazuli was damaged on the vestment of Jesus on throne and of the left Emperor while the gilding was lacking around Jesus.



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Figure 7. µ-Raman spectra of lazurite (B72) and minium (R31) identified by matching with references (blue).

This problem may be related to a superficial organic layer, supporting the hypothesis of a protective varnish, which is in accordance to Van der Werf et al. (2017), who identified fruit tree gum as surface layer. This protective layer would justify also some differences found between the standards and the experimental FORS spectra (Cosentino, 2014).

The data acquired with colorimeter in the CIE-Lab colour space are shown in the following Table 1 for the points analysed on the roll and pointed out on the map (Figure 2).

## CONCLUSIONS

In this study, Bari's parchment roll called Exultet 1 was analysed, for the first time, using non-invasive in situ techniques. The palette employed at the S. Benedetto scriptorium was thoroughly characterized and comprised of red earth, yellow earth, lapis lazuli, green earth, copper resinate and orpiment pigments. Minium and azurite

were also detected but only at some specific points, demonstrating that some miniatures have been restored or modified. Most pigments were identified through FORS. XRF, on the other hand, was useful for identification of traces of gildings and special manufacturing techniques such as the use of organic glue for gold foils and of Pb stylus for preparatory design, or the use of lime for parchment preparation. As was also detected, suggesting the application of a protective varnish. In addition, colorimetric data were collected as a reference for the future acquisitions, to evaluate the status of the conservation.

The results obtained in situ, without moving the artefact from its showcase, shed a new light on the mediaeval Apulian Exultet production, still almost unknown from the materials point of view and would be useful for conservation purposes, but also historical and cultural dissemination.

Sample	L*	a*	b*	C*	h	Sample	L*	a*	b*	C*	h
B31	24.40	-3.33	5.91	6.78	119.38	G41	49.61	-1.24	19.65	19.69	93.62
B33	43.76	-11.06	-1.78	11.20	189.14	G51	58.59	5.09	29.24	29.68	80.12
B34	36.82	-4.12	-7.69	8.72	241.82	G61	53.71	-2.72	17.84	18.05	98.67
B35	37.49	-2.26	-5.70	6.13	248.38	G61bis	51.18	-1.85	19.96	20.05	95.30
B41	32.51	-1.71	2.09	2.70	129.40	G62	64.06	3.29	26.54	26.75	82.93
B51	50.93	-2.54	14.91	15.12	99.66	G63	64.58	2.65	25.04	25.18	83.97
B52	59.41	0.75	26.91	26.92	88.40	G64	63.96	3.69	26.19	26.44	81.98
B53	62.90	1.05	24.39	24.41	87.53	G71	59.60	3.87	26.34	26.63	81.64
B71	46.74	-6.11	7.22	9.46	130.24	G72	61.93	1.40	22.58	22.63	86.45
B72	38.43	-5.84	1.82	6.11	162.68	G73	56.22	-1.52	25.37	25.41	93.43
B73	40.04	-5.06	5.49	7.46	132.66	G74	63.23	3.92	26.37	26.66	81.55
R31	45.22	28.14	27.74	39.51	44.59	G75	45.63	-0.82	22.06	22.07	92.14
R32	42.66	21.05	21.77	30.28	45.96	Br51	39.83	8.75	14.37	16.83	58.68
R33	34.78	14.50	11.57	18.55	38.57	Au41	51.77	12.53	29.99	32.51	67.33
R51	53.16	23.80	30.96	39.05	52.45	P31	54.84	11.63	25.12	27.68	65.16
R52	42.80	14.67	18.29	23.44	51.26	Pk31	58.74	12.12	34.19	36.27	70.49
R53	59.21	15.79	28.12	32.25	60.69	Y51	59.23	8.60	29.73	30.95	73.86
R61	38.67	17.25	19.45	26.00	48.44	Y52	59.32	14.73	45.29	47.62	71.98
R62	46.55	12.31	23.04	26.13	61.88	Y53	62.57	7.60	32.47	33.35	76.83
R71	43.84	13.00	19.28	23.25	56.02						
R72	41.01	23.32	18.79	29.94	38.86						
R73	37.94	13.53	15.56	20.62	48.99						

Table 1. Colorimetric data (CIE L\*a\*b\* and L\*C\*h coordinates) of the points reported on the Map in Figure 2.

#### ACKNOWLEDGEMENTS

The authors would like to thank the "Museo Diocesano di Bari" for supporting during the analyses in-situ and the "Soprintendenza Archivistica e Bibliografica della Puglia e della Basilicata" that authorized the diagnostic study on the parchment. We would like also to thank Dr. Eren Taskin of "University of Bari - Aldo Moro" for the English revision of this article, and Paolo Azzella (Quorum Italia) for photos.

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