

Article

Non-Invasive Study on the *Sinope Gospels*

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Received: 22 September 2020; Accepted: 20 October 2020; Published: 28 October 2020



Abstract: The 6th century *Codex Sinopensis* or *Sinope Gospels* (Paris, Bibliothèque nationale de France) is one of the most precious purple codices that survive from the Late Antique period. Together with the *Vienna Genesis* (Wien, Österreichische Nationalbibliothek) and the *Rossano Gospels* (Rossano Calabro, Museo Diocesano), it has an unusually rich decorative apparatus with scenes representing biblical episodes. It can be, therefore, considered one of the most important preserved artistic productions of the early medieval era. The manuscript has been subjected to a non-invasive diagnostic campaign to evaluate the quality of the colourants used in its decoration, to understand how the parchment was coloured, and to carry out a comparison with the *Vienna Genesis* and the *Rossano Gospels*. The techniques used were UV-visible diffuse reflectance spectrophotometry with optical fibres (FORS), X-ray fluorescence spectrometry (XRF), spectrofluorimetry, and optical microscopy. Analyses highlighted the presence of ultramarine blue, which, besides the use of pure gold for the ink and paint, certifies the high value of the manuscript. In addition, this must be seen as one of the earliest examples of its use in paintings. The purple colour of the parchment was identified as orchil, a dye extracted from lichens, similar to the results of analytical investigations carried out on other purple codices, and not the expected Tyrian purple dye.

Keywords: FORS; XRF; purple; orchil; manuscript; lapis lazuli; silver

1. Introduction

The *Codex Sinopensis* or *Sinope Gospels* (Paris, Bibliothèque nationale de France, ms. Supplement grec 1286), datable to the 6th century CE, is a purple codex, that is, a manuscript written with noble metal inks on purple coloured parchment. These luxurious manuscripts contained biblical texts like the four Gospels and were produced since the 5th c. CE for kings, emperors, popes and other people of noble birth. Like the early textiles dyed with costly Tyrian purple, which indicated the high status of their owners, the purple of the manuscripts also symbolized the suffering of Christ and man's repentance from sin [1]. The *Codex Sinopensis* is one of three Greek purple codices, including the *Vienna Genesis* (Codex Vindobonensis Theol. gr. 31, Wien, Österreichische Nationalbibliothek) and the *Rossano Gospels* (Rossano Calabro, Museo Diocesano), where the text is accompanied by scenes

depicting biblical episodes. It is, therefore, considered to be one of the most important manuscripts of the Late Antique period (3rd–7th century AD).

The manuscript is thought to have once contained all four Gospels within 490 to 500 folios. Today, the fragmentary codex of 43 folios contains portions of the Gospel of Matthew; only five of these folios have decorative features. These small painted illustrations or miniatures represent scenes from the New Testament: Herod's feast and the beheading of St. John the Baptist (f. 10v); the first miracle of the multiplication of loaves (f. 11r); the second miracle of the multiplication of the loaves (f. 15r); the miracle of the two blind men of Jericho (f. 29r); and the miracle of the dried fig tree (f. 30v). These scenes are flanked by images of the prophets of the Old Testament, who hold scrolls containing the text of the prophecies that prefigure them (ff. 10v. and 11r, Moses and David; f. 15r, David and Moses; f. 29r, David and Isaiah; f. 30v, Habakkuk and Daniel).

The manuscript has a varied palette, including black, blue, brown, gold, green, orange, pink, red, silver, white, and yellow for the miniatures, purple for the parchment and gold ink for the text. Of the handful of Late Antique purple manuscripts that survive, the *Codex Sinopensis* is the only one written entirely in gold ink. Given its artistic and historic importance, it was subjected to a non-invasive diagnostic campaign in order to evaluate the quality of the colourants used in its decoration, to understand how the parchment was coloured and to carry out a comparison with the *Vienna Genesis* and the *Rossano Gospels*. The *Vienna Genesis* is thought to have been made in Syria, while the origin of the *Codex Sinopensis* and the *Rossano Gospels* is considered to be either Syria or Palestine. The techniques used for the characterisation of the palette were UV-visible diffuse reflectance spectrophotometry with optical fibres (FORS), X-ray fluorescence spectrometry (XRF), spectrofluorimetry with optical fibres, and optical microscopy.

2. Materials and Methods

Since no sampling of any kind was allowed and the work had to be done on site in Paris, measurements were limited by non-invasive analysis using portable equipment according to a previously developed protocol [2]. FORS was used because it yields responses from most of the colourants used in Antiquity and its spatial resolution (spot of 1.5 mm) is good enough to be highly specific; XRF spectrometry, being an elemental technique, yields responses from metallic pigments, i.e., gold and silver, and adds information on the underlying layers, thanks to its high penetration depth; spectrofluorimetry with optical fibres is useful to confirm the identification of colourants obtained with FORS, having the same spatial resolution; lastly, optical microscopy can help to address the spectral responses yielded by the spectroscopic techniques.

UV-visible diffuse reflectance spectrophotometry with optical fibres (FORS): FORS analysis was performed with an Avantes (Apeldoorn, The Netherlands) AvaSpec-ULS2048XL-USB2 model spectrophotometer and an AvaLight-HAL-S-IND tungsten halogen light source; the detector and light source were connected with fibre optic cables to an FCR-7UV200-2-1,5 × 100 probe. In this configuration, both the incident and detecting angles were 45° from the manuscript's surface, in order not to include specular reflectance. The spectral range of the device (combination of lamp + detector) was 375–1100 nm. Depending on the features of the monochromator (slit width 50 µm, grating of UA type with 300 lines/mm) and of the detector (2048 pixels), the best spectra resolution was 2.4 nm calculated as Full Width at Half Maximum (FWHM) Diffuse reflectance spectra of the samples were referenced against the WS-2 reference tile provided by Avantes and guaranteed to be reflective at 98% or more in the spectral range investigated. The investigated area on the sample had a 1.5 mm diameter. The probe was inserted into an aluminum block, in order to exclude external light and to firmly hold the probe in place. During analysis, the block was laid directly on the folio, the side in contact with the manuscript was covered with Tyvek®, a soft fabric. In all measurements, the distance between probe and sample was kept constant to 2 mm. To visualise the investigated area on the sample, the probe contained a USB endoscope inserted as well in the block. The instrumental parameters were as follows:

10 ms integration time, 100 scans for a total acquisition time of 1.0 s for each spectrum. The whole system was managed by means of AvaSoft v. 8 dedicated software, running under Windows 7™.

X-ray Fluorescence spectrometry (XRF): XRF measurements were performed with an EDXRF Thermo (Waltham, USA) NITON spectrometer XL3T-900 GOLDD model, equipped with an Ag tube (max. 50 kV, 100 µA, 2 W), a large area SDD detector, energy resolution of about 136 eV at 5.9 keV. The analysed spot had an average diameter of 3 mm and was focused by a CCD camera, with a working distance of 2 mm. Total time of analysis was 240 s. The instrument is held in position with a moving stage allowing micrometric shifts, in order to reach the desired probe-to-sample distance; the stage is laid on a tripod. The obtained spectra were processed with the commercial software WinAxil (Canberra Eurisys Benelux, Zellik, Belgium), derived by the academic software QXAS from IAEA (International Atomic Energy Agency, Vienna, Austria).

Spectrofluorimetry with optical fibres: An Ocean Optics (Dunedin, Florida, USA) Jaz model spectrophotometer was employed to record molecular fluorescence spectra. The instrument is equipped with a 365 nm Jaz-LED internal light source; a QF600-8-VIS/NIR fibre fluorescence probe is used to drive the excitation light on the sample and to recover the emitted light. The spectrophotometer works in the range 191–886 nm; according to the features of the monochromator (200 µm slit width) and detector (2048 elements), the spectral resolution available is 7.6 nm calculated as FWHM. The investigated area on the sample is 1 mm in diameter. In all measurements, the distance between probe and sample was kept constant at 12 mm, corresponding to the focal length of the probe. To visualise the investigated area on the sample, the probe contained a USB endoscope. Instrumental parameters were as follows: 2 s integration time, 3 scans for a total acquisition time of 6 s for every spectrum. The system was managed with SpectraSuite™ software under Windows 7™.

Optical Microscopy: A USB Dino-Lite (New Taipei City, Taiwan) AM4113T-FV2W model microscope was used to acquire digital images at 20 ×–90 × magnification ratios. The instrument is equipped with 375 nm and visible LED lights and a digital camera with 1.3 Megapixel resolution

3. Results and Discussion

All the folios of the *Codex Sinopensis* are stored between glass plates and framed, except for those containing miniatures, which are housed in mattes. This was beneficial for the non-invasive analysis of the miniatures, which nevertheless was carried out in the shortest time possible in order not to cause distortions in the extremely thin parchment when exposed to air.

In the following paragraphs, the results of the diagnostic study are described according to the colour (black, blue, brown, gold, green, orange, pink, red, silver, white, and yellow) and to the features (colourants, inks, and parchment) that were analysed.

3.1. Black

Some details (hair, stripes on garments, contours of the haloes) were created using a carbon black pigment, identified by a flat line at nearly 0% reflectance in the FORS spectrum.

3.2. Blue

The artist of the *Codex Sinopensis* used two blue pigments: *ultramarine blue*, the pigment obtained from the semiprecious stone lapis lazuli, and *indigo*, an organic pigment obtained from the leaves of *Indigofera tinctoria*, a plant native of South-eastern Asia, or from *Isatis tinctoria*, a plant widely found in the Mediterranean area; in this case, the pigment is called *woad*. The pigments were identified by FORS according to the typical absorptions at 600 and 650 nm, respectively [3]. Due to the considerable difference in the price of these two pigments, with ultramarine blue being much more expensive than indigo, one would expect the artist to have used them in a selective way, according to the symbolic meaning of the features being depicted. However, this was not found to be the case. On f. 10v, the throne of Herod, the arch, and the garment of David on the right (the same on f. 30v for the garment of Daniel) are painted with ultramarine blue, while the light blue garments of all other figures,

including Herod, are painted with indigo; the same was found for the garments of the figures on f. 15r and 29r. On f. 29r, the foliage on the right is in ultramarine blue, but the dark blue background on f. 11r is painted with indigo. The presence of ultramarine blue, together with the gold ink and the purple coloured parchment, confirms the symbolic importance of the manuscript [4]. Moreover, the presence of ultramarine blue in the 6th century *Codex Sinopensis*, as well as in the two closely related purple codices, is thought to be one of the earliest records of its use in Mediterranean art [5], more precisely from the eastern part, i.e., Syria or Palestine. This means that, as far as we know from the extant witnesses, artists from the Middle East used lapis lazuli for painting at least two centuries before artists from Western Europe.

3.3. Brown

Some features, such as garments, are painted with *yellow ochre*, a very common earth pigment used since prehistory, identified by FORS according to the typical inflection point at 560 nm and the absorption at ca. 900 nm. Yellow ochres can have different hues, from light brown to pure yellow, according to the presence of accessory mineral phases.

3.4. Gold

The text, initials, and certain pictorial features are rendered with ink and paint made from ground gold. In the miniatures, gold paint is used mainly for important features such as the haloes and the robe of Jesus. XRF analysis allowed us to verify that the gold was very pure, with only a negligible amount of silver. However, the composition of the gold paint is slightly different from that of the golden ink (see later), due to a significant amount of iron, which is not present in the latter (see an example on f. 15r in Figure 1).

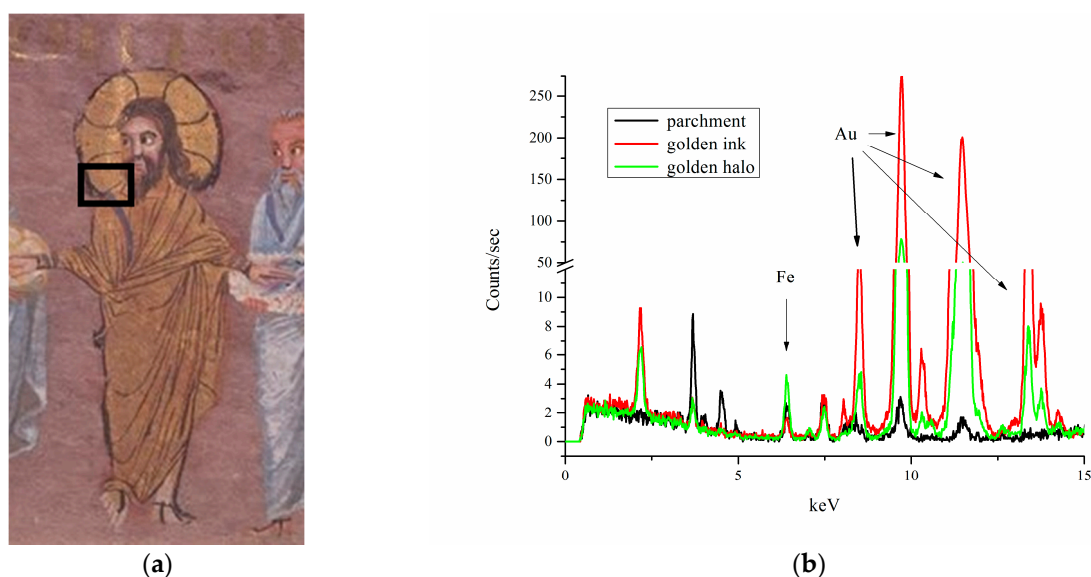


Figure 1. (a) Christ on f. 15r with highlight of the area analysed by X-ray fluorescence spectrometry (XRF); (b) XRF spectra from the golden ink on f. 15r, the golden halo of Christ, and the parchment.

As it can be seen from the spectra (XRF spectra have been normalised on the inelastic scattering curve to minimise the variation in the thickness of the parchment), only the gold paint has iron together with gold, while the golden ink on the same folio is free from iron. All of the other features painted with gold, e.g., the robe of Christ on f. 15r, the haloes of the prophets on f. 10v, etc., have similar features, that is, gold containing a significant amount of iron. The results of FORS analysis exclude a mixture of gold and an iron-containing pigment. Considering that iron is not a typical contaminant of native gold, this evidence might indicate that an iron-containing pigment, such as Armenian bole, is present as a

ground layer under the gold. However, an image under 90 × magnification of the halo of Christ on f. 15r (Figure 2) shows that there is no preparatory layer beneath the gold. Instead, it seems that the artist used a light brown paint, most probably yellow ochre, in subtle brush strokes to give depth to the gilt areas or, in another instance, to define the folds of the tunic of Christ. The discrepancy between the XRF and FORS results can be explained by the spot size that is 3 and 1 mm respectively. The absence of a ground under the gold paint agrees with similar findings for both the *Rossano Gospels* [6] and the *Vienna Genesis* [7,8]. Nevertheless, further study of the manuscript is needed in order to more completely answer this question.

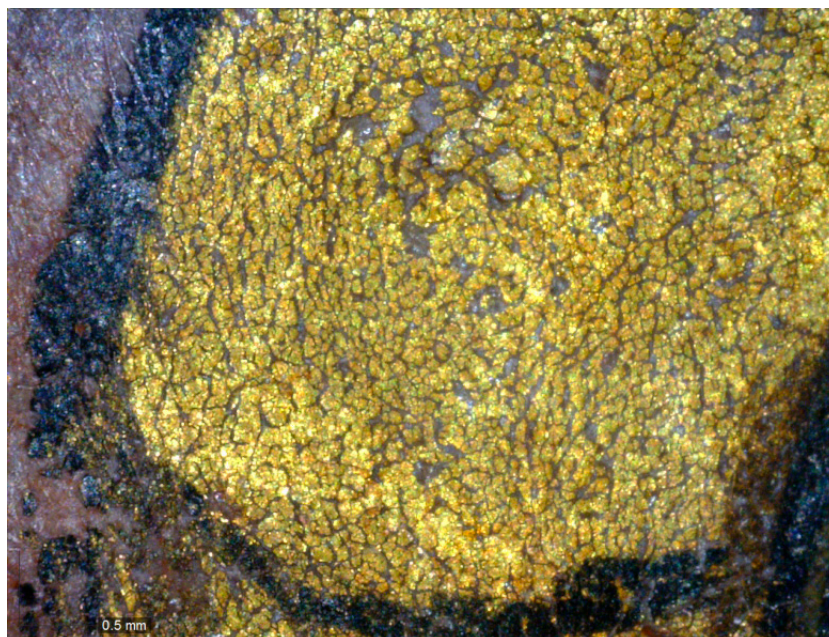


Figure 2. A 90× image of the halo of Christ on f. 15r with only the purple parchment, and no ground layer, showing through losses in the gold (same area as analysed by XRF).

3.5. Green

There are no true green colourants; instead all the green details were rendered with a mixture of indigo and orpiment, a bright yellow pigment obtained from the mineral of the same name. Indigo was identified according to the absorption at 650 nm in the FORS spectrum, while orpiment was identified according to the presence of As and S in the XRF spectrum. This mixture of a blue and a yellow colourant, known as *vergaut*, was very common in the early Middle Ages [9].

3.6. Orange and Red

A few details painted in red and orange were coloured with minium or red lead, a synthetic lead pigment typically found in medieval illuminations and identified according to the typical inflection point at ca. 560 nm in the FORS spectrum; the corresponding XRF spectra contained only Pb without significant amounts of Fe or Hg. The pigment appears to be in good condition, which is unusual due to its tendency to change from orange Pb_3O_4 to brown-black PbO_2 (plattnerite) or PbS (galena).

3.7. Pink

Architectural details (e.g., the throne of Herod on f. 10v) and garments (f. 15r) are painted with madder, a dye extracted from the root of *Rubia tinctorum* and other *Rubiaceae*. It was identified according to the typical absorption band structured into two sub-bands at 510 and 540 nm, and confirmed by spectrofluorimetry according to the strong emission at 600 nm. Madder was certainly used in

Byzantium for textile dyeing and for painting in the early Middle Ages [10], so its presence on the *Codex Sinopensis* is completely consistent. Hues rendered with madder vary from pink to purple.

3.8. Silver

The borders of the scrolls held by the prophets on ff. 10v, 11r, 15r, 29r and 30v, appearing dark grey, were not painted with a black pigment (e.g., carbon or iron gall ink) but rather with silver that contains a small amount of copper. Silver may also have been used in a miniature on fol. 11r, which is in very poor condition and was not analysed. The dark colour of the scroll borders and the presence of sulphur, evidenced by XRF analysis (Figure 3), indicates that the silver had tarnished to blackish Ag_2S , a typical alteration phenomenon. The XRF spectra have been normalised on the calcium content to minimise the variation in the thickness of the parchment.

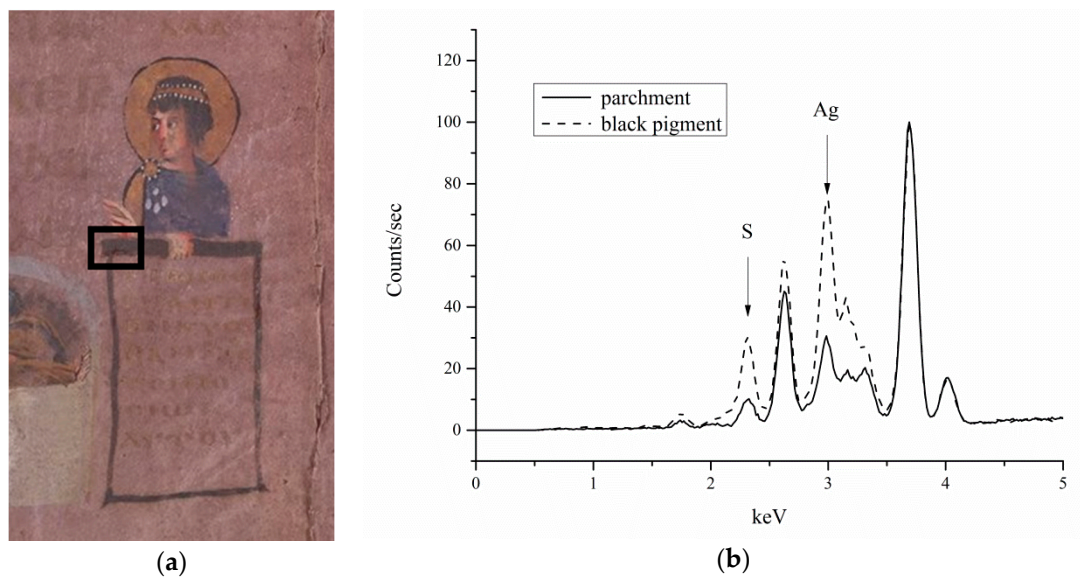


Figure 3. (a) The dark scroll on f. 10v with highlight of the area analysed by XRF; (b) XRF spectra from the black frame and from the parchment.

Splitting of the parchment along these blackened borders (see a 90× image in Figure 4) may have been exacerbated by the copper impurities in the silver.



Figure 4. A 90× image of the dark scroll border on f. 10v (same area as analysed by XRF).

3.9. White

White details were painted with lead white, identified by XRF according to the presence of Pb. Lead white was also mixed with other pigments.

3.10. Yellow

Several details, including the flesh tones (Figure 5, f. 15r), are painted with yellow ochre, in this case a yellower type of ochre. The identification was based on the same spectral features recorded for brown ochres. The choice to use yellow ochre as the base for flesh tones is unusual and contrasts with the techniques seen in the *Rossano Gospels* and the *Vienna Genesis*, where artists used mixtures of lead white and red lead, often with the addition of other pigments, to create predominantly pink tones for the flesh. The artist of the *Codex Sinopensis* used orpiment mixed with indigo to obtain green, but not on its own for yellow.

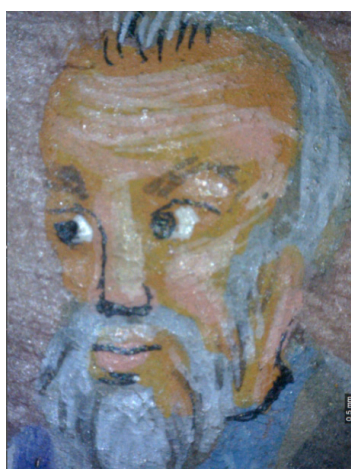


Figure 5. 40× image from a face on f. 15r.

3.11. Inks

The writing ink of the *Codex Sinopensis* is made of pure gold; impurities of silver or intentional additions of copper or other metals are negligible. The characterisation was based on XRF analysis.

3.12. Parchment

The purple colour of the parchment is the only feature of the *Codex Sinopensis* that has been previously investigated. In 1980, Thomas and Flieder [11] analysed a fragment of parchment by means of Gas Chromatography-Mass Spectrometry (GC-MS) and tentatively identified the presence of folium, the dye extracted from the *Chrozophora tinctoria* (L.) A. Juss. plant. Our non-invasive measurements did not confirm the presence of folium but instead suggested that orchil, a lichen dye, was present. In fact, the spectral features of both the FORS and spectrofluorimetric analyses are compatible with orchil and not with folium, according to the absorption bands at 545/588 nm, respectively, and to the emission band at 625 nm (Figure 6; reference samples were prepared as described in Aceto et al. [12]). In addition, this result is consistent with the identification of orchil in nearly all the other purple manuscripts analysed to date [13].

Orchil was in use at least since Greco-Roman times as it was cited by Theophrastus, Dioscurides, and Pliny the Elder for painting and textile dyeing. Several recipes describe its use as a substitute for the more prized Tyrian purple, e.g., in the 3rd century Greek Stockholm Papyrus manuscript. In the early Middle Ages, its main use was for the colouring of parchment for purple codices. It is not possible to determine whether the colour was applied by immersion in a bath or by painting, as there is no clear evidence of either method on the parchment surface.

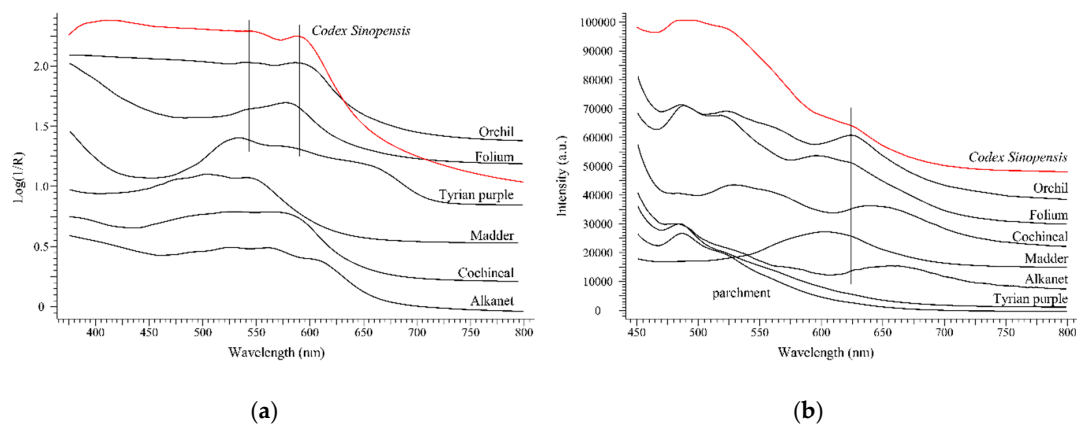


Figure 6. (a) Spectrophotometry with optical fibres (FORS) spectra of the parchment of *Codex Sinopensis* vs. reference samples of purple dyes; (b) Spectrofluorimetry spectra of the parchment of *Codex Sinopensis* vs. reference samples of purple dyes.

4. Conclusions

The *Codex Sinopensis* was decorated with colourants typical of Greek manuscripts produced during the Late Antique period. The presence of ultramarine blue clearly marks the difference of this palette in relation to that of early medieval artists working in Europe, who used indigo instead and started using the precious ultramarine blue at least two centuries later. It is of particular interest to compare the palette of the *Codex Sinopensis* with that of the *Codex Rossanensis* (data from [6]) and the *Vienna Genesis* (data from [7,8]) (Table 1).

Table 1. Comparison of the palette of the *Codex Sinopensis*, *Codex Rossanensis*, and *Vienna Genesis*.

Colour	<i>Codex Sinopensis</i>	<i>Codex Rossanensis</i>	<i>Vienna Genesis</i>
black	carbon *	carbon **	carbon * irongall ink *
blue	ultramarine blue * indigo *	ultramarine blue **	ultramarine blue * ultramarine blue mixed with Egyptian blue * indigo * azurite *
brown	yellow ochre *	yellow ochre **	earth pigments *
gold	shell gold ***	shell gold ***	shell gold ***
green	indigo */orpiment ***	indigo **/yellow ochre ** ultramarine blue **/yellow ochre ** indigo **/orpiment 1**	indigo */orpiment *** azurite */orpiment *** green earth * malachite 1*
grey	carbon */lead white ***	carbon **/lead white **	indigo */carbon *
orange	red lead *	red lead **	red lead *
pink	madder *	elderberry lake ** red lead **/white lead **	madder * red lead*/white lead ***
red	red lead *	red lead ** cinnabar 1**	red lead * cinnabar 1* red ochre 1*
silver	silver 2 ***	silver 3***	silver 3 ***
white	white lead ***	white lead **	white lead ***
violet	-	red lead **/ultramarine blue ** elderberry lake **/ultramarine blue **	madder */ultramarine blue * madder */ultramarine blue mixed with Egyptian blue * madder */indigo 1*
yellow	yellow ochre *	yellow ochre ** orpiment 1**	yellow ochre * orpiment 1***

¹ Occasional use. ² Used only for miniatures. ³ Used only for writing ink, not for miniatures. * Identification by means of FORS analysis. ** Identification by means of Raman analysis. *** Identification by means of XRF analysis.

According to the data reported, it is apparent that the colourants found in the *Codex Sinopensis* are similar to those used by the main artist who worked on the *Vienna Genesis* (note that at least seven different artists have been identified in the *Vienna Genesis* by their palette [8]) and, with slight differences, to those used in the *Codex Rossanensis*. Particularly relevant is the use of madder for a pink colour: the fluorescence spectral features of the madder used in the *Codex Sinopensis* and in the *Vienna Genesis* are very similar in terms of intensity and position of the emission band, which suggests that the main molecules composing the dye could be similar as well as the plant species from which madder was extracted.

Author Contributions: Conceptualization, M.A. and C.F.; methodology, M.A. and A.A.; investigation, E.C., G.F. and M.L.; resources, C.F. and C.D.; data curation, A.A.; writing—original draft preparation, M.A.; writing—review and editing, A.Q.; supervision, C.D. and A.Q. All authors have read and agreed to the published version of the manuscript.

Funding: This research received no external funding.

Acknowledgments: The authors would like to thank Laurence Engel, Présidente de la BnF, for allowing this technical study to have been undertaken on the manuscript.

Conflicts of Interest: The authors declare no conflict of interest.

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