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Paula Nabais
NOVA University of Lisbon

Cecilie Brøns
Ny Carlsberg Glyptotek, Copenhagen

Magdalena M. Wozniak
University of Copenhagen

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Investigating Organic Colorants Across Time: Interdisciplinary Insights into the use of Madder, Indigo/Woad, and Weld in Historical Written Sources, Archaeological Textiles, and Ancient Polychromy

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1. Introduction

Organic dyes have been used from the earliest times to provide color primarily to textiles, but also as a colorant in painting. Such organic dyes could create a wealth of colors, depending on the availability and know-how of resources. These dyes are usually organic in nature, and primarily obtained from different plant sources. Unfortunately, the characterization of natural organic colorants in textiles and artworks is still a challenge. The difficulty of analyzing these materials is sometimes allied to the frequent impossibility of micro-sampling, and the frailty of the objects. Many techniques, such as HPLC (High-Performance Liquid Chromatography) and SERS (Surface-Enhanced Raman Spectroscopy), require the use of a micro-sample, which cannot be recovered after analysis. Moreover, the portable technique Fibre Optic Reflectance Spectroscopy (FORS) can present some challenges in distinguishing between different dye sources belonging to the same molecular family, such as anthraquinone reds. Although no one technique alone can unravel the world of natural dyes, a multi-analytical approach has proven to be far more effective for their identification and characterization.

In the present article, we intend to share insights into three different perspectives and types of source material for the study of the use of organic colorants in ancient and historical times: The first case study presents an 18th-century historical recipe for dyeing textiles, the second case study presents a study of preserved archaeological textiles from Nubia, while the third case study presents the use of organic colorants for the polychromy of ancient Greco-Roman iconography.

These case studies are obviously very different, but they are chosen to illustrate the various source materials and the potential of incorporating the analysis of organic colorants. Moreover, the individual case studies illustrate the various methods used in the analysis of different types of material, as well as their limitations. Combining these approaches may give us a better understanding of the production and use of organic colorants during ancient and historical times as well as the representation of textiles in art. However, it is important to keep in mind that these varied sources are often limited, as we rarely have at our disposal a complete set of information. Allying these methodologies with scientific analysis may provide us with clearer insights into the natural colorants used in ancient and historical textile production and art.

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A wealth of organic colorants were available from antiquity to historical times. These were obtained from numerous sources, including various plants, fungi, lichens, and animal sources. However, to limit the scope of the present study, we focus on only three organic colorants, all obtained from plants: Indigo/woad, madder, and weld. Indigo/woad and madder have been widely used since antiquity, and are therefore obvious candidates for a comparative, interdisciplinary study. The present study is therefore not intended as a comprehensive presentation of all organic colorants used in ancient and historical times, but rather uses selected examples to highlight the potential of including dye analysis in research into ancient textiles and artworks, as well as illustrating the methods used and their implementation.

It is the authors' hope that this contribution will inspire other scholars of ancient and historical textiles to embark on studies of organic colorants and encourage interdisciplinary approaches to the study of ancient colors. This paper, therefore, begins with an overview and presentation of the various methods used to analyze and examine organic colorants in ancient and historical materials.

2. Natural organic colorants

Dyes are water-soluble substances, or substances which may be converted into a water-soluble form. They are mostly of organic origin, and have been used throughout history in three different ways:

i. As a direct dye, which binds directly to the textile;
ii. Complexed with a mordant, a metal ion (usually aluminum), which forms a bridge between the textile fibre and the dye;
iii. As a vat dye, which converts an insoluble dye into a water-soluble form.

Dye-mordant complexes are commonly mentioned in written sources, especially for red and yellow dyes, while the mechanism described for vat dyes is typical for indigo and shellfish (Tyrian) purple. Lichens, such as orchil, are usually used as a direct dye.[1] Besides being used in textiles, dyes could also be used in painting, applied as lake pigments, which are formed by the precipitation of the colorant with a complexing agent, such as alum, converting it into an insoluble pigment in a process analogous to the mordanting of textiles.[2] However, in Antiquity the dye solution could also be adsorbed onto a suitable substrate (such as chalk or white earth) without the reaction of alum, as is mentioned by Vitruvius.

Due to the diversity of natural dyes used for dyeing,[3] we have decided to focus our attention on four examples of some of the most-used dyestuffs in ancient and historical periods in Europe and North Africa: Indigo and woad for the blues, madder for the reds, and weld for the yellows. Because of the multitude of organic colorants used, this contribution will focus on these four only.[4] These were the most common blues, reds, and yellows used and described in European historical sources. For blues, indigo and woad were by far the most used sources. Moreover, as described by Dominique Cardon,[5] in historical times, 'red' meant madder, and weld was the equivalent for yellows that madder was for reds. It is important, however, to stress that many more sources of reds were used (kermes, cochineal, lac dye, *etc.*), as well as yellows (buckthorn berries, saffron, flax-leaved daphne, *etc.*), but that madder and weld were unquestionably unmatched in frequency of use. Moreover, dyestuffs were often mixed or used in combination, which was of course also the case for the colorants under study here.

2.1 *Indigo and woad*

The most significant blue dyestuff from antiquity up until the late 19th century was indigo, which is an organic colorant obtained from the plants woad (*Isatis tinctoria* L.) and *Indigofera* spp. Woad was used as a dyestuff in Europe since the Neolithic. The Greeks and Romans knew about a powerful blue dye from India called *indikon* (*ινδικόν*), Latinized *indicum*, (which gave it the modern name "indigo").[6] The ancient term originally meant a substance from India, indicating the import of indigo pigment to the Greco-Roman world. As an example, the *Periplus of the Erythraean Sea* records trade in *indikon* across the Indian Ocean.[7] The Sanskrit word *nila,* meaning dark blue, was used to designate indigo, which spread from India eastwards into Southeast Asia and westwards to the Near and Middle East, probably both through pre-Islamic trading routes, and with the subsequent diffusion of the product in the Islamic era.

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[1] Kirby *et al.* 2014.

[2] Kirby *et al.* 2014.

[3] Cardon 2007.

[4] For other dyestuffs used in ancient and historical periods, see, e.g., Cardon 2007.

[5] Cardon 2020.

[6] Cardon 2014, 327.

In the late 19th century, synthetic indigo was invented by the German chemist Adolf von Baeyer, and it almost entirely replaced the natural form obtained from indigo plants. In modern times, synthetic indigo is used worldwide as the primary blue dye in the textile industry, and it still has major importance in the 21st century.[8]

The molecule responsible for the intense dark blue color is indigotin (Fig. 1).[9] Unlike the other vegetal dyes, indigo is not soluble in water. It needs to be reduced in an alkaline environment so it can fix onto the textile fibre and then, through the action of oxygen, the pigment is regenerated.[10]

2.2 *Madder*

Dyer's madder is obtained from the plant *Rubia* spp., and has been one of the most-used dyestuffs since antiquity.[11] The root of the madder plant has been widely used for textile dyeing and painting for millennia.[12] Dyeing with madder was probably introduced into Egypt during the Eighteenth Dynasty (1550–1292 BC), most likely from the Levant.[13] Moreover, madder is mentioned in ancient sources such as Strabo and Dioscorides (*De materica medica* 6.3), who uses the term *erythrodanon*, and by Latin authors such as Pliny (*NH* 19.17), who uses the Latin term *rubia* in his description of the use of the roots to extract the dye. During the Medieval period, various names for madder were used.[14]

The Rubiaceae family includes several madder plants, although *Rubia tinctorum* L. is the best known. Other madder plants include Indian madder (*Rubia cordifolia*) and wild madder (*Rubia peregrina*).[15] The color of madder reds vary greatly, depending on the inherent ratios of its main chromophores, alizarin, purpurin, and pseudopurpurin, all belonging to the anthraquinone family (see Fig. 1), and it was used as a dye-mordant complex. The ratios of the different chromophores are also dependent on the age of the plant, how long it has been stored for, and the environmental conditions during its growth, among other factors. Like all natural dyes, the recipe used to extract the color, and the type and quantity of the mordant and other additives, will have a huge impact on the final color.[16]

2.3 *Weld*

Yellow dyes were used in artworks for millennia, up until advances in modern chemistry. *Reseda luteola* L., or weld, was one of the most important dyes in Europe up until the 19th century, and the primary source for organic yellows.[17] Yellow dyes were ubiquitous in the textile industry, not only for creating yellow colors but also as a source for preparing greens, oranges, and other shades. The main chromophores are luteolin and luteolin 7-*O*-glucoside, followed by luteolin 3′,7-*O*-glucoside, and a series of other compounds in smaller amounts. All the chromophores in weld belong to the class of flavonoids[18] (see Fig. 1), and similarly to madder, they are also used in the form of a dye-mordant complex.

Yellow dyes are particularly prone to degradation, making their identification in historical textiles more difficult than other dye sources.[19] Nevertheless, weld has been identified in textiles from Xinjiang,[20] in 17th-century Arraiolos carpets from Portugal,[21] and in Southern Swedish painted wall-hangings from the 18th–19th centuries,[22] as well as in 18th-century French tapestries,[23] testifying to its prevalent use throughout time and across civilizations.

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[7] Balfour-Paul 1998.
[8] Sousa *et al.* 2008.
[9] Kirby *et al.* 2014; Sousa *et al.* 2008.
[10] Cardon 2007.
[11] Cardon 2007; Sandberg 1994; Donkin 1977.
[12] Daniels *et al*. 2014, 13; Cardon 2007, 107–123.
[13] Daniels *et al*. 2014, 13.
[14] Schweppe 2012, 109.
[15] Cardon 2007; Donkin 1977; Schweppe 2012, 115–116.
[16] Cardon 2007.
[17] Cardon 2007.
[18] Santo *et al.* 2023; Veneno *et al*. 2021.
[19] Sharif *et al*. 2022.
[20] Zhang *et al.* 2008, 35, 1095–1103; Liu *et al*. 2011, 38, 1763–1770.
[21] Marques *et al*. 2009, 1216, 1395–1402.
[22] Nyström *et al*. 2015, 60, 353–367.
[23] de La Codre *et al.* 2020, 46, 613–622.

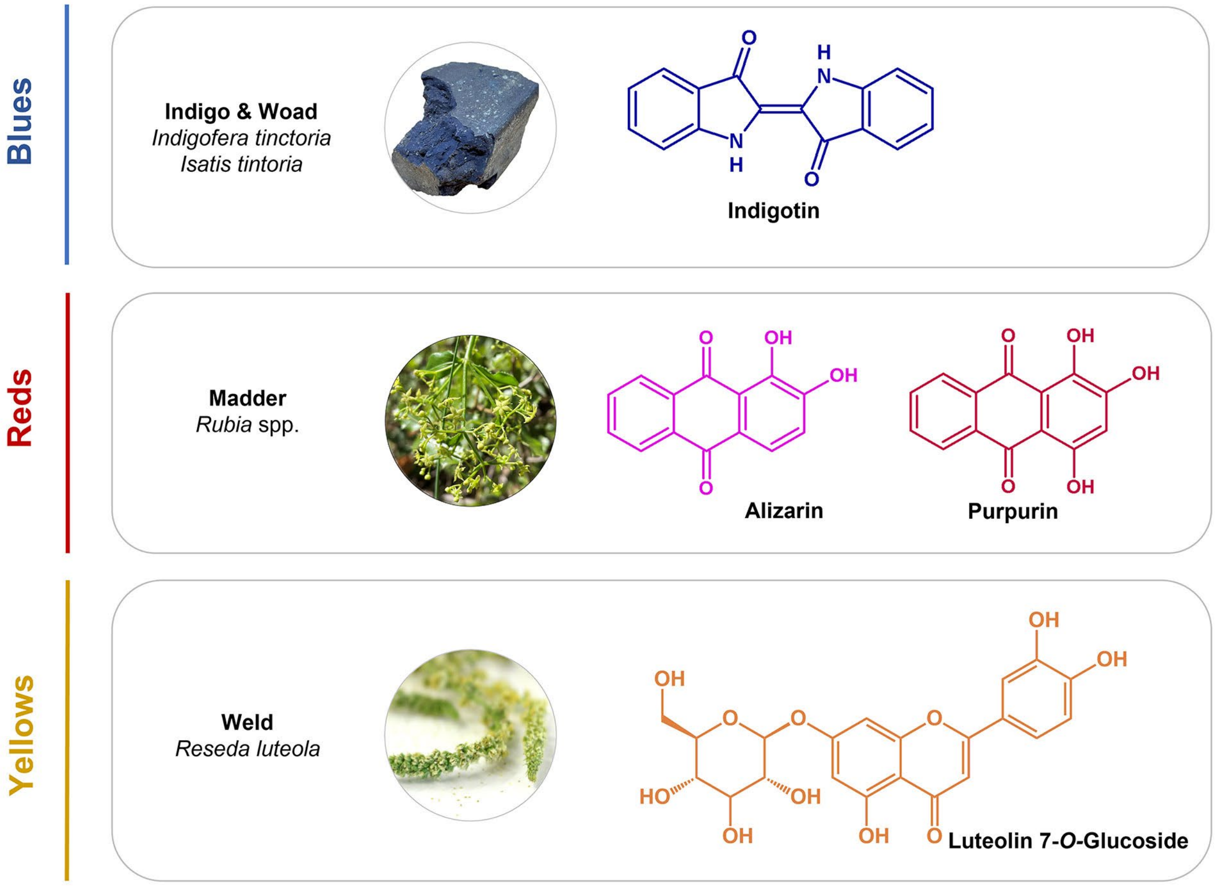

Fig. 1: Structures of the main chromophores for indigo and woad blues, madder reds, and weld yellows, © P. Nabais.

3. Methods for the analysis of organic colorants

The characterization of natural organic colorants in textiles and other types of artifacts continues to be a challenge. The difficulties in analyzing these materials are oftentimes connected to the impossibility of micro-sampling and the frailty of the artworks.

3.1 *High-Performance Liquid Chromatography (HPLC)*

One of the main techniques used for the characterization of organic colorants is High-Performance Liquid Chromatography (HPLC), which, coupled with Diode-Array Detection (DAD) and mass spectrometry, is a very robust technique for the identification of organic colorants.[24] HPLC is generally the most extensively used method for the characterization of textile dyes. This method allows the characterization of all chromophores present, enabling the identification of the dyestuff. HPLC did require a relatively large sample, up until the very recent development of ultra-high-performance liquid chromatography (UHPLC), which has improved in sensitivity thus enabling a smaller sample size.[25] However, for some artworks, the frailty of the object, which is often a consideration for archaeological and historical textiles, prevents us from removing samples large enough for this analysis.

3.2 *Raman Spectroscopy and Surface-Enhanced Raman Spectroscopy (SERS)*

Raman microscopy is another technique commonly used for the characterization of organic colorants. A challenge with this method is that for most organic colorants of natural origin, their intrinsic intense fluorescence may mask the chromophores' Raman signal, hence preventing their identification. This can be overcome by Surface-Enhanced Raman spectroscopy

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[24] Degano *et al*. 2009; Wouters 1985; Zhang *et al*. 2005; Wouters *et al*. 2011; Devièse *et al.* 2011; Degano 2018.

[25] Degano 2018.

(SERS) and other methods.[26] SERS uses silver or gold nanoparticles to quench the fluorescence and enhance the Raman signal, providing a fingerprint identification of the chromophores present. However, it requires a micro-sample that cannot be made available for further analysis.[27]

3.3 *Fibre-Optic Reflectance Spectroscopy (FORS)*

Over the past decade, the development of analytical techniques has proved useful to overcome some of the challenges when analyzing artworks which cannot be sampled. Fibre-Optic Reflectance Spectroscopy (FORS) has proven to be a very valuable technique, enabling the identification of some colorants, namely pigments.[28] However, the identification of many organic dyes can be somewhat challenging, as is the case for red anthraquinone dyes: FORS may be able to distinguish between animal or vegetal-sources, such as cochineal or madder, but not between dyes from a similar source (*e.g.* animal source), such as cochineal and kermes. Moreover, the fact that yellow dyes share many of the main chromophores, *i.e.* flavonoids, makes it difficult, if not impossible, to determine the dye source using this technique.

3.4 *Reflectance hyperspectral imaging*

Reflectance hyperspectral imaging has been recently developed, allowing the identification and analysis of the spatial distribution of colorants without the need to micro-sample, by collecting continuous calibrated spectral images which provide reflectance spectra for every pixel of the image.[29]

3.5 *Microspectrofluorimetry*

Microspectrofluorimetry (in the visible) has been recently explored as a method for the characterization of organic colorants in artworks. It presents several advantages compared with other techniques, such as the simultaneous acquisition of emission and excitation spectra, offering high sensitivity and selectivity, combined with good spatial resolution, and the possibility of in-depth profiling. It can also be used in situ, without any contact with the sample or artwork that is being analyzed.[30] Because the signals obtained are incredibly complex and are sensitive to the environment surrounding the chromophore, such as the pH, additives, and method of extraction, among others, it may be difficult to interpret the data. Therefore, the complementary use of fingerprinting techniques, such as Surface-Enhanced Raman Spectroscopy (SERS), or infrared spectroscopy, has proven to be a robust methodology for not only the identification of the dye source, but also of specific recipes for the same dye.[31]

3.6 *Reconstructions of ancient and historical dye recipes*

Finally, although not a method of analysis per se, the complementary use of analytical methods and accurate reconstructions of historical dye recipes has proven to be extremely useful in the characterization of natural organic dyes in artworks. This also contributes to research into written sources describing technological or scientific knowledge, such as recipe books. The methodology of preparing historically accurate reconstructions of dyes enables the production of reference materials with as much historical accuracy as possible. These references are validated by their "closeness to the true value", the historic material or artwork under study. When studying archaeological and historical materials, it is essential to have access to their methods of production, since today's materials do not represent the formulations used in the past.[32] Hence, even though we will never be able to fully reproduce some parts, these dye reconstructions are prepared according to historical recipes described in written sources, providing us with the closest reference materials to what we might find in artworks.

To conclude, no one technique alone can solve the identification difficulties for natural dyes in historic artworks, but a synergy of complementary techniques is the best approach.

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[26] Rosi *et al*. 2010.

[27] Leona *et al*. 2009; Pozzi 2011.

[28] Picollo *et al*. 2018.

[29] Ricciardi *et al*. 2012; Rosi *et al*. 2013; Delaney *et al*. 2014; Cucci *et al*. 2015.

[30] Melo *et al*. 2010; Claro *et al*. 2008; Claro *et al*. 2010; Castro *et al*. 2014; Nabais *et al*. 2018; Nabais *et al*. 2021.

[31] Nabais *et al*. 2018; Nabais *et al*. 2021.

[32] Carlyle & Witlox 2005.

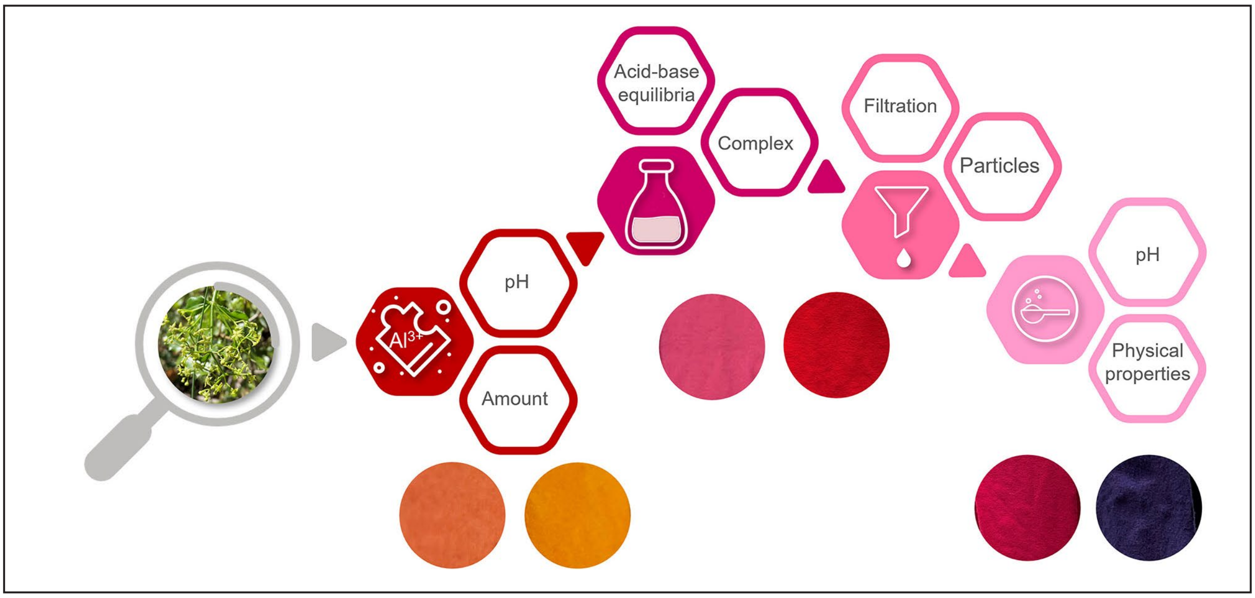

Fig. 2: Organic colorants can provide a huge range of shades and hues. Master dyers knew this, and so they carefully engineered each recipe to produce a very specific color. The mordant type, the amount, the pH of the mordant and the dye bath, the filtration process, and the addition of other materials, will all influence the final color obtained. These are insights that we can gain by reproducing known historical recipes. Represented here are the various hues obtained for madder recipes by changing a few steps within the recipe.

4. Madder, indigo, woad, and weld in historical written sources

While the multi-analytical characterization of archaeological and historical textiles provides important clues on how the textiles were produced and dyed, written sources provide additional evidence. Written documents describing dyeing recipes and processes are valuable sources of information, as they illustrate the practices in use during a particular time. These are sources of technical information contemporary to the historic material under study and may provide a deeper insight into its production. Therefore, the experimental execution of preserved historical recipes that have reached our time can contribute to several areas of heritage research:

i) improved understanding of the *savoir-faire*, through the study of the history of science and technology;
ii) the development of analytical techniques for the identification of specific materials, *e.g.* natural colorants, through the use of references close to historic artworks;
iii) better understanding of the degradation mechanisms and characterization of markers for aged natural colorants;
iv) development of better conservation strategies.

Accurate reconstructions of historical dye recipes may help us understand the recipe and the ingredients used to produce a particular color. Each ingredient and each step of the recipe influences the final color obtained. Hence, the formulation (recipe) can provide a tremendous amount of information on the preparation of dyed textiles and their makers (Fig. 2).

Likewise, as previously mentioned, the reconstructions of historical dyes can also provide insight into degradation mechanisms, providing material for future research into conservation.[33] To understand the degradation mechanisms that are in play in such complex matrices as found in our cultural heritage, it is necessary to have reference materials prepared with as much historical accuracy as possible. These are used to assess the natural evolution of these colors, and simulate, by accelerated aging experiments using a limited number of variables, the aging of these systems. On the other hand, art reconstructions depend on the availability of historical sources, which are scarce for the Classical and Medieval periods, and the Renaissance. Only a small number of treatises are available, which creates a gap in material and technical art history. Several researchers have contributed to making ancient and medieval texts available.[34] The translations and

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[33] Oltrogge 2005; Wallert 2005.

[34] Clarke & Carlyle 2005; Merrifield 1999; Melo & Castro 2016; Clarke & Carlyle 2005; Carlyle & Witlox 2005.

critical edition of the *Liber diversarium arcium*, by Mark Clarke, is an example, as is the Cologne database for painting materials and reconstructions, by Doris Oltrogge.[35]

Considering the economic and social importance of the textile industry, it is no wonder that written records of the technical procedures and recipes for dyeing are abundant.[36] Nevertheless, most technical sources are often just a compilation of recipes, without any systematic and critical transcription and organization. This adds complexity to their understanding and interpretation.

Dominique Cardon has been a pioneer in this research field, taking the first steps in the systematization of textile dyeing recipes, and paving the way for future research.[37] Her recent books on the French master dyers Antoine Janot and Paul Gout are a perfect example of this systematic research. These master dyers specialized in the piece-dyeing of fine wool broadcloths manufactured in Languedoc (Southern France) for exportation to the Levant. They both wrote treatises similarly entitled *Mémoires de Teinture* (Memoirs on Dyeing), illustrated with dozens of dyed textile samples. Antoine Janot's treatise is dated to 1744, while Paul Gout's is from 1763.

These books are full of extremely valuable information and are unique for their time: The books are composed of carefully described recipes for every color, each preceded by a sample swatch. This gives us the opportunity to reproduce the colors from these books and compare the analytical results to the original swatches. The books are, at the same time, a written record and an artwork, providing a complete feedback loop. They were written at a time that was considered the apogee of the organic dyeing industry, after millennia of perfecting recipes and formulations, and just before the advent of synthetic dyes in the 19th century. Moreover, they were conceived by 18th-century master dyers who were knowledgeable about their crafts: Antoine Janot owned a very successful dyeing workshop, while Paul Gout was the master dyer at a Royal Manufactory of wool broadcloths.

Although there are more examples of such complete case studies, such as the Crutchley archive,[38] they mostly correspond to the 17th–18th centuries. Before this, the written sources lack swatches, and are more difficult to interpret with their increasing distance from contemporary times. As an example, the *Liber diversarium arcium*, translated and edited by Mark Clarke,[39] is an excellent source for 14th-century recipes for artists' materials, but the information it contains is less clear or even obscure. It includes a chapter on dyes for wool, linen, and skins, to dye red, green, yellow, purple, and black. In this treatise, although some of the recipes provide measurements, the nomenclature of certain ingredients can be more difficult to interpret. This is the case for the mastic added for the red color (presumably mastic galls) or *glaucus*, the name given to the yellow color dyed with *herba* (possibly weld). Moreover, without swatches, the subjective nature of color is left open to interpretation: "… if you want a more intense green, put repeatedly in indigo, if a little, a little." (§4.20.1B).[40]

To go further back, the Leiden and Stockholm papyri, two fragments belonging to the 3rd century AD, must be addressed; due to their pre-Medieval production, they are frequently cited and very relevant for medieval texts.[41] In these cases, the texts are far less clear, with the names of the materials or the way they were processed being very difficult to interpret. These written sources are also a reflection of their time, with the majority of the recipes for dyeing being exclusively dedicated to the preparation of purple colors.

4.1 *Woad and indigo in historical manuscripts*

In the Stockholm papyrus, there is a long description of how to prepare woad for dyeing, as well as the vat.[42] It is a very complex recipe, taking several days for the vat to be ready for dyeing: "… Dye in blue twice a day, morning and evening, as long as the dye liquor is serviceable." The *Liber diversarium arcium* has no recipe for the preparation of the vat, although it mentions the use of indigo for green dyes.

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[35] Oltrogge 2005; Clarke 2011.

[36] Kirby *et al*. 2014.

[37] Cardon 2013; 2020; Cardon & Bremaud 2020; 2022.

[38] Quye *et al*. 2020.

[39] Clarke 2011.

[40] Clarke 2011, 151.

[41] Radcliffe 1926; Radcliffe 1927.

[42] Radcliffe 1927.

ANTOINE JANOT

Aile de corbeau | Crow's wing
Bleu turquin | Turkish blue
Bleu de Roy | King's blue
Bleu céleste | Celestial blue
Bleu d'azur | Azure blue
Bleu de ciel | Sky blue
Bleu mignon | Cute blue
Bleu déblanchi | Off-white blue

PAUL GOUT

Bleu pers | Persian blue
Bleu turquin | Turkish blue
Bleu de Roy | King's blue
Bleu tanné | Tanned blue
Bleu d'azur | Azure blue
Bleu céleste | Celestial blue
Bleu mignon | Cute blue
Bleu de lait | Milk blue
Bleu déblanchi | Off-white blue

The blue was obtained by immersing the cloth in the woad vat with the addition of indigo pigment. The different shades of blue is entirely dependent on the concentration of the vat, but also the number of times and for how long the cloth is immersed in the vat.

ANTOINE JANOT

Rouge de garance | Madder red
Rouge bruni | Saddened red
Couleur de Roy | King's colour

PAUL GOUT

Garance | Madder red
Garance cramoisillé | Crimson madder
Rouge bruni | Saddened red
Couleur de Roy | King's colour

As stated by Dominique Cardon, "*madder reds are the only colours English dyers call "red" and French dyers call "rouge*"(…)". Madder was indeed a very important dye source, to the extent that Antoine Janot dedicated an entire *memoir* to the dye plant. The reds were prepared using a mordant bath of red tartar and alum.

ANTOINE JANOT

Jaune | Yellow
Jaune citron | Lemon yellow
Jaune soufré | Sulphur yellow
Jaune paille | Straw yellow

PAUL GOUT

Jaune | Yellow
Limon | Lemon
Jaune | Yellow (with old Weld 68%)

Weld yellows were prepared by a mordanting bath of red tartar and alum (and bran in Antoine Janot recipes); followed by a dye bath using different proportions of weld. This dye bath was called *Gaudage*, as in *Gaude* (Weld).

Fig. 3: Blue (woad, left), madder reds (center) and weld yellow (right) reproductions of Paul Gout's recipes, prepared as part of the workshop Les 157 couleurs de Paul Gout, organized by the Vieilles racines et jeunes pousses, and Mara Santo's MSc thesis. For each color the main recipes described by Antoine Janot and Paul Gout are provided, as well as the general procedures. The complete description of the recipes can be found in the books by Dominique Cardon and Iris Brémaud, "Le Cahier de couleur d'Antoine Janot", CNRS 2020, and "Les 157 couleurs de Paul Gout", Vieilles Racines et Jeunes Pousses, 2022.

In the 18th century, Janot and Gout presented a scale for blues made with woad, the common source of indigotin in Europe. They describe the preparation of the vat, and each successive dip into the vat would give a darker shade of blue to the broadcloth. This scale for blues has been common since Medieval times.

4.2 *Madder in historical manuscripts*

A recipe for the preparation of a *Rose color* using madder is already to be found in the 3rd century in the Stockholm papyrus, indicating the use of this dye since ancient times. This is, as expected, very difficult to interpret, with unknown ingredients (*choenix*

Fig. 4: Set of samples produced by the Royal Manufacture of Wool of Portugal, in the 18th century (left) in the National Archives of Torre do Tombo, and the Royal Manufacture of Covilhã (right).

of bean meal, white oil) and obscure steps (*Smear the rolls of wool with ashes, untie them, and wash the wool in the liquid from potter's clay*).

The *Liber diversarium arcium* is, on the other hand, more complete, with measures given for both the cloth and all the ingredients.[43] It is a recipe which, although requiring several steps (premordanting with mastic galls and mordanting with alum overnight; the dyeing is done with galls and madder; the finishing of the colour includes sieved ashes), is possible to reproduce.

In the 18th century, despite describing the use of cochineal, both dye masters gave the throne to madder: They both devote one entire book to the production of madder reds. These colors are the reflection of a much more modern textile industry, with ingredients which were not described in more ancient sources, such as tin liquor.

4.3 *Weld in historical manuscripts*

Finally, for the yellows, the situation becomes more complicated the earlier the source. The Stockholm papyrus only mentions a dark yellow being prepared using golden litharge, and no plant-based yellows appear to be mentioned. In the *Liber diversarium arcium*, on the other hand, *glaucus* is prepared by using *herba*, which is presumably weld. This was also used to prepare the greens mentioned. In the treatise, another source of yellow is mentioned, young fustic, but it is not associated with the term *glaucus*, possibly indicating that the author was differentiating the weld yellow from young fustic.[44]

In the 18th century, weld was either the only source, in Janot, or the main source, in Gout, followed by other yellows such as flax-leaved daphne (*Daphne gnidium*)[45] (Fig. 3).

Considering the valuable source of information that the 18th-century French treatises present, project REVIVE, "The threads of the past weaving the future: The colours from the Royal Textile Factory of Covilhã, 1764–1850,"[46] plans to address this complexity within the Portuguese Manufactory of Wool in Covilhã, following Cardon's pioneering work. REVIVE intends to analyze the contribution of the French wool industry, as the Marquis of Pombal was inspired by the Manufacture des Gobelins, in Paris, during the construction of the Covilhã Factory (Fig. 4). It will be crucial to understand the French transfer of technology in synergy with the Royal Factory's own identity, its singularities, and specificities. REVIVE will also investigate the know-how brought by the network of foreign masters hired to work in the Covilhã Factory.[47]

As Dominique Cardon states, "*as unique sources of inspiration, the ancient colours represented* (...) *are meant to serve as starting points for research and experiments to emulate the chromatic richness and subtlety of natural*

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[43] Clarke 2011, 150.

[44] Clarke 2011, 151.

[45] Cardon 2013; 2020; Cardon & Bremaud 2020; 2022.

[46] 2022.01243.PTDC, funded by the Portuguese Foundation for Science and Technology.

[47] REVIVE project: https://sites.google.com/view/reviveproject/home, accessed 19 February 2024.

Fig. 5: Two shepherds — a detail from a Nativity scene, Faras cathedral, end of the 10th century AD. Sudan National Museum, Khartoum, inv. 24365 © M. M. Wozniak, courtesy of the SNM.

dyes (…).”[48] This is also the ultimate goal of REVIVE: To bring these natural colors back to life, by reproducing and translating these complex procedures so that the general public, designers, and artists can reinvent them.

5. Madder, indigo/woad, and weld in archaeological textiles: Case studies from Nubia

In the field, it is rare for a researcher to have access to all relevant sources of evidence simultaneously: Written sources for the dyeing recipes, the dyeing installations, residual traces of the baths, and the dyed textiles themselves. More often, insights into the dyeing process derive from one, usually fragmentary, source of evidence. Indeed, this medium is not necessarily direct evidence, *i.e.* a painting showing people dressed in colored clothes (Figs. 5 and 6).

Thanks to beneficial environmental conditions in the Middle Nile Valley, organic material, including textiles, is often well preserved. While we do not have a one-to-one match with the costumes represented in the paintings, due to the generally fragmentary condition of the excavated textiles, we have many dyed textile fragments representing the colors documented in the paintings, including reds, yellows, blues, and greens (Fig. 7).

The technical features of the archaeological fragments — wool fibre, S-spun threads, tabby weave, reinforced selvages, sometimes corded edges — point to local Nubian textile production.[49] Regarding the decoration, colored bands are the most common pattern documented in the archaeological textiles; although more rare, fragments with plain colored ground are also attested. Since the written sources from medieval Nubia are very limited, there are no documents currently known that inform us about the dye sources used by medieval Nubian dyers to produce red, yellow, green, and blue clothes. In the settlements excavated so far, there is no evidence of larger installations dedicated to textile dyeing either. The textiles are, for now, the sole and most direct evidence of dyeing activity

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[48] Cardon & Bremaud 2020.

[49] Bergman 1975.

Fig. 6: Dance scene, Old Dongola, Southwest Annex, room 5, north wall, in situ. © M. M. Wozniak, courtesy of NCAM & PCMA UW.

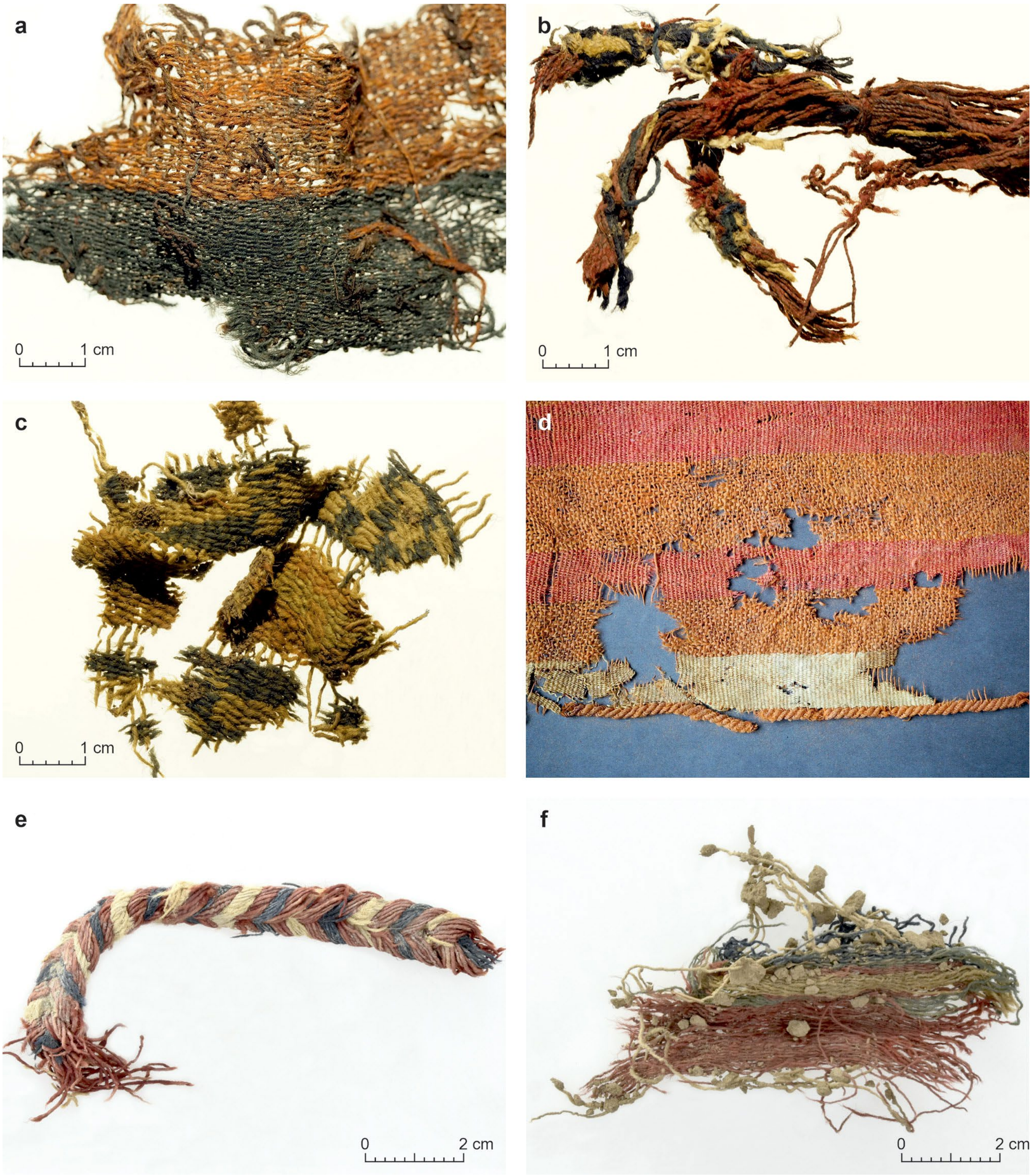

Fig. 7: Dyed textiles from Medieval Nubia (a–c: Meinarti, d: es Sadda 1, e–f: Old Dongola). a–d © M. M. Wozniak, a–c courtesy of the Sudan National Museum; d courtesy of Poznan Archaeological Museum; e–f © M. Skarżynski, courtesy of PCMA UW.

as practiced in medieval Nubia. However, as with the paintings, an optical examination alone gives us no data for identifying the dye source. At best, with the help of a magnifying glass or microscope, the threads can be examined to ascertain whether the fiber was dyed before or after spinning (Fig. 8).[50]

5.1 *Dye analyses of textiles from Nubia*

To identify the dye sources used by Nubian dyers, archaeological samples from medieval Nubia have recently been analyzed with HPLC-MS.[51] In almost all the samples, it was possible to detect the chemical

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[50] So far, piece-dyeing has not been attested in the local textile production. This is a personal observation of the author (MMW), based on the documentation of c. 2500 textile fragments from different Nubian medieval sites.

[51] Wozniak *et al.* 2021.

Fig. 8: Wool fibers dyed before (a–b) and after spinning (c–d), with close-ups to show the various degrees of penetration of dye into the wool fibers (e–f). a, e and f from Old Dongola (© M. M. Wozniak, DinoLite captures, courtesy of PCMA UW); b, c and d from Meinarti (© B. Czaja, DinoLite captures, courtesy of the SNM).

compounds characteristic of plant dyes such as alizarin and purpurin (which are markers of red dyes), apigenin and luteolin (which are markers of yellow dyes), and isatin and indigotin (characteristic for blue dyes). These compounds are common to species growing in Europe, Africa, and Asia.

The identification of the precise species for each plant was delicate and challenging because there is no written evidence of any dyeing recipes used in ancient and medieval Nubia. Moreover, the accessible documentation about local dyeing plants is very limited. In this investigation, the encyclopedic work of

Fig. 9a: Wool textile from Meinarti, Lower Nubia. Sudan National Museum, Khartoum, inv. 18124 © M. M. Wozniak, courtesy of the SNM.

Dominique Cardon (2007) was very helpful for identifying the most probable dye plants used, based on their geographical habitat: We could limit ourselves to the wider range of plants known to be endemic species of the Nile Valley.

5.2 *Madder in Nubian textiles*

For example, we were able to analyze a fragment of red wool thread from a fragmentary textile excavated at Meinarti, Lower Nubia (Fig. 9a). Two main compounds were identified in this sample, namely alizarin and purpurin (Fig. 9b). These markers are characteristic for madder-type roots (*Rubia tinctorum, Rubia cordifolia*) but also for our lady's bedstraw (*Galium*). However, our lady's bedstraw does not grow in Africa, while madder (*Rubia tinctorum*) is indeed an endemic species in North Africa and the Nile Valley.[52] Hence, for this sample, we proposed *Rubia tinctorum* as the most probable dye source used to obtain the red wefts of this textile.

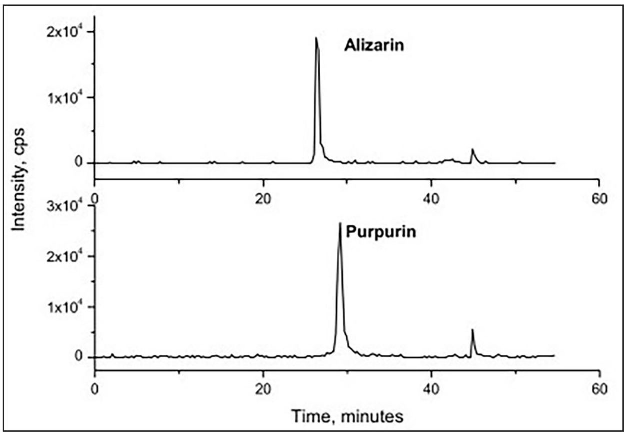

Fig. 9b: Chromatogram for sample from textile inv. 18124 © M. Biesaga.

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[52] Cardon 2014, 123, Fig. 1.

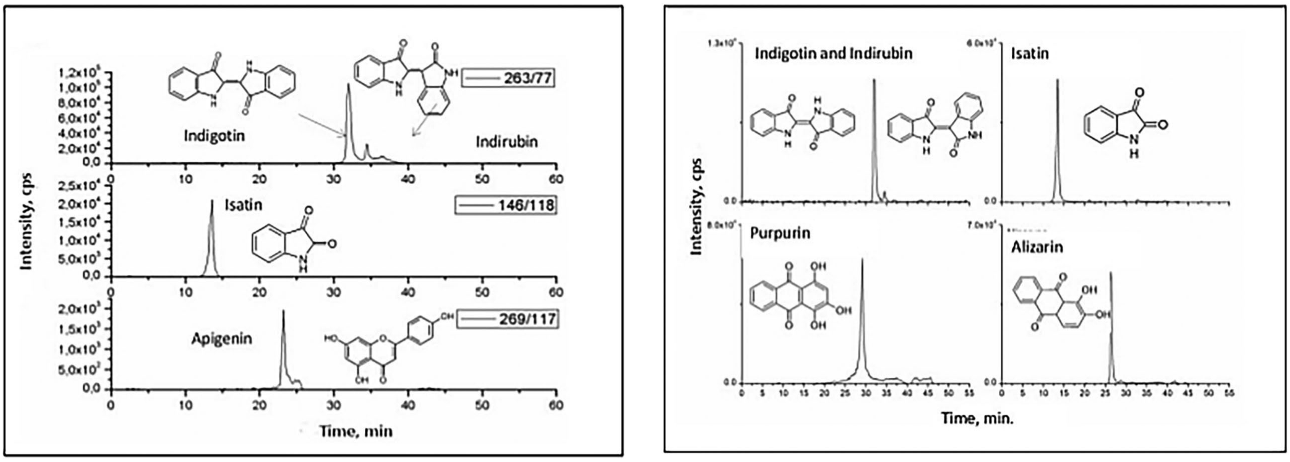

Fig. 10: Chromatograms showing the difference in pick between *Isatis tinctoria* (left) and *Indigofera tinctoria* (right) © M. Biesaga.

5.3 *Woad/indigo in Nubian textiles*

Another challenging interpretation is the presence of isatin and indigotin, which are characteristic for both woad (*Isatis tinctoria*) and indigo (*Indigofera tinctoria*); usually it is not possible to distinguish between these two dye sources. Moreover, as both species can be found in the Middle Nile Valley,[53] geographical provenance cannot be used as a valid criterion for a more precise identification of the dye source. Recently, a method was elaborated by Witkowski *et al.* to tentatively differentiate woad and indigo, based on the isatin/indigotin ratio: If the ratio is above 0.5 it would indicate *Indigofera tinctoria* as the dye source, while low values, below 0.2 would point to the use of *Isatis tinctioria* (Fig. 10).[54] Thanks to this method, it appeared that the majority of our samples had been dyed with woad (*Isatis tinctoria*). The textiles' provenance from Lower Nubia may be significant, but more research needs to be done before confirming a possible regional feature. Interestingly, *Indigofera tinctoria* has also been identified for the first time in a textile excavated in the region of the Nile's 4th Cataract, revealing that, at least in Late Antiquity, two different sources of blue dye were probably known. However, further analyses are needed on additional textiles to confirm the use of *Indigofera tinctoria* in this geographical area.

5.4 *Weld in Nubian textiles*

In some threads, apigenin and luteolin were detected; they are generally considered as markers of weld (*Reseda luteola*),[55] although they are also present in other plants such as dyer's broom (*Genista tinctoria*) or saw-wort (*Serratula tinctoria*).[56] But, among these plants, only weld grows in the area under study. Interestingly, weld often appears only in trace amounts, and was rarely applied as the main dye; it was used mostly in combination with madder and/or woad, to brighten or intensify the red color or to create vivid greens.

5.5 *Research into the dyeing of Nubian textiles*

In a situation where direct evidence for dyeing activity at archaeological sites and in the written sources is absent, dye identification analyses applied to archaeological textiles are the primary method to gain knowledge about landscape resources and the technical *savoir-faire* of the medieval Nubian dyers. HPLC-MS has revealed information about local dye plants, and how to use them, by mastering complex processes such as mordanting and vat dyes. The variety of colors still visible in the textiles also attests the dyers' ability to experiment with dyes, and develop a richer chromatographic palette composed of red, orange, yellow, pink, dark blue, light blue, green, or even black.

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[53] Cardon 2014, 347, Fig. 24.

[54] Witkowski *et al.* 2017; Laursen & Mouri 2021.

[55] Colombini *et al.* 2007; Degano *et al.* 2009; Ferreira *et al.* 2004.

[56] In general, it is usual for other markers to be detected as well, such as genistein (dyer's broom) or methyloquercetin and kaethnoempferol (saw-wort), which allows for a more precise identification.

In the future, close cooperation between archaeologists, botanists, and chemists is planned to provide a detailed geographical mapping of the dyeing plants from the Middle Nile Valley and improve our knowledge of local flora. Further research on the regional availability and seasonality of dyeing plants could be indicative of the degree of specialization within the dyeing activities: Were dyers working exclusively with woad, or did they master different dyeing techniques (dyeing with mordant, vat dye) according to the different plants they had access to?

6. Madder and indigo in ancient polychromy

6.1 *Madder in ancient polychromy*

Madder extracts were not only used for dyeing textiles, but also for preparing pigments for painting. It appears to have been a very common colorant in ancient polychromy and was used to paint different types of artifacts made of various materials, including stone, terracotta and wood.

Because most colorants, including madder, are soluble, they cannot be mixed directly with a binding medium, and therefore cannot be used as a pigment (with the exception of indigo and saffron). Therefore, pigments prepared from soluble, natural colorants, such as lake pigments, are generally formed by extracting the colorant components of the original material, such as the madder root, into water and then precipitating (or adsorbing) the dye onto a colorless or white, insoluble substrate.[57] The white inorganic substrate could be chalk, limestone, ground shells, gypsum, white clay, earth, or the so-called *creta anularia*, which is a mixture of ground glass and chalk. A solid pigment could also be produced by allowing the colorant solution to evaporate, leaving a solid pigment for painting.[58] However, this method of producing lake pigment would require large amounts of madder root, and the resulting pigment would probably not have had much body, nor the bright pink color as is often seen in ancient polychromy.[59] The shades of madder lakes vary from scarlet, carmine red, and pink, to red with a slight bluish tint,[60] the most common being rose-red and pink hues.

As described above, a number of methods can be used to analyze the organic colorants in lake pigments, including Raman spectroscopy and HPLC. However, both methods require samples, which is not always possible for all cultural heritage artifacts. Therefore, for the study of ancient polychromy, UV-induced fluorescence photography is a very useful method as it is non-invasive, and relatively easy to carry out. UVF-photography can reveal the presence and distribution of fluorescent materials, including organic red lakes, such as madder lake. Madder contains purpurin and pseudopurpurin, which both glow a bright coral-red color when exposed to UV light. Hence, when using UV-induced fluorescence photography (with a specific spectrum) on ancient artifacts, red fluorescence most likely indicates the presence of madder, although other organic red lakes from scale insects, such as kermes (from *Kermes vermilio*), Armenian cochineal (from *Porphyrophora hamelii*), and lac (*Kerria lacca*), might be present. These scale insect lakes were used in the ancient world, but there are still only extremely few attestations of such lakes in ancient polychromy, presumably due to the analytical complexities required for their identification.[61] Moreover, such insect colorants were presumably much more costly compared to madder lake, which could also partly explain their rarity in polychromy. One such rare example is the identification of lac in the polychromy of a Hellenistic *oinochoe* (wine jug) from Puglia, Italy.[62]

Since these animal-based lakes are so far only very rarely attested in ancient polychromy, it is usually assumed that the coral-red fluorescence indicates madder lake. This is supported by the fact that the other anthraquinone reds do not fluoresce orange/coral-red in the same way.[63] Moreover, besides organic red lakes, UVF-photography can illustrate the use of a vast number of organic adhesives and surface coatings of original and secondary origin. It is therefore important to stress that the fluorescence

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[57] Kirby *et al.* 2014, 28.

[58] Daniels *et al*. 2014, 17.

[59] We are grateful to Jo Kirby Atkinson for this comment.

[60] Schweppe & Winter 1997, 109.

[61] Insect dyes can be identified by HPLC-DAD. However, as mentioned, this requires sampling, which is not always possible. Moreover, in the case of encaustic paintings, it is advisable to remove the wax from the sample.

[62] Dyer *et al.* 2018.

[63] However, kermes fluoresces slightly pink, while safflower red fluoresces an orange-red color. We are very grateful to Jo Kirby Atkinson for this information.

Fig. 11: Ultraviolet fluorescence (UVF) imaging can detect organic luminescent materials such as lake pigments, and is often used for the examination of mummy portraits. Left: Mummy portrait of a young woman, 2nd century AD. Ny Carlsberg Glyptotek, inv. No. ÆIN 682. Right: UVF image showing the orange-red fluorescence indicative of an organic red lake (photos: Maria Louise Sargent).

only indicates the nature of the fluorescent material, and it is not possible to determine the chemical composition of a given material based on fluorescence alone. Thus, chemical analysis is necessary to make a certain identification.[64]

Such red fluorescence is clearly visible in the UVF images of the Egyptian mummy portraits from the Ny Carlsberg Glyptotek. A portrait from er-Rubayat, dating to c. AD 140–160, depicts a woman wearing a purple garment. In the UVF image, her purple garment fluoresces a bright orange-red color, suggesting that it was painted with madder lake (Fig. 11).[65] Another example is a portrait of a man, still attached to its mummy, recovered from Hawara and dated between AD 100 and AD 125. One of the *clavi* of his tunic is rendered in a dark purple color, which in the UVF image fluoresces bright orange-red, again suggesting the use of an organic red lake, mixed with a blue pigment (Fig. 12).

6.2 *Indigo in ancient polychromy*

Indigo was used in ancient polychromy, although its use for this purpose appears understudied in scholarship. So far, there are only very few attestations of the use of indigo as a pigment for painting in Classical Antiquity.[66] This possibly has to do with the challenges in identifying this colorant in ancient paint layers, which requires sampling.

However, recent advances in Multi-Spectral Imaging (MSI) have provided us with excellent non-invasive methods to evaluate the use and distribution of specific pigments and colorants, as is, for example, also the case for UVF-photography as described above. Another useful method in the study of ancient polychromy is Multiband Reflectance photography (MBR), which can indicate the spatial mapping of indigo. It is based on the strong signal increase when comparing the reflectance values of the spectrum at 635 nm and 735 nm. However, it is important to stress that this method does

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[64] Brøns & Sargent 2019.

[65] The madder lake was mixed with a blue pigment (Egyptian blue) to create the purple hue.

[66] Indigo has, for example, been identified in Romano-Egyptian mummy portraits, see, *e.g.*, Dyer & Newman 2020, 65. See also Schweppe 2012, 98–101 for further attestations of indigo in painting.

Fig. 12: Left: Mummy portrait of man from Hawara, c. AD 100–125. Ny Carlsberg Glyptotek, inv. No. ÆIN 1426. Right: UVF image showing the orange-red fluorescence indicative of an organic red lake (photos: Maria Louise Sargent).

not provide a specific identification of indigo; chemical analysis is still needed. Moreover, MBR photography has several shortcomings. For example, as pointed out by Salas, MBR cannot differentiate between indigo, Maya blue, cobalt-containing pigments, and lapis lazuli, or ultramarine.[67] However, when taking the geographical area, the culture, and the chronological period into consideration, it is often possible to determine which of these is present in the paint.

An example illustrating the use of indigo in ancient polychromy is a votive panel, painted on wood, dating to c. 40 BC to AD 113 (radiocarbon date). The panel renders the goddess Nemesis, identified due to the cubit (measuring stick) she holds in her right hand, with the Hellenistic child-God Harpocrates in the upper left corner. Positive MBR signals were found in several parts of the painting, including the *clavus* of the goddess' tunic and her mantle, which is arranged horizontally across her abdomen (Fig. 12). Interestingly, the MBR image exposes decoration in the shape of a cross at the uppermost part of the *clavus* on her right shoulder, which is not clearly discernible in visible light. The MBR image also reveals elevated counts in some parts of her hair, and in the blue, precious stones in her necklace. Moreover, the two dots on Harpocrates' forehead, his wreath, his sash, and the shading of his white garment near his left thigh, are MBR positive (Fig. 13). FTIR analysis confirmed the use of indigo on Nemesis' grey *clavus*.[68]

6.3 *Weld in ancient polychromy?*

So far, there are no attestations of the use of weld as a colorant in ancient polychromy. However, weld was used as a textile dye in Europe from the prehistoric periods onward, and it is likely to have been cultivated in the Mediterranean area since the Hellenistic period.[69] Vitruvius (7.14.2) mentions the use of weld for painting to create a brilliant green by mixing it with blue. It is thus not impossible that it was used as a lake pigment for painting in Antiquity.

Lack of identification may be related to the difficulties in identifying this organic colorant, as well as the challenges for sampling ancient cultural heritage

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[67] Salas 2020.

[68] Brøns *et al.* forthcoming.

[69] Cardon 2007.

Fig. 13: Left: Votive panel from Egypt, rendering the goddess Nemesis and Harpocrates. C. 40 BC–AD 113 . Ny Carlsberg Glyptotek, inv. No. ÆIN 685. Right: MBR image illustrating the distribution of indigo (Photos: J. Stenger).

artifacts, and the still relatively few research groups focusing on the identification of pigments and colorants in ancient polychromy. Weld was certainly used to produce yellow pigments for artists during later periods, starting at least from the Medieval period and up until the 19th century.[70] For example, weld was used as a lake pigment for the decoration of Medieval manuscripts, such as the Wollaton Antiphonal (University of Nottingham MS250) from c. 1420.[71]

However, it is important to stress that the lack of identification may simply be because this sort of pigment was not commonly used in Antiquity. As the resulting color would probably be very close to a yellow ocher, which was often readily available (and presumably cheaper), as well as much more color-fast, ocher would probably have been the preferred choice of most ancient painters.

Conclusions and future perspectives

Organic colorants are some of the most complex materials to characterize. Their ability to provide different hues and shades depending on their formulation have made them a favorite for dyeing industries throughout history. Moreover, color only recently, after the advent of synthetic colorants, became something rather trivial; during ancient, medieval, and early modern times, color was associated with status and nobility, and for some periods, particular colors, such as purple, were kept for the exclusive use of certain classes.

In this study, we have shared three methodologies which have proven valuable over the last decades for the characterization and study of organic colorants in ancient and historical textiles and artifacts:

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[70] Veneno *et al.* 2021; Cardon 2007, 177; Clarke 2011.

[71] Clarke 2011, 148. for Medieval recipes for making lake pigments from weld, see Kirby *et al.* 2014.

- Written sources give us a glimpse of the technological processes for dyeing with natural colorants. We can better understand the recipes, which may be very specific to certain times and places. By developing analytical techniques which might be sensitive to these formulations, valuable information concerning the making of a dyed textile, but also its makers, can be revealed. Reconstructing dyeing processes helps us to understand the technical abilities of past craftspeople.
- Archaeological textiles provide incredible information on the most likely dye sources. We are able to better understand preferences for certain dye sources and to what extent they were used and for which particular garments. Analyzing dyes from archaeological textiles also helps reconstruct the landscape and environment of past societies.
- The study of polychromy illustrates the use of organic colorants for other forms of arts and crafts besides textiles. Moreover, the analysis of organic colorants in polychromy allows us to better understand the preferred colors for dyed textiles via their representation in art. These artistic renderings can thus potentially fill a gap in our knowledge of the appearance of ancient textiles and dress, given the fact that most textiles do not survive in the archaeological record, or they have lost their color either entirely or partially. Hence, polychromy gives us the chance to reimagine these dyed textiles, providing important clues on the dye sources used.

Future analysis of textiles and polychromy will certainly provide further insights into the use of organic colorants, potentially revealing the use of additional colorants, while studies and experimentation with historical recipes will provide important insights into how these colorants were obtained and used.

As the three case studies presented here have shown, the use of organic colorants attests to overlaps in craft traditions, which have often been considered as separate. Thus, many of the same organic colorants were used for dyeing textiles as well as for painting. This is significant to our understanding of the acquisition of raw materials, trade of colorants, workshop practices, and ancient and Medieval economies.

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