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## Table of contents

<b>Papers</b>	<b>7</b>
<i>Travels and Mysteries of an 18<sup>th</sup> C. French Manuscript on Wool Dyeing with Sample Sheets</i>	<b>8</b>
Dominique Cardon	
<i>An Unknown 18<sup>th</sup>-Century Flemish Dyers Manuscript From Antwerp</i>	<b>9</b>
Emile Lupatini, Bert De Munck, Geert van der Snickt, and Natalia Ortega Saez	
<i>Mémoires de Teinture: The Yellows From an 18<sup>th</sup>-Century Recipe Book by Paul Gout</i>	<b>11</b>
Mara Santo, Mila Crippa, Dominique Cardon, and Paula Nabais	
<i>Practical Dying and Technical Imaging: The Identification of Cochineal Dye and Dyeing Methods Used to Produce a Mesoamerican Feather Insignia</i>	<b>13</b>
Renée Riedler, Julia Zeindl, María Olvido Moreno Guzmán, Nikoletta Sárfi, Martina Griesser, Annette T. Keller, and Leticia Arroyo Ortiz	
<i>Historic Uses of Natural Dyes on Egg Shell in the Ukrainian Craft of Egg Batik and Present Day Adaptations</i>	<b>14</b>
Mar'yana Svarnyk	
<i>Traditional Fish Leather Dyeing Methods with Indigenous Arctic Plants</i>	<b>15</b>
Elisa Palomino, Lotta Rahme, Katrín María Káradóttir, Mitsuhiro Kokita, Sigmundur Freysteinnsson	
<i>In Search of the Orcein Used in the Hanging of the Lady and the Unicorn Recipe, Experimentation and Identification</i>	<b>17</b>
Pauline Claisse, Charlotte Marembert, Rémy Chapoulie, Mohamed Dallel, and Aurélie Mounier	
<i>Towards the Identification of an Unknown Lichen Dye Source in a Medieval Tapestry</i>	<b>18</b>
Rachel Lackner, Kisook Suh, Shirin Fozi, Maria Mieites Alonso, Solenn Ferron, Joel Boustie, H. Thorsten Lumbsch, and Nobuko Shibayama	
<i>Tapestry The Relief of Leiden: History, Materials and Restoration Insights</i>	<b>20</b>
Art Proaño Gaibor and Jori Zijlmans	
<i>Tapestry Manufacture Reverse Engineering: Exploring the Use of Dyes with Hyperspectral Imaging</i>	<b>22</b>
Constantina Vlachou-Mogire, Moira Bertasa, John R Gilchrist, Jon Danskin, and Kathryn Hallett	
<i>The Long Industrial Road to Synthetic Indigo</i>	<b>23</b>

Matthijs de Keijzer and Maarten R. van Bommel	
<i>Natural Indigo Production in Southern India: Tracing History From Pre-Colonial Times to Present Day</i>	24
Smitha Shankar	
<i>Probing Indigo Dye in Japanese Textiles with External Reflection FTIR Spectroscopy</i>	27
Ludovico Geminiani, Francesco Paolo Campione, Cristina Corti, Moira Luraschi, and Laura Rampazzi	
<i>Dyes From the Amazon Forest: Blue Colorants From Ticuna Masks</i>	29
Thiago Sevilhano Puglieri, Laura Maccarelli, and Ashley A. Freeman	
<i>Faking It: Colour and Imitation in Early Modern Italian Fashion</i>	30
Paula Hohti	
<i>Dyestuff Identification in 16<sup>th</sup>-Century Waistcoats: A Multi-Analytical Approach</i>	31
Paula Nabais, Jane Malcolm-Davies, Beatrice Behlen, and Natércia Teixeira	
<i>The Technology of Dyeing Outside the Text</i>	32
Anete Karlson	
<i>Analyses of Methods and Materials Used for Manufacturing of a Silk Wall Hanging From 1714 At Rosenborg Castle, Copenhagen</i>	33
Augusta Møllemand Jensen, Mikkel Scharff, Ina Vanden Berghe, Merethe Kjeldgaard, Anna Sparr, Julie Nogel Jæger, and Annemette Bruselius Scharf	
<i>Natural Dyes in Embroideries of Byzantine Tradition: The Collection of Embroidered Aëres and Epitaphioi in The National Museum of Art of Romania</i>	34
Irina Petroviciu, Emanuela Cernea, Iolanda Turcu, Silvana Vasilca, and Florin Albu	
<i>Mystery, Adventure of Historical Sof Fabric and Their Chemical, Physical and Trade Analyzes</i>	36
Recep Karadag and Muge Burcu Ozdemir	
<i>The History of Κοκκινιάδικη βαφή at Ampelakia, Thessaly</i>	37
Sofia Tsourinaki	
<i>The Shades of Red-Pink Historical Inks: Investigation of the Degradation Pathway of Dyes in A 19<sup>th</sup>-20<sup>th</sup> Century Collection</i>	38
Adele Ferretti, Ellen Hunt, and Ilaria Degano	
<i>From Scarlet to Crimson: Analytical and Historical Insight into Recipes with Lac Dye</i>	40

Mila Crippa, Paula Nabais, Dominique Cardon, and Anita Quye

*Compound Specific Radiocarbon (<sup>14</sup>C)  
Dating of Anthraquinone-Based Colourants in Historical Textiles* 42

L. Hendriks, C. Portmann, N. Haghipour, A. Serrano, and M. Bommel

*Colours of Early Mycenaean Textiles: First Observations* 44

Stella Spantidaki and Christina Margariti

*«A Charlatan's Bogus Dye»?*

*The 7<sup>th</sup>/8<sup>th</sup> Century Coptic Dyeing Recipe Papyrus Berlin P.8316 Revisited* 45

Tonio Sebastian Richter

*Analysis of the Materials in the Late 15<sup>th</sup>-Century Islamic Manuscript by Micro-Destructive Analysis Methods and the Contribution of the Results to Conservation Works* 47

Emine Torgan Güzel and Recep Karadag

*New Dye Analyses of Haithabu Textiles* 48

Ulla Mannering, Charlotte Rimstad, and Ina Vanden Berghe

*Textile Dyes of the Golden Horde Era on the Results of a Research of Archaeological Textile in Kazakhstan and Ukraine* 49

Tatiana Krupa

**Posters** 50

*Identification of the Unknown Type B Brazilein Derivative Marker Found in Brazilwood Dyed Textiles* 51

L. Hendriks, R. Martinent, and C. Portmann

*The Colours of Textiles and Braids in an Early Medieval Hoard Buried in Galloway, Scotland in C. 900 CE* 53

Alexandra Makin, Ina Vanden Berghe, and Susanna Harris

*Effects of Base Material (Silk or Paper) on the Use of Amur Cork Tree as an Indicator of Deterioration* 54

Yoshiko Sasaki and Ken Sasaki

*Organic Pigments in the Excavation Findings and Monuments that are Under the Supervision of the Ephorate of Antiquities of the Thessaloniki Region* 55

S. Vivdenko, M. Tsiapali, L. Achilara, M. Tsimpidou-Avlonitou, A. Vasileiadou, I. Karapanagiotis, and N. Zacharias

*Proteomics Approach to the Biomolecule Degradation of Indigo Dyeing Vessel Deposits* 57

Takumi Nishiuch, Hideki Saito, Natsuki Murakami, Seiji Nakayama,

and Shinya Shoda

- Dressed to Afterlife - Dyes in Late Iron Age Children's Clothing in Finland* 58  
Krista Wright, Jenni Sahramaa, and Ina Vanden Berghe
- Identifying Red Pigments Used in Paintings via High-Performance Liquid Chromatography* 59  
M. Havlová, V. Antušková, and R. Šefců
- Which Pigments for Which Clothes? Reflections on the Colours and Materials Used to Depict Everyday Clothing in Gallo-Roman Wall Paintings* 60  
Nicolas Delferrière and Emeline Retournard
- Colour4CRAFTS Research on the Cultural Tradition of Sustainable Bio-Based Colourants and Dyeing* 61  
Riikka Räisänen, Krista Wright, Riina Rammo, Liis Luhamaa, Debbie Bamford, and Ina Vanden Berghe
- Multi-Analytical Methodology for the Identification of Organic Dyes and Materials in Finnish Karelian Traditional Costumes* 63  
Peppi Toukola, Arvind Negi, Mari Kosunen, and Riikka Räisänen
- Textile Dyes as Samples for the Colouring of Ancient Sculptures* 64  
Dr. Heinrich Piening
- Logwood from the bottom of the sea* 65  
Kari Helene Kullerud
- "Blinded by the Light": Impact of Daylight Fluorescent Pigment Composition on Product Color and Light Stability* 67  
Sarah J. Schmidtke Sobeck, Victor J. Chen, and Gregory Dale Smith
- Traditional Iranian Dyeing Technique: Insights from a Hands-On Workshop Experience* 68  
Malihe Sotoudeh, Paula Nabais, and Vanessa Otero
- Van Gogh's Use of Logwood Ink in France* 70  
Han Neevel

## Papers

## Travels and Mysteries Of An 18<sup>th</sup> C. French Manuscript on Wool Dyeing with Sample Sheets

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It would spoil the fun and suspense if I revealed in what somewhat unexpected place this anonymous, undated manuscript of 48 written folios accompanied by 5 sample sheets with dyed samples of wool broadcloth is now preserved. I intend to publish its critical edition with a translation into English soon after the DHA 42 meeting. At DHA 42 in Copenhagen I propose to present:

- what can be retraced of its history, in the light of my current researches into the context of the entry on « teinture » and related entries on dyeing in Diderot's , d'Alembert's and Jaucourt's Encyclopédie, as part of my contribution to the ENCCRE project<sup>1</sup>;
- what new, unusual contributions this treatise on wool dyeing makes to the history of dyeing techniques and colour names ;
- how it more largely constitutes a new example of the scientific, philosophical and political issues debated in the Age of Enlightenment.

### References

<sup>1</sup><http://enccre.academie-sciences.fr/encyclopedie/>



## An Unknown 18<sup>th</sup>-Century Flemish Dyers Manuscript From Antwerp

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This paper presents the historical analysis of a unique dyers manuscript preserved within the Museum of Industry in Ghent, Belgium. The manuscript, originally from a dyer based in Antwerp, dates from the late 18<sup>th</sup> century and contains an extensive collection of natural dyes recipes. The study of this manuscript allows researchers and conservator-restorers to better grasp the practices of traditional dyeing techniques and materials in the region during that time.

Most of the manuscript revolves around the dyeing of wool fabrics into different shades of red, brown, and blue. Approximately 60 of the 120 recipes focus on achieving red and its hues. Furthermore, over 30 other recipes employ red as an undertone or in combination with other dyes to attain orange, brown and black hues. Fifteen recipes involve blue dyeing techniques. Surprisingly, a minimal number of recipes deal with black (five), which was very diffused at that time in Antwerp (Thijs, 1978).

Concerning the materials, the document extensively mentions the use of madder, brazilwood and cochennille. In order to create a variety of red shades, the dyer describes how fabrics were treated with different mordanting compounds with tin and alum as the main ingredients and how the dyeing solutions were prepared. The resulting colours comprise ‘madder red’, ‘formal red’, ‘crimson’, ‘scarlet’, ‘Turkish red’, ‘fire colour’ and ‘carnate’.

In addition to the dyeing recipes, the manuscript contains various accounting documents and correspondences between the dyer, customers, and suppliers. Additionally, over 50 recipes are illustrated by original colour samples, which are rare for manuscripts of that time (Quye et al., 2020) .

In this paper, the contents of the artefact will be disclosed, including recipes with attached samples and correspondence. Also, various findings resulting from archive research will be included, contextualizing and placing the dyer in its urban and social context. The paper concludes by discussing the potential limitations and provides an avenue for future research.



Figure 1: Full page recipe, to create 'piss-blue'.

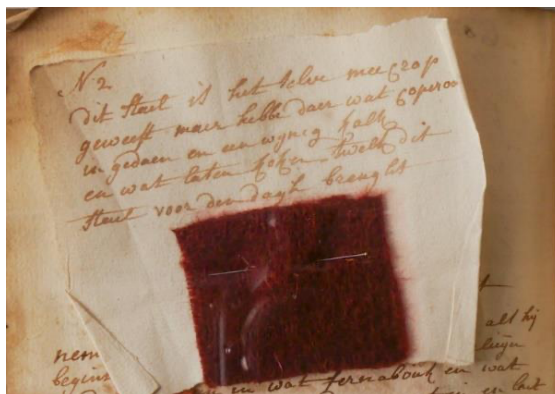


Figure 2: Detail of a recipe to dye 'madder red'.  
(V37828-001), Museum of Industry collection. Museum  
of industry, Ghent, 9000, Belgium

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## Mémoires de Teinture: The Yellows from an 18<sup>th</sup>- Century Recipe Book by Paul Gout

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Organic dyes have been used for artworks such as textiles, for millennia, and have great artistic and historic value. They may provide clues to the understanding of the technology behind an artwork's production. The characterization of natural organic colorants in artworks is still a challenge to this day, and of the natural dyes used in cultural heritage, yellows are some of the most difficult to identify<sup>1,2</sup>. Hence, the use of historically accurate reconstructions may provide us with a clue about the manufacture of yellow-dyed textiles.

To test this approach on yellow dyes, we have reproduced yellow-dyed textiles made with weld (*Reseda luteola* L.) using recipes from French master dyers of the 18<sup>th</sup> century, Antoine Janot and Paul Gout<sup>3-6</sup>. They wrote treatises similarly entitled *Mémoires de Teinture* (Memoirs on Dyeing), illustrated with dozens of dyed textile samples. When analyzing these books, one can't help but notice the considerable percentage of recipes for yellows & light browns. In Gout's book, weld figures in 33 % of the 157 recipes still illustrated with samples.

In a previous DHA presentation, we have shown our first results on the reconstruction of these dyeing processes, preliminarily analyzed by colorimetry, reflectance spectroscopy and high-performance liquid chromatography. We have shown that molecular fluorescence in the UV-VIS is highly sensitive and selective, allowing the disclosure of recipes' specificities, leading to chronological and location particularities, and enabling a better understanding of the making of the artists' materials<sup>1,2</sup>. It was able to distinguish between both master dyer's recipes, but also on the use of particular ingredients such as red tartar or bran.

More recently we had the opportunity to study the beautiful manuscript of *Mémoires de Teinture* by Paul Gout. In this presentation we intend to share our results of such a study, and also to compare with our data acquired on the historical reconstructions, focusing on the yellow dyed swatches. This will provide key knowledge on the technological processes for dyeing with weld from these 18<sup>th</sup> c. French masters.

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# Practical Dying and Technical Imaging

## The Identification of Cochineal Dye and Dyeing

### Methods Used to Produce a Mesoamerican Feather

### Insignia

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A colonial feather insignia from New Spain (16<sup>th</sup>/early 17<sup>th</sup> century, Mexico) made with natural colored and dyed bird feathers with is currently under investigation at the Weltmuseum Wien

(<https://www.weltmuseumwien.at/en/science-research/feather-fashion-and-cross-cultural-exchange/>). The study of material and techniques has the final aim of making a faithful replica, which will be exhibited at the National Museum of Anthropology in Mexico City.

The insignia represents a highly skilled feather art called *amantecayotl* in Nahuatl. Historical sources such as the Florentine Codex are documenting the individual processes of feather selection and preparation by *amantecas* (feather workers) who incorporate a list of birds, which are symbolically coded. While the brilliant color of most of the feathers is based on biopigments and structural phenomena, less precious feathers had been dyed with organic dyes.

To make a faithful replica, it was critical for the researchers to identify the bird species and the dye and dyeing process used to dye feathers scarlet red and light pink. Based on historical sources, visual examination and previous analyses on comparative feather work from the same artisanal tradition, the red dye used had been most likely cochineal (*Dactylopius coccus* Costa).

In order to confirm this assumption without relying on costly analytical equipment, the research team consulted imaging techniques like technical photography and multispectral imaging (Rainbow MSI Solution, PhaseOne) in combination with Fiber Optics Reflectance Spectroscopy (FORS). The prerequisite was the dyeing of reference materials based on different recipes and raw materials. A dye workshop was set up at the Weltmuseum Wien and reference samples were dyed and documented. Finally, these reference samples were brought to Mexico in order to discuss the results and to compare raw materials, dye process and resulting feather color with experienced feather dyers on site.

## Historic Uses of Natural Dyes on Egg Shell in the Ukrainian Craft of Egg Batik and Present Day Adaptations

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The craft of wax resist on eggs, sometimes referred to as “egg batik”, has been traditional in several European countries, mostly in Slavic communities. The documented tradition in Ukraine is complex, using a variety of techniques, with motifs, patterns, and color combinations specific to different ethnographic regions within the country. A fair number of eggs dyed with wax resist have been preserved in museums, the oldest egg in a museum collection in Ukraine is dating back to 15<sup>th</sup>-16<sup>th</sup> century. Several large ethnographic albums of eggs with color plates have been published as early as 1899, they contain some of the earliest reports about the dyes used on eggs, and already at that time the authors are lamenting that the craft itself, as well as the knowledge about the use of natural dyes on eggs is gradually being lost.

This paper will first summarize the information about the use of specific natural dyes on eggs in Ukraine that we find in the late 19<sup>th</sup> century publications. It will then address the current availabilities of these dye stuffs and the potential substitutions for the dyes that have become commercially unavailable (e.g. *Porphyrophora polonica* or *Paubrasilia echinata*). It will share the results of recreating some of the traditional egg patterns with wax resist and natural dyes listed in the late 19<sup>th</sup> century publications (or their substitutions). Lastly, the paper will outline several potential avenues of further research that would be required to better understand both the historic uses of natural dyes on eggs and the possibilities for current applications and adaptations.

## Traditional Fish Leather Dyeing Methods with Indigenous Arctic Plants

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Along the Arctic and sub-Arctic coasts of Alaska, Siberia, north-eastern China, Hokkaido, Scandinavia and Iceland, people have been dressed or shod in fish skin for millennia. These items were decorated with a rich colour palette of natural dyes provided by nature. Minerals and raw materials of plant origin were collected from the riverbanks and processed by Arctic seamstresses who operated as designers, biochemists, zoologists and climatologists at once. During our research, an international team of fashion, tanning and chemistry researchers used local Arctic and sub-Arctic flora from Sweden, Iceland and Japan to dye fish leather. Several plants were gathered and sampled on a small scale to test the process and determine the colours they generated based on historical literature and verbal advice from local experts.

The paper describes the process and illustrates the historical use of natural dyes by Arctic groups originally involved in this craft. The project builds on traditional cultural heritage that has enabled us to develop sustainable dyeing processes, reinforced by the use of state-of-the-art analytical technologies. The physical and rheological properties of the different traditional dyeing recipes were measured to test the stability and suitability of the colour. The results are promising and confirm the applicability of these local plants for dyeing fish skins, providing a basis for a range of natural dye colours from the local Arctic flora. The aim is to develop moderate-sized industrial production of fish leather in this colour palette to replace current unsustainable chemical dyeing processes. The project represents an innovation in materials design driven by traditional technologies, addressing changes in interactions between humans and with our environment. The results indicate that new materials, processes and techniques are often the fruitful marriage of historical research into traditional methods and fashion, helping the industry move towards a more sustainable future.

**Keywords:** Historical Arctic Fish Skin; Traditional Natural Dyes; Indigenous Arctic and Subarctic Flora; Local Cultural Heritage; Analytical Characterisation and Identification; Food Industry By-Product; Sustainable Fashion.



Figure 1: 2003-43-9 Nivkh coat. Fish skin applied. Amur River, Siberia. Penn Museum. Philadelphia,



Figure 2: © Lotta Rahme's testing on fish skin dyeing with bearded lichen (*Usnea dasopoga*) and horsehair lichen (*Bryoria capillaris*) from western and southern Sweden.



## In Search of the Orcein Used in the Hanging of the Lady And The Unicorn Recipe, Experimentation and Identification

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The Lady and the Unicorn tapestry (14<sup>th</sup> c.) was acquired by the Musée de Cluny in 1882. The set comprises 6 tapestries representing an allegory of the five senses. These tapestries have deteriorated, especially the colours that require several dyes, such as green or violet. These degradations result in a significant loss of information to understand the history of the tapestry's creation. Bibliography reports on the state of deterioration of the tapestry carried out by the Bregère studio in 1942, indicating the mauve colour disappearance that had become light blue on the subject's dress of the tapestry "La Vue".

In the Middle Ages, it was common for the violet colour to be made with a blue dye bath (woad) followed by a red bath which could be madder, kermes or orcein. The latter is obtained from lichen, either of the Rocella types from the Mediterranean basin (sea orcein) or the land orcein found inland. This dye has been used since antiquity and was rediscovered in the Middle Ages in France in the 14<sup>th</sup> century. It is known to be very unstable and seems to have been used to make the violet of the dress' Lady turned blue today.

Our study focuses on this orcein. Thus, it was necessary to experiment by extracting its dyestuff to optimise wool dyeing before analysing the samples using non-contact methods (colourimetry, hyperspectral imaging and fluorimetry). This methodology allowed recording orcein's reference spectra depending on the recipe used to constitute a database. In addition, a light degradation study was carried out on the samples before comparing the results with the signal obtained on the tapestry. This comparison would help us confirm the hypothesis on the identification of this dye and have some information on this dyeing technique.

## Towards the Identification of an Unknown Lichen Dye Source in a Medieval Tapestry

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The Nine Heroes Tapestries, a set of wool textiles that celebrate several notable Classical, Jewish, and Christian figures, are among the oldest surviving Medieval tapestries in the world. Five of the Heroes are on permanent display at the Cloisters at the Metropolitan Museum of Art (the Met) and are the subjects of a long-term conservation campaign. Fragile and fragmented upon their arrival to the Met, these works survived reappropriation as curtains,<sup>1</sup> a fortuitous move that preceded the bombing of their former home,<sup>2</sup> and an ambitious post-WWII restoration.<sup>3</sup>

As part of the Met's conservation campaign, one of the set, Julius Caesar and Attendants (MA.47.101.3), was analyzed using liquid chromatography quadrupole time-of-flight mass spectrometry (LC-qTOF-MS). The goal of this analysis is to identify the dyes used in the weaving of the tapestry, to assess the fragments' relationship to each other, and to develop our understanding of the series' origins and history.

Dye analysis revealed the presence of typical medieval dyes, such as madder and weld.<sup>4</sup> Both natural and synthetic dyes used for restoration were also detected. Notably, in several brown samples we identified chlorinated norlichexanthone compounds of the thiophanic acid chemosyndrome that have not yet been documented in the dye analysis literature. These markers are unique metabolites produced by certain crustose lichens, primarily from the *Lecanora* and *Lecidella* genera.<sup>5</sup> Comparison with reference samples allows us to confirm the identity of thiophanic acid and related metabolites and to propose a possible dye source. To the best of our knowledge, this is the first time that chemical markers from a non-orchil lichen dye source have been unambiguously identified in a historic textile, despite extensive documentation of their use throughout history.<sup>6</sup> This presentation will discuss efforts to identify the lichen species and explore how chemistry can inform our understanding of a textile's history, even when little or no documentation survives.

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## Tapestry *The Relief of Leiden*: History, Materials and Restoration Insights

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During the conflicts of the Eighty Years' War and the Anglo-Spanish War, between 1573 and 1574, the Siege of Leiden took place. The Spanish forces, led by Francisco de Valdez, tried to capture the defiant city of Leiden in the Netherlands. However, the siege was unsuccessful as the city was effectively relieved in October 1574.<sup>1</sup>

The *Relief of Leiden* tapestry was created in 1587, thus thirteen years after the liberation. It was commissioned by the cities magistrate to a Delft weaver, Joost Jansz. Lanckaert, and is a remarkable example of northern Netherlandish tapestry craftsmanship from the 16<sup>th</sup> century.

The tapestry illustrates a two-month period, depicting the breaching of the Maas and IJssel dikes by troops of Prince William of Orange, and the naval operation of the Sea Beggars, culminating with the relief of the city of Leiden on October 3<sup>rd</sup>.<sup>2</sup>

The tapestry is based on a chorographical map of Holland drawn by Hans Liefvrick and the ornaments were designed by Isaac Claesz. van Swanenburg. Upon its creation, the *Relief of Leiden* tapestry quickly became a beloved masterpiece and retained its status throughout the war years. It honours the city's courageous actions during the Eighty Years' War and has been carefully preserved and displayed since 1872 at De Lakenhal Museum in Leiden.

Major tapestry weaving cities, like Brugge, Ghent and Antwerp, had their own wool dyeing rules. However, after the Spanish Fury in 1576, many weavers migrated to cities like Delft, Leiden or Haarlem, where no dyeing regulations existed. The lack of rules for tapestry dyes could lead to deceptive practices.

This study of the *Relief of Leiden* tapestry, using liquid chromatography, provides chemical proof of these early fraudulent practices. It sheds light on the materials used in the tapestry's production, together with the early and modern restorations, and shows as well the evolution of dyeing practices in northern Netherland in the 16<sup>th</sup> century.<sup>3</sup>



*Tapestry The Relief of Leiden, 1587-1589 Jan Joost Lanckaert, (297 × 366 × 1cm)  
De Lakenhal Museum, Leiden (The Netherlands)*

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# Tapestry Manufacture Reverse Engineering: Exploring the Use of Dyes with Hyperspectral Imaging

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Hyperspectral imaging (HSI) has emerged as an excellent analytical method for the optical characterisation of artworks at high spatial and spectral resolutions. It combines both optical spectroscopy and imaging capabilities at high measurement speeds across large areas or entire surfaces of the artwork in a non-contact and non-destructive manner, which is a highly significant application for historic tapestries.

This collaborative research deployed a high-resolution ClydeHSI Art Scanner, with a push-broom visible to very near-infrared (VNIR; 400–1000 nm) and near infrared (NIR 900–1700 nm) hyperspectral cameras, for the characterization and analysis of several 16<sup>th</sup> century tapestries on display at Hampton Court Palace<sup>i</sup>. Wool and silk model tapestry samples, designed for the MODHT<sup>ii</sup> EU research project and dyed based on original medieval recipes, were also scanned and used as an external reference library<sup>2</sup>.

In this work, further processing of the hyperspectral reflectance cubes with Spectral Angle Mapping (SAM) algorithm will be discussed alongside the individual mapping of each dye on the tapestry surface. This insight will enable the study of the tapestry weaving techniques using coloured yarns for the production of high-quality design formation. Furthermore, results of the SAM processing of the MODHT samples following light ageing will provide information on the impact of degradation phenomena on the analytical results exploring the advantages and potential limitations of this emerging analytical method. Finally, the deployment of complementary analytical techniques such as HPLC will be used to provide further evidence on the HSI results with a particular focus on the characterisation of the yellow dyestuffs and the identification of brazilwood.

<sup>i</sup> These tapestries are part of the Royal Collection [www.rct.uk](http://www.rct.uk)

<sup>ii</sup> Monitoring of Damage to Historic Tapestries

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## The long industrial road to synthetic indigo

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The presentation will cover the various ways, problems and solutions to produce indigo industrially. Surprisingly, although a large number of synthetic dyes were already on the market by the late 1860s, no synthetic substitute for indigo had been found. In the early days of synthetic dyes, chemistry was largely empirical, with vague ideas about how atoms in a molecule were linked. The carbon theories developed by Friedrich August Kekulé provided chemists with a useful model for determining the structure of dyes. In 1865, Adolf Baeyer began investigating the structure and synthesis of indigo, but it took him 18 years to reveal the indigo formula.

Between 1878 and 1882, he succeeded in establishing three indigo processes, the first from isatin, the second from cinnamic acid (propionic acid process) and the last synthesis from o-nitrobenzaldehyde (Baeyer-Drewson process); these routes were not attractive from a commercial point of view. In 1890, Karl Heumann discovered two processes. In the first Heumann synthesis, N-phenylglycine was fused with potash. Unfortunately, the high temperature led to a low yield of indigo. The second Heumann synthesis, based on N-phenylglycine-o-carboxylic acid, was the economically viable process. Between 1890 and 1897, many technical processes were developed to obtain the basic materials for the synthesis of indigo. In 1897, BASF produced synthetic indigo on an industrial scale applying an improved process based on N-phenylglycine-o-carboxylic acid. By 1900, four different commercial routes to synthetic indigo were in use.

In 1901, Johannes Pfleger, working for Degussa, discovered a more efficient way of producing indigo on an industrial scale. This process used the condensing agent sodium amide for the ring closure to form indoxyl at a lower temperature, resulting in a 90% yield of indigo. Although this process was advantageous, the inexpensive blue sulfur dyes of the German factories Cassella, Kalle and Bayer were strong competitors to synthetic indigo, and BASF created its own competitor with the discovery of Indanthrone blue BS by René Bohn in 1901. After the price of synthetic indigo fell to 7 German marks per kilo in 1904, BASF and Farbwerke Hoechst shared the Pfleger-process and then formed the Second Indigo Convention to control the indigo market. Over time,



the demand for synthetic indigo decreased and many companies ceased production, and by the mid-1960s, even BASF wanted to stop the production. In the late-1960s, the demand increased significantly, as it was used to dye fabrics for blue jeans, even today.

During the presentation, we will discuss some of the synthetic routes developed, their advantages and limitations, providing a better historical insight into this still very important colourant.

## Natural Indigo Production in Southern India: Tracing History From Pre-Colonial Times to Present Day

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The paper discusses the ancient method of producing indigo dye from the *Indigofera tinctoria* plant using local production methods that are unique to the Indian subcontinent, especially to southern India. Greek historians Herodotus and Pliny have recorded that India was home to the blue dye referred to as “Indikon” in ancient historical accounts. Marco Polo was the first to note that Indigo was produced from plants in India and was not a metal as originally thought in the western world.

The production of blue dye from the plant has been recorded in great detail by the East India company for whom Indigo was a noted commercial product that earned a handsome revenue. Their records show this dye was produced locally by growers of the Madras and by farmers of the Bengal presidencies respectively. Arguably, the oldest known dye to mankind, indigo dye cakes sometimes referred to as “Blue gold”, were exported from India into the west and used world over to colour textiles and other media.

The discovery of synthetic indigo in the early 1900s led to a crash in the demand of plant based indigo and synthetic indigo nearly completely replaced this once coveted Indian cash crop. The ancient methods of farming and indigo production were completely local and sustainable where production wastewater was traditionally used to irrigate food crops. The paper will describe in detail the production methods of this dye that remains unchanged over centuries and present a farm in Southern India that follows the very same process to this day. The paper will also describe the chemical reaction responsible for the emergence of the characteristic blue of indigo as part of the production process.

## Probing Indigo Dye in Japanese Textiles with External Reflection FTIR Spectroscopy

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Indigo has always represented a fundamental material in Japanese tradition<sup>1</sup>. It was used to piece-dyeing (*aizome*) or to obtain patterned indigo dyeing, by using both tie-dye and stencil dyeing. As for the first one, called *shibori*, it involves shaping and securing the fabric before dyeing to create patterns. The method of stencil dyeing, the so-called *katazome* is a method of dyeing historically done on cottons using a paste-resist. Besides, indigo was used also as a pigment in some stencil dyeing techniques and in *ukiyo-e* prints, where it was mixed with binders such as animal glue<sup>2</sup>.

A novel non-invasive method for the detection of indigo dye in leather and textiles is presented. ER-FTIR (External Reflection FTIR) spectroscopy is rapid, portable, and widely employed in the cultural heritage field, but rarely applied to the study of textiles. In the present case, the measurements were made on site with on a variety of materials coming from traditional samurai armours (Morigi Collection) and Japanese folk art artifacts (Montgomery Collection), during their exhibition at Museo delle Culture (Lugano, Switzerland).

Firstly, the spectrum of indigo was studied deeply in order to highlight diagnostic bands. ATR-FTIR and ER-FTIR spectra were compared to highlight differences between instrumental responses and to get a complete band assignment, based on ATR-FTIR literature. Finally, ER-FTIR was tested with success for the first time as a fast and reliable technique to suggest the occurrence of indigo on a great variety of applied art supports, also on aged materials. ER-FTIR spectroscopy also allowed to characterise the bulk, and to obtain useful information about the dyeing techniques on the basis of traces of animal glue and rice starch.

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## Dyes From the Amazon Forest: Blue Colorants From Ticuna Masks

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This work is part of a long-term initiative that explores Indigenous artistic traditions and preservation of the Americas. The studies focus on Technical Art History and Conservation Science and examine ways of collaborative practices that can enhance the social impact of their scientific outcomes. A manuscript is being prepared that combines historical documents and scientific literature to explore the natural materials used in Indigenous paints from the Brazilian Amazon Forest. The investigation of Ticuna masks is a project inside this initiative and focus on the colorants used in their production.

The Ticuna are the biggest Indigenous group in Brazil, and their decorated masks and bark clothes, used in rituals, are in collections of museums worldwide. They are made of bark and colored mainly with natural organic dyes. Human and social scientists have investigated these masks and rituals extensively, but not from their physical-chemical perspectives, resulting in a gap in artistic and cultural understanding and preservation.

Colors like green, yellow, orange, red, pink, purple, white, black, and brown are present, and different colorants have been used for each color. However, this paper focus on blue. Descriptions from historical documents resulted in the hypothesis of the existence of a new man-made blue pigment, which is here investigated. Masks from the 1940s to 1960s from institutions like the Fowler Museum (USA), Peabody Museum (USA), and Museum of Archaeology and Ethnology of the University of São Paulo (Brazil) were considered. Chemical analyses were carried out with samples and natural materials using mainly XRF, Raman, FTIR, and HPLC-DAD.

## Faking It: Colour and Imitation in Early Modern Italian Fashion

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Sixteenth century was a period of rapid growth of technological innovation in fashion and textile industry. One of the important areas of innovation was colour. By 1500, Italian dyers had not only developed methods to obtain the deepest shades of expensive reds and blacks - the ultimate colours of power and fashion- but they had also developed sophisticated techniques to imitate these, by mixing and applying new dyestuffs that were available on the market. This offered potential for fraud. In 1524, for example, Venetian mercers sold 'pavonazzo' fabrics dyed with madder and orchil, claiming these were dyed with the more expensive method 'in grain'. However, colour imitations were also used to produce fabrics at a cheaper price for a broad spectrum of consumers. This paper investigates how colour in textiles was imitated and faked in sixteenth- and seventeenth- century Italy, and how such practices of imitation provided a new opportunity for ordinary men and women to appear fashionable.

### **Short bio**

Paula Hohti is a professor and a fashion and material culture historian at Aalto University. Her research focuses on the Italian Renaissance, with a special focus on dress and decorative arts at the lower artisanal levels. Hohti has been a principal investigator within the international *The Material Renaissance* and *Fashioning the Early Modern* -projects, and a Marie Curie Research Fellow at the Centre for Textile Research in Copenhagen. In 2016, she received a 2-million euro ERC consolidator grant to develop material hands-on experimentation and historical reconstruction as a methodology in dress and fashion history.

## Dyestuff Identification in 16<sup>th</sup>-Century Waistcoats: A Multi-Analytical Approach

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No technique alone can solve the complex problems encountered in the analysis of dyes in cultural heritage artefacts. This paper proposes a protocol for sampling textiles and the identification of dyestuffs using a combination of several techniques.

Early modern materials are not well represented in dye and mordant analyses despite extensive documentary evidence suggesting the enormous demand for coloured fabrics, even among those below the elite. Non-wovens likewise receive less attention than woven textiles despite their ubiquity in the early modern historical record. Knitted garments, in particular, have rarely been subjected to dye analysis. One garment is noteworthy for its colourfulness, despite not being visible in formal wear. Men throughout society wore knitted undergarments known as waistcoats from the late sixteenth century.

The waistcoats under investigation here are from the collections at the Museum of London, the Grimsthorpe and Drummond Castle Trust, Scotland, and English Heritage. Two are made of silk and are now a pale blue-green colour. The third is a fragmentary archaeological brown wool garment thought to be a waistcoat from Lindisfarne Priory, UK.

Small samples were taken from each and subjected to a series of analytical techniques: X-ray fluorescence, Micro-Raman spectroscopy, UV-Vis microspectrofluorimetry and high-performance liquid chromatography (HPLC) coupled with a mass spectrometer. Using this protocol, it was possible to characterize the dyes and mordants in all three waistcoats by ensuring that maximum information gleaned from a sample before it was exhausted.

This multi-analytical approach presented here demonstrates the potential for early modern items with previously hidden histories to be contextualised with new knowledge about the dyes and mordants used.

## The Technology of Dyeing Outside the Text

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The research of textile dyeing technologies is an important part of the history of textiles, including clothing. One of the sources for the study of the cultural history of the Baltic (Latvia, Estonia), including the clothing of the local people, are the drawings and their descriptions of Johan Kristof Brotze (1742–1823) at the turn of the 18<sup>th</sup>/19<sup>th</sup> century. They also contain information about dyes and dyeing methods used by peasants. Brotze's fixed information, although fragmentary, is valuable because researchers lack documentary sources about the ancient dyeing methods used in the 18<sup>th</sup> century in the territory of modern Latvia. Additional research allows for more information than what is mentioned in the description. The report will present how the particular dyeing technique used in the dyeing process with *Bixa orellana* to produce the orange color of linen or cotton fibers mentioned in the description was determined by experimental method.

The information recorded by Brotze, supplemented with experimentally obtained dyeing results, allows us to establish that:

- 1) In the 18<sup>th</sup> century, Latvian peasants used not only local plants but also dyes imported from abroad, such as *Bixa orellana*, for dyeing parts of honor clothes;
- 2) Technology with the usage of alkali was applied in the dyeing process, although this is not mentioned in the written text;
- 3) Latvian peasants combined both old and new knowledge in textile dyeing - using traditional dyeing methods with new, previously unknown dyes from abroad.



*Figure 1. Cotton threads dyes with Bixa Orellana in plain water.*



*Figure 2. Cotton threads after dyeing with Bixa Orellana treated with an alkali solution.*



## Analyses of Methods and Materials Used for Manufacturing of a Silk Wall Hanging From 1714 at Rosenborg Castle, Copenhagen

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A unique silk wall hanging covers most of the walls in Christian IV's Writing Room at Rosenborg Castle in Copenhagen, Denmark. It was acquired by Frederik IV in 1714 and has covered the walls ever since except for brief periods of conservation treatments. It consists of c. fifty-six meters of block printed (and possibly painted) silk moiré, featuring a "bizarre" pattern rapport that covers the entire width of the silk (c. 45 cm) and repeats every c. 55 cm. There is no climate control in the room causing variable fluctuations over the year. There are three northeast facing windows resulting in much exposure to UV radiation and visible light.

The silk textile is in a deteriorated condition, fragile, with voids, the moiré-pattern mostly ceased to be visible, and the colour of the textile has changed. The bizarre patterns are still visible, similarly in a poor condition, partly cracked and partly flaking off, and the colours has changed as well, compared with their appearance along the selvage, where the fabric is sewn together.

The present study builds upon a previous study and archival information. It attempts to further clarify questions concerning the origin, composition, and manufacture of the silk wall hanging, determine the possible degradation parameters, and suggest preservation plan and future means to present the wall hangings.

Preliminary identification of manufacturing methods and materials are presented, using imaging techniques (VIS, UVIL, IRR), examining textile and pigment cross-sections by light microscopy, SEM-EDX, analysis of organic pigments and colorants by HPLC-DAD, as well as identifications of binding materials and adhesives using FT-IR.

Results so far includes the identification of the textile fibres, consisting of linen and dyed silk, a specific moiré-pattern manufacturing technique following the weaving, covered by a repeated printed and possibly painted pattern with colours including elements such as Ag, Cu, Al, S, Zn, Pb and colorants, and possibly using oil and other vegetable binders. The printed (and possibly painted) textile fabric was sewn together along the selvages and nailed along the borders of Christian IV's Writing Room.

## Natural Dyes in Embroideries of Byzantine Tradition The Collection of Embroidered Aëres and Epitaphioi in The National Museum of Art of Romania

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The medieval textiles collection of the National Museum of Art of Romania (MNAR) has been built since 1865 and nowadays preserves about 1000 medieval and pre-modern tissues and embroideries. These extremely valuable objects, dated between the 14<sup>th</sup> and the 19<sup>th</sup> centuries, are mainly religious embroidered garments and veils with special significance in Byzantine liturgy. Ecclesiastical embroideries of Byzantine tradition are characterized by a complex technique: metallic threads with silk core, metallic wires and coloured silk threads are couched over padding on layers of silk and cellulosic supports so that to create relief through light reflection<sup>1</sup>. The silk supports and sewing threads are coloured, mainly in red, blue, green and yellow hues, and analytical investigation of dyes used in embroideries preserved in the MNAR, in Putna and Sucevița Monasteries have been released in the last years<sup>2-5</sup>.

The present work continues the approach with studies of dyes in about 30 aëres and epitaphioi from the MNAR collection. Considering their privileged function in the liturgical ritual, these luxurious pieces embroidered with silver, gilded silver or coloured silk threads and decorated with pearls, sequins or semi-precious stones are the most faithfully description of the stylistic and technological evolution of the art of post-Byzantine embroidery in the Romanian provinces. The original data resulted within the present research will improve the knowledge regarding this topic.

Dye analysis was performed by liquid chromatography with diode array and mass spectrometric detection. The biological sources identified - lac dye, madder, kermes, carminic acid-based dyes, weld, young fustic, indigo based dyes, tannin sources, and others - will be discussed together with those previously reported on Byzantine embroideries in Romanian collections and similar objects preserved at Mount Athos<sup>6</sup>.

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## Mystery, Adventure of Historical *Sof* Fabric and Their Chemical, Physical and Trade Analyzes

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*Sof* a mohair silky, fine and deluxe fabric woven with the yarn spun from mohair goats, one exclusively bred in around Ankara. *Sof*, produced exclusively in and around Ankara since the 15<sup>th</sup> century was a commodity of choice for Ottoman, Venetian, Polish and British tradesmen, as well as Italian and French princes and princesses. It was considered the most important commercial product of Ankara, which was a textile center of the Ottoman period.

There are many mohair historical art objects in the Topkapi Palace Museum. However, these objects were identified by applying different analysis methods to determine which of these objects might actually have been produced with Angora goat mohair.

Belonging to the 16<sup>th</sup> century caftan, blanket, etc. *Sof* objects were analyzed in the Topkapi Palace Museum. Dyestuff and fiber analyses were done with HPLC-DAD and SEM- EDX. The historical sample analyzes were compared with other types of hair such as Angora Goats. This comparison had been made with historical mohair objects in Topkapi Palace Museum. According to the dye analysis and morphological analysis results, the objects are similar to the hair of Ankara Goats.

The production and trade of *Sof* fabric using Angora goat mohair played an important role in the economic development of the region around Ankara during the Ottoman period and also brought wealth and revenue into the area, further boosting its economy. The production of this commodity, which was interrupted for a while, has become valuable for the industry again today.

## The History of *Κοκκινιάδικη βαφή* at Ampelakia, Thessaly

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Ampelakia, a small town in central Greece, became famous during the 18<sup>th</sup> century for *κοκκινιάδικη βαφή*/dyeing with *ρίζαρι* or *έρυθρόδανον*. The Manufacturing Association of Ampelakia was founded in 1778 and the town became wealthy by making the famous red color «in the Thessalian way» with locally grown madder (*Rubia peregrina* L). The complex method involved many processes that required almost a month to carry out and some of the stages had a mystical, ritualistic or symbolic character that was kept as a guild and family secret. The carefully prepared process was the pre-mordanting of cotton skeins within a solution of rancid oil and soda ash and the addition of sheep's dung to «animalize» the dye bath. The skeins were left in the mixture to ferment and dried and the process was repeated several times until the oxidized fatty acids thoroughly penetrated the fibers. Then the skeins were treated with gall tannin, re-mordanted with alum and finally dyed with madder and sheep's blood. Madder as a source of red dye has been identified in several clay vessels at Alatzomouri-Pefka in Crete (c. 1750–1700 BCE). Moreover, textual evidence confirms a rich terminology of related nouns and adjectives as early as Classical times, thus testifying to the significant role of the colour: a) the verb *βάπτειν* «to dip, immerse», widely attested in all periods, lives on in Modern Greek as *βάφω* b) the term *κόκκινον*/red is testified in the epigraphic sources from Hellenistic Delos and may designate a red dye of vegetable (madder?) and not animal origin such as purple/*πορφύρεος* c) *έρυθροδανόν*/to dye red with *έρυθρόδανον* makes a clear terminological distinction between the material used for dyeing and the resulting red colour itself. For the scope of this research project, the complex process was reproduced by the author and compared with ethnographic material where this was possible. Epigraphic evidence and literary sources with instructions for dyeing will also be discussed.

## The Shades of Red-Pink Historical Inks: Investigation of the Degradation Pathway of Dyes in a 19<sup>th</sup>-20<sup>th</sup> Century Collection

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Over the centuries, ink's composition has undergone changes, leading to the introduction of synthetic dyes in their formulation in the late 19<sup>th</sup> century<sup>1</sup>. This recast has allowed ink manufacturers to obtain better performing inks than those made with natural dyes, opening up a wide palette of different shades and bright colours. From an analytical point of view, ink analysis is challenging due to the variety of materials and recipes, amongst which many are patented. Moreover, the ink dyes undergo degradation processes during ageing, further complicating their analytical characterisation in historical samples<sup>2</sup>. Thus, the study of the collections of historical-artistic materials is fundamental for improving our knowledge of technologies, synthesis and ink's composition of the early days of synthetic chemistry<sup>3</sup>. Moreover, the simulation of ageing processes of ink collections could lead to important insights for authentication and dating purposes<sup>4</sup>.

In the present work, a red-pink inks collection produced in France in the late 19<sup>th</sup> – early 20<sup>th</sup> century was fully characterized. For simulating ageing process that could naturally occur in historical manuscripts and ink drawings, reference ink mock-ups (cast as water dispersion on Whatman filter paper, pure cellulose) were exposed at room temperature (T=25°C) to natural indoor light, at RH c.a 50%. Liquid chromatography – tandem mass spectrometry (HPLC- MS<sup>2</sup>) characterization of unaged inks enabled us to highlight specific trends, showing that the formulations from the early days of synthetic industry were mainly based on rhodamine B and 6G, eosin Y, rose Bengal, cotton scarlet and amido naphthol red G. Moreover, by comparing the unaged and aged ink's molecular profile significant differences were detected, proving that different rates of fading and semi-quantitative variations occur depending on ink's composition and recipes. Finally, degradation products of several dyes were determined.

Our investigation supports art technological studies of historical ink collections, and it could bring important insights into the field of dye degradation by expanding the dataset available for ink identification in manuscripts and drawings.

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## From Scarlet to Crimson: Analytical and Historical Insight into Recipes with Lac Dye

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Stick lac, a resin-like substance produced by scale insects was traded into Europe since antiquity being the source of lac dye, a red dyestuff used for dyeing textiles. The colour diversity and fastness to light of lac dye were unanimously recognised by ancient dyers who developed throughout the time their recipes and unique skills in extracting the dyestuff from the resinous matrix and using it to obtain scarlet-to-crimson fabrics<sup>1</sup>.

Although the practice of dyeing textiles spans millennia, the number of historical documentary sources comprising recipes for dyeing with lac dye is scarce and the material and technical content of such books is yet to be fully explored. In this presentation, we will attempt to fill this knowledge gap by addressing the processes and formulations for dyeing woven wool with lac dye as recorded in dye books from the 18<sup>th</sup> and 19<sup>th</sup> centuries, including the unique Crutchley Archive<sup>2,3</sup>. To do so, we first tackled complex instructions and missing information by interpreting amounts, ingredients, additives, times and colour descriptions. Secondly, we developed a tentative approach for the historically accurate reproduction of lac-based recipes which provided a better understanding of the technical know-how as well as of similarities and differences between the books. Finally, the reconstructions were analysed through a multi-analytical approach using colourimetry, FORS, high-performance liquid chromatography, infrared and Raman spectroscopies and molecular fluorescence in the visible. The results were compared and validated with a database of reference lac-dyed broadcloths which enabled to pinpoint the recipes' specificities.

This study offers a first insight into the variety of molecular compositions of lac-based dyeing formulations, supporting advances in art technological source research while fostering future research on the characterisation and preservation of the original dyed patterns still present in the dye books.

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## Compound Specific Radiocarbon (<sup>14</sup>C) Dating of Anthraquinone-Based Colourants in Historical Textiles

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Any observer of an artwork speculates about two details: who is the artist and when was the object created. Answering these queries is not an easy task, however the second question may be addressed by radiocarbon (<sup>14</sup>C) dating. Such information, however, can only be obtained by sacrificing a sample from the object, a critical step owing to the unique and irreplaceable nature of art objects. Organic compounds isolated from natural sources are potential chronological markers for the object's life. Omnipresent in cultural heritage objects, organic colourants are found in a wide range of substrates, from different cultures, displaying different artistic techniques and preservation states, covering different time periods from 5000 BCE till today<sup>1</sup>. Carbon rich and short lived, the carbon isotopic ratio of organic colourants represents a snapshot of the atmospheric CO<sub>2</sub> during the years of growth of the organism (plant or insect) from which it was isolated. As such, these compounds represent ideal material for <sup>14</sup>C analysis and can serve as proxies for dating the creation of a coloured object, yet until today no such analysis has ever been conducted.

This work aims to contribute with new metrics to date cultural heritage objects<sup>2</sup>. With the focus on red anthraquinone dyes used in historical dyed textiles, we discuss preliminary results in the development of a new protocol combining dye analysis by ultra-high-performance liquid chromatography (HPLC)<sup>3</sup> and compound-specific radiocarbon analysis (CSRA). Blank assessment and constant contamination modelling are key parameters in highlighting the associated <sup>14</sup>C constraints within the different steps of the methodology covering the chemical extraction, chromatographic separation, and final <sup>14</sup>C analysis. The complementary combination of both techniques has the potential to support art historical interpretations about the origins of the objects, offering a more specific chronological time window on the objects' production, and furthering discussion on historical dyeing traditions.

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## Colours of Early Mycenaean Textiles

### First Observations

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Red and purple dyed textiles were a strong symbol of wealth and power throughout antiquity. The last years, more and more textile fragments dyed in red and purple hues are being discovered and studied in Greece, pushing the evidence for dyed textiles backwards in history. Such instances include the recent find of purple dyed fabrics in Stamna, Aetolia (MBA to LBA) and the textiles discovered in the so-called Heroon of Lefkandi (LBA).

A new scientific project entitled *The Fabric of Kings. Funerary Textiles from Mycenae and the early Mycenaean Textile Production (FAROS)*, funded by the Hellenic Foundation for Research and Innovation (2023-2025), aims to study the characteristics of the early Mycenaean textile industry in order to create a better understanding of the Mycenaean textile culture and its evolution through time. Through the study of newly re-discovered, unpublished textile fragments from the Grave Circles A and B of Mycenae (c. 16<sup>th</sup> c. BC) in the National Archaeological Museum storerooms and early Mycenaean textile tools, as well as the examination of the palatial iconography and the data mentioned in the Linear B archives, there is indeed a first-time opportunity to dive into the secrets of the early Mycenaean textile production.

Preliminary research demonstrates that the textiles deposited in the Shaft Graves were of the same high quality as the other rich offerings. Fragments from both Burial Graves preserve traces of red, as well as purple colour and the first analyses suggest the presence of real purple, symbol of power, and hematite. These fragments constitute the earliest red and purple dyed textiles in Greece. We shall discuss the evidence of colour, as well as the following actions to take to continue the study.

# «A Charlatan's Bogus Dye»?

## The 7<sup>th</sup>/8<sup>th</sup> Century Coptic Dyeing Recipe Papyrus Berlin P.8316 Revisited

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The Coptic Papyrus Berlin P.8316 provides a recipe — in fact *the most detailed* extant recipe from (late) antiquity — for the treatment of wool fibers with a mordant and subsequently with a vegetable dyestuff, most likely madder. Its significance was not realized by the first editor (Erman 1895, 1899, 1904) and completely mistaken by Stoyanova 2008. In a reedition (Richter 2020) I established the technical procedure as described in the papyrus and identified its epistemological setting in proto-alchemical traditions otherwise known from texts such as the two 3<sup>rd</sup>-/4<sup>th</sup> century papyrus quires kept at Leiden and Stockholm (Halleux 1981) and *The Four Books of Pseudo-Democritus* (Martelli 2013). Beyond the detailed account of a common ancient dyeing practice, Papyrus Berlin P.8316 offers recipe evidence for Pliny's report (*b.n.* xxxv, 42) about certain baffling artifices brought about by the dye workers of Egypt. The aim of the proposed paper is to bring this hitherto widely overlooked late ancient written testimony of dyeing technology to the awareness of a community of experts.

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## Analysis of the Materials in the Late 15<sup>th</sup>-Century Islamic Manuscript by Micro-Destructive Analysis Methods and the Contribution of the Results to Conservation Works

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In this study, the animal encyclopedia at an Ottoman Library, consisting of 62 volumes, and written on behalf of lives from the end of the 15<sup>th</sup> century to the beginning of the 16<sup>th</sup> century (1487-1501), was examined analytically. This historical object is the most comprehensive encyclopedic object on the animal/living world. The samples consisting of paper, ink, dye, and leather samples were analyzed using high-performance liquid chromatography with a diode array detector (HPLC-DAD). After all, information on material characterization and deterioration product(s) was obtained. As a result of the analysis, vanillic acid, and its derivatives due to the natural aging of the paper and chrysophanic acid and its derivatives caused by fungi, which are the result of unfavorable environmental factors, were determined. It was concluded that the ink used in the manuscripts was iron-gall ink. In addition, it was determined that madder, weld, buckthorn, redwood, rhubarb, or dock plants were used as dyestuffs in paper and leather samples. Moreover, it was deduced that the leather samples were tanned with the vegetable tanning method using tannins.

Since tannic acids were determined in the dyestuff analysis of three paper samples with ink, elemental analysis was performed on black ink by scanning electron microscope- energy dispersive X-ray spectroscopy (SEM-EDX). As a result of this analysis, the element of iron has been detected and it has been confirmed that the black ink is iron-gall ink. It is inevitable that this study will guide conservation experts in restoration and conservation studies.

## New Dye Analyses of Haithabu Textiles

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The archaeological excavations at Haithabu in Schleswig have yielded one of the most important collections of textiles dated to the Viking Age. Several larger garment fragments have been identified, that altogether provides an important foundation for exploring the clothing and cloth types used in this period. When the textiles were analysed in the 1980s and 1990s, only few of them were screened for dye stuffs with the results being that many had been undyed or dyed with walnut or other tannins. Since then, dye analysis methods have evolved immensely, with HPLC (high performance liquid chromatography) being the most accurate method, requiring ever smaller samples. In 2023, a new set of dye analyses have been performed using this method on a selection of textiles from Haithabu. The results provide an up-to-date picture of the dye stuffs used in the production of these Viking Age textiles, but possibly also of the preservation conditions and the conservation processes after their recovery.



## Textile Dyes of the Golden Horde Era on the Results of a Research of Archaeological Textile in Kazakhstan and Ukraine

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My acquaintance with the textiles of the Golden Horde era began in 2003. It was material from the excavations of the Ukrainian archaeologist G. Toshchev in the Zaporozhye region, which he brought to my laboratory in V.N. Karazin Kharkiv National University. In 2019, I received an offer to head a scientific laboratory in Kazakhstan.

To date, we have studied textiles of this time from the territory of Ukraine (4 archaeological sites) and Kazakhstan (3 archaeological sites).

The main part of the studied fabrics is archaeological silk. This is due to the peculiarities of the resistance of silk textiles to the aggressiveness of the archaeological environment.

Research methodology it's conducting chemical-technological research. We widely use characteristic reactions, which allow dyes to be attributed by the test method. This greatly facilitates our study of archaeological textiles.

The leading place is occupied by the study of **red** textile dye. The main thing here is the use of dyer's madder (*Rubia tinctorum*). There is also cochineal (*Dactylopius coccus*).

I researched blue and yellow textiles. The **blue** color was obtained by using indigo (*Indigofera*). **Yellow** textile dyes are the most common in the plant kingdom. We have attributed, for example, mignonette (*Reséda*). There are others.

Investigating the madder, it was found that the same plant can produce a different color. That is, the predominance of alizarin or purpurin depends on the place of growth of plant materials. We have established this experimentally. The assortment of fabrics and textile dyes coincides over a wide area of Eurasia. This is connected with the system of caravan roads (the Great Silk Road) and with the geopolitical processes of that time in Eurasia.

## Posters

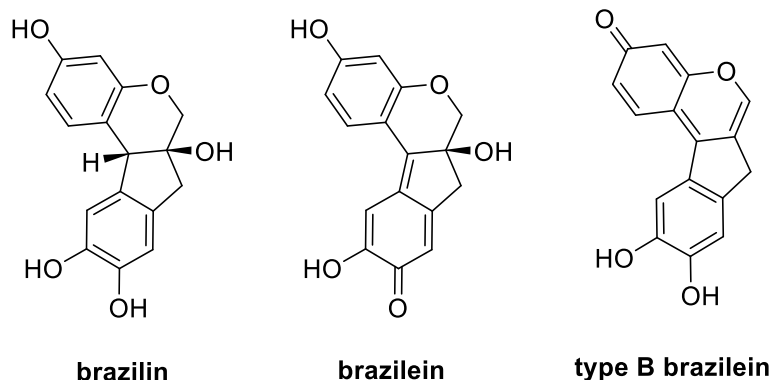
## Identification of the Unknown Type B Brazilin Derivative Marker Found in Brazilwood Dyed Textiles

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Natural red dyes may be distinguished in two classes. Anthraquinone derivatives, which can be extracted from plant sources of the *Rubiaceae* family and from scale insect belonging to the *Coccidea* family, or neoflavonoids found in redwood dyes extracted from various tree species collectively termed as brazilwood. The principal colouring matter of the latter is brazilin, which oxidizes to brazilain as the main chromophore. Similarly to haematoxylin and haematein, both analogs found in logwood, brazilin and brazilain are rarely identified in extracts of historical textile samples when using standard hydrochloric acid extraction methods.<sup>1,2</sup> The identification of the redwood dye is based on the presence of an unknown brazilain derivative (referred to as Bra' or type B) together with another yellow compound (referred to as compound C)<sup>3,4</sup> that was identified by Peggie *et al.* (2018) as being a benzochromenone, urolithin C.<sup>5</sup> This work unambiguously established the unreported structure of the type B brazilain derivative with a combination of synthesis, UV spectroscopy, high-resolution MS and NMR spectroscopy. The results support the hypothesis that brazilain upon HCl treatment was dehydrated similarly as hematein<sup>2</sup>.



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## The Colours of Textiles and Braids an an Early Medieval Hoard Buried in Galloway, Scotland in C. 900 CE

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The Galloway Hoard is a mixed material deposit buried c. 900CE in Dumfries and Galloway, now in modern-day Scotland, then part of early medieval Northumbria. The hoard includes a lidded vessel that contained numerous objects, some of which were wrapped individually, others in bundles.

The objects in two bundles, containing respectively a re-worked rock crystal and a set of socketed mounts, were protected by layers of plant fibre textile, silk and leather. One of the bundles, that holding the socketed mounts also contained one or more tubular braids. Similar braids were used in the vessel wrappings. A silk compound weave (samite), a weaving technology produced in Central Asia, Syria, and Egypt, is typically woven in multiple colours. Preserved in archaeological conditions, the textiles and braids are currently various shades of brown, some with faint traces of colour.

As part of the Arts and Humanities Research Council project Unwrapping the Galloway Hoard, samples of the textiles and braids were sent to the Textile Laboratory at the Royal Institute for Cultural Heritage in Belgium, for dye analysis. This poster outlines the initial dye analysis results in relation to the different textiles and braids in the hoard. The dye analyses provide the opportunity to understand the analytical characterization of the pigments, application of dyes and use of colours of the textiles and braid in the hoard, and potentially reveal information about their origins.

## Effects of Base Material (Silk or Paper) on the Use of Amur Cork Tree as an Indicator of Deterioration

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In a previous study, the components of Amur cork tree extracted from cultural textile were analyzed by HPLC, and it was shown that the sum of the chemical deterioration products of berberine could be used as an indicator of deterioration. Fluorescence lifetime measurement of Amur cork tree from cultural textile was also reported as an effective method for non-destructive analysis of the deterioration state of silk<sup>1</sup>. Here, we attempted to evaluate the deterioration of cultural property dyed textiles and ancient sutras dyed with Amur cork.

When the base material was silk, the deterioration products X1 and X2 of berberine are observed and fluorescence lifetime measurements report a shorter  $\tau_2$ . When the base material was paper, both X1 and X2 are observed, or only X2<sup>2</sup>. The fluorescence lifetime of  $\tau_2$  was shorter as in the case of silk. The obtained relationship between  $\tau_2$  and the amount of deterioration products of berberine showed a correlation with  $\tau_2$  when both X1 and X2 were observed, regardless of the base material, but not a strong correlation when only X2 was observed.

### **Acknowledgement**

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## Organic Pigments in the Excavation Findings and Monuments that are Under the Supervision of the Ephorate of Antiquities of the Thessaloniki Region

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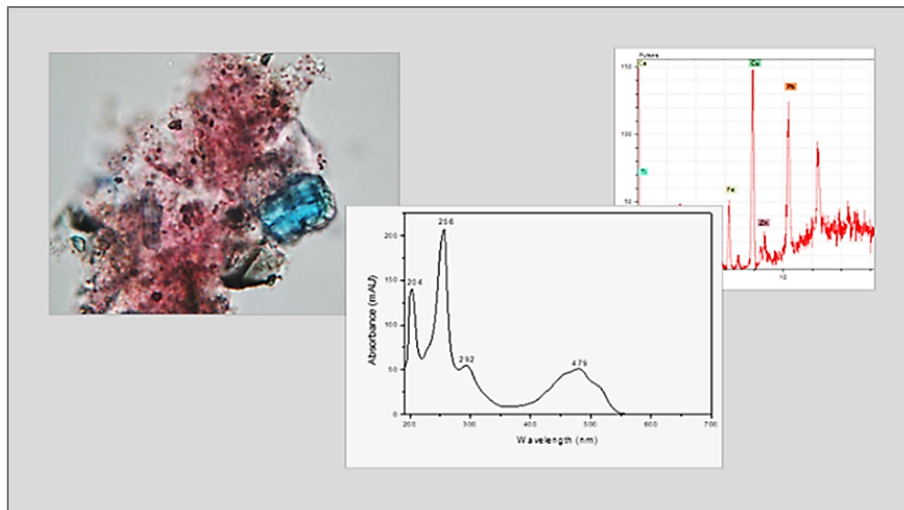
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The study presents the examination results of the organic pigments detected in excavated findings and monuments that are under the supervision of the Ephorate of Antiquities of the Thessaloniki Region. The examined organic pigments derived from the decoration of figurines and bird-shaped glass vessels that were discovered in the tombs of the eastern and western cemeteries of the Thessaloniki during the excavations at the site of the International Exhibition of Thessaloniki and the METRO of Thessaloniki. A pink organic pigment was identified in an architectural element (anthemion) in the circular tomb of Derveni. A sample of the ancient fabric from the cist-tomb in Lakkoma was also examined. The organic pigments from the decoration of the churches of the region of Thessaloniki were also submitted for examination. These are blue and pink colors in the iconostasis of the church of Agios Georgios in Sochos and a pink color in the mural of the church of Agios Andreas in Peristera. The analytical techniques m-XRF, Raman, and HPLC were used. The examination of the thin section of the samples with the polarizing microscope allowed the optical distinction of the dyestuffs from the pigments and other components of the sample. This examination also gave us information about the substrate of the dyes (microcrystalline calcite - chalk, lead white, kaolinite, etc.). The first identification of the main chromophores and other minerals of the samples was made by polarized optical microscopy. The optical properties of the pigment grains of the samples were examined (color, pleochroism, texture, refractive index, birefringence, polarization color, extinction, etc). The above-mentioned physicochemical analyses helped to determine the pigments and other components of the examined samples. Finally, the following organic pigments were identified: 1) madder and cochineal in the figurines, 2) madder in the bird-shaped glass vases 3) shellfish purple in the fabric, 4) madder in the circular tomb of Derveni, 5) beta-naphthol in the pink color from the mural of the church of Agios Andreas in Peristera and 6) madder, cochineal and indigo in the pink and blue color of the iconostasis of the church Agios Georgios in Sochos.



*Figure 1. Pink color on the figurine. Madder. Egyptian blue pigment grain. Polarizing microscope, m-XRF and UV/Vis spectrum (HPLC).*

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## Proteomics Approach to the Biomolecule Degradation of Indigo Dyeing Vessel Deposits

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Indigo dyeing occupies an important place in traditional Japanese dyeing, but the historical evolution of the production process remains unclear. To shed a new light on this aspect, this paper presents the initial results of proteomics research on excavated artefacts, museum collections, experimental objects for better understanding of biomolebular identification of dying vessels as well as the degradation process of organic materials related to indigo dyeing. Extracted proteins were digested by trypsin using FASP (Filter assisted sample preparation), then trypsin-digested peptides were analysed by nano LC-MS/MS (Thermo Orbitrap QE plus). The MS/MS searches conducted against uniprot all living species database using PEAKS software. False-discovery rate (FDR) of protein identification was set at 1%. More than 30 proteins were identified from excavated artefact samples. These identified proteins were derived from plant leaves and diverse microorganisms. Interestingly, the small acid-soluble spore proteins (SASPs) from *Bacillus* species were commonly found in excavated artefacts, museum collections and experimental object. It is known that SASPs play a role in the DNA protection from environmental stresses. Thus, we have confirmed good correspondence of microbe assemblage between excavated and current samples. In addition, the presence of indigo dye and related compounds was confirmed in modern vessels used for indigo dyeing.

## Dressed to Afterlife

### Dyes in Late Iron Age Children's Clothing in Finland

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Four Late Iron age (900–1200 CE) child burials from south-western Finland with textile remains were examined to deepen the knowledge of child clothing in the Viking Age and Crusade period Finland. The analysed textiles were from graves Masku Humikkala 15, Turku Kirkkomäki 35, Pöytyä Anivehmaa 13 and Salo Rikala 22.

As child burials are rare, the microarchaeological examination of their woollen textile fragments have brought new information of prehistoric children's costumes and colourants. The dye analysis focused on two twill fabrics, a bronze spiral decorated twill apron, a spiral decorated braid and a colourful tablet-woven band originating from a spiral decorated miniature shawl. Although the textile fragments are small, they revealed interesting colourants as well as also probable use of natural pigmented, but undyed yarns.

Of the 13 analysed samples, the HPLC analysis detected natural colourants in 5 yarns. The detected colourants were indigoids in visually blueish yarns and a reddish yarn, as well as three unidentified compounds in visually reddish yarns. It is not clear if these compounds are connected to the blue dyeing or to another biological dye source. These unknown colourants have been detected several times in the Finnish Late Iron Age textiles, but without any match to any reference dyeings so far.

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## Identifying Red Pigments Used in Paintings Via High-Performance Liquid Chromatography

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High-performance liquid chromatography is widely used for pigment identification in many fields, such as food industry, conservation of historical materials, and others<sup>1,2</sup>. In this work, HPLC is used for determination of red pigments in paintings and polychromy.

Two methods using HPLC were developed, one using methanol and water, and second using acetonitrile and water as mobile phases. For these two methods, several standards were measured and from these measurements a database of common red pigments was created. In this database, retention times for both methods and UV-VIS spectra are included. Aside from that, for several of the measured red pigment standard (such as alizarin, purpurin, carminic acid, and some modern dyes), the detection limit was determined.

As a second step, real samples from paintings were measured. Colorants from these samples were extracted and measured by the best optimal methods. The obtained chromatograms and spectra were compared with those measured for standards and evaluated.

### **Acknowledgements**

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## Which Pigments for Which Clothes? Reflections on the Colours and Materials Used to Depict Everyday Clothing in Gallo-Roman Wall Paintings

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Among the various representations on the wall paintings of Gaul, several belong to the register of everyday life. Within these very varied scenes, various types of clothing can be identified and correspond to the occupation or social class of the person represented. Whether they are lightly coloured, very colourful or sometimes patterned, these garments were painted by craftsmen using which pigments? This is one of the questions that will interest our paper, by listing the colours present but also by using the results of the pigment analyses that have been carried out and by comparing the images with the realia. Do the archaeological textile remains of clothing in Gaul correspond to the same reality in the use of dyes as those painted on the walls of buildings? Do we find the same pigments used for the same colour, in particular red, one of the most present colours?

## Colour4CRAFTS Research on the Cultural Tradition of Sustainable Bio-Based Colourants and Dyeing

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The project Colour4CRAFTS - ‘Colour for Combining, Re-engineering, Applying, Futuring, Transforming, Stretching!’ (2023-2026) is a multidisciplinary project that combines the traditional dyes and textile dyeing with the development of cutting-edge technology. Research groups from a range of fields and development businesses are joining forces to produce novel information on the use of dyes in historical times and further generate this knowledge for new high-tech applications of bio-based dyes. The project is hosted by the Faculty of Educational Sciences of the University of Helsinki, Finland. It is funded by a €4 million EU consortium grant under the Horizon Europe framework.

The poster introduces the historical part of the project that focuses on textile dyes and dyeing traditions in the North-Eastern Baltic (e.g. present-day territories of Estonia, Latvia and Finland) from a long-term perspective. Within the project, archaeological samples will be analysed with HPLC-MS and other methods to understand the history of colouration in this geographic area. The region is inhabited by diverse ethnicities, mainly Finno-Ugric and Baltic groups, allowing the evaluation of contacts between different subareas.

The primary sources of research materials are archaeological textiles from the 10<sup>th</sup> century onwards. In order to trace the continuing tradition of trades, the project also includes ethnographic records, mainly from the 18<sup>th</sup>-19<sup>th</sup> centuries. Although in the 19<sup>th</sup> century, synthetic dyes were introduced to the region of the study, the local traditional dyeing recipes persisted to a small extent until the 20<sup>th</sup> century. In order to trace the continuing tradition of trades, the project also includes published and unpublished ethnographic records, mainly from the 18<sup>th</sup>-19<sup>th</sup> centuries. Through the research, new information is expected to be found on the local and traded dyes.

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## Multi-Analytical Methodology for the Identification of Organic Dyes and Materials in Finnish Karelian Traditional Costumes

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Traditional costumes and crafts are fundamental marks of human civilization. They are works of art with significant historical, family and identity value, and are crucial for preserving the legacy of traditional culture. The scientific analysis of their materials brings insight into the interpretation of the artifacts themselves and amplifies the knowledge in the fields of archaeological and craft sciences.

As part of the multidisciplinary TRACTion (Traditional costumes and crafts in action) and Colour4CRAFTS projects, our research team is focusing on analysing the dyes and textile materials used in the traditional folk costumes of the Eastern Karelian Äyrämöinen women to shed light on the region's traditional dyeing and clothing making practices.

The Äyrämöinen women used to make and embroider dresses passing on their knowledge to the next generations and preserving many ancient features of prehistoric times in their costumes. The distinctive feature of the otherwise plain costumes was the colorful embroidery, for which all the dyes were derived from the local sources.

Our work aims to characterise the dyes and textile materials in the costumes (18<sup>th</sup>-20<sup>th</sup> century) using complementary non-invasive (hyperspectral imaging (HSI)) and minimally invasive analytical (real-time mass spectrometry (DART-MS)) methods. We will use HSI to map and characterise the chemical nature of the dyes and textile fibres used in these costumes. This technique combines spectral fingerprinting and imaging, allowing for investigation of large surface areas. DART-MS will identify the types of dyes used in such materials. Importantly, DART-MS is minimally invasive and relatively non-destructive, requiring only a single fibre for analysis while preserving the sample for possible further analysis.

Our study will assist in retrieving and expanding the inherited knowledge of the use of dyes in historical Karelian textiles. The analysis methodology we employ will provide new information about the composition of these valuable textiles while still preserving their integrity.

## Textile Dyes as Samples for the Colouring of Ancient Sculptures

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Antiquity was colourful. This fact has been widely accepted since the exhibitions " Gods in Colour, Polychromy of ancient Sculpture". In a large number of examples, historical colour settings could be analytically recorded and reconstructed. It became visible that colour-graded pigments and different roughnesses in the surface of the stone surfaces have a direct relationship to the textile to be depicted. Rougher surfaces with a high depth of colour, for example, can be interpreted as woollen fabrics, where lighter, smooth surfaces, on the other hand, tend to indicate finer types of fabric.

Dyeing experiments with traditional natural dyes from Greece, Turkey or the Middle East, based on very simple ancient recipes and applications show characteristic dyeing on different fabrics such as wool, cotton, linen or silk. The direct comparisons with the reconstructed colour settings on stone show that these simple dyeing have served as textile templates for the implementation on ancient stone sculptures. The textile sources are often not only used in their patterns, but also the coloration corresponds to the textile originals in an astonishing way.

The poster will show ways of non-destructive colour analysis and colour reconstruction as well as the colour comparison between pigment version and textile originals based of examples.



## Logwood from the bottom of the sea

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In 1768, *Fredensborg*, a Danish slave ship, wrecked practically on our doorsteps.

The ship was heading for Copenhagen after a trip in the triangular trade between Africa, the Caribbean and Europe, when a big storm ended the journey on the shore of Tromøy, close to Arendal in southern Norway.

In September 1974 it was discovered by local divers. An excavation took place, followed by much research, before an exhibition was made at the museum, where I work, in the

1990s. The divers found dyewood that was described both as redwood and logwood in the documents which the crew saved from the sea. The ship was holding a cargo of 50 000 kg of dyewood when it left St. Croix. It was still intact after more than two hundred years on the bottom of the sea, and the divers had noticed it gave a red color on the deck of the diving boat.

Several years afterwards, I received a piece of the logwood that came from the excavation of *Fredensborg*.

I also got a piece of logwood from another shipwreck. The *Lyra*, which wrecked in 1915 on its way home from the Baltic Sea, was discovered in 1965. This piece of logwood had been on the bottom of the sea for fifty years.

In our experiment, the two pieces of logwood, that had stayed on the bottom of the sea, was used to dye different pieces of fabric and yarn.

They were compared with new logwood that had not been in the water. The logwood from *Fredensborg* gave more colour than the logwood from



Lyra. These practical experiments were performed in a workshop with two other experienced dyers: Emily Halvorsen, who is a textile artist and dyer, and Ellen Bøe who is a museum teacher at the Stavanger Archaeological Museum.

## “Blinded by the Light”: Impact of Daylight Fluorescent Pigment Composition on Product Color and Light Stability

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Daylight fluorescent pigments were developed in the 1930s and in the subsequent decades have been featured by an array of artists. They are challenging materials for conservation due to their complex formulations consisting of a mixture of dyes with varying stability and optical brighteners impregnated in a polymer resin. Furthermore, these colorants are often intended to be displayed under blacklights which accelerate fading processes<sup>1,2</sup>.

Through LC-MS analysis the organic dyes and additives in different pigment sets were identified allowing for a molecular level understanding of the unique optical properties of the materials. For example, energy transfer can occur between components leading to an enhanced “pop” of the color. Unfortunately this can also lead to a greater perceived light fading during exhibition. Differences in the dyes, resin, and matrix additives were identified between manufacturers. Some of these variations have potential uses for conservators to identify and track treatments<sup>3</sup>. In addition, the light stability of daylight fluorescent pigments were investigated. The fading behavior of historic samples were compared with modern mock-ups to evaluate variations in stability between product formulations and to assess optimal exhibition conditions for preservation of the materials’ distinctive colors. Overall our work explores the fundamental photochemistry of daylight fluorescent pigments and how it can be used to inform preservation of art that features these vibrant materials<sup>3</sup>.

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## Traditional Iranian Dyeing Technique: Insights From a Hands-On Workshop Experience

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Iranian expertise in creating beautiful carpets and rugs is attributed to their expert dyeing techniques that have been passed down through generations as an inherited activity. However, the lack of scientific research has prevented the full disclosure of historical dyeing techniques in Iran. To address this, we visited Yazd, an ancient city and UNESCO World Heritage Site in Iran, to participate in a workshop with a Persian master dyer and to investigate the locally available Persian dyes and dyeing techniques. Based on interviews with the master dyer at the workshop, fifteen plant sources were selected for dyeing. These sources include oak (*Quercus persica*), saffron (*Crocus sativus*), madder (*Rubia tinctorum*), weld (*Reseda luteola*, collected in Iran and Afghanistan), prangos (*Prangos ferrulacea*), turmeric (*Curcuma longa*), henna (*Lawsonia inermis*), walnut hulls (*Juglans regia*), almond hulls (*Prunus eburnea*), pomegranate peel (*Punica granatum*), wild pistachio (*Pistacia atlantica*), vine leaves (*Vitis vinifera*), desert rod (*Eremostachys laevigata* (*gandal*)), myrobalan (*Terminalia chebula* (*halileh*)), and sumac (*Rhus coriaria*). However, traditional dyers may not know the scientific names of the plants used for natural dyes, which can make it difficult to provide accurate information on the sources of these dyes.

As part of the research in this workshop, we formulated dye mixtures with a number of listed dyes along with various types of mordants - including alum, which was the most commonly used option, as well as ferrous sulfate and copper sulfate. We also used mixtures without mordants to compare the effects of mordants on color.

For the study of cultural heritage collections, knowledge of the chemistry underlying the dyeing process is essential<sup>1</sup>. Particular emphasis was given to the use of saffron (*Crocus sativus*), as it is one of the world's most prized flowers, its use was widespread in Persia in the classical era<sup>2</sup>. The saffron flower consists of three main parts - the stigma, the petal, and the stamen. In this Workshop, extracts from these parts of the flower were used to dye wool. Analytical characterization of the different parts of the saffron flower and how the different mordants affect the final product will be carried out by colorimetry and high-performance liquid chromatography-diode array detector. To conclude, this study will allow a better understanding of this ancient knowledge that is mostly preserved through oral transmission, and we hope to gain insights that will help us to better understand the regional plants and dyeing techniques.

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## Van Gogh's Use of Logwood Ink in France

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Analysis by X-Ray Fluorescence Spectroscopy (XRFS) of the organo-metallic inks on 51 drawings and 26 letters, made by Vincent van Gogh, showed that, in France (1886-1890), he used chrome-logwood ink, whereas, in the Netherlands (1880-1885), he mainly used iron-gall ink. There is no indication from his letters that he knew which ink type he was using for writing and drawing. The German chemist Runge introduced chrome-logwood ink in 1847 as an alternative for iron-gall ink, because the acidity of the latter attacked the steel writing nibs. The results of XRFS-analysis of the writing inks on early 20<sup>th</sup> C. Dutch and French postcards, show that the former mostly were written with iron-gall ink, while the latter were written with chrome-logwood ink. This raises the question why, around 1900 in France, chrome-logwood ink was more popular than iron gall ink.

Since the 16<sup>th</sup> C., in Europe, logwood extract was used for dyeing textile. The Spanish brought the dyewood from Mexico to Europe. In 1765, the English chemist Palmer obtained a beautiful violet-brown colour 'Prune de monsieur', on wool with logwood extract by using tin salts as mordants. This became very popular in France. Around 1730, the French had brought the logwood tree to Saint-Domingue, present-day Haiti. In the 18<sup>th</sup> C., Saint-Domingue became France's most prosperous colony by the forced labour of over half a million enslaved people from French West-Africa. In 1791, self-liberated slaves started a revolution against French colonial rule, resulting in Haiti becoming independent in 1804. Due to the independence war, its economy collapsed and in, 1824, the government signed an unprofitable trade treaty with France. France then could import cheap logwood from Haiti, which explains why chrome-logwood ink was cheaper and became more popular than iron gall ink in France.

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