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# Cochineal Reds in Iberia and France: A Comparative Study of 18th Century Tin-Mordant Recipes to Dye Wool

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## Abstract

The Royal Textile Factory of Covilhã, founded in 1764, is the perfect example of the Portuguese Industrial and Cultural Heritage. Despite its historical significance, comprehensive studies on the dyeing techniques employed in the 18th century remain scarce. Given the influence of French technology on Portuguese wool production, this study presents a comparative analysis of French and Spanish dyeing recipes to understand their influence on the practices adopted by the Portuguese wool industry. Focusing on the production of red dyes from cochineal insects, one of the main colours used in Covilhã until the late 19th century, this work presents the reconstruction of selected 18th-century scarlet recipes. Quantitative and qualitative differences between French and Spanish methodologies were analysed, particularly regarding the use of mordants, the quantities of cochineal, and the role of pH and tin liquor in achieving scarlet shades. The results highlight that although both traditions relied heavily on cochineal, significant variations existed in recipe composition and application. This work contributes to a better understanding of historical dyeing techniques and supports future conservation and reproduction efforts for Portuguese textile heritage.

**Keywords:** wool; cochineal reds; historical recipes; dyed textiles; natural dyes

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## 1. Introduction

### *1.1. The Royal Manufacture of Covilhã*

Founded in 1764, during the reign of King José I (r. 1750–1777), the Royal Textile Factory of Covilhã (*Real Fábrica de Panos*) stands as a significant landmark of the Portuguese Cultural and Industrial Heritage. Operating until 1885, it remains a symbol of the Lusitanian wool industry and was recognised by the Portuguese legislation as a Property of Public Interest in 1982 [1,2].

Covilhã, a city with a deep-rooted connection to the textile industry, is located in the district of Castelo Branco, in the Beira Interior region of central-eastern Portugal (Figure 1), situated on the slopes of Serra da Estrela, a place that offers ideal conditions for wool production due to its rich pastures [3,4]. Despite recent developments to safeguard this

inheritance, uncovering the methods used to produce and dye textiles in Covilhã during the 18th century remains challenging, as documentation regarding the 121 years of production remains scattered around various archives and collections [1].

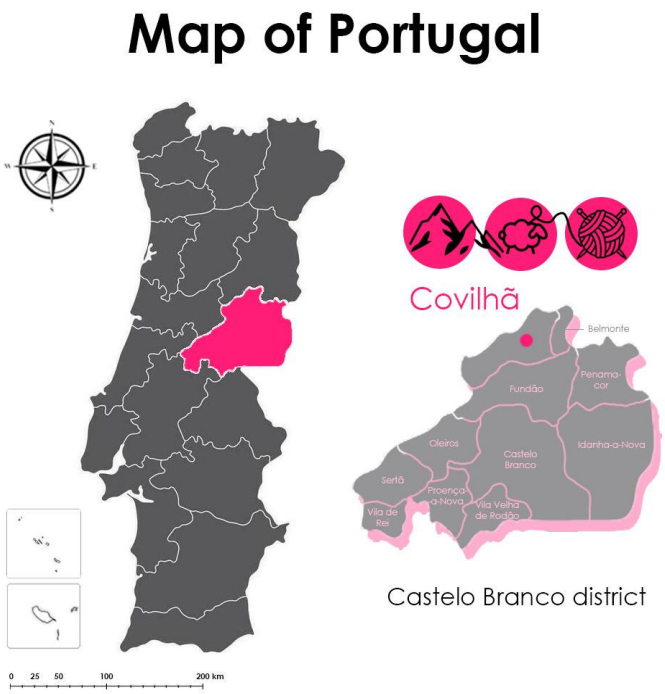



**Figure 1.** (**Left**): Map of Portugal. The Castelo Branco district is highlighted in pink, where Covilhã is located. (**Right**): Counties of Castelo Branco, highlighting Covilhã (pink dot). ©Mara Espírito Santo, 2025.

The establishment of the Royal Textile Factory was part of a broader industrial reform led by Sebastião José de Carvalho e Melo, Marquis of Pombal, then Secretary of State for Internal Affairs[1]. Highly influenced by the French industry, particularly the Manufactures des Gobelins and French ministers Cardinal de Richelieu and later Jean-Baptiste Colbert, Pombal aimed to modernise Portuguese industries, particularly the textile sectors (silk, flax and wool) [5]. Wool manufacture was considered "the most important, because by the universal consumption of their fabrics, woollen manufactures absorb, like sponges, most of the people's substance (income)"—(*a mais importante, porque pelo universal consumo dos seus tecidos absorvem as manufaturas de lã, como esponjas, a maior parte da substância dos povos*)[2] [1]. From this revival, Pombal was able to support the army's needs for uniforms and increase the country's wealth by diminishing the importation of English wool broadcloths [1].

To rejuvenate textile production in Covilhã, the Portuguese government recruited experts from all over Europe, including Ireland, England, Italy, France and Spain [6–8]. Among them were the Spanish master dyer Bernardo Rodriguez (1759–1764) [6] and the French master dyer Jean-Baptiste Salessis (1774–1791) [9], both of whom were brought to Covilhã to supervise the Factory's dyeing processes, working alongside local labourers and apprentices [1]. We know, for instance, that a Portuguese master dyer from Porto made a kind of internship with French master Salessis at Covilhã, during which he learned all the recipes for dyeing "fine" colours and blue as well [8].

Although recent research has explored Covilhã's industrial heritage in depth, no documentation has yet been found detailing the specific dye formulations used in the production of 18th-century woollen textiles. Nevertheless, the documented presence of Spanish and French dyers, together with the strong influence of French manufacturing practices, clearly indicates that a process of technological transfer did occur, shaping local

dyeing techniques. Particular attention should be given to the likely use of tin-based mordants, essential for producing vibrant scarlet reds.

In 1667, a new method for producing scarlet dye, using cochineal paired with a tin-mordant, was introduced in France by the Dutchman Jean Glucq, who had been invited by Colbert to establish a dyeing workshop in Paris, near the Gobelins. Shortly afterwards, his brother-in-law, François de Julienne, founded a cloth factory nearby. Their mutual nephew, Jean de Julienne (1686–1766), became involved in his uncle's workshops from a very young age, where he learned the full range of dyeing techniques [10].

A recently discovered letter in the Portuguese national archive, Torre do Tombo[3], reveals that Jean de Julienne offered his services to the King of Portugal at the time, stating that he would bring with him the *secret d'écarlate*—the new cochineal-based formulation using tin liquor. Although the letter is undated, it must have been written before 1729, when Julienne was formally granted control of his uncle's factories in Paris [10]. While his proposal appears not to have been implemented at the time, later records from the Royal Textile Factory of Covilhã, dated 1788, confirm the presence of tin-based materials at the site [8]. This indicates that such dyeing methods were eventually adopted and suggests that techniques originating in France and Spain did find their way into Covilhã's dyeing practices, even if only some decades later.

### *1.2. Historical Sources on Scarlet Dyeing*

This study aimed to assess the recipes used to produce scarlet reds, a colour popularised in the mid-17th century, where cochineal was used to obtain a bright red, alongside a tin mordant. In the absence of Portuguese documentation concerning these formulations, and considering the strong likelihood of technological transfer, four primary sources from countries of influence—namely France and Spain—were selected for analysis. These written sources were selected based on their accessibility and historical significance as they represent the few surviving 18th century documents on the art of dyeing wool. The two French sources are furthermore unique in that they illustrate the recipes for each colour with corresponding samples of dyed wool broadcloth. They also are particularly relevant in the Portuguese historical context of development of a wool textile industry since they were written by dyers who were dealing with large numbers of pieces per day (from 8 to 12 in average) and per year (more than 6000 pieces in average) and who had to strictly conform to high standards of quality and reproducibility [11–14].

#### 1.2.1. French Sources

Antoine Janot is the author of *Mémoire—on trouvera les operations de la teinture du grand et bon teint des couleurs qui se consomment en Levant avec la quantité et qualité des drogues qui les composent* (Memoir—on the processes of dyeing the colours that are consumed in the Levant in the good and fast mode of dyeing, with the quantities and qualities of the drugs from which they are obtained), dated 31 May 1744.

Born in Carcassonne in 1700, Janot was a master dyer whose contributions to textile dyeing in 18th-century France remain invaluable to the understanding of historical dye technologies. Trained under his father Jacques Janot, a master dyer at the Royal Manufacture in Bize, Antoine Janot continued the family trade by establishing a dyeing workshop in the textile town of Saint-Chinian. Operating at the heart of one of France's major wool broadcloth production centres, Janot served local clothiers by dyeing up to a dozen pieces of fine wool fabric daily, each intended for export to the Levant via Marseille [11,12,15].

His practice was strictly regulated under royal edicts issued in 1669 and 1671 by Louis XIV's minister Colbert, and updated in 1737, requiring the use of approved materials and methods to ensure colourfastness. Janot authored two more treatises, in 1744 and

in 1747. Together, his three memoirs documented some seventy recipes across a wide chromatic spectrum, each illustrated with dyed textile swatches. His detailed descriptions of the mordanting and dyeing processes constitute a crucial reference for both historical reconstruction and technical analysis of early modern dyeing practices [11,12,15].

Paul Gout is the author of *Mémoires de teinture* (Memoirs on Dyeing), dated from 1763.

Active during the mid-18th century, Gout distinguished himself both as a master dyer and as director of the Royal Manufacture in Bize, producing *Mémoires de Teinture* in 1763, an illustrated manuscript including four memoirs two of which are illustrated with dyed textile samples. It reflects the technological and ideological shifts in the Enlightenment era and is similar to Antoine Janot's manuscript in structure. Unlike Janot, whose practice was governed by rigid royal regulations, Gout enjoyed greater latitude in formulating dyes, enabled by his dual role as technical expert and factory director. This allowed him to experiment with innovative dyeing techniques and colour combinations, including those previously restricted by royal edicts [13,14].

Gout's writings exhibit a refined understanding of dyeing chemistry and market demands, particularly for exports to the Eastern Mediterranean. His documentation of both standard and experimental formulations has become an essential source for the study of 18th-century textile production, offering insights into evolving industrial practices and the transmission of artisanal knowledge within France's royal manufacturing system [13,14].

#### 1.2.2. Spanish Sources

Luís Fernández is the author of *Tratado instructivo, y práctico sobre el arte de la tintura: Reglas experimentadas y metódicas para tintar sedas, lanas, hilos de todas clases, y esparto en rama* (Instructive and Practical Treatise on the Art of Dyeing: Tried and methodical rules for dyeing silks, wools, threads of all kind, and raw esparto), published in 1778 [16].

A native of Toledo, Luís Fernández played a pivotal role in promoting dyeing reform in 18th-century Spain. His early training within the traditional textile sector enabled him to master the craft of dyeing, ultimately attaining the status of master dyer. This technical expertise, combined with his commitment to strengthening the domestic textile industry in the face of foreign competition and the support he received from the Five Major Guilds of Madrid, facilitated his relocation to Valencia, where he established his own dye workshop [16].

His professional reputation and network allowed him to assume key positions, including Inspector of Dyes and Director of the Royal Factory of the Five Major Guilds of Madrid in Valencia. His most prominent publication, *Instructive and Practical Treatise on the Art of Dyeing* (*Tratado instructivo y práctico sobre el arte de la Tintura*, 1778), stands as a testament to his technical acumen and practical experience. In his writings, Fernández consistently advocated for the modernisation of the Spanish textile industry, the establishment of manufacturing facilities throughout the kingdom, and expressed concern over the neglect of dyeing practices in the face of foreign industrial competition [16].

Miguel Jerónimo Suárez y Nuñes is the author of *Arte de teñir las lanas, sedas, hilo, y algodon, ò Compendio Universal de la teorica, y practica de la tintura, y quanto a ella corresponde. Tomo I.* (The Art of Dyeing Wool, Silks, Threads and Practice of Dyeing, and all that pertains to it. Volume I), published in 1779 [17].

Born in Madrid in 1733, Miguel Suárez emerged as a pivotal figure in Spain's economic and industrial development during the 18th century. Despite lacking familial ties to the textile industry and receiving no formal training in the field until later in life, Suárez may be characterised as a largely self-taught author in technical and scientific matters [17].

His association with the Royal Board of Trade, where he served as a fiscal agent and archivist, facilitated his advocacy for vocational education through the establishment of

schools and workshops. In 1764, he was appointed director of the silk factories of La Concepción in Cádiz, marking the beginning of his involvement in the textile sector. From 1766 onward, he conducted inspections of textile operations across Andalusia, Valencia, and Aragon to assess the sector's stagnation, and undertook study tours in Catalonia and France to acquire advanced knowledge of dyeing techniques, particularly in silk and wool. As both a writer and translator, Suárez rendered several French technical treatises into Spanish, including *The Art of Dyeing Wool (Arte de teñir las lanas*). He died in Madrid on the 21st of December 1791 [17].

### *1.3. Scarlet Reds*

Colour, often linked to social status and moral or spiritual qualities, can be considered a cornerstone of culture [18,19], and, as Goethe noted, "Red has always had a profound and magnificent effect" [19].

Among the abundance of reds that can be obtained from insects and plants, such as madder (*Rubia* spp.) and brazilwood (*Paubrasilia echinata* and *Biancaea sappan*) from plant sources, and from insect sources, kermes (*Kermes vermilio*), lac-dye (*Kerria* spp.), early sources of cochineal: *Porphyrophora* spp., including Polish cochineal (*P. polonica*) and Armenian cochineal (*P. hamelii*), and then American cochineal (*Dactylopius coccus)* [20,21]; one red stood out in the 18th century—the scarlet red. Historically, this bright, brilliant colour was obtained from red-dye insects. Colours derived from animal sources, despite being less abundant, are often highly concentrated in chromophores, resulting in high chromatic quality and good lightfastness. They are especially efficient for dyeing animal fibres [22–24]. This let to high demand, increased prices, and a strong association with royalty, prestige, and wealth [24–26].

One red stood out in the 18th century for its brightness and flame-like tone: scarlet red (see Figure 2). Historically, this brilliant red was obtained from the kermes insect, *Kermes vermilio*. The substitution of kermes for cochineal for dyeing scarlet still was somewhat recent in the history of European dyeing technology and has only been allowed by the invention of the tin mordant, discussed in this article. To produce these scarlet hues, typically a two-step dyeing process is needed: mordanting of the textile, to introduce the mordant agent to the textile, allowing the material to have a chemical predisposition to the chromophore, enhancing its affinity to the natural colour and form a stable dye-metal complex; and the dyeing, the addition of the colour to the fabric. Without these two steps, the dye would not bind as effectively to the textile, as opposed to a one-step process, for this colour, compromising both the colour and its durability.

Cochineal is among the dyeing materials documented at the Royal Factory of Covilhã in the 18th century, along with madder and, brazilwood [27,28].

Cochineal insects are plant parasites found across Europe, Asia, Africa, and Americas. This insect belongs to the family Coccoidea, with American cochineal (*Dactylopius coccus Costa*) being one of the most famous and desired sources of colour [25,26,29–31]. There are a variety of *Dactylopius* species, and it is not to be ruled out that in some cases, a mixture of wild and domestic cochineals may have been sold. Despite their differences, all cochineal species produce some carminic acid as an antimicrobial and anti-predatory defence mechanism [29].

*D. coccus* is usually found on cacti, such as Opuntia ficus-indica [25,26] or other species of Opuntia [29,31]. Although native to South America, its cultivation spread to South African and Mediterranean regions after the arrival of the Spanish Army to the New World [32,33]. The use of dye insects rich in carminic acid, to dye crimson to purplish shades of red, was already known and practiced by dyers both in Europe and Asia well before the 16th century [19,23]. The crimson-dyeing insects of the Old

World are known as *Porphyrophora*, belonging to the Margarodidae family that includes many species, many still being discovered and described today [26]. Among them are the "Polish cochineal" (*Porphyrophora polonica*) and "Armenian cochineal" (*P. hamelii*) [25,26,34]. Although chemically close to the American cochineal, these insects yield carminic acid in much lower quantities. Polish and Armenian cochineal yield only 0.6% and 0.8% of their dry body weight, respectively, and in contrast, the domesticated American cochineal produces approximately 20% of its dry body weight in carminic acid—about 25 times more than its European counterparts [20,29,31,35].



**Figure 2.** Standard for scarlet red on Londres Large cloth, preserved in the Archives départementales de l'Hérault, ref. C/2239. In 18th century France, such reference standards for the most important fast colours were preserved and commonly used in the control offices of all the main textile centres. Very few have been preserved. Photo copyright C. Cordier/Archives départementales de l'Hérault.

Carminic acid was first isolated in 1818 by French chemists Pelletier and Caventou. Like several other hydroxyanthraquinones, it is known for its photoreactivity [36]. The colour it produces is directly influenced by the position and number of hydroxyl substituents [37]. This polyprotic acid [37] is a β-C-glycosyl derivative of 9,10-anthraquinone [37,38]. Among anthraquinones, carminic acid has received significant attention due to its biological activity [39,40] and widespread use as a colourant in food, cosmetics, paintings, and textiles [36,41,42].

One of carminic acid's most notable characteristic is its sensitivity to pH changes, a feature that has attracted considerable scientific attention. Early studies such as Jørgensen & Skibsted (1991) and Favaro et al. (2002) explored its behaviour in solution, including photophysical properties and photodegradation, laying the groundwork for later research into its acid-base equilibrium [36,43].

Favaro et al. detected five deprotonation states between pH 0.9 and 13. They noted that the fully protonated form was visible in fluorescence spectra at pH ~1 [36]. At approximately pH 3, a monoanionic species ($CA^{-}$) appears, which aligns with the findings of Cañamares et al. Their results also revealed di-anionic ($CA^{2-}$) and tri-anionic ($CA^{3-}$) forms within pH ranges 4–7 and 9–12, respectively [39]. Similarly, Vitorino's identified five de-protonatable protons: one at the carboxyl group (position 2), and four on phenolic hydroxyls (positions 3, 5, 6, and 8) [37]. Using spectrophotometric and fluorometric titrations, she identified five distinct species within a pH range of 1 to 13, with the fully protonated form observed at pH 1 [36]. There are multiple proposed deprotonation pathways involving both carbonyl and hydroxyl groups, making the molecule highly responsive to metal ion bonding.

A possible acid-base equilibrium is shown in Figure 3, demonstrating the changeability of this chromophore under different pH values. These transformations highlight the chromophore's strong dependence on hydroxyl positioning, a trait shared among hydroxyanthraquinones [39,42]. Additionally, the arrangement of these hydroxyl groups makes carminic acid prone to complexation with metal ions, such as $Al^{3+}$ [37,44].

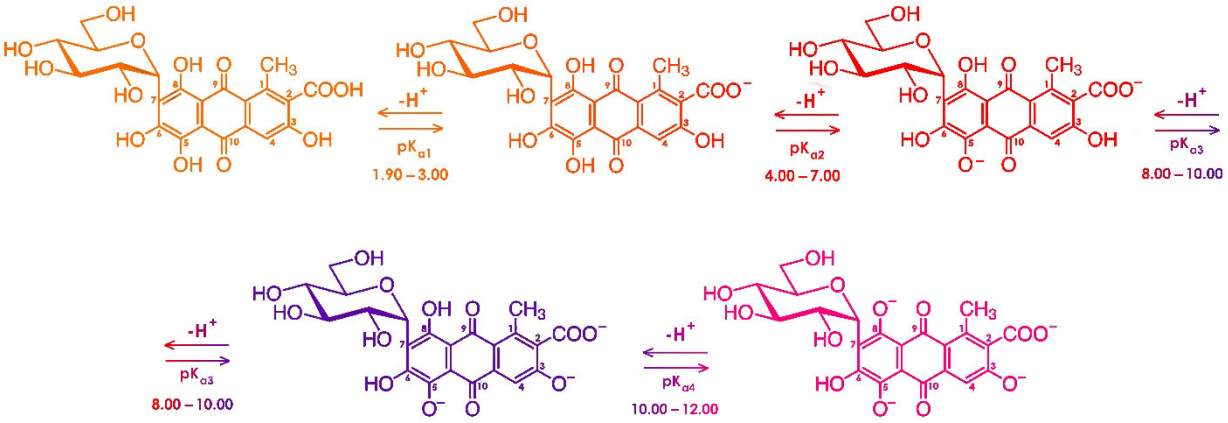



**Figure 3.** A possible acid-base equilibrium of carminic acid, based on data from Favaro et al. [36], Danfei Liu et al. [45], and Dapson [46].

The effect of pH on carminic acid's molecular structure within the dyeing context will be discussed further in the Results & Discussion section.

The other key ingredient for the scarlet red is tin liquor, shifting the typical crimson colour of cochineal to the most desirable bright scarlet red. Its discovery is attributed to the Dutch inventor Cornelius Drebbel (1572–1633), who worked as an engineer for the Kings of England [47]. While Drebbel is credited with the discovery, it was his apprentices, the Kuffler brothers, who refined the process and commercialised the resulting dye [47]. By the mid-17th century, this distinctive red had become known in London as *Color Kufflerianus* or *Écarlate de Bow*, and in Paris as *Écarlate d'Hollande* [25,26]. As previously noted, the method was introduced in France in 1667 by Dutchman Jean Glucq. It soon spread across Europe, with each master dyer preparing their own version using water, cochineal, salt (NaCl or $NH_4Cl$), metallic tin (often from Cornwall, England), and *aqua regia*. See Supplementary Material for more information.

## 2. Materials and Methods

### *2.1. The Historical Sources*

The primary sources used in this study, which contain the dyeing recipes selected for analysis and reconstruction, are as follows.

From France:

- Title: Memoir—on the processes of dyeing the colours that are consumed in the Levant in the good and fast mode of dyeing, with the quantities and qualities of the drugs from which they are obtained (*Mémoire—on trouvera les operations de la teinture du grand et bon teint des couleurs qui se consomment en Levant avec la quantité et qualité des drogues qui les composent)*. Year: 1744. Author: Antoine Janot; manuscript in Archives départementales de l'Hérault, Montpellier, France, reference C/5569, https://archivespierresvves.herault.fr/ark:/37279/vta4c4f2666d9aa0cef/daogrp/0/111 (accessed on 4 September 2025). Edition: D. Cardon, *Des Couleurs pour les Lumières—Vie et Oeuvres d'Antoine Janot (1700–1778)*. Paris: CNRS Editions, 2019 [11].
- Title: Memoirs on Dyeing (*Mémoires de teinture).* Year: 1763. Author: Paul Gout; manuscript, private collection. Edition: D. Cardon, *Mémoires de teinture—Voyage dans*

*le temps chez un maître des couleurs*. Paris: CNRS Editions, 2013; translation into English: D. Cardon, *The Dyer's Handbook—Memoirs of an 18th century Master Colourist*. Oxford and Philadelphia: Oxbow Books, 2016; new ed. 2020 [14].

From Spain:

- Title: Instructive and Practical Treatise on the Art of Dyeing: Tried and methodical rules for dyeing silks, wools, threads of all kind, and raw esparto (*Tratado instructivo, y práctico sobre el arte de la tintura: Reglas experimentadas y metódicas para tintar sedas, lanas, hilos de todas clases, y esparto en rama)*. Year: 1778. Author: Luis Fernandez. Place: Madrid; Publisher, Imprenta de Blas Roman.
- Title: The Art of Dyeing Wool, Silks, Threads and Practice of Dyeing, and all that pertains to it. Volume I (*Arte de teñir las lanas, sedas, hilo, y algodon, ò Compendio Universal de la teorica, y practica de la tintura, y quanto a ella corresponde. Tomo I)*. Year: 1779. Author: Miguel Jerónimo Suárez y Nuñes; Place_ Madrid; Publisher: Imprenta de Pedro Marin.
  Conversions were based on Manuel Carrera Stampa's article: "The Evolution of Weights and Measures in New Spain" (1949) [48].

### *2.2. The Materials*

Millipore ultrapure water was used for the mordant baths and the dye baths. Potassium aluminium sulphate dodecahydrate ($AlK(SO_4)_2 \cdot 12H_2O$) and tin(II) chloride dihydrate ($SnCl_2\ 2H_2O$) were purchased from Sigma-Aldrich (Darmstadt, Germany); wheat starch ($(C_6H_{10}O_5)_n$) was purchased from José M. Vaz Pereira, S.A. in Lisbon; citric acid 1-hydrated ($C_6H_8O_7\ H_2O$) was purchased from PanReac (Barcelona, Spain); ammonium chloride ($NH_4Cl$) was purchased from Riedel-deHaën® (Seelze, Germany); and cochineal and turmeric were purchased from KremerPigmente® (Aichstetten, Germany). Crude tartar, white and red ($KC_4H_5O_6$) was collected from barrels of organic wine produced by Patrick and Alexis Maurel's winery Terres du Pic, Mas de La Liquière, 34380 Mas-de-Londres, France.

The wool was 100% pure wool broadcloth woven from the Ile-de-France breed of sheep at Eric Carlier's workshop: Le Passe-trame, rue du Moulin Gau, 81660 Payrin-Augmontel, France. The density in the warp is 14 threads per cm, and in the weft is 15 threads per cm.

### *2.3. Analytical Techniques*

#### 2.3.1. Colourimetry

For measuring colour, a handheld spectrophotometer Lovibond® TR 520 (Amesbury, UK) with a diffused illumination system, 8° viewing angle, and 48 mm integrating sphere was used. The measuring aperture was 8 mm in diameter. Equipment calibration was performed with white and black references. Colour coordinates were calculated defining the D65 illuminant and the 10° observer. The colour data are presented in the CIE-Lab 1976 system. In the Lab Cartesian system, L*, relative brightness, is represented by the z-axis. Variations in relative brightness range from white (L* = 100) to black (L* = 0). The (a*, b*) pair represents the hue of the object. The red/green y-axis plots a*, ranging from negative values (green) to positive values (red). The yellow/blue x-axis reports b* going from negative numbers (blue) to positive numbers (yellow). All samples were measured three times, and an average was calculated (see Supplementary Materials Table S1).

#### 2.3.2. pH Measurements

For the pH measurements, a Sartorius Docu-pH Meter (Göttingen, Germany) was used, calibrate with two points (buffers 4 and 7 at 25 °C). The solutions were measured once at room temperature, around 25 °C.

### *2.4. The Recipes*

The recipes analysed and reconstructed in this study (Table 1) were translated and interpreted from the historical sources listed above. All the values presented, from the Spanish printed sources, were calculated using the quantities used before the 19th century in Castile, given that the authors were both based in Madrid, Spain. (see Supplementary Materials Table S2).

### *2.5. The Methodology*

Initially, fifteen recipes were selected for analysis (Table 1). These were chosen based on the recipe name—named "Scarlet" or "Flame Colour"; the final colour—recipes where the name was not one of the two above described, but presented a similar shade of red (in the case of the French recipes, where original dyed samples were available); and lastly, the ingredients used—recipes containing cochineal and tin liquor. The aim was to analyse similarities and differences between recipes, offering a critical comparison of each author, highlighting national patterns, and assessing how French and Spanish traditions diverge.

From the recipes listed, seven were selected for reconstruction: Antoine Janot's *Écarlate*; Paul Gout's *Écarlate* in his first Memoir; Miguel Suárez's *Escarlata color de fuego* and *Escarlata de Venícia*; and Luis Fernández's *Escarlata*, *Color de Zereza*, and *Color de Punzó* (Figure 4). The selection of these recipes was based mainly considering the name of the recipe, "Scarlet", in the case of the French master dyers. For the Spanish recipes, there are no samples available that would allow verifying the original colour, and so all recipes were selected to be reconstructed.

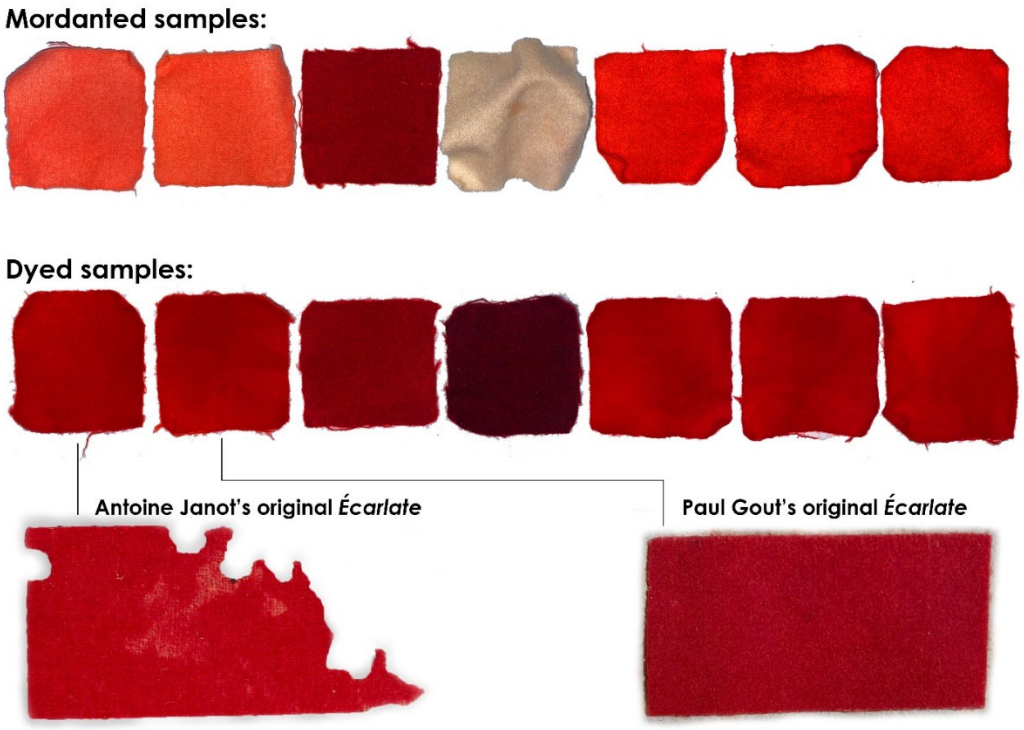



**Figure 4.** (**Upper**) and (**middle**) rows: Mordanted and dyed samples obtained during the reconstruction of the recipes provided by the following master dyers/authors: (from left to right) Antoine Janot's *Écarlate*, Paul Gout's *Écarlate*, Miguel Suárez's *Escarlata color de fuego* and *Escarlata de Venícia*, and Luís Fernández's *Escarlata*, *Zereza* and *Punzó*. ©Mara Espírito Santo 2025. (**Bottom left**): Antoine Janot's scarlet, folio 8 verso of his first *Memoir*. Archives départementales de l'Hérault, ref. C/5569. Photo copyright C. Cordier/Archives départementales de l'Hérault; (**Bottom right**): Paul Gout's scarlet in his second *Memoir*, page 45 of the manuscript. Private collection. Photo copyright D. Cardon.

**Table 1.** Recipes for dyeing scarlet red from the master dyers Antoine Janot (1744–1747), Paul Gout (1763), Luis Fernández (1778) and Miguel Jerónimo Suárez y Núñes (1779). Values presented are in %WOF: Percentage of weight of fabric.

| Master/Author: | Recipe Name: | Mordant Bath: | | | | | | | | | Dye Bath: | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Cochineal | Tin Liquor | Red Tartar | White Tartar | Alum | Acid Water | Ammoniac Salt | Young Fustic | Remaining Dye Bath * | Cochineal | Tin Liquor | Starch | Alum | Turmeric | Bran |
| Janot | Écarlate | 0.5 | 18.5 | | 8 | | | | | | 7 | 9.25 | 8 | | | |
| | Écarlate de feu | 0.5 | 18.5 | | 8 | | | | 4 | | 6 | 9.25 | 8 | | | |
| | Jujube | 0.5 | 18.5 | | 8 | | | | 16 | | 3 | 9.25 | 8 | | | |
| Gout | Écarlate | 0.37 | 22 | | 8 | | | | | | 6.6 | 16 | | | | |
| | Écarlate 2 | | 24 | | 8 | | | | | Écarlate | 7 | 16 | | | | ? ** |
| | Couleur de feu | | 16 | | 8 | | | | 16 | Écarlate | 6 | 16 | | | | |
| | Couleur de feu 2 | | 16 | | 8 | | | | 16 | Couleur de feu | 6 | 20 | | | | |
| | Fleur de grenade | | 2 | | 8 | | | | 24 | Écarlate 2 | 5 | 16 | | | | |
| | Jujube | | 12 | | 8 | | | | 24 | Écarlate 2 | 4 | 8 | | | | |
| | Jujube 2 | | 16 | | 4 | | | | 20 | Pourpre | 3 | 8 | | | | |
| Fernández | Escarlata | 3 | 66 | | 12.5 | | | 1.6 | | | 6 | 33 | | | | |
| | Zereza | 4.7 | 66 | | 12.5 | | | 2 | | | 6 | 33 | | | | |
| | Punzó | 4.7 | 66 | | 12.5 | | | | | | 6 | 33 | | | 1.6 | |
| Suárez | Escarlata de Venícia | | | 10 | | 20 | 1/5 | | | | 75 | | | 0.1 | | |
| | Escarlata color de fuego | 18.8 | 12.5 | | 12.5 | | | | | | 4.7 | 12.5 | 3 | | | |

Note * The mordant bath reuses a previous cochineal dye bath, the recipe for which is specified in each case; ** No quantities mentioned.

The recipes selected for reconstruction were reproduced only once, using the same quantities, temperatures, and durations in the mordant and dye baths described in the recipes available. To confirm reproducibility, more experiences need to be conducted, as the main goal of this paper was to determine how similar the French and Spanish traditions are. Future studies may include reconstructions of the recipes first explored in the present paper, as well as a well thought-through recipe variations, to assess the influence each ingredient has.

#### 2.5.1. The Mordanting

For all the recipes, except for Miguel Suárez's *Escarlata de Venícia*, the following method was used[4]: Approximately 350 mL of ultrapure water (Milli-Q®, Darmstadt, Germany) was added to a 600 mL capacity beaker, and heated to 40 °C. Once this temperature was reached, all the ingredients[5] indicated in the recipe, except the tin chloride[6], were added to the water, maintaining constant stirring. As the temperature rose to 80–90 °C (just below boiling), the tin chloride was added, turning the colour of the solution from a pink shade to a red hue. When the bath reached 100 °C, the wool samples[7] were immersed and kept under these conditions for the following durations: 1 h—Luis Fernández's *Escarlata*, *Zereza*, and *Punzó;* 1 h 30 min.—Miguel Suárez's *Escarlata color de fuego*; and 2 h—Antoine Janot's and Paul Gout's *Ecarlate*. At the end of the mordanting period, samples were taken out, gently squeezed, and then washed with ultrapure water only, and air-dried. A scheme of this process is illustrated in Figure 5, below.

For Miguel Suárez's *Escarlata de Venícia*, the recipe was originally conceived for use with kermes of "extreme quality for tapestry", but the author notes that cochineal could be used instead, either partially or entirely, with the addition of acid water to the mordant bath. The latter option was selected for this study, substituting the kermes entirely. The process began by adding 300 mL of Milli-Q® water to a 600 mL beaker, followed by the addition of 80 mL of acid water (ultrapure water with 0.5 g of citric acid). Separately, alum was dissolved in 20 mL of water, and added to the beaker, along with the ground cochineal, at around 40 °C. Once the solution reached the boiling point, the wool sample was added, and the temperature and agitation were maintained for 2 h. After this time, the wool sample was taken out of the water, squeezed gently, and put in a linen bag to dry for 5–6 days, allowing the mordant to be fully absorbed.

#### 2.5.2. The Dyeing

For all the recipes, except for Miguel Suárez's *Escarlata de Venícia*, the following method was employed[8]: Roughly 300 mL of ultrapure water was added to a 600 mL beaker, enough to fully submerge the samples. All the ingredients required for each recipe could be added before the water was hot, ensuring constant mixing from the beginning. When the solution reached a temperature close to 90 °C (just before boiling), the tin chloride was added and allowed to dissolve completely. Once the bath reached the boiling point, the mordanted wool samples were introduced and dyed according to the following durations: For Janot and Gout's *Écarlates* the time required is dependent on the quality of the bath, and so the samples were dyed for 20 min, until the desired colour was achieved; for the colours of Luis Fernández, each dye bath had a duration of 30 min; and for Miguel Suárez's *Escarlata color de fuego* the dye bath lasted for 1 h 30 min. After dyeing, the samples were taken out, washed with ultrapure water, and allowed to dry at room temperature. A scheme of this process is illustrated in Figure 5, below.

For *Escarlata de Venícia* of Miguel Suárez, the dyeing process started the same way as its mordanting process. 280 mL of ultrapure water was added to a 600 mL beaker. Separately, alum was dissolved in 20 mL of warm water, and then added to the main beaker

at around 40 °C. Just before the solution reached the boiling point (80 °C), the grounded cochineal was incorporated into the solution. As soon as the temperature reached 100 °C, the mordanted wool was submerged in the dye bath and left to dye for 1 h, under constant agitation and temperature. At the end of the process, the sample was gently squeezed, rinsed with ultrapure water, and left to dry.

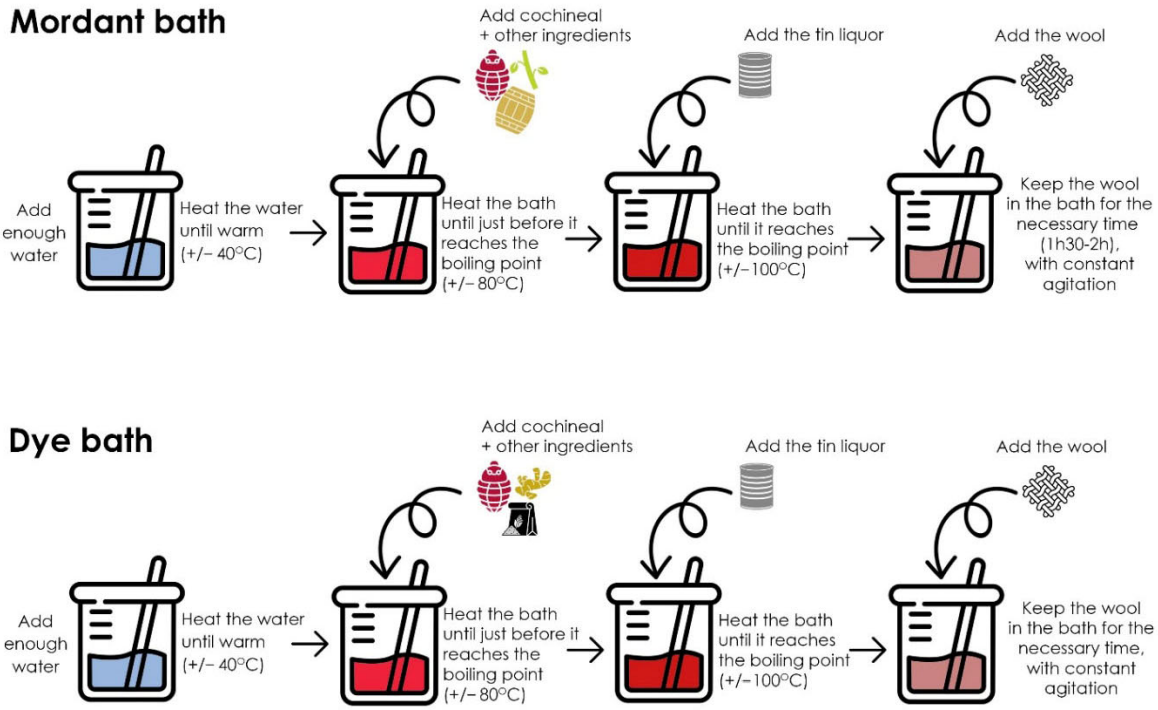



**Figure 5.** Scheme illustrating the process to mordant and dye a typical scarlet red ©Mara Espírito Santo 2024.

## 3. Results & Discussion

### *3.1. Comparative Study of the Recipes*

Although a reduced number of recipes were selected to reconstruct, it is important to assess all the formulations found to produce a scarlet red and all the various shades, since this study aims for a meaningful comparison between countries.

Analysing the fifteen recipes selected for this comparative study (Table 1), at first glance, only half (53%) present the use of cochineal in both the mordanting and dye bath. However, in six of Paul Gout's recipes, the mordant bath reuses a previous cochineal dye bath. If this is taken into account, all but one—Suárez's Escarlata de Venícia—use cochineal in both stages (93.3%). Interestingly, the French recipes use considerably less cochineal in the mordant bath than the Spanish recipes, with the average being around 0.47%, whereas the Spanish present a 7.8% average. In the dye bath, all the recipes use cochineal, with various quantities. For the French recipes, the average of cochineal used in the dye bath is 5.4%; for the Spanish, this is around 19%. However, Miguel Suárez's *Escarlata de Venícia* stands out, using 75% of cochineal. By contrast, the average amount of cochineal used in the remaining Spanish recipes is only 5.2%, making Suárez recipe much more concentrated in dye material, highlighting its divergence from the typical practice to dye a scarlet red with cochineal. In this instance, the amount of cochineal used by the Spanish authors in the mordant bath is considerably higher, and the quantity used in the dye bath is very similar between all four authors, except for *Escarlata de Venícia.*

Tin liquor appears in 93% of the recipes, used in both mordanting and dyeing steps. Average concentrations are 27.3% and 17.1%, respectively. In Paul Gout's manuscript, these

quantities vary from 2% to 24%, relating to the redness of the colour. The redder the shade, the greater quantity of tin liquor is employed in the mordant bath. This can be seen in his *Fleur de grenade* where only 2% is used, and *Écarlate 2*, where 24% of tin liquor is employed, plus the remaining dye bath of the previous *Écarlate*, where this ingredient was already present. In Antoine Janot's recipes, as well as Luis Fernández's, the amount of tin used remains consistent in both processes, and the final shade is manipulated by the quantity of cochineal and/or by adding a yellow dye. However, there are notable variations in the quantities of tin liquor used. Among the authors studies, Luis Fernández stands out for using exceptionally higher amounts—66% in the mordant bath; and 33% in the dye bath.

Once again, *Escarlata de Venícia* from Miguel Suárez stands out, being the only one that does not make use of a tin liquor but instead uses acid water and alum to mordant the wool cloths. It is interesting that by manipulating the pH of the baths, lowering it significantly to pH = 1.95, the colour obtained is indeed red; however, it is not a scarlet red.

The recipes found for tin liquor are typically brief and lack precise instructions, particularly regarding the concentration of *aqua regia*. The solution was usually prepared the day before use and applied within 15 days. Paul Gout, for example, noted the importance of balance in its composition, though exact proportion remains unknown. Nevertheless, this study reinforces the notion that tin liquor was very much a particular secret for each master as Miguel Suárez stated, "there is no master dyer who doesn't have a unique recipe to make the scarlet, and each one is convinced that their own is the best of all" (*No hay Tintorero que no tenga una receta particular para hacer la escarlata, y cada uno está persuadido à que la suya es la mejor de todas*).

Despite its historical importance, no systematic study has explored the chemistry of tin liquor. Each formulation likely affected the final colour's brightness, hue, and lightfastness. Modern reconstructions using tin(II) chloride as a safer substitute have shown that a proportion of 3%WOF allows to obtain scarlet samples with DE2000 differences of between 1 and 2 with scarlet samples in Janot's and Gout's manuscripts [12], the use of tin liquor raises concerns regarding its environmental toxicity. While this ingredient was essential to achieve the desired scarlet shade, its use in conservation studies and artisanal reproductions must balance history with sustainability.

Regarding the use of tartar, all the recipes presented make use of this ingredient in the mordant bath, and every single one, except Miguel Suárez's *Escarlata de Venícia*, make use of white tartar, instead of the red used in this recipe. The quantities vary, and notably, the Spanish recipes tend to use more of this ingredient, consistently using 12.5%, apart from Suárez's *Escarlata de Venícia*, where only 10% of red tartar is used. The French recipes use less, with quantities, most using 8%, and one recipe using 4% of tartar.

As mentioned, some recipes make use of a yellow dye to shift the hue of the colour, most precisely eight recipes: seven from the French master dyers and only one from the Spanish author Luis Fernández. Interestingly, both Antoine Janot and Paul Gout use young fustic (*Cotinus coggygria*) for this purpose, and Luis Fernández uses turmeric (*Curcuma longa*). It is noteworthy that none of these recipes are labelled as "scarlet," suggesting the use of yellow additives was reserved for creating alternative red tones.

### *3.2. Analysis of the Reconstructions*

Seven recipes were selected and reproduced from the historical sources, resulting in a range of red hues (Table 2). Colorimetry measurements (Figure 6) show that five of the recipes successfully produced what can be categorised as scarlet red, which is established between the ranges of CIE L*a*b*of 33–43, 45–55, and 24–34, respectively. These values were considered based on Domique Cardon's work, [11] page 196. The remaining two, both by Miguel Suárez, yielded darker or non-scarlet tones (see Supplementary Materials Table S1).

**Table 2.** Results from the reproduction of the selected recipes to dye scarlet red. Values presented are in %WOF: Percentage of weight of fabric.

| Master/ Author: | Recipe Name: | Colour: | Mordant Bath: | | | | | | | | Dye Bath: | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | Cochineal | Tin Liquor | Red Tartar | White Tartar | Alum | Acid Water | Ammoniac Salt | pH * | Cochineal | Tin Liquor | Starch | Alum | Turmeric | pH * |
| Janot | Écarlate | | 0.5 | 18.5 | | 8 | | | | 3.74 | 7 | 9.25 | 8 | | | 2.47 |
| Gout | Écarlate | | 0.37 | 22 | | 8 | | | | 3.69 | 6.6 | 16 | | | | 2.35 |
| Fernández | Escarlata | | 3 | 66 | | 12.5 | | | 1.6 | 3.66 | 6 | 33 | | | | 2.46 |
| | Zereza | | 4.7 | 66 | | 12.5 | | | 2 | 3.51 | 6 | 33 | | | | 2.56 |
| | Punzó | | 4.7 | 66 | | 12.5 | | | | 3.47 | 6 | 33 | | | 1.6 | 3.03 |
| Suárez | Escarlata de Venícia | | | | 10 | | 20 | 1/5 | | 1.95 | 75 | | | 0.1 | | 3.38 |
| | Escarlata color de fuego | | 18.8 | 12.5 | | 12.5 | | | | 3.55 | 4.7 | 12.5 | 3 | | | 3.13 |

* pH measured from the reconstructions made, using $SnCl_2$.

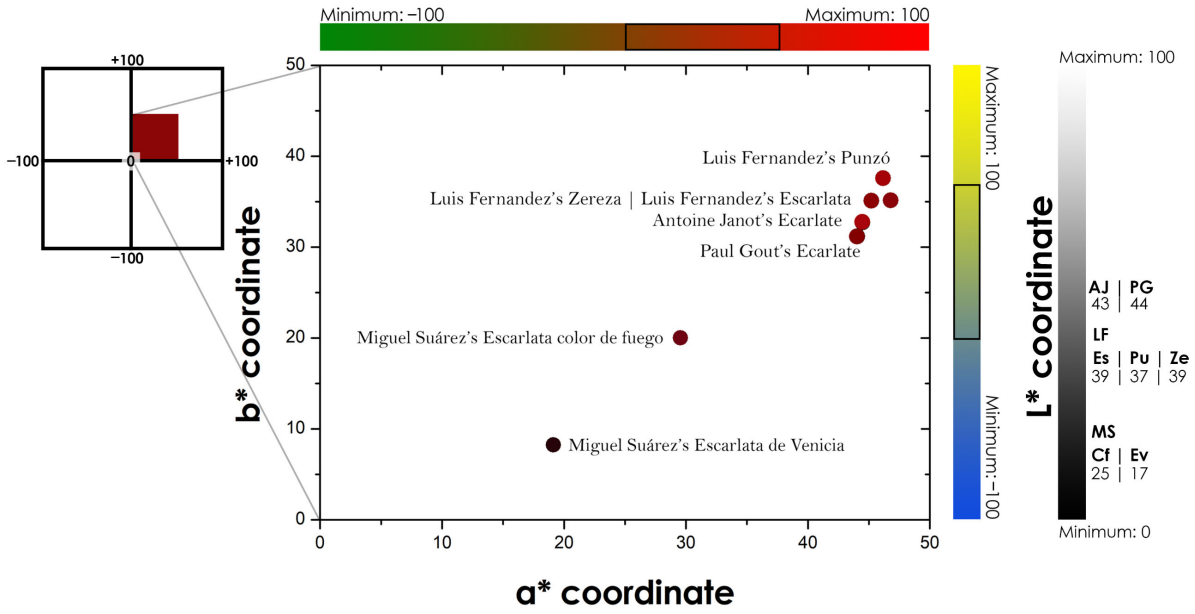



**Figure 6.** Colorimetry measurements from the samples obtained by the historical reproduction of the masters' written recipes. AJ—Antoine Janot's *Écarlate*; PG—Paul Gout's *Écarlate*; LF—Luís Fernández; Es—*Escarlata*; Pu—*Punzó*; Ze—*Zereza*; MS—Miguel Suárez; Cf—*Escarlata color de fuego*; Ev—*Escarlata de Venícia*. ©Mara Espírito Santo 2024.

Photographs of the reconstructed mordanted and dyed samples can be found in Figure 3, Section 2.5. *The Methodology*. Photographs of all the samples of Antoine Janot and Paul Gout can be found in Dominique Cardon's books: *Des Couleurs Pour Les Lumières—Antoine Janot, Teinturier Occitan, 1700–1778*, and *The Dyer's Handbook—Memoirs of an 18th-Century Master Colourist*, respectively.

Among the samples, those obtained from Miguel Suárez's recipes diverged the most. This was expected from his *Escarlata color de Venícia* since it is an adaptation from a known recipe used to dye tapestries with kermes and alum. This recipe is the only one that does not make use of the very important tin liquor, using instead acid water and alum. This supports the assertion that tin mordants are fundamental in shifting cochineal's typical crimson colour to the vibrant scarlet red most desired in the 18th-century wool industry. In *Escarlata color de fuego*, although the basis of the recipe is the same when compared to the other five recipes reproduced, a considerably higher amount of cochineal is used in the mordant bath, making the final colour darker.

Luis Fernández's three recipes yield very similar results. In fact, to obtain the *Zereza* colour, after describing the way of the scarlet, he says: *if you want this colour to have a berry-like shade (moratéo, called the colour of cherry) (si se quiere que este color tenga moratéo, llamado color de Zereza)* add a little bit more cochineal and ammoniac salt. This adjustment results in a slightly less red shade compared to *Escarlata*. To get the *Punzó* colour, Fernández suggests using the same quantities of the *Zereza* colour, but "take out the ammoniac salt, and add turmeric in the dye bath". As expected, this results in a slightly more yellow hue. Though subtle, these changes are detectable in the colorimetric data.

Both Janot's and Gout's *Écarlates* are very close in shade, with Antoine Janot's being slightly redder, likely due to his use of slightly more cochineal in both the mordant and dye baths. Since original samples dyed by these two French masters are available, further experiments will aim at establishing and proposing a process which may allow the reproductions to consistently match the historical scarlet samples. At present the closest reproduction of Paul Gout's first scarlet, obtained in the course of a different study, showed a ΔE 2000 colour difference of as little as 0.9 with the sample in the manuscript [12].

The pH ranges from the reconstruction of the selected recipes for scarlet red, fall within a range of 1–3 and 2–3 for the mordant and dye bath, respectively. Considering this and relating to the data from Vitorino, Favaro et al., and Cañamares et al., presented in Table 3 (see also Figure 2), it is possible to suggest that the carminic acid present in the solutions of these reconstructions is either $CA^-$ or $CA^{2-}$. The fully protonated form of carminic acid is not as available to complex with a metal ion as are the $CA^-$ or $CA^{2-}$. While Miguel Suárez's *Escarlata de Venícia* recipe produced a red shade, the absence of the tin mordant prevents it from achieving a true scarlet tone, even though the carminic acid has the right environment to complex with the wool textile.

**Table 3.** Protonation states of carminic acid as a function of pH, relating to the data from Vitorino, Favaro et al., and Cañamares et al.

| Species | Charge | pH Range | Deprotonated Groups | Sources |
|---|---|---|---|---|
| $CAH_5$ (Fully Protonated) | 0 | ~1 | None | Vitorino [37], Favaro [36] |
| $CA^-$ (Monoanionic) | −1 | ~3 | Carboxyl group (C-2) | Vitorino [37], Cañamares [39] |
| $CA^{2-}$ (Dianionic) | −2 | 4–7 | Carboxyl + one phenol (likely C-8) | Cañamares [39], Vitorino [37] |
| $CA^{3-}$ (Trianionic) | −3 | 9–12 | Carboxyl + two phenols | Cañamares [39], Favaro [36] |
| $CA^{4-}/CA^{5-}$ (Highly Deprotonated) | −4 to −5 | >12 | Most phenolic groups | Vitorino [37], Favaro [36] |

Note: The precise order of deprotonation among phenolic groups may vary depending on environmental conditions and the presence of metal ions. Structural positions are estimated based on available spectral data.

While various molecular structures have been suggested [37], the exact configuration of carminic acid remains unresolved. In studies of 18th-century scarlet reds in textiles, tin-based mordants were used instead of aluminium. Understanding the role of tin liquor at a molecular level is crucial, as it is key to transforming the typical crimson colour of cochineal into a vibrant red. Further research into 18th-century tin liquor formulations would provide greater historical accuracy and reveal the full chemical nuance of scarlet dyeing techniques.

## 4. Conclusions

This study offers a comparative analysis of 18th century Spanish and French cochineal dyeing recipes, using tin as a mordant, highlighting key methodological and material differences. Its relevance becomes particularly evident when contextualising the Portuguese Royal Textile Factory in Covilhã, which was strongly influenced by French industrial policies. The dyeing techniques adopted at the factory were likely shaped by both French and Spanish traditions, as suggested by the presence of two master dyers—Spanish Bernardo Rodriguez and French Jean-Baptiste Salessis—and by evidence pointing to processes of technological transfer. While both traditions relied on cochineal, Spanish recipes used significantly larger quantities of dye, in both in the mordant and dye baths. In contrast, French masters achieved comparable chromatic results with smaller amounts of dye, but greater precision in the use of tin and pH control, making these recipes more cost effective.

The author that most diverges among the recipes analysed is Miguel Suárez. Unlike the remaining authors, Suárez was not a master dyer by family lineage, and this is reflected in the final colour results.

It is possible that the recipes used in Covilhã combined elements from both traditions. Future research should prioritise the analytical study of the recipes reproduced, such as

artificial degradation, to further understand the differences between these two traditions, and assess how they compare to existing samples from the Portuguese textile industry, such as the ones from the contemporary Royal Textile Factory of Portalegre, currently under analysis by the REVIVE project (The threads of the past weaving the future: The colours from the Royal Textile Factory of Covilhã, 1764–1850, funded by the Portuguese Foundation for Science and Technology).

Tin liquor emerged as a key factor in shifting cochineal typical crimson colour towards the vivid scarlet tones highly prized in the 18th century. Although this study used tin(II) chloride as a substitute, further work is needed to reconstruct historical formulations of these solutions made by each particular dyer, to understand how this substitute compares to the tin liquor formulations. Despite its historical importance, no systematic study on the formulations of tin liquor has been published, which would help us understand how these formulations influence the colour brightness, hue, and lightfastness.

By bridging archival documentation with experimental reconstruction, this study shed light on the technical sophistication of early modern dyers. Their deliberate manipulation of variables such as concentration, pH, and dye content demonstrates a high level of empirical knowledge and craft expertise.

Ultimately, this work contributes to the field of heritage science by supporting more informed conservation, exhibition, and revival of traditional dyeing practices.

**Supplementary Materials:** The following supporting information can be downloaded at: https://www.mdpi.com/article/10.3390/heritage8090375/s1, Table S1: Colourimetry measurements from the dye bath recipes selected to reconstruct; Table S2: Table of conversions used to reproduce the Spanish recipes [48].

**Author Contributions:** Conceptualization, M.E.S., R.D.H., L.G.F., J.S., D.C. and P.N.; methodology, M.E.S., R.D.H. and P.N.; validation, D.C., V.O. and P.N.; formal analysis, M.E.S., V.O. and P.N.; investigation, M.E.S., R.D.H., L.G.F., J.S. and P.N.; resources, P.N.; data curation, M.E.S.; writing—original draft preparation, M.E.S.; writing—review and editing, M.E.S., R.D.H., L.G.F., J.S., D.C., V.O. and P.N.; visualisation, M.E.S., R.D.H. and P.N.; supervision, D.C., V.O. and P.N.; project administration, P.N.; funding acquisition, P.N. All authors have read and agreed to the published version of the manuscript.

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**Data Availability Statement:** Dataset available on request from the authors: The raw data supporting the conclusions of this article will be made available by the authors on request.

**Conflicts of Interest:** The authors declare no conflicts of interest.

## Notes

1 Secretary of State for Internal Affairs between 1756 and 1777. In 1770, he received the title of Marquis of Pombal. He is particularly well known for coordinating the reconstruction of downtown Lisbon according to Enlightenment ideas after the earthquake of 1 November 1755.

2 Translation by the authors, from Father Manuel Cabral de Pina in 1734 [1].

[3] Arquivo Nacional Torre do Tombo (ANTT), Condes de Linhares, mç. 16, doc. 41. This document was uncovered by Manuel Conceição, a grantee from UBI under the supervision of Rita Salvado, during the archival research carried out as part of the REVIVE project.
[4] All the mordant baths, from the recipes reconstructed, were made separately.
[5] The cochineal used was grounded beforehand, as well as the tartar, to ensure a homogeneous colouring from the start.
[6] Tin chloride was used as a substitute for the tin liquor (a toxic solution); since this is a much-concentrated compound, a reduction of 97% was calculated.
[7] The samples measure 6 × 6 cm, and weigh about 1.20 g.
[8] All the dye baths, from the recipes reconstructed, were made separately.

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