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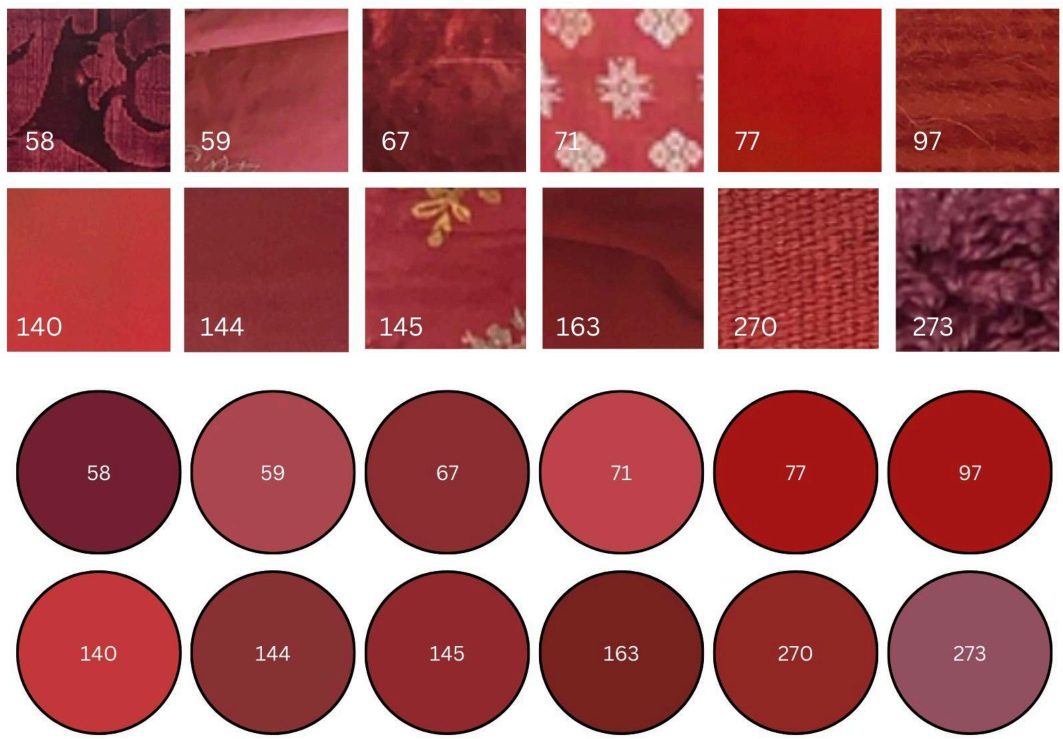
Making and Knowing in Early Modern Europe: Hands-On History

Fall 2024

Red

Experiments in Cochineal Dyeing Techniques with Varying Mordants



Shades of cochineal-dyed red color, selected from *A Red Like No Other* labeled by page number; see Appendix A.

Prized in early modern Europe for its potency and vibrant red hue, the way in which cochineal outperformed ‘old world’ red dyestuffs in the European textile market after its introduction from the colonized Americas is practically legendary. Cochineal is an insect that lives and feeds on the nopal or prickly pear cactus (Opuntia) native to Central and South America, the female of which produces a high concentration of carminic acid that, when crushed, produces a bright red dye. Many scholars of the subject write about it with a sort of reverence, emphasizing its superiority and the rich scarlet red textiles it produces. In *Cochineal Red: The Art History of a Color*, Phipps cites the “deep crimson color, ease of use, and abundant supply" as the causes of its immense popularity, and luxurious red velvets and silks, colorful tapestries, and even the iconic military uniforms of the British redcoats were all colored with the bug dyestuff.[1]

Despite all the examples of cherry, blood, and scarlet reds in extant textiles, however, the reconstructions thus far of the Making and Knowing Project have yielded instead many shades of pink and purple:

Rosenkranz Cochineal Workshop, Brown Dyeing Workshop 2023, Rosenkranz (https://teaching640.makingandknowing.org/resources/case-studies/).

Thus, my objective in this project is to recreate the shades of red that made cochineal a staple of luxury textile manufacturing in Europe up until it was usurped by synthetic dyes in the 19th century. There are different ways to adjust the color results of a natural dye, but the focus of this project is on mordanting as the most significant and common. Mordants are materials, such as metal salts or tannins, that chemically bond to the textile in order to increase the effectiveness, fastness, and color result of the dye, which in turn bonds to the mordant. Of the mordants used historically in the textile industry, aluminum salt or alum is the most common and has been used since antiquity. For this series of experiments, I'm using oak galls (a source of tannins) and four metal salts: aluminum, copper, iron, and tin. Additionally, I am testing a method that uses cream of tartar, or potassium bitartrate, in conjunction with potash alum (an aluminum salt) that is recommended to help achieve that red color and as a post-dye treatment.

One important thing to note about cochineal specifically is that pH has a significant impact on the coloration. A more acidic dyebath makes for a more orange-toned outcome, while a more basic one tends to lean purple. Additives to the dyebath itself and post-dye treatments could therefore be used to further alter the end result of dyeing with cochineal. Additionally, a mix of dyestuffs is indicated in dye analysis of many pieces and seems to have been a common method to achieve the desired color or budget. For example, Ms. Fr. 640 recommends purchasing high-quality cloth and dyeing it

[1] Elena Phipps, "Cochineal Red: The Art History of a Color," 10.

cheaply with “pure scarlet pastel woad & a little cochineal” as an alternative to buying lower quality pre-dyed cloth at the same price[2]. For the purposes of this project, I’ve narrowed down the variables to just the one (the mordant), but like any craft, each practitioner had their own method of producing the desired result. Here, I’ll be using the measurements outlined in *Natural Colorants for Dyeing and Lake Pigments* (Kirby et al) for mass, temperature, and time.

Mordants used, left to right: alum, cream of tartar, copper sulfate, iron sulfate, oak galls

Mordant	Chem Name	Formula	Mass per 10g textile[3]	Notes
Aluminum salt	Potash alum or potassium alum	$KAl(SO4)_2$	2g	
Cream of tartar	Potassium Bitartrate	$KC_4H_5O_6$	0.7g	Optional, added to alum mordant solution
Oak galls	gallotannic acid	Organic material	1g	Crushed
Iron salt	iron(ii) sulfate	$FeSO_4\ 7H_2O$	1g	
Copper salt	Copper sulfate	$CuSO_4\ 5H_2O$	2g	
Tin salt	tin(ii) chloride	$SnCl_2$	0.25g	Very toxic

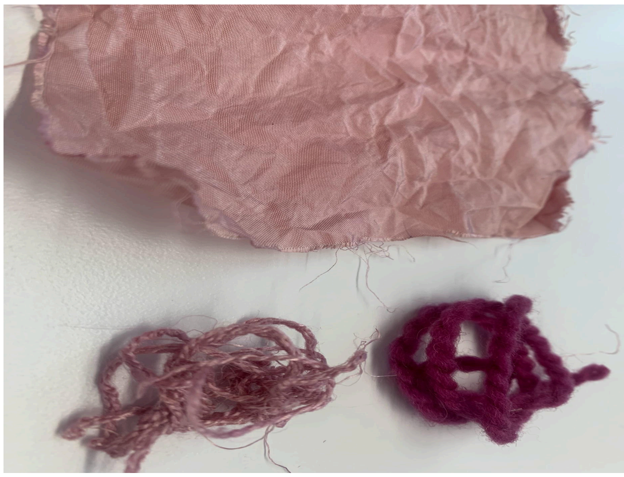
To maintain continuity and get the best possible comparison of the different mordants, the process would ideally be as close to identical for each bath. The textile mass used per bath came out to about 5g: one large and two small squares of silk taffeta, some fine silk yarn, a length of wool yarn, and a piece of cotton cloth. The mordant bath would be made with 250mL of water and the prescribed mass of mordant, heated to 40C, the textiles added and then soaked for one hour at about 90C before removing from the heat

[2] Making and Knowing Project et al., *Secrets of Craft and Nature*, 38v.
[3]Kirby et al., *Natural Colorants for Dyeing and Lake Pigments*, 50.

to cool and soak overnight. The next day, the textiles would be rinsed and then added to 500mL of pre-prepared cochineal dyebath[4] at 90C to simmer for one hour before being removed, rinsed, and left to air-dry.

Control: Unmordanted Textile

The unmordanted textile samples, subjected to the same dyeing process but no mordanting the day prior, produced the least saturation across textile groups and a very pink color.



Silk (above) and yarns silk (left) and wool (right) dyed without mordant

Potash Alum

Aluminum is the most prominent metal salt used as a mordant for textile dyeing, and it's been in use since antiquity for its easy natural availability and the brightening effect it has on colors. Potash alum, the most common aluminum salt used for the purpose and often referred to simply as 'alum', is also used as a substrate in the production of lake pigments and medicinally.

In the lab, I used 1g potash alum and 250mL water to create a colorless, mildly acidic solution that had no visual effect on the textiles after they were rinsed. The cochineal took very well to the silk and wool and moderately well to the cotton, resulting in a dark purple-ish-pink color.

[4] This large batch of cochineal dyebath was prepared in the lab on September 20, 2024 in preparation for the 2024 dyeing residency of Bertha Estrada Huipe and Mateo Rodriguez Estrada.

Clockwise from top left: cotton, wool yarn, silk yarn, silk dyed with potash alum mordant

Additionally, I also made a (slightly more acidic) mordant solution using potash alum in combination with an added 0.35g cream of tartar, a technique recommended in both modern and historical recipes to achieve a more reddish color result and to soften the texture of wool. Easily found in the baking aisle of any grocery store, cream of tartar, or potassium bitartrate, is a precipitate byproduct of wine-making that can be scraped from the barrels after fermentation and has been noted in English toll records toward the end of the 13th century.[5] One 1706 recipe provided in Natural Colorants (Kirby et al.) calls for "ashes of pressed wine-grapes"[6], and the modernized recipe for mordanting later in the chapter recommends using cream of tartar in conjunction with potash alum specifically in cases where demineralized water is not being used.

The resulting color was a bit less purple in tone, but it also made for a much less concentrated color on the silk and cotton samples; the wool seemed unaffected by this loss of saturation. I'm not entirely certain why this is, though it's worth noting that many sources recommend the cream of tartar specifically when mentioning wool, and the sale description on the Botanical Colors website[7] additionally warns that in some cases it can "inhibit the development of certain shades."

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[5]Carus-Wilson, "The English Cloth Industry in the Late Twelfth and Early Thirteenth Centuries," 39.
[6] Kirby et al., *Natural Colorants for Dyeing and Lake Pigments*, 40.
[7] Botanical Colors, "Cream of Tartar."

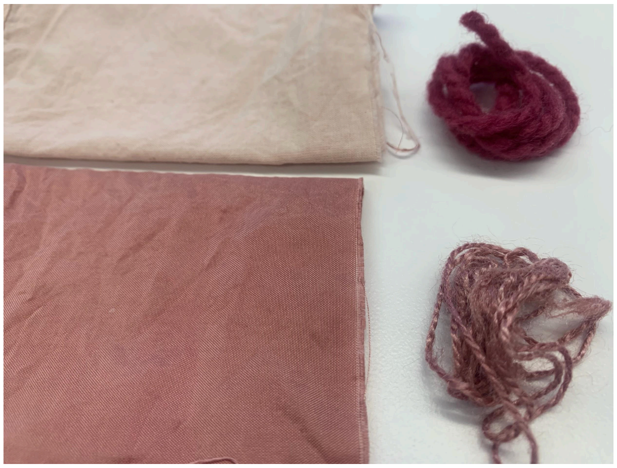
Clockwise from top left: cotton, wool yarn, silk yarn, silk dyed with potash alum and cream of tartar mordant

Oak Galls

Oak galls, also called Aleppo galls, gall nuts, or oak apples, are formed on oak trees as a result of a parasitic relationship with gall wasps, which lay their eggs in the tree's leaf buds. Because of their high tannin content, they're used in dyeing as a mordant and for the making of iron gall ink. I used 0.5g of crushed oak galls with 250mL of water for mordanting, which was very mildly acidic and dyed the textiles a light brown color.

Textiles after mordanting in Oak Gall solution

After dyeing with cochineal, the oak-gall-mordanted silk came out a pale but pleasant salmon pink, with the wool taking a darker color and the cotton coming out only slightly tinted.



Clockwise from top left: cotton, wool yarn, silk yarn, silk dyed with oak gall mordant

Iron Sulfate

Iron sulfate, or ferrous sulfate, was historically known as green vitriol or copperas, and it has been used in dyeing as a mordant and 'saddening' agent (meaning it makes the colors darker) for thousands of years, with the earliest identifiable textiles dating back to 1500-1300BC in Egypt.[8] It was also used in the making of inks, notably iron gall ink. In the lab, 0.5g of the greenish crystals with 250mL of water produced a somewhat acidic brown-tinted solution that left the mordanted textiles a warm brown color.

Textiles mordanted with iron sulfate solution

After dying, the textiles were a cool purple color, with the wool taking it especially well to a very dark shade and the cotton coming out rather pale and grayish. The silk was a rather lovely shade of purple.

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[8] Kirby et al., *Natural Colorants for Dyeing and Lake Pigments*, 22.

Clockwise from top left: cotton, wool yarn, silk yarn, silk dyed with iron sulfate mordant

Copper Sulfate

Copper sulfate, also called blue vitriol, is often found historically in association with iron salts as a result of the impurity of the salts used.[9] I used 1g of the blue copper sulfate crystals with 250mL of water to make a solution with a bright blue-green tint and a mild acidity. The mordanted textiles came out a pale turquoise color.

Textiles mordanted with copper sulfate solution

After dyeing with cochineal, the cotton and silk were a lighter and darker shade of dull pink, respectively, and the wool was a dark slightly purple-ish shade of the same.

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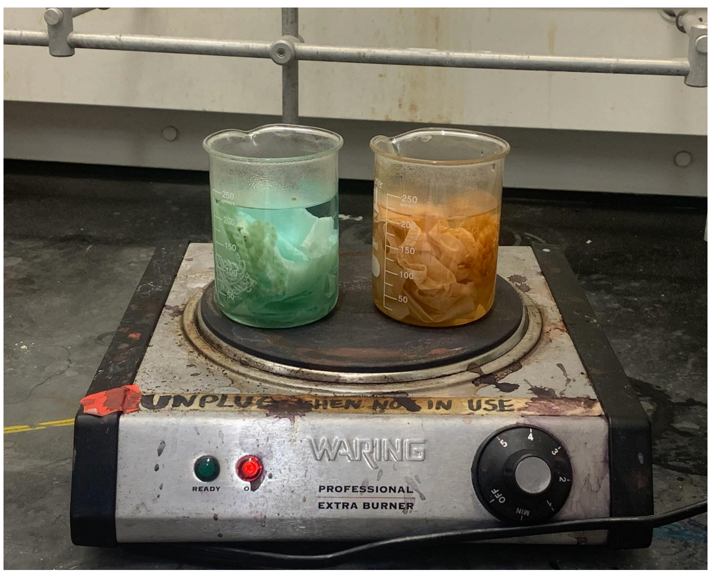
[9] Kirby et al., *Natural Colorants for Dyeing and Lake Pigments*, 22.

Clockwise from top left: cotton, wool yarn, silk yarn, silk dyed with copper sulfate mordant

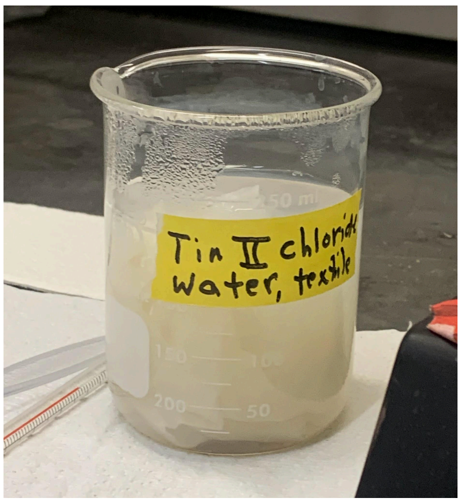


Copper sulfate (left) and iron sulfate (right) solutions with textiles

Tin Chloride

Tin chloride is a metal salt that brightens reds and yellows and is particularly noted for its ability to give cochineal a bright scarlet color when used as a mordant. Slightly later to the game, tin was used as a mordant starting in the 17th century when its properties as a mordant were discovered in a lab, and it often comes with a warning that it can make wool feel hard or brittle. It is also highly toxic and can cause skin irritation. Lab safety information is available in the field notes (Appendix B).

In the lab, the tin chloride, which proved a bit of a nuisance to measure, produced a cloudy white acidic solution. Even when rinsing the mordanted cloth, the wool yarn broke into several pieces in my hands with even slight tension, and the solution left a film on the inside of the beaker. It’s possible that the higher concentration of tin added to this effect, as the tin chloride stuck to itself when measuring and made it nearly impossible to accurately measure out the tiny quantity needed. When the rinsed textiles were added to the heated dyebath, it produced a reaction and boiled over, possibly a result of contact between the acidic tin chloride on the cloth and the more basic cochineal dye bath.



Tin chloride solution (left) and film left in beaker (right)

The resulting textiles were slightly more orange-toned, but still distinctly pink. The cotton was pale, and the silk was a warm orange-pink. The wool yarn took on a darker reddish color, and it was much brittler and less fluffy to the touch than the other wool yarn samples; in handling, it shed quite a bit, which can be seen in the image.

Clockwise from top left: cotton, wool yarn, silk yarn, silk dyed with tin chloride mordant

mordant	mass	water	solution pH
alum potash	1g	250mL	5
&COT	0.35g	250mL	4
oak gall	0.5g	250mL	6
iron	0.5g	250mL	4
copper	1g	250mL	5
tin	0.3g	250mL	2.5

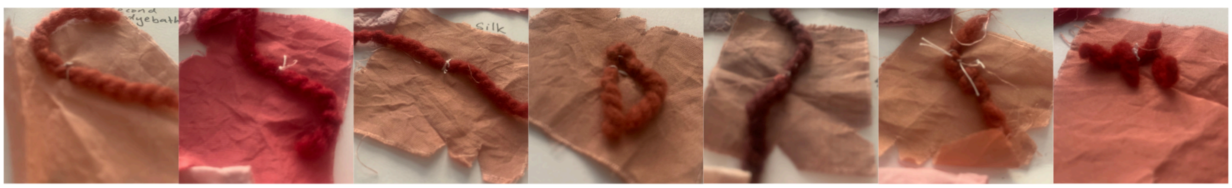
Second Dyebath and Post-Dye Treatment

A small sample of silk cloth and wool yarn from each mordant sample was subjected to a second dyebath, made with dried cochineal and cream of tartar, and a separate sample of the same to a post-dye treatment of cream of tartar dissolved in water.

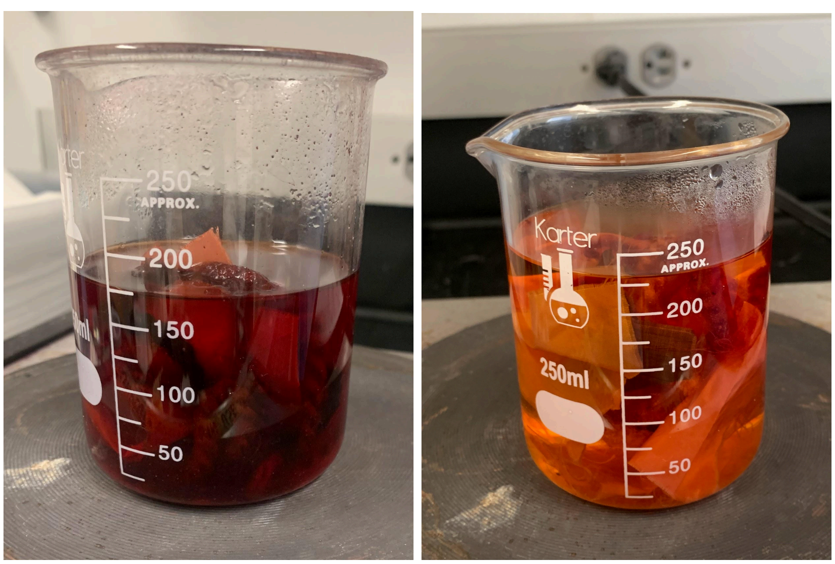
The secondary dye bath, which contained cream of tartar, was more orange and acidic than the pre-prepared bath used during the first round. I used the method described by Kirby et al for the preparation, but I substituted alum for the cream of tartar to test the results of a stronger acidity. The silk samples showed a notable increase in saturation of color for nearly all mordants as did several of the wools, and the effect was generally warming across the board.

Samples after second dyebath, left to right: no mordant, potash alum, potash alum + COT, oak gall, iron sulfate, copper sulfate, tin chloride

The cream of tartar post-dye bath, made with 0.6g of cream of tartar dissolved in water and heated similarly to the second dyebath above, was acidic and initially colorless but turned a light pinkish orange over time as the textiles soaked. The textile samples, both wool and silk, became paler for all mordant groups, perhaps speaking to the wash-fastness of cochineal in an acidic environment. The bath also had a warming effect on the color of the samples to varying degrees.



Samples after post-dye treatment, left to right: no mordant, potash alum, potash alum + COT, oak gall, iron sulfate, copper sulfate, tin chloride



Dyebath with textiles (left) and cream of tartar bath with textiles (right)

An additional sample of silk and wool yarn was dyed using only the second dyebath with no mordant as a control, and the color was warmer than that of the more alkaline pre-prepared bath without a mordant.

Silk and wool samples exposedonly to the second dyebath (left) and also to the post-dye treatment (right)

Treatment	Textile Mass	Coch. Mass	COT Mass	Water	PH
Secondary Dyebath	2.8g	0.6g	0.4g	250mL	5
Post-Dye Treatment	2.3g	-	0.6g	250mL	3.5

Conclusions and Ongoing Questions

The dyeing experiments yielded a wide spectrum of colors, from the cool purple of the iron to the red of the tin, and were ultimately successful in achieving the red color that was sought in the end. Even then, the dyeing yielded less red tones than I had hoped for, and many of the questions I had at the beginning remain: Why is red, rather than purple or pink, the color cochineal is so inextricably tied to when it seems to be the most difficult to produce? What practical knowledge or skills might we be missing to achieve that color consistently? Additionally, several factors presented themselves as possible further routes of questioning.

Water: During the dyeing workshop done as a group in September, we discussed the possibility that the water itself plays a role in the results of dye. New York City water in particular is lauded for its freshness and often credited with the distinctive quality of bagels made in the city, and it makes sense that the variations in the makeup of tap water could create variations in the results of experimentation. Additionally, on page 50 of *Natural Colorants,* the recipe suggests adding cream of tartar specifically if one is unable to use demineralized water for the dyeing. These experiments were carried out using the tap water in the lab, though all from the same sink for consistency's sake.

Dyebath: Aside from the second dyebath to which a few samples were subjected, all of the dyeing done in this experiment used the bath prepared a few months previous in the lab for the group dyeing workshop. When it was first prepared in September, the bath was a deep blood-red before being heated with the textile, which made it more purple;

when I was using it in November it was a dark purple, smelled quite awful, and was growing mold on the inside of the bucket. Additionally, it seemed to become more basic as time went on; during the first dyeing session, the pH was reading at about a 7 and by the end, a few weeks later, it was leaning toward a 9. Thus, I'm curious what effect this had on the results of my experiments. The initial pH at the time of its making was not recorded.

Acidity: While I avoided making any changes to the dyebath to avoid having too many variables, cochineal as a pH-sensitive dyestuff has a lot of potential for further experimentation. As discussed above, a higher acidity is purportedly linked to more crimson and even orangey shades of red, and this is somewhat visible in the results of the experiments here, especially with the post-dye treatment. It could be interesting to see if further increasing the acidity of the mordants or dyebath affected the results.

Limitations of dye analysis: The ability to detect specific dyestuffs used in museum textiles is a developing technology but still very limited, and it's even more difficult to identify any of the mordants used. As such, it's hard to know just how many of the extant textiles in museum collections are dyed with cochineal—red and otherwise—and, of those that have been tested, how many may have been dyed with multiple dyestuffs in combination that didn't show up in the chemical analysis.

Non-European practices: The research and experiments conducted for this project were limited to historical European practices. Prior to their export to Europe, though, cochineal bugs had been used for dyeing by indigenous peoples in the Americas for centuries, with their own techniques and practices.

Results of dyeing experiments, clockwise from top right: oak gall, alum with COT, alum, copper, iron, tin

Note on photography: As with all digital images, colors may appear different depending on the screen on which they are viewed. Additionally, the images themselves appear different from the precise colors as they appear in life, and this consideration should be taken regarding both the photos taken of samples dyed during this project and the photos used as reference for the color of red that this project aimed to recreate.

Further Reading

General: Brown, Reece. "Dyeing Textiles with Cochineal." The Making and Knowing Project: Research and Teaching Companion, 2023. https://teaching640.makingandknowing.org/resources/student-projects/sp23_brown-reece_final-project_dye-workshop/.

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Appendix A - Color Reference Images

These images were used as a reference for the red color that is attributed to cochineal in many sources, including in *A Red Like No Other*, from which these images are sourced. Items are labeled by the page number on which they appear.

58 - Fragment
Italy, sixteenth century
Cut silk velvet with silver thread in green selvage
Metropolitan Museum of Art

59-Cloak
France, 1580-1600
Red satin, couched and embroidered with silver, silver gilt, and colored silk threads; trimmed with silver gilt and silk thread fringe and tassel; lined with pink linen
Victoria and Albert Museum, London, 793-1901

67-Liturgical Vestments
Eighteenth to nineteenth century, collected in Guatemala
Velvet, metallic embroidery over silk satin
Museum of International Folk Art

71-Songket textile
Bali, Indonesia, nineteenth century
Metallic foil-wrapped thread; supplementary weft patterning on plain-weave silk ground
Museum of International Folk Art, IFAF Collection, FA.

77-Red wool jinbaori surcoat with Nabeshima ginger crest
Japan, nineteenth century
Dutch rasha wool, stenciled leather edging, silk cord tie, and embroidery on the Nabeshima crest
Arthur M Sackler Gallery, Smithsonian Institution, Washington DC

97-Ana Roquero, reproduction following Jean Hellot's 1752 recipe for scarlet. Wool.

140-Armchair (fauteuil) from the council room (salle du conseil)
Malmaison, France, 1800
Painted and gilded beech; original under-upholstery and red wool show cover (silk, velvet, and gilded silver trim)
New York Historical Society

144-Medias (stockings)
Spain, eighteenth century
Silk, gold thread
Museo del Traje, Madrid

145-Jubón (corseted jacket)
Spain, ca. 1740-1760
Silk, flax, hemp, metal, taffeta, French falla
Museo del Traje, Madrid

163-Coatee of General Sir William Napier
Great Britain, early nineteenth century
Wool with cotton lining, metal buttons and fasteners
National Army Museum, London

270-Diamond-twill skirt fabric
San Antonio Castillo Veliz, Oaxaca, Mexico, ca. Mid-1950s
Wool, cochineal

273-Full Integration Design, *Art, of Course, tocado* (fascinator)
Jute, cochineal, garnet, metal
Enesimapotencia, Consultoria y Producción Cultural, Madrid.
(modern)

Padilla, Carmella, Barbara Anderson, and Blair Clark. *A Red Like No Other: How Cochineal Colored the World: An Epic Story of Art, Culture, Science, and Trade*. New York, NY: Skira Rizzoli, 2015.

Appendix B - Field Notes

Textile Specifics

material	dimension	mass	per mordant
silk taffeta	2x2in	~0.2g	2
silk tussah yarn	4ft	~0.13g	1
wool yarn	2ft	~0.45g	1
silk taffeta	~5x4in	~1.15g	1
cotton hankie	1/2	2.9g	1
total		5g	6pc

Mordanting and Dyeing Data

The textiles were all mordanted on one day and then dyed the second, as reflected by the times in the tables. The date listed is the day it was dyed. Temperature was taken periodically and textiles were agitated every five minutes or so. The textiles were left in the mordant solution overnight and rinsed the next day before dyeing.

mordant	mass	real mass	water	textile mass	temp 1 (add)	PH	mordant time	temp 2 (remove from heat)
alum potash	1g	1.03g	250ml	5.15g	65C	5	30min	88C
&COT	0.35g	0.36g	250ml	5.03g	70C	4	30min	89C
oak gall	0.5g	0.50g	250ml	4.81g	70C	6	30min	89C
iron	0.5g	0.5g	250mL	4.8g	58C	4	39min	88C
copper	1g	1.01g	>250mL	5.0g	55C	5	39min	91C
tin	0.13g	0.3g	250mL	5.3g	50C	2.5	38min	85C
unmordanted	-	-	-	-	-	-	-	-

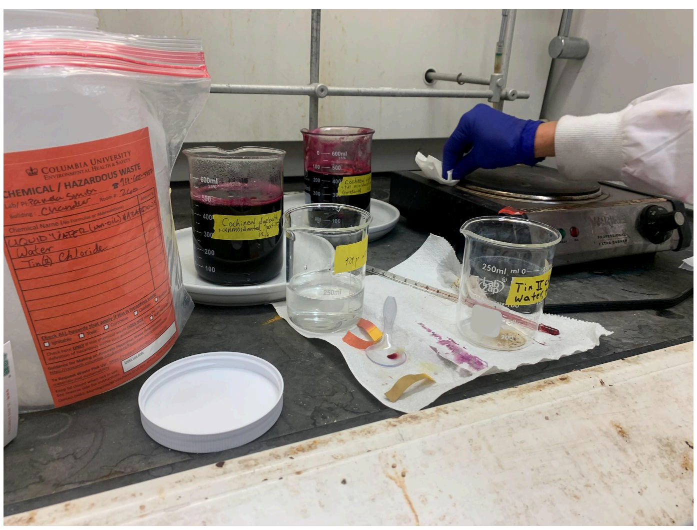
(cont'd)

mordant	Date	dyebath	time 1 (add textiles)	temp 1	time 2 (remove heat)	temp 2	Rinse
alum potash	11/20	500ml	11:11	90C	12:04 (53)	89C	12:15

&COT	11/20	500ml	11:14	90C	12:05 (51)	87C	12:15
oak gall	11/20	500ml	11:15	90C	12:06 (51)	85C	12:16
iron	11/27	500ml	10:40	87C	11:30(50)*	84C	11:40
copper	11/27	500ml	10:41	86C	11:30 (49) *	84C	11:40
tin	12/6	500ml	12:26	85C	1:15(49)**	81C	1:30
unmordanted	12/6	500ml	12:25	85C	1:15(50)**	89C	1:30

*When dyeing the iron and copper-mordanted textiles, there was an issue with the hotplate that caused the beaker to jump and spill a bit, resulting in the heat being turned off early. Because of residual heat, this did not end up having a significant change to the temperature.

**When dyeing the plain and tin-mordanted textiles, there was a reaction that caused the latter dyebath to boil over very quickly at 12:35. Both were removed from the heat entirely and allowed to cool for five minutes. The tin samples were transferred into a larger beaker before being returned to the heat.



Tin chloride dyeing workspace after temporarily removing dyebaths from the heat

Secondary Dyebath and Post-Dye Treatment

The textile samples were marked to make sure they didn't get mixed up, since they were all dyed together.

second dyebath silk	mordant	wool yarn
one notch	alum	one knot at end
two notches	COT	two knots at end
three notches	gall	three knots at end
one notch two sides	iron	one knot each end
two notches two sides	copp	two knots each end
two/one two sides	tin	shorter piece
plain	no	one knot in middle
long	fresh	long

Date	textile mass	coch mass	COT mass	water	start	95C	pH	temp added	time added	temp remove	time remove	rinse time
12/10	2.8g	0.6g	0.4g	250 mL	10:38	11:05	5	90C	11:18	85C	11:58	12:05
12/12	2.3g	0g	0.6g	250 mL	11:27	-	3.5	73C	11:38	49C		12:30

Lab Safety

Gloves, goggles, and the fume hood were used when working with the copper, iron, and tin salts, and the solutions and textile rinse water were labeled and disposed of in the hazardous waste stream. Additionally, the dyebath for the tin-mordanted textiles was disposed of as hazardous, as well as the gloves, paper towels, pH strips, and other supplies used. Gloves were changed regularly and the workspace was thoroughly wiped down after use. Safety Data Sheets are available for each substance here:

Potash Alum - Potash Alum (Aluminum Potassium Sulfate).pdf

Copper Sulfate - Copper Sulfate Pentahydrate MSDS.pdf

Iron Sulfate - Iron (ii) Sulfate Heptahydrate MSDS.pdf

Tin (ii) Chloride - tin II chloride 0_5M_undefined_USA_EN.pdf

Cochineal Dyebath Observation

date	cochineal ph
11/20	~7
11/27	~8?
12/6	8.5/9?
12/10	8.5?

Note: It was difficult to determine the pH very precisely using the color-coded strips, since the solution is itself very dark in color, as well as the fact that I was not focusing on the dyebath itself at the time.

Several other dyebaths were made at the same time as the cochineal in class, and all were revealed to be molding at least a bit during the time of my experiments.

Cochineal used in both the pre-prepared dyebath and in the secondary dyebath. There was some conversation as to whether the source of cochineal resulted in different results, so care was taken to use the same bag for secondary baths.