Experiment: Palette of Purples on Silk
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For the velvet weaving project, we created a palette of exquisite purples with natural dyes available in Early Modern Europe. For the material, we applied the highest quality French mulberry silk yarn.

Next, I will introduce the dyes and processes we applied, so that it is possible to reproduce the experiment. I will also analyse why the results were not always as expected. We selected natural dyes based on their availability in Early Modern Europe, and due to them not requiring lengthy processes: Cochineal (*Dactylopius coccus*), woad (*Isatis tinctoria* L.), brazilwood (*Caesalpina* L.), madder (*Rubia tinctoria* L.), and logwood (*Haematoxylum campechianum* L.).

The image Circle of Purples (1_ circle of purples) shows the resulting range.

Circle of Purples, clockwise from the top:

1. Cochineal
2. Woad & cochineal
3. Woad & brazil wood
4. Woad & madder
5. Logwood & madder
6. Logwood
7. Cochineal & logwood

This numbering of the skeins is withheld throughout the text and all photos. The skeins are not always in a numerical order in following photos, but the above fixed numbering makes it easier to follow the dyeing process.

Next, I first introduce the materials applied, followed by the necessary process of mordanting, and finally the natural dyes, recipes, and dye processes applied.

Material

We had the privilege of working with finest French mulberry silk organzine, 60/66 (3 ply fibroin of 20/22). Upon request, the manufacturer kindly reeled our order of 140 grams of silk yarn into seven skeins, twenty grams each, and degummed them for us beforehand to be ready for dyeing. With such a delicate

and fragile silk, this was a prerequisite for our laboratory dye experiment to succeed in our brief time frame.

Mordanting with Alum and Cream-of-Tartar

The word mordanting refers to preparing the fibres to better absorb the natural dyes, and bind them with chemical bonds, to improve colourfastness against light and washing. Certain natural dyes are very colourfast, while others are more likely to fade, so it is practical to follow pre-tested recipes.

The Early Modern dyers would have used various metal salts for achieving bright shades from natural dye sources. However, these hazardous processes would have caused health damage to the dyers and even the wearers of the dyed garments, not to mention polluting the effluent waters discharged into rivers. For such reasons we excluded tin, chrome, and copper from our experiment. Moreover, while an iron mordant could have provided us with an even wider range of purples, it might have harmed our delicate silks, so we excluded iron from our process, too. Consequently, we only worked with alum mordant, supported with cream-of-tartar.

Subsequently, I prepared the mordant solution by first dissolving the weighed *alum* and *cream-of-tartar* in half-a-litre of boiling water, and then pouring this in a kettle with three litres of cool water, to start the mordanting process in a lukewarm solution. The recipe for the mordant bath is provided below.

Skein	g	Mordant bath for silk	Mordant temp / time for silk	pH
1-7	140	Alum 10 % (= 14 g) Cream of Tartar 5 % (= 7 g)	50°C / 60 min, let cool and soak overnight	< 7

I added the silk skeins to the mordant bath, and warmed up the solution to 50°C, maintaining this temperature for one hour. I left the silks soak in the mordant solution until the next day, when we continued with the dyeing.

(image 2_mordanting)

As such, indigo dyeing (introduced below) does not require mordanting, since this type of dye does not bind with the fibres chemically but attaches to its surface with physical bonds. However, to create purple, we would dye the indigo

blue skeins further with shades of red, for which it was necessary to mordant all the skeins.

Dyes and dyeing

The selected dye sources were used in this experiment as follows: Two of the skeins were dyed with only one dye; cochineal (1) or logwood (6), while the other skeins were dyed in two consecutive dye baths, first either indigo blue with woad and second with shades of red from cochineal (2), brazil wood (3) or madder (4); or, first with logwood and second with madder (5) or cochineal (7) as detailed in Table 1 below:

Table 1: The dyes used for each of the seven skeins.

Skein	**Bath 1**	**Bath 2**	Base recipes (for wool) Kirby et al (2014) page(s)
1	Cochineal	-	
2	Woad	Cochineal	65,
3	Woad	Brazil wood	65, 62
4	Woad	Madder	65, 55-58
5	Logwood	Madder	64, 55-58
6	Logwood	-	64
7	Cochineal	Logwood	64

As our starting point for the dye process, I took the practicable, simplified dye recipes provided by Kirby et al (2014) with some modifications as detailed below. In general, as wool and silk are both protein fibres, the same processes can be applied without risk of damage to the fibres, but since we were only working with silk, I made some further modifications. Also, some recent research suggests enhanced processes for colourfastness and sustainability, which I considered.

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(image 3_Dried woad balls and powder)

Dyeing Indigo Blue with Woad Powder

In Early Modern Europe, the professional dyers would have used fresh or dried woad (on the left), but nowadays woad is mostly provided in a ground powder form (on the right), which we utilised.

In three of our recipes, woad (*Isatis tinctoria*) was the base for purple as shown in Table 2 above. The process of indigo dyeing, referred to as vat-dyeing, was the most complicated and time consuming in this experiment, so we began with it. Also, it is recommended to start with the indigo dyeing process, since the mordant dyes are often sensitive to alkalinity, which cannot be avoided in the indigo dyeing process. Moreover, as explained in the mordanting section above, for achieving colourfast purple, the fibres needed to be mordanted. It is best to do before dyeing with indigo, because this way the mordant will fix more evenly with the fibre and allow for more even dyeing with the second dye following indigo.

From the chemical perspective, the indigotin compound of the dye is not water soluble. Its soluble precursor is indigan, which is the blue colorant in both woad and in imported natural indigo (*Indigofera* ssp), while woad also includes two other precursors (isatan A and B), which can be enzymatically hydrolysed into indigotin. Indigotin, then, must be first reduced into a water-soluble form to be able to attach to the fibres, and then oxidized back into the non-soluble, visible blue colour. Other reasons for possible, slight differences in the blues between the two types of plants are due to other minor dye compounds, such as the purplish indirubin, which varies in quantity.

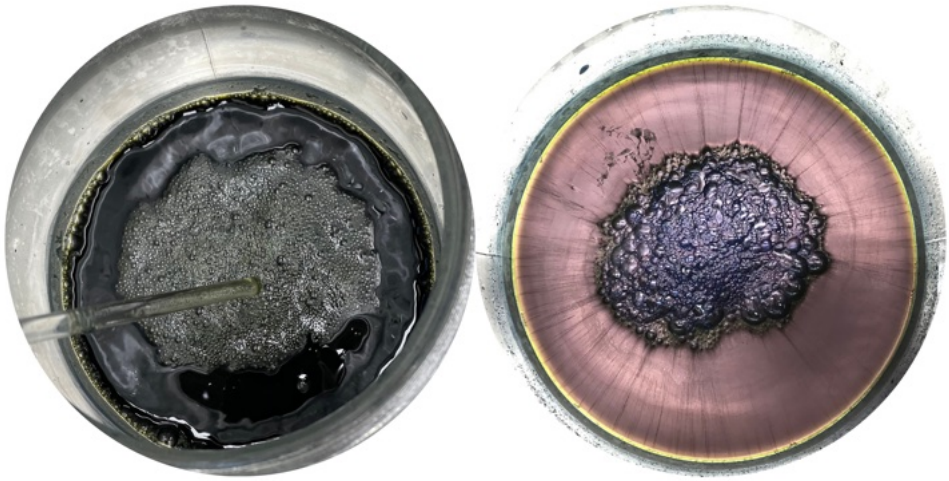
We used the following recipe for extracting dye from powdered woad and dyeing with it:

Skein	g	Bath 1	Extraction temp / time	Assist	pH	Reducing agent	pH	Reducing temp / time
2	60	Woad (powder)	40-50°C	Sodium hydroxide 10 % (= 6 g)	10-11	Sodium dithionate 60 % (= 36 g)	8-9	55°C/10 min Let settle for 30 mins
3								
4								

As the above explanation suggests, preparing the indigo vat and the magic of dyeing with it was a skill mastered by specialist dyers only. The skill was passed on under the apprenticeship of an experienced Master, a qualified member of the dyers' guild. Nowadays, the chemicals and recipes are easily available, and it is possible even for laypeople to experiment with the dye in a small scale. Dyeing delicate silk yarn evenly, however, is challenging to say the least, as the dye fixes itself onto the surface of the material instead of permeating the fibre. For safety reasons, as the process discharges detrimental fumes it is important to do it in an effectively ventilated space, to wear a protective mask, and if working inside, to leave the room while the chemicals react and the vat settles.

The woad powder is gently whisked into the dyebath, after which the pH is set for alkaline by adding pre-diluted sodium hydroxide NaOH (or sodium carbonate

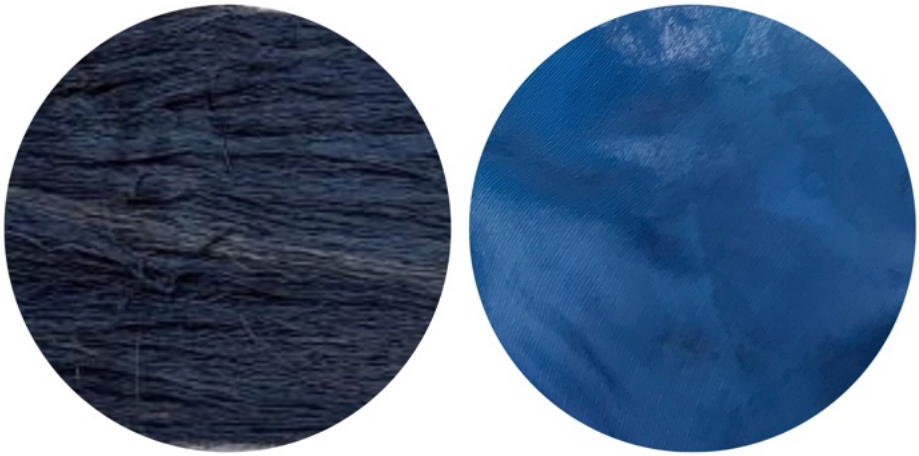
Na_2CO_3) to start the process. To reduce the indigotin dye precursors into a leuco form that can attach to the fibres the use of a reducing agent is required. We applied an economical and effective, but tainting sodium dithionite $Na_2S_2O_6$ in our lab experiment. Maintaining an even temperature (50–55°C) is another key to successful reduction and dyeing process. The dye vat is ready when it forms a metallic surface, and a foam poetically called the flower (shown on the photo below on the right). Then it is let to settle for 20-30 minutes for the chemical process to complete and any residue to fall on the bottom of the vat.



Preparing the indigo vat (left) and the reduced vat ready for dyeing (right).

The skeins are dipped into the vat for 1-10 minutes, depending on how deep blue is desired, avoiding oxygen to get into the dye bath, because oxygen reverses the reduction process. If necessary, the vat could be reactivated by adding some more alkaline, and some more reducing agent. Upon lifting the skeins from the vat, they are yellow-green, and start their transformation into blue in the air due to an oxidation process – the true magic of indigo dyeing.

Out of the three skeins, the first one became evenly dyed (2), but with two others (3, 4) we were not quite as successful. This is interesting because we followed the same process for all three of them. Apparently, we did not handle the three skeins in an identical way during the dyeing process, even though we tried to do so. We utilized a cotton cloth to protect the silk skeins from contact with the dye residues when dipping the skeins into the dyebath, and for this it worked well, but it may have constrained the space for the skeins in the dye bath to be moved around and the dye to reach the fibres uniformly thus preventing an even result. Utilising a larger vessel or adding more liquid to the dye bath might have solved this issue. In general, indigo attaches best onto cellulosic fibres, and in higher alkaline levels (pH 10-11), which work well for cellulosic fibres but are detrimental to silk and other protein fibres.



Mulberry silk yarn (left) and industrial silk fabric (right, still damp) dyed in the same indigo bath. You can see the dye result is not even.

Also, the various types of materials each may require specific dye processes, which is suggested by the above sample of an industrial, bleached silk fabric (on the right) that we dyed simultaneously with the exquisite yarns.

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Experimenting with Logwood for Purple

Second, we wanted to experiment with the purple colour available from logwood (*Haematoxylum campechianum* L.), which was a novel dye material in the Early Modern Europe. Logwood purple is created by haematoxylin which oxidizes into haematein. Its purple shade was known for its inclination to fade already at that time, but it contributed to beautiful blacks. A more recent Japanese study suggests instructions for more stable purple results on silk (Sakata 2008), which I experimented with, according to the recipe A) below:

Skein	g	Bath 1	Extraction temp / time	Assist	pH	Dye temp / time
5 6	40	Logwood 333 % (= 133 g)	70°C / 60 min	A*) - B) potash (K_2CO_3) 5 g	A*) 7 B) 9	40°C / 120 min 80°C / 60 min

Note: receipe A) proved unsuccessful for purple in this experiment!

Skein 6: Logwood dye sample after the second dye, recipes A + B.

Consequently, I started with the neutral pH and a temperature of 40 degrees for 120 minutes, respectively (recipe A). To my surprise, the result was not even near a purple colour, but a rich, milk-chocolaty brown. Unfortunately, I did not

photograph this phase as I was too anxious to try and find a fix for the colour. Next, I changed the pH of the dye bath first to pH 8, which did not make any change to the colour, and then to 9 (recipe B), when the colour finally changed towards purple. I re-dyed the silks in this dye bath at 80°C for one hour, achieving a greyish purple colour. Probably, had I not made the first dye round, the colour could have been closer to the targeted shade.

Whereas indigo dyeing is displeasing to one's nostrils, to say the least, logwood as well as brazilwood introduced later, offer lovely aromas for the dyer to enjoy. During our experiment, the scents from logwood induced hints of sweet caramel, herbs, and hay.

Skein 7 turned out beautiful, first dyed with cochineal and then overdyed with logwood. This combination is good also, because as the logwood dye fades, the colourfast cochineal will keep, so the colour will be beautiful even if it may change somewhat over time.

Skein	**g**	**Bath 2**	**Extraction temp / time**	**Assist**	**pH**	**Dye temp / time**
7	20	Logwood 333 % (= 66 g)	70°C / 60 min	A) - B) potash (K_2CO_3) 5 g	A) 7 B) 9	40°C / 120 min 80°C / 60 min

Skein 7: Cochineal-dyed (see next section for details) and a white skein (6) going to logwood bath (on the left), and cochineal overdyed twice with logwood (recipes A + B) (on the right).

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Dyeing with Cochineal

Cochineal *Dactylopius coccus,* a new and ten times richer than the Old-World sources for bright carmine red in Early Modern Europe, is one of the easiest classic dyes to use. Its beautiful carmine colour is relatively consistent even between different dye batches. Also, the colour is not sensitive to minor acid-alkaline differences, or changes in temperature. However, the colour fixes most effectively in slightly acidic conditions and at a temperature of 90 degrees. The applied mordanting is particularly recommendable for cochineal dyeing, as both alum and cream-of-tartar are acidic. Also, it is useful to pay attention to the temperature, because at lower temperatures not all the dye will fix to the fibre but will go to waste.

We applied the recipe and process given below:

Skein	g	Bath 1 & 2	Extraction temp / time	Assist	pH	Dye temp / time
1 2	60	Cochineal 12,5 % (= 7,5 g)	100°C / 60 min	none used	6	90°C / 60 min
7						

Skein 1: Silk dyed with cochineal.

This shade could have been further toned towards more purplish by after-

mordanting in a mild iron solution. However, we wanted to protect the delicate silk fibre for the later weaving process, so we left the colour as it is. The iron mordant would have made the silk more prone to fracturing during weaving. Iron would also have slightly dampened the brightness of the colour.

Skein 2: Woad-indigo overdyed with cochineal. Skein 7: Cochineal overdyed with logwood.

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Overdyeing Blue with Brazilwood

Brazilwood (*Caesalpina brasiliensis* L.) was another red dye newly imported to Early Modern Europe from the New World. Its dye compounds include brazilin and are extracted from the chips of this hardwood (and other *Caesalpina* species), which during history has added to the chopping down of redwood forests. The quantities of wood chips necessary for dyeing a small sample are enormous, and the colour is not lightfast, so even for research purposes larger samples are not recommendable.

To improve dye extraction from the wood chips, I had poured boiling water on them and let them steep overnight before starting the heat extraction process

the following day. The dye would also benefit from repeated extraction.
We applied the following recipe:

Skein	g	Bath 2	Extraction temp / time	Assist	pH	Dye temp / time
3	20	Brazil 500 % (=100 g)	100°C / 60 min	Potash (K_2CO_3) 12,5 % (= 2,5 g)	8-9	80°C / 60 min

In an alkaline dyebath, brazilwood produces a rich, plum-red dye, which fixes with silk fibre due to alum mordanting and readily oxidizes into brazilein. The colour could be deepened by after-mordanting with iron, but for the velvet weaving process we refrained from risking the silk. On our dark indigo base, the brazilwood dye was quite faint even with 500 % weight of fibre, as shown in the image below. There was also some loose residue of indigo dye left on the silk after washing, which may have impeded the bonding of the brazilwood dye on the yarn. The result on skein 3 was a greyish, even a little bit greenish blue rather than purple.

Skein 3, Uneven woad blue overdyed with brazilwood.

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Overdyeing Blue with Madder

Madder (*Rubia tinctorum* L) is a classic dye plant, and its roots develop a multitude of dye molecules, the quantity of which may vary due to soil quality and other environmental factors. Already the Early Modern dyers knew the varying qualities of madder from different geographical sources and adjusted their recipes accordingly. Alizarin is the most bountiful red compound in madder root, providing the warm tones of red madder was mostly coveted for. In addition to alizarin, madder contains tens of other dye molecules, such as purpurin, which dissolves in alkaline solution, as well as munjistin and rubiadin. Madder therefore affords a wide range of red tones that are colourfast.

To achieve purple instead of brownish tones, the dyer wants to avoid raising the temperature to above seventy degrees centigrade on the one hand, and to avoid fixing the yellow molecules to the material, on the other. The latter would be most recommendable to do by dyeing another material soaking up yellows after first extraction. To save time, the pieces of madder root can also be rinsed to get rid of most of the yellow dye molecules, and this is what we did.

So, after first rinsing the madder roots with boiling water and then with cold water, we proceeded with the following recipe:

Skein	g	Bath 2	Extraction temp / time	Assist	pH	Dye temp / time
4 5	40	Madder 100 % (= 40 g)	100°C / 30 min	Potash (K_2CO_3) 6 % (= 2,5 g)	8-9	70°C / 60 min

Madder root dye could also be modified towards a more purplish tone by after-mordanting with iron. But as repeated already before, we did not want to risk the delicate silk.

Skein 4, Woad and madder (left), skein 5, logwood and madder (right).

So, that is how we achieved our purples. The last pair of photos shows how differently silk reflects natural light (left) and flashlight (right). In Early Modern

Europe silk was seen under sunlight or in soft candlelight, which would have glimmered differently on these exquisite silks.

As an afterthought: Since these natural-dyed yarns showed to be much more durable for weaving that those with synthetic dyes, we could have used even smaller skeins (10 g each) and applied iron mordanting on half of them to have a wider selection of purples. The smaller skein size would also have facilitated a more even dye result with indigo.

References:

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