Purple from Shellfish

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Imagining a Recipe

There seem to be no *detailed* publications surviving either on the process of dyeing with *Murex* dyes, or on an exact recipe to use. For our experiment, I developed a recipe building on the suggestions of Mr. Ghassen, who provided us with the dried glands; examining existing literature on historical and more recent murex-dyeing; and literature and experience on indigo-dyeing, since also *Murex* dyes are indigoids in chemical structure.

Here is the recipe we chose to apply:

Dried mollusc glands	g	Extraction	Assist	pH	Extraction temp / time	Reducing agent	pH	Reducing temp / time
Stramonita haemastoma	60	Water H_2O 400 ml	Sodium hydroxide NaOH 6 g	10-11	40-50°C / 30 min	Sodium dithionate Na_2S_2O6 10 g	8-9*	55°C / 10 min
Bolinus brandaris	15							

Hexaplex trunculus	10							Let settle for 30 mins
TOTAL	85							

* If you need to adjust the pH, add a tiny amount of citric acid (if your solution is too alkaline, pH > 9) or soda ash (if your solution is too acidic, pH < 8). In our experiment, we did not need to make any adjustments.

Carthaginian colour, light, and oxygen

Mr. Ghassen has worked with reviving the tradition of murex-dyeing on the Southern Mediterranean in Tunis, ancient Cartagena, since 2007. His interest dates even further back to his school history class where he first learned of this magical purple dye. Nowadays, as he recounted in our online workshop, imperial purple is his passion. Yet, he does not follow an exact process when he dyes with *Murex*. He learned the skill by experimenting without detailed notes and has, consequently, developed a feel for how to achieve the best possible outcome at a given time.

He says the colour is influenced by several variables: The season of harvesting the *Murex,* the combination of the three species of *Murex* available, differences between individual molluscs, the water, the chemicals used, and even the amount of sunlight during the process. In sum, his focus has not been on developing a fixed recipe by meticulous testing of the multitude of options to achieve a particular shade of purple on a particular fibre, since the circumstances always vary.

First, when the dye is applied **fresh during the spring and autumn harvesting seasons**, the outcomes are most vibrant. This may be due to some of the dye getting destroyed when the glands are dried, or during some other phase of the process, but the fresh glands provide the deepest purples.

Second, discussing **the three species of *Murex*** depicted in the opening photo, Mr Ghassen explained, that *Stramonita haemastoma* provides imperial purple, but in vat-dyeing it needs another species to develop a permanent dye, because it lacks an enzyme necessary in the process. *Bolinus brandaris,* on the other hand, includes all that is necessary, but on average it contains merely five to six times less dye than the two other species, so the number of shellfish needed would be gigantic. Finally, *Hexaplex trunculus* is most abundant and therefore least costly of the three, but it produces a blue shade than the two others, due to a lower content of bromine than of indigo.

Third, the quality of **water and chemical**s applied influence the outcome. This is no news to experienced natural dyers since it is the case even with less delicate colorants. Waters and tap waters differ around the world, and adjusting the alkalinity of the solution, and reducing agent used to render the non-soluble dye precursors soluble may vary, too.

And last but by far not the least, **the impact of light**. This seems to have been the most controversial topic in literature, until archaeo-chemical research started to gain ground. These molluscs include indigoid dye molecules, which provide blues (*Tekhelet,* in particular). For purple, the dyer needs to coax the reddish tones, which are developed from the photo sensitive bromines. Only during the past ten years have archaeo-chemists been able to fully track the process for wool dyeing. In short, both the extraction and the oxidation processes should be conducted in subdued light, or, preferably, in total darkness.

Based on Mr. Gassen's tips and published information, and completed with experience on the processes for dyeing with *Isatis Tinctoria* and lichen dye *Xanthoria parietina*, this is the process and recipe we decided to experiment with:

Preparing the vat (about 30 minutes)

1. Measure the material and the dried glands: 20 g of fibre -> 85 g dried mollusc glands, combination of *Stramonita haemastoma* 60 g + *Bolinus brandaris* 15 g + *Hexaplex trunculus* 10 g.
2. Grind the dried glands, each type in a separate mortar. (We made a separate vat for each to get samples for later chemical analysis.)
3. Dilute 6 g soda ash Na_2CO_3 to 400 ml water. Share the solution into three vessels (300 ml, 50 ml, 50 ml) for the three types of molluscs, in a water bath in a larger kettle. Stir in the ground glands, check pH = 10-11.
4. Heat to 50°C, keep for 30 minutes. (The one-kettle and water bath system helped us control the heat).
5. Turn off the lights. Add 10 g (7,5 g/1,25 g/1,25g) of sodium dithionite $Na_2S_2O_4$, and stir.
6. Whisk gently without introducing bubbles (i.e., oxygen) into the dye vats. Take samples of each mollusc vat for chemical analysis, and gently pour the smaller vats into the bigger one. Cover the kettle with an opaque lid.

Letting the dye vat settle

7. Let settle in the dark for 30 minutes or until the solution has cooled down to ambient temperature, the colour of the solution is now yellow-green.

Dyeing the textiles

8. Still keeping the lights minimal, measure pH, should be about 8. (If over 9, add citric acid, a tiny quantity at a time. If 7 or under, add a pinch of soda ash, a tiny quantity at a time. Stir gently until completely dissolved and measure the pH again.)
9. Soak the textile samples in the vat in the dark, under an opaque lid:
    - Silk samples I for 45 minutes
    - Silk samples II for 120 minutes
    - Wool samples for 120 minutes
10. Let oxidise in air.
    - Silk samples I oxidised for 45 minutes in the light.
    - Silk samples II and wool samples oxidised 50 minutes in the dark.

11. Rinse with water, with a little bit of vinegar added to the last rinsing water.
12. Let dry.

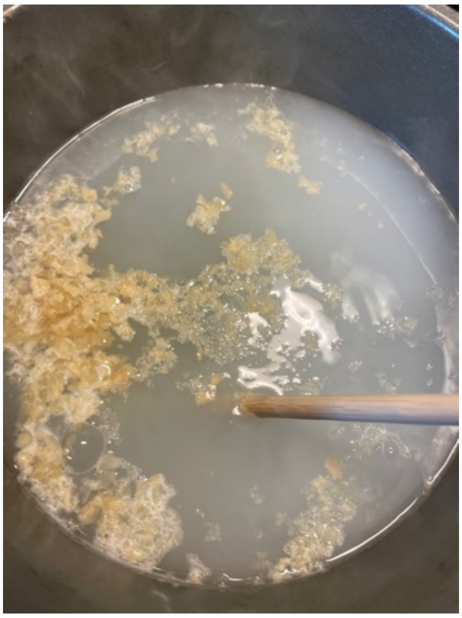
Results and discussion:
The recipe and process worked well for wool. The samples turned a beautiful, reddish purple, as shown in the photos.

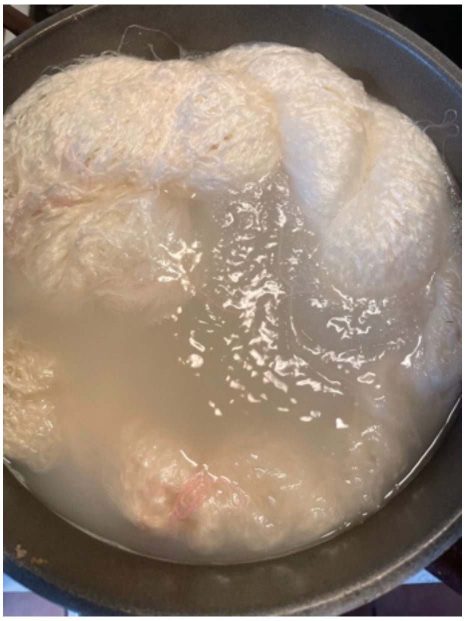
However, as predicted by Mr. Ghassen, silks ended up with a much more bluish tone, as evidenced in the next photo. Subsequently, there still seem to be secrets to be revealed for achieving stable imperial purple on silk.

Materials used:
Calabrian silk yarn 14,2 g (degummed beforehand, see below)
Habotai silk fabric 0,8 g
Wool yarn 2,5 g
Wool fabric 2 g

The Calabrian Silk yarns were degummed before the workshop, i.e., sericin was removed from them, as follows:

1. Measure the silk yarns
2. Put them in clean water and let soak overnight
3. Dilute Marseille soap, 25 % of the weight of the silk, into hot water.
4. Add the silk and bring to boil. Let boil for 90 minutes.
5. Rinse thoroughly and let dry.





References:

Ramig, K., Islamova, A., Scalise, J., Karimi, S., Lavinda, O., Cooksey, C., ... & Karapanagiotis, I. (2017). The effect of light and dye composition on the color of dyeings with indigo, 6-bromoindigo, and 6, 6′-dibromoindigo, components of Tyrian purple. *Structural Chemistry*, *28*, 1553-1561.