KdG Research
University of Applied Sciences and Arts

Research project **Biocolor**: natural dyes from plant materials and agricultural by-products

Introduction

Color is an important criterion of attractiveness and acceptability of many products, such as textiles, cosmetics and food. While dyes were exclusively natural in the past, this changed in the 1850s when mauveine was the first synthetic dye to be produced. Due to high expectations concerning the performance of colorants, the majority of dye substances used in several industries nowadays have a synthetic origin. A disadvantage of the synthetic dyes is its potential to cause health problems and its harmful effects on the environment. Therefor, regulations for the use of colorants has become more strict during the last decades. Coupled with the ecological awareness of consumers, the development of high-performing natural dyes is in high demand.

In this context the Biocolor research project (2020-2022) was launched. In collaboration with its industrial and academic partners, the project examined the potential of plant materials and agricultural by-products as a source for high-performing colorants. Textiles, food and candles were chosen as case studies to apply the produced dyes. The agricultural residues and biomass that will be used for this project are principally available in Belgium and the Netherlands.

Textile

This case study describes the complete cycle of the production of dyed textiles, beginning with the drying of the plant materials and finishing with dye bath application.

This report will present the following steps:

- Drying of plant materials
- Production of dye bath
- Pretreatment of textiles
- Dyeing protocol

Drying of plant materials

Depending on the plant material, a drying step is necessary to prevent deterioration of the biomass during its storage. Wet materials like green leaves, flowers and roots can quickly become moldy when not taken care for properly. Some agricultural by-products however are dry by itself. A good example of this case are onion peels, which are separated from the bulb during the sorting process once harvested.

During this project, freeze drying was used to dry all materials to be able to compare the coloring capacity of each material on a dry weight basis.

Once dried, the plant material is milled to reduce particle size and increase the contact surface for extraction purposes.

Production of dye bath

Solid liquid extraction (SLE) was used to extract the coloring substances from the plant materials and agricultural by-products. To do so, a 25 g L^{-1} solution was made of each dried material, using water as the extraction solvent. The solution was stirred at 90 °C during 60 minutes. Afterwards the solution was sieved to remove the raw material. The remaining dye bath was used to dye the pretreated textiles.

Pretreatment of textiles

For this project silk, wool and cotton were used. The textiles could be pretreated in three ways:

- No pretreatment
- Potassium aluminum sulphate (alum) pretreatment - $KAl(SO_4)_2.12H_2O$
- Iron sulphate pretreatment – $FeSO_4$

For the alum pretreatment the protocol was as follows: 20% alum was weighed relative to the weight of the fabric to be pretreated. The alum was dissolved in water at 90 °C. For the pretreatment, 1 L of water could be used to treat 30 g of textile. Once the alum was dissolved, the textile was added and left to soak for 60 min at 90 °C while stirring occasionally. Afterwards, the textiles were rinsed with clear water and left to dry in open air until further use.

The protocol for iron pretreatment was similar, however only 1% $FeSO_4$ needed to be used relative to the weight of the fabric to be pretreated.

Dyeing protocol

The textiles were added to the dye bath at a ratio of 1:50, meaning 50 g of dye bath was used for each gram of textile. The textiles were left to soak in the dye bath for 60 min at 90 °C while stirring occasionally. Afterwards, the textiles were rinsed with clear water and left to dry in open air.

Results of textile dyeing case study

In total, 29 samples of plant materials and agricultural by-products were examined for their capacities to dye textiles (table 1). 14 samples gave only little color to the textiles. On the other hand, some samples showed to be promising by delivering bright and vibrant colors to the textiles. Madder (red), calendine (yellow) and yellow onion (orange) are examples of this.

Table 1: List of plant materials and agricultural by-products used to dye textiles without pretreatment.

Plant species	Scientific name	Plant part used	Color obtained
Mexican marigold	Tagetes sp.	Flower	Yellow/green
Calendine	*Chelidonium majus*	Aerial parts	Yellow
Pomegranate	*Punica granatum*	Peel	Yellow
Tansy	*Tanacetum vulgare*	Flower	Yellow
Weld	*Reseda luteola*	Aerial parts	Yellow
Yellow chamomile	*Anthemis tinctoria*	Flower	Yellow
Madder	*Rubia tinctorum*	Root	Red
Cleavers	*Galium aparine*	Root	Pink
Thyme	*Thymus vulgaris*	Aerial parts	Orange/yellow
Wild privet	*Ligustrum vulgare*	Leaves	Orange/yellow
Yellow onion	*Allium cepa*	Peel	Orange/yellow

Plant species	**Scientific name**	**Plant part used**	**Color obtained**
Avocado	*Persea americana*	Seed	Orange/pink
Docks & sorrels	Rumex sp.	Root	Orange
Walnut	*Juglans regia*	Black husk	Orange
Elderberry	*Sambucus nigra*	Fruit	Blue/purple
Black cumin	*Nigella sativa*	Press cake	No/little color
Blackcurrant	*Rubus allegheniensis*	Press cake	No/little color
Californian cypress	*Chamaecyparis lawsoniana*	Aerial parts	No/little color
Carrot	*Daucus carota*	Leaves	No/little color
Common marigold	*Calendula officinalis*	Flower	No/little color
Elderberry	*Sambucus nigra*	Press cake	No/little color
Nettle	*Urtica dioica*	Aerial parts	No/little color
Red beetroot	*Beta vulgaris*	Root	No/little color
Red cabbage	*Brassica oleracea*	Leaves	No/little color
Sage	*Salvia officinalis*	Aerial parts	No/little color
Spinach	*Spinacia oleracea*	Aerial parts	No/little color
Tomato	*Solanum lycopersicum*	Fruit	No/little color
Walnut	*Juglans regia*	Brown shell	No/little color
Woodbine	Parthenocissus sp.	Fruit	No/little color

A striking observation was the big color change that can be caused by the textile pretreatment. Alum tends to brighten colors, while iron darkens the colors. The textile type itself also greatly influences the color outcome.

Textile pieces dyed with yellow onion extract. From left to right: cotton alum – cotton iron – silk alum – silk iron – wool alum – wool iron.

Color fastness, meaning resistance to rubbing, washing, light exposure and perspiration, is a important feature putting the natural colorants to the test. Unfortunately, the vibrant colors given by some dye baths did not withstand UV-light exposure. Most colors faded, but again the textile type was a big contributor. The wool and silk samples presented better color stability compared to cotton.

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Evaluation of the case study

The use of plant materials and agricultural by-products for textile dyeing shows promising results. A surprising observation is the fact that colors on textile are difficult to be predicted. Pomegranate for example gives a bright yellow color to wool while the peels used to produce a dye bath were bright pink. This results in the need to test as many biomass sources as possible to diversify the color palette that can be produced. Attention should be given to the fact that all results presented in this report were obtained by performing experiments in a laboratory setting on a non-industrial scale.

Some restrictions concerning the use of plant materials were identified during the project:

- To obtain sufficient quantities of raw materials
- To obtain regular supply of raw materials, since some are only available during a limited time of the year
- To obtain stable color results, which do not fade due to UV-light exposure
- To obtain consistent color results, despite differences in the source of the same material

These limitations should be addressed before plant materials can be seen as a perfect alternative to replace synthetic colorants for textiles.

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In collaboration with Centexbel, Delphy, HoGent, Ilvo Foodpilot, Inagro and Masureel