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Natural dyes for textile dyeing: A comparison of methods to assess the quality of Canadian golden rod plant material

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Abstract

The introduction of natural dyes into modern textile dyehouses requires the classification of products of standardised quality with regard to colour depth and shade of the dyeings. Canadian golden rod was chosen as a representative example to test the methods that are available to assess the quality of different crops of plant material which had been collected over a period of five years. Aqueous solutions containing the extracted flavonoid dyes were characterised by means of direct photometry, measurement of absorbance after addition of $FeCl_2$, analysis of total phenolics (TPH) in the extract and dyeings on wool yarn.

TPH calculated as gallic acid varied from 62 g/kg to 97 g/kg of plant material; only one sample exceeded this range with a value for TPH of 142 g/kg. Correlation among TPH, photometry in the presence of $FeCl_2$ and lightness of the dyeings can be used to characterise samples. However, correlation between the photometric results and colour depth of dyeings is not sufficient to permit characterisation of the plant material with regard to the final dyeing. At present, a combination of laboratory dyeings and CIELab coordinates was found to be suitable to establish an experimental basis for standardisation of plant material.
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1. Introduction

With the appearance of synthetic dyes the use of natural dyes for textile dyeing almost disappeared. The wide range of colours available with good fastness properties at moderate costs was the main reason for the replacement of natural dyes by their synthetic counterparts [1].

Nowadays, there is a growing interest in the revival of natural dyes in textile dyeing; arguments based around keywords such as sustainability, green chemistry, improved eco-balances and thereby leading to niche products for special markets [2,3].

The introduction of natural dyes into textile dyehouses is coupled to several requirements which have to be fulfilled:

- adaptation of traditional processes on modern equipment [3,4]
- supply of dyehouses with an appropriate amount of plant material [4,5]
- selection of materials leading to products with acceptable fastness properties [6–9].

An additional important aspect which also has to be considered by an imaginary supplier of natural dyes has been identified during an extensive study of possible future use of natural dyes [10,11], namely that the plant material which contains the natural dye needs the same level of standardisation as modern synthetic dyes already have achieved at present. Important parameters which have to

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be adjusted to a fixed level and confirmed within defined limits by analysis and standardisation procedures are as follows:

- tinctorial strength of the plant material
- shade of the dyeing on various textile substrates
- fastness properties of the dyeing.

Although numerous papers have described the selection of plant raw materials, dyeing procedures, shade of dyeings and fastness properties [12–18], only little information is available in the literature concerning:

- variations among different crops of the same plant
- reproducibility of dyeings
- simple techniques to analyse and standardise a given plant material.

Standardisation of the plant material requires the elaboration of methods to evaluate the properties of a certain batch of plant material with regard to particular dyeing properties (colour strength and shade). Such procedures will be based on analytical methods which depend on the type of plant material. In an ideal case the characterisation of the extract permits the manufacturer of the natural dyestuff production to adjust a certain batch of material to a desired standardised colour strength and shade, analogous to the standardisation of a synthetic dyestuff during the finishing of dyestuff.

In this work, Canadian golden rod was chosen as a representative source for a plant based yellow natural dye. The dyes found in Canadian golden rod are the flavonoid dyes quercetin (C.I. Natural Yellow 10) and kaempferol (C.I. Natural Yellow 13,10) [1]. A set of simple analytical procedures and variations in application of the dyes were compared with regard to a possible correlation of the dyeing results. Different photometric methods, including an analysis of the total phenolic components (TPH) extracted from the material, were studied to predict the shade and colour strength in the following dyeing procedure [19–21].

Variations in batches of plant material were monitored during a period of over five years.

The methods should enable the supplier of the natural dye to produce batches of plant material with similar, i.e. almost identical dyeing properties.

2. Experimental

2.1. Chemicals and reagents

Analytical grade chemicals were used for the phenol analysis (Na_2CO_3, Folin–Ciocalteau reagent (Sigma–Aldrich Chemie, Steinheim, Germany)).

Technical quality chemicals were used for the dyeing processes: $FeCl_2$ (33% aqueous solution, BASF AG, Ludwigshafen am, Germany), $FeSO_4 \cdot 7H_2O$ (technical grade >96% purity, Riedel-de-Haen, Seelze, Germany), alum, $KAl(SO_4)_2 \cdot 12H_2O$ (puriss. pa Fluka, Buchs, Switzerland).

2.2. Plant material

To investigate the variations in the dyestuff content during different years a number of plant samples were collected from different sites and during a period of several years (1999–2003) (Table 1). The plants were collected as a whole including buds, part of stem and upper part of leaves.

The material was dried at room temperature and stored in dark.

2.3. Extraction of dye

A weighed amount of dry plant material was extracted with distilled water in a beaker. In the standard procedure the ratio of mass of plant material to the volume of liquid was 1:20; extraction was performed for approximately. 60 min at 95 °C in an open stainless steel beaker. Due to the rather high liquor ratio some manual stirring was sufficient to distribute the plant material in the liquid during the extraction period. Volume loss due to evaporation was compensated by the addition of water at the end of the extraction period to obtain the initial volume.

All extracts were freshly prepared before analysis. The extracts were analysed by measurement of the λ_{max} using a 10 mm cuvette and a diode-array spectrophotometer (Zeiss CLH 500/MCS521 UV–vis, Carl Zeiss (Jena), Germany).

Total soluble phenolics (TPH) in the extract were determined with Folin–Ciocalteau reagent according to the method of Slinkard and Singleton using gallic acid as standard [19–22]. The extracts were diluted with distilled water to adjust extinction within the range of the calibration curve. Results were expressed as both $mg\,L^{-1}$ of phenols in extract or g/kg of dry plant material calculated as gallic acid equivalents [19–21]; for extraction and photometric analysis of phenols least two repetitions were performed.

In another analytical method the extract was characterised by formation of the Fe(II)–phenol complex which then was quantified by measurement of absorbance at $\lambda = 600$ nm. A volume of 1 mL of freshly prepared extract was diluted with 10 mL of deionised water and the absorbance of the solution was measured at 600 nm before and after addition of 0.5 mL of diluted $FeCl_2$ solution ($50\,mL\,L^{-1}$ commercial $FeCl_2$ solution) to develop the final colour. As partial flocculation occurs after addition of the Fe(II)-salt, the solution was measured a short time after addition of the Fe(II)-salt.

2.4. Dyeing experiments

As material for the dyeing experiments, washed and bleached wool yarn ready to be dyed was used in the form of small hanks (Schoeller Wool, Hard, Austria), and desized,

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