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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.

Keywords: Flavonoid dyes; Natural dye; Textile dyeing; Wool; Quality determination; Phenolics

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,

Table 1
Results of standardised dyeing experiments with different plant material using Fe(II)-mordant on wool yarn

No.	Year	Mordant	L^*	a^*	b^*	TPH mean ($\mathrm{g\,L^{-1}}$)	Std. dev. ($\mathrm{g\,L^{-1}}$)	TPH plant (g/kg)	Abs. (λ nm)	Abs. (λ_{max} nm)	Abs. (600 nm)	Abs. (600 nm) Fe(II) added
1	1999	$\mathrm{FeCl_2}$	27.8	−0.5	+8.8							
		$\mathrm{FeCl_2}$	26.1	−0.1	+9.0	3.82	1.55	76.4	1.3 (370)	1.8 (405)	0.1	0.7
		$\mathrm{FeSO_4}$	32.4	−1.1	+10.7				1.3 (390)			
2	2000	$\mathrm{FeCl_2}$	27.6	+1.0	+12.6							
		$\mathrm{FeCl_2}$	26.1	+0.8	+11.2	3.37	0.30	67.4	1.3 (390)	2.7 (450)	0.1	1.4
		$\mathrm{FeSO_4}$	43.5	−1.1	+13.7				1.3 (390)			
3	2001	$\mathrm{FeCl_2}$	27.0	+0.4	+11.4							
		$\mathrm{FeCl_2}$	21.5	+1.3	+10.3	3.10	0.16	62	1.3 (380)	2.4 (440)	0.1	1.1
		$\mathrm{FeSO_4}$	30.2	+0.5	+12.7				1.25 (390)			
4	2002	$\mathrm{FeCl_2}$	28.1	+1.1	+12.8							
		$\mathrm{FeCl_2}$	26.2	+1.2	+12.3	3.57	0.92	71.4	1.35 (375)	2.2 (420)	0.1	0.85
		$\mathrm{FeSO_4}$	38.3	−1.4	+14.5				1.3 (390)			
5	2003	$\mathrm{FeCl_2}$	23.9	+1.3	+9.9	4.87	2.62	97.4	1.5 (385)	3.6 (490)	0.1	3
		$\mathrm{FeSO_4}$	32.3	−0.6	+13.2				1.3 (390)			
6	2001	$\mathrm{FeCl_2}$	24.3	+0.5	+9.6							
		$\mathrm{FeCl_2}$	25.5	+0.7	+11.2	3.73	0.92	74.6	1.3 (385)	1.4 (400)	0.1	0.6
		$\mathrm{FeSO_4}$	31.9	−0.6	+11.2				1.3 (390)			
7	2002	$\mathrm{FeCl_2}$	24.3	+1.3	+10.2	4.13	0.40	82.6	1.35 (390)	2.1 (425)	0.15	0.7
		$\mathrm{FeCl_2}$	27.7	+0.7	+11.9							
		$\mathrm{FeSO_4}$	31.5	−0.7	+13.1				1.3 (390)			
8	2002	$\mathrm{FeCl_2}$	26.5	+0.7	+11.4							
		$\mathrm{FeCl_2}$	23.3	+1.3	+9.4	7.10	2.89	142	1.4 (375)	2.2 (410)	0.1	1.4
		$\mathrm{FeSO_4}$	31.9	−0.6	+14.4				1.3 (390)			
9	2003	$\mathrm{FeCl_2}$	22.7	+1.4	+9.1				1.5 (400)	4 (480)	0.1	3.2
		$\mathrm{FeCl_2}$	21.4	+1.3	+8.3	3.85	1.07	77.0	1.5 (390)	4.1(470)	0.1	3.5
		$\mathrm{FeSO_4}$	28.1	−0.4	+11.5				1.3 (390)			
		$\mathrm{FeSO_4}$	30.2	−0.7	+12.2				1.3 (390)			

Data include year of collection, type of mordant, CIELab coordinates, TPH in extracts, TPH calculated per kilogram of dry plant material, absorbance of the extracts (wavelength of measurement), absorbance after addition of $\mathrm{FeCl_2}$ (600 nm). Analytical data of absorbance on line with the respective dyeing.

scoured and bleached cotton fabric (Getzner Textil AG, Bludenz, Austria) was used as cellulose material.

Dyeing was performed using a liquor ratio of 1:20–1:22 at 95 °C in open beakers with manual agitation of the material. The wool yarn or the cotton fabric were added to the extract, temperature was raised to 95 °C within 10–15 min and maintained at that temperature for 50 min. As a meta-mordating procedure was used, the calculated amount of mordant was added to the dyebath after 15 min at 95 °C. No adjustment of pH was made.

In case of mordanting, $\mathrm{FeCl_2}$, $\mathrm{FeSO_4\cdot 7H_2O}$ or alum, $\mathrm{KAl(SO_4)_2\cdot 12H_2O}$ was added to the dyebath to give a final concentration of 4.5 $\mathrm{mL\,L^{-1}}$ $\mathrm{FeCl_2}$ (0.016 $\mathrm{mol\,L^{-1}}$ Fe), 4.5 $\mathrm{g\,L^{-1}}$ $\mathrm{FeSO_4\cdot 4H_2O}$ (0.016 $\mathrm{mol\,L^{-1}}$), or 4.5 $\mathrm{g\,L^{-1}}$ alum mordant, respectively. The detailed dyeing procedure is given in literature [3,21]. At the end of the dyeing period at 95 °C the bath was cooled down to approximately 60 °C and the dyebath was removed as to start with rinsing.

After dyeing, excess dye was removed from the dyeings by rinsing three times with cold water.

The CIELab coordinates for the dyeings were measured using a Minolta Chroma-Meter CR 210, sample diameter 10 mm (geometry *d*/0°). The L^*, a^*, b^* were calculated for light source D65. The values are calculated from three repetitive measurements at the same place of sample.

For extraction and photometric analysis of phenols least two repetitions were performed, average and standard deviation are given in Table 1.

3. Results and discussion

3.1. Extraction step

Extraction of Canadian golden rod yields several flavonoid dyes; representatives are quercetin, kaempferol and the corresponding glucosides [1]. Simple techniques were tested and compared for their suitability as analytical procedures to standardise the quality of a plant material by extract analysis because full analysis of the complex mixture of compounds extracted would extend the possibilities of a natural dye producer by far.

A 60 min extraction at the boil was used as a standard procedure for the extraction of natural dyes from plant matter [3,21]. To confirm that extraction equilibrium had been established during this time, both the release of coloured material and the extraction of phenolic components were monitored during extraction.

An example for the increase in absorbance as a function of extraction time can be seen in Fig. 1 where the extraction of sample no. 9 as a function of time is shown. In parallel with this, the determination of extracted phenols, TPH, by the Folin–Ciocalteau method [19–21] was performed. As can be seen in Fig. 1 for both analytical parameters a considerable part of the extractable material was found in solution within 20 min extraction time. The major part of coloured substances was extracted within the first 30 min of the 60 min extraction time at temperature of 95 °C.

The absorption spectra of the extracts are shown in Fig. 2.

Due to the high surface and good accessibility of the plant material to be extracted only a relative short extraction time was sufficient.

For the standardised extraction an extraction period of 1 h was defined because structural differences in investigated plant material then are of minor relevance for the final concentrations achieved in the extract.

The aqueous extracts could be used in different variations of the dyeing process, e.g. as a direct dye or with the addition of a mordant. Ecological requirements limit metal based mordants mainly to Fe(II)-salts and alum [3,23]. In Table 2 representative results of dyeings with extracts are given. The label "direct" indicates that the extract was used as dyebath directly without further addition of chemicals for fixation, thus the procedure is similar to the application of a direct dye. As examples for application of the extracted dyestuff as mordant dye, examples with the addition of Fe(II)-mordant and alum on cotton and wool are shown.

As can be seen the obtained colour varies from yellow ("direct" dye and alum mordant) to olive (Fe(II)-mordant). Selected fastness properties are given in the literature [3]. Dyeings on cotton showed acceptable fastness to light and water for all three variations; on wool only the dyeing with use of Fe(II)-mordant exhibited sufficient high fastness to be of interest for textile dyeing.

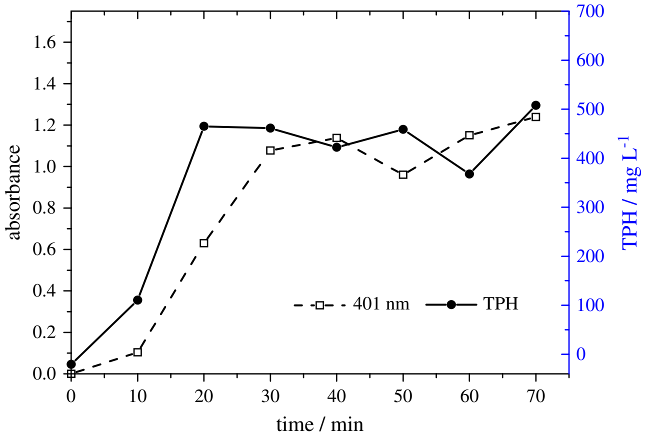

Fig. 1. Extraction of plant material no. 9 – absorbance of extracts (dilution 1 mL extract plus 10 mL deionised water) and TPH (mg L^{-1}) as a function of extraction time.

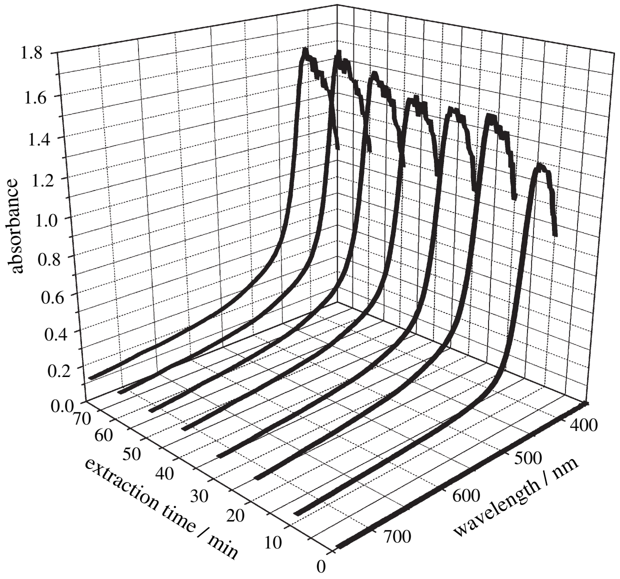

Fig. 2. Extraction of plant material no. 9 – absorption spectra of extracts as a function of extraction time (dilution: 1 mL extract plus 10 mL deionised water).

Shade and colour depth of the dyeing with use of Fe(II)-mordant on wool was of considerable interest for a colour gamut based on natural dyes, thus this dyeing was taken as a basis for experiments to investigate methods to standardise the plant materials by means of chemical analysis of the extract.

3.2. Characterisation of extract

The following analytical procedures based on the analysis of the aqueous plant extract were studied with regard to their value as a possible method for quality assessment and standardisation.

- photometry of the extract in the range of 350–700 nm
- photometry of the extract with addition of Fe(II)-mordant
- analysis of total soluble phenolic compounds (TPH) in the extract

3.2.1. Photometry of the extract in the range of 350–700 nm

For normal synthetic dyes initial measurement of the absorbance of a dyebath in the visible range light correlates with the

Table 2
Representative results of dyeings from dried Canadian golden rod no. 5 on wool yarn (Wo) and cotton fabric (Co)

Material	Mordant	*L**	*a**	*b**
Wo	Direct	69.4	−1.5	+34.9
Wo	$FeSO_4$	36.1	−1.6	+13.6
Wo	Alum	74.1	−3.9	+69.4
Co	Direct	89.2	−3.1	+13.0
Co	$FeSO_4$	57.6	−4.0	+13.1
Co	Alum	78.3	−6.6	+52.9

Direct: application as "direct dye"; mordants: 4.5 g L^{-1} $FeSO_4$, 4.5 g L^{-1} alum.

final colour depth of the dyeing, and thus this method was chosen as a simple method to classify different extracts.

In Table 1 values of the absorbances registered in the range of 385–400 nm are given and also readings at the wavelength of maximum absorbance are presented. However, no correlation between the variations in absorbance of the extracts and the colour of the dyeings could be identified. Of course for a given extract such a correlation could be expected, however, resolution of the method was not sufficient to identify variations between the different extracts with regard to the final colour of the dyeing.

A problem in the photometric analysis arose from the fact that turbid solution was formed during the extraction and thus part of the absorbance was due to the presence of insoluble substances.

3.2.2. Photometry of the extract with addition of Fe(II)-salt

Addition of Fe(II)-salts to the extract yielded the corresponding Fe(II)-complexes of the extracted flavonoid components, which resulted in increased absorbance. Thus a variation in the photometric analysis was carried out, where Fe(II)-solution was added to the diluted extract and absorbance was measured at 600 nm. For comparison the absorbance of the diluted extract before addition of Fe(II)-salt is also shown in Table 1.

In Fig. 3 the correlation between lightness value of the dyeings and absorbance measured at 600 nm is given. Considerable variation between photometric data and results of the corresponding dyeing experiments was observed.

The found variation can be explained with several reasons:

- variation in the composition of the extracted solutions
- precipitation of finely dispersed and colloidal dissolved material by added Fe-salts
- not all substances in the extract which form coloured Fe-complexes actually were exhausted as natural dyestuff in the dyeing step.

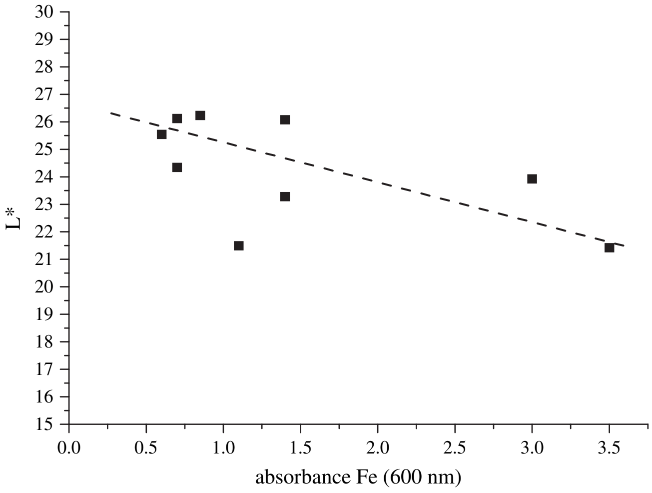

Fig. 3. Dependence of L^* of dyeing as a function of absorbance of extract measured at 600 nm after addition of $FeCl_2$ solution.

3.2.3. Analysis of total soluble phenolic compounds (TPH) in the extract

The flavonoid compounds contain phenolic groups in their chemical structure. According to the literature the Folin–Ciocalteu method was applied to determine the total phenolic content in the extract [19–21].

The results of TPH analysis of the different extracts and corresponding L^* of the dyeings are shown in Table 1. A graphical representation of the L^* of the standard dyeings as a function of the TPH content is shown in Fig. 4. The type of mordant, $FeCl_2$ or $FeSO_4$, is distinguished in Fig. 4.

Dyeings with used $FeCl_2$ showed remarkably darker shades compared to dyeings with $FeSO_4$. In Fig. 5 the data of the dyeings with $FeCl_2$ are shown in an enlarged presentation and related to the TPH content of the plant material. Eliminating the data obtained from extracts with extremely high TPH of 7.1 $g\,L^{-1}$, which in practice would be excluded from the process of standardisation, the data of other dyeings follow a general trend for darker dyeings (lower L^*) with increasing TPH content. Data points from different batches of plant material were found remarkably close together within a relatively narrow part of the CIELab-colour space. From the TPH values of the extracts an amount of TPH per kilogram of dry plant material could be calculated. This concentration of TPH in the solid plant material varied between the different plant batches from 62 g/kg to 97.4 g/kg, and for one sample (no. 8) a maximum value of 142 g/kg was analysed.

This indicated at least a limited suitability of the TPH analysis to be of value for a rapid evaluation and selection of material in the standardisation process.

The limited correlation between TPH and L^* could be explained by the fact that part of the extracted phenolic components was not participating in the formation of coloured components on wool, which is similar to the results obtained from UV–vis photometry and photometry of Fe-complexes (Fig. 1).

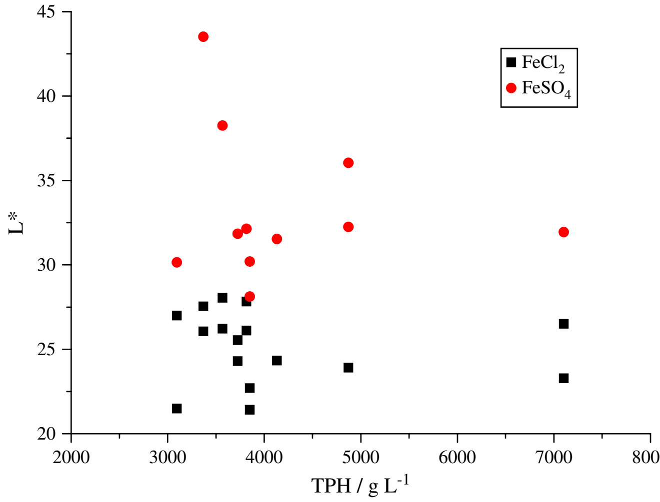

Fig. 4. L^* of dyeings using $FeCl_2$, and $FeSO_4$ mordants as a function of total phenol concentration (TPH) ($g\,L^{-1}$).

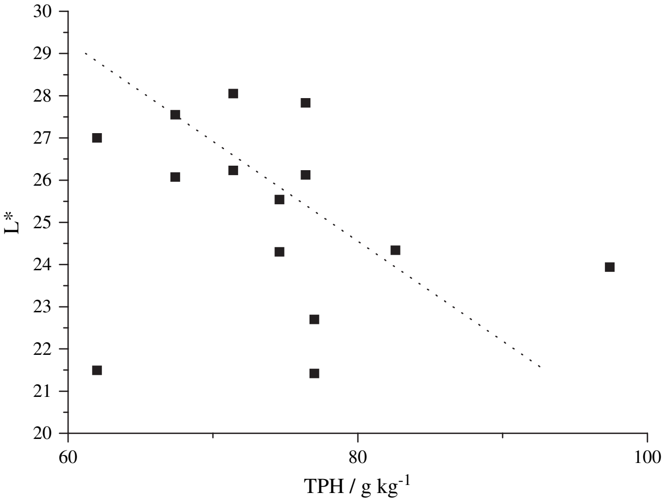

Fig. 5. L^* of dyeings using $FeCl_2$ mordant as a function of total phenol concentration (TPH) in plant material (g/kg^{-1}).

Only a certain share of the extracted phenolic components thus could be considered to be of value as natural dyestuff which shows affinity to the woolen substrate.

3.3. Standardised laboratory dyeing

In the laboratory dyeing experiment the aqueous extract was used in standardised dyeing experiments on wool, with use of Fe-mordant. The dark olive shade of the dyeing was chosen because of its higher sensitivity to variations in the concentration of the extract, compared to the yellow colour obtained when alum mordant is added.

As can be seen in the CIELab coordinates shown in Table 1 distinct differences exist between dyeings obtained with $FeCl_2$ and $FeSO_4$ mordants.

In Fig. 6 all dyeings are presented as pair of CIELab coordinates either a^* vs. L^* or b^* vs. L^*.

The mordants $FeCl_2$ and $FeSO_4$ are represented with different signs.

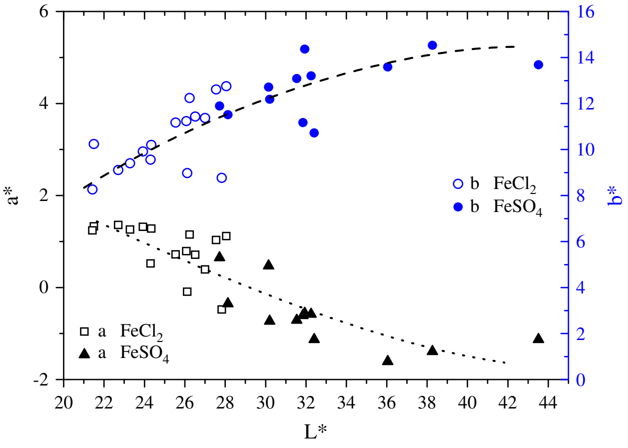

Fig. 6. Variation in L^*, a^* and b^* of test dyeings (use of $FeCl_2$ or $FeSO_4$ mordant).

Independent of plant material and Fe(II)-mordant used two general lines can be seen for a^* vs. L^* and b^* vs. L^*. This indicates that differences between the different sorts of plant material could be compensated by correction of the amount of extracted material.

From the point of technical dyeing using synthetic dyes a colour difference (ΔE) of ± 1 is accepted as tolerable colour difference between two dyeings. The introduction of natural dyes into textile dyeing processes yields products which in a first step address to consumers who are interested in ecological production, sustainability and renewable resources. This permits at least for such niche products some argumentation with possible variations due to non-synthetic raw material [11]. Thus for natural dyeing a somewhat wider colour difference of $\Delta E = \pm 2$ could be argued with the use of natural resources.

In Fig. 6 for $FeCl_2$ mordant the major part of dyeings can be expected to be adjustable by varying the amount of plant material extracted, within a range of $\Delta E = \pm 2$ around a set mean value for L^*, a^* and b^* coordinates, which defines the standard quality.

Some materials will not be adjustable to this standard, which then will have to be excluded from further processing.

4. Conclusions

From an analysis of the dyeing properties of Canadian golden rod samples collected over a period of five years only relatively small differences in colour depth and shade were found among the major parts of the different materials collected.

At present for dried Canadian golden rod standardised laboratory dyeing experiments were found to be the most relevant procedure to select and adjust a batch of raw material as to set level of colour depth and shade.

UV–vis photometry with or without addition of Fe(II)-salt or determination of TPH could support identification of material which will exhibit large deviation from the standard average quality, however, a quality standardisation based only on analytical determination of these two parameters cannot be recommended.

Considerable variation in absorbance of the extracts in the wavelength interval from 350 to 700 nm was found and TPH concentrations varied from 3.10 g L^{-1} to 7.10 g L^{-1} while CIELab coordinates of the laboratory dyeings on wool did not change to the same extent. This could be explained with presence of other coloured components, and the presence of the phenolics in the extract which were not of value as natural dye for textile substrates.

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