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Dyeing Properties and Analysis by Rp-Hplc-Dad of Silk Fabrics Dyed with Madder (*Rubia tinctorum* L.)

Karadag Recep[1,2]*, Torgan Emine[2] and Erkan Gökhan[3]

[1]*Laboratory for Natural Dyes, Faculty of Fine Arts, Marmara University, Istanbul, Turkey*
[2]*Turkish Cultural Foundation, Natural Dyes Research and Development Laboratory, Istanbul, Turkey*
[3]*Gökhan Erkan, Dokuz Eylül University Textile Engineering Department, Izmir, Turkey*

Abstract

Madder (*Rubia tinctorum* L.) has been used for dyeing textiles since the Stone Age. Common madder produces pigments in its roots, such as alizarin, pseudopurpurin, purpurin, munjistin, rubiadin, xanthopurpurin, purpuroxanthin, lucidin, chinizarin, christofin, and antrhagallol. Madder gives a unique red colour to textiles. The aim of this study is to understand the effect of different mordant concentration on silk dyeing by using madder extract. Degummed and bleached, woven silk fabric was used. Alum $[KAl(SO_4)_2.12H_2O]$ was used as mordant. All reagents were analytical grade. Silk fabrics were scoured by soap (35% w/w) at 90°C for 1.5 hours. Liquor ratio was 100:1. Dyeing was performed at 65°C, 100% owf, for 2 hours. Mordanting procedure was achieved at different mordant ratio, for 2 hours, at 65°C. All color measurements were performed using Minolta 3600D spectrophotometer (D65 illuminant, specular included, 10° observer angle). Colour fastness to washing, light, perspiration and rubbing was performed according to ISO 105 C06, ISO 105 B02, ISO 105 E04 and ISO 105 X12 respectively. Each dyed silk fabrics were analysed RP-HPLC-DAD. Semi-quantities of colouring compounds were determined in the dyed silk fabrics, depending on the amounts of mordant metal.

Keywords: Madder; *Rubia tinctorum* L.; Silk fabrics; Colour measurement; Alizarin; Purpurin; Fastness; HPLC-DAD

Introduction

Natural dyes are obtained from dye plants and dye animals in nature [1-9]. They were the primary colour source of textiles until the mid - to late 19th century [10-13]. For a long time, natural dyes have been used for purposes such as the colouring of wool, mohair, cotton and silk fibres as well as fur and leather [10,11,14-18]. Anthraquinones, naturally occurring in the madder roots (*Rubia tinctorum* L.), have been used for dyeing fabrics or fibres especially to the colour red and they have also been used as a lake pigment rarely since ancient times [19]. In addition to their antioxidant, antimicrobial, antifungal and hypotensive effects; anthraquinones have also been known for their various effects such as anticancer and skin diseases [20]. Madder (*Rubia tinctorum* L.) and other alizarin-containing plants belong to the most important group of red dyestuffs found in nature all over the world. Many of these belong to the large plant family of the Rubiaceae [20-23]. The dye plant is mentioned in early literature on dyeing and textiles dyed with a madder type, dated to 3000 BC, have been found in Mohenjo-Daro in the Indus valley. Madder type dyestuffs belong to the group of mordant dyes which need a pre-treatment with a metal salt. The metal salts most in use are alum compounds.

The main colouring compounds are alizarin and purpurin. Together with alizarin and purpurin, a number of anthraquinones such as xanthopurpurin, pseudopurpurin, rubiadin and munjistin are present. In the drying process of madder roots, pseudopurpurin is probably converted into purpurin. In addition, the metal compound used for mordanting, such as aluminium or iron, will influence the uptake of the various colouring components [21].

High Performance Liquid Chromatography (HPLC) using Diode-Array Detection (DAD) is ideally suited for identification of natural dyestuffs present in these materials [22-26].

The colour of the fibre is the result of three combined factors: the spectrum of the light source, the spectral reflectivity of the fibre colour, and the spectral sensitivity of the eye. The CIELAB (1976) system was introduced to describe the colour that results from these three factors. The system is a three dimensional space, with coordinate axes L*, a* and b*. The L* axis denotes the lightness of the colour (L* of 0 corresponds to black, while L* of 100 denotes white), a* represents the green–red axis (a* negative: green, a* positive: red), and b* represents the blue–yellow axis (b* negative: blue, b* positive: yellow). Each fibre colour can be represented as a set of values for L*, a*, and b*, and consequently as a point in this colour space [27]. Colour and fastness tests of the natural fibers dyed with different plants is worked by most researches [28,29].

Materials and Methods

In this study, 100% silk, sateen weave S 4/1 (3) fabrics that were ready for dyeing were used. The sateen weave used a 4/1 weaving ratio, floating each weft thread under four warp threads, then over one thread, and the rotation used was 3. The warp density per cm of the fabric was 160 and the weft density per cm was 60. The weight of the fabric was 74 g/m^2. The silk used in this study was obtained from the Armagan Company (Turkey).

All reagents were analytically graded, unless stated otherwise. Madder (*Rubia tinctorium* L.) was obtained from the Turkish Cultural Foundation, Cultural Heritage Preservation and Natural Dyes Laboratory. The following standard dyes have been used as references: alizarin from Carl Roth (Germany); rubiadin, and purpurin, which were synthesised by the University of Jordan. Alum $[KAl(SO4)_2.12H_2O]$,

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***Corresponding author:** Karadag Recep, Laboratory for Natural Dyes, Faculty of Fine Arts, Marmara University, Istanbul, Turkey, Tel. +902163262667; Fax: +902163269129; E-mail: rkaradag@marmara.edu.tr

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hydrochloric acid and methyl alcohol were obtained from Merck (Germany).

The mordanting and dyeing of silk fabrics

Sixteen sample silk fabrics were cut, which had about 16×20 cm in size and 2 grams. These fabrics were washed at constant temperature (90°C) with 35% soapy water 100:1 ratio of the water for 2 hours. Before and after washing the fabrics, the pH of soapy water was measured. After the washing the fabrics were thoroughly rinsed with the deionised water and allowed to dry. Than except for one fabric, the other 15 samples were weighted separately for the mordanting process again.

Fifteen samples of fabrics, which had different percent of mordant, were mordanted a constant temperature of 65°C for 2 hours. After the mordanting process, the fabrics were waited in the mordant solution at room temperature for 24 hours. At the end of this period the fabrics that were mordanted and unmordanted were dyed at a constant temperature of 65°C with 100% madder (*Rubia tinctorum* L.) according to their weights by mixing for 2 hours.

After the dyeing process the fabrics were allowed to dry at the room temperature (25°C) after in order of washing at 25°C at 45-50°C and in finally 25°C in deionized water.

Extraction procedure for HPLC analysis

The extraction of according to the different mordant percent in the dyed silk fabrics with madder were performed with a solution mixture of% 37 HCl:MeOH:H_2O, (2:1:1; v:v:v) for 8 minutes at 100°C in open small tubes to extract dyestuffs. After cooling under running cold tap water, the solution was evaporated just to dryness in a water bath at 65°C under a gentle stream of nitrogen. The dry residue was dissolved in 200 µl of the mixture of MeOH:H_2O (2:1; v:v) and was centrifuged at 4000 rpm for 10 min. 50 to 100 µl supernatant was injected into the HPLC apparatus.

HPLC instrumentation

Chromatographic measurements were carried out using an Agilent 1200 series system (Agilent Technologies, Hewlett-Packard, Germany) including G1322A Degasser, G1311A Quat pump, G1329A autosample, G13166 TCC, and G1315D Diode Array Detector. PDA detection is performed by scanning from 191 to 799 nm with a resolution of 2 nm, and the chromatographic peaks were monitored at 255, 268, 276, 350, 491, 520, 580 and 620. Column: A Nova Pak C18 analytical column (39×150 mm, 4 µm, Part No WAT 086344, Waters) was used. Analytical and guard columns were maintained at 30°C and data station was the Agilent Chemstation. Two solvents were utilized for chromatographic separations of the hydrolysed samples. Solvent A: H_2O-0.1% TFA and solvent B: CH_3CN-0.1% TFA. The flow rate was 0.5 mL/min, and following elution program was applied (Table 1).

Time (min.)	Flow rate (ml/min)	H_2O-0,1% TFA (v/v)	CH_3CN-0,1% TFA (v/v)
0.0	0.5	95	5
1.0	0.5	95	5
20	0.5	70	30
25	0.5	40	60
28	0.5	40	60
33	0.5	5	95
35	0.5	5	95
40	0.5	95	5
45	0.5	95	5

Table 1: HPLC analysis is performed using the following gradient elution.

Trial No.	Percent of alum mordant	Identified dyestuff	Percent of d yestuff
1	0	alizarin	70.979
		purpurin	25.890
		rubiadin	3.130
2	1	alizarin	63.878
		purpurin	34.142
		rubiadin	1.981
3	2	alizarin	60.649
		purpurin	37.685
		rubiadin	1.666
4	3	alizarin	67.247
		purpurin	30.794
		rubiadin	1.959
5	4	alizarin	62.223
		purpurin	36.015
		rubiadin	1.762
6	6	alizarin	65.296
		purpurin	32.699
		rubiadin	2.005
7	9	alizarin	71.339
		purpurin	26.876
		rubiadin	1.785
8	12	alizarin	62.864
		purpurin	35.024
		rubiadin	2.112
9	14	alizarin	53.192
		purpurin	45.145
		rubiadin	1.664
10	18	alizarin	62.336
		purpurin	35.583
		rubiadin	2.081
11	21	alizarin	57.288
		purpurin	41.123
		rubiadin	1.590
12	24	alizarin	59.347
		purpurin	38.790
		rubiadin	1.862
13	27	alizarin	66.263
		purpurin	31.325
		rubiadin	2.411
14	30	alizarin	55.425
		purpurin	42.438
		rubiadin	2.137
15	33	alizarin	73.270
		purpurin	24.094
		rubiadin	2.636
16	36	alizarin	56.099
		purpurin	41.942
		rubiadin	1.959

Table 2: Variation semi quantities percent of alizarin, purpurin and rubiadin are in the dyed fabrics.

Colour measurements of silk fabrics

All colour measurements were performed using Minolta 3600D spectrophotometer (D65 illuminate, specular included, 10° observer angle). The spectrophotometer was equipped with software, which was able to automatically calculate CIEL*a*b* and colour strength (K/S)

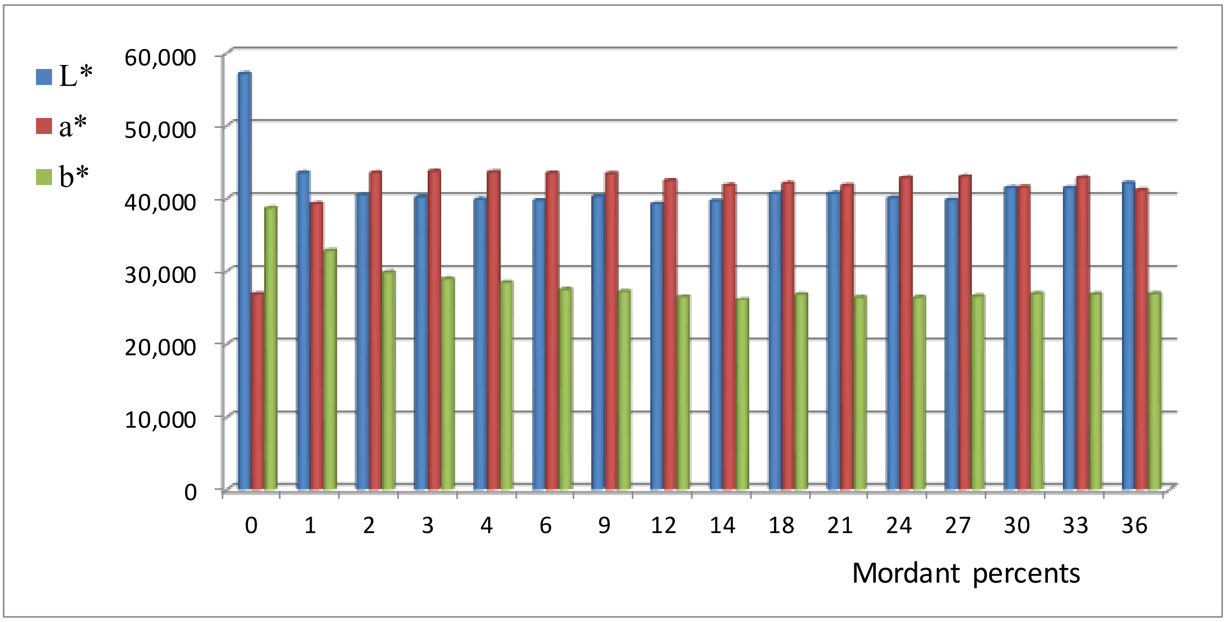

Figure 1: Colour Strength Values of Fabrics.

Trial No.	Mordant percentage (w/w)	L*	a*	b*	C*	H°
1	0	57.151	26.875	38.681	47.101	55.209
2	1	43.539	39.285	32.810	51.184	39.867
3	2	40.488	43.541	29.809	52.767	34.396
4	3	40.218	43.751	28.927	52.449	33.472
5	4	39.882	43.643	28.450	52.097	33.099
6	6	39.740	43.517	27.511	51.484	32.301
7	9	40.268	43.435	27.218	51.248	32.079
8	12	39.266	42.424	26.480	50.010	31.972
9	14	39.672	41.771	26.078	49.243	31.977
10	18	40.688	42.048	26.824	49.876	32.536
11	21	40.702	41.738	26.441	49.409	32.354
12	24	40.065	42.789	26.426	50.292	31.699
13	27	39.803	42.994	26.605	50.560	31.749
14	30	41.439	41.551	26.930	49.514	32.948
15	33	41.430	42.819	26.866	50.549	32.106
16	36	42.097	41.129	26.917	49.155	33.203

Table 3: Colorimetric values of fabrics.

values from the reflectance values at the appropriate-wavelength for each dyeing.

Colour strengths of fabrics were determined by using the Kubelka-Munk formula (Eq. 1), which is shown below.

$$K/S\ (1-R)^2/2R \qquad (1)$$

where,

K is the scattering coefficient

S is the absorption coefficient

R is the reflectance

Fastness measurements of silk fabrics

Washing fastness, light fastness and rubbing fastness of the dyed silk fabrics were performed according to ISO 105 C06, ISO 105 B02, ISO 105-X12 respectively [30-33]. The specific tests were applied by using the following instruments: Atlas Xenotest Alpha for light fastness, Atlas rubbing fastness tester and Atlas Linitest for washing fastness. The changes in shades and staining to adjacent multi fibres fabrics and rubbing fastness fabrics were related to the standard Grey Scale rating (where 1 is poor and 5 is excellent). The changes in shades under artificial light were evaluated according to standard blue wool fabrics (SDC) protocols. ECE non-phosphate standard detergent was used in washing fastness trials.

Results

Each dyed silk fabrics were analysed using a RP-HPLC-DAD. Quantities of colouring compounds were determined in the dyed silk fabrics, depending on the amounts of mordant metal (Table 2). Table 2 shows colorimetric values of samples. The darkest hue was obtained at 12% of mordant concentration. Redness of fabrics was increased by mordanting. Highest a* value (redness) was obtained at 3% mordant concentration. Vividness of fabrics was increased by adding mordant. The results were seen suitable with [17,23].

Colour strength values of fabrics are depicted in Figure 1. If the percent area of spectra of each dye is compared with the colour strength (Kubelka-Munk) values, a correlation between the purpurin concentration and colour strength appears. The color strength of the fabrics was increased with the increasing of purpurin concentration. However increase of concentration of alizarin yields decreasing of the colour strength values. This fact may be due to the amount of hydroxyl groups of both alizarin and purpurin. Alizarin has two hydroxyl groups and purpurin has three hydroxyl groups. Thus the alum can have more chance to bind with purpurin rather than the alizarin. Mordant addition increases the colour strength of the fabrics. The highest colour strength was obtained at 4% of mordant concentration.

Colorimetric values of fabrics are presented at Table 3. L* values were compatible with the colour strength values of the highest lightness and was observed in the case of non-mordant fabric due to the binding effect of mordant. Highest red colour was obtained in the case of 3%

Trial No.	Fastness to Light	Rubbing Fastness		Colour Fastness to Washing							Colour Fastness to Perspiration (Acidic)							Colour Fastness to Perspiration (Basic)						
				Colour Change	Bleeding						Colour Change	Bleeding						Colour Change	Bleeding					
		Dry	Wet		CA	Co	PES	PA	PAC	Wo		CA	Co	PES	PA	PAC	Wo		CA	Co	PES	PA	PAC	Wo
1	3-4	5	4	2-3	4	3-4	3-4	3	4-5	2-3	1-2	2	2-3	3-4	1-2	4	1-2	1-2	2	2	3	1-2	2-3	1
2	4	2	2	3	3	4	4	3	4-5	2-3	3-4	2-3	2-3	4	1-2	4	1-2	3	2	2	3	1-2	2-3	1
3	5	3-4	3-4	4	3	4	4	3-4	4-5	3	4	3	3	4	2-3	4	2-3	3	2-3	2-3	3-4	2	3-4	2-3
4	4-5	4	3-4	4-5	4	4	4-5	3-4	4-5	3	4-5	3-4	3-4	4-5	2-3	4-5	3	4	2-3	2-3	4	2	4	2-3
5	5	4-5	4	4-5	4	4	4-5	4	5	4	4-5	3-4	3-4	4-5	3	4-5	3-4	4-5	2-3	2-3	4	2	4	2-3
6	6	4-5	4-5	4-5	4	4-5	4-5	4	5	4	4-5	3-4	4	4-5	3	4-5	3-4	4-5	2-3	2-3	4	2	4	2-3
7	5-6	4-5	4-5	4-5	4	4-5	4-5	4	5	4	4-5	3-4	4	4-5	3	4-5	3-4	4-5	2-3	2-3	4	2	4	2-3
8	5-6	4-5	4-5	4-5	4	4-5	4-5	4	5	4	4-5	3-4	4	4-5	3	4-5	3-4	4-5	2-3	2-3	4	2	4	2-3
9	5-6	4-5	4	4-5	4	4-5	4-5	4	5	4	4-5	3-4	4	4-5	3	4-5	3-4	4-5	2-3	2-3	4	2	4	2-3
10	5-6	4-5	4-5	4-5	4	4-5	4-5	4	5	4	4-5	3-4	4	4-5	3	4-5	3-4	4-5	2-3	2-3	4	2	4	2-3
11	5-6	4-5	4-5	4-5	4	4-5	4-5	4	5	4	4-5	4	4	4-5	3	4-5	3-4	4-5	2-3	2-3	3-4	2	3-4	2-3
12	5-6	4-5	4-5	4-5	4	4-5	4-5	4	5	4	4-5	3-4	4	4-5	3	4-5	3-4	4-5	2-3	2-3	4	2	4	2-3
13	5-6	4-5	4	4-5	4	4-5	4-5	4	5	4	4-5	3-4	4	4-5	3	4-5	3-4	4-5	2-3	2-3	4	2	4	2-3
14	5	4-5	4	4-5	4	4-5	4-5	3-4	5	3-4	4-5	3	2-3	4-5	2-3	4-5	3	4-5	2-3	2-3	3-4	2	3-4	2-3
15	5-6	4-5	4-5	4-5	4	4-5	4-5	3-4	5	3-4	4-5	3	3	4-5	3	4-5	3-4	4-5	3	3	4	3	4	3
16	5-6	4-5	4-5	4-5	4	4-5	4-5	3-4	5	3-4	4-5	3	3	4-5	3	4-5	3	4-5	3	3	4	3	4	3

PA: Polyamide, WO: Wool, PAC: Polyacrylic, PES: Polyester, CO: Cotton, CA: Cellulose.

Table 4: Fastness Properties of Fabrics.

mordant concentration. However no significant variation was observed between the concentration levels of mordant. As a general statement alizarin and purpurin have a red colour. Variations at the amounts of alizarin and purpurin did not affect the redness of the fabrics. However yellowness of the fabrics was affected by rubiadin concentration. Decreasing of concentration of rubiadin yielded the decreasing of b* values of fabrics. The highest b* value was observed at non-mordant dyeing.

Fastness properties of fabrics are listed at Table 4. Wet fastness properties are related to binding mechanism of dye into fibre. Thus, addition of mordant gave higher fastness results than the non-mordant dyeing. Washing fastness results increased with increasing of mordant concentration. However poor results were observed in the case of basic perspiration fastness tests. This fact can be due to the exceed hydroxyl ions in the perspiration solution. When compared with basic perspiration fastness test results, relatively better results were observed for acidic perspiration fastness tests. According to the all fastness results, the best result is dyed fabric with 6% alum mordant.

This study shows that the colour strength and colorimetric values of dyed fabrics are strongly affected by colouring components of natural dye solution. Colour strength values increased by increasing of amount of alizarin. Yellowness (b*) of the fabrics were changed by amount of rubiadin. Future studies in this field should focus on the role of the each component of the natural dyes. Affinity and dye uptake of each component of natural dyes should be studied.

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