

Dyeing efficiency of *Lawsonia inermis* (Henna) and ferrous sulphate on silk fibre

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This study evaluates the dyeing efficiency of *Lawsonia inermis* (Henna) at varying concentrations on silk fibres using 3% ferrous sulfate as a mordant. Preliminary trials suggest 3% of ferrous sulphate as the optimal mordant concentration, while henna dye percentages of 10%, 15% and 20% are assessed. The dyed samples are analyzed for absorbance, colour coordinates, colour strength and fastness properties. Results indicate that 15% henna dye, along with 3% ferrous sulphate, achieves better dyeing performance on silk fibres.

Keywords: Dye fastness, Henna dye, Mordant, Natural dyes, Silk fibre

1 Introduction

Natural dyes are colouring materials extracted from natural sources, used for the colouration of various textile materials¹. Many studies worldwide have investigated the extraction, optimization and application of natural dyes to overcome the limitations associated with synthetic dyes in the textile industry². Natural dyes have been used since ancient times for dyeing carpets, rugs, and garments, with sources ranging from roots, stems, barks, leaves, berries, and flowers³. Henna is well-known for its chief constituent, lawsone or hennotannic acid, a red-orange pigment found in its leaves⁴. Dyeing of textile materials with natural colourants is a promising area that needs to be explored systematically and scientifically to produce diversified value-added products.

Most natural dyes are substantial dyes that need mordants to keep colour from fading off the yarn due to washing and exposure to light⁵. Mordants increase the fastness properties by forming an insoluble complex of the dye and the mordant within the fibres, thereby improving colour stability⁶. For the successful commercial use of natural dyes, it is essential to standardize scientific techniques for optimal mordant and dyeing concentrations. Thus, the present study is designed to optimize the concentration of henna dye

(10%, 15% and 20%) for silk fibre dyeing using 3% ferrous sulphate as the mordant.

2 Materials and Methods

2.1 Materials

Silk fibre was procured from the College of Temperate Sericulture, SKUAST-K. Dried henna leaves were obtained from the local market in Srinagar. The mordant and all other chemicals used were of laboratory grade. The shade-dried henna leaves were washed thoroughly with water to remove dirt and dried in a tray dryer at 80°C for two hours before being grounded into a fine powder. The latter was passed through a BSS-14 standard test sieve and stored for further studies. Different concentrations of henna dye (10%, 15% and 20%) were screened for optimization.

2.2 Methods

2.2.1 Scouring

The silk fibre was scoured using 2% non-ionic soap (Labolene) at 50 °C for 20 min with a material-to-liquor ratio of 1:50. The scoured fibre was rinsed thoroughly with tap water and air-dried. Before dyeing, the scoured material was soaked in distilled water for 30 min.

2.2.2 Extract Preparation

Henna leaf colourants were extracted in an alkaline medium following a modified version of the technique

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described by Yusuf⁷. Powdered henna leaves (10%, 15%, and 20% on weight of fibre (owf)) were added to an aqueous solution of Na₂CO₃ with a material to liquor (M:L) ratio of 1:20. The mixture was heated at 80-85 °C for 1h with periodic stirring. After cooling, the mixture was filtered through a clean cotton cloth and centrifuged at 10000 rpm for 10 min at 25°C. The resulting reddish orange filtrate was made neutral (pH 7) using HCl and used for dyeing silk fibres.

2.2.3 Dyeing of Silk Fibres

Dyeing was conducted using 10%, 15%, and 20% owf of henna dye concentrations as per the method of Mohammad⁸. An M:L ratio of 1:40 was maintained. Simultaneous mordanting was carried out using 3% ferrous sulfate. The dye bath, containing silk fibres, distilled water, extracted liquid dye, and mordant, was heated to 93 °C and maintained for 1h with regular stirring to ensure uniform dyeing. The dyed samples were washed with 0.5% Labolene (non-ionic detergent), rinsed with tap water, and air-dried in the shade at 25°C.

2.2.4 Percent Absorption of Dye

The per cent absorption of the dye was calculated by recording the optical density of the dye solution before and after the dyeing process using a UV-visible absorption spectrophotometer at 560 nm. It was calculated using the following equation⁹.

Percent absorption =

$$\frac{\text{O.D. before dyeing} - \text{O.D. after dyeing}}{\text{O.D. before dyeing}} \times 100$$

2.2.5 Colour Analysis

The CIE colour coordinates (L*, a*, b*, C*, h°, ΔL, Δa, Δb and ΔE) and colour strength (K/S values) were determined using a computer colour matching system.

2.2.6 Fastness Properties

The colour fastness is usually rated either by loss of depth of colour in the original sample or is expressed by staining scale. The fastness rating was assessed using the grey scale as per ISO-105-A02 (loss of shade depth) and ISO-105-A03 (extent of staining). The light fastness was evaluated as per the

ISO 105-B02 method. Samples of the dyed material were exposed simultaneously to a light source of Xenon arc lamp using a Xenon test chamber (Model Xe-1-S, Q-SUN) under specified conditions for 10 h. The rubbing fastness (dry and wet) was assessed as per ISO-105-X12 method using a manually operated PROLIFIC Crock Meter with the finger of 1.6 cm diameter moving to and fro in a straight line over a 10±0.30cm track on the specimen, with a downward force of 9 N. The washing fastness was carried out as per the ISO 105-C01 method.

3 Results and Discussion

3.1 Optimization of Henna Dye Concentrations with 3% Ferrous Sulphate as Mordant

3.1.1 Percent Absorption of Silk Fibre

The highest dye absorption (91.7%) is observed with 15% henna dye, followed by 10% (91.0%) and 20% (90.6%), using 3% ferrous sulfate as a mordant (Table 1). Beyond a certain concentration, the yarn reaches equilibrium, limiting further dye uptake. It aligns with earlier findings that dye uptake plateaus at higher concentrations¹⁰.

3.1.2 Colour Coordinates

It is observed that increasing dye concentration reduces the lightness (L*), with values decreasing from 48.836 (10%) to 46.292 (15% and 20%). The highest ΔL is recorded in 20% (-57.937), followed by 10% (-47.921) and 15% (-47.449), respectively (Table 1). Negative ΔL values indicate darker shades. These results are in agreement with the previous study⁸. The a* values are highest for 15% dye (4.551), followed by 10% (4.489) and 20% (4.141). The highest Δa is recorded in 15% (2.582), followed by 10% (2.499) and 20% (2.472). Similarly, b* values peak at 15% (19.801), 10% (18.978) and 20% (18.236). The highest Δb is recorded in 15% (2.700), followed by 10% (2.506) and 20% (2.464). All henna-dyed samples are seen in the red-yellow zone. The C* values are highest for 15% henna dye (19.766), followed by 10% (19.227) and 20% (18.999). The h° values follow a similar trend, with 15% showing the highest at 78.095 followed by 10% (76.635) and 20% (75.095). Total colour change (ΔE) is highest at 15%

Table 1 — Colour coordinate values of silk fibre dyed with different concentrations of henna using 3% ferrous sulphate as mordant

Dye conc. (%)	Dye absorption	L*	a*	b*	C	h°	ΔL	Δa	Δb	ΔE	K/S
10	91.0	48.836	4.489	18.978	19.227	76.635	-47.921	2.499	2.506	47.621	2.571
15	91.7	46.292	4.551	19.801	19.766	78.095	-47.449	2.582	2.700	48.099	3.637
20	90.6	46.292	4.141	18.236	18.999	75.097	-57.937	2.472	2.464	48.011	3.635

(48.099), followed by 20% (48.011) and 10% (47.621) (Table 1).

3.1.3 Colour Strength (K/S)

The colour strength of henna-dyed silk fibres increases with the increase in the dye concentration. The maximum K/S value is observed at 15% (3.637) followed by 20% (3.635) and 10% (2.571). Higher colour strengths are seen in shades obtained with higher dye concentration. These results are in conformity with Yusuf⁷ who reported higher k/s value with higher henna dye concentrations in dyed woolen yarn samples. Mohammad⁸ also reported similar results with henna dye on woolen yarn samples.

3.1.4 Colour Fastness Properties

The overall fastness properties i.e., fastness to washing (fading and staining), lightness and rubbing (dry and wet) of all samples are presented in Table 2. Washing fastness achieves grade 5 for color change and staining at 10% and 15% dye concentrations. Light fastness grades are similarly excellent at 10% and 15%. Dry fading records excellent (5) grade across all concentrations, while wet rub fastness is highest at 15% and 20%. The formation of dye-mordant complexes enhances higher fastness grades of the dyes and mordants when applied on the fabric¹¹. The washing fastness of the dye is influenced by the rate of the diffusion of the dye and state of the dye inside the fibre¹². Good light fastness is due to the formation of complex with the mordant which protects chromatophore from photolytic degradation¹³. The higher grades of the wet rub fastness than dry rub fastness may be due to dissolving of water-soluble dye molecules which make them easier to be removed from the fibre by rubbing¹⁴.

4 Conclusion

The study demonstrates the effective use of henna dye (*Lawsonia inermis*) for silk fibre dyeing, with 3% ferrous sulfate as a mordant. Among the tested concentrations, 15% henna dye achieves optimal results, exhibiting the highest dye absorption (91.7%),

Table 2 — Effect of different concentrations of henna on colour fastness properties of silk fibre

Dye conc, %	Washing		Light	Rubbing	
	Fading	Staining		Dry	Wet
10	5	4/5	5	5	4/5
15	5	5	5	5	5
20	4/5	4/5	4/5	5	5

superior color strength ($K/S = 3.637$), and enhanced brightness and chroma values. Color fastness properties, including washing, light, and rubbing fastness, are excellent at 10% and 15%, highlighting the stability of the dye-mordant complexes. The findings suggest that 15% henna dye, combined with 3% ferrous sulfate, is ideal for achieving vibrant, durable, and high-quality coloration on silk fibres. This optimized protocol can serve as a potential alternative to harmful synthetic dyes for dyeing of silk fibre and holds promise for sustainable dyeing practices in the textile industry.

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