Effect of Dyebath pH on Colour Characterization of Silk Fabric Dyed with Natural Dyes Using Cold-Pad-Batch Dyeing Technique

Priyank Patel[1] Devang Panchal[2] Bharat Patel[3]
[1,2,3]Department of Textile Chemistry
[1,2,3]Faculty of Technology & Engineering, The Maharaja Sayajirao University of Baroda, Vadodara, India

Abstract — Silk is considered as Queen of textile fiber owing to its smooth and lustrous appearance. Traditionally it can be dyed with synthetic as well as natural dyes to impart a variety of colours. In recent years finding of researches conducted shown that chemicals used in synthetic dyes may contain toxic elements. These dyes may cause an environmental problem as well as adversely affect the health of living beings. To overcome the adverse effect of synthetic dyes researchers all over the world have been focused on substitute preparation of synthetic dyes. Dyes of natural origin are environment-friendly in nature and have no side effects on the skin. Natural colorants are often criticized for limitations in the available colour range. The present study deals with the application of two natural dye extracts from Madder and Kamala by a Cold–pad-batch technique of dyeing. The effect of different auxiliaries on the dyeing has also been investigated by selecting two mordents applied by the meta-mordanting technique. Commercially available natural dyes have been applied with four different formulations to produce a variety of shades. Shades of pink to dark red have been produced using Madder and Shades of Cream to dull yellow have been produced by Kamala as a natural dye. The dyeing characterization of silk dyed with Madder and Kamala using the Cold-Pad-Batch technique has been done in terms of colour strength value (K/S) as well as the overall colour fastness properties. The samples dyed with energy-efficient Cold-Pad-Batch technique give different shades on silk fabric using natural dyes. The different formulations of dye liquor result in a change in shades of dyed fabric with satisfactory light, wash, and rubbing fastness properties.

Keywords: Natural Dye, Cold-Pad-Batch Dyeing, Mordents, Colour Fastness, Silk, Madder, Kamala

I. INTRODUCTION

Silk, called the 'Queen of fibres', is a continuous protein fibre produced by the silkworm to form its cocoon [1]. Silk is dyed using various dyes such as Acid dyes, metal-complex, reactive dyes, etc. acid dyes are more suitable for silk and wool, and the direct and vat dyes are more suitable for cotton and so on. Dyeing and printing make it possible to bring about the choicest colours and shades on silk leading to the value addition of the silk materials. Silk offers a wide colouration possibility covering almost the entire spectrum of colours and hues due to its ready acceptability for a large range of dyestuffs. The exceptional capacity to absorb moisture from the air, the comparatively simple and orderly arrangement of fibroin molecular structure, and the abundance of hydrogen bonds and electrostatic bonds render silk fibre an ideally suited substrate with a very good dye affinity. Anionic dyestuffs namely acid and direct dyes form a 'Dye-Fibre' complex by electrostatic and hydrogen bonds. Silk can also be dyed with basic, metal-complex, and reactive dyes. Acid dyes are widely used for the dyeing of silk. Using this class of dyestuff, a wide range of bright shades can be obtained. These dyes are sodium salts of organic acids (mostly sulphonic acid) and are applied from an acidic medium.[2]

Recently, due to environmental awareness throughout the world, much work has been reported on the dyeing of silk with natural dyes [3,4]. Literature is also available on the dyeing of silk with *Madder* and *Kamala* as natural dyes [5,6]. *Madder* is one of the oldest dyestuffs known. It is most frequently used to produce turkey reds, mulberry, orange-red, terracotta, and in combination with other dyes and dyeing, procedures can yield crimson, purple, rust, browns, and near black. The primary dye components are alizarin and munjistin which are found in the roots. Madder is a complex dyestuff containing many colourants. By manipulating mordanting, pH, and temperature a range of shades can be obtained. There are many historic recipes including one from Turkey which brings out the purpurin from madder resulting in a purple [7]. *Kamala* – is a powdery substance obtained from the fruit of Mallotus philippinensis, a small evergreen that is also known as the monkey-face tree (because monkeys are said to rub their faces in the fruit). Kamala is found throughout tropical India. Kamala dye is very similar in behavior and colour to annatto. Kamala dyes golden yellows to tangerines with moderate lightfastness on cotton. Lightly deeper shades are obtained on protein fibres. When iron is added deep moss green is obtained. Over or under dyed with indigo produces forest greens [8].

In earlier research, we have reported the effect of natural dyes on the antimicrobial and functional properties of textiles [9,10]. In the present investigation, an attempt has been made to explore energy-efficient methods to produce a variety of shades on silk fabric. The silk fabric has been dyed with two natural dyes, namely, Madder and Kamala with different formulations by the cold-pad-batch technique of dyeing. Two mordants viz., alum and potassium dichromate has been used. The dyed samples have been evaluated in terms of change in colour strength value and fastness properties using standard testing methods.

II. MATERIAL

Silk fabric: EPI =128 & PPI = 105, GSM = 43 g/m^{2}was obtained from Khadigram udyog, kothi char rasta, Baroda, Gujarat, India for the study.

A. Dyes and chemicals

Two commercially available natural dyes namely, Madder and Kamala were procured from the Ahmedabad dye market and were used without further purification. Metal salts like Alum (Al_2SO_4, $2H_2O$) and Potassium dichromate ($K_2Cr_2O_7$) were used along with caustic soda(NaOH), acetic acid(CH_3COOH), soda ash (Na_2 CO_3) from Suvithinath laboratories, India, and non-ionic detergent.

B. Methods

1) Dyeing of silk fabric

Dyeing of silk samples was carried out with two natural dyes on laboratory two bowl padding mangle. Padding liquors for Madder and Kamala were prepared at an individual concentration of 50 gpl dye, then immediately passed through two bowl padding mangle by 2-dip 2-nip padding technique using 65% padding expression. (12-16 hr for Batching)

Here Shade was obtained by padding the silk fabric with different dye liquors in succession for example dipping and padding a cloth into different 5 dyebaths, FI, FII, FIII, FIV, FV Contain Madder, and Kamala respectively.

Cold pad batch dyeing is closely related to continuous dyeing but it only requires the use of a padding mangle and a means of winding fabric onto a roll. The fabric is dipped in a trough containing different 5 formulations, FI, FII, FIII, FIV, FV, as follows;

- FI- 50 gpl Madder + 10 gpl Alum / 10 gpl $K_2Cr_2O_7$ + 10 gpl Citric acid
- FII - 50 gpl Madder + 10 gpl Alum / 10 gpl $K_2Cr_2O_7$ + 10 gpl Citric acid + 10 mlpl Methanol
- FIII - 50 gpl Madder + 10 gpl Alum / 10 gpl $K_2Cr_2O_7$ + 10 gpl Sodium carbonate
- FIV - 50 gpl Madder + 10 gpl Alum / 10 gpl $K_2Cr_2O_7$ + 5 gpl Sodium hydroxide
- FV - 50 gpl Madder + 10 gpl Alum / 10 gpl $K_2Cr_2O_7$ + 2 gpl Sodium hydroxide + 2 gpl Sodium hydro sulphite

The fabric was mangled and then wound on a roll without drying it. The roll is covered in plastic sheets to stop the fabric drying and the batch is slowly rotated for 24 hours to allow the dye to fix at room temperature. After dyeing the fabrics can be washed off using batch processes.

C. Testing and analysis

1) Colour measurement

The dyed samples were assessed for *L* a* b** colour coordinates and *K/S* values (illuminant D65 /10^0 observer) on spectra scan 5100 (RT), spectrophotometer (premier colourscan instrument)

D. Fastness determination

1) Fastness to light

Colour fastness to light was evaluated by exposing the dyed samples to sunlight for 12 hours in a normal manner (According to AATCC test method 16-B- 1977) to see the effect of fading of colour due to sunlight. Lightfastness was evaluated by comparison of the exposed portion with the unexposed portion of the material. They were graded from 1 to 8 (1 means poor and 8 means excellent fastness to light).

2) Fastness to Washing:

ISO standard test no. III was used for the determination of the washing fastness of dyed fabrics. This was done on Launder – O – meter where a fabric sample of 5 X 2 cm^2 was placed between two adjacent undyed fabrics of the same size. They were sewn from all four sides to form a composite specimen. The composite specimen was placed in a glass jar containing 5 gpl soap solution and 2 gpl soda ash solution, keeping material to liquor ratio 1:50. Jars were then closed and placed in Launder – O – meter. The machine was then run for 30 minutes at 60 ± 2^0 C. After completion of treatment, the samples were removed and washed with water, squeezed, and dried in air. By using Greyscale the change in the shade was assessed and graded from 1 to 5 (1 means poor and 5 means excellent fastness to washing.

3) Fastness to rubbing

A device for the rubbing test has a finger having a diameter in the range of 1-6 cm moving to and fro in a straight line covering a 10 cm track on the specimen with a forward force of 6N; done on a crock-meter. The rubbing cloth against which the specimens were tested of dyed silk cloth cut into 5X5 cm^2 size. Piece of material to be tested were prepared in two pairs of pieces, one for the dry rubbing and the other for the wet rubbing test. In both cases, it travels 10 times in 10 seconds along the track. In the wet rubbing test, the cloth has been wetted and squeezed until it contains its weight of water. The staining of the rubbing cloth was assessed with greyscale and graded from 1 to 5 (1 means poor and 5 means excellent fastness to rubbing). Rubbing fastness (both wet and dry) on crock meter (BS1006; NOX12; 1978).

III. Results and Discussion

Silk fabric was dyed with three individuals as well as with the combination of the dyes at 50 gpl concentration level by a hot padding method. The results in terms of *L* a* b** and *K/S* values are presented in Table-1 where *L** represents the lightness or darkness of the samples, the variation in *a** values shows the redness of dyed samples, while *b** is indicative of the yellowness of the sample.

Dye	Formulation	*K/S* values	*L**	*a**	*b**	C*	H*
Madder	FI	16.491	30.892	8.703	12.732	15.422	55.623
	FII	1.356	18.097	4.844	2.045	5.258	22.879
	FIII	8.512	17.530	9.614	0.938	9.660	5.570
	FIV	2.653	18.248	7.300	2.461	7.704	18.623
	FV	0.433	21.071	2.168	4.583	5.070	64.657
Kamala	FI	4.798	32.365	7.822	14.449	16.430	61.546
	FII	3.658	31.397	7.530	13.398	15.369	60.639
	FIII	1.914	27.113	5.943	9.752	11.420	58.618
	FIV	3.912	30.827	8.650	13.092	15.691	56.524
	FV	2.860	34.316	10.281	15.602	18.685	56.594

Table 1: Colours developed as a result of dipping in individual and combined dyebathsusing alum

FI- 50 gpl Madder + 10 gpl Alum / 10 gpl $K_2Cr_2O_7$ + 10 gpl Citric acid; FII - 50 gpl Madder + 10 gpl Alum / 10 gpl $K_2Cr_2O_7$ + 10 gpl Citric acid + 10 mlpl Methanol; FIII - 50 gpl Madder + 10 gpl Alum / 10 gpl $K_2Cr_2O_7$ + 10 gpl Sodium carbonate; FIV - 50 gpl Madder + 10 gpl Alum / 10 gpl $K_2Cr_2O_7$ + 5 gpl Sodium hydroxide; FV - 50 gpl Madder + 10 gpl Alum / 10 gpl $K_2Cr_2O_7$ + 2 gpl Sodium hydroxide + 2 gpl Sodium hydro sulphite

Dye	Formulation	*K/S* values	*L**	*a**	*b**	C*	H*
Madder	FI	9.458	34.828	8.084	12.272	14.695	56.603
	FII	11.652	35.975	12.799	13.740	18.778	47.012
	FIII	6.505	23.887	8.867	2.041	9.099	12.957
	FIV	2.695	21.317	9.505	0.104	9.506	0.627
	FV	3.489	20.804	16.003	-0.619	16.015	357.786
Kamala	FI	4.900	41.540	10.473	18.223	21.018	60.089
	FII	8.029	47.242	9.624	21.611	23.657	65.969
	FIII	2.734	40.692	7.575	17.060	18.666	66.031
	FIV	3.199	43.356	9.299	19.062	21.209	63.970
	FV	3.024	41.954	10.534	17.983	20.841	59.615

Table 2: Colours developed as a result of dipping in individual and combined dye baths using $K_2Cr_2O_7$

FI- 50 gpl Madder + 10 gpl Alum / 10 gpl $K_2Cr_2O_7$ + 10 gpl Citric acid; FII - 50 gpl Madder + 10 gpl Alum / 10 gpl $K_2Cr_2O_7$ + 10 gpl Citric acid + 10 mlpl Methanol; FIII - 50 gpl Madder + 10 gpl Alum / 10 gpl $K_2Cr_2O_7$ + 10 gpl Sodium carbonate; FIV - 50 gpl Madder + 10 gpl Alum / 10 gpl $K_2Cr_2O_7$ + 5 gpl Sodium hydroxide; FV - 50 gpl Madder + 10 gpl Alum / 10 gpl $K_2Cr_2O_7$ + 2 gpl Sodium hydroxide + 2 gpl Sodium hydro sulphite

From the results, it can be seen that a variety of shades viz, yellow, red, blue, orange, and purple can be produced on silk fabric using two natural dyes namely, Madder and Kamala using the cold-pad-batch process. It can also be seen from Figure 3 that dipping silk fabric into individual or several different dyebaths various shades can be produced on silk fabric by the use of mordant and also by using different formulations.

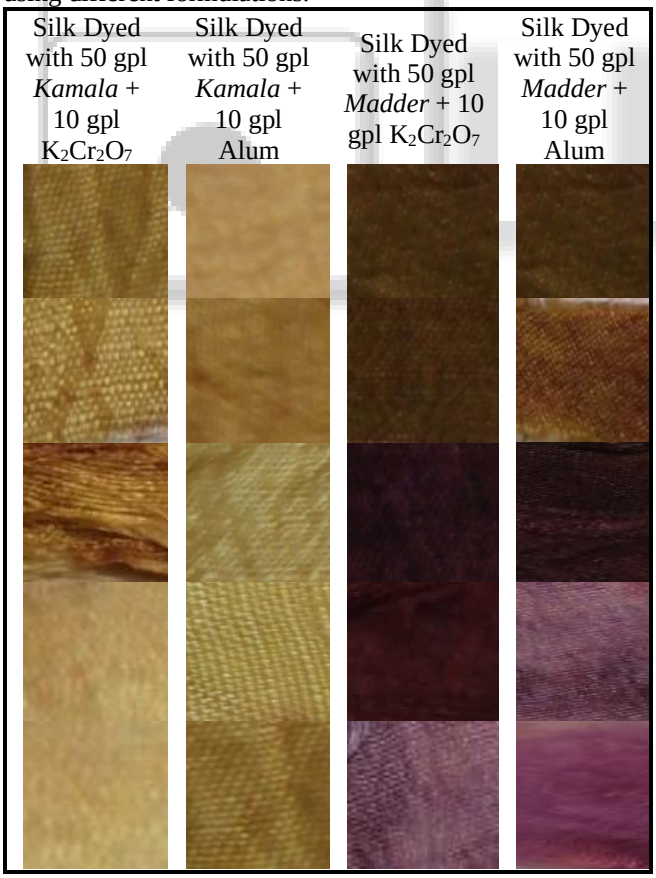

Fig. 1: Various shades produced by different formulations of Cold-Pad-Batch liquor using natural dyes on silk fabric

Fastness to light, washing and rubbing of samples dyed individual mordants and or with different formulations are given in Table 3. From the table, it can be seen that all samples dyed either individually using different formulations with Madder or Kamala dye or with the combination of mordants, exhibit adequate all-around fastness properties.

Dye	Formu -lation	LF	WF			RF	
			SS	SC	CC	SD	SW
Madder	FI	5-6	4	4/5	4	3	3
	FII	6	4	4	4/5	3	3
	FIII	6-7	3-4	4	4	3-4	3-4
	FIV	6-7	3-4	4	3-4	3-4	3-4
	FV	6-7	3-4	4	4	4	3-4
Kamala	FI	6	4	4	4	3	3
	FII	6-7	4	4	4	4	3-4
	FIII	6-7	4	4	4	4	4
	FIV	6-7	4	4	4	4	4
	FV	6-7	4	4	4	4	4

Note: WF is washing fastness, RF is rubbing fastness, SS is Stain on silk, SC is Stain on cotton, CC is Colour change, SD is Stain on dry cotton and SW is a stain on wet cotton.

Table 4: Fastness ratings of dyed samples

IV. Conclusion

Silk fabric can be dyed successfully with Kamala and Madder. Various shades of beautiful yellow and red can be produced by the cold-pad-batch padding technique. The use of Alum and $K_2Cr_2O_7$ as mordents further increase the colour range with minor improvement in fastness properties. The most significant part of the study is that the hot padding method, where the requirement of energy is too low.

Cold pad batch dyeing can save energy and water for the whole process as there is no need to apply heat and this is a low water dyeing process. So, the process can be considered as cost-effective and best suited for small-scale industries or cottage dyeing.

References

[1] Agarwal BJ, Patel BH. Reincarnation of natural colourants. *Chem. Weekly* 2000;45: 139-146.
[2] Patel BH. Natural Dyes, Handbook of textile and industrial dyeing: Principles, processes and types of dyes; Volume 1. Cambridge, UK: Woodhead Publishing Ltd; 2011. p 395 - 421.
[3] Agarwal BJ, Patel BH, Patel HM. Dyeing of nylon with babool bark extract. *Textile Asia* 2002; 33: 34- 42.

[4] Patel BH, Patel PB. Dyeing of polyurethane fibre with *Ocimum sanctum. Indian Journal of Textile Research* 2006; 31: 474-476.
[5] Patel KJ, Patel BH, Naik JA, Bhavsar AM. Eco- friendly dyeing with Tulsi leave extract, *Man-made Textiles in India*, 2002;45(11):420- 425.
[6] Patel BH, Agarwal BJ, Patel PG.Dyeing of silk with babool, *Indian silk*, 2003;42(4):17-21.
[7] Patel BH, Bhatia KB, Parekh UD. Environmental-friendly & cost-effective method to create various shades on silk. *Indian silk*, 2005; 44 (6):24-25.
[8] Patel BH. Expanding the range of colors on silk fabric with natural dyes. *Asian Dyer* 2012;9:50-52.
[9] Chattopadhyay DP, Patel BH, Functional properties of silver nano-sol treated wool and silk fabric. *Man-made Textiles in India* 2014; 42 (9):340-344.
[10] Patel BH, Tandel MG, Antimicrobial finish to cotton fabric by natural extracts. *Man-made Textiles in India* 2005;48 (12): 473-478.

