Modified acid colour dyeing of silk for energy preservation

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***Abstract*—Textile wet processing, particularly dyeing, consumes considerable amounts of water and thermal energy. In the recent times, both water and thermal energy are rapidly becoming more and more expensive and therefore there is a need to conserve them for better tomorrow. Various modifications in the dyeing system have been adopted to make the energy conservation feasible. Performing the dyeing process at lower dyeing temperature and for short duration can lead to the conservation of energy. This can be done by the utilization of an appropriate solvent for physical modification of the substrate and subsequently dyeing it at lower temperatures and for shorter duration in order to conserve energy. The solvent used for the pretreatment of silk substrate is acetophenone. Commercial anionic acid dyes have been applied on the solvent pretreated silk substrates at different dyeing temperatures, ranging from room temperature to 70º C and the results compared with conventionally dyed samples, in terms of colour strength values and fastness properties of the dyed samples. The solvent treatment not only aids in reducing the dyeing time and temperature but also retains the aesthetic values associated with silk fibre**

Keywords— solvent, silk, acid dyes, fastness characteristics, energy conservation

I. INTRODUCTION

The dyeing process is regarded as one of the most important parts of textile wet processing, which utilizes very large amounts of energy. In general, dyeing involves adsorption of dye molecules/ions on the fibre surface from the solution phase (i.e. dyebath), followed by the diffusion of the adsorbed species into the fibre substance, and finally interaction of these species into the fibre substance. These processes are influenced by controlled conditions of pH, temperature, dye concentration, presence of dyeing assistants (viz. leveling or exhausting agents), liquor ratio, etc. Temperature plays a very key role in the economics of the dyeing process [1 – 3]. Hence, one of the main objectives of a successful dyer is to lower the temperature of dyeing in order to conserve energy. This can be achieved by various methods, viz. graft polymerization, redox system, solvent dyeing etc. [4 – 13].

Since time immemorial, man has been marveled by the beauty of silk owing to its scientific, technological and aesthetic values. . It is a non-keratin type of protein filament fibre. The cocoon of a silkworm contains about 360 to 1200 meters of continuous twin filament [14], joined together by silk gum sericin. Silk has always been regarded as the "Queen of textile fibres"; the luster, handle and the draping qualities of silk are superior to those of many other textile fibres. The natural luster and smoothness, possessed by silk fibre, is something unique, which is usually not observed in any natural textile fibre [15]. The conventional dyeing of silk by exhaust dyeing method, with commercial acid, basic, reactive and other dyes, is usually performed at near boiling temperature, which substantially damages the silk fibre, loses its magnificent luster and deteriorates its qualities [16, 17]. Therefore, low-temperature dyeing is an attractive approach in order to decrease this potential damage.

In this work, an effort has been made to dye Tasar silk fabric with commercial metal-complex acid dyes at lower dyeing temperatures in order to preserve its luster and other qualities, which give an aesthetic appeal to silk. The silk substrate was pretreated with acetophenone solvent and subsequently dyed with metal-complex acid dyes at different temperatures for varying lengths of time. The physic-mechanical changes in the fibre were evaluated by various testing and analysis methods, viz. tensile strength, shrinkage behavior and weight analysis. The colour strengths (in terms of K/S values) of such dyed samples were measured spectrophotometrically and the results were compared with those of conventionally exhaust dyed samples. Fastness characteristics of various dyed samples were also evaluated and compared with each other.

II. MATERIALS & EXPERIMENTAL METHODS

A. *Materials*

(1) Fabric : Plain weave Tasar silk fabric (65 gm/sq. m.) having 80 ends/inch and 60 picks/inch was used for the present investigation. The grey fabric was procured from the Khadi Gram Udhyog, Vadodara. For experimental work, silk fabric was cut in a square dimension, which exactly weighed 1 gram on electronic balance. Before dyeing, the fabric was first degummed (scoured) to remove the natural gum sericin and other natural impurities so as to ensure uniform application of the colour. The degumming bath was prepared with

5 gpl	Soap solution
0.5 gpl	Soda ash

using liquor ratio of 50:1. The treatment was given in the above bath at 90° - 95° C for 1 hour. The fabric was then thoroughly washed with hot and cold water and air-dried.

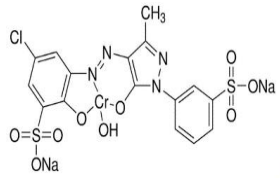
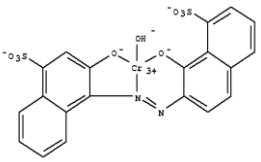
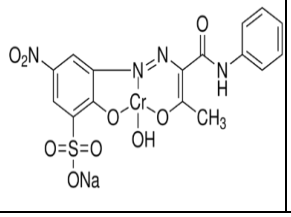
After scouring, the fabric was bleached to improve its whiteness. Degummed silk was treated with a solution containing

10 gpl	Hydrogen peroxide (30 volume)
2 gpl	Sodium silicate
5 gpl	Soda ash

using liquor ratio of 50:1. The bleaching process was continued for 60 minutes at 80 ± 2° C. After bleaching, the fabric was thoroughly washed with cold water and treated with 0.01 N hydrochloric acid to neutralize the residual alkali present in the fabric. Finally, the fabric was washed thoroughly to free it from the acid and dried at ambient temperature.

(2) Dyestuffs: Three commercial 1:1 metal-complex acid dyes, namely were selected for the present study. Various important characteristics of these dyes are incorporated in Table I.

TABLE I. VARIOUS SPECIFICATIONS OF 1:1 METAL-COMPLEX ACID DYES

1:1 Metal-complex Acid dye	Chemical structure	Colour Index No.	Molecular Formula (Mol. wt.)
D I Acid Red GRE		C. I. Acid Red 183	$C_{16}H_{11}ClN_4Na_2O_8S_2$ (532.48)
D II Acid Blue M2G		C. I. Acid Blue 158	$C_{20}H_{14}N_3NaO_7S_2$ (495.46)
D III Acid Yellow MGR		C. I. Acid Yellow 99	$C_{16}H_{13}CrN_4NaO_6S$ (496.35)

The dyes were purified by the following method before use–

The commercial metal-complex dye was dissolved in sufficient quantity of Dimethyl formamide (DMF) solvent and filtered through Whatman Filter Paper No. 1 so that all inorganic impurities insoluble in DMF are separated from the dye. The dye was then separated from the above solution with the help of trichloroethylene solvent. The quantity of trichloroethylene solvent used was about 5 to 6 times volume of DMF used. The precipitated dye was collected on the Whatman Filter Paper No. 1 by filtering the solution and finally drying the purified sample at about 105° C temperature in hot air dryer for about 2 hours. The dried sample was then kept in a vacuum desiccator over P_2O_5 at room temperature for several hours.

(3) Chemical and Auxiliaries: The solvent, acetophenone (98%; mol. wt. 120.15), used for the work was of Analytical Reagent grade. All other chemicals used were of Laboratory Reagent grade.

B. Experimental Methods

Silk fabric was pretreated with the acetophenone solvent and subsequently dyed with the metal-complex acid dyes as follows:

(1) Preparation of dye solution: 1gm of dye was weighed exactly on an electronic balance and pasted with distilled water and finally the volume was made 100 ml. This solution was used as stock solution of the dye having strength 1:100.

(2) Pretreatment with Acetophenone: Silk fabric samples were treated with 4% acetophenone (v/v) at room temperature for different intervals of time, viz. 10, 20 and 30 minutes. The fabric samples were then washed thoroughly with cold water and dried in oven at 60° C.

(3) Dyeing Procedure: Dyeing of treated as well as untreated silk fabric samples was performed on Laboratory constant temperature water bath (Model: Paramount Pvt. Ltd.). The dye bath was prepared as follows –

2 %	Metal-complex acid dye (*owf*)
3 %	Acetic acid (*owf*)

M: L ratio = 1: 40

Well-wetted untreated samples were introduced in the above dye baths at room temperature and kept for 10 minutes. For one set, the dyeing was continued at room temperature for another 45 minutes. Similar dyeings were also performed at Room temperature, 40°, 50°, 60° and 70° C temperatures respectively.

Simultaneously, untreated silk fabric was also dyed by conventional dyeing method (at 90° C for 60 min) for comparing the dyeing performance of solvent treated and subsequently dyed samples.

(4) Washing Procedures: After dyeing, all dyed sample were rinsed thoroughly with cold water; then soaped at 60° C for 15 min using non-ionic detergent (Lissapol D; 5 g/l); again washed thoroughly with cold water and dried at ambient temperatures.

C. Testing and Analysis

(1) Measurement of Physico-mechanical Properties :

(a) Tensile Properties: The treated as well as untreated samples were tested for breaking load and elongation at break on Instron 1121 Tensile

Tester (UK) using 200 mm/min extension rate and 500 mm gauze length. An average of 10 readings was taken.

(b) Shrinkage Behavior: The shrinkage due to the pretreatment with acetophenone was determined by measuring the length before and after the pretreatment. Consequently, the percentage shrinkage was calculated using the following formula:

$$\text{Percent shrinkage} = \frac{x - y}{x} \times 100$$

where x and y are the initial and final lengths of the samples before and after the pretreatment

Weight Analysis: The change in weight due to the pretreatment was also measured in the same manner as shrinkage by taking weights of the samples before and after the pretreatment. The percentage change in weight was calculated as follows:

$$\text{Percent change in weight} = \frac{W_1 - W_2}{W_1} \times 100$$

where W_1 and W_2 are the initial and final weights of the samples before and after the pretreatment.

(2) Evaluation of Dyed Samples:

(a) Colour measurement: Dyeing performance of various dyed samples was assessed by measuring the relative colour strength (*K*/*S* value) spectrophotometrically. These values are computer calculated from reflectance data according to Kubelka - Munk equation [18].

(b) Assessment of Fastness Properties: All the dyed samples were evaluated for fastness to various agencies like washing, light and rubbing using standard methods [19].

- Fastness to Washing: Wash fastness of different dyed samples was assessed on Launder-o-meter using ISO standard Test No. 3. The change in shade was visualized using Grey scale and graded from 1 to 5; where 1 indicates poor and 5 excellent fastness to washing.
- Fastness to Light: Colour fastness to light was evaluated by exposing the dyed samples to sunlight according to AATCC test method 16B-1977. They were graded from 1 to 8; where 1 indicates poor and 8 excellent fastness to light.
- Fastness to rubbing: This method is intended for determining the resistance of the colour textiles of all kind to rubbing off and staining other material. Two tests were made, one with a dry rubbing cloth and other with a wet rubbing cloth.

III. Results & Discussion

In order to perform dyeing at lower temperatures, silk fabric was pretreated with 4 % acetophenone at room temperature for 10, 20 and 30 minutes. The treatment caused some physico-mechanical changes to occur in the silk substrate. These changes were determined in terms of tensile properties, shrinkage behavior and weight analysis. Dyeing of metal-complex acid dyes was performed on the pretreated samples at different temperatures (room temperature, 40°, 50°, 60°, 70° C) for 45 minutes. The colour strength and fastness characteristics were compared with untreated samples dyed conventionally.

A. Effect of Solvent Pretreatment on Physico-mechanical Properties of Silk Fibre

The tensile strength (dry) in terms of breaking load and elongation-at-break of untreated as well as solvent pretreated silk fabric are mentioned in Table II. It can be clearly seen from the table that the solvent treatment decreases the tensile strength of silk fibre. The breaking load of untreated silk is 67.08 g while that of acetophenone treated silk is 59.63 g when the treatment was given for 10 minutes showing 2.44 % decrease in strength of the fibre, which is marginal. There is also a marginal reduction in the elongation at break for the 10 minutes treated samples as compared to the untreated silk sample. However, when the treatment time is increased to 20 and 30 minutes, there is further reduction in the values of breaking load (up to 11.1 %) as well as elongation at break. The decline in the tensile properties of silk due to solvent pretreatment largely depends on the chain length of the macromolecule and also on the inter-chain hydrogen bonds. Breaking down of these weak hydrogen bonds during the treatment with the solvent may be responsible for the substantial decrease in the tensile strength due to pretreatment.

The shrinkage analysis has been carried out before and after the treatment with the solvents. The values for the change in length of silk due to the solvent pretreatment, both in the warp and weft directions, are represented in Table II. It may be postulated that the solvent swells the silk fibre to a considerable extent and thereby gets absorbed by the fibre replacing the pre-occupied water molecules. Thus, the solvent gets diffused into the fibre structure resulting in the molecular breakdown so that the molecular chains become free for relaxation and cause shrinkage to occur.

TABLE II. EFFECT OF SOLVENT PRETREATMENT ON PHYSICO-MECHANICAL PROPERTIES OF SILK

Physico- mechanical properties		Sample			
		Control	Solvent treatment time (min)		
			10	20	30
Breaking load (g)		67.08	65.44	62.38	59.63
Elongation-at-break (%)		11.23	10.65	9.76	8.24
Shrinkage (%)	warp	--	1.67	2.66	3.86
	weft	--	2.89	3.84	5.12
Weight loss (%)		--	5.7	6.3	7.4

The solvent treatment is also found to cause weight reduction of the silk fibre. The percent weight loss of silk is 5.7 %, 6.3 % and 7.4 % corresponding to the solvent treatment for 10, 20 and 30 minutes respectively. The weight loss is found to increase slightly with the increase in the time of treatment. The decrease in weight may be due to the removal of sericin (gummy matter) from the fibrous material due to the solvent treatment. The extent to which the sericin is removed by the solvent decides the percent weight loss due to solvent treatment.

B. Effect of Solvent Pretreatment on Colour Strength of Silk Fibre Dyed with Metal-complex Acid Dyes

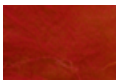
The colour strength (in terms of K/S values) of conventionally dyed samples and solvent pretreated samples with different 1:1 metal-complex acid dyes has been mentioned in Table III. The colour strength (K/S) values of conventionally dyed silk substrates with D I, D II and D III dyes are 13.67, 14.29 and 8.36 respectively. Temperature plays a significant role on the dyeing behaviour of acetophenone solvent treated silk dyed with various metal-complex acid dyes. It can be visualized from the table that there is an increase in the colour yield with an increase in the dyeing temperature for various solvent treated and dyed samples. Dyeing performed at room temperature gives uniform dyeing results but the colour strength is considerably lower than the conventionally dyed sample. However, the impact of temperature varies from dye to dye. The dyeing results for solvent induced modified dyeing at 40° C and 50° C, for all the three dyes under study, are quite comparable with conventionally dyed samples. But when the dyeing on solvent treated sample is performed at 60° and 70° C, there is substantial enhancement in the colour strength values, particularly when the treatment has been given for 10 and 20 minutes.

TABLE III. COLOUR STRENGTH (IN TERMS OF *K/S* VALUES) OF SILK FABRIC DYED WITH INDIVIDUAL METAL-COMPLEX ACID DYES FOR PRODUCTION OF PRIMARY SHADES

Dye	Dyeing temp. (° C)	*Shades & K/S* values for			
		Control	Solvent-treated samples		
			10 min	20 min	30 min
D I: Acid Red GRE	90	13.67	–	–	–
	RT	–	11.54	11.13	10.57
	40	–	13.54	13.33	12.47
	50	–	14.69	14.11	13.68
	60	–	15.43	15.87	14.84
	70	–	16.65	16.32	14.86
D II: Acid Blue M2G	90	14.29	–	–	–
	RT	–	12.56	12.21	11.89
	40	–	14.53	14.12	13.67
	50	–	15.87	15.13	14.61
	60	–	16.98	17.06	15.36
	70	–	17.66	17.52	16.75
D III: Acid Yellow MGR	90	8.36	–	–	–
	RT	–	6.33	5.98	4.84
	40	–	9.25	8.76	6.75
	50	–	10.53	9.48	8.25
	60	–	11.77	11.43	9.69
	70	–	12.28	12.02	10.44

The shades obtained for the three metal-complex acid dyes used for the investigation can be visualized from Table IV. It can be seen from the samples that the depth of the shade enhances when the acetophenone solvent pretreatment has been given to the silk substrates. However, sometimes the shade may become dull as observed from the shade for the 30 min solvent treated and subsequently Acid Blue M2G dyed silk fabric at 70° C.

TABLE IV. SHADE COMPARISON OF SILK FABRIC DYED WITH INDIVIDUAL METAL-COMPLEX ACID DYES DYED BY CONVENTIONAL AND SOLVENT-ASSISTED DYEING METHODS

Dye	Control sample (dyeing temperature: 90° C)	Samples treated with Acetophenone for (dyeing temperature: 70° C)		
		10 min	20 min	30 min
D I	 (K/S = 13.67)	 (K/S = 16.65)	 (K/S = 16.32)	 (K/S = 14.86)
D II	 (K/S = 14.29)	 (K/S = 17.66)	 (K/S = 17.52)	 (K/S = 16.75)
D III	(K/S = 8.36)	 (K/S = 12.28)	 (K/S = 12.02)	(K/S = 10.44)

It has been reported in the literature that the molecular weight of the metal-complex acid dye has a great influence on the colour strength values of the samples dyed by solvent-induced modified dyeing system. The increase in the complexity or the molecular weight of metal-complex acid dye affects the affinity of the dye for the silk fibre. However, in the present work, the three dyestuffs selected possess nearly similar molecular weights and therefore a nearly similar trend has been observed in the dyeing behavior of these dyes. Further, the dye uptake in case of solvent pretreatment followed by metal-complex acid colour dyeing also depends upon the solubility of the dye in acetophenone solvent and low water solubility of a particular dye used.

The time of treatment with acetophenone solvent also has an influence on colour strength during application with metal-complex acid dyes on silk. It can be observed from the Table 3 that on increasing the time of treatment of the solvent from 10 minutes to 30 minutes, followed by dyeing with metal-complex acid dyes, there has been a substantial decrease in the K/S values; this may be due to extensive swelling. The solvent-water system may partially modify the fibre structure by regrouping the hydrogen bonds, thereby enhancing the dye penetration inside the fibre and thereby causing higher shrinkage at longer duration of treatment. Due to more swelling, the openings of the pores in the fibrous structure will be more. During subsequent dyeing, the molecules cannot only penetrate inside the fibrous structure easily, but it can also

come out rapidly. However, there is reduction in the final colour strength of the dyed sample when the treatment time is increased.

C. Effect of Solvent Pretreatment on Fastness Properties of Silk Dyed with Metal-complex Acid Dyes

Table V indicates the washing, light and rubbing (both dry as well as wet) fastness characteristics of various silk samples dyed by conventional dyeing method as well as acetophenone solvent modified system. The table clearly shows the significance of solvent treatment as the samples treated with acetophenone and subsequently dyed with metal-complex acid dyes have better grades in many cases, particularly at 60° and 70° C dyeing temperature as compared with conventional samples dyed at 90° C.

TABLE V. EFFECT OF SOLVENT TREATMENT ON FASTNESS PROPERTIES OF SILK DYED WITH METAL-COMPLEX ACID DYES

Dye	Dyeing temp. (° C)	Fastness grades for															
		Control				Solvent-treated samples											
						10 min				20 min				30 min			
		Wash	Light	Rub		Wash	Light	Rub		Wash	Light	Rub		Wash	Light	Rub	
				Dry	Wet			Dry	Wet			Dry	Wet			Dry	Wet
D I: Acid Red GRE	90	4-5	7	4-5	4	–	–	–	–	–	–	–	–	–	–	–	–
	RT	–	–	–	–	4-5	6-7	3	4	4	6	3-4	4	4	6-7	3-4	4
	40	–	–	–	–	4	7-8	4-5	3-4	4	6-7	3-4	4	4-5	7	3-4	4
	50	–	–	–	–	4	8	4-5	4	4-5	7	4	4	4-5	7	4	4
	60	–	–	–	–	4-5	7-8	5	4-5	4-5	7-8	4	4-5	5	7-8	4	4
	70	–	–	–	–	5	8	4-5	4-5	4-5	7-8	4	4-5	5	8	4	4-5
D II: Acid Blue M2G	90	4-5	7	4-5	4-5	–	–	–	–	–	–	–	–	–	–	–	–
	RT	–	–	–	–	4	6	4-5	3-4	4	7	3-4	4	4-5	6-7	3-4	3-4
	40	–	–	–	–	3-4	6-7	4	3-4	4	7	3-4	4	4-5	6-7	4	3-4
	50	–	–	–	–	4	7	4	4	4	7-8	4	4-5	4	7	4	4
	60	–	–	–	–	4	7-8	4-5	4-5	4	8	4	5	4-5	7-8	4	4
	70	–	–	–	–	4-5	7-8	4-5	5	5	8	4-5	5	5	7-8	4	4-5
D III: Acid Yellow MGR	90	4	6-7	4	3-4	–	–	–	–	–	–	–	–	–	–	–	–
	RT	–	–	–	–	3	6-7	3	3-4	4	6	4	3	3-4	6-7	3	4
	40	–	–	–	–	3	7-8	3-4	4	4	6-7	4-5	3-4	4	7	4	3-4
	50	–	–	–	–	3-4	7-8	4	4	4-5	7	4-5	4	4	7-8	4	4
	60	–	–	–	–	4-5	7	4-5	4-5	4-5	7	4-5	4-5	4	8	4-5	4
	70	–	–	–	–	4-5	7-8	5	4-5	4-5	7-8	4-5	4-5	5	8	4-5	5

The washing grades are in the range of 3 to 5 indicating that washing fastness ranges from good to excellent. A washing grade of 3 is particularly observed for treated sample dyed at room temperature, which indicates that proper dye fixation has not been achieved on the fibre at such a low dyeing temperature. Excellent washing fastness grades (4/5) are observed when the dyeing temperature is slightly increased from 40° C to 70° C. In most of the cases, the wash fastness is quite comparable with that of conventionally dyed sample.

The light fastness grades ranges from 7 to 8 exhibiting excellent light fastness of various acetophenone treated and dyed samples. However, solvent treated samples dyed at room temperature exhibit fastness grades in the range of 6 to 7 (good to very good), indicating that the fixation of metal-complex acid dye on silk substrate is adequate but not to that extent as observed for other dyeing temperatures.

The rub fastness characteristics, both in dry and wet conditions, are quite adequate and are comparable with those of conventionally dyed samples.

CONCLUSIONS

The morphological structure of silk fibre is modified by solvent pretreatment, which removes sericin (silk gum) from the fibre, thereby causing reduction in tensile strength, shrinkage and weight loss. The dyeability of the fibre towards metal-complex acid dyes is also improved to a greater extent. Dyeings performed at lower temperatures ranging from room temperature to 70° C have shown better results. At 60° and 70° C, all metal-complex acid dyes under study gave comparatively improved dyeing performance than even conventionally dyed samples. The fastness characteristics, viz. wash, light and rub (both dry and wet) fastness are adequate and quite comparable with conventionally dyed samples.

Silk is usually dyed at 90° C for 60 to 90 min. However, by solvent pretreatment, it is possible to reduce the dyeing time to 45 minutes and dyeing temperatures up to 60° or 70° C in order to achieve optimum dyeing results. This will lead to energy conservation and considerable saving of time, which will subsequently reduce the cost of dyeing and make the process economical.

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