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A Comparative Study on Fastness of Silk Fabric Dyed with Reactive Dye and Metal Complex Dye

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Abstract

Poor colour fastness in textile products is one of the major sources of customer dissatisfaction and leads to fading or bleeding of textile colour by various type of external influences like water, perspiration, rubbing etc., during textile manufacturing and in daily use. Moreover, poor colour fastness may also prove hazardous due to the bleeding and subsequent penetration of the dye molecules and metal ions into the human skin. This article has tried to find a solution to mitigate the poor colour fastness to perspiration particularly for dark shade on silk fabrics. This paper shows a comparative analysis of different type of fastness properties of natural silk fabrics and their strength loss while dyeing with reactive and metal complex dyes following different national and international standards like ISO, ASTM standards etc. In this study, it has been revealed that colour fastness to perspiration of reactive dyed silk fabric against all types of fibres is comparatively better than that of metal complex dyed silk fabric. Percentage of strength loss is not affecting the subsequent processes of weaving preparatory and weavability of dyed silk yarn. The colour fastness to perspiration is notably achieved by using reactive dye fulfilling the customer's demands.

Keywords: Perspiration fastness, rubbing fastness, metal complex dye, reactive dye, perspirometer, crock meter.

Introduction

Silk is a naturally occurring protein fibre produced by the worms and has been potentially used as a textile material for over five thousand years. Natural raw silk is composed mainly of sericin about (22-25 %), fibroin about (62.5-67.0 %) and the rest is of water and mineral salts (***Mahmoodi, Moghimi, Arami, Mazaheri, 2010***). Fibroin is a single protein which is insoluble in hot water. On the other hand, sericin is primarily amorphous and acts as a gum binder to maintain the structural integrity of the cocoon, which makes it more water-soluble than fibroin (***Asakura and Kaplan, 1994***), (***Shenai and Saraf, 1993***), (***Chopra and Gulrajani, 1994***), (***Jiang, Wang, Huang, Guo, 2006***), (***Freddi, Mossotti, Innocenti, 2003***). This delicate filamentous fibre is well known for its sheen texture, water absorbency, dyeing affinity, thermal tolerances along with insulation properties (Sheikh et al., 2006). Silk is basically a strong fibre having strength 3.6-4.0 cN/tex which can be attributed for its linear and beta configuration and considerable crystalline polymer system (***Ghol and Vilensky, 2005***). Apart from this, silk fibre contains $-NH_2$ and –COOH groups at either ends of its chemical structure for which it can be dyed with acid dyes, reactive dyes, metal–complex dyes etc. Amongst all of these dyes, acid and metal-complex dyes possess better affinity for fibre and get easily absorbed by the fibre but have poor to moderate washing fastness. Reactive dyed silk fabric shows high perspiration fastness (***Chakraborty, 2010***). But the main objective of colouration of a textile material is the permanent retention of the colour even after exposure to certain chemical agencies without damaging the material constituents (***Makkar, Singh, Rose, 2013***), ***(Gulrajani and Maulik, 2002)***. This can be further illustrated that the process of colouration should not destroy the textile colour during processing and usage leading to a useful life of the coloured fabric showing satisfactory washing, light, rubbing, perspiration fastness etc. The colour fastness has a direct relation on the consumer's choice for certain colours and fabrics. During dyeing of silk material, an operation or a series of operation is carried out by means of which uniform colour of a

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permanent nature is produced on the substrate. This implies that the process of colouration should neither allow the colour to fade due to perspiration, rubbing, washing away during laundering nor should it fade rapidly when exposed to light. Wide variations in the fastness properties of dyes have been observed which depends on a number of factors for example, chemical construction of the dye-stuff, nature of the substrate, depth of shade, method of application, auxiliary chemicals added during the dyeing process etc.***(Jain, 1988)***.

1.1 Dyeing of silk material with metal-complex dye

Premetalised dyes, also known as metal-complex dyes, are the incorporation of the metal into the dye molecule for ready application of the dye onto the material substrate expediting the process of dyeing. Generally, there are two types of premetalised dyes one is 1:1 premetalised dyes having one dye molecule for each metal ion and the other one is 1:2 metal complex dye having one metal ion complex for every two dye molecules ***(Ghol and Vilensky, 2005)***. Reaction mechanism of silk with metal complex dye is illustrated below-

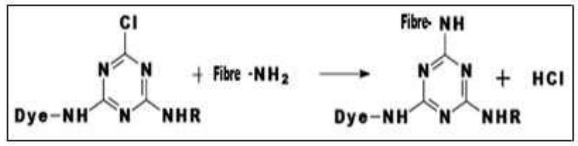
$$\text{Fibre-NH}_2\ (s) + H^+\ (aq.) + HSO_4^-\ (aq.) = \text{Fibre-NH}_3^+\ HSO^-\ (s)$$

$$\text{Fibre-NH}_3^+\ HSO_4^-\ (s) + \text{Dye-}SO_3^-\ (aq.) = \text{Fibre-NH}_3^+\ \text{Dye-}\ SO_3^-(s) + HSO_4^-\ (aq.)$$

In this reaction, ionic links are formed between positively charged amino group and anionic dye radical.

1.2 Dyeing of silk fibre with reactive dye

Reactive dyes are so called because their molecules react chemically with the fibre polymer to form a co-valent bond. Reactive dyes produce bright shades with good wash, light and perspiration fastness properties due to its reaction with $-NH_2$ group of silk; a covalent bond formation takes place between $-NH_2$ group of silk and reactive groups of dye ***(Ghol and Vilensky, 2005)***. Reaction mechanism of silk fibre with reactive dye ***(Uddin and Hossain, 2010)*** shown below-



Reactive dyes basically belong to two classes-substitutive and additive classes. Substitutive dyes include mono and dichlorotriazines and additive type includes mainly vinylsulphone dyes (remazol dyes) possessing a general formula $DSO_2CH=CH_2$.These dyes do not give any by-product during reaction with cotton or water, follows a neucleophilic addition mechanism and are applied through application of heat. Chemical attachment takes place in alkaline pH (Chakraborty, 2010). Some of the commonly used reactive dyes are Dichlorotriazine-Procion MX, Aminochlorotriazine monochlorotriazine)-ProcionH, Aminofluoro triazine (monofluorotriazine)-CibacronF, Cholorodifluropyrimidine-Drimarene K, Sulphatoethylsulfone Remazol, Trichloropyrimidine DrimareneX, etc.

Materials and methods

In this study two ply 20/22 warp and four ply 40/44 weft degummed 100% filature silk yarns have been selected for dyeing. The total samples have been divided into two parts. Metal complex dye (1:2 metal-complex dye) has been applied on one part of the sample, while the other part is subjected to the reactive dye Drimaren Black CLS (C.I. no reactive black 5). The part of the sample that has been dyed with metal complex dye is nomenclatured as *Sample-A*, while the other sample dyed with reactive dye is named as *Sample-B.*

2.1 Pre-treatment process of silk yarn before dyeing

Silk yarn was treated to degum with a solution of (Material is to Liquor Ratio) M.L.R 1:20 containing degumming agent enzyme for weft and soap for warp along with other respective chemicals as specified in Table 1.

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Table 1- Recipe for Degumming of Silk Yarn

	Recipe	Quantity for weft	Quantity for warp
Degumming Process MLR=1:20, Time=30 min. at 90°c, Ph=10-11	Wetting and stain removal agent	2gm/lit	2gm/lit
	Lubricating agent	2gm/lit	2gm/lit
	Wetting agent	1gm/lit	1gm/lit
	Enzyme, degumming agent	3.5gm/lit	Nil
	Soap, degumming agent	Nil	5gpl

2.2 Dyeing Process

The Dyeing process has been carried out in a full scale commercial Arm Dyeing Machine by following the exhaust method. *Sample-A* has been subjected to this dyeing method. In this method the material to liquor ratio is maintained at 1:20. One gram per litre of levelling agent is added in the machine at 30^0C - 35^0C and the entire material is allowed to run for 10 minutes. This is followed by the addition of metal complex dye (shade of black having depth 8%) at the same temperature followed by running the bath for 10 minutes. 2 grams per litre of acetic acid to be added in the machine to maintain the Ph of the bath at 3 to 4. After the addition of the ingredients the temperature of the bath has been raised to 86^0C at the rate of 2^0C per minute and once the temperature has been attained it has been kept holding at that temperature for about 30 minutes. After the stipulated holding time the liquor is drained out followed by hot wash. A dye-fixing agent of 4 grams per litre has been added at 50^0C and run for 10 minutes. This is followed by a subsequent draining of the bath. Fresh water is then introduced followed by scrooping with acetic acid along with antistatic agent of 2 grams per litre together with antifungal agent of 4 grams per litre at room temperature. Similarly the Arm Dyeing Machine has been prepared for carrying out the dyeing of *Sample-B* with reactive dye stuff. The material to liquor ratio in this case is also maintained the same as the previous sample. 1 gram per litre of levelling agent is added in the machine at 30^0C - 35^0C and the entire material is allowed to run for 10 minutes. The reactive dye stuff (shade of black having depth 8%) is then added followed by running of the bath for 10 minutes. This is followed by elevation of the temperature to 70^0C at the rate of 2^0C per minute and maintained at that temperature for 30 minutes. Glauber Salt of 50 gram per litre is then added in two steps of 25 gram per litre per step for controlled rate of dyeing decreasing thereby the hydrolysis of dyes. The temperature of the dyeing bath is then brought down to 50^0C followed by addition of soda ash and sodium bicarbonate of 15 gram per litre each. The temperature of the bath has been again elevated to 70^0C and kept there for half an hour. This operation is then followed by hot wash and cold wash of the material and subsequent scrooping with acetic acid, antistatic agent and antifungal agent as has been adopted for the earlier sample.

2.3 Fabric Preparation

Fabric from above dyed yarn has weaved on a Rapier Loom with following parameters as shown in Table 2.

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Table 2 - Fabric specifications

Sl. No.	Fabric parameters	Values
1.	Weave construction	8 end satin
2.	Thread density (ends/cm × picks/cm)	140 × 44
3.	Area density (gsm)	91.00
4.	Warp(degummed and dyed) ply and resultant denier	2 ply, 34 den
5.	Weft(degummed and dyed) ply and resultant denier	4 ply,136 den

2.4 Fastness Testing

Colour fastness to perspiration has been evaluated by ISO 105-E04:2008 method with artificial perspiration solutions of acidic and alkaline type while fastness to water has been evaluated by ISO 105-E01:2013 method. In this method, both the specimens (Sample-A and Sample-B) are brought in contact with multi fiber fabric stripes as shown in figure 1a. The samples of dimension 100 mm by 40 mm adhered with the multi fibre stripes is wet in simulated acid and alkali perspiration solutions and distilled water in room temperature, taken out and then subjected to a fixed mechanical pressure and allowed to dry slowly at a slightly elevated temperature of 37±20C in a perspirometer which are shown in figures 1b, 1c and 1d respectively.

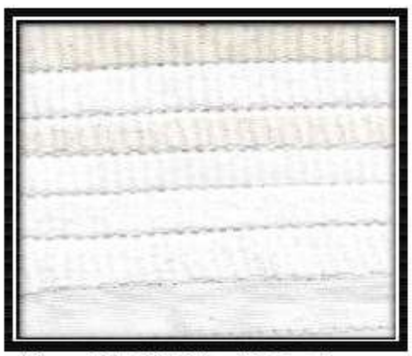
Figure 1(a) - Multi fibre fabric stripes

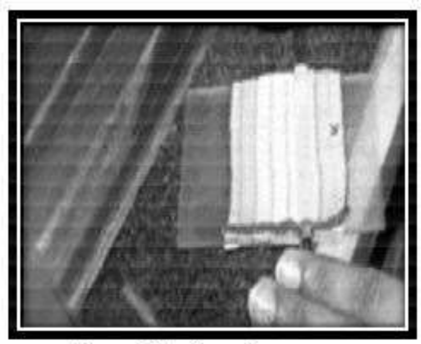
Figure 1(b) - Squeezing excess Liquor from sample


Figure 1(c) - Loading the Perspirometer

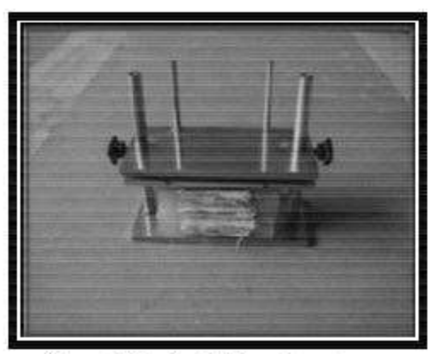
Figure 1(d) – Loaded Perspirometer

The sample has been kept in this condition for 4 hours after which the sample is taken out and subjected to conditioning at standard temperature and relative humidity. After conditioning, the specimen is evaluated for colour change and for

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colour transfer from the sample to the multi fibre fabric stripes. Two sets of standard grey scales have been used to assess the colour staining and colour change of the samples. Rubbing fastness has been evaluated by ISO 105 – X12: 2002 method with a Crock Meter which is shown in figure 2.

Figure 2 – a typical Crock Meter

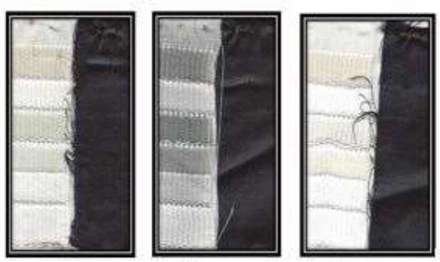
This test method is designed to determine the amount of colour transferred from the surface of coloured textile materials to other surfaces by rubbing. Crock Meter has a finger of 1.6 cm diameter and can be moved to and fro in a straight 10 cm track on the specimen.

2.4 Yarn strength testing

Yarn strength test was carried out by a single yarn strength tester following the standard method of ASTM D2256. Single strand yarn specimens have been tested on a tension testing machine at a predetermined elongation rate and the breaking force has been determined adjusting the rate to 300±10 mm/min for 250 mm (10 inches) gauge length as per the above stated standard method.

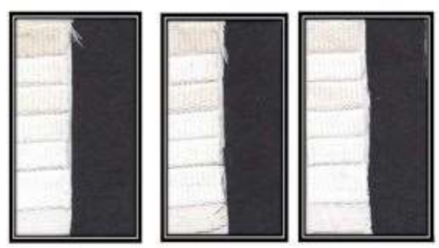
Result and discussion

In this study, it has been revealed that colour fastness to perspiration of reactive dyed silk fabric against all types of fibres is comparatively better than that of metal complex dyed silk fabric. This observation is supported by the tables for assessment result of staining and colour change [Table nos. 3(a), 3(b) and 4 respectively) and figures of the dyed fabric specimens (figures 3 and 4 respectively).



A **B** **C**
A = staining against acid
B = staining against alkali
C = staining against water

Figure 3 – Staining of Metal complex dyed sample - A



D **E** **F**
D = staining against acid
E = staining against alkali
F = staining against water

Figure 4 – Staining of Reactive dyed sample - B

Table 3(a) - Assessment result for staining of Metal complex dyed silk fabric

Multi fibre fabric	Grey scale value	
	Metal complex dyed Sample	
	Alkali	Water
Wool	2-3	4
Viscose	3	4-5
Silk	2	4
Polyester	2-3	4
Cotton	3	4
Acetate	3-4	4-5

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Nylon	3	4

Table 3(b) - Assessment result for staining of Reactive dyed silk fabric

Multi fibre fabric	Grey scale value	
	Metal complex dyed Sample	
	Alkali	Water
Wool	2-3	4
Viscose	3	4-5
Silk	2	4
Polyester	2-3	4
Cotton	3	4
Acetate	3-4	4-5
Nylon	3	4

Table 3(b) - Assessment result for staining of Reactive dyed silk fabric

Multi fibre fabric	Grey scale value	
	Reactive dyed Sample	
	Alkali	Water
Wool	4	4-5
Viscose	4	4-5
Silk	4-5	4-5
Polyester	4	4-5
Cotton	4	4-5
Acetate	4-5	4-5
Nylon	4	4-5

Table 4 - Assessment result for colour change of dyed silk fabric

Samples	Grey scale value		
	Acid	Alkali	Water
Metal complex dyed Sample-A	4-5	4-5	5
Reactive dyed Sample-B	5	5	5

A set of grey scales is used for measuring staining. The values have been rated as per the grey scale. Fastness rating 5 (five) indicates that there is no staining which means there is no difference between the treated and untreated materials. Similarly, fastness rating 4 (four) indicates slightly stained, while fastness rating 3 (three) stands for noticeable staining, fastness rating 2 (two) denotes considerable staining and fastness rating 1(one) indicates much stained of the dyed silk fabric specimen. Study on colour fastness to rubbing of reactive dyed silk fabric shows a marginal improvement over that of metal complex dyed fabric as found from Table 5.

Table 5 - Assessment of colour fastness to rubbing

Samples	Grey scale value	
	Wet	Dry
Metal complex dyed Sample -A	4	4
Reactive dyed Sample-B	4-5	4

The results of the yarn strength test shows that metal complex silk dyed yarn sample of two ply 20/22 denier (resultant yarn count of 34 denier after degumming) requires 120 gf breaking force which is more than that required for reactive dyed silk (100 gf) as shown in figure 5.

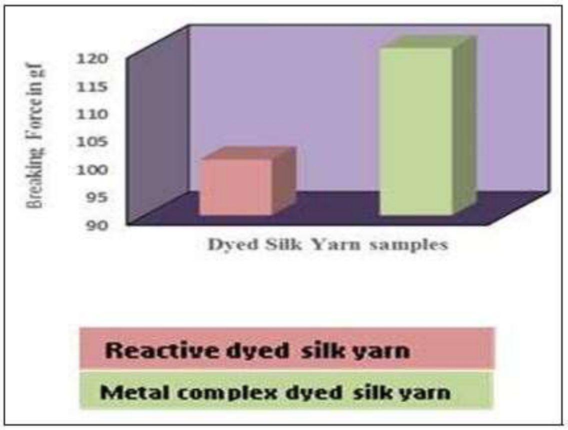

Figure 5 - Breaking force in gram force (gf) for reactive dyed and metal complex dyed silk yarn

Reactive dyeing of silk material is normally carried out in alkaline medium. As silk fibres are very sensitive to alkaline medium, above result shows that metal complex dyes depicted slightly better results than reactive dyes. There is a strength loss of 20 percentages in the reactive dyed silk yarn as compared to that of metal complex dyed silk yarn. But it has been observed that this percentage of strength loss is not affecting the subsequent processes of weaving preparatory and weavability of dyed silk yarn. Moreover, the colour fastness to perspiration is notably achieved by using reactive dye fulfilling the customer's demands.

4.0 Conclusion

The entire study reveals that the fastness properties of dyed silk materials such as colour fastness to perspiration colour fastness to rubbing gives more preference to reactive dye stuff over metal complex dye stuff particularly for higher shade depths. Though the strength loss has been observed to a certain extent for dyeing silk material with reactive dye over metal complex dye, yet this percentage of strength loss is neither found to be considerable in affecting the subsequent processes of weaving preparatory and weavability respectively nor encroaching the quality of the finished fabrics. During the combination of different dyes to obtain a particular shade, application of reactive dye may lead to considerable strength loss of the silk yarn due to repeated corrections and addition of dye in alkali medium, in order to match with the perfect shade. But the probability of occurrence of this type of problem is less while dyeing silk material with single dark shades such as black shade. Therefore, in order to achieve notable colour fastness to perspiration and rubbing at the expense of very less strength loss, single coloured high shade depth reactive dye on silk fabrics is found to be favourable than metal complex dye fulfilling thereby the customer's demands.

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