Al-Azhar Bull. Sci. Vol. 18, No. 1 (June.): pp. 1-7 , 2007.

PRINTING OF DIRECT DYE ON SILK FABRIC WITH IMPROVED DYE FIXATION AND WET –FASTNESS

WAGIH A. ABDALLAH AND ABEER I. FATH ALLA

Textile printing, Dyeing and Finishing Department, Faculty of Applied Arts, Helwan University, Cairo, Egypt.

Abstract

Application of direct dyes on silk fabric suffer from problems of poor fastness properties as well as decrease in dye fixation .A formaldehyde donor agent, i.e. Hexamine, (hexamethylene tetramine), was used for improving dye fixation and wet fastness when added to the printing paste during printing silk fabric with a direct dye containing free amino groups (Diamine Supra Blue GRL200%).

Key words

Crosslinking agent; Hexamine; silk fabric; direct dyes ; printing; wet fastness; levelness.

Introduction

Silk belongs to one of the most important groups of animal fibres. It has an affinity for many dye classes and the selection of them depends on various factors ,like regular availability of colors, shade, brightness, fastness standards(1), affordable input cost and end- use (2). Direct dyestuffs are well adapted to the printing of silk fabric. The wet fastness of these dyes is undesirable for many purposes, particularly for materials which will be expected to withstand washing . Wash fastness depends on the dye –fibre interaction, molecular weight as well as the solubility of the dye in water (3) . Direct dye on silk suffer from problems of poor fastness properties as well as dye fixation. Attempts have been made to increase the molecular weight of direct dye, after it has been adsorbed by the fibre, to render it less soluble in water and therefore more fast to wet treatment. Some fastness properties specially wash fastness of these dyes can be improved by treatment with dye-fixing agent.

The present study investigate the feasibility of utilizing hexamine, as a formaldehyde donor, in fixing amino containing direct dye onto silk structure during printing process . The method adopted is to incorporate hexamine in a silk printing paste containing Diamine Supra Blue 200% (as a direct dye containing free amino groups in its structure). The effect of hexamine concentration and steaming time on the extent of dye fixation is investigated.

Experimental

Materials

Degummed and bleached plain woven natural silk fabric , weighing 50 g/m² was used. Hexamine, commercial grade ,as well as urea, citric acid and sodium dihydrogen phosphate analytical grades, were used. Diamine supra blue GRL 200%(direct-Ciba) as well as Meypro-gum NP 16 thickening agent (Meyhall chemical AG, Switzerland) and Primasol AE New 2 [BASF] (non-ionic detergent)were also used.

Printing method

Printing of silk fabric was carried out by manual flat screen printing method using the following recipe :

Direct dye (diamine supra blue GRL 200%)-----	40	g
Urea -------- -------------------------------------	100	g
Hexamine --	(0-100)	g
Thickener (Meypro gum NP 16) ,(12%)---------	600	g
Balance (water or thickener)---------------------	X	g
	1000	g

The pH value of the print paste was adjusted at weak acid to neutral medium [pH: 5.5 -7], using citric acid and sodium dihydrogen phosphate to effect good substantivity to silk. [8]

After printing, the samples were dried at room temperature and steamed in saturated steam at 100-110 °C for 30 minutes. Each print was rinsed with hot water (70-80°C) and cold water and soaped using 2g/l Primasol AE New 2 at 80-90 °C for 15 minutes followed by rinsing in cold water and finally dried in air

Measurements

Color yield measurement

The color strength of silk printed fabric was measured using a U.V-visible spectrophotometer model ICS [Texcion Ltd., Kennestide park, Newbury, Berkshire , UK]. The reflectance expressed as K/S value was calculated by the Kubelka-Munk equation [6] $K/S = [(1-R)^2/2R] - [(1-R_o)^2/2R_o]$.

Were R &R_o are the decimal fraction of the reflectance of each colored and uncolored samples respectively, K is the absorption coefficient and S the scattering coefficient .Dye fixation was determined by stripping the printed samples with an aqueous solution of 50% dimethyl formamide [DMF] at 80- 90 °C for 15-20 min. The stripped samples were rinsed and dried, then the color yield (K/S), was measured .The value of fixed dye was calculated according to the following equation

$$\text{Dye fixation [D\%]} = \frac{A}{B} \times 100$$

Where: A and B are the color strength ,K/S, of prints after and before stripping respectively .

Color fastness measurements:

Fastness properties to washing and perspiration were conducted according to the AATCC Test Method : 61-1996 and 15-1997 respectively [7].

Results and Discution:

-Effect of Hexamine ,(hexamethylene-tetramine), concentration on the dye-fixation .

Hexamine is a condensation product of formaldehyde with ammonia [9] which considered as a formaldehyde donor product because it splits off at 100°C. Thus hexamine can be employed to fix direct dye containing amino groups on silk [5]. Released formaldehyde can unites with amino groups contained in the chemical structure of the direct dye producing large dye molecule. This can lead to dye fixation and fastness properties. The reaction possibilities which take place to illustrate the formation of bonds between the dye and silk fibres induced by hexamine , through formaldehyde release, during steaming the prints are given below:

$$D\text{-}NH_2 + HCHO + H_2N\text{-}Si \xrightarrow{H^+} D\text{-}NH\text{-}CH_2\text{-}NH\text{-}Si + H_2O \quad (1)$$

$$D\text{-}NH_2 + HCHO + HS\text{-}Si \xrightarrow{H^+} D\text{-}NH\text{-}CH_2\text{-}S\text{-}Si + H_2O \quad (2)$$

$$2D\text{-}NH_2 + HCHO \xrightarrow{H^+} D\text{-}NH\text{-}CH_2\text{-}NH\text{-}D + H_2O \quad (3)$$

Where : Moreover ,the formaldehyde released can crosslink silk as follws:

$$2Si\text{-}NH_2 + HCHO \xrightarrow{H^+} Si\text{-}NH\text{-}CH_2\text{-}NH\text{-}Si + H_2O \quad (4)$$

Where :D:direct dye - Si:silk

Nature of the chemical structure of formaldehyde donor crosslinking agent , hexamine, as well as its concentration additive included in the printing paste had significant effects on the printing properties of silk fabric i.e. wet fastness and dye fixation.

To investigate the validity of dye fixation on to silk structure according to the aforementioned tentative mechanism different concentrations of hexamine were added to the printing past ,and color strength of prints was determined before and after stripping, and the extent of dye fixation .Data obtained are shown in table (1).

Table (1) Effect of hexamine concentration on dye fixation on to silk fabric

Hexamine conc. (g/Kg)	Color strength (K/S)		Dye Fixation(%) [D%]
	Before stripping [B]	After stripping [A]	
0.0	5.04	3.47	68.8
5	5.43	3.79	69.7
10	5.80	4.16	71.7
20	6.11	4.45	72.8
30	6.81	5.16	75.7
40	6.89	5.37	77.9
50	7.05	5.92	83.9
60	7.58	6.57	86.6
70	8.43	7.54	89.4
80	8.40	7.35	87.5
90	8.33	6.25	87.0
100	8.29	6.14	86.1

Printing paste: see experimental part, steaming at 110°C for 30 min.

It is clear ,table (1),that increasing hexamine concentration in the printing paste is accompanied by increasing the extent of dye fixation ,reaching a maximum at 70 g/Kg, then slightly decreases by further increase in concentration up to 100g/kg.It seems, beyond a concentration of 70 g/Kg , that the extent of both dye-hexamine reaction (reaction3) and silk crosslinking (reaction 4) become appreciable, which leads to dye loss and unaccessible silk structure, thereby decreasing the extent of dye fixation in that range.

2-Effect of steaming time on the dye fixation .

In this phase of study, a printing paste containing hexamine at a concentration of 70 g/Kg was selected .A temperature at 110°C was also selected as an ideal steaming temperature according to Lei etal[5]. The effect of steaming time on the color strength and the extent of dye fixation is shown in table(2).

Table (2) Effect of steaming time on color strength and dye fixation.

Steaming time (min.)	Color strength (K/S)		Dye fixation(%) [D%]
	Before Stripping	After Stripping	
10	8.14	6.71	82.4
20	8.53	7.74	90.7
30	8.40	7.51	89.4
40	8.29	7.26	87.5
50	8.04	6.89	85.6
60	7.93	6.57	82.8

printing paste:- direct dye 40 g/Kg, urea 100 g/Kg, hexamine 70 g/Kg, thickener 600g/ Kg- steaming temp. 110°C.

It is obvious, under the conditions employed, that prolonging the time up to 20 min. is accompanied by increasing the extent of dye fixation , expressed as K/S after stripping and D%. Beyond a time of 20 min. and up to 60 min. the extent of dye fixation decreases. The later can be ascribed to the breakdown of "dye-formaldehyde –silk" bonds under the action of extra energy furnished by subjecting the prints to prolonged steaming time.

3-Effect of hexamine on the wet –fastness properties of the prints.

Silk specimens printed with the direct dye in absence and presence of hexamine using the optimum conditions were assessed to wet fastness tests ,i.e. washing and perspiration , and the results are formulated in table (3).

Table(3): Wet fastness properties of silk prints in Absence or presence of hexamine.

Absence or presence of hexamine in the print paste	Wet-fastness properties					
	Fastness to washing		Fastness to perspiration			
			Acidic		Alkaline	
	Alt.	Sta.	Alt.	Sta.	Alt.	Sta.
Absence of hexamine in the print paste	4	4	3/4	4	4	4
presence of hexamine in the print paste	4/5	5	4/5	5	4/5	5

printing paste:- direct dye 40 g/Kg, urea 100 g/Kg, hexamine 70 g/Kg, thickener 600g/ Kg- steaming temp. 110°C ,steaming time 20 min.

Alt. : alteration- Sta. : staining

The above table reveals that fastness to washing and perspiration in the case of hexamine presence are better than corresponding samples printed without adding it in the printing paste .This could be ascribed to the high extent of dye fixation , i.e. complete reaction between hexamine and each of dye and substrate which bounded chemically together with increasing the molecular size of the dye inside silk fibres.

Conclusions

Hexamine,as a formaldehyde donor, can bind Diamine Supra Blue GRL 200% direct dye on to silk structure giving rise to improved dye fixation during printing. A hexamine concentration of 70g/Kg in a printing paste containing ingredients, viz. the dye (40g/Kg),urea(100g/Kg),thickener(600g/Kg),water(190g/Kg) , at a pH of 5.5-6(using citric acid /sodium dihydrogen phosphate) as well as steaming at 110°C/20 min. represent optimum printing conditions. Washing and perspiration fastness were higher in presence than in absence of hexamine in the printing paste.

References

1. B.R. VENUGOPAL, “Dyeing of silk ” , Colouradge ,P. 36 ,June (1991).

2. M.L. GULRAJANI , Rachna , Venna Kapoor , Deepali Agarwal , “ Studies in dyeing of silk with different classes of dyes , part I- yellow dyes” Colouradge ,p.51 , May (1995) .

3. E.R.TROTMAN , “ Dyeing and chemical technology of textile fibres ” , forth edition , p. 420 , (1971) .

4. Chambers,s Technical Dictionary , Revised and Greatly , England edition , p.415 .

5. X. P. LEI, D. M. LEWIS, X. M. SHEN &Y. N. WANG, “crosslinking nucleophilic dyes on woll ” Dyes and Pigments , Vol. 30 , No. 4, P. 272 , (1996).

6. P. KUBELKA , & F. Z. MUNK , Technol . Phys. 12, P.593(1931).

7. Technical Manual of the American Association of Textile Chemist and Colorists (AATCC), Vol. 74 , 1999 .

8. V. A. SHENAI , “ Technology of textile processing ” , Technology of printing , Vol. I.V , Third Edition , P. 379 , (1990) .

الملخص العربي

طباعة الحرير الطبيعي بالصبغات المباشرة مع تحسين كل من نسبة الصبغة المثبتة وخواص الثبات للبلل

تعاني طباعة الحرير الطبيعي بالصبغات المباشرة من بعض المشاكل مثل عدم الحصول علي خواص ثبات مرضية مع قلة نسبة الصبغة المثبتة داخل الخامة .

في هذا البحث تم استخدام مادة الهكسامين $[(CH_2)_6N_4]$ Hexamine كمادة معطية للفورمالدهيد والذي ينطلق منها كغاز اثناء تبخير الطباعات لاجراء عملية تثبيت الصبغة المباشرة علي خامة الحرير الطبيعي والتي تستطيع تكوين روابط عرضية عن طريق الارتباط بمجموعات الامين "amino groups" المتواجدة في كل من الحرير الطبيعي والصبغة المباشرة وبالتالي تزداد نسبة الصبغة المثبتة ويزداد معا خواص الثبات للغسيل والاحتكاك "wet properties" .

تم دراسة مجموعة من العوامل المؤثرة علي درجة ثبات الصبغة Dye fixation وهي :

- **استخدام تركيزات مختلفة من مادة الهكسامين لاختيار افضل التركيزات التي تعطي افضل النتائج .**
- **دراسة تأثير تثبيت الطباعات (التبخير عند درجة حرارة 110°م) لفترات زمنية مختلفة للوصول الي أفضل زمن لتثبيت الطباعات .**

كما تم عمل تجارب لقياس درجة ثبات الاقمشة المطبوعة بالظروف المثلي لتركيز مادة الهكسامين المضافة لعجينة الطباعة والظروف المثلي للتثبيت ، وكانت النتائج جيدة جدا من حيث ثبات الاقمشة المطبوعة لكل من الغسيل والاحتكاك ، وتعتبر هذه النتائج إضافة جديدة لخواص الثبات للاقشمة المطبوعة بالصبغات المباشرة علي اقمشة الحرير الطبيعي .