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Innovative Applications in Natural Fabrics Coloration with Anionic Dyes

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ABSTRACT

A novel application involving printing anionic dyestuffs, namely reactive, direct and acid dyes on cotton as well as 70/30 cotton/wool substrates is fulfilled using a cationic reactant. This technique involves using two steps: the first step is printing with a recipe that includes only the thickening agent and the cationic reactant followed by steaming or thermofixation and eventually washing off. The second step is carried ou dyeing using the anionic dye at specific pH value and temperature. The mentioned sequence ensures the prevention of prints' bleeding on backgrounds and reduces energy consumption required for dye fixation on the bicomponent fabric as well as saving dye and electrolyte amounts that pollute the water effluent. In order to illustrate the union shade with sharp outlines achieved via using this technique, several design applications are carried out using different anionic dyes. All parameters and measurements that may affect the results of this work are discussed in detail, such as: cationic reactant concentration, fixation method of prints, nitrogen content percentage, tensile strength and fastness properties.

Key words: Amination, anionic dyes, cationic reactant, colour yield, fastness levels and substantivity.

Introduction

Reactive dyes are molecules that combine chromophores with a reactive group that will form covalent bonds with the fibre via reaction with hydroxyl groups. Alkaline pH (~11) is required for this reaction to proceed. Significant amounts of electrolytes (up to 100% of fabric weight) are necessary to force reactive dyes to exhaust to the cotton fibres. Excellent wash fastness properties are obtained once the dyed fabric is thoroughly washed off. This washing step is crucial since a significant percentage of the reactive dye (50 %) will react with water to form a dye molecule that has virtually no affinity for the fibre and must be removed. As is the case with direct dyes, the bulk of chemical discharge is from electrolytes, but the effluent is also coloured from the significant amounts of hydrolyzed reactive dyes (Hauser, 2000). A fibre treatment applied before dyeing that would leave the cotton fibres with cationic charges would seem to be a possible solution to both of these problems, (Lewis and Lei, 1989) have reviewed this approach.

The reurgent interest in textiles made of cotton/wool blends has led to developments in union dyeing (same shade on cotton and wool) that hold promise for reopening this market for wool, adding greater value to cotton and providing the consumer with products that have unique aesthetic appeal, fabric hand and comfort (Cardamone and Turner, 2000). Introduction of amine groups into the cellulose structure produces a fibre that may be considered analogous to wool. Unlike cellulose, wool has a natural substantivity towards anionic dyes, especially under acidic conditions (Lewis and Mcllroy, 1997). Methods for amination of cellulose have been known for quite some time. The motivation to modify wool is usually to increase or decrease dye absorption so that, for example, differential dyeing effects can be obtained (Welham, 1992). Cationic reactants can interact with fibre components of the fabric (vis., cotton and wool) in different ways as per the following:

i) Interaction with the cellulose component: the cationisation of cotton cellulose using amino compounds takes place as shown in scheme 1 (Hauser, 2000):

First an epoxide is formed in the presence of alkali (as a base catalyst). Second, this epoxide reacts with a hydroxy group in the cellulose. Aminoalkyl celluloses are recognized to be capable of enhancing dyeability with most cotton dyes. They also offer the opportunity for introduction of compounds which give properties of flame retardancy, rot resistance and increased ion-exchange capacity (Lewis and Mcllroy, 1997).

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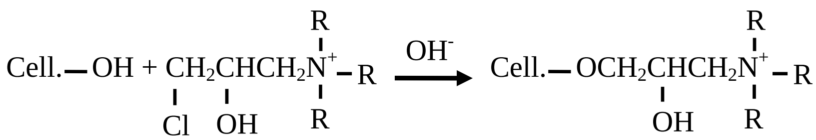

Scheme 1: Cationically modified cotton when a cationic reactant reacts with cellulose under alkaline conditions.

ii) Interaction with the protein component: The interaction of keratin wool with the cationic reactant takes place as in scheme 2. Carboxyl groups (I) dissociate into the carboxylate ions and protons as shown in reaction 1, and the cationic reactant (II) dissociates into the corresponding cation and hydroxyl ions as shown in reaction 2. Reaction 3 shows the binding of the cationic reactant onto wool fibre through a salt linkage (Abo-Shosha, 2007):

$$\underset{\text{I}}{\text{Wool} - \text{COOH}} \longrightarrow \text{Wool} - \text{COO}^- + \text{H}^+ \quad (1)$$

$$\underset{\text{II}}{\text{R-N}} + H_2O \longrightarrow \text{R-NH}^+ + \text{OH}^- \quad (2)$$

$$\text{Wool} - \text{COO}^- + \text{R} - \text{NH}^+ \longrightarrow \text{Wool} - \text{COOH.N-R} \quad (3)$$

Scheme 2: Interaction between wool keratin and the cationic reactant.

In the present study, a route is adopted to obtain union shade prints on cotton as well as 70/30 cotton/wool fabrics by omitting the cationisation step through printing while dyeing procedure is applied afterwards using anionic dyes. Subsequently, dye particles are intensively attracted to the cationic sites located only in design area which results in union coloration of fabrics without bleeding. All factors and measurements that may influence or explain the results of this work are studied.

2. Experimental:

2.1. Materials:

1. Two scoured, bleached, plain 1/1 substrates are used throughout the present work: 100% cotton fabric having a weight of 154 g/m^2 supplied by El-Mahalla for Spinning and Weaving, Egypt. The other substrate is 70/30 cotton/wool having a weight of 235 g/m^2 supplied by Goldentex Co., Egypt.
2. A reactive dye, Levafix Tuquoise Blue PN-G (C.I. Reactive Blue 197) is used in investigating various parameters as well as measurements of the present study. Moreover, another reactive dye is utilized in printing design applications, namely: Levafix Marine Blue PN-G.

   A direct dye, Sirius Scarlet S-G, is used throughout the study. Another dye is used in design applications namely: Sirius Yellow S-2G. An acid dye, Supranol Blue BLU, is used throughout the study. Another acid dye is used in design applications namely: Supralan Grey S-GL. All dyestuffs are kindly supplied by DyStar Textilfarben, Germany is used in washing off'.
3. A cationic reactant namely: Sera Fast C-B which is a polyalkyl amine is used throughout the current study and is also kindly supplied by DyStar Textilfarben, Germany.
4. Mypro gum NP-16 (Meyhall): which is a non-ionic thickening agent based on modified plant seeds gum is used with a concentration of 80 g/kg.
5. A non-ionic detergent, Sera-Wash M-RK, manufactured by Dystar Textilfarben, Germany.

2.2. Cationisation procedure:

Cotton and cotton/wool substrates are cationised via printing with a paste that contains the thickening agent as well as 100 g/kg Sera Fast C-B cationic reactant. The specimens are exposed to either steaming or thermofixation at 110 °C for 10 min. eventually, the cationized specimens are washed off using 2g/l detergent at a liquor ratio of 1:50. Washing is carried out at 70 and 60 °C for cotton and cotton/wool fabrics, respectively and lasted for 10 min. for both fabrics.

2.3. Dyeing procedure:

For reactive dyeing, both substrates are dyed, with and without cationization step, using 2% (owf) dye at pH values of 9 and 7 for cotton and cotton/wool fabrics, respectively. Dyeing procedure is carried out at 90 and 75 °C for both substrates, respectively by elevating the temperature slowly and lasted for 60 min. maintaining material-to-liquor ratio at 1:30. The pH is adjusted at the required value using sodium carbonate powder (of analytical grade).

Similar dyeing conditions (of dye concentration, pH value and temperature) are followed using direct dyes. After 15 min. of elevating temperature, sodium chloride (2% owf) is added to the dyebath. Concerning acid dyes, both fabrics are dyed in neutral medium using the same dyeing conditions of reactive dyeing. Attraction of dye molecules towards the printed areas, having cationic sites, can be easily noticed at the beginning of dyeing process. The dyed samples are rinsed in hot then cold water after which they are washed off at 90 and 70 °C for 10 min. for cotton and cotton/wool fabrics, respectively using 2 g/l non-ionic detergent.

2.4. Measurement and analysis:

- Colour strength expressed as K/S is measured according to a previously reported method (Judd and Wyszenki, 1975) by the light reflectance technique, and the relative colour strength is calculated by applying the following Kubelka-Munk equation:

Colour strength $K/S = \frac{(1-R)^2}{2R}$

where R is the decimal fraction of the reflectance of the coloured fabric, K is the absorption coefficient and S is the scattering coefficient.

- The nitrogen content is determined by the (Cole and Parks, 1949) modification of the micro Kjeldahl method.
- Tensile strength (Tenacity and elongation-at-break) are determined according to (ASTM Standard Test Method, 1994).
- Fastness properties in terms of rubbing, washing and perspiration are measured according to (Standard methods for the assessment of colour fastness of textiles, 1995).

Results and Discussion

3.1. Influence of cationisation on colour yield:

The purpose of the cationisation of cellulosic as well as protein fabrics is to increase the neutral substantivity of anionic dyes for fibres by introducing positively charged sites on fibres' surface (El-Molla, 2011). One of the drawbacks of blended fabrics is the difficulty of attaining a union (solid) shade upon coloration with the same dye at the same step. The preferential dye absorption by the woolen component in the blended fabric is a major cause of this drawback. This situation is different in case of printing process of cotton/wool blends, where cationisation can afford union shade, but the problem of staining on the white ground of the prints appears. This problem starts during washing off the prints to remove the spent printing paste out of the fabric. Unfixed dye molecules or agglomerates dissolve in the washing liquor and then recombine with the cationic sites of the white ground (Rashad et al., 2007).

In the present study, cationic sites are imparted to cotton and cotton/wool substrates through printing process. As previously mentioned in the experimental part, printing is followed by either steaming or thermofixation in order to guarantee cationic reactant penetration inside the fibres since these substrates are subsequently exposed to washing off to remove the thickener film adhered to fibres' surface and thus, prevent the potential of harsh handle as well as obtaining coloration levelness all over substrates. It is important to note that, cationisation process is carried out in a neutral medium since minor variations in K/S values are obtained in alkaline medium. After drying, the substrates are dyed with Levafix Turquoise Blue PN-G, washed off and the K/S values are assessed. In order to investigate the effect of cationisation on the colour yield of the dyed fabrics, different concentrations of the cationic reactant are incorporated in the paste (0, 20, 40, 60, 80, 100 and 160 g/kg) and the results are plotted in Fig. 1.

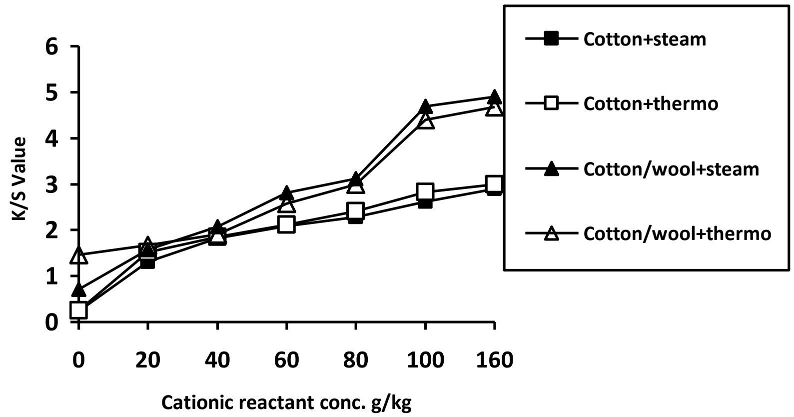

Fig. 1: Effect of Sera Fast C-B concentration (using steaming or thermofixation after cationisation) on K/S values of dyed cotton and cotton/wool fabrics with the reactive dye.

It can be observed from the figure that, increasing cationic reactant concentration is accompanied by improvements the colour yield of the dyed fabrics regardless of both: fabric type as well as the fixation method that proceeds cationisation process. It should also be noted that, greater enhancements in colour yield occur on cotton/wool prints compared to cotton prints which is referred, as mentioned earlier, to that wool is characterized by a greater affinity towards anionic dyes. Moreover, cationic reactant concentration should be limited and not to exceed 100 g/kg to maintain fastness levels (particularly the rubbing fastness) and tensile strength, as will be shown later on.

3.2. Miscellaneous applications on anionic dyes:

It is established that, alkaline medium is essential in cellulosic fibre's coloration to produce ionisation of accessible cellulose hydroxyl groups, which can then react with the dye (Broadbent, 2001). On the other hand, protein fibres are colorated in an acid medium since the rate of dye fixation increases by increasing the pH value. At lower pH values, dye concentration increases on the available fibre surfaces by increasing the number of ammonium ion sites. The positive charge always present on fabric surface forces the fabric to be dyed at any desired pH (Draper et al., 2003). Consequently, neutral medium is used on dyeing both substrates with acid dyes as well as on dyeing cotton/wool fabric with both reactive and direct dyes. Most acid dyes are known to have very little affinity for cotton and therefore, cationized cotton can be readily dyed with acid dyes (El-Molla et al., 2011). The sulphonic acid groups present in the dye molecules can interact with the cationic groups in the modified cotton. Meanwhile, both fabrics are cationized (using 100 g/kg Sera Fast C-B) and dyed with two more anionic dyes, direct and acid dyes (2% owf), and the colour yield of the dyed samples is listed in Table 1.

It can be concluded from the table that, enormous improvements in K/S values of cationized substrates take place compared to substrates without cationisation, regardless of: kind of dye, substrate and curing method. On the other hand, fixation method of the cationic reactant (through steaming or thermofixation) has a neglectable influence on K/S values regardless of both a kind of substrate and dye type. Consequently, thermofixation can be chosen as a fixation method proceeding cationisation, in order to minimize the spent energy in steaming.

The table also reveals that, better K/S values are achieved for dyed substrates using direct dyes compared to reactive and acid dyes without cationisation. This result is referred to the great affinity of direct dyes towards cotton and wool substrates due to its large molecular weight as well as its high hydrophilicity.

Moreover, the three anionic dyes (reactive, direct and acid) are used in coloration of cotton and cotton/wool fabrics using a diverse set of designs in order to clarify the potential results of the current technique, as shown in Fig. 2.

It is concluded from the figure that, applying cationisation on both fabrics via printing results in accomplishing more brilliant colours, sharp outlines and prevents bleeding occurrence. Dye bleeding is the essential feature that limits cationisation to dyeing but according to the achieved results, it is applicable also in printing. Besides, increasing dye concentration results in obtaining a dark dye colour in both: the printed areas as well as the dyed background.

Table 1: K/S values of cotton and cotton/wool substrates, with and without cationisation dyed with the three anionic dyes.

Substrates Status	K/S Value	Substrates Status	K/S Value	Substrates Status	K/S Value
Dyed cotton + reactive dye + without cationisation	0.25	Dyed cotton + direct dye + without cationisation	5.20	Dyed cotton + acid dye + without cationisation	0.09
Dyed cotton + reactive dye + Cationisation + steaming	2.62	Dyed cotton + direct dye + Cationisation + steaming	9.91	Dyed cotton + acid dye + Cationisation+steaming	1.54
Dyed cotton + reactive dye + Cationisation + thermofixation	2.83	Dyed cotton + direct dye + Cationisation + thermofixation	9.93	Dyed cotton + acid dye + Cationisation+thermofixation	1.57
Dyed cotton/wool + reactive dye + without cationisation	0.70	Dyed cotton/wool + direct dye + without cationisation	2.24	Dyed cotton/wool + acid dye + without cationisation	0.75
Dyed cotton/wool + reactive dye + Cationisation + steaming	4.69	Dyed cotton/wool + direct dye + Cationisation + steaming	5.73	Dyed cotton/wool + acid dye + Cationisation+steaming	2.41
Dyed cotton/wool + reactive dye + Cationisation + thermofixation	4.40	Dyed cotton/wool + direct dye + Cationisation + thermofixation	5.37	Dyed cotton/wool + acid dye + Cationisation+thermofixation	1.99

Fig. 2: Design applications of the new technique on cotton and cotton/wool fabrics using the three dyes all with optimum conditions.

3.3. Influence of cationisation on nitrogen content:

Cationic textile fabrics modified with cationic reactants contain amino compounds. The main objective of amination is making cellulose more substantive to anionic dyes and enabling much less electrolyte and non-alkaline fixation conditions to be adopted for anionic dyeing. The great majority of processes involve increasing the nitrogen content (i.e. the basicity) of the cellulose, there being an analogy here with the better dye-sorption properties of wool that naturally contains such built-in nitrogen (Shore, 2002). The reaction mechanism of cationic agents with cellulose fibres is similar to that of reactive dyes. Therefore, depending on the pretreatment conditions, some of the cationic reagent will react with water and produce a hydrolyzed compound that cannot react with the fibre. The percentage of nitrogen content on cationised fibres is a measure of the concentration of the cationic reagent attached to them (Kanik and Hauser, 2004). Table 2 illustrates the effect of cationisation using 100 g/kg Sera Fast C-B on nitrogen content of both fabrics.

The table reveals that, cationisation of both fabrics results in increasing nitrogen content regardless of the applied fixation method after cationisation.

Table 2: Effect of cationisation on nitrogen content percentage of cotton and cotton/wool substrates.

Substrates Status	Nitrogen Content %
Cotton + without cationisation	0.04
Cotton + cationisation + steaming	0.29
Cotton + cationisation + thermofixation	0.38
Cotton/wool + without cationisation	0.20
Cotton/wool + cationisation + steaming	0.69
Cotton/wool + cationisation + thermofixation	0.74

3.5. Tensile strength and fastness properties:

Table 3 summarizes the effect of cationisation using 100 g/kg Sera Fast C-B on tensile strength of both fabrics dyed with the three dyestuffs.

Table 3: Effect of cationisation on tensile strength of cotton and cotton/wool substrates dyed with anionic dyes.

Substrates Status	Tensile strength		Substrates Status	Tensile strength	
	Tenacity kg	Elongation %		Tenacity kg	Elongation %
Dyed cotton + reactive dye + without cationisation	39.98	23.75	Dyed cotton/wool + direct dye + without cationisation	36.45	15.25
Dyed cotton + reactive dye + Cationisation + steaming	31.34	20.75	Dyed cotton/wool + direct dye + Cationisation + steaming	41.99	16.00
Dyed cotton + reactive dye + Cationisation + thermofixation	44.46	25.00	Dyed cotton/wool + direct dye + Cationisation + thermofixation	47.64	16.25
Dyed cotton/wool + reactive dye + without cationisation	45.50	21.50	Dyed cotton + acid dye + without cationisation	42.79	16.75
Dyed cotton/wool + reactive dye + Cationisation + steaming	44.59	18.51	Dyed cotton + acid dye + Cationisation+steaming	38.35	17.50
Dyed cotton/wool + reactive dye + Cationisation + thermofixation	31.83	18.50	Dyed cotton + acid dye + Cationisation+thermofixation	37.34	20.50
Dyed cotton + direct dye + without cationisation	37.30	20.75	Dyed cotton/wool + acid dye + without cationisation	37.60	20.25
Dyed cotton + direct dye + Cationisation + steaming	30.36	20.35	Dyed cotton/wool + acid dye + Cationisation+steaming	38.35	17.60
Dyed cotton + direct dye + Cationisation + thermofixation	43.80	22.75	Dyed cotton/wool + acid dye + Cationisation+thermofixation	37.79	16.75

It is observed from the table that, slight changes in tensile strength occur in cationized substrates regardless of both: kind of substrate and the dye used. Moreover, the overall fastness ratings in terms of rubbing, washing and perspiration of cotton and cotton/wool fabrics uncationized as well as cationized with 100 g/kg Sera Fast C-B via printing (using steaming and thermofixation) and dyed with 2 % (owf) using reactive, direct and acid dyes are listed in Table 4.

Table 4: Fastness properties of cationized versus uncationized cotton and cotton/wool substrates dyed with the three anionic dyes.

Substrate Status	Rubbing		Washing		Perspiration			
					Acidic		Alkaline	
	Dry	Wet	St.	Alt.	St.	Alt.	St.	Alt.
Dyed cotton + reactive dye + without cationisation	4-5	3-4	4-5	4-5	4-5	4-5	4-5	4-5
Dyed cotton + reactive dye + Cationisation + steaming	4	3	4-5	4-5	4-5	4-5	4-5	4-5
Dyed cotton + reactive dye + Cationisation + thermofixation	4	3	4-5	4-5	4-5	4-5	4-5	4-5
Dyed cotton/wool + reactive dye + without cationisation	4	4	4-5	4-5	4-5	4-5	4-5	4-5
Dyed cotton/wool + reactive dye + Cationisation + steaming	3-4	3	4-5	4-5	4	4	4-5	4-5
Dyed cotton/wool + reactive dye + Cationisation + thermofixation	3-4	3	4	4	4	4	4	4
Dyed cotton + direct dye + without cationisation	4-5	4-5	3-4	3-4	3-4	4	4-5	4-5
Dyed cotton + direct dye + Cationisation + steaming	4	3-4	3-4	4	3-4	4	4	4

Dyed cotton + direct dye + Cationisation + thermofixation	4	3-4	3-4	4	3-4	4	4	4
Dyed cotton/wool + direct dye + without cationisation	4-5	4-5	4	4	4	4	4-5	4-5
Dyed cotton/wool + direct dye + Cationisation + steaming	3-4	3-4	4-5	4-5	4-5	4-5	4-5	4-5
Dyed cotton/wool + direct dye + Cationisation + thermofixation	3-4	3-4	4	4	4	4	4-5	4-5
Dyed cotton + acid dye + without cationisation	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5
Dyed cotton + acid dye + Cationisation + steaming	4-5	4	4-5	4-5	4-5	4-5	4-5	4-5
Dyed cotton + acid dye + Cationisation + thermofixation	4	4	4-5	4-5	4-5	4-5	4-5	4-5
Dyed cotton/wool + acid dye + without cationisation	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5
Dyed cotton/wool + acid dye + Cationisation + steaming	4	3-4	4-5	4-5	4-5	4-5	4-5	4-5
Dyed cotton/wool + acid dye + Cationisation + thermofixation	4-5	4	4-5	4-5	4-5	4-5	4-5	4-5

St. = Staining on cotton, Alt. = Alteration

It can be concluded from the table that, fastness ratings in terms of washing and perspiration for colour change and staining range from very good to excellent. Concerning colour fastness to rubbing, fastness levels range from good to very good which is may be referred to the higher dye concentration and higher surface coloration (lower penetration) on cationic fibres.

4. Conclusion:

Sera Fast C-B is a commercial cationic reactant that is incorporated alone (100 g/kg) with a thickening agent to print 100% cotton as well as 70/30 cotton/wool substrates. Cationic reactant concentration should not exceed the former concentration to maintain both: tensile strength and fastness levels of the cationized substrate. Steaming or thermofixation are applied afterwards at 110 °C for 10 min. followed by washing off in order to remove the thickener film that causes harsh handle and uneven coloration. The objective of the curing step is to allow penetration of the cationic reactant into fibres prior to the washing step. Cationized substrates are then dyed with 2% (owf) Levafix Turquoise Blue PN-G reactive dye at pH 9 and 7 for cotton and cotton/wool substrates, respectively. Dyeing procedure lasts for 60 min at temperatures of 90 and 75 °C for both substrates, respectively. Attraction of dye particles towards printed areas can easily be observed at the beginning of dyeing procedure. All prints are characterized by brilliant colours and sharp outlines besides the occurrence of no bleeding. Traditional cationisation using the padding technique results in omitting cationic charges all over fabrics which leads to bleeding after printing and washing. This novel technique also guarantees energy reduction due to achieving a union shade on a bicomponent fabric (cotton/wool) using one class of dye as well as minimizing electrolyte addition and its easy application. Furthermore, several design applications have been carried out using reactive, direct and acid dyes. All measurements that may enrich discussion are investigated, i.e. colour yield, nitrogen content percentage, tensile strength and fastness properties. Nitrogen content measurement reveals that, cationisation results in increasing the nitrogen content in cotton substrate which enhances its affinity to anionic dyes. Moreover, cationisation leads to neglectable changes in tensile strength besides, prints of both fabrics are characterized by very good fastness ratings.

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