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One-bath process of enzymatic pretreatment and trichromatic reactive dyeing of viscose fabric

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The viability of reactive dyeing in the same bath without draining the liquor after enzymatic bio-treatment has been investigated. The comparative impact of applying the one-bath bio-treatment and dyeing method using two sets of commercially available polyfunctional and bifunctional trichromatic reactive dyes has been studied and the findings are compared with the two-bath conventional process. Three dyeing recipes in Maroon, Olive and Brown colour combinations of dyed fabrics are evaluated with respect to the colour strength, colourimetric data and fastness properties. The produced one-bath bio-treated and dyed samples show fairly high colour strength and satisfactory colour difference values as compared to the conventional method. The polyfunctional trichromatic reactive dye class is found more sensitive to the type of pretreatment and dyeing method used, imparting slightly higher colour difference values as compared to those of the bi-functional reactive dye class. The design and application of the one-bath Bio-method could provide a significant approach for demanding viscose fabric pretreatment and trichromatic reactive dyeing at reduced time, water and energy parameters.

Keywords: Enzymatic pretreatment, Fastness properties, One-bath process, Trichromatic reactive dyes, Viscose fabric

1 Introduction

Nowadays, the exhaust reactive dyeing of regenerated cellulosic textiles represents one of the major sectors of textile coloration process[1-7]. Among this process, reactive dyes, in particular, are characterized by the necessity of using large amounts of sodium sulphate or sodium chloride and alkali to exhaust and fix the dye molecules onto cellulosic fibres[8-12]. However, this exhaust dyeing method could lead to substantial environmental impact with higher consumption of water, energy, chemicals and emission of large dye effluents[13-19]. Therefore, it is necessary to achieve a more favorable exhaust process with satisfactory levels of productivity and process optimization concerns, especially in shortening the dyeing time, saving chemicals and care of the residual amounts of salt and alkali in the dyebath effluents that badly affect the environment[20-23]. It has been reported that the enzymatic pretreatment of raw viscose fabric with an optimized cold pad-batch[20] and exhaust reactive dyeing[5] in one-bath process have greatly saved chemicals, time and energy, and reduce the environmental pollution. To make the one-bath exhaust process industrially viable, it is necessary to study the effects of enzymatic pretreatment of raw viscose fabric on the subsequent dyeing in one-bath exhaust process with different reactive dye mixtures. With the rapid development of reactive dyes, trichromatic dyes are generally introduced as a mixture of dyes that behave like a single dye at the same salt and alkali concentration, temperature and time[13, 24, 25], contributing to high reactive dyeing performance. Moreover, a process that promotes high dyeing performance with reduced consumption of water, chemicals, energy and total dyeing time is urgently desired with the aim of cleaner reactive dyeing of regenerated cellulose fabrics. In addition, the development in processing these dyes using one-bath process as an alternative to the conventional two-bath processing of cellulosic fabrics is still of special interest, because it may decrease the operating costs and demand high productivity.

In this study, considering that the raw viscose fabric is conventionally dyed using two-bath process, a concurrent pretreatment and dyeing raw viscose fabric are usually adopted using the exhaustion method on jet machines not using the continuous pad

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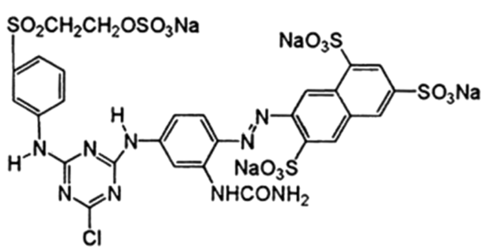
batch method, making it much relevant and more challenging to reduce the high consumption of water for the batchwise technique. A facile and efficient one-bath enzymatic pretreatment and trichromatic reactive dyeing has been studied as an alternative method for dyeing viscose fabric that simultaneously reduces the water and energy consumption. The dyeing performance of two sets of commercially available trichromatic reactive dyes is revealed through the comparison of the proposed one-bath process with the two-bath conventional method. Dyeing is performed by mixing trichromatic dyes of the same polyfunctional and bifunctional reactive groups for estimating the feasibility of this approach. The characterization of dyeing effluents taken after one-bath pretreatment and dyeing method has been compared to the two-bath conventional method. Saving water and processing time of the one-bath Bio-method is also estimated, and the results are compared with the two-bath conventional method.

2 Materials and Methods

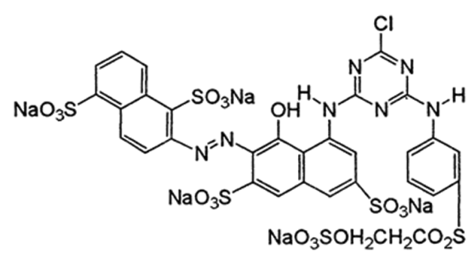
A plain-woven viscose gray fabric (115 g/m^2, 360 ends/10 cm and 280 weft yarns/10 cm) was supplied by Modern Kobba Co. (El-Obour City, Egypt). Two sets of trichromatic commercial reactive dyes [Synozol® Ultra DS polyfunctional dyes (Class A) and Synozol® HF bifunctional monochlorotriazine/vinylsulphone (MCT/VS) dyes (Class B)], supplied by KISCO, Kyung-In Synthetic Company, South Korea, were used for dyeing viscose fabric. The commercial names of all dyes used are shown in Table 1. The available chemical structures of the Synozol® HF dye (class B) is illustrated in Fig. 1. Soda ash (sodium carbonate anhydrous), sodium sulphate anhydrous and glacial acetic acid were supplied by El-Nasr Pharmaceutical Chemicals Company, Egypt. An efficient α-amylase enzyme, namely Bactosol® HPA Liq. was purchased from Archroma, Egypt. A nonionic wetting agent (GB detergent SRM Conc.) and a mild oxidizing agent (Ludigol) were purchased from GB Chemical Products Co., Egypt. Other auxiliaries, viz. sequestering agent (WAT SEQ-10) and APEO free non-ionic wetting/detergent (Detergent WAT-870 ECO), supplied by Al Wataneya for Trade & Chemicals Co., Egypt) as well as anionic leveling agent (Moral 45, Istanbul Chem., Egypt), were used. All chemicals and dyebath auxiliaries were used as received.

Table 1 — List of commercial reactive dyes used in dyeing viscose fabric

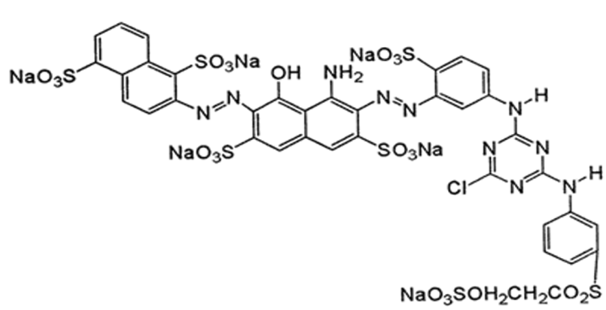
Class	Dye stuff commercial name	Reactive system
A	Synozol Ultra Yellow DS (RY DS) Synozol Ultra Red DS (RR DS) Synozol Ultra Navy DS (RB DS)	Polyfunctional
B	Synozol Yellow HF-2GR 150% C.I. Reactive Yellow 145 (RY 145) Synozol Red HF-6BN 150% C.I. Reactive Red 195 (RR 195) Synozol Navy Blue SHF-BR C.I. Reactive Blue 222 (RB 222)	Bifunctional MCT/VS



C.I. Reactive Yellow 145 (**RY145**)



C.I. Reactive Red 195 (**RR 195**)



C.I. Reactive Blue 222 (**RB 222**)

Fig. 1 — Chemical structures the bifunctional MCT/VS trichromatic Synozol®HF dyes

2.1 Fabric Pretreatment and Dyeing Procedures

Enzymatic pretreatment and dyeing of viscose fabric were carried out on a jet dyeing machine (Thies HT, Germany) at a liquor ratio 10:1. The processing stages were performed by the two-bath conventional method and the enzymatic one-bath Bio-method according to the profiles mentioned in Fig. 2(a) and (b), respectively. The total times (min) assigned for the fabric pretreatment and dyeing methods are given below:

(a) Conventional Method (Two-baths- Total Time 360 min)

Pretreatment stage: One-bath/two stages – Total time 190 min

- Desizing (2 g/L Bactosol® HPA Liq., 2 g/L detergent WAT-870) – 90 min
- Scouring and bleaching (2g/L H_2O_2 50%, 1g/L stabilizer, 1 g/L WAT SEQ-10, 1 g/L GB detergent SRM conc., 2g/L Soda ash) – 70 min
- Cold neutralization (1 g/L acetic acid) /hot and cold rinsing – 30 min

Dyeing stage: *One-bath – Total time 170 min*

- Reactive dyes (% owf) – 10 min
- Levelling agent, Moral 45 (1 g/L), Glauber's salt (45-60 g/L) – 40 min
- Soda ash(15-20 g/L) – 90 min
- Cold neutralization (1g/L acetic acid) /hot and cold rinsing – 30 min

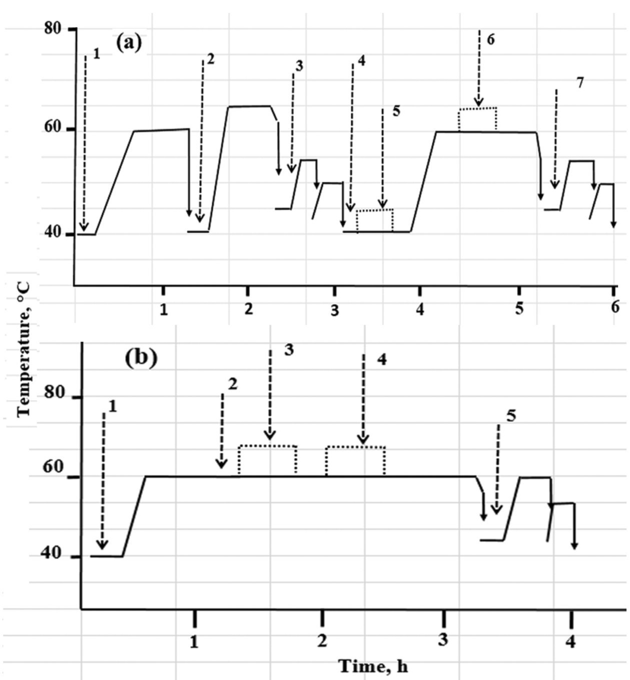


Fig. 2 — Temperature/time profile of (a) two-bath process (conventional method) and (b) one-bath process (Bio-method) for dyeing viscose fabric using reactive dyes

(b) Bio-method (One-bath/two stages - Total time 240 min)

- Bio-pretreatment (2 g/L Bactosol®HPA Liq., 1 g/L detergent WAT-870, 1 g/L GB detergent SRM conc., 2 g/L Ludigol) - 80 min
- Reactive dyes (% owf) – 10 min
- Levelling agent, Moral 45 (1g/L), Glauber's salt (45-60 g/L) – 30 min
- Soda ash (15-20 g/L) – 90 min
- Cold neutralization (1 g/L acetic acid) /hot and cold rinsing – 30 min

In this study, three different trichromatic reactive dye recipes (% owf) using two sets of polyfunctional and bifunctional reactive dye classes A and B, respectively, were examinedas listed in Table 2.

The conventionally and enzymatically pretreated fabrics were dyed in three different dye shades to produce Maroon, Olive and Brown (Table 2). The amounts of sodium sulphate and soda ash used for the three reactive dye batches were used out in accordance to the recipes listed in Table 3.

2.2 Measurements

The colour strength (*K*/*S*) of the dyed samples was determined using UltraScan PRO spectrophotometer with a D65 illuminant and 10° standard observer at the maximum wavelength of each dye in accordance with the Kubelka–Munk equation. The colour readings of all dyed fabrics were also expressed in the CIELAB colour space system (often denoted as L*, a*, b* coordinates); L* represents lightness or darkness of the sample (a higher lightness value represents a lower colour yield), a* denotes redness if positive value or greenness if negative, b* represents yellowness if positive or blueness if negative, and C* specifies chroma and *h*° denotes hue angle.

The total colour difference values (ΔE) of the Bio-method dyed sample as compared to the conventional one were calculated using the following equation:

$$\Delta E = \sqrt{(L^*_1 - L^*_2) + (a^*_1 - a^*_2)^2 + (b^*_1 - b^*_2)^2)} \quad \ldots (1)$$

where L^*_1, a^*_1 and b^*_1 are from bio-method dyed sample and L^*_2, a^*_2 and b^*_2 are from the conventional method dyed sample.

Laboratory analysis of the chemical oxygen demand (COD) of the residual dyebath was carried out in accordance with standard methods for examination of water and waste water[26]. Samples of the residual dyebath were refluxed in strongly acidic solution with known amount of potassium dichromate in the presence of sulphuric acid for 2h at 125 °C.

Table 2 — Colour formulations of commercial reactive dyes used in dyeing viscose fabric

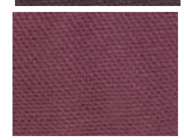
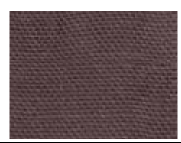
Dye class	Dye	Dye recipes, % owf	Colour	Dyed sample	
				Bio-method	Conventional method
A	RY DS	1	Maroon		
	RR DS	2			
	RB DS	0.4			
	RY DS	1.1	Olive		
	RR DS	0.2			
	RB DS	0.9			
	RY DS	1.4	Brown		
	RR DS	0.9			
	RB DS	1			
B	RY 145	1	Maroon		
	RR 195	2			
	RB 222	0.4			
	RY 145	1.1	Olive		
	RR 195	0.2			
	RB 222	0.9			
	RY 145	1.4	Brown		
	RR 195	0.9			
	RB 222	1			

Table 3 — Concentration of salt and alkali used in dyeing of viscose fabric with different recipes of trichromatic reactive dyes of classes A and B

Dye class	Colour's batch*	Sodium sulphate (Na_2SO_4), g/L	Soda ash (Na_2CO_3), g/L
A	Maroon	50	18
	Olive	45	15
	Brown	50	18
B	Maroon	60	20
	Olive	50	18
	Brown	60	20

*Recipes of reactive dyes used in samples are given in Table 2.

After digestion, the remaining dichromate was titrated with standard ferrous ammonium sulphate to determine the amount of potassium dichromate consumed and the total oxidized organic species calculated in terms of oxygen equivalent.

2.3 Fastness Testing

The dyed viscose fabrics, after washing-off using 2 g/L nonionic detergent at 80°C for 15 min, were tested for fastness in accordance with ISO standard methods. The wash fastness test was assessed according to the standard method ISO 105-C06 B2S:2010 (4g/L of ECE detergent, 1 g/1 of sodium perborate, 25 steel balls) at 50 °C for 30 min and at a liquor ratio of 50:1. Fastness to acidic and alkaline perspiration was evaluated with a perspirometer at specific pressure, temperature and time in accordance with ISO 105-E04:2008. Any change in colour of the dyed specimens (CC) and colour staining on the adjacent cotton (SC) and wool (SW) multi-fibers was then assessed with the corresponding ISO grey scales for colour change and staining. Light fastness was also assessed using a Xenon arc lamp test in accordance with ISO 105-B02:2013.

2.4 Cost Analysis

To validate the developed one-step Bio-method, the cost efficiency of bulk scale production was estimated for pre-treatment and dyeing of 500 kg viscose fabrics with trichromatic reactive dye classes A and B in Maroon shades. Comparative cost savings of time and water for the one-bath Bio-method and two-bath conventional method was calculated according to the total dyeing time and the amount of water used, except the amount used in washing-off the dyed fabrics.

3 Results and Discussion

The *K*/*S* values (Fig. 3) clearly confirm the positive effect of the Bio-method on the trichromatic reactive dyeing of the enzymatically pretreated viscose fabric, if compared with the conventional method. The K/S values of the dyed samples indicate that the trichromatic dyes of the polyfunctional reactive dye (class A) show higher dye uptake than the bifunctional (class B) dye. These findings could be associated with the high efficiency of the polyfunctional dye (class A). As the name implies, the polyfunctional reactive dye chromophore may contain more than two pendant reactive groups corresponding to the bifunctional monochloro-triazine/vinyl sulphone MCT/VS reactive groups in the dye structure of class B. Thus, the polyfunctional dye can react more readily with viscose, imparting much more chance for dye fixation on the viscose fabric through the chemical reaction between the dye polyfunctional reactive system and the fibre hydroxyl groups. Providing these bonds formed encountered to the dyeing of (class B) (Scheme 1), the polyfunctional dye-fibre fixation mechanism will be higher. Interestingly, for all dyed samples produced in Maroon, Olive and Brown shades, the Bio-method developed relatively high performance of dye build-up and colour strength values as compared to those of the conventional method.

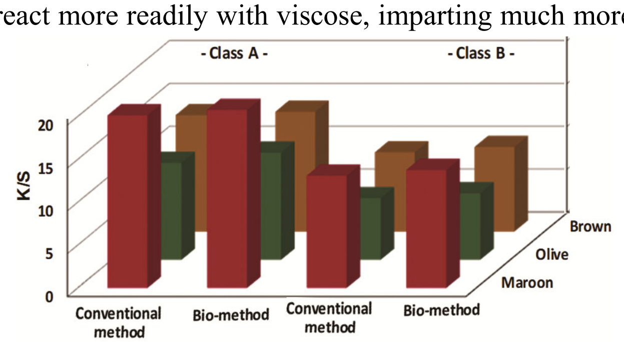
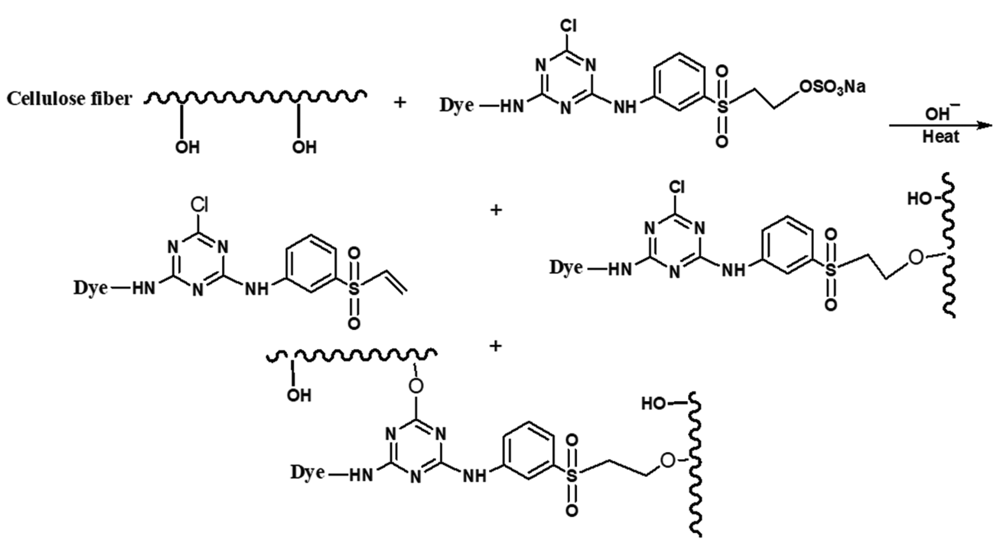


Fig. 3 — Colour strength data of the dyed viscose fabric with the trichromatic reactive dyes of class A and B using the Bio- and conventional methods

Table 4 shows the colourimetric data secured for the three dyed batches in Maroon, Olive and Brown colours applied by both Bio- and conventional methods. The produced bio-treated and dyed samples show satisfactory colour difference values as compared to those of the conventional method. The results also depict that the reactive dye (class A) is more sensitive to the type of pretreatment and dyeing method used; imparting slightly higher colour difference values as compared to those of (class B). This seems reasonable as the reactive dye (class B) having a polyfunctional reactive system, is highly resistant to hydrolysis during the dyeing stage, and the extent of the dye which can react with the fibre thus mainly results in high dye uptake and dye-fibre fixation. Moreover, the enzymatic pretreatment conditions can assist the removal of short fibres, fuzz and spinning oil, which, in turn, improve the water absorption and fabric softness, promoting lower dye aggregation and higher penetration of dye molecules into the vicinity of the substrate during the dyeing stage. It is also observed that the colour differences calculated from the colourimetric data for Maroon,



Scheme 1 — Dye-fibre fixation reaction of bifunctional MCT/VS dyes of class B

Table 4 — Colourimetric data for conventional and Bio-method

Dye class	Colour	Method	*K*/*S*	L*	a*	b*	C*	*h*	ΔE
A	Maroon	Conventional	20.14	25.66	28.79	4.91	29.20	9.69	
		Bio	20.77	25.34	26.43	3.72	26.69	8.01	2.66
	Olive	Conventional	11.3	28.68	-3.86	7.88	8.77	116.12	
		Bio	12.5	28.02	-5.55	5.97	8.15	132.93	2.63
	Brown	Conventional	13.58	22.22	6.05	1.38	6.21	12.85	
		Bio	13.98	21.96	7.83	1.88	8.05	13.49	1.86
B	Maroon	Conventional	13.1	30.44	27.01	3.33	27.21	7.02	
		Bio	13.77	30.70	25.79	2.67	25.93	5.90	1.40
	Olive	Conventional	7.18	36.41	-6.21	8.85	10.81	125.05	
		Bio	7.76	34.80	-6.03	8.08	10.08	126.71	1.80
	Brown	Conventional	9.25	29.27	5.55	4.38	7.07	38.30	
		Bio	9.87	28.86	4.87	3.81	6.19	38.07	0.98

Olive and Brown shades applied using the Bio-method and conventional method show higher values with dye type A on comparison with those of dye B. This is advantageous, as less amount of trichromatic reactive dyes of (class A) would need to be applied in viscose dyeing using Bio-method to achieve a specific *K*/*S* value, thus reduces the production cost and environment pollution by reducing the concentration of dye in the effluent due its high fixation efficiency.

It is also believed that the polyfunctional reactive dyes of type A can further assist the suitability of the Bio-method as compared to the conventional one. However, the polyfunctional reactive groups in these dyes would possibly promote higher dye reactivity and more dye-fibre bond stability, resulting in the polyfunctional dyes being more robust to the conditions of the one-bath method than the bifunctional dyes of type B. While in the conventional method it is necessary to drain the pretreatment bath prior to dyeing, this step is omitted in the case of Bio-method and the starting dyeing temperature is relatively high. Starting the dyeing at 60 ^{o}C could favour more reaction through the polyfunctional reactive group in dye A because of being more resistant to hydrolysis reaction, resulting in higher rate of fixation as compared to the bifunctional dye B.

3.1 COD Measurements

The COD values of the dyeing effluents of both conventional and Bio-methods were further studied to evaluate the environmental impact of the two processes. One of the three dye recipes studied, indicated by Maroon colours of trichromatic reactive dye classes A and B has been investigated. The results of dye effluents for both Bio-and conventional methods are illustrated in Fig. 4. It is observed that the enzymatic one-bath pretreatment and dyeing process causes a significant reduction in COD of the

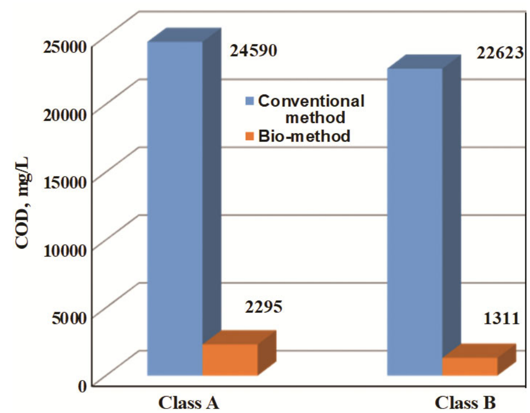


Fig. 4 — COD values of dyeing effluents of both conventional and Bio-methods with maroon recipes of trichromatic reactive dye A and dye B

trichromatic reactive dyeing effluent, which, in turn, imparts much better impact to the environment, as also expected. The dyeing effluent of the Bio-method shows more than 90% reduction in COD values. It is also clear that the dyeing effluent of the Maroon dye recipes formulated by class B exhibits better results of approximately 94% reduction in COD while those of class A shows approximately 91%, inferring that the polyfunctional reactive dye secured lower dyeing effluent as the dye is adequately reacted with viscose fabric. It is anticipated that the compounding of ludigol in the dye bath, acting as a mild oxidizing agent, can convert the desized products of the raw viscose fabric by the enzymatic treatment into gluconic acid and thus lowering the environmental load.

3.2 Fastness Properties

The colour fastness properties of the dyed viscose samples indicate very satisfactory ratings to wash, perspiration and light fastness using the Bio-method (Table 5). The results obtained are found approximately similar to those dyed using conventional method. This

Table 5 — Fastness properties of dyed viscose in three different recipes of trichromatic reactive dye classes A and B using conventional and Bio-methods

Class	Sample	Dyeing method	Wash fastness*			Perspiration fastness*						Light fastness
						Acidic			Alkaline			
			CC	SC	SW	CC	SC	SW	CC	SC	SW	
A	Maroon	Conventional	4	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4
		Bio	4	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4
	Olive	Conventional	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4	4-5	4
		Bio	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4	4-5	4
	Brown	Conventional	4	4-5	4-5	3-4	3-4	3-4	4	3-4	3-4	3-4
		Bio	4	4-5	4-5	3-4	3-4	3-4	4	3-4	3-4	3-4
B	Maroon	Conventional	4-5	4-5	4-5	3	3-4	4-5	4	4-5	3-4	4-5
		Bio	4-5	4-5	4-5	3	3-4	4-5	4	4-5	3-4	4-5
	Olive	Conventional	4-5	5	4-5	4-5	4-5	4-5	4-5	4-5	5	4
		Bio	4-5	5	4-5	4-5	4-5	4-5	4-5	4-5	5	4
	Brown	Conventional	4-5	5	4-5	4-5	4-5	4	4-5	4-5	4-5	4
		Bio	4-5	5	4-5	4-5	4-5	4	4-5	4-5	4-5	4

* CC = Change of colour, SC = Staining on cotton, and SW =Staining on wool.

Table 6 — Total time and water required in processing one-bath Bio- and conventional methods of 500 kg viscose fabrics

Parameter	Conventional method	Bio-method	Savings
Total time, min	360	240	120
Total amounts of water, m^3	30	5	25

indicates that the reactive dyed samples by one-bath Bio-method give the same effect as that of the two-bath conventional method.

3.3 Cost Analysis

The one-bath Bio-method seems to reduce the total cost of viscose dyeing because of the time and water savings as compared to the two-bath conventional method. The savings of costs has been calculated in perspective of El-Kobba Group Co., Egypt.

3.3.1 Processing Time and Amounts of Water Required

As in the one-bath Bio-method, the stages of pretreatment and dyeing performed in the same bath and no water draining is done, it saves time and water. Here, the required time and amounts of water for processing 500 kg fabrics at a liquor ratio 10:1 using one-bath and two-bath methods are listed in Table 6.

A look to both the methods stated in Figs. 2 (a) and (b), the time consumed in the conventional pretreatment processing, bath drainage and re-starting the dyeing cycle relative to the time necessary to carry out the bio-treatment prior to dyeing without bath drainage is potentially reduced from 200 min to 80 min, thereby saving 120 min in the Bio-method. On the other hand, the water consumed in the conventional method is almost five times of the Bio-method, and all the stages, namely desizing, scouring/bleaching and intermediate neutralization/rinsing steps are carried out with recharging the bath in each step with fresh water till the dyeing stage. So, the one-bath Bio-method saves at least 120 min (2 h) of time and 25,000 liters (25 m^3) of water.

3.3.2 Energy Cost Saving for Extra Time

The costs have been calculated by using the following:

- The average electricity rate per hour ≈1.25 LE/1kWh
- Dyeing machine power = 60 kW
- Total electricity cost per hour = 75 LE (approx.)

So, the one-bath Bio-method can approximately save 150 LE the production process, saving 2h time in the processing can effectively maximize the total production.

3.3.3 Cost Saving for Extra Water

The amount of water saved in one-bath bio-method is equal to the difference between the amounts used individually in each method. Amounts of 25,000 liters of water are saved using the Bio-method and the total cost of this extra water amount is estimated as follows:

- Cost of 1.0 m^3water amount ≈ 9 LE (approx.)
- Extra cost of water treatment 1.0m^3≈ 7 LE (approx.)
- Total cost of water used in conventional method = 25 m^3× 16 = 400 LE (approx.)
- Total cost of water used in Bio-method = 5 m^3× 16 =80 LE (approx.)

So, the one-bath Bio-method can approximately save 320 LE (0.64 LE/kg fabric).

It can be concluded that the bio-method offers positive response in terms of ecological and ecofriendly issues, as no water and/or effluent treatments are required for the extra saved 25 m^3 of water.

4 Conclusion

Dyeing of viscose fabric with two classes of trichromatic reactive dyes has been successfully carried out by one-bath Bio-method. A comparative impact of processing three recipes of polyfunctional and bifunctional-based trichromatic shades such as Maroon, Olive and Brown is investigated on the production scale. The produced dyed samples show satisfactory colour difference values on the application of the Bio-method as compared to the conventional method. The polyfunctional reactive dye class is found more sensitive to the type of pretreatment and dyeing method used, imparting slightly higher colour difference values as compared to those of the bifunctional reactive dye class. The enzymatic one-bath pretreatment and dyeing process causes a significant reduction in the COD of the dyeing effluent of trichromatic reactive dyeing with Maroon recipes, imparting much better impact to the environment, as expected. Also, the dyed samples of one-bath Bio-method show the same fastness properties to those obtained by the two-bath conventional method. The one-bath bio-method is also found cost effective and demanding viscose fabric pretreatment and trichromatic reactive dyeing at reduced time, water and energy parameters.

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