USE OF TETRA-SODIUM ETHYLENE DIAMINE TETRA-ACETATE IN PAD-STEAM DYEING OF COTTON WITH REACTIVE DYES

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ABSTRACT

Reactive dyeing of cotton textiles generates high levels of dissolved solids and oxygen demands in the disposed effluent due to the use of inorganic salt (sodium chloride or sodium sulphate) and alkali (sodium bicarbonate, sodium carbonate or sodium hydroxide). Considerable efforts are being undertaken within the textile industry to reduce effluent loads and to comply with environmental regulations. This paper presents results where the inorganic salt (sodium chloride) and alkali (sodium bicarbonate or sodium carbonate) are replaced by a biodegradable organic chemical, tetrasodium ethylene diamine tetra-acetate also known as sodium edate. The dyeing method selected for the study was pad-steam in which the solution containing the dye, the salt and the alkali is applied on cotton fabric by impregnating the fabric in the solution following squeezing the fabric to a designated pick-up of solution and steaming to achieve penetration and fixation of the dye to the fibres within the fabric. The study showed that the colour yield, dye fixation and ultimate colourfastness achieved by using sodium edate were closely comparable to those obtained using inorganic salt and alkali. An industrial trial produced the same findings where the dyeings made with sodium edate produced significant reductions in total dissolved solids, chemical oxygen demand and biochemical oxygen demand of the effluent.

1. INTRODUCTION

Dyeing cotton with reactive dyes is widely practised because the covalent bond that is formed between the fibre and the dye molecules produces excellent colourfastness to washing. However, all reactive dye systems require considerable quantities of inorganic salt (sodium chloride or sodium sulphate) and alkali (sodium bicarbonate, sodium carbonate or sodium hydroxide) to ensure efficient utilisation and fixation of reactive dyes [1-2]. Irrespective of the application method of the reactive dye, almost all of the salt and alkali is discharged to effluent. This creates high levels of dissolved solids and oxygen demands in the effluent, which is environmentally undesirable [3-4].There have been a number of developments for improving the quality of effluent for reactive dyeing of cotton. Development of better dye structures and modified dyeing processes offered considerable reductions in the amount of inorganic salt [5-8]. Cationization of cotton fabric before reactive dyeing has been revealed to be capable of eliminating the use of inorganic salt, and alkali in some instances [9-11]. However, cationization is an additional process step and the practice has yet to be adopted by industry.

Organic salts have been explored and found to be effective alternatives to inorganic salts [12-14]. Ahmed [15] has reported a method for exhaust reactive dyeing of cotton using the organic amine salt, tetrasodium ethylene-diamine-tetraacetate, also known as sodium edate, as an alternative to traditional inorganic salt and alkali. This paper presents findings of a study of the potential for sodium edate as more sustainable substitute to the inorganic salt and alkali in pad-steam dyeing of cotton with reactive dyes. In this study, unlike Ahmed's work, the scoured and bleached fabric was not further boiled with sodium carbonate and non-ionic detergent before dyeing.

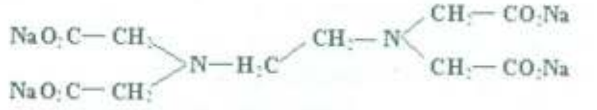

Figure 1: tetrasodium salt of ethylene diamine tetra-acetic acid

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2. EXPERIMENTAL

2.1 MATERIAL

(a) Cotton fabric

A commercially prepared (bleached) 100% cotton woven fabric (280 g/m^2, twill) was used for all dyeings.

The ready-to-dye fabric had an absorbency of 4 sec (AATCC 79 – 1995), pH of 7.7 and a CIE whiteness index of 84.0.

(b) Dyestuff & chemicals

A difluorochloropyrimidine dye, CI Reactive Red 147, and a sulphatoethylsulphone dye, CI Reactive Blue 250, were used. A non-ionic emulsifying detergent (Felosan RGN-S, CHT) was used for washing-off. The sodium chloride, sodium bicarbonate, sodium carbonate and Sodium edate were analytical grade.

2.2 METHODS

(a) Pad-steam dyeing

Fabric samples were dyed (20 g/l dye and the relevant salt and alkali or organic amine salt) by padding (two dip-two nip, BENZ laboratory padder, 70% liquor pick-up). The padded fabrics were then steamed (wet-temperature of 101 – 102°C, 100% moisture, Mathis laboratory steamer) for 60, 90 and 120 sec. For the conventional dyeings, the alkali used was sodium bicarbonate for the difluorochloropyrimidine (Red 147) and sodium carbonate for the sulphatoethylsulphone (Blue 250).

(b) Washing-off

The dyed fabrics were rinsed with cold then hot water, soaped with 2 g/l non-ionic detergent at the boil for 15 min, and then rinsed with hot water until bleeding stopped. The fabrics were finally rinsed with cold water and dried.

(c) Industrial scale dyeing

In order to validate and compare the effectiveness of sodium edate under production conditions two 50 m lengths of bleached cotton woven fabrics (156 g/m^2, plain weave) were dyed (20 g/l of Reactive Red 147) on a Benninger pad-steam range in an industrial dye-house. The new dyeing was carried out using sodium edate (100g/l). The conventional dyeing was carried out with the mill's standard recipe of 30 g/l sodium chloride and 15 g/l sodium bicarbonate. Commercial grade chemicals were used for the trial. The fabric was padded (70% liquor pick-up, ambient temperature), steamed (wet-temperature of 100-102°C, 100% moisture, 60 and 120 sec), washed-off (see Table 1) and then dried.

Table 1: Washing-off conditions on Benninger Dyeing Range

Parameters	Washing tank 1	Tank 2	Tank 3	Tank 4	Tank 5	Tank 6
Temp (oC)	35	80	90	90	90	35
Soaping	-	-	2 g/l detergent	-	-	-
Flow rate (litre/min)	40	30		30		

*slightly anionic (Perlavin RIS, Dr Petry)

2.3 MEASUREMENTS AND ANALYSIS

(a) Colour yield and dye fixation

Colour yield (*K/S* value) was determined using a Datacolor 600 spectrophotometer at the maximum absorption. The specific measurement settings were: 30 mm sample aperture, illuminant D65, UV included, specular component included, reflectance mode and 1964 (10°) CIE Supplementary Standard Observer. Colour yield (*K/S*) was measured as final *K/S* value after washing-off. The approach for determining the extent of dye fixation using *K/S* values, used by other researchers [16-19], was followed. The percentage of reactive dye fixed on the fabric was measured using the equation;

$$\%F = [\,(K/S)\,/\,(K/S_{before\ washing\text{-}off})\,] \times 100. \qquad (1)$$

(b) Colourfastness testing

Fabric samples, dyed and washed-off, were tested for colourfastness to rubbing (AS 2001.4.3 – 1995), to washing (AS 2001.4.15C – 2006) and to light (AS 2001.4.21 – 2006). For comparing colourfastness properties, the shade depth of samples dyed using optimum sodium edate were matched with the samples dyed using optimum conventional chemicals.

(c) Effluent testing

Sodium edate and conventional dyeing effluent samples were collected from washing tanks during the industrial production run and tested for *pH*, *TDS* (Eutech Instruments), *COD* (HACH 8000) and *BOD* (HACH 10099).

3. RESULTS & DISCUSSIONS

As in Ahmed's work [15], sodium edate acts as an electrolyte when dissolved in water and can substitute the inorganic salt for promoting dye exhaustion. Because the amino groups in sodium edate provide a pH of 10 – 12 when dissolved in water, it can also replace the inorganic alkali used to activate dye-fibre reaction. The dye-fibre reaction between dye molecules and cellulose using sodium edate is believed to be same as using an inorganic alkali, i.e. a nucleophilic substitution mechanism in case of difluorochloropyrimidine dye and nucleophilic addition mechanism in case of sulphatoethylsulphone dye [1-2].

Following discussion identifies the optimum chemical concentrations for conventional pad-steam dyeing. Then it presents the effect of sodium edate concentration and steaming time on results of pad-steam dyeing of cotton with reactive dyes. The optimum results are compared with the optimum conventional pad-steam dyeing. The discussion concludes with results of an industrial trial with its effluent analysis.

3.1 OPTIMUM CONCENTRATIONS FOR CONVENTIONAL PAD-STEAM DYEING

Optimum colour yield and dye fixation were obtained with 50 g/l sodium chloride and 15 g/l sodium bicarbonate for CI Reactive Red 147 and 15 g/l sodium carbonate for CI Reactive Blue 250 at 60 sec steaming (Fig. 1).

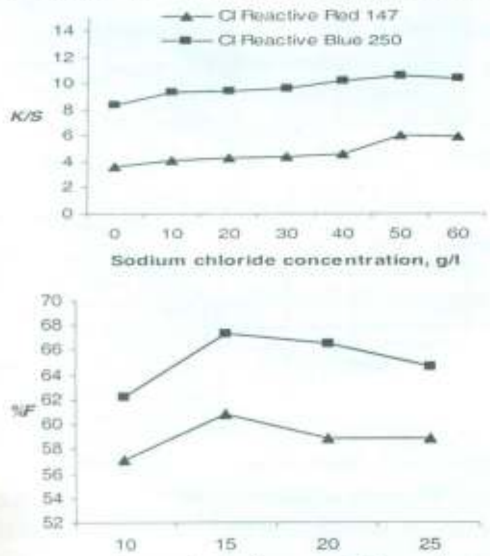

Figure 1: Effect of inorganic salt and alkali concentrations on pad-steam dyeing results

3.2 EFFECT OF SODIUM EDATE CONCENTRATION

Figure. 2 shows that both colour yield and dye fixation increased then decreased with increasing concentration of sodium edate. The optimum result is obtained at about 100 g/l of sodium edate. The optimum concentration of sodium edate was higher than that of inorganic chemicals in conventional dyeing. This is because the aqueous ionic strength of each of the organic salts is lower than that of sodium chloride and sodium bicarbonate / sodium carbonate together; thus, more organic amine salt is needed to achieve the required electrolytic effect for dye sorption [20-21].

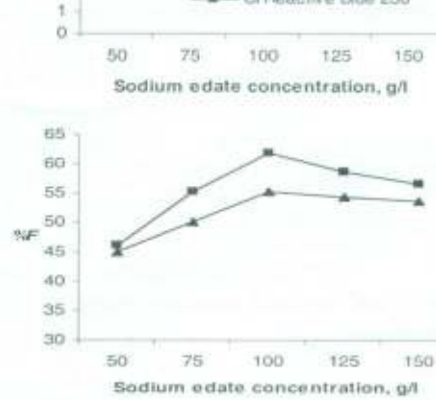

Figure 2: Effect of sodium edate concentration on colour yield and dye fixation at 60 sec steaming

3.3 EFFECT OF STEAMING TIME

Steaming of the fabric padded with reactive dye expedites dye-fibre reaction and promotes dye penetration into the fibre in presence of an electrolyte [22]. The steaming conditions in conventional pad-steam dyeing are always fixed, i.e. temperature of 102 – 104°C and 100% moisture. Therefore, the key process parameter is steaming time. To study the effect of steaming time, the range of up to 120 sec was selected, which is the maximum time for the industrial pad-steam dyeing machines. The effect of steaming time on colour yield and dye fixation with the optimum concentration of sodium edate is given in Fig. 3. The figure shows that colour yield and dye fixation continued to increase with steaming time.

Steaming time to maximum yield and fixation was not determined because the time exceeds standard industry practice.

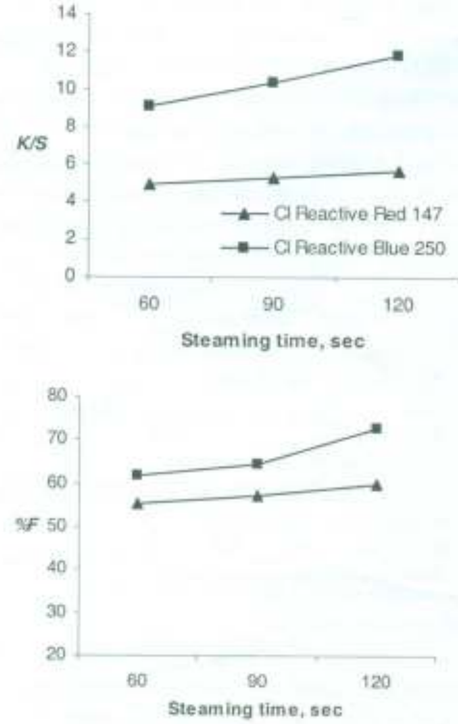

Figure 3: Effect of steaming time on colour yield and dye fixation with optimum concentration of sodium edate

4. COMPARISON BETWEEN SODIUM EDATE AND CONVENTIONAL DYEINGS

(a) Colour yield and dye fixation

The colour yield and the dye fixation of dyed fabric using optimum sodium edate and inorganic chemicals were compared and analysed at 60 and 120 sec steaming. Figure. 4 shows that the yield and fixation results for the sodium edate and conventional dyeings were significantly comparable. Use of sodium edate resulted better at 120 sec steaming where colour yield and dye fixation were effectively identical to those obtained in conventional dyeings.

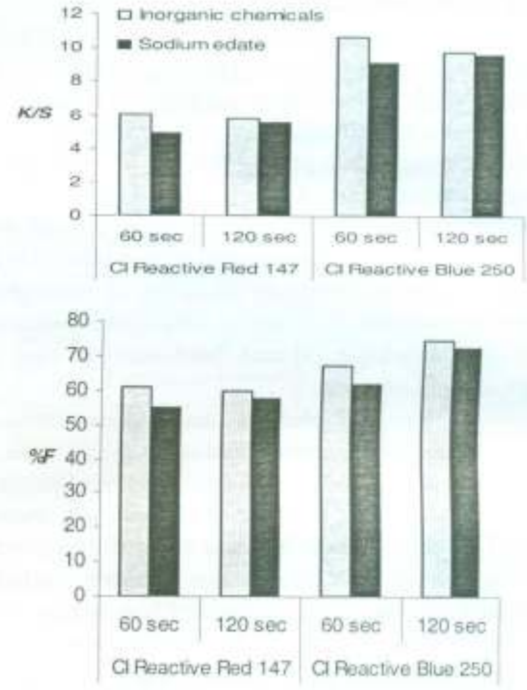

Figure 4: Colour yield and dye fixation of optimum sodium edate and conventional dyeings

(b) Colourfastness

As shown in Table 2, the colourfastness to rubbing, washing and light of the sodium edate dyeings are generally good to excellent and similar to that of the conventional dyeings. This encourages the use of sodium edate for successful pad-steam dyeing of cotton with reactive dyes.

Table 2: Colourfastness of dyed cotton fabrics using optimum chemical concentrations

Dye	Dyeing	Rubbing fastness (Grey scale rating)		Washing fastness (Grey scale rating)		Light fastness
		Dry	Wet	Change in colour	Staining on white*	Blue wool reference
CI Reactive Red 147	Conventional	4-5	4	4-5	4-5	5
	Sodium edate	4-5	4	4-5	4-5	5
CI Reactive Blue 250	Conventional	4-5	4	4-5	4-5	3-4
	Sodium edate	4-5	4	4-5	4-5	3-4

All adjacent white fibres, secondary cellulose acetate, cotton, polyacrylonitrile, polyester, polyamide and wool, had same value (4-5).

5. INDUSTRIAL DYEING RESULTS

Figure. 5 shows the colour yield and fixation obtained with sodium edate were closely equivalent to those obtained with sodium chloride and sodium bicarbonate at 120 sec steaming. The colour yield and fixation achieved under production conditions were higher than those obtained on laboratory scale because the fabric used for production had a lower mass per unit area.

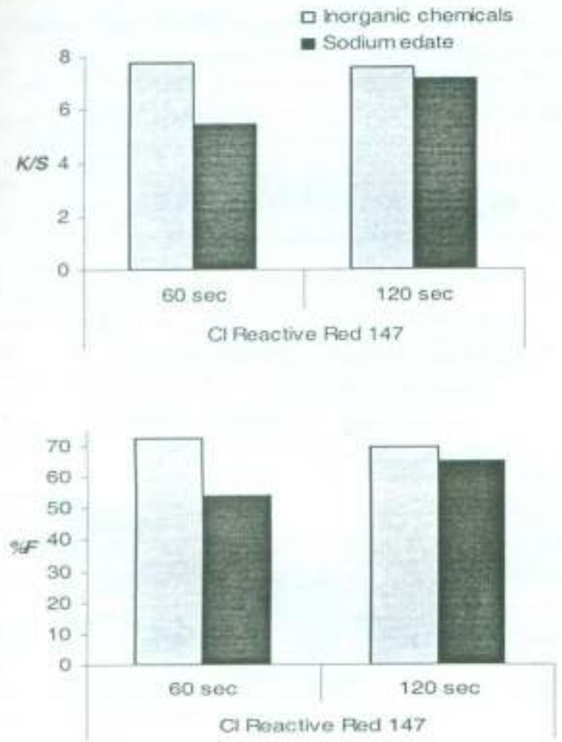

Figure 5: Colour yield and dye fixation of optimum sodium edate and conventional dyeings at production scale

(a) Effluent analysis

The results are shown in Table 3. It can be seen that sodium edate provided a 30% reduction in *TDS* and significantly lower levels of *COD* and *BOD* in the effluent. These results are highly encouraging because the higher cost of sodium edate relative to inorganic chemicals may be offset, in whole or part, by the costs of effluent purification or by load-based penalties on more polluted effluent.

Table 3: Effluent test results

Effluent sample	*pH*	*TDS* (ppm)	*COD* (ppm)	*BOD* (ppm)
Conventional dyeing	8	1000	82	28
Sodium edate dyeing	8	700	15	6

6. CONCLUSIONS

This investigation has shown that tetrasodium ethylene diamine tetra-acetate can be used for pad-steam dyeing of cotton with reactive dyes to improve the quality of dyeing effluent. Colour yield, dye fixation and colourfastness results of sodium edate dyeing are equivalent to those of conventional dyeings. On one hand the proposed dyeing using sodium edate is comparatively more expensive but on other hand the effluent characteristics, when using sodium edate, are markedly improved. Thus, it is suggested that refinements to the use of organic amine salts may lead to opportunities for further reductions in effluent loads from cotton dye-houses.

ACKNOWLEDGEMENT

We very gratefully acknowledge the support of Mr Shehbaz Ali Malik and Mr Sirajuddin Memon, Popular Fabrics Pvt Ltd, Karachi, Pakistan, for permission to conduct the industrial trial in their mill.

REFERENCES

[1] A. D. Broadbent, "Reactive dyes," in Basic principles of textile coloration, A. D. Broadbent, Ed., First ed: Society of Dyers and Colourists, pp. 332-357, 2005.

[2] J. Shore, "Dyeing with reactive dyes," in Cellulosics dyeing, J. Shore, Ed., First ed: Society of Dyers and Colourists, pp. 189-245, 1995.

[3] M. Bide, "Environmentally responsible dye application," in Environmental Aspects of Textile Dyeing, R. M. Christie, Ed., First ed: Woodhead Publishing Ltd, pp. 74-92, 2007.

[4] B. Smith, "Wastes from textile processing," in Plastics & The Environment, A. L. Andrady, Ed., First ed: John Wiley & Sons, Inc., pp. 293 - 295, 2003.

[5] Anon, "Ciba introduces low-salt reactive dyeing," Textile Industries Dyegest Southern Africa, vol. 15, pp. 5-8, 1996.

[6] D. Hinks, et al., "Cationic fiber reactive dyes for cellulosic fibers," AATCC Review, vol. 1, pp. 43-46, 2001.

[7] C. B. Anderson, "Dyeing reactive dyes using less salt," American Dyestuff Reporter, vol. 83, pp. 103-105, 1994.

[8] K. Van Wersch, "Processes for dyeing cellulosic fibres with reactive dyes," International Textile Bulletin. Dyeing/Printing/Finishing, vol. 43, pp. 21-24, 1997.

[9] D. P. Chattopadhyay, et al., "Salt-free reactive dyeing of cotton," International Journal of Clothing Science & Technology, vol. 19, pp. 99-108, 03 2007.

[10] M. S. S. Kannan and R. Nithyanandan, "Salt & alkali free reactive dyeing on cotton," ATA Journal, vol. 17, 2006.

[11] S. Zhang, et al., "Continuous dyeing of cationised cotton with reactive dyes," Coloration Technology, vol. 121, pp. 183-186, 2005.

[12] H. G. Prabu and M. Sundrarajan, "Effect of the bio-salt trisodium citrate in the dyeing of cotton," Coloration Technology, vol. 118, pp. 131-134, 2002.

[13] Y. Guan, et al., "Application of polycarboxylic acid sodium salt in the dyeing of cotton fabric with reactive dyes," Journal of Applied Polymer Science, vol. 105, pp. 726-732, 2007.

[14] J. W. Rucker and D. M. Guthrie, "Salt substitute for dyeing cotton with fiber reactive dyes," Sen-I Gakkaishi, vol. 53, pp. P256-P260, 1997.

[15] N. S. E. Ahmed, "The use of sodium edate in the dyeing of cotton with reactive dyes," Dyes and Pigments, vol. 65, pp. 221-225, 2005.

[16] L. Wang, et al., "Preparation of cationic cotton with two-bath pad-bake process and its application in salt-free dyeing," Carbohydrate Polymers, vol. 78, pp. 602-608, 2009.

[17] H. Wang and D. M. Lewis, "Chemical modification of cotton to improve fibre dyeability," Coloration Technology, vol. 118, pp. 159-168, 2002.

[18] K. Srikulkit and P. Santifuengkul, "Salt-free dyeing of cotton cellulose with a model cationic reactive dye," Coloration Technology, vol. 116, pp. 398-402, 2000.

[19] D. M. Lewis and E. L. Gillingham, "Fixation of aspartyl-triazine dyes on cellulosic fibers," Textile Chemist and Colorist, vol. 28, pp. 76-77, 1996.

[20] G. E. Krichevskii and I. M. Movshovich, "Use of methods of heterogeneous chemical kinetics and nonequilibrium thermodynamics for the quantitative evaluation of the rate of fixation of reactive dyes by fibrous materials," Textile Research Journal, vol. 40, pp. 793-802, 1970.

[21] J. A. Dean, "Electrolytes, Electromotive Force, and Chemical Equilibrium," in LANGE'S HANDBOOK OF CHEMISTRY, J. A. Dean, Ed., Fifteenth ed: McGRAW-HILL, INC., 1999.

[22] K. Hunger, "Textile dyeing," in Industrial dyes, K. Hunger, Ed., Third ed: Wiley-VCH, pp. 339-357, 2003.