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Use of Biodegradable Organic Salts for Pad-Steam Dyeing of Cotton Textiles With Reactive Dyes to Improve Process Sustainability

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Abstract. Cotton textiles are dyed mostly with reactive dyes because they produce a wide gamut of bright colours with excellent colourfastness to washing. However, the reactive dyeing requires considerable quantities of inorganic salt and alkali for efficient utilization and application of dyes. These salts and alkalis when drained to effluent generate heavy amounts of total dissolved solids leading to environmental pollution. Substantial remedies are being considered within the textile processing sector to reduce the effluent pollution and to fulfill the environmental regulations. This paper is a part of such efforts and presents results where any of the three biodegradable alkaline polycarboxylic sodium salts, tetrasodium ethylene diamine tetra-acetate (namely sodium edate), trisodium nitrilo triacetate (namely trisodium NTA) and tetrasodium N,N-*bis*(carboxylatomethyl)-L-glutamate (namely tetrasodium GLDA), can be used as alternative to traditionally used nonbiodegradable inorganic salt and alkali in the dyeing solution. Two widely used dyes, CI Reactive Red 147 and CI Reactive Blue 250, and pad-steam dyeing method were used in this study. The dyeing was carried out by impregnating the cotton fabric in the solution containing the dye, salt and alkali, and squeezing it to a 70% pickup, followed by steaming for dye penetration and fixation to the fibres. It has been found in this study that the ultimate colour-yield and colourfastness properties obtained by using the biodegradable organic salts were closely comparable to those obtained with inorganic salt and alkali. A significant increase in the colour-yield was obtained when tetrasodium GLDA was used for CI Reactive Blue 250. The dyeing effluent showed reductions in total dissolved solids content with sodium edate and trisodium NTA.

Keywords: Biodegradable organic salt, dyeing, cotton textiles, reactive dyes

1. Introduction

The textile colouration and finishing industry is one of the major contributors to environmental pollution[1, 2]. This is mainly due to the discharge of nonbiodegradable inorganic salts, alkalis, other processing aids and organic matter such as dyes to the effluent. The effluent treatment can play a significant role in reducing discharge pollution. However, these treatments are expensive and produce highly concentrated solid wastes[3]. Therefore, the better approach would be to improve the textile processing technologies and chemistry for reducing the discharge pollution.

Reactive dyes have become a default choice for colouration of cotton textiles, because they provide a wide range of inexpensive bright colours with excellent washing fastness[4]. The excellent washing fastness is due to the covalent bonding between the fibre polymers and the dye molecules under alkaline pH conditions [5]. The inorganic salt, such as sodium chloride or sodium sulphate, for dye transfer to and penetration into the fibre, and inorganic alkali, such as sodium bicarbonate, sodium carbonate or sodium hydroxide, for dye-fibre reaction, are required in substantial quantities to accomplish the dyeing process. Irrespective of the dyeing method using reactive dyes, just about all of the salt and alkali is drained to effluent. Such effluents are characterised by high levels of dissolved solids which is environmentally

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undesirable [2, 5-7]. A number of developments have been proposed for reducing the effluent pollution by cotton dyeing systems with reactive dyes.

Developments in dye structures, dyeing processes and machinery have offered reductions in the amount of inorganic salt and improved dye fixation [8-14]. Cationisation of cotton fibre before dyeing process has been proved to be capable for abolishing the requirement of inorganic salt, and also alkali in few cases [15-19]. Nevertheless, cationisation is an extra process and has yet to be taken on to industrial level. Organic chemicals have been shown to be meaningful substituent to inorganic salts in exhaust dyeings [20-23]. Tetrasodium ethylene diamine tetraacetate (sodium edate) is a biodegradable alkaline polycarboxylic salt and has been studied as an alternative to inorganic salt and alkali in reactive dyeing of cotton by exhaust method[24]. However, the high alkalinity of sodium edate can cause reactive dye hydrolysis in the dyebath during dye exhaustion. This can result in the difficult control of dyeing and reduced dye fixation [22]. The use of the alkaline organic salts in continuous dyeing of cotton with reactive dyes would be of interest.

This paper presents the results of a study where sodium edate and two more alkaline polycarboxylic sodium salts, trisodium nitrilo-triacetate (trisodium NTA) and tetrasodium N,N-bis(carboxylatomethyl)-Lglutamate (tetrasodium GLDA) have been used in the continuous pad-steam dyeing of cotton with reactive dyes to improve effluent quality by replacing the traditional inorganic salt and alkali. These salts are biodegradable and are commonly used in the detergent and the cosmetic industries as chelating agents [25-27].

2. Material and methods

A commercially prepared ready-to-dye cotton woven fabric (282 g/m2) and two widely used commercial reactive dyes, CI Reactive Red 147 and CI Reactive Blue 250, were used for this study. A nonionic detergent, Felosan RGN-S, was used for soaping during washing-off. The sodium edate, trisodium NTA, tetrasodium GLDA, sodium chloride, sodium carbonate and sodium bicarbonate were analytical grade.

Fabric pieces were padded (70% pick-up, Benz padder) with aqueous dyebath having 20 g/l dye and the related salt and alkali or an organic salt. The padded fabric pieces were then subjected to steaming (100% moisture, 101 – 102oC, Mathis steamer) for 60, 90 and 120 sec. In case of traditional dyeing processes, sodium bicarbonate was used for CI Reactive Red 147 and sodium carbonate for CI Reactive Blue 250 as an alkali. To remove unfixed dyes and residual chemicals, the dyed fabrics were rinsed with cold water then with hot water, soaped with 2 g/l Felosan RGN-S at boil (15 min), and then rinse with hot water until dye desorption stopped. Finally, the fabrics were washed with cold water and oven dried.

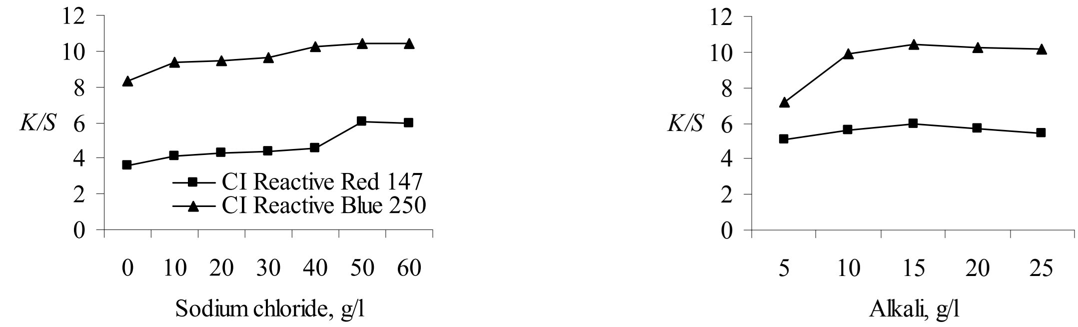
The final fabric samples were tested for colour-yield, K/S value, using a Datacolor 600 spectrophotometer and colourfastness to rubbing (ISO 105 - X12), to washing (ISO 105 - C02) and to light (BS 1006: 1990 UK-TN).

Each optimum dyeing recipe was diluted 100 times as representative of the dyeing effluent. The TDS of the representative dyeing effluent was measured using a Eutech laboratory TDS meter.

3. Results and discussion

3.1. Optimum inorganic salt and alkali

Salt is used for increasing dye levelness throughout the fibre and the resultant colour-yield, in pad-steam dyeing [6, 28, 29]. Fig. 1 (a) shows that the maximum colour-yield was obtained with 50 g/l sodium chloride for 20 g/l dye. The reaction between the reactive dye molecules and the hydroxyl groups of cotton cellulose occur at alkaline pH conditions. The dye can also react with the hydroxide ions in an aqueous dye solution [6, 28, 30]. The excessive amount of alkali can increase dye hydrolysis at the cost of dye-fibre reaction, thus, reducing the ultimate colour-yield. Fig. 1 (b) shows that 15 g/l alkali provided the optimum colour-yield for 20 g/l dye.



(a) Effect of sodium chloride concentration on colour-yield at constant alkali (15 g/l)

(b) Effect of alkali concentration on colour-yield at constant sodium chloride (50 g/l)

Fig. 1: Optimum sodium chloride and alkali (sodium carbonate / sodium bicarbonate) concentrations for 20 g/l dye at 60 sec steaming

3.2. Effect of biodegradable organic salt concentration

Fig. 2 shows the effect of alkaline polycarboxylic sodium salt concentration on colour-yield of the fabric dyed with 20 g/l dye. The yield passed through maximum values with increasing concentration of the salt. For CI Reactive Red 147, the optimum concentration was around 100 g/l of the sodium edate or trisodium NTA and 75 g/l of the tetrasodium GLDA. And for CI Reactive Blue 250, the concentration was 125 g/l of the sodium edate or trisodium NTA and 100 g/l of the tetrasodium GLDA. Lesser concentrations of tetrasodium GLDA were required to achieve optimum yield which was better than that obtained with the other two organic salts. The colour-yield of CI Reactive Blue 250 obtained by using optimum tetrasodium GLDA was significantly higher than that obtained with other organic salts. This is highly significant increase comparing to the possible lower colour-yield in the traditional reactive dyeing of cotton by pad-steam process because of excessive dye hydrolysis and lower ultimate dye-fixation levels [28]. This may be attributed to the higher ionic-strength and better stability (at elevated pH) of tetrasodium GLDA for reaction between a bis(sulphatoethylsulphone) dye and the cotton cellulose.

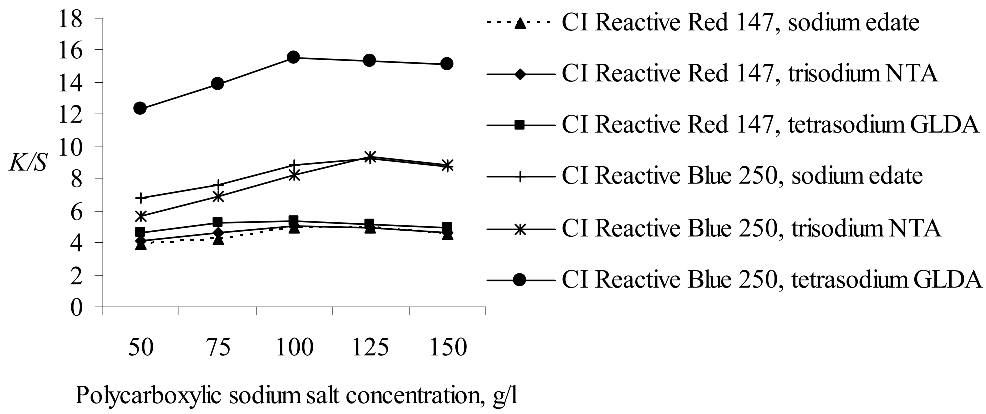


Fig. 2 Effect of alkaline polycarboxylic sodium salt concentration on colour-yield at 60 sec steaming

3.3. Dyeing using organic salt versus conventional dyeing

Comparative effect of steaming time

Steaming time of the range of 60–120 sec was selected. This is because industrial pad-steam dyeing machines generally function within this range. Fig. 3 shows the effect of steaming time on colour-yield having the optimum concentrations of inorganic and organic chemicals. The figure shows that the colour-yields continuously increase slightly with steaming time in all cases. The steaming time to maximum yield at longer steaming time was not determined because the time exceeds standard industry practice.

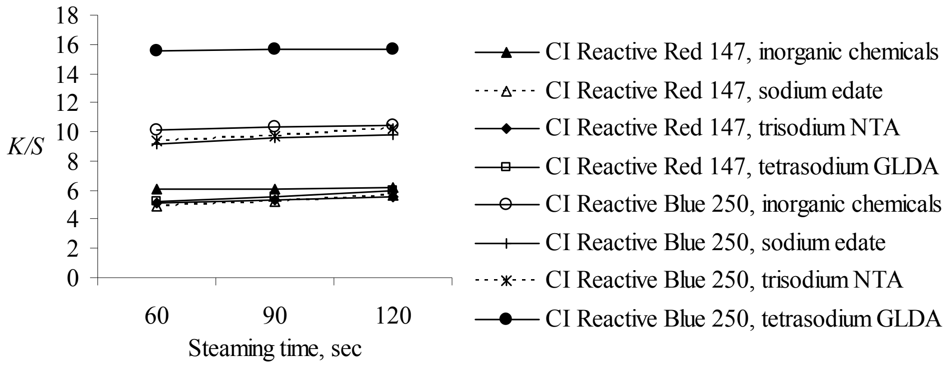


Fig. 3: Effect of steaming time on colour-yield of 20 g/l CI Reactive Blue 250 (20 g/l) at constant alkaline polycarboxylic sodium salts and inorganic chemicals concentrations

Comparative colour-yield and colourfastness

Fig. 4 shows the comparative colour-yields for the alkaline polycarboxylic sodium salts and traditional dyeings. The figure shows that the colour-yield results for the alternate and traditional dyeings were considerably comparable. However, interestingly, the tetrasodium GLDA gave considerably higher yield with CI Reactive Blue 250, as described in an earlier section for Fig. 3. These results obtained by using tetrasodium GLDA for the bis(sulphatoethylsulphone) dye worth a further study.

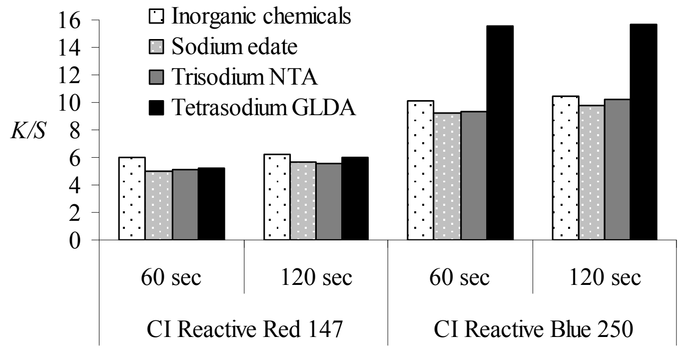


Fig. 4 Colour-yield using optimum traditional salt and alkali, sodium edate, trisodium NTA and tetrasodium GLDA concentrations at 60 and 120 sec steaming

Table 1 shows that the rubbing, washing and light fastnesses achieved by of the organic salt dyeings are generally very good and the same as those obtained by the traditional dyeings. These results are motivating. The lower lightfastness of CI Reactive Blue 250 is same as that notified on the dye manufacturer's literature. The 40 g/l of tetrasodium GLDA (not an optimum concentration) was used for colourfastness testing to have the matching colour-yields with traditionally obtained yields.

Table 1 Colourfastness of cotton fabrics dyed with 20 g/l CI Reactive Blue 250 at 60 sec steaming

Dye (20 g/l)	Chemical concentrations	Colour-yield (*K/S* after washing-off without DMF treatment)	Rubbing fastness		Washing fastness		Light fastness
			Dry	Wet	Change in colour	Staining on white *	Blue wool reference
CI Reactive Red 147	50 g/l sodium chloride 15 g/l sodium bicarbonate	5.85	5	4-5	5	5	6
	100 g/l sodium edate	5.65	5	4-5	5	5	6
	100 g/l trisodium NTA	5.54	5	4-5	5	5	6
	75 g/l tetrasodium GLDA	5.67	5	4-5	5	5	6
CI Reactive Blue 250	50 g/l sodium chloride 15 g/l sodium carbonate	10.34	4-5	4	4-5	4-5	3-4
	125 g/l sodium edate	10.12	4-5	4	4-5	4-5	3-4

	125 g/l trisodium NTA	10.27	4-5	4	4-5	4-5	3-4
	40 g/l tetrasodium GLDA	10.82	4-5	4	4-5	4-5	3-4

* Secondary cellulose acetate, cotton, polyacrylonitrile, polyester, polyamide and wool

Comparative effluent analysis

Table 2 shows that sodium edate and trisodium NTA provided around 24–29% TDS reduction in the effluent of CI Reactive Red 147 and around 8–14% reduction in the case of CI Reactive Blue 250. The lower percent reduction in TDS for CI Reactive Blue 250 is due to the higher concentration of alkaline polycarboxylic salt needed to obtain optimum colour fixation and ultimate colour-yield. Tetrasodium GLDA did not provide any significant reduction in the effluent TDS. However, tetrasodium GLDA is still safer in terms ecological footprint than sodium edate and trisodium NTA principally because of its better biodegradability [27]. Further, it provided significantly higher colour-yields for CI Reactive Blue 250. This means the amount of unfixed dye washed-off to effluent will be reduced significantly.

Table 2 TDS of dyeing effluents

Dye (20 g/l)	Effluent samples (Dyebath recipes diluted 100 times)	TDS (mg/l)
CI Reactive Red 147	50 g/l sodium chloride 15 g/l sodium bicarbonate	1380
	100 g/l sodium edate	1010
	100 g/l trisodium NTA	1040
	75 g/l Tetrasodium GLDA	1370
CI Reactive Blue 250	50 g/l sodium chloride 15 g/l sodium carbonate	1400
	125 g/l sodium edate	1100
	125 g/l trisodium NTA	1230
	40 g/l Tetrasodium GLDA	1350

4. Conclusions

This research has revealed that the biodegradable alkaline polycarboxylic sodium salts, such as sodium edate, trisodium NTA and tetrasodium GLDA, can effectively be used for reactive dyeing of cotton by pad-steam process to reduce the dyeing-effluent pollution. The alternate dyeing system produced colour-yields and colourfastness comparable to those obtained using traditional salt and alkali. Further, increased colour-yield of the CI Reactive Blue 250 was achieved with tetrasodium GLDA. This was a significantly higher increase in colour-yield with no change in the colourfastness of dyed fabrics comparing to the lower yields obtained traditionally because of lower dye-fixation levels.

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