

Application of Polycarboxylic Acid Sodium Salt in the Dyeing of Cotton Fabric with Reactive Dyes

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ABSTRACT: In this study, the effects of polycarboxylic acid sodium salt on the dyeing of cotton with reactive dyes were evaluated by measuring and comparing the K/S values and dyeing fastnesses of the dyed cotton fabric samples. Results showed that the K/S value and dyeing fastness of cotton fabrics dyed with polycarboxylic acid sodium salt, substituting inorganic salts as exhausting agent were close to that of with sodium chloride when dip-dyeing process was used. While, in pad-dry dyeing, the K/S value of cotton fabric samples dyed with polyacid salts as exhausting agent was higher than that of with sodium sulfate, and the dyeing fastnesses of these samples were nearly the same. The dyeing mechanism of cotton fabric with reactive dye, using poly-

carboxylic acid sodium salt as exhausting agent was analyzed. The dyeing exhausting mechanism of reactive dye seems different when the inorganic salt and polycarboxylic acid sodium salt were used as exhausting agent in the dyeing of cotton fabric with reactive dye. The polycarboxylic acid sodium salt, as weak electrolyte, increased the dye-uptake of reactive dye on cotton fabric not only by screening negative charges on cotton surface, but also by the effect of salting-out or hydrophobic combination. © 2007 Wiley Periodicals, Inc. *J Appl Polym Sci* 105: 726–732, 2007

Key words: polycarboxylic acid sodium salt; cotton fabrics; reactive dye; low salt dyeing

INTRODUCTION

The increasing awareness of environmental protection drives dyer to explore new eco-friendly textile processing techniques. In dyeing process, large amount of inorganic salts are used to increase the dye-uptake of reactive dye, but the inorganic salts in dyeing wastewater are difficult to be biodegraded¹ and leads to the basification of soil.²

Dyers have made great efforts to decrease the amount of inorganic salts used in dyeing process of textiles for a long time. As far as the dye structure is concerned, Ciba Special Chemicals developed some new reactive dyes such as Cibacron F monofluorotriazine, Cibacron LS difluorotriazine, Cibacron C monofluorotriazine, and vinyl sulfone reactive dye etc. These dyes exhibit many good dyeing properties, for examples, they can be used in low-salt, low-alkali dyeing, and have high fixation.^{3–5} But these new type of dyes are expensive compared with the normal reactive dyes used by interpreters.

The experimental results of S.M. Burkinshaw, Peter J. Hauser, and coworkers showed that the cotton pre-treated with cationic surfactants could be dyed with

reactive dye without using inorganic salts as exhausting agent.^{6,7} But the drawback of this method is likely to lead the uneven of dyeing and the increase of pre-treating process.

In recent years, many dyeing workers tried some biodegradable and non poisonous dyeing additives to substitute inorganic salts as exhausting agent in dyeing of cotton with reactive dye. Nahed S.E. Ahmed used Sodium salt of Ethylenediamine tetraacetic Acid (sodium edate) as exhausting agent in dyeing of cotton with reactive dye.⁸ Unfortunately, the high alkalinescence of sodium edate would make reactive dyes to hydrolyze in dyeing bath, which results in the dyeing condition hard to control and the fixation ratio of dye to decrease. Moreover, not only some dyes are easy to change their hue due to the chelate effect of the sodium edate (for example C. I. Reactive Blue 74), but also sodium edate is too costly to be employed in dyeing of fabrics extensively. Meanwhile, the use of sodium edate is forbidden in European Union because of the toxicity of its degraded products.⁹ Gurumallesh Prabu et al. adopted trisodium citrate as exhausting agent and good dyeing effect was obtained,¹⁰ unfortunately it was also costly for fabric dyeing.

With the development of polymer science, polymer technology and products have been applied in textile industry extensively. In addition of textile materials,

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polymer was also used as auxiliary in dyeing and finishing processes. Polyacrylic acid, polyacrylate, polyacrylamide, and polyvinyl alcohol are used as size in warp sizing.^{11,12} Polyacrylate and poly(ethylene oxide) alkyl ether are employed in fabric printing as paste thickener.^{1,13,14} In the finishing process of textile, polymer such as polysiloxane, polyethylene emulsion, polyurethane, polyurethane/polyaniline, and poly(ethylene oxide) are applied as finishing auxiliary to improve the quality or performance of fabric.¹⁵⁻¹⁷

Because polycarboxylic acid sodium salt have many advantages such as low poisonous, cheap, easy to get, and biodegradable.¹⁸ They have been used as free-formaldehyde antcrease agent, antimigrating agent, paste thicker and nonfoam washing agent in fabric dyeing, and finishing processes.¹⁹ But reports about polycarboxylic acid sodium salt used in fabric dyeing is seldom found. This study is aimed at investigating the possibility of using polyacrylic acid sodium salt (PA) and polymaleic acid sodium salt (PM) to substitute inorganic salts as exhausting agents in the dyeing of cotton fabric with reactive dyes. For this purpose, K/S values and dyeing fastnesses of the dyed cotton samples were measured and compared. The dyeing exhaustion mechanism of reactive dye was also analyzed, when polyacid salts was used as exhausting agent in the dyeing of cotton fabric with reactive dye.

EXPERIMENTAL

Materials

Scoured and bleached, fluorescent brighter-free woven cotton fabric (150 g/m²) was used in the experiment.

Commercial monochlorotriazine reactive dyes as listed in Table I, were used without further purification. Sodium hydroxide, sodium chloride, sodium sulfate, and sodium carbonate were A.R. grade. PA sodium salt and PM sodium salt were synthesized in laboratory according to the references.^{20,21} PA and PM solution were cooled to room temperature and 30% sodium hydroxide solution was added dropwise under stirring, and the neutralization was allowed to proceed until a constant pH 7 ± 0.1 was obtained. The concentration of PA sodium and PM sodium was 50.3 and 47.9% respectively.

Dyeing

The dip dyeing process was depicted in Figure 1. The pH value of the dyeing bath was adjusted to 10.5 ± 0.1 by adding sodium carbonate solid.

In pad-dry dyeing process, cotton fabric was padded by the laboratory pad mangle under the condition of room temperature, liquid ratio of 10 : 1, pickup 80%, concentration of the dye 20 g/L, and concentration of sodium carbonate 20 g/L. Then the padded cotton

TABLE I
The Structures of Dyes

Commercial name	C.I. generic name	Structure
Reactive Yellow K-4G	C.I. Reactive Yellow 18	
Reactive Red K-2G	C.I. Reactive Red 15	
Reactive Blue K-3R	C.I. Reactive Blue 74	

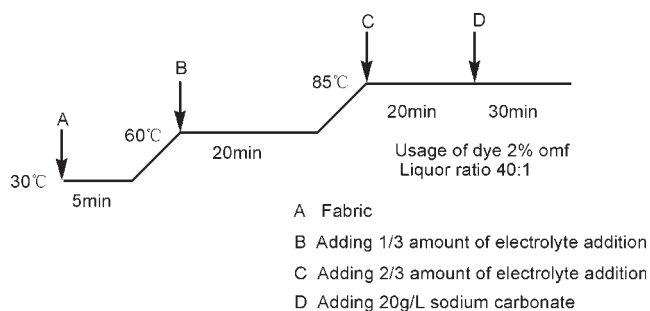


Figure 1 Dip dyeing method.

fabric was predried at 80°C for 3 min and baked at 160°C for 100 s. Finally the dyed cotton was washed in 2 g/L nonionic detergent (liquor ratio 20 : 1) at 95°C for 15 min, then washed thoroughly and dried.

Measurement

Color strength

The reflectance of dyed samples was measured with the Reflectance Spectrophotometry (Spectra Flash SF450, Data Color) under the illuminant D_{65} using 10° standard observer. The color strength of dyed samples was expressed with K/S value calculated by the Kubelka–Munk equation.

Dyeing fastness

The rubbing fastness of dyed samples was tested according to ISO 105-X12: (1993). The washing fastness of dyed samples was tested according to ISO 150-C02: (1989).

Specific conductance

The specific conductance of electrolyte solution was tested at the different temperatures such as of 30, 60,

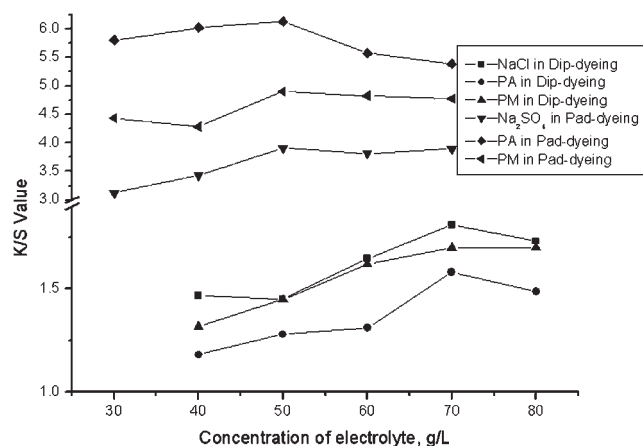


Figure 2 Effect of electrolyte concentration on the K/S value of the sample dyed with C.I. Reactive Yellow 18.

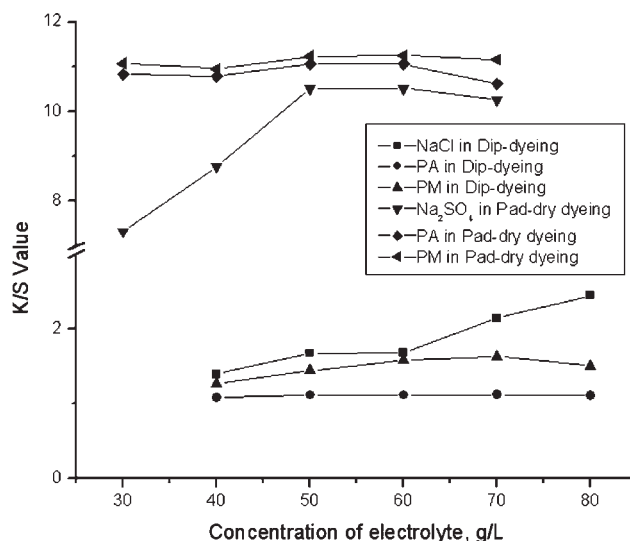


Figure 3 Effect of electrolyte concentration on the K/S value of the sample dyed with C.I. Reactive Red 15.

and 85°C respectively, with DDS-A specific conductance instrument (Chengdu Wuke instrument, China).

Surface tension

The surface tension was measured with Sigma 703 surface tension instrument (KSV instrument, Finland). The solution of surfactant and electrolyte was put into a cleaned polytetrafluoroethylene circular shape container with an apparent area of 50.26 cm², and the half-dry hanging slice was put in the solution just contacting with the solution. The surface tension value showed on the instrument was recorded.

Viscosity

The flow time was used to evaluate the viscosity of solution. The flow time of solution was measured by a thoroughly cleared and dried using Ubbelohde viscometer with a capillary length of 110.0 mm and diameters of 0.30 mm in a water bath of (30 ± 0.01)°C. The flow time was recorded using a topwatch with an accuracy of 0.01 s. Three parallel experiments for each concentration have to be done for the determination of viscosity data.²²

RESULTS AND DISCUSSION

Effect of electrolyte concentration on the K/S value of the sample

The K/S values of dyed cotton fabric changed with the increase of the electrolyte concentration in dye bath as showed as the curves in Figures 2–4. The K/S values of cotton fabrics dyed by pad-dry dyeing process were higher than that of cotton fabrics dyed by

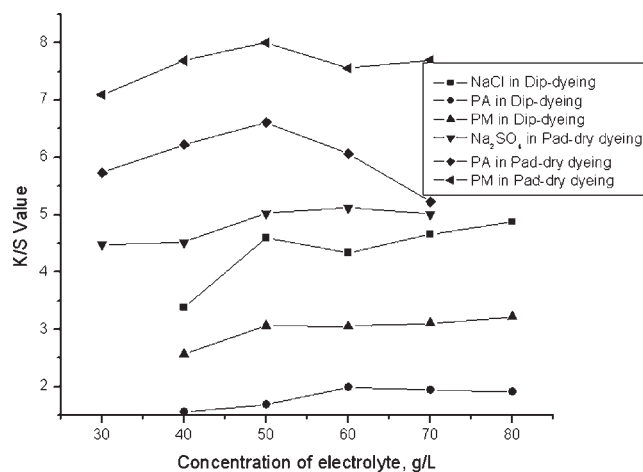


Figure 4 Effect of electrolyte concentration on the K/S value of the sample dyed with C.I. Reactive Blue 74.

dip-dyeing process. And the K/S values of these samples showed a maximum value with the concentration of electrolyte increasing. After the maximum K/S value, a negative increase of K/S value was observed with the concentration of electrolyte continual increasing.

In dip dyeing, because PA and PM as weak electrolytes could not ionize completely in dye bath to provide enough sodium ions to screen the negative charges on the surface of cotton, the K/S values of dyed cotton samples both with PA and PM as exhausting agents were a little lower than that of dyed cotton samples with sodium chloride. In pad-dry dyeing, the K/S values of dyed cotton samples with PA and PM as exhausting agent were higher than that of dyed cotton samples with sodium sulfate as exhausting agent, because the added PA or PM was intended to increase the viscosity of the pad-dry dyeing bath (data in Table II), which would benefit to reduce the dye migration during baking. But, there was an optimum polyelectrolyte concentration for the pad-dry dyeing bath. If the polyelectrolyte concentration of pad-dry dyeing bath exceeded the optimum concentration, the viscosity of the dyeing bath would be too high, which would prevent the dye from adsorption

upon cotton fabric and result in the K/S value decreasing.

PA and PM as exhausting agents exhibited different effects on the K/S value of cotton samples dyed with different dyes. As showed in Figure 5, the data bars indicated that the K/S value of the cotton samples dyed with C. I. Reactive Yellow 18, Reactive Red 15 were nearly the same, in spite sodium chloride or polycarboxylic acid sodium salts was used as exhausting agent. But, when polycarboxylic acid sodium salts were used as exhausting agent, the K/S value of the cotton samples dyed with C. I. Reactive Blue 74 were lower than that when sodium chloride was used as exhausting agent. Dye-uptake of reactive dye was related to the ratio of its salt sensitivity. The salt sensitivity of reactive dye varies with the ratio of the amount of electronic charge to its molecular weight if the dyeing condition is definite.²³ In other words, under the same concentration of electrolyte, the higher the ratio the higher is the dye uptake of reactive dye. From the dye structures in Table I, it could be found that the ratio of electronic charge to mass was 4.417×10^{-3} , 4.569×10^{-3} , and 2.730×10^{-3} for Reactive Yellow 18, Reactive Red 15, and Reactive Blue 74, respectively. Obviously the effect of polycarboxylic acid sodium salt on the K/S value of cotton samples dyed with Reactive Blue 74 should be smaller than that of cotton samples dyed with Reactive Yellow 18 or Reactive Red 15 under the same dyeing condition.

The mechanism of PA or PM working as an exhausting agent

Comparing data bar A, B, C, and D in Figure 6, it could be found that the specific conductance of polycarboxylic acid sodium salt solution were lower than that of sodium chloride solution and the effect of temperature on the ionization degree of polycarboxylic acid sodium salt was smaller than that of sodium chloride solution too. It was suggested that the PA and PM as weak electrolytes could not provide enough sodium ion to screen the negative charge on cotton fabric surface and promote reactive dye to absorb on cotton fabric effectively during exhaustion phase. So, it

TABLE II
Effect of Concentration of PA on the Viscosity of the Pad-Dry Dyeing Bath

Test Number	Flow time (s)										
	Pure water	Concentration of PA (g/L)					Concentration of PM (g/L)				
		30	40	50	60	70	30	40	50	60	70
1	112.0	220.0	235.8	262.2	280.4	307.1	131.1	135.7	143.5	156.1	163.3
2	111.9	218.6	235.4	263.8	280.2	306.6	132.0	134.4	144.0	153.0	162.9
3	112.2	219.2	234.6	263.4	280.8	307.3	131.4	137.1	143.4	153.9	163.3
Average	112.0	219.3	235.3	263.1	280.5	307.0	131.5	135.7	143.6	154.3	163.2

Concentration of dye 20g/L, temperature $(30 \pm 0.2)^\circ\text{C}$, pH value of solution 7.0 ± 0.1 .

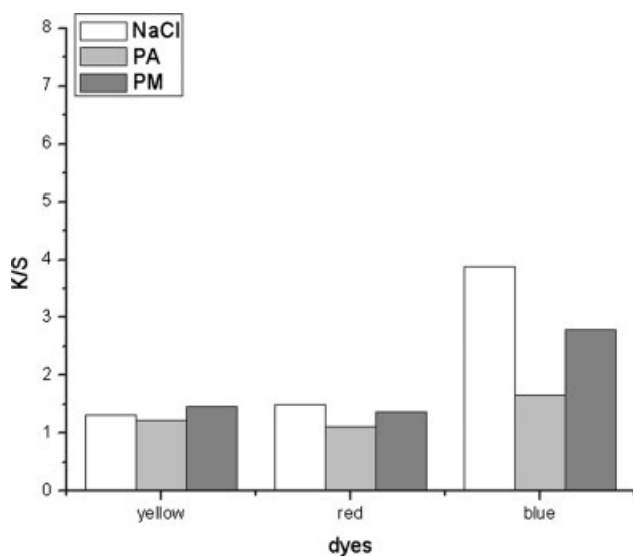


Figure 5 K/S values of samples dyed with different dyes in dip dyeing process. The concentration of dye solution 0.4 g/L, the concentration of the NaCl, PA, and PM respectively, 50 g/L.

was possible for some other reason to promote dye to up take cotton fabric.

Curves in Figures 7 and 8 showed that both the surface tensions of ionic and nonionic surfactant solutions were decreased when electrolytes were added in the surfactant solutions. Similarly, in Figure 9, the values of surface tension of the dye solution were also decreased by adding electrolytes. As a strong electrolyte, sodium chloride would cause the surface tension of surfactant solution and dye solution to decrease through the screen of negative charge of surfactant

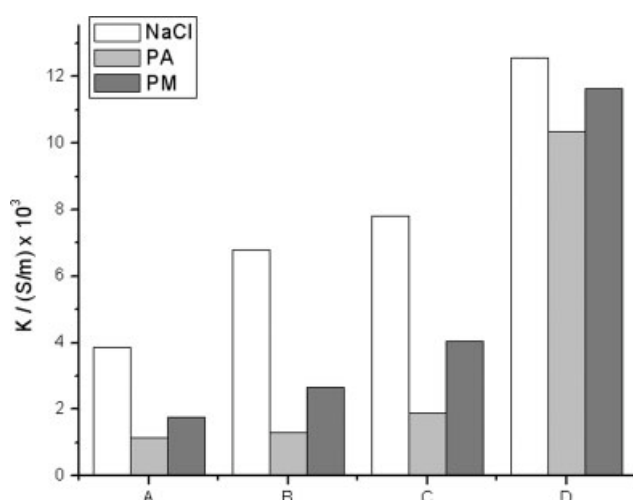


Figure 6 Effect of temperature on specific conductance of different electrolyte. A, B and C was the specific conductance of the electrolyte solution under 30, 60, and 85°C respectively ($[\text{Na}^+] 0.02608 \text{ mol/L}$, pH value 7.0 ± 0.1) D was the specific conductance of the electrolyte solution at 85°C ($[\text{Na}^+] 0.02608 \text{ mol/L}$, pH value 10.5 ± 0.1).

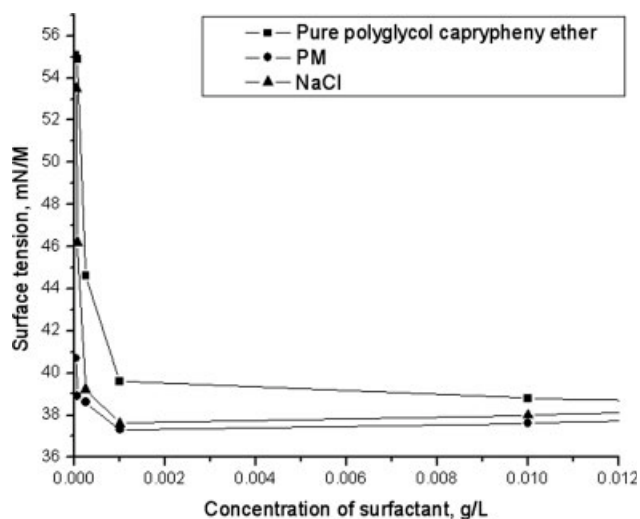


Figure 7 Effect of PM and NaCl on the surface tension of polyglycol caprypheny ether ($n = 10$) solution (Temperature 30°C, $[\text{Na}^+] 0.8547 \text{ mol/L}$).

and dye by ionized sodium ion, which resulted in the salting-out of surfactant and dye in the solution.^{24–26}

The way polycarboxylic acid sodium salt leading the surface tension of surfactant and dye solution to decrease seems a little different from that of sodium chloride did. Because PM as a weak electrolyte could not be ionized completely like sodium chloride did in neutral solution at room temperature, the surface tension decreasing of surfactant solution and dye solution caused by adding PM could not all at the ionized sodium ion leading the salting-out of surfactant and dye in the solution. Another reason for the surface tension decreasing of surfactant or dye solution caused by adding PM may be at the PM added in the solution destroyed the highly ordered cluster structure formed between surfactant or dye molecules and water mole-

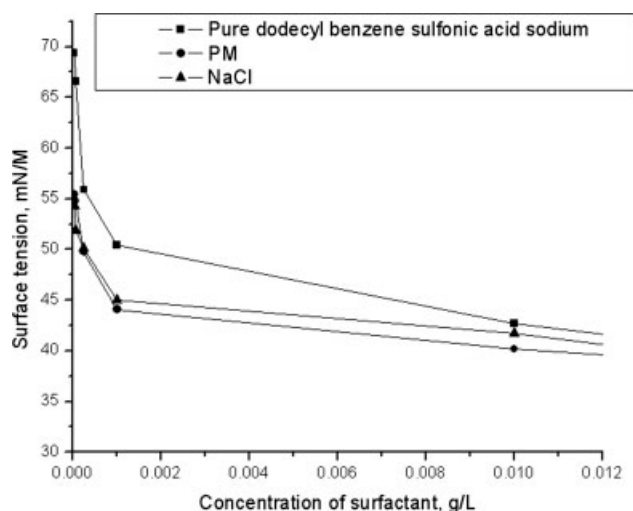


Figure 8 Effect of PM and NaCl on surface tension of dodecyl benzene sulfonic acid sodium solution (Temperature 25°C, $[\text{Na}^+] 0.8547 \text{ mol/L}$).

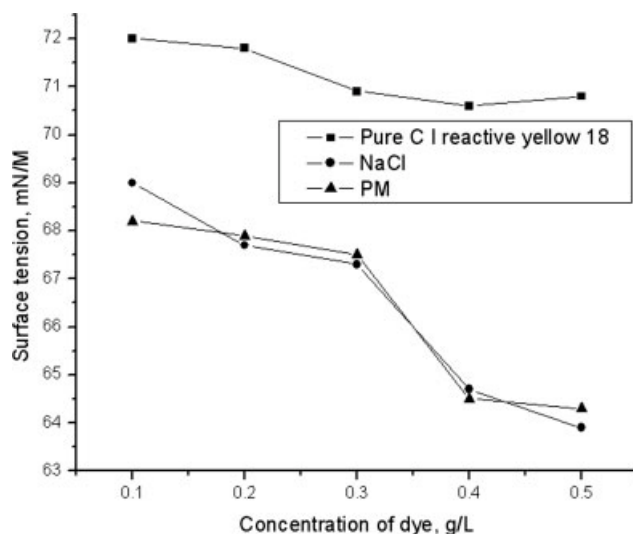


Figure 9 Effect of NaCl and PM on the surface tension of the dye solution (Concentration of NaCl and PM 50 g/L, temperature 30°C, pH value 7.0 ± 0.1).

cules which would result in the hydrophobic combination of surfactant or dye, and then the cluster assembled by surfactant or dye molecules migrated from inner phase to air-liquid interface of the solution.^{27–29} The hydrophobic combination of surfactant or dye molecules in the solution contained PM was the result of entropy driving.

According to Donnan membrane balance theory,²⁹ the osmotic pressure established by the dyeing system containing polycarboxylic acid sodium salts should be larger than that did by the dyeing system containing

sodium chloride, because the molecular weight of polycarboxylic acid was much larger than that of chloride ion and the polycarboxylic acid molecule could not penetrate into cotton fiber like chloride ion did. The higher osmotic pressure of dye containing polycarboxylic acid sodium would make more sodium ion penetrating into cotton fiber, which would help dye molecule in up taking cotton fabric.

The data bar D in Figure 6 showed that the specific conductance of all the electrolyte solutions were nearly close to each other under the alkaline condition at 85°C. Under this condition, the macromolecule chains of polycarboxylic acid sodium salts almost stretched completely and the ionization of sodium ion on the chain was adequate.³⁰ So, at the fixing phase of reactive dye, the screen of the negative charge on cotton surface by the sodium ion ionized from polycarboxylic acid sodium salt played an important role.

From above, it could be found that the working principle of polycarboxylic acid sodium salt in dyeing of cotton with reactive dye was relatively complex. Screening of the negative charge on cotton surface, hydrophobic combination of dye, osmotic pressure increasing of the dyeing system, and salting-out effect of dye may play a role in promoting reactive dye up taking cotton at different dyeing stages.

Effect of the different electrolyte on the dyeing fastness of the dyed samples

Data in Table III indicated that the fading and staining fastnesses of the dyed fabrics with polycarboxylic acid

TABLE III
Effect of the Different Electrolyte on the Dyeing Fastness of the Dyed Samples

Reactive dyes	Electrolytes	Washing		Rubbing			
		A	B	Wrap direction		Weft direction	
				Dry	Wet	Dry	Wet
Reactive Yellow K-4G (in dip-dyeing)	NaCl	4–5	4–5	4–5	4–5	4–5	4–5
	PA	4–5	4–5	4–5	4–5	4–5	4
	PM	4–5	4–5	4–5	4	4–5	3–4
Reactive red K-2G (in dip-dyeing)	NaCl	4–5	4–5	4–5	3–4	4–5	4
	PA	4–5	4–5	4–5	4–5	4–5	4
	PM	4–5	4–5	4–5	4	4–5	3–4
Reactive blue K-3R (in dip-dyeing)	NaCl	4	4–5	4–5	3–4	4–5	4
	PA	4	4–5	4–5	4	4	3–4
	PM	4	4–5	4	4	4–5	3–4
Reactive Yellow K-4G (in pad-dry dyeing)	Na ₂ SO ₄	4–5	4–5	4–5	4–5	4–5	4–5
	PA	4–5	4–5	4–5	4–5	4–5	4
	PM	4–5	4–5	4–5	4–5	4–5	3–4
Reactive red K-2G (in pad-dry dyeing)	Na ₂ SO ₄	4–5	4–5	4–5	3–4	4	4
	PA	4–5	4–5	4–5	4	4–5	4
	PM	4–5	4	4–5	3–4	4–5	3–4
Reactive blue K-3R (in pad-dry dyeing)	Na ₂ SO ₄	4	4–5	4–5	4	4–5	4
	PA	4–5	4–5	4	4	4	3–4
	PM	4–5	4–5	4–5	4	4–5	4

The concentration of the NaCl, PA, and PM 50 g/L.

A. The fading fastness of the sample; B. The staining fastness of fabric.

sodium salts as exhausting agent were near to that of dyed fabrics with sodium chloride or sodium sulfate as exhausting agent, except of wet rubbing fastness. The wet fastnesses of the dyed fabrics with polycarboxylic acid sodium salts as exhausting agent were a little lower than that of dyed fabrics used sodium chloride or sodium sulfate. Because the polycarboxylic acid sodium salt was a nonfoam washing agent for dyeing and printing cotton fabric dyed with reactive dye,¹¹ the remainder of polyacid salts on fabric after washing may caused to the decrease of the wet fastness.

CONCLUSIONS

It is possible with the polycarboxylic acid sodium salt to substitute inorganic salts as exhausting agent both in dip-dyeing and pad-dry dyeing of cotton fabric with reactive dye. The K/S values and fastnesses of dyed cotton fabric with polycarboxylic acid sodium salt as exhausting agent were close to that of with sodium chloride when dip-dyeing process was adopted. When the pad-dry dyeing process was used, the K/S values of dyed cotton fabrics with polycarboxylic acid sodium salt as exhausting agent were higher than that of with sodium sulfate and the dyeing fastnesses of dyed cotton fabric were nearly the same.

The mechanism of polycarboxylic acid sodium salt to promote dye up taking cotton fabric was a little different from that of inorganic salts. All of the screen of negative charge on cotton surface, hydrophobic combination of dye, osmotic pressure increasing of dyeing system and salting-out effect of dye may played a role in promoting reactive dye up taking cotton at different dyeing stage when polycarboxylic acid sodium salt was used as exhausting agent.

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