TECHNICAL BULLETIN



COTTON INCORPORATED

6399 Weston Parkway, Cary, North Carolina, 27513 • Telephone (919) 678-2220

TRI 3016

DYEING CATIONIC PRETREATED COTTON

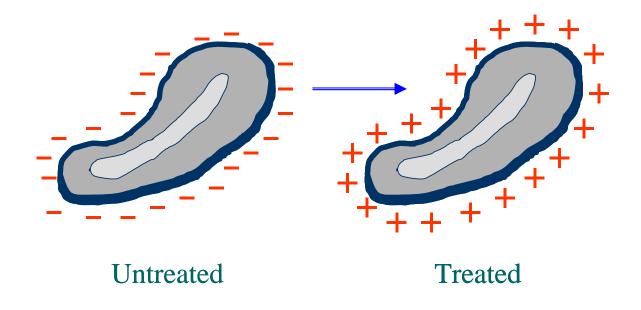
© 2005 Cotton Incorporated. All rights reserved; America's Cotton Producers and Importers.

TABLE OF CONTENTS

Introduction	2
Overview of Dye Selection and Dyeing Procedure	.2, 3
Evaluation of Dyes	4, 5
Effect of Cationic Concentration on Depth of Dyeing	6
Ability to Achieve Tone on Tone Dyeings	6
Levelness of Dyeings	6
Comparison of Depth and Cast of Dyeings Cationic versus Non-Cationic	6
Afterwashing of Direct and Reactive Dyed Shades on Cationic Cotton	6
Perspiration Fastness of Dyed Cationic Pretreated Cotton	7
Tinting of Cationic Treated Yarn	7
Sources for Cationic Reagent	7
Sources for Cationic Pretreated Cotton	
Recommendations For Investigating The Dyeing of Cationic Cotton	.10

INTRODUCTION

Research has been conducted for at least twenty years on the application of cationic reagents to cotton to change the charge on cotton from anionic to cationic (negative to positive). The changing of the ionic nature of cotton increases the affinity of anionic dyes for cotton. A change in ionic nature has been desired to overcome certain problems related to dyeing cotton by conventional means. These problems included the need for high levels of electrolyte (salt), the relatively high level of hydrolysis of reactive dyes in water which reduces the amount of dye that can be fixed onto cotton, the substantial afterwashing time required on reactive dyeing, and the poor wetfastness of direct dyes. One of the cationic reagents extensively studied has been 2,3 epoxypropyltrimethylammonium chloride.



Very little research has been conducted and published on the dyeing of cationic cotton. This has been a major focus at Cotton Incorporated though virtually all the research has been on exhaust dyeing. Initial trials at Cotton Incorporated have shown fiber reactive and direct dyes to be better candidates for dyeing cationic pretreated cotton than vat and acid dyes; however, a great deal of further research and actual mill practice need to be done to optimize the process.

OVERVIEW OF DYE SELECTION AND DYEING PROCEDURE

Most small-scale laboratory work at Cotton Incorporated and scale-up trials to sample machine processing have involved peroxide bleaching the cationic treated cotton prior to dyeing.

Laboratory experiments have shown that cationic cotton can be dyed within a wide range of temperatures from 100°F (38°C) to 200°F (93°C). However, within this range of temperatures, there is considerable difference in the length of time required to exhaust the dye onto cationic cotton and this is proportional to the bath temperature. In some cases it is actually faster to dye the cationic cotton at an elevated temperature including the ramp time than dyeing at a lower

temperature. It has also been observed that dyeing with reactive dyes at low temperatures results in a ring dyed effect.

Conventional dyeing procedures for cotton are not appropriate for dyeing cationic treated cotton and may have a negative effect on certain advantages that the cationic treatment can provide. For cationic pretreated cotton, salt is not necessary for dyeing with direct or fiber reactive dyes nor is alkali needed for reactive dyes.

There are differences in the rate at which dyes exhaust onto cationic cotton. Temperature, concentration of the cationic pretreatment, concentration of the dyestuffs, and possibly pH of the dyebath impact the exhaust rate.

Because of the differences in the dyeing rate of the various dyes, laboratory dyeings have all been made at a ramp rate of $4^{\circ}F$ ($2^{\circ}C$) and a final bath temperature of $200^{\circ}F$ ($93^{\circ}C$) for 30 minutes. In many cases, the dye was completely exhausted well before the end of 30 minutes at temperature and in some cases before the final temperature was ever reached.

Many dyes were evaluated for use for dyeing cationic cotton and some were found to have better performance than others. Colorfastness, staining of untreated cotton, and the effect of the dye on the surface appearance of the fabrics were criteria used in judging performance in order to select dyes as ones a dyer would want to use for shade matching.

Following is a table showing the results found on a number of direct, reactive, and acid dyes. It was generally found that reactive dyes stain untreated cotton far less than direct dyes. Some reactive dyes are better than others for minimizing the stain on untreated cotton. There does not appear to be an adverse effect on lightfastness with the cationic treatment.

TABLE OF DYES EVALUATED

IIA Stain Stain 3.5 5.0 5.0 4.5 5.0 4.0 4.5 4.5 4.0 4.5 4.5 5.0 4.5 4.5 5.0 4.5 4.5 5.0 4.5 4.5 5.0 4.5 4.5 5.0 4.5 4.5 5.0 4.5 4.5 5.0 4.5 4.5 5.0 4.5 4.5 5.0 4.5 5.0 5.0 4.5 5.0 5.0 4.5 5.0 5.0 4.5 5.0 5.0 5.0 5.0 5.0 5.0 5.0 5.0 5.0 5.0 5.0 5.0 5.0 5.0 5.0 5.0 5.0 5.0 5.0 5.0 5.0 5.0 5.0 5.0 5.0 5.0 5.0 5.0 5.0 5.0 5.0 <
COMMENTS Stain on untreated Light Fastness IIA co co co co co co co co co co co co co
Stain on Light 4.0Light FastnessIIA CCStain CCIIA Stain Stain Stain Stain Stain Stain StainIIA Stain Stain Stain Stain Stain StainIIA Stain Stain Stain Stain Stain StainIIA Stain Stain Stain Stain Stain Stain Stain Stain StainIIA Stain Stain Stain Stain Stain Stain Stain Stain Stain Stain Stain Stain
$ \begin{array}{ccccccccc} \mbox{IIA} & \mbox{IIA} & \mbox{Stain} & Sta$
$\begin{array}{llllll} \label{eq:linear} \begin{tabular}{ll} Stain \\ Stain \\ 5.0 \\ $
IIA IIA Stain Stain 3.5 5.0 4.5 5.0 4.5 4.0 4.5 4.0 4.5 4.0 4.5 4.0 4.5 4.0 4.5 4.0 4.5 4.0 4.5 4.0 4.5 5.0 4.5 5.0 4.5 5.0 4.5 5.0 4.5 5.0 4.5 5.0 4.5 5.0 4.5 5.0 4.5 5.0 4.5 5.0 5.0 5.0 5.0 5.0 5.0 5.0 5.0 5.0 5.0 5.0 5.0 5.0 5.0 5.0 5.0 5.0 5.0 5.0
Nyion 5.0

41	39	23	34 34	ວ <u></u> →	17 28 31	10 16	DYEING
Orange Cibacron Orange F-BR	Green Cibacron Green FN-BL	Grey Solophenyl Grey 4GL	Solophenyl Brown RL SLF Brown LRL	BROWN Sumifix Supra Yellow Brown EXF	TURQUOISE Remazol Bnt Green 6B Intralite Turq GRLL Remazol Turq GA	NAVY Cibacron Navy FN-B Procion Navy HEXL	NAME
reactive	reactive	direct	direct	reactive	reactive direct reactive	reactive reactive	TABLI
dull	bluish	greenish grey	bluish brown lighter, less blue than (24)	yellow brown	greenish turq. bright turq. heaver, bluer than (17)	greenish navy (fuzz) redder navy	TABLE OF DYES EVALUATED (CON
3.0	3.5	3.5	2.5 3.5	4.0	5.0 4.0	3.5 3.5	NT'D) Stain on Untreated
5.0	5.0	4.5	5.0 5.0	5.0	4.5 4.5	2.5 3.5	Light Fastness
4.5	5.0	5.0	5.0 5.0	5.0	5.0 5.0	4.5 5.0	COC IIA
2.5	4.5	5.0	4.5 5.0	4.5	4.5 5	3.5 4.0	IIA Stain COT
4.0	5.0	5.0	4.0 5.0	5.0	5.0 4.5	4.0 5.0	IIA Stain Nylon
4.5	5.0	5.0	5.0 5.0	5.0	5.0 3.5	4.5 4.5	IIA Stain Poly

Effect of Cationic Concentration on Depth of Dyeing

The depth of shade that can be achieved in dyeing cationic cotton is directly related to the concentration of cationic reagent on the cotton. Heavy shades cannot be obtained on cotton that has been treated with a relatively low level of cationic. A trial was run in which the depth of shade obtained on two dyeings was nearly the same even though the dye concentration of one bath was double the strength of the other. The dye in the low dye concentration dyeing was almost completely exhausted, the higher concentration dyeing left a great deal of dye in the bath. This would imply that there is a level of saturation or dye concentration above which no additional color is developed. In addition, washing is more difficult since there is unfixed dye in the yarn or fabric.

Ability to Achieve Tone on Tone Dyeings

As a result of achieving a certain depth of shade on a specific level of cationic treatment, one can create fabrics using yarns treated with different levels of cationic reagent then dyeing the fabric or the garment resulting in the yarns dyeing to different depths of color. The depth of shade is directly proportional to the level of cationic treatment. This feature works well in creating stripes, jacquard patterns, chambrays, oxfords, and if the cationic is applied to cotton fiber, a heather can be created. It is particularly important to select reactive dyes that do not stain the untreated cotton if the fabric contains untreated cotton. If all of the yarn or fiber in the fabric is cationic treated then direct dyes can perhaps be used to match the desired shade since staining of untreated cotton is not a concern.

Levelness of Dyeings

Level dyeings on a fabric containing cationic pretreated cotton can only be achieved within a range of dye concentrations based on the level of cationic treatment. Too little dye can cause unlevelness. This means that a cationic cotton substrate cannot be dyed just any depth of color, but must be dyed within a range of dye concentrations to achieve a level shade. The use of too much cationic pretreatment is more than just a waste of chemical, but a guarantee of unlevel dyeing. Trials in a jet have shown that levelness can be improved by circulating at 100°F (38°C) for 15 minutes before beginning the rate of rise.

Effect on Depth and Cast of Dyeings

There can be a significant difference in the amount of dye required to obtain a shade on cationic cotton compared to dyeing the same shade conventionally on untreated cotton. Our comparison of ten dyes showed the amount of dye required on cationic cotton to be 8 to 60% less than on untreated cotton. There is also a slight cast change typically from cationic cotton to conventionally dyed untreated cotton. Unfortunately, this means that there is not a simple adjustment that can be made to dye formulas to convert from conventional dyeing to dyeing of cationic cotton.

Afterwashing of Direct and Reactive Dyed Shades on Cationic Cotton

The washing of cationic cotton after dyeing is greatly simplified compared to rinsing and soaping reactive dyed untreated cotton. Only one wash at 160°F (71°C) is generally required followed by a cool rinse to reduce the fabric temperature for unloading. If a significant excess of dye is used in the dyeing, extended rinses might be required. If direct dyes are used, the rinsing process is similar.

Perspiration Fastness of Dyed Cationic Pretreated Cotton

Colorfastness tests on cationic cotton dyed with direct or reactive dyes have shown results as good as or better than conventional dyeing of untreated cotton except for perspiration fastness. AATCC Test Method 15-1985 Color Fastness to Perspiration was used. This test showed that color change was quite good on the 11 dyes tested (ratings of 4.5-5.0), but the stain on cotton and nylon was rated at 1.5-3.5 on some of the dyes. Trials showed that the ratings could be improved to 3.5-5.0 by adding after the dyeing step, an alkaline wash (3 g/l of soda ash at 200°F/93°C for 10 minutes) and rinsing.

<u>Tinting of Cationic Treated Yarn</u>

A potential problem related to the use of cationic pretreated yarn in fabric formation (knitting, weaving, tufting, etc.) is the identification of special yarns which have the same appearance as the other yarns within the fabric. A 30/1 yarn treated with a cationic reagent has the same appearance as a 30/1 yarn that has not been treated. Yarns treated with various levels of the chemistry make the problem even greater. The problem goes beyond merely the identification of the cone of yarn, but the yarn itself in the case of a yarn break in the machine. Tinting the yarn would solve the identification problem and would also allow the pattern in the fabric to be seen as it is being formed. The tint would have to be fugitive, i.e. removable in a preparation process before dyeing.

One technique is to apply basic dyes in a package machine. This approach was tried and can be used but since basic dyes have no affinity for cotton, the dyes can cause a housekeeping problem when the super saturated packages are removed from the package machine and the dye solution drains onto the floor and other surfaces. If the package machine is equipped with a vacuum or if the packages can be "blown out" in the package machine to remove the excess solution, the approach becomes more feasible. Maxilon[®] Yellow GL, Maxilon[®] Blue SL, and Maxilon[®] Red GRL from Ciba were used in a trial and were found to be removed with a peroxide bleach or alkaline scour.

Another approach tried utilized tints from Peach State Labs specially selected for this purpose. They exhibit enough affinity for cotton to exhaust onto the cotton thus eliminating the problem of draining dye. These tints were also found to bleach or scour out completely.

Sources for Cationic Reagents and Technology

Three potential suppliers for the cationic chemistry are:

Peach State Labs, Inc. 180 Burlington Road	The Dow Chemical Company 2301 Brazosport Blvd.	Degussa Corporation QUAB Global Business Co
Rome, GA 30162	Freeport, TX 77541	Peroxygen Chemicals
Tel. 706-291-8743	Tel. 979-238-2011	379 Interpace Parkway

QUAB Global Business Center Peroxygen Chemicals 379 Interpace Parkway PO Box 677 Parsippany, NJ 07054-0677 Tel. 973-541-8353

Sources for Cationic Pretreated Cotton

LIST OF CATIONIC YARN SUPPLIERS

CloverTex, LLC

(Sells heathered cotton yarns made from cationic-treated cotton fiber) 1401 Gunn Street Clover, SC 29710 Contact: Andy Long Tel: (704) 477-2184 E-mail: <u>along@clovertex.com</u>

Parkdale Mills

(Sells heathered cotton yarns made from cationic-treated cotton fiber) P.O. Box 1787 Gastonia, NC 28053 Tel: (704) 864-8761 (Ask for yarn sales)

Patrick Yarns

(Sells heathered cotton yarns made from cationic-treated cotton fiber) 700 S. Railroad Avenue Kings Mountain, NC 28086 Contact: David Possage Tel: (704) 739-4119 Email: <u>david@patrickyarns.com</u>

R.L. Stowe Mills, Inc.

(Sells cationic-treated cotton yarn) 1101 South Watkins Street Chattanooga, TN 37404 Contact: Michael Slocumb Vice President of Marketing, Specialty Yarn Division Tel: (423) 493-1000 Ext. 3907 E-mail: mslocumb@rlstowe.com

LIST OF CATIONIC YARN SUPPLIERS (CONT'D.)

Spectrum Dyed Yarns

(Applies cationic treatment to cotton yarns) 136 Patterson Rd. P.O. Box 609 Kings Mountain, NC 28086 Contact: Ken Kanipe Vice President Manufacturing Tel: 704.739.7401 Ext. 111 E-mail: ken.kanipe@sdy.com

Tintoria Piana U.S., Inc.

(Sells cationic-treated cotton fiber) 220 S. Erwin Street Cartersville, GA 30120 Contact: Andrea Piana Tel: (770) 382-1395 E-mail: <u>tpdyer@aol.com</u>

Tuscarora Yarns, Inc.

(Sells heathered cotton yarns made from cationic-treated cotton fiber) 8760 E. Franklin St. P.O. Box 218 Mt. Pleasant, NC 28124 Tel: 704 436 6527

To assist your company, Cotton Incorporated has compiled this list of potential suppliers. This list represents companies that Cotton Incorporated understands to be reputable; however, this list is not exhaustive and there may be additional suppliers with the necessary capabilities. Cotton Incorporated cannot guarantee that each company has the ability or willingness to deliver a particular product or service. Although we make every attempt to keep this list up to date, there may be cases where a company's capabilities or status may have changed.

Cotton Incorporated is providing this list as a courtesy, but makes no warranty or representation, either expressed or implied, with respect to the suppliers contained herein. Likewise, no statement contained herein shall be construed as a recommendation to use a specific supplier and Cotton Incorporated assumes no responsibility for any negligence on the part of any supplier included in this list.

<u>Recommendations For Investigating The Dyeing of Cationic Cotton By A Mill, Dyestuff</u> <u>Supplier, Apparel Manufacturer, Or Retailer</u>

Knit fabric normally was used in the research at Cotton Incorporated on dyeing of cationic cotton because of our in-house knitting capability. Woven fabrics, yarns, and tufted rugs have also been dyed. Fabric and /or yarn have been dyed in a Mathis Labomat, Unimac garment dye machine, Roaches Pyrotec, and jet dye machines. A fabric favored in trials was a jersey stripe made from yarns treated with different levels of cationic reagent such as 0, 5, 15, 45 g/l. The 0 level allows one to judge the amount of stain on untreated cotton. The Mathis Labomat is an infrared heated device and is quite easy with which to work. For a full evaluation, start with evaluation of single dyes, then go to three dye combinations. A basic universal dye procedure is shown below:

Liquor ratio: 15:1 Mathis Labomat Knit fabrics made from 18/1 four stripe repeat 0, 5, 15, 45 g/l cationic pretreatment

<u>Formula</u>

0.5% Direct or acid dye or 1.0% Fiber reactive dye

Procedure

- 1. Add predissolved dyes to water at 80°F (27°C).
- 2. Load fabric or yarn.
- 3. Heat to 200°F (93°C) at 4°F /min (2°C/min).
- 4. Hold for 30 minutes.
- 5. Cool to 180°F (82°C).
- 6. Rinse at 160°F (71°C) until bath is clear.
- 7. Rinse cool.

The use of current preferred dyes for untreated cotton, may not give the best results. Each dye must be re-evaluated. The list of dyes evaluated at Cotton Incorporated shown previously in this bulletin may be a good starting place. Dyes must be selected based on the criteria important to your location.

The use of a fabric knit from one level of cationic treatment is useful in assessing levelness of dyeing.

For more information on the reactive dyeing of cationic pretreated cotton without salt, please contact Peach State Laboratories.

The statements, recommendations and suggestions contained herein are based on experiments and information believed to be reliable only with regard to the products and/or processes involved at the time. No guarantee is made of their accuracy, however, and the information is given without warranty as to its accuracy or reproducibility either express or implied, and does not authorize use of the information for purposes of advertisement or product endorsement or certification. Likewise, no statement contained herein shall be construed as a permission or recommendation for the use of any information, product or process that may infringe any existing patents. The use of trade names does not constitute endorsement of any product mentioned, nor is permission granted to use the name Cotton Incorporated or any of its trademarks in conjunction with the products involved.

RESEARCH AND TECHNICAL SERVICES

Cotton Incorporated is a research and promotion company representing cotton worldwide. Through research and technical services, our company has the capability to develop, evaluate, and then commercialize the latest technology to benefit cotton.

- Agricultural research leads to improved agronomic practices, pest control and fiber variants with properties required by the most modern textile processes and consumer preferences. Ginning development provides efficient and effective machines for preservation of fiber characteristics. Cottonseed value is enhanced with biotechnology research to improve nutritional qualities and expand the animal food market.
- Research in fiber quality leads to improved fiber testing methodology and seasonal fiber analyses to bring better value both to growers and then mill customers.
- Computerized fiber management techniques result from in-depth fiber processing research.
- Product Development and Implementation operates programs leading to the commercialization of new finishes and improved energy and water conserving dyeing and finishing systems. New cotton fabrics are engineered -- wovens, circular knits, warp knits, and nonwovens -- that meet today's standards for performance.
- Technology Implementation provides comprehensive and customized professional assistance to the cotton industry and its customers -- textile mills and manufacturers.
- A fiber to yarn pilot spinning center allows full exploration of alternative methods of producing yarn for various products from cotton with specific fiber profiles.
- The Company operates its own dyeing and finishing laboratory, knitting laboratory, and a laboratory for physical testing of yarn, fabric, and fiber properties including High Volume Instrument testing capable of measuring micronaire, staple length, strength, uniformity, color, and trash content.

For further information contact:

COTTON INCORPORATED WORLD HEADQUARTERS 6399 WESTON PARKWAY CARY, NC 27513 PHONE: 919-678-2220 FAX: 919-678-2230 COTTON INCORPORATED CONSUMER MARKETING HEADQUARTERS 488 MADISON AVENUE NEW YORK, NY 10022-5702 PHONE: 212-413-8300 FAX: 212-413-8377

Other Locations • Los Angeles • Mexico City • Osaka • Shanghai • Singapore •

Visit our website at: www.cottoninc.com



COTTON INCORPORATED