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**TRI 3003** 

# **GARMENT DYEING OF**

# **COTTON APPAREL**

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#### **INTRODUCTION**

Garment dyeing has been practiced for many years, both commercially and as a home procedure. Recently, interest in this technique has intensified. One reason for the increased interest is the fashion trend towards a wrinkled or worn appearance for casual clothing. Garment dyeing of 100% cotton is a way to easily achieve this look. Commercial equipment, which is readily available, is well suited to the dyeing of 100% cotton garments.

Garment dyeing is an important consideration for modern textile and apparel manufacturers, especially since it can be used as a method for achieving rapid response to consumer demand. With the utilization of garment dyeing, lead times and excess inventory can be significantly reduced. In addition, it can be used as a method for short lot dyeing, where basic shades are supplemented with fashion shades. With garment dyeing, the expense of apparel fabric waste is reduced, since the waste fabric is undyed goods with minimal or no finish.

In order to successfully establish a profitable garment-dyeing operation, all manufacturing steps must be carefully controlled. A vertical organization should have the best chance for control and, hence, a greater chance for success. The next best situation is a textile mill that supplies ready-to-dye fabrics to the garment manufacturer, and then provides a commission dyeing service once the fabrics are cut and sewn. Finally, the third situation is that of a commission dyehouse which improves its chances for success by maintaining a close liaison with the customer.

Involvement in a garment-dyeing program requires a review of certain parameters. Some items that warrant consideration are listed below. This information, which is not necessarily complete, is intended to be used as a guide to cotton garment dyeing and is not intended to cover every situation that may be encountered in the dyeing of cotton garments.

#### FABRIC SELECTION

Garments of 100% cotton provide an excellent substrate for dyeing. A diverse range of fabrics can be successfully dyed in garment form, ranging from lightweight knits to heavy twill bottom weights. The option to mix various knitted and woven fabrics in multi-component garments is a valuable styling tool. Some shade differences will occur between the different components, but they should be minimal and often can be used as a styling advantage.

Novel effects can result from the combination of an all-cotton fabric with a blended fabric in a garment. The synthetic fiber component is left undyed, yielding a tonal appearance. Tone-on-tone effects can also be achieved by combining mercerized and non-mercerized cotton yarns or fabrics.

#### FABRIC PREPARATION

Greige goods can be used for garment dyeing, but subsequent shrinkage and the longer length of time required in the garment-dyeing machine for preparation before dyeing should be taken into consideration. Garments can be cut oversized to compensate for shrinkage, but seam appearance may be adversely affected.

Fabric preparation prior to garment manufacture serves two functions: (1) it produces a clean fabric for dyeing, and (2) it relaxes the fabric so that consistently sized garments can be made. Most wovens are singed, desized, scoured, and for lighter shades, bleached. Some fabrics are mercerized as well. Generally, continuous or semi-continuous preparation systems are used. Minimal fabric shrinkage is desired and, with wovens, is normally achieved by mechanical compaction. A smoother appearance can be achieved by pretreatment with liquid ammonia.

Knitted fabrics are pre-relaxed to minimize torque, puckering of seams, and shrinkage during garment dyeing and drying. Becks, jets, and continuous rope ranges are normally used for scouring and bleaching of knits. Conveyor type relaxation dryers and/or compacting are used to minimize residual fabric shrinkage.

Fabrics for garment dyeing can be pigment printed or surface finished. Yarn-dyed fabrics can be overdyed by garment dyeing.

Chemical finishes, such as resins and some softeners, generally interfere with garment dyeing and should not be applied to the prepared fabric unless, upon evaluation, they are found to be satisfactory. Level dyeing can best be achieved with a clean absorbent base.

#### GARMENT PREPARATION

Garment dyeing is applicable to a wide range of apparel items. The list includes, but is not limited to, T-shirts, jeans, pull-overs, sweaters, dresses, bathrobes, casual jackets, skirts, and hosiery. With garment dyeing, these items can easily be color coordinated.

**Threads:** 100% cotton or corespun cotton/polyester threads are recommended, so that the thread will dye to nearly the same shade as the garment. Mercerized thread may be used for effect with non-mercerized fabric since it will dye slightly darker and should always be used with mercerized fabric.

**Facing/Interlining:** This portion of the garment may not show, but some blended interlinings will pill and mat during garment dyeing. Excessive shrinkage may cause the waistband and other areas of the garment to look crumpled.

**Zippers:** No problems other than dye acceptance have been encountered with nylon or polyester zippers. A colored zipper can be selected to eliminate this problem. Additional flexibility is possible with nylon zippers when fiber reactive dyes are used since the pH can be lowered with acetic acid at the end of the dye cycle, and the hydrolyzed dye can then be used to dye the nylon. Non-ferrous based metal components, such as nickel-plated brass zippers, should be used since they are less affected by chloride ions from the electrolyte, and they give some protection to copper-containing dyestuffs.

**Pocketing:** As with interlining, this is not visible when the garment is worn. All-cotton and cotton/polyester blended fabrics have been used successfully. With a blended pocketing fabric, the cotton portion dyes, but the resulting overall shade is not the same as the garment shade when a dye formulation is selected to only dye the cotton.

**<u>Trim</u>**: By selecting different fabric constructions for the trim, an unlimited number of styling effects can be developed. Dyeability of the trim should always be taken into consideration. Delicate trims may be damaged during garment dyeing.

**Buttons:** Plastic buttons usually present no problems during dyeing and drying. In some instances, delicate buttons are sewn on after dyeing due to breakage problems or for color coordination. Metal buttons could present the same types of problems as metal zippers, although pewter buttons are acceptable. Additionally, these metal parts may damage the inner surface of the dyeing vessel and eventually cause excessive abrasion to the garments.

**Labels:** Labels may be added after dyeing, or dye-resistant labels can be used. Dye sensitive labels are available, which have a unique look after dyeing. With lighter shades, special provisions for labels may not be required since legibility may not be a problem.

Many garments are dyed in open chamber rotary type machines, where the primary concerns are tangling and abrasion prevention. In some cases, it may be advisable to invert the garment and attach seams together by tacking or by the use of tag fasteners. This will help to reduce tangling, which could cause resist marks or unlevelness. Generally, the best appearance of the garment is preserved if the garment is inverted prior to dyeing and then reverted to the correct side after drying.

#### **DRYING AND PRESSING**

Drying in a tumble dryer yields a soft hand due to mechanical action. Hanging garments on a rack and drying in a conveyor oven is another option. Pressing dyed garments on form boards or a hothead press is sometimes necessary to obtain a smooth garment.

#### **EQUIPMENT**

Paddle machines and rotary drums are the two types of equipment regularly used for garment dyeing. Rotary drum machines have become more predominant, but paddle machines are sometimes preferred for garments that require gentler handling, such as sweaters. A higher liquor ratio is required for paddle machines, which is less economical and may limit shade reproducibility.

Sophisticated rotary-dyeing machines, which incorporate state-of-the-art technology, have been developed by many machinery companies. The load capacity of some of these rotary machines approaches 600 pounds, although generally these machines are rated somewhat higher than practical capacity. As a rule, better performance is achieved at 80% or less of the rated capacity.

Advancements in machinery design have given enormous potential to the future of garment dyeing. Features of modern rotary-dyeing equipment include the following:

- 1. lower liquor ratio
- 2. gentle movement of goods and liquor (minimizes surface abrasion)
- 3. rapid heating and cooling
- 4. centrifugal extraction
- 5. variable drum speed with reversal capability (adaptable to a wide variety of goods)
- 6. continuous circulation of goods (improves migration control)
- 7. ease of sampling
- 8. variable water levels with overflow rinsing capabilities
- 9. large diameter feed and discharge lines (minimizes filling and draining time)
- 10. microprocessor controls
- 11. lint filters
- 12. pressure dyeing
- 13. auto-balancing drums

One feature that can be used to reduce abrasion on delicate garments or to minimize tangling is a compartmental chamber, sometimes referred to as a "Y" pocket.

Efficiency is improved by features such as multiple add-tanks, which allow the operator to assemble all the necessary dyestuffs and auxiliaries in advance, add them to the appropriate tank, and then allow the dye cycle program to take over the operation. Labor requirements are minimized by features such as the ability to tilt the drum for ease of loading and unloading and by the use of conveyor belts for transporting garments. The net result of all these modern features is a machine that is capable of efficiently producing high-quality dyed garments.

#### **DYE SELECTION AND DYEING PROCEDURES**

Direct, fiber reactive, sulfur, and vat dyes may be used for cotton garment dyeing. Specific dyeing procedures are determined by the garment type, shade desired, equipment, and dye system. Cotton Incorporated has undertaken a careful evaluation of the various dye classes and their application techniques for cotton garments. The background and results of this evaluation follow.

## GARMENT DYEING COTTON APPAREL

## A Study of Applicable Dye Classes

**Procedure:** Two Milnor washing machines (front loading rotary type) were modified to serve as dyeing machines for this study. Each machine had a capacity of 10-15 lbs. (4.5-6.8 kg.), indirect heating, cooling capabilities, auto-reversing, and 17 rpm rotation speed.

The 100% cotton items used in this study were as follows:

## PANTS--2 Pair/Dye Lot

3/1 Right Hand Twill 8/1 Ne Warp, 6/1 Ne Filling 67 ends/inch × 43 picks/inch 11.9 oz./square yard (404 grams/square meter)

## FABRIC SQUARES--6 Squares/Dye Lot

Dimensions: 22 in. × 22 in. (56 cm. × 56 cm.) 3/1 Left Hand Twill 14/1 Ne Warp, 11/1 Ne Filling 104 ends/inch × 51 picks/inch 8.4 oz./square yard (285 grams/square meter)

## KNIT SHIRTS--2 Shirts/Dye Lot

Single Pique, 28 cut 50/2 Ne Combed Peeler Mercerized 5.0 oz./square yard (170 grams/square meter)

Before being constructed into garments or cut into sample squares, all fabrics used in this study were yarn mercerized (knit) or piece mercerized (wovens) and bleached in preparation for dyeing. The pant's fabric was mechanically preshrunk, which is evident in the low shrinkage results. The fabrics used for the  $22 \times 22$  inch squares and the knit shirts were not finished for shrinkage control, which is also evident in the resulting shrinkages. In practice, fabric shrinkage should be controlled.

The dye classes and shades evaluated are listed below:

Dye Class	Shade 1	Shade 2
Direct	Khaki	Light Blue
Fiber Reactive	Turquoise	Navy
Sulfur	Black	Navy
Vat	Khaki	Navy (regular and Hydron)

All dyeings of fabrics and garments were done in an open chamber machine without divisions. Pants and shirts were inverted to minimize surface abrasion during dyeing. Prior to all dyeings, a wet-out procedure was followed by adding 0.5 g/l of non-foaming wetting agent and circulating for 10 minutes at 90-100 F (32-38 C), followed by a drop and refill.

The dyeing procedures used are outlined, and comparisons of time, water, and dye/chemical costs were recorded. After dyeing, all the samples were extracted centrifugally and tumble dried. The fabric squares dyed with each set of garments served as test fabrics for all colorfastness testing.

#### **DYEINGS**

#### **Direct**

A total cycle of 155 minutes with two drops and fills, and one overflow was used.

#### Formulations:

Khaki		Light Blue	
0.50 g/l 1.50 g/l 0.05 g/l	Anionic Dispersant Leveling Agent Soda Ash	0.50 g/l 1.50 g/l 0.05 g/l	Anionic Dispersant Leveling Agent Soda Ash
0.28% owf 0.20% owf 0.07% owf 5.00 g/l	Direct Yellow 106 Direct Blue 109 Direct Red 79 Glauber Salt	0.10% owf 3.00 g/l	Direct Blue 80 Glauber Salt

Softening and aftertreatment were done using 0.1 g/l acetic acid, 1.0% cationic aftertreatment, and 2.0 g/l cationic softener (10% solution).

### DIRECT DYEING PROCEDURE

- 1. Set bath @  $90^{\circ}F(32^{\circ}C)$ .
- 2. Add auxiliary chemicals.
- 3. Circulate 5 minutes.
- 4. Add dyes.
- 5. Circulate 5 minutes.
- 6. Raise to  $200^{\circ} F (94^{\circ} C)$  @  $3^{\circ} F (1.7^{\circ} C)/minute$
- 7. Circulate 15 minutes.
- 8. Add predissolved salt slowly (over 3-5 minutes).
- 9. Cool to  $180^{\circ}F(82^{\circ}C)$  either by radiation or using heat exchanger.
- 10. Circulate 15 minutes.
- 11. Sample.
- 12. If satisfactory, cool to  $160^{\circ}F(71^{\circ}C)$  @ maximum rate.
- 13. Drop.
- 14. Overflow rinse cold until clear.
- 15. Set bath volume.
- 16. Add aftertreatment and/or softener.
- 17. Raise to  $110^{\circ}$ F (43 $^{\circ}$ C) @ maximum rate.
- 18. Circulate 10 minutes.
- 19. Unload.

#### FIBER REACTIVE

An "all-in" method was used for a total cycle of 235 minutes with three drops and fills and two overflow rinses.

Formulations:

Turquoise		Navy	
0.5 g/l	Anionic Dispersant	0.5 g/l	Anionic Dispersant
75.0 g/l	Common Salt	75.0 g/l	Common Salt
4.0 g/l	Sodium Bicarbonate	2.0 g/l	Sodium Bicarbonate
8.0 g/l	Soda Ash	4.0 g/l	Soda Ash
2.0% owf	Reactive Blue 21	2.3% owf	Reactive Blue 73
2.0% owf	Reactive Blue 181	1.5% owf	Reactive Black 5

1.0 g/l nonionic soaping agent was used for soaping off the unfixed dye. For softening, 0.1 g/l acetic acid (56%) and 2.0 g/l of cationic softener (10% solution) were used.

Due to dyestuff reactivity on the fabric to be dyed, other fiber reactive procedures may be more suitable. If penetration is difficult, then the alkali could be omitted from the dyebath until the reaction temperature has been reached, and circulation is maintained for 15-20 minutes after which the alkali is added over a 5 minute period and dyeing continued for 30-45 minutes. The equipment used for this procedure should have good addition capabilities. It is normally referred to as the *"Migration Method."* 

Another procedure may be useful if the dyestuff reactivities are suitable. This procedure also requires a machine with controlled addition capabilities. Hot-dyeing fiber reactive dyes, such as monochlorotriazine and others with similar reactivities, are best suited for this method. This method is a common technique for dyeing yarn in package machines. It is normally referred to as the "Constant Temperature Method."

These two methods are listed on the following pages.

#### FIBER REACTIVE DYEING PROCEDURE ALL-IN METHOD

- 1. Set bath @  $90^{\circ}F(32^{\circ}C)$ .
- 2. Add auxiliary chemicals, including salt and alkali.
- 3. Circulate 5 minutes.
- 4. Add dye slowly over 5 minutes.
- 5. Circulate 20 minutes.
- 6. Raise to reaction temperature @  $2^{\circ}F(1^{\circ}C)/minute$ .
- 7. Circulate 50 minutes.
- 8. Sample.
- 9. If satisfactory, drop.
- 10. Overflow rinse warm  $100^{\circ}$ F (38°C) until clear.
- 11. Check pH (should be 6.5-7.5).
- 12. Set bath volume.
- 13. Add soap-off chemicals.
- 14. Raise to  $190^{\circ}F(88^{\circ}C)$  @ maximum rate.
- 15. Circulate 10 minutes.
- 16. Drop.
- 17. Overflow rinse warm  $100^{\circ}$ F (38°C) until clear.
- 18. Set bath volume.
- 19. Add softener.
- 20. Raise to  $120^{\circ}F(49^{\circ}C)$  @ maximum rate.
- 21. Circulate 10 minutes.
- 22. Unload.

#### FIBER REACTIVE PROCEDURE MIGRATION METHOD

- 1. Set bath @  $90^{\circ}F(32^{\circ}C)$ .
- 2. Add dyeing assistants and salt.
- 3. Circulate 5 minutes.
- 4. Add dye over 5 minutes.
- 5. Raise to reaction temperature @  $4^{\circ}F(2.3^{\circ}C)/minute$ .
- 6. Circulate 20 minutes.
- 7. Add predissolved alkali over 5 minutes.
- 8. Circulate 45 minutes (less for lighter shades).
- 9. Sample.
- 10. If satisfactory, drop.
- 11. Overflow rinse until nearly clear (should take 10-15 minutes).
- 12. Add soap-off chemicals.
- 13. Raise to  $190^{\circ}$ F (88 $^{\circ}$ C) @ maximum rate.
- 14. Circulate 10 minutes.
- 15. Drop.
- 16. Overflow rinse warm until clear.
- 17. Adjust bath volume.
- 18. Add softener.
- 19. Raise to  $120^{\circ}F(49^{\circ}C)$  at maximum rate.
- 20. Circulate 10 minutes.
- 21. Unload.

#### FIBER REACTIVE PROCEDURE CONSTANT TEMPERATURE METHOD

- 1. Set bath @ 90-140°F (32-60°C).
- 2. Raise to reaction temperature @ maximum rate.
- 3. Add dyeing assistants and salt while heating.
- 4. Circulate 5 minutes.
- 5. Add dye over 5 minutes.
- 6. Circulate 20 minutes.
- 7. Add predissolved alkali over 5 minutes.
- 8. Circulate 45 minutes (less for light shades).
- 9. Sample.
- 10. Overflow rinse until nearly clear (should take 10-15 minutes).
- 11. Add soap-off chemicals.
- 12. Raise to 190°F (88°C) @ maximum rate.
- 13. Circulate 10 minutes.
- 14. Drop.
- 15. Overflow rinse until clear.
- 16. Adjust bath volume.
- 17. Add softener.
- 18. Raise to  $120^{\circ}F(49^{\circ}C)$  @ maximum rate.
- 19. Circulate 10 minutes.
- 20. Unload.

#### **SULFUR**

For the black shade, a total time of 205 minutes completed the cycle. For the navy shade, a total of 180 minutes was required. Both shades involved three drops and fills and three overflow rinses.

Formulations:

Black			Navy
5.0 g/l	Sulfide Solution	5.0 g/l	Sulfide Solution
24.0% owf	Liquid Sulfur Black 1	24.0% owf	Liquid Sulfur Blue 19
30.0 g/l	Common Salt	30.0 g/l	Common Salt

Oxidation of each shade utilized 1.0 g/l of hydrogen peroxide (35%). The soaping cycle used 0.5 g/l soda ash and 1.0 g/l nonionic detergent.

Foam buildup during the dye cycle can be controlled by the addition of 0.5-1.0 g/l of a selected defoamer.

#### SULFUR DYEING PROCEDURE

#### <u>Steps</u>

- 1. Set bath @ 90-100°F (32-38°C).
- 2. Add Sodyefide B, 4.0-7.0 g/l (more is needed with medium-light shades).
- 3. Circulate 3 minutes.
- 4. Add dye.
- 5. Circulate 5 minutes.
- 6. Raise temperature @ 4°F (2.3°C)/minute to that required for dyeing: 120°F (49°C) for blue and navy shades 160°F (71°C) for other colors 190°F (87°C) for black
- 7. Circulate 5 minutes.
- 8. Add predissolved salt slowly over 5 minutes.
- 9. Circulate 30 minutes.
- 10. Overflow rinse cold until nearly clear--never drop.
- 11. Adjust bath volume.
- 12. Add oxidation chemicals.
- 13. Raise to required temperature for oxidation.
- 14. Circulate 10 minutes.
- 15. Drop.
- 16. Overflow rinse cold 5 minutes.
- 17. Adjust bath volume.
- 18. Add soap-off chemicals.
- 19. Raise to  $190^{\circ}F(88^{\circ}C)$  @ maximum rate.
- 20. Circulate 10 minutes.
- 21. Drop.
- 22. Fill @ 90-100°F (32-38°C).
- 23. Overflow rinse warm 100°F (38°C) for 5 minutes.
- 24. Adjust dye bath volume.
- 25. Apply softener. (Do not use a cationic softener. Sodyeco softener A-100 is recommended by Sandoz.)
- 26. Unload.

<b>NOTES:</b>	A.	Dark shades, particularly black shades, should be unloaded slightly alkaline
		at a pH of 8.0 to reduce potential for acid degradation during storage.
	D	Detwoon stone 5 and 6 20 20 collitar of ammonium hydroxida may be

B. Between steps 5 and 6, 2.0-3.0 cc/liter of ammonium hydroxide may be added to prevent premature oxidation.

#### **DIRESUL (EXHAUST) FORMULATION**

Liquor Ratio: 10:1

Dyes/Chemicals:	
Sodium Carbonate	1.00 g/l
Sulfalox 100 <sup>®</sup>	1.00 g/l
Penetrant EH <sup>®</sup>	0.25 g/l
EDTA 100	1.00 g/l
Antimussol C <sup>®</sup>	0.25 g/l
Sodium Sulfate	25.00 g/l
Sodium Carbonate	4.00 g/l
Sodium Hydroxide (50%)	5.00 g/l
Sandozol Reducer RDT-L <sup>®</sup>	7.00 g/l
Diresul Black 4G-EV <sup>®</sup>	18.00 % owf
Oxidizer A	1.00 g/l

#### Procedure:

- 1. Scour with Sodium Carbonate, Sulfalox 100<sup>®</sup>, and Penetrant EH<sup>®</sup> at 180°F (82°C) for 15 minutes.
- 2. Rinse until clear.
- 3. Add auxiliaries to bath at  $100^{\circ}$ F (38 $^{\circ}$ C) and circulate 15 minutes.
- 4. Add Reducer then add Dye.
- 5. Purge with Nitrogen (3 purges).
- 6. Heat to  $200^{\circ}$ F (93 $^{\circ}$ C) at 5.0 $^{\circ}$ F (2.8 $^{\circ}$ C) per minute.
- 7. Circulate 30 minutes and cool to  $170^{\circ}F(76^{\circ}C)$ .
- 8. Overflow rinse for 15 minutes at 120°F (49°C), drop.
- 9. Fill at 80°F (27°C).
- 10. Add 1.0 g/l Sodium Carbonate and Oxidizer A.
- 11. Heat to  $120^{\circ}F(49^{\circ}C)$  and circulate 15 minutes.
- 12. Drain.
- 13. Fill at 80°F (27°C).
- 14. Heat to 100°F (38°C).
- 15. Add Sodium Carbonate and circulate 5 minutes.
- 16. Drain.

## <u>VAT</u>

Total machine time for the vat shades was 185 minutes with two drops and fills and two overflow rinses.

Formulations:

Khaki		Navy	
0.06% owf	Vat Blue 64	7.0% owf	Vat Blue 18
0.20% owf 2.20% owf	Vat Green 49 Vat Brown 1		
18.00 g/l 2.00 cc/l 12.00 g/l	Caustic 50% Ammonium Hydroxide 28% Sodium Hydrosulfite	18.0 g/l 2.0 cc/l 12.0 g/l	Caustic 50% Ammonium Hydroxide 28% Sodium Hydrosulfite

Formulation:

Navy (Vat-Hydron)		
12.0%	Vat Blue 43	
14.0 g/l	Caustic 50%	
2.0 cc/l	Ammonium Hydroxide 28%	
10.0 g/l	Sodium Hydrosulfite	

Oxidation of each shade utilized 2.0 g/l of hydrogen peroxide (35%).

The soaping cycle used 0.5 g/l soda ash and 0.5 g/l nonionic detergent.

Softening at the end of the cycle used 2.0 g/l cationic softener (10% solution) and 0.1 g/l acetic acid (56%).

If foaming occurs, the addition of 0.5-1.0 g/l of a selected defoamer can be used for control.

#### VAT DYEING PROCEDURE

#### <u>Step</u>

- 1. Set bath @  $90^{\circ}F(32^{\circ}C)$ .
- 2. Add dye.
- 3. Circulate 5 minutes.
- 4. Add caustic.
- 5. Circulate 5 minutes.
- 6. Raise to  $175^{\circ}F(80^{\circ}C) @ 4^{\circ}F(2.3^{\circ}C)/minute$ .
- 7. Circulate 5 minutes.
- 8. Add ammonium hydroxide.
- 9. Circulate 1 minute.
- 10. Add sodium hydrosulfite.
- Circulate 25 minutes. (Check reduction-must maintain reduction at least 20 minutes.)
   Overflow ringe 10 minutes or until nearly clear
- 12. Overflow rinse 10 minutes or until nearly clear.
- 13. Add hydrogen peroxide.
- 14. Raise to  $120^{\circ}F(49^{\circ}C)$  @ maximum rate.
- 15. Circulate 10 minutes.
- 16. Add soap-off chemicals.
- 17. Raise to 180-190°F (82-88°C) @ maximum rate.
- 18. Circulate 10 minutes.
- 19. Drop.
- 20. Overflow rinse warm  $100^{\circ}$ F (38 $^{\circ}$ C) for 5 minutes.
- 21. Adjust dye bath volume.
- 22. Add softener chemicals.
- 23. Raise to  $120^{\circ}$ F (49 $^{\circ}$ C) @ maximum rate.
- 24. Circulate 10 minutes.
- 25. Unload.

**NOTE:** For optimum results, dye selection is important when using this procedure with ammonium hydroxide. Individual manufacturer's recommendations should be followed.

#### **INDIGO PROCEDURE**

Dyeings can be conducted with or without a pretreatment procedure prior to the dyeing with the reduced indigo dye. Satisfactory results have been achieved by dyeing the garments directly following the prescour procedure. Dyeings conducted on garments that have been pretreated with a cationic salt exhibited a slight variation in cast compared to garments, which have not been pretreated.

#### Example Procedure

The following is the procedure used for the application of the indigo to cellulosic garments in an enclosed rotating vessel:

1. Prescour garments at  $180^{\circ}F(82^{\circ}C)$  for 10 minutes in the following bath:

3.0 g/l	Sodium Hydroxide (50%)
1.5 g/l	KieralonTX-199 <sup>®</sup> Detergent
1.0 g/l	Basokol NB-S <sup>®</sup> Dispersant

2. Prepare a \*stock vat at 120°F (49°C) for 30 minutes containing up to 6% BASF Indigo Pure<sup>®</sup> on the weight of the cellulose and in the stock vat concentrations and order listed:

x.x g	BASF Indigo Pure <sup>®</sup>
1.20 g/g dye	Sodium Hydroxide (50%)
0.26 g/g dye	Sodium Carbonate
0.81 g/g dye	Sodium Hydrosulfite

(\*The volume of water used in the reparation of the stock vat should be based on the solubility of the indigo dye -100 g/l.)

- 3. Hot rinse the garments sufficiently and end with a cooling rinse.
- 4. Fill with an 80°F (27°C) water bath and add the following dyebath concentrations of dyeing auxiliaries based on a final dyebath liquor to goods ratio of 10:1 in the order listed:

2.0 g/l	Basol WS <sup>®</sup> Dispersant
1.0 g/l	Vitexol K <sup>®</sup> Defoamer
80.0 g/l	Sodium Chloride
7.0 g/l	Sodium Carbonate
1.0 g/l	Primasol FP <sup>®</sup> Penetrant

- 5. Bring the dyebath volume to nearly 10:1 liquor ratio. Close the dyeing vessel and purge its air with nitrogen gas.
- 6. Add to the vessel the reducing agent solution of the following:
  - 3.0 g/l Sodium Carbonate
  - 5.0 g/l Sodium Hydrosulfite
- 7. Rotate vessel 2 minutes at  $80^{\circ}$ F (27 $^{\circ}$ C) and add stock vat "2" above over 10 minutes.
- 8. Rotate vessel 10 minutes at  $80^{\circ}F(27^{\circ}C)$ .
- 9. Drain and centrifugally extract unbound dye liquor while supplying nitrogen to fill eliminated liquor volume. Collect liquor in a closed tank purged with nitrogen.
- 10. Open rotating vessel to exposed garments to air for 5 minutes or until oxidation is complete.
- 11. Test collected spent liquor and restore original indigo, sodium hydrosulfite concentration, and pH with a vat like item "2" above. Save replenished dyebath for dyeing of subsequent batches of garments.
- 12. Wash in 80-100°F (27-38°C) water a sufficient number of times until clear rinse water is achieved.
- 13. Wash in hot water at  $130^{\circ}$ F (54 $^{\circ}$ C) to remove alkali.
- 14. Perform garment washing procedures (enzyme wash, stonewash, etc.) and/or apply softeners and fixatives to achieve desired handle, fastness, and "washdown" appearance.

#### References

- <u>BASF Manual Cellulosic Fibers</u>, BASF Corporation.
- Hughey, Claude, "Indigo Dyeing Review," BASF Corporation.
- Sello, Stephen, "Simultaneous Warp Sizing and Dyeing," J.P. Stevens and Co., Inc.
- Kunihiro, T., "Review of Indigo Dye," Japan Textile News, July 1977.

#### TEST RESULTS

COLORFASTNESS TESTS (Color Change Only)								
Dye Type	Shade	Wash Tests (AATCC) <sup>1</sup>		Wash Tests (Machine) <sup>2</sup>			Light 20/40 <sup>3</sup>	
		2A	<b>3</b> A	<b>4</b> A	5	10	20	
Direct	Khaki Lt. Blue	2-3 4	2 3-4	1-2 3	2 2-3	1 2	1 1	3-4/2 4-5/3-4
Reactive	Turquoise Navy	4-5 5	4 4-5	3-4 4	4-5 4-5	4 4-5	3 4	5/4-5 5/4-5
Sulfur	Black Navy	4-5 4	4-5 3	4-5 2-3	4-5 4	4 3	4 2-3	5/5 5/5
Vat	Khaki Navy Navy (Hydron)	4 4 4	3-4 3-4 3-4	3 3-4 3	3 4 4	2-3 3 3-4	2-3 2-3 3	5/4-5 5/5 5/4-5

#### NOTES:

A. Washfastness ratings based on Gray Scale for color change of 1-5 ("5" being best).

B. Lightfastness based on Xenon Arc Exposures for 20 and 40 AFU's.

C. Frosting<sup>4</sup> ratings ranged from 4-5 with exception of Sulfur (3 to 3-4).

D. Cold water bleed<sup>5</sup> and perspiration<sup>6</sup> ratings all 4-5 to 5.

E. Dry crocking<sup>7</sup> ratings all 4-5. Wet crocking Class 3 or better with exception of Sulfur and Hydron, which range from 2-3.

\*HLTD's = Home Launder/tumble dry cycles.

<sup>&</sup>lt;sup>1</sup>AATCC Test Method 61-1985: Similar to 1S0 105-C06

<sup>&</sup>lt;sup>2</sup>AATCC Test Method 135-1978 II.B, 5-10-20- HLTD's\*

<sup>&</sup>lt;sup>3</sup>AATCC Test Method 16E-1982

<sup>&</sup>lt;sup>4</sup>AATCC Test Method 119-1984

<sup>&</sup>lt;sup>5</sup>AATCC Test Method 107-1981

<sup>&</sup>lt;sup>6</sup>AATCC Test Method 15-1979

<sup>&</sup>lt;sup>7</sup>AATCC Test Method 8-1981

#### PREDICTING GARMENT DYEING SHRINKAGE

% Shrinkage During Garment Dyeing (L x W)	*AATCC Test Method % Shrinkage (L x W)
1.3 x 2.2	1.5 x 3.5
22.3 x 5.6	21.5 x 6.0
12.6 x +0.5	11.5 x +0.5
	Garment Dyeing (L x W) 1.3 x 2.2 22.3 x 5.6

\*Shrinkage after 5 HLTD's @  $120^{\circ}F(50^{\circ}C)$  using AATCC Test Method 135-78 IIB on undyed garments and fabrics.

AATCC Test Method 135-IIB accurately predicted the shrinkage of 100% cotton garments and fabrics during garment dyeing in this study.

RELATIVE COST FACTORS				
Dye Class	Shade	*Cost/lb.	**Water Usage	Time (Hrs.)
Direct	Khaki	\$0.13	2/1	2.6
Direct	Lt. Blue	\$0.08		
Fiber Reactive	Turquoise	\$0.83	3/2	3.9
Fiber Reactive	Navy	\$0.74		
Sulfur	Black	\$0.23	3/3	3.4
Sulfur	Navy	\$0.31		
Vat Vat Vat (Hydron)	Khaki Navy Navy	\$0.45 \$0.82 \$0.57	2/2	3.1
*Dye and chemical cost only. (Based on 1987 dye and chemical costs.) **Relative water use (drops and fills/overflow rinses).				

#### AFTERTREATMENT

**Softeners:** The application of softeners at the end of the dye cycle improves the appearance, hand, and ease of pressing of the dyed garments after tumble drying. Durable press appearance ratings for 100% cotton woven garments after garment dyeing and tumble drying are usually around 1.0-1.5. The addition of a polyethylene softener in the final bath after dyeing generally increases this rating to 1.5-2.0. Use of a softener formulation with 2.0% polyethylene and 2.0% aminofunctional silicone in the final rinse bath typically results in appearance ratings of 2.0-2.5, and ratings as high as 3.0 have been achieved for knit garments. As with all cationic softener systems, yellowing during drying may be a problem, and care must be taken to avoid unacceptable shade changes on white and pastel shades.

**<u>Resins</u>**: After the garments are dyed, they are rinsed and extracted. With some dyes, such as directs and some reactives, a cationic dye fixative may be applied at this stage to prevent bleeding of the dye in the finish bath. For a neater appearance, a postcure resin finish may be applied to dyed garments while still in a wet state. A durable press finish is pumped into the wet, extracted, dyed garments. The liquor ratio should be as used in the dyeing procedure. The concentration of the finishing components will depend upon the amount of water in the wet garments after extraction of the finish, and the quantity of finish required for an acceptable balance of durable press and physical properties. Agitation of the wet garments for 15-20 minutes in the finish bath should dilute the finish by the quantity of water left in the garments. This dilution can be determined from the weight of the dry garment (measured before dyeing) and the water retained after extraction (measured by previous experimentation). A typical finish may consist of the following:

#### % On Weight of Bath

Etherified DMDHEU (45%)	5.0-15.0
Magnesium Chloride Hexahydrate (64%)	1.5-4.0
Acetic Acid (56%)	0.5-1.0
Polyethylene Softener (30% active)	2.0-5.0

The acetic acid is recommended to neutralize any residual alkalinity in the garments. Other softeners such as cationics and silicones may be used. To avoid any possible contamination of subsequent dyeings, it may be advisable to use a separate machine for finish applications.

After agitation in the finish bath, the garments are extracted, and the finish is pumped into a holding tank where it is reconstituted to original concentration for reuse. The degree of dilution can be determined by knowledge of the quantity of water in the wet garments, by measurements such as density or dry solids, or by previous experience.

The extracted garments are dried by normal methods, but care must be taken that garment temperature does not exceed 240°F ( $115^{\circ}$ C). It is preferable to leave at least 5.0-10.0% moisture in the garments. Sensors are available to control temperature of the drying garments. Pressing is conducted in conventional fashion, followed by postcuring.

## **ROTARY DYEING MACHINE SUPPLIERS**

Manufacturer	U.S. Sales Agent
American Laundry Machinery, Inc. Textile Systems Division 5050 Section Avenue Cincinnati, OH 45212 Tel: (513) 731-5500 Fax: (513) 731-0090	
Ashby Industries Inc. P.O. Box 3866 Rte. 57 N. Keehler Road Martinsville, VA 24113	Commercial Textile Machinery, Inc. 4 Leuconia Court Clifton Park, NY 12065
Consolidated Laundry Machinery Co. 1151 29 <sup>th</sup> Street Los Angeles, CA 90011 Tel: (213) 232-2417 Fax: (213) 231-8312	
Ellis Corporation 1400 West Bryn Mawr Avenue Itasca, IL 60143 Fax: (708) 250-9241	
Flainox S.R.L. 5, Via Leopardi I-133010 Quaregna-Italy	Craven, Inc. 224 Datura Street Suite 1414 West Palm Beach, FL 33401
G.A. Braun, Inc. 461 E. Brighton Avenue Syracuse, NY 13205 Tel: (800) 432-7286	Martint Equipment Co. 4455 Morris Park Drive Suite B Charlotte, NC 28227 Tel: (704) 573-1625 Fax: (704) 573-1725
Pellerin Milnor Corporation           P.O. Box 400           Kenner, LA 70063           Tel: (504) 467-9591 ext. 222           Fax: (504) 468-3094	<ul> <li>Wink Davis Dyeing Equipment Co. 650 Pressley Road Charlotte, NC 28217</li> <li>Pellerin Laundry Machinery, Inc. P.O. Box 1137 Kenner, LA 70063</li> <li>Western State Design 17026 Marquardt Avenue Cerritos, CA 90703</li> </ul>

Proll & Lohmann Maschinen andAnlagen GmbHTiegelstr. 6P.O. Box 569D-58005 HagenGermanyRoaches Engineering Ltd.	Henderson Machinery, Inc. P.O. Box 19403 Greensboro, NC 27419 Crosrol Inc.
Upperhulme, Near Leek Staffordshire, ST. 13 8ty England	Box 6488 Greenville, SC 29606
Rome Machine & Foundry Co., Inc. P.O. Box 5383 Rome, GA 30162	
<b>UniMac Company</b> Division of Alliance Laundry Systems 3595 Ind. Park Drive Marianna, FL 32445	WTMC Inc. P.O. Box 400 Gaffney, SC 29342
<b>The ELX Group/ Washex Machinery Co.</b> 5000 Central Freeway Wichita Falls, TX 76306	Alliance Equipment Co. 3838 South State Salt Lake City, UT 84115
	<b>Greentex, Inc.</b> 5475 Pare. #209 Montreal Quebec Canada H4PIP7
	<b>Texchine, Inc.</b> P.O. Box 188 207 Beaufort Chapin, SC 29036
	<b>Texchine, Inc.</b> P.O. Box 6295 (30065) 1691-F Enterprise Way Marletta, GA 30067

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- 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 the grower and his 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 from specific cotton fiber profiles for various products.
- The Company operates its own dyeing and finishing laboratory, knitting laboratory, and a laboratory for testing, including High Volume Instrument testing capable of analyzing micronaire, staple length, strength, uniformity, color, and trash content.

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