TECHNICAL BULLETIN

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ISP 1015

CHEMISTRY OF FIBER REACTIVE AND REACTIVE/DISPERSE DYES IN THE PRINTING PROCESS

This report is sponsored by the Importer Support Program and written to address the technical needs of product sourcers.

INTRODUCTION

Textile Printing: To stamp or impart a pattern or design onto a textile substrate by means of coloration. It is the art or process of printing textile substrates. Dyestuff printing would add to this definition the phrase: "A localized form of dyeing."[1]

The print systems employed in the textile industry in the last 30 years are listed below. In each case, these dyes or combinations thereof are involved with the coloration of cotton and other cellulosic fibers and blends. These print systems are:

1. Fiber Reactive Dyes
2. Disperse Dyes
3. Vat Dyes
4. Azoic Dyes
5. Acid Dyes
6. Pigment Colorants

For this bulletin, only the Fiber Reactive and Disperse Dye systems will be considered. These two systems are used to color 100% cotton and blends of cotton and polyester.

FIBER REACTIVE DYES & THEORY

Fiber reactive dyes react with the cotton fiber through covalent bonds. The major developments in fiber reactive dyes follow:

1. April, 1956, ICI introduced Procion® dyes, first range of fiber reactive dyes. These dyes are monochlorotriazine (MCT) chemistry. (DyStar, LP).

2. June, 1957, CIBA introduces Cibacron® dyes, a new range of reactive dyes (also MCT) and the first to compete with ICI's Procion® series. (Ciba Specialty Chemicals).

3. 1957, Hoechst introduces Vinyl Sulfone® dyes, the first reactive dyes based on vinyl sulfone (VS) chemistry. (DyStar, LP).

Fiber reactive dyes are colored organic substances, which are water-soluble. These dyes will react with cellulose under certain conditions of pH, temperature, solution, and time as shown in Table I.

[1]Pigment Printing Handbook AATCC

Table I. Effect of Dyebath Conditions on the Dyeing Process

Condition	What happens
pH	Less than pH 7.0 not much happens. Greater than pH 7.0 reaction begins to occur.
Temperature	Temperature range: 0º–100°C (32°-212°F). With alkali present, fiber reactive dyes will react with cotton even at very low temperatures though slowly. As temperature increases, reaction speeds increase.
Time	Proportional to temperature
Solution in water	Fiber reactive dyes are very soluble in water.

REACTIVE DYES ON COTTON

The reaction mechanism of the two dye classes – MCT and VS – differs slightly and produces two different end products. These differences provide flexibility in printing processes and procedures. Therefore, we will examine the reaction mechanism and look at the various processes for making this happen.

1. Monochlorotriazine (MCT) dyes reaction with cotton:

   MCT–Dye + **Cotton** ➔ **Cotton – MCT**–Dye

The link between the cotton and the MCT-dye is an ESTER linkage, which is not stable to acid.

2. Vinylsulfone (VS) dyes reaction with cotton:

   VS–Dye + **Cotton** ➔ **Cotton – VS**–Dye

The link between the cotton and the VS-dye is an ETHER linkage, which is not stable to alkali.

Because of the different chemistry of the two classes of dye, the printed fabric also would have different characteristic as seen in Table II.

Table II. Fiber Reactive Print Comparison: MCT vs. VS

Properties	Monochlorotriazine– (MCT)	Vinyl Sulfone – (VS)
Chemical linkage	Ester	Ether
pH sensitivity of print	Not stable to acids	Not stable to alkali
Molecular weight	Generally high	Generally low
Print paste stability: one-phase	1 to 4 weeks	1 to 10 days
Print paste stability: two-phase	Unlimited	Unlimited
Color space	Good	Good
Process suitability: one-phase	Yes	Limited paste stability
Process suitability: two-phase	Poor	Yes
After-washing prints	Difficult to get good results	Good washing results
Environmental – effluent loading	High (Urea & MCT)	Moderate

Both processes undergo the same path to reaction. First the dye must be dissolved in water and applied to dry fabric. Water must be present when fixation takes place. Fiber reactive dyes have little affinity for cotton as long as the pH is neutral or slightly acid. Alkali must also be present for the dyestuff to become reactive. As alkali is added, the dye has more affinity for the cotton.

As the temperature increases, the dyestuff completes the reaction with the cotton in the presence of alkali. The results of the reaction are new molecules. The dye reacts with the hydroxyl (-OH) groups on the cotton molecule. These hydroxyl groups or radicals also exist as a component of water.

The problem with this scenario is since both cotton fabrics and water have these -OH groups; there can also be a reaction with the (-OH) of the water. This is called dye hydrolysis. This new molecule is still soluble but will no longer be able to react with the cotton. The fabric is deprived of some color when this happens. Since this hydrolysis is accelerated by alkali, the alkali is not added until the proper time. Common in printing, this is accomplished by using sodium bicarbonate, a weak alkali in the print paste. After printing and drying, the fabric is steamed or baked to decompose the sodium bicarbonate to sodium carbonate (a stronger alkali) to complete the fixation.

THICKENER SELECTION

Reaction with cotton is the desired result but other similar molecules are often present, such as starch or sugar. The dyestuff will readily react with these contaminants. This makes selection of the thickener and other ingredients in the print paste a critical matter. Thickeners based on starch are inexpensive but cannot be used. Sodium alginate is a thickener obtained from seaweed (Kelp) and does not react with the dyestuff. Some of the Carbopol®[2] polyacrylic acid based resin thickeners have also been used to print reactive dyes. There are some additional products that have been tested.

The key reason for using sodium alginate is its ‘rewetting’ characteristic. It can be washed out after fixation. The synthetic thickeners are difficult to remove in washing due to poor rewetting characteristics. Sodium alginate is also stable to the electrolytes, or salts, present in the alkali, which is included in the print paste. Many synthetic thickeners, which are based on polyacrylic acids, experience a dramatic decrease in viscosity when electrolytes such as the alkali are added to the mix. More information on sodium alginate can be found in the Troubleshooting section.

PRINTING SUBSTRATES

Many cellulosic textile substrates have been used to produce decorated and colored articles. For minimum results, the following should be a guide. Fabric preparation should include the following:

- A scour with detergent and alkali to remove oils and waxes from the fabric and make it absorbent is required.
- For cotton and cotton blends, an additional bleaching process may be included to further whiten the fabric and remove motes.
- In the case of cotton woven fabric, caustisizing or mercerizing may be required to maximize color value, increase the luster of the fabric, minimize imperfections, and provide better coverage of immature cotton. The concentration of sodium hydroxide (NaOH) needed to caustisize cotton is 22ºBé. To produce mercerized fabric, the NaOH concentration needs to be increased to 28-32ºBé.

[2]Noveon, Inc.

Preparation for Printing

The dyestuff printer needs a fabric that has the following properties:

- Uniform absorbency
- Clean
- Uniform white bleach base
- Uniform width
- No creases
- Neutral pH (7.0 - 7.5 is best for all printing)
- Alkalinity of 0.05 or less
- Caustisized or mercerized

PRINTING PROCESS

A fiber reactive print is applied by one of two printing procedures:

A. One-phase printing or direct printing
B. Two-phase printing

Both methods have been in use since the earliest days of fiber reactive printing. There are limitations and drawbacks as well as advantages to each. Table III contains a comparison of both systems.

Table III. Comparison of Reactive Dye Fixation Processes

	One-Phase (MCT[3])		Two-Phase (VS[4])
1	Print paste with alkali + urea	1	Print paste no alkali – no urea
2	Print	2	Print
3	Dry	3	Dry
4	Fixation - steam or bake	4	Pad alkali
5	Wash	5	Fixation – steam
6	Dry	6	Wash
		7	Dry

One-Phase Printing

The terminology suggests a single process but this is not the case. 'One-Phase' refers to the alkali-dyestuff relationship where the dyestuff and alkali are mixed together and applied to the fabric. After application the wet, printed fabric is dried and then put through a third process to accommodate fixation of the dyestuff. This is accomplished through either an atmospheric steamer or a curing oven. Once the print has undergone fixation, the fabric is then washed to remove the by-products of the fixation step, and then dried. The fabric is now ready for finishing.

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[3] Procion® - Cibacron® - Basilen® Dyes
[4] Remazol® Vinyl Sulfone® Dyes

Table IV. Stock Thickening Formula for One-Phase Printing

Chemical	**Steam**	**Bake/Cure**	**Details**
Thickener	700 g	700 g	Sodium alginate (4%-10% depending on grade)
Sodium bicarbonate	30 g	30 g	Sodium bicarbonate: mild alkali needed for fixation
Oxidative compound[5]	10 g	10 g	Sodium metabezinesulfonate
Urea	50 – 100 g	150 – 200 g	Humectant – More is needed for baking / curing
Water softener	10 g	10 g	Sodium hexametaphosphate
Water	Bulk	Bulk	Water to total volume
Total	1000 g	1000 g	

Table V. Print Paste Formula

Chemical	**Amount**	**Details**
Fiber reactive dye	10 – 200 g	Liquid or powder fiber reactive dyes can be used
Stock thickening	700 g	+/- 50 g depending on viscosity requirement
Water	Bulk	Water to total volume
Total	1000 g	

Tables IV and V contain print paste formulations for one-phase printing. The concentration of sodium bicarbonate is determined by the amount of fiber reactive dye in the print paste. Heavy shades may require a slight increase in the amount of sodium bicarbonate to achieve good fixation. All reactive dyes for printing are more or less interchangeable. The same print paste could be used for VS and MCT dyes. One-phase printing favors the slower reacting MCT dyes due to their improved stability in the presence of alkali. But each manufacturer warns against the practice of mixing the products together in the same print paste because of relative rate of reaction and molecular weight differences, to name a few. On the whole, formulations are similar.

1. Print Paste Formulation (See Tables IV and V)

2. Print
   - Print with lighter colors first, then build to heavier colors.
   - The black will be applied last and may require a double screen lay down.
   - Screen mesh should be 80-100.

3. Drying
   - Drying temperatures should be controlled to a uniform 125º-130ºC (257°-266°F).
   - The dried fabric should be fixed as soon as practical. Storing printed fabrics containing fixation chemicals may produce mark-off, especially in humid conditions.

4. Steam Fixation
   - The printed and dried goods are steamed.
   - MCT dyes require a steaming time on the order of 8-10 minutes.
   - Steam should be saturated and slightly overheated at 103ºC (217°F). This prevents water spotting in the steamer.

[5] This oxidative compound releases oxygen during the fixation process and prevents impurities from destroying the dyestuff (Prevents dye reduction). Examples: Ludigol®, Reserve Salt, Basitol®

5. Washing Fiber Reactive Prints
   Ingredients For Successful Print Washing Are:
   - Power sprays – Open width
   - Rope washing – Hot water washing – No detergent
   - Minimum 95ºC (203ºF)
   - Final cold rinse

No acid is needed to neutralize the alkali used in fixation. If the washing procedure was designed properly, there will be very little residual alkali. Additions of acid can lead to strength problems with resin finishing and acid catalyst degradation of cotton fibers.

6. Opening and Drying Fabric for Finishing.
   - The washed fabric should be dried open width.

Fixation by dry heat is sometimes called "Baking" or "Curing". The process is similar to the steam fixation process where the fixation apparatus uses hot dry air to heat the fabric and make the reaction proceed. Most baking or curing is now done in ovens heated by natural gas burners. The stock paste contains 25% - 50% more urea, which is needed as a humectant to retain water on the printed cotton so that a good reaction between fiber reactive dye and cotton is achieved (see Table IV).

Two-Phase Printing

This process takes advantage of the increased stability of the dyestuff when alkali is absent. The dyestuff is mixed with only thickener and water then applied to the fabric. The wet fabric is dried as before. The alkali in solution is now applied to the fabric by a chemical pad and the wet fabric immediately passes into a "fixation" apparatus, usually a steamer. Step six is the washing of the "fixed" fabric and then, as before, drying is the final step in the process.

The foremost consideration in print process design is to maximize the reaction of fiber reactive dyestuff with the cotton fiber versus hydrolysis with water. If dye and alkali are not combined until the last minute, more dye should be available for reaction with the fiber (less hydrolysis). Preparation of print paste for two-phase fixation is as follows in Table VI and Table VII.

Table VI. Stock Thickening Formula for Two-Phase Printing

Chemical	Amount	Details
Thickener	700 g	Sodium alginate (4%–10% depending on grade)
Oxidative compound	10 g	Sodium metabezinesulfonate
Water softener	10 g	Sodium hexametaphosphate
Water	Bulk	Water to total volume
Total	1000 g	1000 grams

Table VII. Print Paste Formula for Two-Phase Printing:

Chemical	Amount	Details
Fiber reactive dye	10 – 200 g	Liquid or powder fiber reactive dyes can be used
Stock thickening	700 g	+/- 50 g depending on viscosity requirement
Water	Bulk	Water to total volume
Total	1000 g	1000 grams

Two-Phase Steaming

Two-phase steaming method refers to the equipment required and consists of a pad mangle and a loop steamer, which must work together continuously. After printing and drying, the printed fabric will pass through a chemical pad containing the fixation bath. Care must be taken in two-phase procedures to effect a rapid immobilization of the soluble dyestuff and start the fixation process immediately. This is accomplished by using stronger forms of alkali in conjunction with high amounts of electrolyte.

The two-phase printing process is flexible in that a variety of fixation methods may be used. The two-phase procedures that will be outlined are:

1. Two-phase loop steaming method
2. Two-phase flash age steaming method

Two-Phase Loop Steaming Method:

Table VIII contains the alkali and electrolyte requirements for two-phase steaming. Constant specific gravity is at least as important as the composition of the fixation liquor. The fixation liquor has a specific gravity of 28°Bé. Dilution of this liquor to 20°Bé, causes the dye to dissolve into the fixation liquor and results in poor fixation. Time required for fixation will vary. It is possible to steam from 45 seconds to 2–3 minutes in a large loop steamer with fabric supported by insulated rods on the back (unprinted) side of the fabric. This insures that the face of the fabric does not come in contact with any rollers or other surfaces. Excessive steaming will discolor the print and result in poor quality.

As for the composition of the padding liquor, only the concentration of caustic soda varies: 20 ml/L caustic soda 38°Bé is required for a steaming time of 40 seconds and 40 ml/L caustic soda 38°Bé for a steaming time of 20 seconds. The loop steamers in use today with this fixation method operate at speeds that allow steaming times of 1–3 minutes. Faster fixation times can be used, but this is limited by the mechanical considerations at higher speeds.

Table VIII: Alkali – Electrolyte Formulation for Two-Phase Steaming

Chemical	**Amount**
Sodium carbonate	150.0 g
Common salt or Glauber's salt	150.0 g
Potassium carbonate	50.0 g
Sodium hydroxide, 38° Bé	10.0 – 50.0 ml
Bulk with water to total volume	1.0 liter

This formula will give satisfactory results with all types of equipment and prints.

It is critical that the printed fabric should not dry out during steaming and that the prints feel moist when leaving the steamer. The risk of drying-out is particularly great if the steam supply is not adequate. Heat coils in the steam chamber will dry out the inside of the box and result in fabric and color damage. In such a case, the steam supply should be increased. This will assure adequate steam for condensation and reduce the temperature in the steamer. Two-phase steamers are now using internally heated superheated steam to effect a more rapid condensation on the fabric and thus a more rapid fixation of the dye.

A more stable padding liquor is the Alkali – Silicate system, as shown in Table IX. With this system, the prints are padded though the bath and steamed in the same manner as the electrolyte system. The danger of drying out or discoloring is much less of a problem. The two-phase loop steamer will handle knitted fabrics and fabrics with poor dimensional stability. This method is more universal and could accommodate both woven and knitted styles.

Table IX: Alkali – Silicate Formulation for Two-Phase Steaming

Chemical	**Silicate Solution**	**Silicate Plus Solution**
Sodium silicate, 42° Bé [6]	900 ml/L	900 ml/L
Sodium hydroxide, 50% [7]	100 ml/L	10 ml/L
Sodium carbonate	0 g/L	0-50 g/L as needed
Bulk with water to total volume	1 liter	1 liter

Two-Phase Flash Age Steaming Method:
The flash age steaming method uses the short tower flash ager. This method takes advantage of the rapid fixation possibilities with reactive dyes. The print paste is the same as for other two-phase procedures and the alkali formulation choices are contained in Table X.

Table X: Alkali Formulation for Two-Phase Flash Age Steaming

Chemical	**Electrolyte Solution**	**Silicate Solution**
Common salt	150 g/L	
Sodium carbonate	150 g/L	50 g/L (if needed)
Potassium carbonate	50 g/L	
Sodium hydroxide, 50%	72 cc/L	90 cc/L
Sodium silicate, 42° Be'		700 cc/L
Bulk with water to	1 liter	1 liter

The fabric is padded and then is transported to a flash ager and steamed 8-15 seconds @ 130°C (266°F). Washing is done immediately after steaming. Best results occur if the padder, steamer, and washer are in tandem. Details on washing are contained in the next section.

Most woven fabrics are suitable for both the two-phase loop steamer and the faster fixation two-phase flash ager.

PRINT WASHER SET-UP

All fiber reactive dye printing requires one common processing component: good after washing.

Ingredients For Successful Print Washing Are:

- Water utilization of 25-30 liters/printed yard
- Temperature gradation of 90°-95°C (194°-203°F) to get the wash fastness results
- First open width - then rope to remove all residual products

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[6] The amount of sodium hydroxide is dependant on the grade of sodium silicate. The needed ratio is: Sodium Silicate 42°Bé (1:3.0) @ 20-30°C. The ratio of 1:3.0 refers to the NaO_2% vs. SiO_2% Ratio.

[7] Pad bath may need adjustment with sodium hydroxide or heating to produce a minimum wet pick-up.

The following six points should be part of an adequate washing range set up to get good dyestuff print washing results.

- Power sprays are very useful in re-wetting and removal of thickener, alkali and dye.
- Water is essential. Rather large volumes are necessary for washing fiber reactive printed cotton fabric. Water use of 25-30 liters per yard processed is the volume needed to adequately get good wash results.
- Temperature and counter flow are the controlling factors in good washing.
- Detergent use must be avoided to get good fiber reactive print washing.
- An emulsifier can be used with cotton/polyester fabrics to remove and keep all disperse dye particles in suspension.
- The best test to see how well the washer is doing is the "accelerated color transfer test". Take a sample right after washing. Do not dry. Lay the sample face down on a white fabric and press with a hot hand iron 10 seconds. The white fabric should remain clean.

PRINT SYSTEM: DISPERSE DYES

Background on Disperse Dyes

Many dispersed dyestuffs are either azoic-coupled pigments or vat-based colorants that are easily dispersed into synthetic polymer fibers. Disperse dyes are organic compounds which are colorants that are not water soluble but are easily dispersed in a synthetic fiber by heat and the use of dispersing compounds. These colorants have affinity and solubility in the synthetic fiber.

Disperse dyes are classified as low, medium, or high-energy dyes. This classification refers to the amount of energy required to fix these dyes on the fiber. Disperse dyes are solids that will convert to a gas without going through a liquid state at a certain elevated temperatures. This process is called "sublimation" and the temperature for each dye is its sublimation point. This process is the means by which the dye disperses into the polyester fiber. Low energy dyes have a low sublimation point and high-energy dyes have a higher sublimation point. In order to get good results in shade control and run-ability, the energy level of the dyes used in combination must be in the same range. Dyes that are easy to fix will come off the fiber just as easily. The expression used to describe this is "Easy On – Easy Off".

Although disperse dyestuffs are not water-soluble; all primary dyeing methods are water based applications. This goes for printing as well. The dye is carried to the fiber in a print paste which is water based. All the chemicals in the print paste are water-soluble or easily mix with water (miscible) with the one exception being the disperse dye. The disperse dye is dispersed by surfactants that are easily dissolved in water (hydrophilic) and aid in the miscibility of the disperse dye. This same process is used for vat and pigment colorant dispersions.

The disperse dye print paste contains:

- Water
- Thickener
- Disperse dye fixation agent (optional)
- pH control agent
- Anti-reducing agent (if needed)
- Disperse dye

Stock Thickeners and Print Paste

Disperse dyes can be printed with a combination of synthetic and natural gum thickeners or with a straight natural gum thickener. Natural gum thickener is added to the synthetic thickener formula to improve print flow characteristics. Tables XI and XII detail the stock formulations and Table XIII contains the print paste formulation.

Table XI. Synthetic Thickener Stock

Chemical	**Amount**
Water	900 ml/kg
Sequestering agent[8]	5 g/kg
Natural gum thickener[9]	10-30 g/kg
Synthetic thickener conc.	30 g/kg
Bulk with water to	1 kilogram

Table XII. Natural Gum Thickener Stock

Chemical	**Amount**
Water	800 ml/kg
Sequestering agent	5 g/kg
Citric acid	5 g/kg
Fixing agent[10]	10-20 g/kg
Sodium alginate (low viscosity)	80 g/kg
Starch ether	20 g/kg
Bulk with water to	1 kilogram

Table XIII. Print Paste Formulation

Chemical	**Amount**
Stock thickener	600-800 g/kg
Disperse dye liquid[11]	X g/kg
Bulk with water to	1 kilogram

Since fixation of disperse dyes on polyester is almost always done by high temperature steaming or by baking in an oven at high temperature (Thermasol), an anti-reduction agent is not needed. If the fixation is to be done by pressure steaming, 5 - 10 g/kg of sodium chlorate should be added to the print paste. *Navy* and *black* shades are especially affected by the reducing nature of steam.

There are several steps needed to achieve dye fixation in the printing process. These steps are detailed in Table XIV. Once the fabric is printed, water is removed by drying. Next the fabric will be put through a steamer or oven and energy, as heat, is applied to disperse the dye into the fiber. After the fabric passes through the fixation apparatus, the fabric must be washed to remove the remnants of the print paste and any unfixed dyestuff.

[8] Phosphate based water softener. Sodium Hexametaphosphate

[9] Starch ether, sodium alginate or Guar gums are used alone or in combination to modify viscosity flow and performance characteristics.

[10] Agents available form Ciba, Clariant, Lanxess, etc.

[11] Disperse Liquid Dispersions are available form most dyestuff manufacturers.

Table XIV: Procedure and Flow

	High Temp Steam	Thermasol	Pressure Steam
Print			
Dry	95º-130ºC (200º-265ºF)	95º-130ºC (200º-265ºF)	95º-130ºC (200º-265ºF)
Fixation	7 min @ 175ºC (347°F)	60-90 sec. @ 200-220ºC (392°-428°F)	30-45 min. @ 3 bar
Rinse	Dispersing agent and sequesterant		
Reductive scour	Sodium hydrosulfite and sodium hydroxide		
Final wash	Dispersing agent		
Dry			

Since fixation on polyester is almost always with high temperature steam, the time in the steam chamber varies depending on the process and colors. Steaming time is in the range of 5-10 minutes. The condensation of the steam on the fabric provides the heat for fixation. This process enables good fixation without overriding fabric heat-set memory. Disperse dyes have a tendency to stain polyester after fixation in the washing step. This stain is hard to remove by washing only, and a reductive scour is incorporated in the washing procedure to chemically destroy the color.

PRINT SYSTEM: FIBER REACTIVE/DISPERSE DYES PRINTING

Printing Cotton/Polyester Blended Fabrics

This segment of the printing industry is large, with some estimates as much as 1 – 2 billion meters of cotton/polyester being printed yearly. Much of this total is printed with pigments, which consigns much of the product to lower profit lines of fabrics. Many 100% cotton fabrics are in higher profit lines due to fiber reactive dyestuff prints. In many cases, soft handle dictated that these dye printed fabrics be non-resin finished. This may result in shrinkage or home laundry appearance issues. Cotton can be blended with polyester to enable the fabric to be heat-set for improved dimensional stability. Printers can print these blended fabrics with mixtures of disperse and fiber reactive dyes and duplicate to an extent, the 100% cotton, high end product that commands a premium in the market.

Print Paste Formulation

The colors used for this process are formulated by the print plant to include the following color inventory in Table XV.

Table XV: Stock Color Pallet

Neutral Yellow	Violet	Brown
Orange	Navy	Black
Scarlet	Blue	Gray
Red	Turquoise	

The mix ratio of reactive/disperse dyes can be handled two ways for plant simplification. In both cases color palette and fastness considerations are conditional on customer requirements.

- Method –1: Dyestuff manufacturer can specify which dyes to use. Plus, they can provide formulas for compounding stock colors.
- Method –2: A Do-It-Yourself system would require selecting dyes and making sample prints to determine proper mixing ratios.

Selected liquid disperse dyes and vinyl sulfone fiber reactive dyes are mixed in the print paste at predetermined amounts and printed in a neutral print paste. The special requirements for the disperse dye printing means that alkaline print pastes have to be avoided or the resultant prints are at best second quality. This special neutral print paste requirement is detailed in Table XVI, and the print formula in Table XVII. The printed fabric is first high temperature steamed and then two–phase flash aged.

The wet and dry fastness testing is better than with one-phase fixation with alkali and the process does not affect the colors adversely. In fact, in many cases brighter colors can be achieved.

Table XVI. Neutral Stock Thickener Formulation

Chemical	**Amount**
Sodium alginate low viscosity (LV)	60 g/kg
Sodium hexametaphosphate[12]	10 g/kg
Oxidative compound	10 g/kg
MSP (mono-sodium-phosphate)[13]	4 g/kg
Stain control agent[14]	20 g/kg
Bulk with water to	1 kg

Table XVII. Print Paste for Reactive/Disperse Dyes

Chemical	**Amount**
Stock thickener	600 – 800 g/kg
VS fiber reactive dye liquid	Y g/kg
Disperse dye liquid	X g/kg
Stain control agent[15]	10 – 20 g/kg
Bulk with water to	1 kg

Disperse dyes have a tendency to stain cotton fibers in the printing process. These stains are difficult to remove in washing and affect the finished shade of the print. The use of an anti-staining compound, or amphoteric agent, is very helpful in getting good results from this process. This low staining characteristic allows easy removal of the unfixed dye in the washing procedure after dyeing. The advantages of disperse dyes fixed independently of the reactive dyes coupled with the two-phase procedure gives the printer an ample tool to produce high quality prints on cotton/polyester fabrics.

[12] Sodium hexametaphosphate (Calgon-T) sequestering agent to prevent hard water ions, especially calcium, form forming insoluble alginate salts which prevent thickener removal in washing.

[13] Buffering agent to maintain pH of print paste less than 7.0.

[14] Remol® ASN (Clariant) dispersing and amphoteric agent

[15] Amphoteric agent is a dispersing agent with both acid and basic properties and thus can prevent certain anionic – cationic reactions.

Process Recommendations for Cotton/Polyester Printing

1. Print Paste Formulation: Disperse and fiber reactive dyes formulated in correct ratio for shade requirement. Must have anti-stain agent.
2. Print: Print as for fiber reactive dyes. Print black heavy blotch shades in last position.
3. Drying: For disperse dyes, watch for excessive drying on fabrics, which will cause yellowing or scorching of the cotton.
4. High Temperature Steaming Conditions: 6-8 minutes @ 165°-175°C (329°-347°F)
5. Pad Alkali:
    a. Sodium hydroxide + electrolyte fixing bath
    b. Sodium hydroxide + silicate fixing bath
    c. Use either bath.
6. Flash Age Steam or Two-phase Steaming:
    a. Steaming for woven fabrics can be done in a "Flash Ager". 30 to 60 seconds at 125°-130°C (257°-266°F) is recommended. This varies with equipment and capacity and with printed fabric construction.
    b. Both woven and knitted items can be processed on a two-phase loop steamer. Steaming conditions are 1 to 3 minutes at 125°-130°C (257°-266°F).
7. Spray: Power sprays are crucial to getting the brightest shades.
8. Wash: Wash using a good emulsifying detergent.
9. Dry and Finish: Fabric should be dried open width. Finishing may include resin or addition of softener but will always include framing to finish width.

TROUBLESHOOTING

What Is Print Paste Stability? Reactive Dyes have a limited stability due to hydrolysis. Some of the dyes are only stable for 1-2 days if the temperature is above 26ºC (80ºF). A good rule of thumb is to print patterns within 24 – 48 hours and it is important that a sample "Strike-Off" print be checked before printing production yardage.

Are There Special Considerations For Shade Matching? The metameric matches required for some end uses will not be attainable if optical brightener is used in the finish mix. Fabric pH should be 6.0 - 7.5. Not much variation either way (acid or alkaline) should be tolerated.

What Is The Ideal Print Paste Viscosity? Flow characteristics are much more important to good color lay down than viscosity numbers alone. Use of a low viscosity sodium alginate will provide an adequate coverage on most cotton fabrics. The use of a high viscosity sodium alginate, on the other hand, provides a print paste that can produce very sharp lines.

Sodium alginate is a natural thickener and is very resistant to shear stress. This means that the thickener will flow at a certain rate due to temperature and thickener concentration but will not be influenced by shear stress the way pigment thickeners react. The standard viscosity for the alginate based print paste should be 1500 cps – 2500 cps at 20º– 30ºC (68°-86°F) when measured on a Brookfield viscometer at 20 rpm with a # 6 spindle.

- Outlines should be at the upper end of this scale at 2400 cps – 2500 cps.
- Most pattern work should be in the middle of the range or 2000 cps.
- Blotches may require two screens for good coverage. The first screen should be 1500 cps – 1600 cps. While the second screen will cover better on this platform with a viscosity in mid or upper range: 1800 cps – 2500 cps.

One phase print pastes contain alkali, which deteriorate the thickener in one specific way. This effect results in lower viscosities over time. This condition is exacerbated by heat of 30ºC (86ºF) and upwards.

What Precautions Should Be Observed In Processing Fastness Checks? The washed fabrics should be dried and checked for cold-water bleeding and wet and dry crock. If needed, a good cationic fix should be included in the pad on the tenter frame in drying. If the goods do not need a fix, they can be left in buggies to drain.

What is Two-Phase Wet Fixation? This two-phase method uses a stainless steel chamber filled with a heated fixation solution. The fabric is passed through the chamber and supporting rollers keep the fabric in contact with the solution for up to 12 seconds. The fabric exits the chamber and goes immediately into an after washing as for two-phase prints.

ADDITIONAL PROCEDURES AND FORMULATIONS

Two-Phase Wet Fixation

Although not commonly used in the United States, two-phase wet fixation is an extremely rapid process. The alkali formulation for this process is found in Table XIII.

Table XIII: Alkali Formulations for Two-phase Wet Fixation:

Chemical	**I**	**II**	**III**
Common salt	100.0 g		250.0 g
Glauber's salt		100.0 g	
Sodium carbonate	150.0 g	150.0 g	
Potassium carbonate	50.0 g	50.0 g	
Sodium hydroxide, 38°Bé	70.0 g	70.0 g	70.0 g
Bulk with water to final volume	1 liter	1 liter	1 liter

These liquors have a specific gravity of 25-28°Bé at about 95°C (203°F). Constant specific gravity is essential for the reliability of the process. If the fixation liquor becomes diluted with condensation water, which may occur when the trough is heated directly or water taps are not tightly shut off, considerably more dyestuff dissolves from the printed fabric into the fixation liquor, particularly if the specific gravity is allowed to fall below 20° Be.

Two-Phase Cold Dwell

Another less frequently used means of fixation is the Two-Phase Cold Dwell. Prints can be fixed by this method without heat treatment. The print pastes are the same as for two–phase printing and contain no alkali or urea, as outlined in Table XIX. After drying, the printed fabric is padded either with an alkaline-electrolyte solution or a sodium silicate solution.

Table XIX. Alkali Formulations for Two-phase Cold Dwell Fixation:

Chemical	**Alkaline – Electrolyte Solution**	**Sodium Silicate Solution**
Sodium Sulfate	180 g	
Potassium Carbonate	50 g	
Sodium Carbonate	150 g	
Sodium Hydroxide 38°Bé	100 ml	
Cold Water	700 ml	100 ml
Sodium Silicate 50°Bé	---	900 ml
Total	1000 ml.	1000 ml

Process Outline for Two-Phase Cold Dwell

1. Print
2. Dry
3. Pad, crease-free
4. Air passage, 20 seconds
5. Batch, covered, for 3-24hrs at 20°C(68°F)
6. Wash
7. Dry

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Importer Support Program

The Cotton Board and Cotton Incorporated are funded by U.S. upland cotton growers and importers of cotton and cotton products (this includes raw cotton, piece goods, and finished apparel). A percentage of the importer funds are devoted for importer specific programs organized under the Importer Support Program. Examples of projects funded from this fund include training schools, educational programs, focus groups, economic meetings, and research initiatives.

Selected technical issues have been identified by importer members as relevant to their business. This report is a condensed, less technical report of those issues intended to provide the reader with basic, yet useful information on the topic.

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