

TECHNICAL BULLETIN



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ISP 1016

PRINT CHEMISTRY OF VAT DYES AND VAT DISCHARGE

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

INTRODUCTION

This bulletin will outline the chemistry and considerations for both the fixation of printed vat dyes and discharge printing with vat dyes. Vat colors are substances that can undergo a reversible chemical reduction and oxidation. The oxidized form of the dyestuff is an insoluble pigment whereas the reduced compound is soluble in alkaline solution and has an affinity for cotton (cellulose) and exhibits a drastically different shade compared to the oxidized state.

Since vat dyes are manufactured as the insoluble pigment known also as the oxidized form of the dye, they must be chemically reduced to the soluble or “leuco” form of the dye. This leuco form of the dye allows the transfer of the dye from the surface of the fiber to the interior region of the cotton fiber. After this transfer, the system is chemically converted so that re-oxidation of the dye can take place, thus trapping the dye in the fiber. The process then requires a hot wash procedure 93°C (200°F) to develop the crystalline form of the dye and the final desired shade.

Vat dyes are divided into the two following classes:

1. Anthraquinone derivatives
 - The major group of dyes
 - Superior in fastness, especially light fastness, to the Indigoid based dyes
2. Indigoid derivatives
 - Limited use in dyeing and only used for special shades due to fastness
 - Easily converted to the leuco form and used in discharge printing

These designations indicate molecular structural differences, which determine the reactivity and printing (dyeing) characteristics and the relative suitability of the vat dye to give the desired fastness result.

While vat dyes are unable to achieve shades of extreme brightness, they do offer a wide range of shades and have innately good fastness. A vat pallet, listing with dyestuff suitability for the one-phase and two-phase procedure appears at the end of the bulletin.

Vats are printed using either the one-phase or two-phase printing process. Two-phase printing is not sensitive to air degradation and is much more forgiving during printing, drying, and storage. Due to the sensitivity of the vat reducing agent to air, the one-phase process is not used for printing on white grounds very much anymore. However, the one-phase process is used widely for discharge printing of dyed fabrics. The fabrics to be printed must have been dyed with a dischargeable dye. These are listed in Table XV and XVI.

PREPARATION FOR PRINTING

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

Preparation includes 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 moles.
- In the case of cotton woven fabric, it is advantageous although not necessary that the fabric be causticized or mercerized to maximize color value and print appearance as well as increasing fabric luster and sheen. The concentration of sodium hydroxide (NaOH) needed to causticize cotton is 22°Bé. To produce mercerized fabric, the NaOH concentration needs to be increased to 28-32°Bé.

The dyestuff printer needs a fabric with the following properties:

- Uniform absorbency
- Clean (no lint)
- White
- Uniform width
- No creases
- Neutral pH (7.0 - 7.5 is best for all printing)
- Alkalinity ≤ 0.05
- Causticized or mercerized

PRINTING PROCESS

Vat dyes can be printed by the following procedures:

1. One-Phase Printing
2. Two-Phase Printing

The two-phase process accounts for almost 100% of vat print application. The resultant color prints are very fast to washing and rubbing. The majority of vat prints are found in the home market in upholstered goods, drapery, and decorative items, which requires these vat printed fabrics to meet their color fastness specifications. The only apparel market that requires similar fastness specifications is the military camouflage apparel sector.

One-phase printing is used almost exclusively in vat “discharge” applications for the fashion apparel market. Discharge is where one color is destroyed and another may be put in its place.

PRINT SYSTEM: TWO-PHASE PRINTING

Two-phase printing follows the typical range dyeing procedure where the vat dyestuff in oxidized pigment form is printed without reducing chemicals and dried. Then the fabric is treated with a chemical bath containing the reduction chemicals and alkali, after which, it is steamed and washed. These flow and process details are listed in Table I.

Table I. Two-Phase Printing Process – Flow

Process	Description
1. Print	No reducing chemicals
2. Dry	No restrictions
3. Pad	Reducing agent & alkali applied
4. Steam – Flash Age	Immediately after padding
5. Spray & Rinse	To reduce alkalinity and begin oxidation
6. Oxidize	To convert vat back to pigment form
7. Soap	To achieve final shade
8. Dry	Dried and finished in open-width form

The two-phase process was developed to print vat dyes with a high redox potential. Redox is short for Reduction Oxidation and is a measure of a system’s capacity to reduce and oxidize material. Dyes with a high redox potential have better fastness properties. These dyes require a stronger reducing chemistry; therefore, sodium hydrosulfite or “Dyeing Hydro” is used in two-phase printing.

If the reducing agent is not strong enough to completely reduce the dye, poor color yield is the result. The two-phase print paste formulation contains no reducing agent or alkali because the reducing agent and alkali are applied after printing. See Tables II and III for details.

Table II. Two-Phase Stock Thickener Formula

Chemical	Amount, g/kg
Water	825 - 905
Starch ether ¹	55
Guar or guar ether ²	40
Sodium alginate (8% low viscosity) ³	(0 – 80)
Total	1000

Table III. Two-Phase Print Paste Formula

Chemical	Amount, g/kg
Vat Dye ⁴	1-80
Stock Thickener	550 – 750
Auxiliaries ⁵	X
Water	Bulked with water to
Total	1000

Notes:

- 1 - Promotes surface coverage printing and thereby increasing color value.
- 2 - Promotes a longer flow that increases print coverage uniformity.
- 3 - May need to be used to give better print outlines and edges. Blended products are available.
- 4 - The use of excessive dye may cause fixation problems.
- 5 - These auxiliaries include various as-needed products to improve running and printing properties.

The viscosity of the print paste is maintained between 5000 and 8000 centipoises (cP) @ 20 rpm on a Brookfield Viscometer using spindle number 6. In cases where the printer has incorporated a blend of natural thickener and synthetic thickener, the viscosity is maintained more like a pigment print paste: 17,000-18,000 cP using the same measurement parameters.

One advantage to the two-phase process is the increased reliability of printing, drying, and storage of the printed and unfixed fabric. No specific handling is required and storage can be indefinite if the fabrics are protected from water spots and other storage area contaminants. Storage on large A-frames is possible but is not practical with the one-phase system.

After printing, the fabrics are padded face-side out using the kiss roll application technique and usually only the printed face is in contact with the fixation solution as shown in Figure 1. The fixation solution formula is detailed in Table IV.

If the fabric is completely immersed in the fixation solution prior to the nip roll then the wet-pick-up will be increased to the point that flushing of the print can occur. Flushing is a horizontal wicking of the print motif.

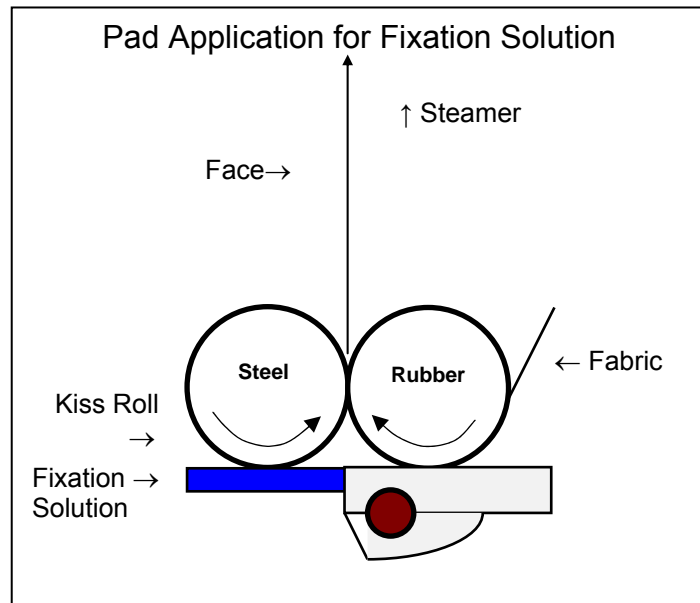


Figure 1. Face Only Padding

Table IV. Fixation Solution

Chemical	Amount
Water	600 ml/L
Sodium hydrosulfite	100 g/L
Sodium hydroxide, 50%	70 ml/L
Sodium carbonate ⁶	50 – 100 g/L
Auxilliaris ⁷	X g/L
Water	Bulk with water to
Total	1000 ml/L

The percent of active reducing agent in the fixation bath can be determined by titration. The optimum fixation range is 50–70g/L hydrosulfite. A spot test is available using a reduction paper, but this only tells if hydrosulfite is present, not how much is present.

Notes

6 - Soda ash is used only when necessary to improve outlines.

7 - Auxilliaris include surfactants, metal salts, and other agents to assist processing the fabric to improve fixation and quality.

After padding, the fabric is guided into the flash ager. The ager is designed to use super-heated steam at 125°-130°C (257°-266°F). The fabric is supported on the back and travels vertically for 2.0-6.0 meters and then reverses direction and vertically descends to an exit. At this point, for the first time, the face of the fabric is contacted by a transport roller and is conveyed out of the steamer exit. The face of the fabric is untouched until the dye fixation is complete and no chance of smearing or other defiling of the print can occur. In this steamer design, the total steaming time can vary from 20-30 seconds to 45-60 seconds depending on fabric weight and print design.

After exiting the steamer, the fabric usually gets a short, forced air cool-down. Then the fabric is passed through an aggressive open-width spray compartment where it is impinged with both fresh and re-circulated water to reduce pH and alkalinity.

Still in open width configuration, the fabric is passed through an oxidation bath. A constant pump fed mix of hydrogen peroxide and acetic acid is used. Concentration in the bath:

Hydrogen Peroxide, 35%	5.0-8.0 ml/L
Acetic Acid, 56%	2.0-3.0 g/L

The concentration of hydrogen peroxide in the oxidation bath can be controlled by titration. A minimum acceptable range of 2.0 g/L should be maintained.

At this point, the fabric enters a hot wash. Washing with detergent and sodium carbonate, constant agitation, and temperatures of 90°-95°C (194°-203°F) are needed to develop the true shade.

Bath Concentration:

Sodium Carbonate	5.0-10.0 g/L
Detergent	2.0-3.0 g/L

The fabric is then passed through a hot rinse at 40°– 50°C (104°-122°F).

Table XIV contains a pallet with recommendations for shade production.

PRINT SYSTEM: ONE-PHASE PRINTING FOR VAT DISCHARGE

Discharge printing is the term used to describe the destruction of a dyed shade by a chemical process in such a manner that a pattern is imparted to the dyed fabric. The sequence begins with a dyed fabric. Vinyl sulfone (VS) fiber reactive dyes are the products chiefly suited for ground dyeing, but direct dyes and naphthol-azoic dye combinations have also been used.

The discharge agent, sodium sulfoxylate formaldehyde, is applied to the dyed fabric via printing. The dyed fabric is then processed to achieve the discharge. First, the color chromophore is reduced and eliminated. Then the dye linkage to the fiber is broken and these by-products of discharge are removed in washing. Vinyl sulfone dyes, as a class, are readily color dischargeable and the vinyl sulfone link is easily destroyed by an alkaline print paste.

If no color is added to the discharge print paste, the result is a white discharge. However, if a vat dye is added to the discharge print paste, the discharge agent simultaneously reduces the vat dye and enables it to color the cotton fiber. In this case, the vat dye is the illuminating or discharge color.

Discharge is only carried out by reduction. The introduction of sodium sulfoxylate formaldehyde, Rongalit® C by BASF, in 1905 made a decisive contribution towards simplifying dyestuff discharges. Since then, this product and its competitors have played the predominant role in the discharge of reactive dyes on cotton fabrics. The different steps in the discharge printing process are outlined in Table V.

Table V. Discharge Printing Process Flow

<ol style="list-style-type: none"> 1. Production of the finished ground shade dyeing. 2. Treatment with mild oxidizing agent⁸ to prevent contamination and halos. 3. Print with discharge print paste. 4. Fixation in air-free steam of the print with destruction of the ground shade. 5. Oxidation and washing.

Fabric Requirements

Ground shades for discharge must be produced with dyes that are dischargeable. Dyed fabrics must be padded with a mild oxidation (anti-reduction) compound⁸ to prevent halos and produce sharp discharges. The fabric is then framed and dried before printing.

Preparation of The Printing Pastes

The one-phase vat printing process is the basis for the vat discharge process. Enough reducing agent is needed to achieve both an adequate discharge and good fixation of the illuminating color. Table VI contains the stock thickener formula for discharge printing.

Table VI. Stock Thickener Discharge Paste Formula

Chemical	Amount, g/kg	Details
Thickener ⁹	120	Guar/starch blend
Water	300	
Humectant ¹⁰	30-60	Glycerin
Potassium Carbonate ¹¹	50-70-120	
Sodium Carbonate ¹²	70-40-30	
Printing Hydro ¹³	220	Reducing agent
Water (bulk)	Bulk with water to	
Total	1000	

Notes:

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

9. The blended products are sometimes prepared as a concentrate, which is a wet slurry, and the amount must be determined by trial.

10. This amount may vary, also, due to fabric construction and steam quality.

11. This amount may vary depending on steamer quality and fabric construction.

12. Sodium carbonate is sometimes required to improve the discharge only. Notice that as the potassium carbonate is increased the sodium carbonate is decreased. This balance works well.

13. The amount required for discharge and good dye fixation is sometimes double the amount needed only for fixation. The product is as for one-phase printing – Sodium Sulfoxylate Formaldehyde.

One-phase printing uses a stabilized form of the reducing agent known as “printing hydro”. The print paste contains this printing hydro, alkali, and a humectant with the vat dyestuff in the oxidized pigment form. Table VII contains the discharge print paste formula.

Table VII. Discharge Print Paste Formula

Chemical	Amount, g/kg	Details
Vat Dye ¹⁴	X	
Stock Thickener	700	Guar/starch blend
Reducing agent ¹⁵	0-60	Sodium sulfoxylate formaldehyde
Water	Bulk with water to	
Total	1000	

The print viscosity will be maintained in the 4500 – 8000 cps range (Brookfield Viscometer: 20 rpm with spindle # 6). When using the premixed slurry concentrates, the printer has minimal control over viscosity but these products are formulated to get consistent results.

One-Phase Discharge Print Process

The discharge printing process and details are as follows:

1. Print
 - a. On dyed ground using the formulas found in Tables VI and VII.
2. Dry
 - a. Care is taken to minimize exposure to air preventing premature decomposition of the printing hydro.
 - b. After drying, fabric should not be stored but steamed as soon as possible.
3. Steam Fixation
 - a. Steam 10 minutes at 102°C (215°F).
 - b. Steam in air-free steamer with good moisture. Steam quality is critical for good results. In all cases, it is desirable that the steam be re-saturated by means of auxiliary saturating device such as a steam saturator to prevent fabrics from drying¹⁶.
4. Storage
 - a. Storage of steamed, printed fabrics should be minimized.

Notes:

14. The concentration of the vat dye, in most cases, should be controlled and limited to 80-100 g/kg of a single strength paste.

15. Additional reducing agent may be added depending on discharge requirements.

16. A steam saturator is an enclosed chamber used to obtain saturated steam using boiler produced and transported steam. Steam from the boiler is entered into a water sump and then allowed controlled passage upwards to an outlet that feeds the cloth steamer. In other words the line steam is condensed and then re-boiled as saturated steam. This requirement is critical for achieving good results in discharge printing.

5. Rinsing

- a. Cold water in power sprays should be used initially if at all possible. Utilizing recycled water plus fresh water is a possibility. In any event, the fabric is processed in open width.
- b. Next, pass through saturator which is kept at 40°C (104°F) with a constant pump feed mix of:

Hydrogen Peroxide, 35%, 5.0-8.0 g/l
Acetic Acid, 56%, 2.0-3.0 g/l

6. Hot Wash

- a. Washing with constant agitation at 90°–95°C (194°-203°F) with detergent and sodium carbonate is needed to remove by-products of discharge and develop vat dye shade. Bath concentration recommendation:

Sodium Carbonate 5.0-10.0 g/l
Detergent 2.0-3.0 g/l

- b. Followed by a hot rinse at 40°–50°C (104°-122°F)

7. Opening and Drying Fabric for Finishing

- a. The washed fabric should be dried open width.

A selection of suitable vat dyes is found in Table XIII. *Vat Dyestuffs Suitable For Printing Application*. Table XIV contains vat dyes suitable for the two-phase printing process. A selection of dischargeable dyes is provided in Table XV. *Pallet Listing: Dischargeable Fiber Reactive Dyes*. Furthermore a guide for the production of dischargeable ground shades is found in Table XVI. *Selection For Dischargeable Ground Shade Production: Fiber Reactive Dyes*.

Pallet Selection: Vat Dyes

Table XIII. Vat Dyestuff Suitable For Printing Application:

Dyestuff Name	Color Index #	2-Phase	Discharge
Flavone GC	Yellow #2	-	Yes
Yellow F3GC	Yellow #33	Yes	-
Gold. Yellow RK / Gold. Orange 5G	Orange #1	Yes	Yes
Orange GR	Orange #7	Yes	Yes
Bordeaux RR	Red #15	Yes	Yes
Brill. Pink RB	Red #1	(Max.20 g/kg)	Yes
Brill. Red F6B / Red TFBB	Red #10	Yes	Yes
Red Violet 2RN	Violet #3	-	Yes
Indigo Blue O4B	Blue #4	-	Yes
Blue TCLF	Blue # 66	Yes	
Blue GC	Blue #14	Yes	-
Blue BC	Blue #6	Yes	-
Navy Blue TG	Blue # 16	Yes	
Brill. Green FBB / TFFB	Green #1	Yes	Yes
Brown HRR	Brown #57	Yes	Yes
Olive T / TT	Black #25	Yes	-
Printing Black G	Mix	Yes	Yes
Sulfur Carbon CLG	Sulfur Black #7	Yes	Yes

Table XIV. Two-Phase Printing Recommendations For Shade Matching: Vat Dyes

Lemon Yellow	Yellow / Golden Yellow / Gold / Bronze
Yellow 33 Red 15 Orange 1 Green 1	Yellow 33 Red 15 Orange 1 Green 1 Brown 57 Black 25
Orange / Scarlet / Red / Rust	Red / Ruby / Wine / Burgundy
Orange 7 Yellow 33 Red 15 Orange 1 Black 25	Yellow 33 Red 15 Orange 1 Red 10 Violet 3 Blue 66
Maroon / Violet / Purple	Royal / Blue
Red 15 Orange 1 Red 10 Violet 3 Blue 66 Brown 57	Red 15 Orange 1 Red 10 Violet 3 Blue 66 Green 1 Black 25
Jade / Emerald / Green / Turquoise	Forrest Green / Bottle Green
Blue 66 Green 1 Black 25 Yellow 33 Red 15	Blue 66 Green 1 Black 25 Yellow 33 Red 15 Brown 57 Blue 16 Orange 1
Olive / Khaki / Taupe / Grey	Navy
Green 1 Black 25 Yellow 33 Red 15 Orange 1	Blue 16 Black Mix Violet 3 Brown 57

Continued next page...

Table XIV. Two-Phase Printing Recommendations For Shade Matching: Vat Dyes (*continued*)

Brown / Charcoal Brown	Black
Brown 57	Black Mix
Red 15	Orange 7
Orange 1	Yellow 33
Black 25	Green 1
Blue 66	Blue 66
Green 1	Blue 16
Black Mix	
Stable Tertiary (3 Dye) Combination – # 1	Stable Tertiary (3 Dye) Combination – # 2
Yellow 33	Orange 1
Red 15	Red 15
Black 25	Black 25
All shades less than 1.0 g/kg total dye	
Yellow 33	
Red 15	
Orange 1	
Green 1	
Black 25	

Pallet Listing: Dischargeable Fiber Reactive Dyes

Table XV. Pallet Listing: Dischargeable Fiber Reactive Dyes

Vinyl Sulfone Dyestuff	Color Index	Discharge	Max.Conc.	Good Results
Br. Yellow 4GL 150	Y160	Yes	50 g/l	*
Br. Yellow GL	Y37	Yes	50 g/l	*
Yellow FG	Y42	Yes	50 g/l	
Yellow GR	Y15	Yes	50 g/l	*
Gold. Yellow RNL	O107	Yes	50 g/l	
Gold. Yellow 3RA	O96	Yes	50 g/l	*
Br. Orange 3R	O16	Yes	50 g/l	
Br. Orange R-2R	-	Yes	50 g/l	
Br. Red 5B	R35	Yes	60 g/l	*
Br. Red F3B	R180	Yes	60 g/l	*
Br. Violet 5R	V5	Yes	30 g/l	
Blue BB New	BLU220	Yes	30 g/l	*
Turquoise G	B21	Partial	10 g/l	Very limited use
Navy Blue R-GB	-	Yes	60 g/l	*
Black B	Blk 5	Yes	80 g/l	*
Black GF-A	Blk 5	Yes	80 g/l	*

The use, even very sparingly, of both the VS Turquoise G-A (Bl 21) and the VS Brilliant Blue R (Bl 19) is not recommended if a consistently dischargeable ground is desired. Table XVI contains recommendations for matching shades with dischargeable dyes.

Table XVI. Selection For Dischargeable Ground Shade Production: Fiber Reactive Dyes

Black	Black B Navy R-GB Golden Yellow RNL Brill. Red F3B Yellow FG Br. Orange 3R	Navy	Black B Navy Blue R-GB Brill. Red F3B Br. Orange 3R EF Navy GG
Dark Green	Yellow FG Orange R-GG Navy Blue R-GB EF Navy GG Brill. Blue BB New	Blue	Brill. Blue BB New Navy Blue R-GB
Royal	Brill. Blue BB New Brill. Red F3B Brill. Violet 5R	Turquoise	Turquoise G-A Max.1% 10 g/L Brill. Blue BB New Navy Blue R-GB
Bright Green	Brill. Yellow 4GL 150 Brill. Blue BB New	Teal	Brill. Yellow 4GL 150 Brill. Blue BB New Navy Blue R-GB EF Navy GG
Bright Red	Brill. Red F3B Brill. Orange 3R Golden Yellow RNL	Red	Brill. Red F3B Brill. Orange 3R
Wine	Brill. Violet Brill. Blue BB New Brill. Red F3B	Maroon	Brill. Violet 5R Brill. Orange 3R Brill. Red F3B
Violet	Brill. Violet Brill. Red F3B Brill. Blue BB New	Scarlet	Brill. Red F3B Brill. Yellow GL Brill. Orange 3R
Orange	EF Orange GG Brill. Orange 3R EF Brill. Orange G	Golden Yellow	Yellow FG Golden Yellow RNL
Yellow	Yellow GR 110 Yellow FG	Lemon Yellow	Brill. Yellow 4GL 150
Beige-Taupе-Grey	Yellow GR 110 Brill. Red F3B Brill. Blue BB New	Brown	Yellow FG Brill. Red F3B Navy R-GB

TROUBLESHOOTING

How does air in the steamer affect the vat print? Air in the steamer immediately disables the reducing agent so little or no fixation will occur. The steamer box is a dynamic piece of equipment. Steam is passing through it at high volume. This motion can draft air into the box and the combination of air and steam results in very poor operating conditions. Finding and sealing leaks, and having a positive atmosphere to push steam out rather than pull air in will be helpful.

Poor fixation of vat dye usually results from one or two causes

- As mentioned, air in the steamer is a number one problem.
- Exceeding dyestuff recommendations is another possible cause. This is especially true in discharge printing. The reducing agent, in discharge printing, is required to bring about discharge of the ground shade as well as reduce and fix the vat dye. Discharge of the ground will occur first and the dyestuff fixation will be affected if the amount of vat dye has exceeded recommended levels.

Why did the ground shade not discharge completely to white? Poor discharge of the ground shade is most commonly the result of using a dyestuff that is not recommended for the discharge procedure. Most dyestuffs labeled as vinyl sulfone (VS) dyes are both color dischargeable and linkage dischargeable. There are dyestuffs that have multiple reactive groups, such as VS and monochlorotriazine (MCT) on the same dye molecule. The MCT group will not discharge and must not be used in any concentration for ground shades at all.

What can cause color specks, especially in pale shades, in two-phase vat printing? Vat dyes are manufactured in their oxidized form as a pigment. This requires that the dyestuff be ground to a fine particulate state and then dispersed by a surfactant into a water based paste or thickened liquid package. These dispersions are stabilized with a mild alkali and standardized by a printing or dyeing procedure. Most dispersion problems show up when the pigment particles begin coalescing into large agglomerates that show up as specks. These specks are especially noticeable in pale shades. However, the specks do not show up until after fixation has occurred. The problem can usually be corrected by redispersing the vat dyestuff package.

What can cause print to flush in two-phase processing through the fixation solution padder? Flushing is a common problem in two-phase printing and has common causes. Some of these are:

- Improper thickener selection with insufficient solids to keep the print sharp.
- Pad solution that is low in electrolyte will allow the print paste to flush even though the dyestuff is insoluble at the time of padding.
- Too much padding solution on the fabric due to extended contact with the fixation bath before the nip. Heavier fabrics may require immersion to get good fixation. The fixation solution should only be applied to the face side of lighter weight fabrics.

What causes water spots in the steamer? Water spots in steamer are almost always due to improper or insufficient maintenance. The steamer is designed to have closed steam coils in the entry and exit as well as in the roof and at other locations to prevent condensation of steam. When these coils become inaccessible due to the clogged traps, steam can condense and the

condensate can drip onto fabric before the dyestuff is fixed. Once the steam traps are cleaned and steam is reintroduced, condensate will not be formed.

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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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