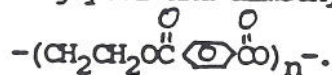


Dyeing Polyester: Disperse Dyes for Textile Conservation Work

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Abstract: In order to achieve both strength and invisibility in textile conservation treatments, sheer plain weave or net fabrics of nylon, polyester, or silk are often employed. "Stabiltex," the most stable sheer fabric, is only available in a limited solid color range, thereby restricting its utilization for the treatment of textiles. Dyeing methods for coloring this polyester fabric were not conducive to laboratory work until the development of thermofixation, now used industrially. This presentation will discuss two methods of thermofixation as a way of achieving a broad colorway and infinite design range on polyester fabrics needed in a textile conservation laboratory. The thermosol method will be recommended for larger quantities of fabric because the dye is padded onto the fabric before the heat treatment. For unique designs or odd shapes, the heat-transfer process would be used. Disperse dye is painted or printed on a release paper, then transferred to the fabric under heat.

History: Polyester is a synthetic polymer, generally made by the condensation of ethylene glycol and dimethyl terephthalate:



As a film, it is widely known by the Dupont tradename "Mylar;" as a fiber, polyester may be marketed as Dacron (DuPont), Kodel (with a slightly different chemical formulation), Fortrel (Celanese), Trevira (Hoechst), or Terylene (ICI). Commercial production of polyester began in the 1950's although it was discovered by two English chemists in 1941.

It is a highly oriented, tightly packed polymer, unaffected by water, (with a very low moisture regain of about 0.4%) and a relatively high glass transition temperature. Compared to natural fibers and to many other synthetic ones, it has good resistance to sunlight, to acids and bases, and to solvents [17].

In developing polyester as a commercial fiber, it was found that the "acetate dyes"—dyes suitable for cellulose

acetate—might be applicable with some modifications, because polyester is even more hydrophobic than cellulose acetate [3]. When the "acetate dyes" were introduced in the 1920's, dye application methods began to change.

For thousands of years, dyes had been applied to fibers from dye baths in which the dyes were in solution, as colored water or "dye liquors," and the fibers absorbed under certain conditions of temperature and pH. With these new synthetic fibers, which are unaffected by water (hydrophobic), solubilization was less important. As long as the dye was evenly "dispersed" in the water, that was adequate. Dyes without solubilizing groups were ground or milled with dispersing agents and sold as liquids or re-dispersible powders [3].

While these disperse dyes worked well with cellulose diacetate, cellulose triacetate and polyester fibers require a swelling agent or "carrier" in order to achieve good depth of shade. Carriers increase the amount of dye absorbed into the fiber. They are often based on phenolic compounds, like *o*-phenyl phenol (the active ingredient in Lysol[®]), but at high temperatures the fumes may be unpleasant or toxic. However, high temperatures promote dye exhaustion into the fiber.

Two application methods for polyester dyeing gained industrial success at the outset: 1) aqueous dyeing with 2-5% *p*-phenyl phenol for 60 minutes and 2) pressure steaming or high temperature dyeing without a carrier, but at 15-25 lb/in² pressure (1.0-1.8 kg/cm²) for 30-90 minutes at 110°-120°C. Characteristics of polyester fibers, such as their hydrophobic nature and dense molecular structure, make them difficult to dye. The rate of diffusion into the fibers is increased by heat (pressure dyeing or thermofixation) or by chemical additives (carriers). That is, heat or a carrier causes a loosening of the molecular structure. This swelling allows the dye to diffuse into the fibers. Dyebath temperatures of 265-280°C. are necessary for pressure dyeing. These conditions cannot be achieved with the low temperature, atmospheric dyeing equipment for wool, silk, or cotton found in most textile conservation laboratories. Likewise, the carriers are often toxic and noxious, making them unacceptable in small dyeing facilities.

A third method of dyeing polyester was introduced in 1958, called "thermosol" or thermofixation. Using the same dye class of disperse dyes, the dye is padded onto the fabric by dipping the cloth in the dye liquor and allowing the dispersed dye to dry on the cloth. The cloth, with the unfixed dye dried on it, is then passed through an oven heated to 390° to 425° F. for

45-90 seconds [6, 12, 13]. In the oven, the high temperature substitutes for a swelling agent while concomitantly causing the dye to diffuse into the fiber.

The best disperse dyes for this method are those of suitable molecular weight and size, which can penetrate the fiber at the proper time. Disperse dyes sublime—they change from a solid (the dye particle dispersed in water and then on the fabric) to a gas, without going through a liquid phase. If the dye is too volatile, it can sublime before the fiber reaches its proper (and most open) temperature. If the dye is too non-volatile, it will not sublime well enough at the highest temperature to dye the fiber [9, 11, 17]. However, disperse dyes with high resistance to sublimation are preferable to those with low resistance when permanent press, heatsetting, pleating, and ironing are going to be in the subsequent finishing of the fabric [6, 20]. Thus, the end-use of the fabric as well as the dyeing method have to be considered.

Dyes of high heat stability (for treatment at temperatures of 390^o-400^o F.) are suggested: C.I. Disperse Yellow 42, C.I. Disperse Yellow 88, C.I. Orange 77, C.I. Disperse Orange 145, C.I. Disperse Red 153, C.I. Disperse Red 305, C.I. Disperse Red 339, C.I. Disperse Blue 27, C.I. Disperse Blue 77, C.I. Disperse Blue 79, and C.I. Disperse Blue 337 [14].

A fourth means of dyeing polyester incorporates the use of low temperature subliming dyes with the concept of thermofixation or dry heat. It is referred to as the Heat Transfer printing process. In this method, the disperse dyes are printed or applied to cellulosic paper—a substrate that has no affinity for the disperse dyes. The dyes are allowed to dry on the paper and then this "transfer paper" is placed, dye side down next to polyester fabric. The combination is then heated. As the sandwich of paper, dye, and fabric warms, the dye sublimates and is absorbed into the polyester substrate. Disperse dyes suitable for this work are sold by major dye manufacturers: Bafixan (BASF), Catulia (Franken-Donders), Dispersol TP (ICI), Lurafix (BASF), Seriplas (Yorkshire Chemicals Ltd), Sublaprint (Keystone), Teraprint (Ciba-Geigy), and Transetile (American Cyanamid). Among the disperse dyes suitable for heat transfer printing are: C.I. Disperse Yellow 3, C.I. Disperse Yellow 8, C.I. Disperse Yellow 54, C.I. Disperse Orange 3, C.I. Disperse Red 9, C.I. Disperse Red 15, C.I. Disperse Red 60, C.I. Disperse Violet 1, C.I. Disperse Blue 14, and C.I. Disperse Blue 56 [9].

Clearly, if two or three disperse dyes are mixed to achieve a certain color, the size and volatility of the dyes must be

comparable or the color will not match the prediction. Also, the fastness properties may be altered. In addition, not all dyes are listed in the Color Index, and not all have known chemical constitutions (and therefore molecular weights), known vapor pressures, or known vapor concentrations. For these reasons, a careful review of a manufacturer's recommendations is urged for whatever application method is chosen.

Experimental work on the use of thermofixation—by thermosol and by transfer paper—in textile conservation was carried out in England at the University of Manchester, Institute of Science and Technology (UMIST) under the direction of Dr. L.W.C. Miles as early as 1977 [11]. There he suggested that conservators pad and heat set polyester in experimental ovens, or alternatively, consider transfer paper. Among the advantages he foresaw to conservation laboratories were: reduced in-house fabric stocks (and costs), avoidance of extensive training or equipment, and the possibility of cutting print motifs from transfer papers. He also pointed out some limitations: only polyester will have suitable lightfastness (disperse dyes color nylon with only poor fastness), subsequent pleating or ironing will be limited, and there are repeat and width problems.

Further experimentation by the authors of this paper began in 1986 when Patsy Orlofsky of the Textile Conservation Workshop pointed out that the limited shade range of sheer polyester did not provide adequate colorways for cotton patchwork quilts and other antique textiles in need of repair. Work done at UMIST in 1977 had shown that Thermosol fixation worked well, but that improvised padding with tightly woven polyester produced unlevel results. The present project was designed to determine whether the technologies of the Thermosol process and of transfer printing onto polyester could be modified to fit the needs and facilities of a textile conservation laboratory.

Experimental. Two polyester fabrics were purchased from Testfabrics, Inc. of Middlesex, New Jersey. One was a polyester batiste with a filament warp and a spun weft filling (#733); the other, a 100% Dacron polyester type 54 spun polyester challis (#767). These two fabrics are both considerably denser than a polyester tulle. This density was intentional: it is far easier to match a color from a dense weave to a loose weave, than to deal with color matching from translucent fabrics. Moreover, any unleveling or skittering of dyes is much more apparent on a denser fabric.

The fabrics were cut into 8 1/2" x 11" rectangles. In addition, white paper, used for photocopying purposes, was

obtained. The fabric was weighed for reference purposes. The dyes were then prepared. Initially, an anti-migrant, "Superclear N-100," was used. After some discussion and literature review, it was decided to eschew the "Superclear N-100" and subsequent dye work excluded this assistant and any carrier from the master recipe [3, 6]. Superclear N-100 was so viscous a liquid that it had to be measured in an aluminum cup (after tare) and the entire weighing submerged in the fixed amount of deionized water in order to dilute the Superclear and achieve solubility. The aluminum cup could then be retrieved and reused.

Spraying was done on acid free glassine paper and, eventually, on Kraft brown paper, with the samples taped at the corners. A Binks Wren air brush was attached to the compressed air supply in the textile conservation laboratory. While wet, the fabrics or papers were kept horizontal to prevent undue migration or running. Masks or respirators and latex gloves were worn at any time during preparation when the possibility of spray or vapor containing unfixed dye could be in the air.

The thermofixation was prepared by sandwiching the sprayed fabric or the fabric-dyed unit between two layers of aluminum foil. This sandwich was then placed on two or more acid free blotters, which isolated the heat of the ironing from the padded textile laboratory table. The sublimation of the dyes was achieved by using a normal, domestic iron heated to a temperature of 420°F.—the temperature setting associated with ironing cotton or linen. The temperature was measured with temperature indicator papers. The color of the disperse dye changes both in hue and in intensity during thermofixation. The color of the disperse dye—wet or dried—may not resemble the fixed color at all. This color change provides a handy identification of missed, unheated, sections. The dyed fabrics were allowed to cool and then rinsed. The fabric was then allowed to dry in air.

Discussion. Working with disperse dyes does not entirely resemble laboratory work with other classes of dyestuffs used in textile conservation. Basing work methods on laboratory standard dyeing operations, the fabric samples were weighed to four places (to tenths of milligrams) after notching for identification. Both of these procedures were found to be extraneous, except to obtain a general idea of the sample weights to establish a liquor ratio for clearing baths (see below).

All measurements of dyes and assistants should be treated by weight. The wetting agent is most easily treated this way, and the disperse dyes also. Measuring the dyestuffs by volume is not recommended even with dye pipettes (white markings on the pipettes) and graduated cylinders because of the viscosity of the commercial dye solutions. It is simpler to measure weight rather than volume.

The samples were not scoured prior to dyeing. There was some dye differential (Dispersol Red 4GPC or Dispersol Brown 3G-PC) on the verso, although the recto did not show signs of unlevel dyeing. Spraying dry fabric samples does not seem to affect the quality of the leveling per se. A soiled (oily) fabric sample, however, could compromise the dyeing quality. Similarly, there may be a problem if the substrate onto which the samples are laid cockles or buckles when the moisture from the dye solution penetrates it. The acid free glassine paper did cockle and may have been the source of some uneven dye distribution. In addition, there may be some problem if the dye solution is not well dispersed or shaken before use. The black disperse dye, Dispersol Black D-2BA, seemed to have this problem: the paper transfer technique showed evidence of unlevel dyeing.

There are, however, unexpected advantages with disperse dyes. The diluted, made up dye solutions can be stored at room temperature for up to a year without degradation, although some shaking or mixing should be done prior to spraying. Moreover, paper transfers can be stored in archival folders indefinitely. These properties will allow conservators to dye fabrics only when required, without having to set up the entire dyeing procedure each time. Paler shades can be made up by dilution and strength of spray on the transfer paper. Finally, the transfer papers themselves can be used more than once in certain circumstances.

A smooth and level distribution of the dye was achieved quite successfully with an air brush. This constitutes an improvement over a pad batch method. Alternatively an inexpensive foam brush works superbly. The quantity of dyestuff spread onto a fabric or paper surface can be controlled quite well. Shape lines or patterns can be realized by using the paper transfer techniques and cutting the appropriate shape from the paper once the disperse dye has dried. It may be possible to use silkscreen techniques to stencil patterns (i.e. a pattern repeat) to match a large antique textile in need of variegated polyester net repair. Such work would require a thickener system and this was not explored due to lack of time although viable

systems are known to exist.

The major limitation of the dyeing process involved the thermofixation of the dyes. Heat setting with an iron is a slow and tedious technique. Temperature test papers ("thermopapers") permitted an accurate measurement of the temperature achieved and assured fixation of the dyes, but did not eliminate the need for a timed fixation. The time was reduced from 90 seconds to 60 seconds in an experimental trial: the reduction in time did not appear to reduce the quality of fixation. Later experiments seemed to indicate, however, that a full 90 seconds was preferable. Still, the shape of the domestic iron does not allow for a simple repeat and overlap. Because aluminum foil is needed to isolate the iron from the subliming dyes, the processor cannot see the object in question. The shape of the iron's imprint on the foil does help to determine whether or not an area has been treated

The vacuum hot tables used by paintings conservators were considered as an attractive alternative, but the temperature range generally does not exceed 165-175°F. The tables are not designed to reach the 420°F. that the Thermosol process requires. Tacking irons were also considered but again they did not reach temperatures high enough for thermosol sublimation. It may be that heat transfer papers could be used with these tools, if the disperse dyes were carefully chosen. Another possibility involves the use of the microwave oven. Whether or not the microwaves could generate the sublimation of the disperse dyes was not explored since the only available oven of this type was restricted to use for food preparation by laboratory staff, and not meant for experimental use.

The anti-migrant, Superclear N-100 produces a distinct noxious odor perhaps in conjunction with the dispersing agent in the liquid dyes. The Superclear N-100, manufactured by Diamond Shamrock, is described in the literature as a refined natural gum. Be that as it may, the odor that occurs during the thermofixing with this ingredient in the recipe requires a respirator with organic cartridges to prevent its inhalation by staff in class proximity. By avoiding the use of the anti-migration agent, hazardous fumes were very much reduced. Not using an auxiliary other than a wetting agent, lessened odors considerably, although "elephant nose" exhaust hoses were always used during thermofixation.

Clearing baths were easily made up. There was some question as to the necessity of including sodium hydrosulfite in the clearing bath, since textile conservators rarely use this

reducing agent except at room temperature. One set of samples with Dispersol Navy F-26 was divided and half was cleared with sodium hydrosulfite, half was cleared with only the soda ash and non-ionic surfactant. A slight color difference is perceptible: the sample cleared with the sodium hydrosulfite was discontinued for the rest of the clearing baths. However, no clearing is necessary if the selection of disperse dyes is made carefully [13].

Two other changes were made to the recipe for the rinse bath. A much smaller percentage of non-ionic surfactant was used because the textile conservation laboratory does not have continuous dyeing capabilities and can only use fixed baths and then continuous cold or hot water rinsing under the tap (or with dionized water). Triton X-100, manufactured by Rohm-Haas, was substituted for the Synthrapol WN-K recommended by ICI Americas. This was done to conserve the laboratory's supply of Synthrapol WN-K. No difference was observed between samples cleared by the different non-ionic surfactants, or between samples cleared with a smaller quantity of surfactant.

After final rinsing, the samples were allowed to dry on acid free blotters. With the exception of samples of Dispersol Black D-2BA, no color transfer was observed on the blotter after drying. It may be that these samples were simply not thermofixed as well as was required; it may be that the black dye required a longer treatment of heat; it may be that it was not rinsed as well, although the rinsing was fairly consistent.

Evaluation of the colors for crocking or bleeding was not made. Lightfastness and wetfastness tests are summarized by the manufacturer. It may be that textile conservators will want to test their own samples after dyeing to assure themselves of the stability of their particular samples.

It may also be useful eventually to develop a color map of the disperse dyes using the CIE $L^*a^*b^*$ system. This would allow a textile conservator to evaluate the color on a historic textile prior to dyeing. With color map indicating the limits of the disperse dyes available (stored on transfer paper), a simple comparison of the color numbers of the antique textile to the map could determine whether or not that color was within the boundaries (possibilities) of the disperse dyes.

Finally it must be noted that the disperse dyes selected for experimental purposes in 1986 do not entirely conform to the recommendations in the literature. Dispersol Black D-2BA, as well as some of the others, did not sublime entirely at the

temperatures used. Certainly, the disperse dyes which work well for Thermosol heatsetting will not reach their proper temperatures as dyes on transfer paper. Readers are urged to explore dye selection thoroughly and carefully with the manufacturers' representatives. Alternatively, readers may wish to restrict their selection to those disperse dyes available commercially through small distributors as crayons or pre-made transfer papers. Whichever route is taken, readers should inquire about Material Safety Data Sheets (MSDS) and fastness ratings to insure a safe and suitable choice of dyes.

Conclusion. The aim of this project was to determine if the technologies of the Thermosol process and of transfer printing would fit within the confines of a textile conservation laboratory. The experimental work suggests that there is no obstacle inherent in the method to prevent successfully dyeing polyester tulle in a textile conservation laboratory. Minor peccadilloes like the shape of a domestic iron could be improved upon with further experimentation. The flexibility of the thermofixation system and stability of the dyestuff are tremendous advantages for any textile laboratory. The possibility of producing custom pattern in the tulle to match the major elements on a patterned piece of quilt patchwork was not imagined at the outset of this project, but such designing now seems reasonable.

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Supplies Required

Hot plates for rinsing finished work. (Fisher Scientific)
 Deionized water
 Temperature indicator strips for 400°F., 410°F.,
 435°F., 450°F. (Talas)
 Aluminum foil
 Aluminum weighing cups or pans; or pipettes
 Disperse dyes (see Dye Supplies)
 Superclear 100-N (ICI)
 Polyester fabric of choice, undyed
 Non-ionic surfactant (Triton X-100, Rohm-Haas)
 Thermometer for dye baths
 Hand Iron
 Glass stirring rods
 Glass beakers 250ml
 1000ml
 Weighing balance to 1/100 gram
 Air brush apparatus
 Small (60ml capacity) bottles to fit air brush mechanism
 Soda ash (aka washing soda or sodium carbonate, optional)
 Blotting paper
 Substrate: poly(ethylene) sheeting or Kraft paper*

Safety equipment:

3M Dust and Mist Respirator #08710, 3M, St. Paul,
 Minnesota.
 Dual Cartridge half-mask Respirator Facepiece, #7200,
 3M, St. Paul, Minnesota.
 Two organic vapor cartridges #7241, 3M, St. Paul, Minnesota
 Latex Gloves, prepowdered, disposable, ambidextrous,
 Fisher Scientific, #11-394-1A.
 "Elephant Noses" or Fume extractors, Nederman, Inc. 24719
 Crestview Court, Farmington Hills, MI.

*Do not use mylar film or a fiberglass-resin surface as a non-porous
 substrate: use disposable Kraft paper or poly(ethylene) sheeting.

Dye Supplies

1. Crayola Heat Transfer Crayons (8 colors) 1 box \$2.75 + shipping/handling
Cerulean Blue Ltd.
P.O. 21168
Seattle, Washington
98111-3168 tel: (206) 443-7744

2. Dye Transfer Papers 6 solid colors, each sheet 8 1/2" x 17"
\$1.30/sheet + shipping/handling
Cerulean Blue Ltd.
P.O. 21168
Seattle, Washington
98111-3168 tel: (206) 443-7744

3. Dyes with manufacturers' commercial names (and corresponding Color Index number if any), product literature, and material safety data literature sources can be found in the Buyers Guide, published annually and free to members of:
American Association of Textile Chemists and Colourists
P.O. Box 12215
Research Triangle Park
North Carolina, 27709. tel: (919) 549-8141.
The Buyers Guide costs \$66.00 to non-members; membership (including a subscription to Textile Chemist and Colorist) costs less. Write or call for application forms.

Master Recipe

Total bath: 250ml

Disperse dye total: ^{2.5g} (12.5g or 12.5 ml if 5% of total bath)
 Disperse dye _____ (Dye Name, type, C.I.# if available)

Monosodium phosphate, optional: used to insure a uniform pH for marginally prepared fabrics (i.e. below pH of 5.5)
 2.3g/l or here, 0.95 grams

Synthrapol WN-K, wetting agent 3.8g/l or here, 0.95 grams

Total ingredients: 14.4g

Total deionized water required: 235 ml.

Procedure: Measure 100ml of deionized water, add the Synthrapol WN-K (wetting agent) and monosodium phosphate (optional). Stir gently to dissolve, but do not agitate. Add half of dye. Stir gently, do not agitate (avoid formation of any foam). Add remainder of water. Stir gently. Add remainder of dye.

Note: if grains or powder are used that contain a dispersing agent, then dilute the dye with water by shaking carefully

Safety equipment: latex gloves
 dust/mist mask

1 Yellow BGG	2.50018	
2 Red D-2GA	2.50037	² 250
4 ORANGE B2R	2.50016	.05
		12.50
3 BLUE BR	2.50035	
5 Navy BT	2.50010	

0.027.

#1	0.0969
#2	0.0965
#3	0.0974
#4	0.094
#5	0.096

Procedure for Spraying

Transfer well mixed dye solution to clean air brush bottle.
Test for air pressure on scrap paper or textile. Use pressure that will provide a diffuse, even spray.
Prepare substrate sheet.
Scotch taper corner of paper or fabric on to substrate.
Spray.

Depth of shade: achieved by quantity of spray on fabric on paper surface.

Dichromatic or trichromatic shades achieved by the following:

- 1) overspraying
- 2) measured quantities of dyes sometimes mixed together and diluted as necessary.

Safety equipment: dust/mist mask
latex gloves

Procedure for Heat Setting

Pre-heat iron

Test iron temperature with temperature indicator strips, °T to reach 420°C

Sandwich fabric (and paper transfer) between sheets of aluminum foil. Use at least two layers of aluminum foil beneath the active polyester sublimation reaction to prevent scorching by the iron.

Safety equipment: respirator or "elephant nose" fume exhaust.

Procedure for Clearing, if Desired

Liquor bath: 1:75

Ingredients:

deionized water

3.5g/l soda ash

0.5g/l non-ionic surfactant

Enter goods into bath at 50°C

Gradually (10 minutes) raise temperature to 83°C-95°C

Hold for 90 seconds

Rinse thoroughly: one bath warm water
one bath cool water

Safety equipment: latex gloves
respirator or fume exhaust

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