DANIELS, MICHAEL DEAN. Factors Affecting the Behavior of Selected Disperse Dyes Under High Temperature Dyeing Conditions. (1977) Directed by: Dr. Victor S. Salvin. Pp. 70.

The major purpose of this study was to investigate the changes in dyeing behavior of selected disperse dyes resulting from variations in the conditions under which the dyes were used. The variations introduced into the dyeing system were: variations in pH; variations in dyeing times at the top temperature; and variations in the surfactant used.

The procedure used to make the dyeings was chosen because it would subject the dyes to severe conditions which would aid in differentiating among the various dyes and dyeing conditions by magnifying any potential instability of a given dyeing system. The procedure consisted of conducting dyeings of each of the fifteen dyes chosen for the study under four conditions. Each of these four conditions was further divided into dyeings held at varying times at the top temperature of 265° F (130° C).

Filter tests were conducted on blank dyebaths which were held at the top temperature without fabric for three hours for examination of particle size change.

Ratings of the depth of shade of the dyed swatches were done visually and by spectrophotometer. A change in the depth of shade was considered an indication of a change in the dyeing properties of the dye. Ratings of the filter tests were done visually only. Variations in ratings on these tests gave a good indication of the dyeing properties of the dye under the various conditions.

The results of the investigation showed that the dyeings made at pH 4.0 were the darkest in shade, and the dyeings made with the nonionic surfactant were the lightest. The dyeings made at pH 8.5 and those made with the anionic surfactant were intermediate in depth, and were approximately equal.

The filter tests did not show any significant differences in the filtering properties of the dyes among the four conditions. This was an indication that the particle size of the dyes after dyeing did not change significantly under these conditions.

The results showed that the conditions used did affect the equilibrium of the dyes in the investigation, and that these changes could be detected by spectrophotometric examination of the dyed swatches resulting from the various conditions. However, the filter tests could not be used to detect the same differences among the four dyeing conditions, even though there were large differences in the results of the filter tests among the various dyes.

FACTORS AFFECTING THE BEHAVIOR OF SELECTED

DISPERSE DYES UNDER HIGH TEMPERATURE

DYEING CONDITIONS

by

Michael Dean Daniels

A Thesis Submitted to
the Faculty of the Graduate School at
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Approved by

Victor S. Salvin

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APPROVAL PAGE

This thesis has been approved by the following
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CHAPTER I

Introduction

Disperse dyes are being used widely today in the dyeing of polyester, acetate, acrylic and nylon fabrics and yarns. As a result of this widespread use, many problems are encountered daily by those involved in attempting to dye fabrics with these disperse dyes. Some of the common problems encountered in production of fabric dyed with these dyestuffs are: dye spots on the fabric; streaky dyeings; fastness problems; and dye residue on package dyed yarns. Some of these problems have been attributed to the chemical decomposition and the physical form of the various disperse dyes used in the dyeing process.

There have been studies made on these problems, but most of these have been made on very specialized equipment or were limited to a very small specific aspect of the dyeing process. Part of the objectives of this investigation was to study dyes under general dyeing conditions which could be reproduced in any reasonably well-equipped textile plant laboratory.

Statement of the Problem

The purpose of this study was to investigate the effects of various dyeing conditions on the dyeing

properties of selected disperse dyes. The objectives of the research were to:

1. Determine the effects of pH on selected disperse dyes.

2. Determine the effects of surfactants on selected disperse dyes.

3. Determine the effects of time at top temperature (265° F) on selected disperse dyes.

It was assumed that the relative stabilities of the selected disperse dyes could be measured by the various methods employed in this study. It was further assumed that decomposition or particle size change of the dyes could be detected by changes in the depth of shade of the swatches dyed by the dye in question or by the filter tests used in this study.

This investigation is limited by the fact that the dyes used were not selected at random and any conclusions or results based on the study of these dyes cannot be extended to cover disperse dyes in general. The dyeing conditions used in this research are not all inclusive, but rather were chosen to relate to the conditions which might be encountered in the industrial-scale dyeing of fabric.

The following null hypotheses were tested:

1. That there are no significant differences in the behavior of dyes due to differences in time at the dyeing temperature.

2. That there are no differences in dye behavior as a function of pH.

3. That there are no differences in dye behavior as a function of type of surfactants.

4. That there are no differences in dye behavior as shown by variations in particle size caused by variations in pH or surfactants which are detectable by filter tests.

5. That there are no differences in dye behavior as a function of delayed introduction of fabric into the dyeing system.

Definition of Terms

The following definitions have been included for clarification of terms relating to this study.

Concentration of dyestuff on the fiber weight. Refers to the weight of dyestuff used expressed as a percentage of the weight of the fabric being dyed.

Concentration of surfactant on the fiber weight. Refers to the weight of surfactant used expressed as a percentage of the weight of the fabric being dyed.

Depth of shade. Refers to the perceived amount of darkness or lightness of the color of a fabric.

Disperse dyes. These dyes are nonionic dyestuffs which are slightly soluble in water and are used to dye acetate, nylon, acrylic or polyester fibers.

Dye auxiliaries. Any chemical which may be used to aid in the dyeing of a fiber.

Residual lubricant. Refers to the knitting or coning oil which remains in the fabric after it is scoured.

Surface dye. This is the dye which has not been absorbed into the interior of the fiber but is loosely attached to the exterior of the fiber.

Surfactant. Refers to the type of dyeing auxiliary which is used to reduce surface tension between components of the dye bath.

CHAPTER II

Review of Literature

The literature cited in this chapter gives a general background on disperse dyes, as well as reviewing recent research relating to this study. This chapter includes a short history of the development of disperse dyes, as well as information on the characteristics of the dyes, mechanism and kinetics of dyeing with these dyes, and application of these dyes on the industrial level. Recent research on the stability of these dyes is cited as a background to the present study.

Disperse Dyes, A General View

History

Disperse dyes have had a long and colorful existence. These dyes were first developed in the 1920's for use in the first hydrophobic man-made fiber, cellulose acetate. This fiber was initially very difficult to dye, due to a lack of effective dyes as well as a lack of knowledge for the mechanism of dyeing the fiber.

The problem of how to dye this fiber was finally solved almost simultaneously by four British chemists. They discovered that dyes could be formed by using sulpho-fatty acids to produce very fine aqueous dispersions of

certain insoluble azo, diphenylamine, and anthraquinone dyestuffs. The difficult-to-dye cellulose acetate could by dyed directly from baths containing these dyes.[1]

These early disperse dyes were made in the form of pastes. These pastes had the distressing tendency to settle out and dry up, which made them very difficult to use.[2] Eventually, the Germans developed a method of mixing the dye filter cake with concentrated dispersing agent, then processing this mixture through what is now known as a roller mill. This process ground the large dye particles down to a useable size in the micron range. This mixture was dried and ground further to produce a satisfactory powder dyestuff.

Before 1953, these dyes were known as "acetate dyes." In that year, it was decided that the term "disperse dyes" was more appropriate for describing this family of dyes, and this name is still in use today.

There have been many improvements in disperse dyes since those early days. Production of these dyes soared with the coming of nylon, polyester and acrylic fibers, which, along with acetate, are dyeable with disperse dyes. According to Fourness, by 1972, disperse dyes constituted over twenty-five percent of the United Kingdom's total

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[1]Robert K. Fourness, "The Development of Disperse Dyes for Acetate Fibres," Journal of the Society of Dyers and Colourists 90 (January, 1974): 15.

[2]Ibid, p. 18.

dyestuff production.[3] This percentage has since increased and is comparable to that of the remainder of the dyestuff producing countries. This is a far cry from the initailly limited use of these dyestuffs on cellulose acetate.

Characteristics of Disperse Dyes

Disperse dyes have certain characteristics which may generally be used to distinquish these dyes from other types of dyes. One of these characteristics is that of the very slight water solubilities of the various dyes. Because of this relative insolubility, these dyes are used in a stage of fine dispersion in the dyebath, instead of being in a true solution as is common with other types of dyes. The small amount of water solubility which a disperse dye does exhibit, commonly 10-40 milligrams per liter, is a very important part of the mechanism of dyeing with these dyes, a point which will be discussed further.

Disperse dyes generally have high melting points. The dyes will usually vaporize before melting, a property which enables thermosol dyeing and heat transfer printing to be successful.

Disperse dyes, particularly the blue anthraquinone dyes, have some tendency towards fume and ozone fading. According to Trotman, this is especially critical when the

[3]Ibid., p. 18.

dyes are applied to acetate fibers.[4] Disperse dyes have otherwise generally good fastness properties. Disperse dyes vary in their dyeing rate whether on acetate or polyester.

Mechanism and Kinetics of Dyeing

The accepted mechanism of dyeing with disperse dyes is that of dyeing by solid solution. Salvin stated that the dyestuff diffuses into the fiber, where it is held by hydrogen bonding, van der Waals forces, dipole interaction and physical entrapment.[5] The degree to which the dyestuff is held within the fiber determines the fastness of the dye-fiber combination. The old saying, "Easy come, easy go," is very appropriate to describe this interaction. If the dye goes into the fiber easily, it will come out of the fiber almost as easily. This is frequently demonstrated by poor washfastness of some dyes on nylon or acetate fibers which can be related to their rapid diffusion rates into the fiber.

From consideration of the mechanism of disperse dyeing it is appropriate to follow with a discussion of the kinetics of disperse dyeing. In order to do this, one must first briefly consider the structures of the fibers

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[4]E. R. Trotman, Dyeing and Chemical Technology of Textile Fibers (London: Griffin, 1964), p. 479.

[5]V. S. Salvin, "Dyeing Behavior of Disperse Dyes on Hydrophobic Fibers," American Dyestuff Reporter 49 (August 22, 1960): 603.

involved. Here, only cellulose acetate and polyester will be discussed, although nylon and acrylic fiber can also be dyed with disperse dyes.

In both the cases of cellulose acetate and polyester, the nature of the fiber dictates the dyeing conditions and the properties of the finished goods. In the case of cellulose acetate, the fiber is relatively easy to dye under comparatively mild conditions. This is because the structure of cellulose acetate fiber is not as crystalline as that of polyester fiber. Each of these fibers contain regions where the molecules are more or less randomly arranged in a parallel fashion. It is in these less oriented, so-called amorphous regions where the dyeing takes place, since very little of the solution containing the dyestuff can penetrate the more crystalline regions. Polyester contains more of these crystalline regions; therefore, more severe conditions are needed to force the solution into the fiber to dye it successfully.

The characterization of polyester as a hydrophobic fiber means that water penetrates the fiber slowly and higher energy conditions are required to accelerate the rate at which the waterborne dye enters the fiber.

In the dyeing of these hydrophobic fibers, the dye must first dissolve in the dyebath before it can penetrate the fiber. This is the point at which the small amount of water solubility of disperse dyes becomes vital.

Although these dyes are very slightly water soluble, this small amount of water solubility is necessary for attaining practical dyeing rates on either cellulose acetate or on polyester. The dye first dissolves in the water, migrates to the fiber surface, then diffuses into and throughout the fiber for the remainder of the dyeing cycle. The dissolving of the dye in the dyebath, along with the diffusion of the dye throughout the fiber are said to be the rate determining steps of dyeing, since the fiber can be dyed no faster than the dye can be made available to it in solution and no faster than the dye can diffuse into the fiber.[6] Where the dye has very limited water solubility or this solubility is reduced by formation of large crystals, the dyeing rate will be reduced.

Application of Dyes in the Textile Plant

There are many methods of applying disperse dyes to polyester, acetate, nylon or acrylic fibers. One of these is known as atmospheric dyeing, which is carried out in an aqueous system at temperatures at the boil or below. This process is often used for dyeing cellulose acetate, since this fiber dyes more readily at lower temperatures than polyester. The weaker acetate fiber also may not withstand higher temperatures.

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[6]H. U. von der Eltz, "On the Theory and Practice of Dyeing Texturized Polyester Filaments," Textilveredlung 6 (1971): 196.

Disperse dyes are usually applied to polyester at high temperatures and/or high pressures. Also, it may be dyed with the aid of a carrier at lower temperatures if necessary.[7] The normal dyeing temperatures without carrier for polyester are from 240 to 275° F (115 to 135° C) with holding times at these temperatures from as little as one minute to one hour or longer.

The machines used for this high temperature dyeing range from pressure becks to the more often used "jet" machines. The use of a jet dyeing machine, which moves the fabric by means of rapidly circulating dye liquor, can call for careful selection of dyestuffs to prevent problems in dyeing. A partially flooded jet dyeing machine is one of the most efficient foam-producing machines yet devised by modern engineering. For this reason, dyes and auxiliaries must be selected which have low foaming characteristics. Also, the high shear produced by the pumps in these machines can break down the dyestuff by stripping it of its dispersing agent. This can and often will result in agglomeration of the dye, with the result that the fabric being dyed may have dye spots. Any fabric with defects such as this must

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[7]Dye carriers are chemicals which act to swell the fiber in dyeing which makes the interior of the fiber more accessible to the dye liquor, thus allowing dyeing in shorter times or at lower temperatures. In the swelling of the fiber, the free energy forces which cause the fiber molecules to pack closely are released. The fiber molecules then have increased open areas where water containing dissolved dye can enter.

be redyed, thus costing much money and causing inconvenience to the dyer.

These fibers may also be dyed in yarn form in machines which also operate at elevated temperatures and pressures with similar long holding times at the top temperature. The use of these machines in dyeing may make the selection of dyestuffs more critical, since the pumps in some of these machines have more shearing action on the dye than the pumps on a jet dyeing machine. Also, as a result of the manner in which the yarn is loaded into the machines, the yarn package tends to act much as a filter paper. A dye which has any tendency towards instability or particle size change may filter out under these conditions, thus creating spots of dye on the yarn package. Dyes are usually tested for filtration properties before they are used on such a machine.

Many problems in addition to those discussed above may be encountered in a commercial operation where disperse dyes are being used to dye any of the fibers dyeable with these dyes. However, the problems which seem to surface most often in day-to-day use of these dyes is the tendency of these dyes to crystallize, agglomerate, filter out or to give nonreproducible shades during the dyeing of the fabric or fiber. All of these conditions may result in unmerchantable goods, therefore these problems are to be avoided by any and

all means possible. If it would be possible to predict the behavior of dyes before using them in production machines, this would be a great help to the fabric dyer because it would allow him to select dyes which would minimize his problems, thus maximizing production and profit. Much work has already been done in this area, and this study is an attempt to further this work.

Disperse Dyes--Research on Stability

Little work has been done with the specific subject covered in this study, however there has been some research on the stability of disperse dyes under various conditions.

Apperly stated that instability in the dyebath is promoted by the tendency of dyestuff particles which are very small to aggregate. This occurs due to the high free energy of their surface area due to the large surface area of the small particles. Larger dye crystals may also grow as a result of absorbing the smaller particles in the dye liquor. In addition, aggregation of the various sized particles may occur. The latter condition may be controlled somewhat with proper surface treatment of the dyesstuff particles.[8]

According to Leube and Uhrig, there are many factors which can affect the stability of dispersions of disperse

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[8]T. W. J. Apperly, "Investigation of Factors Affecting the Particle Properties of C. I. Disperse Yellow 3," Journal of the Society of Dyers and Colourists 85 (December 1969): 562.

dyes. They said that thermodynamically, bodies with a large surface area, such as very fine particles of disperse dyes, are less stable than those with a smaller surface area. Therefore, finely divided disperse dyes would tend to gradually form more coarse crystals. Crystallization is favored by high purity of the dye crystals, large differences in particle size, products that increase the solubility of the dyes, and repeated heating and cooling.[9]

Two of the other problems discussed by Leube and Uhrig are agglomeration and aggregation. They indicated that these problems are aggravated by any factor which diminishes the stabilizing effect of the dispersing agent, reduces the electrostatic repulsion between the disperse dye particles, and any factor which increases the kinetic energy of the dye. Any of these factors increase the probability that agglomeration will occur. It can be seen that the conditions encountered during the dyeing cycle would tend to promote aggregation and agglomeration since the dyeings are carried out under high temperature conditions which would increase the kinetic energy of the dye and therefore the chances for collision of the dyestuff particles.[10]

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[9]H. Leube and H. Uhrig, "Test Methods for Selection of Disperse Dyes," Canadian Textile Journal 91 (March, 1974): 5.

[10]Ibid., p. 6.

Biedermann stated that aggregation could be caused by a number of factors, including high dye concentration, salts, unsuitable dispersing agents, fast liquor flow and mechanical shear from the pump in the dye machine. He said that aggregation was most likely to occur at temperatures of 100 to 130° Celcius. Also, other problems likely to be encountered include crystal growth, where there is no change in the structure of the crystals, or crystallization on cooling. The crystallization on cooling was stated to be more likely to occur when there is a high concentration of dye and/or no fabric in the bath. Another change which might occur was said to be a change from an amorphous state of the dye to a crystalline state. He said that these crystals might be submicroscopic in size, and thus be detectable only by electron microscope.[11]

In the Palanil Handbook for disperse dyes, it is stated that high concentrations of dye increases the possiblity of agglomeration, since a large number of collisions between the particles of dyestuff. Some of thse collisions would be sufficiently energetic to result in agglomeration. This agglomeration proceeds slowly at first, then goes more rapidly with increasing time at elevated temperature.[12]

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[11]W. Biedermann, "Classification of Types of Precipitation Occurring in Disperse Dyeing," Journal of the Society of Dyers and Colourists 88 (September 1972): 331.

[12]BASF, Palanil Handbook (BASF, n.d.), p. 6.

The tendency to agglomerate is lessened somewhat, according to Kuehni and Phillips, by the use of an anionic dispersing agent, used by many dyestuff manufacturers, which gives each dye particle a positive charge. Agglomeration is slowed due to the like-charged particles being mutually repulsed.[13] Richter stated that the dispersing agent forms a protective sheath around the dyestuff molecule, which slows the agglomeration.[14]

The Palanil Handbook stated that variations in pH, within a range of 4.5 to 5.5, do not have any effect on the dispersion of disperse dyes, but that an alkaline pH should be avoided since hydrolysis of the dye might result. Lower pH values could also decrease the stability of the dispersion, since the dispersing agent is not as effective at a very low pH.[15]

Von der Eltz said that changes in pH may adversely effect the disperse dyeing process. These changes may manifest themselves in the form of changes in the yield of the dyes may be affected.[16]

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[13]Rolf G. Kuehni and R. Eugene Phillips, "Performance Characteristics of Disperse Dyes for Exhaust Dyeing of Polyester" (n.d.), p. 3.

[14]Paul Richter, "The Quality of Finish--Decisive Criterion in the Selection of Disperse Dyes for More Economical High Temperature Dyeing of Polyester Fibers,' Melliand Textilberichte (English Edition) 3 (October, 1974): 772.

[15]BASF, Palanil Handbook, p. 6.

[16]Von der Eltz, p. 197.

Pomfret stated that disperse dyes may be considered to dissolve in polyester without the dye molecules themselves being associated with ionized groups within the fiber molecule. For this reason, he says, the absorption of disperse dyes in polyester is not normally affected by the presence of acid in the bath. He does state, however, that some dyes may be affected by the presence of alkali in the bath and their substantivity thereby lessened. Certain dyes contain phenolic groups which form sodium salts which favor a shift of the solubility equilibrium point to the dyebath. Pomfret also stated that specific dyestuffs may be affected by the addition of acid, alkali, or electrolyte, but that the atypical behaviors of these dyes could be explained by the study of the chemical structures of those dyes.[17]

Murray and Mortimer discussed the use of nonionic surfactants in disperse dyeing. They pointed out that certain nonionic surfactants could increase the solubility of disperse dyes in water. This could affect the leveling properties as well as the fiber penetration and yield of the dyes. Certain of these products with low cloud points may also adversely affect the filtering properties of a dye or affect the crystallization of the dye in the

[17]R. E. Pomfret, "Some Theoretical and Practical Aspects of the Dyeing of Polyester Fibre With Disperse Dyes," Canadian Textile Journal 80 (September 27, 1963): 53.

dyebath.[18] Certain anionic surfactants also give increased solubility to dyes.

Von der Eltz said that, in a dyebath, the dye in dispersed form is in equilibrium with the dye in solution in the dyebath. When the dyebath is raised to a dyeing temperature of 250 to 265° F. (120 to 130° C.), the dye becomes more soluble in the dyebath. If the dye is not exhausted at this stage, when the bath is cooled it may tend to crystallize. When there is fiber in the bath, as in a normal dyeing, this dye may be deposited on the fiber. In an experiment such as that undertaken in the present study, where the dyebath contains no fabric, the dye will crystallize and may be filtered upon cooling of the dyebath.[19]

Von der Eltz also said that the temperature at which the dye is initially dispersed before the addition to the dyebath should be 122° F. (50° C.) or less. Temperatures above this can cause recrystallization of the dye in the bath.[20] For this reason, warm water was used in the present study for all initial dispersions.

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[18]Murray and K. Mortimer, "Dye Auxiliaries in the Application of Disperse Dyes to Man-made Fibres," Journal of the Society of Dyers and Colourists 87 (June 1971): 175.

[19]Von der Eltz, p. 197.

[20]Ibid., p. 196.

CHAPTER III

Procedure

The procedure for this study is divided into five
main parts: Description of the equipment used; selection
and preparation of the dyes and chemicals; selection and
preparation of the fabric; description of the dyeing
conditions; and rating of the results.

Equipment

The equipment for the study was selected mainly on
a basis of availability to the researcher, although it
was the type of equipment which would usually be found
in a commercial dyehouse. All of this equipment was
located in the laboratory of a commercial dyehouse, or
in the research facilities of a major dyestuff manufacturer.
A list of the equipment used and any necessary explanations
follow:

An Atlas High Temperature Laundrometer Model LHD-
HTP, with 3" x 5" high temperature containers was used for
making dyeings. This model could be programmed for rate
of rise, time at top temperature and rate of cooling.

The shades of the swatches were rated visually
under a Macbeth Executive light, under illuninant C.

Color comparisons were made with a Diano-Hardy
Recording Spectrophotometer with a Diano-Hardy color
computer and a standard teletype output.

All weighings were made on a Mettler balance, Model P160, to 0.001 gram accuracy.

The pH meter was a Beckman Model Phasar-1, digital readout meter, with a standard calomel reference electrode. The meter was standardized prior to each set of measurements to insure accuracy.

Initial cutting of swatches was done with a Maimin Rotoshere L.

400 milliliter glass beakers.

Glassine weighing paper.

Despatch Oven.

Water used was softened and filtered with a total hardness of 20 to 30 parts per million.

Dessicator.

Weighing bottles.

1000 milliliter small-mouth bottles.

Soxhlet extraction apparatus.

Standard shears for cutting fabric.

Sears Kenmore Model 600 washer and Model 600 Dryer.

Pocket calculator for statistical analysis.

Bock extractor.

Buchner funnel.

Water aspirator.

No. 2 Whatman filter paper.

Dye resisting markers.

Other assorted glassware.

Dyes

The dyes to be tested were chosen on the basis of expected performance based on personal experience and advice from colleagues in the textile industry. Some dyes were chosen which were expected to be unstable under the conditions of the study, while others were selected which were expected to be relatively stable. While the expectations of the researcher concerning the performance of these dyes were not borne out by the results of the study, the dyes selected did provide a wide range of performance levels in the tests made.

The dyes chosen for the study are given in Table 1, listed by Color Index number and also by the number used in this study, which was assigned for convenience in the statistical analysis. All of the dyes used in this study were used as received from the various dyestuff manufacturers. These dyes were all in powder form, and were standard strength products. Commercial powder disperse dyes generally contain surfactants, dispersing agents and sodium sulphate (or other diluent used for standardization of shade). It is assumed that these dyes contained approximately forty percent of actual dyestuff, since this is an average amount for a commercial disperse dye.

TABLE 1

Dyes Used in the Study

C. I. Number of Dye	Dye Number Used in Study
C. I. Disperse Red 17	1
C. I. Disperse Orange 21	2
C. I. Disperse Blue 3	3
C. I. Disperse Blue 7	4
C. I. Disperse Red 17	5
C. I. Disperse Blue 95	6
C. I. Disperse Yellow 42	7
C. I. Disperse Orange 95	8
C. I. Disperse Red 5	9
C. I. Disperse Red 30	10
C. I. Disperse Red 60	11
C. I. Disperse Yellow 3	12
C. I. Disperse Yellow 33	13
C. I. Disperse Red 159	14
C. I. Disperse Red 275	15

Stock solutions of the dyes were used for convenience and accuracy in measurements. These stock solutions were prepared as follows: A 4.000 gram quantity of the dye was weighed on the Mettler balance using a glassine weighing paper. The dye was transferred to a 400 milliliter glass beaker containing approximately 250 milliliters

of 120º F (50º C) water. Any dye particles adhering to the glassine paper were washed into the beaker by use of water from a plastic wash bottle. The dye was then stirred with a teflon coated stirring rod until no lumps of dye were evident in the beaker. The solution was then transferred to a 1000 milliliter volumetric flask, and the total volume increased to 1000 milliliters with room temperature water. The flask and contents were shaken thoroughly to insure satisfactory dispersion of the dye. The stock solution was then transferred to a 1000 milliliter small mouth bottle, from which it was dispensed for the dyeings.

Fabric

The fabric chosen for the study was 150 denier texturized polyester doubleknit. This fabric, which was chosen for the following reasons: the fabric had a very subdued pattern which allowed easy perception of small differences in shade; a sufficient quantity of the fabric was available so that the same roll of fabric could be used for the entire testing procedure; and the fabric exhibited very little or no barre, which was a further aid in comparison of shades.

The fabric was prepared by washing in a Model 600 Sears Kenmore washing machine. It was washed using no detergent, for fourteen minutes at 140º F (60º C) on the

normal cycle, followed by a double rinse cycle at 105° F
(40° C). The fabric was then tumble dried in a Model
600 Sears Kenmore electric dryer for thirty minutes.

Three solvent extractions were then made on the
fabric to determine the amount of solvent extractables
remaining on the fabric. These extractions were run on
a standard Soxhlet extraction apparatus. The solvent
used was 1-1-1-trichloroethane, and the extraction was
run at the boil for three hours.

In conducting this extraction process, three swatches
of approximately ten grams each were placed into weighing
bottles. The bottles containing the swatches were placed
into a dessicator charged with "Drierite" and allowed
to remain for one hour to allow the moisture content of
the swatches to become constant. The bottles were removed
quickly from the dessicator and the lids were placed
snugly on the bottles. The bottles containing the swatches
were then weighed on the balance to the nearest milligram,
and the weights recorded. The swatches were then removed
from the bottles and placed into the extraction apparatus.
The extraction was run the required time, then the swatches
were removed from the apparatus and placed back into the
bottles. The bottles and swatches were placed in a
Despatch oven at 300° F (150° C) for one hour to evaporate
the solvent. The bottles and swatches were removed from
the oven and again placed in the dessicator for one hour.

The bottles and swatches were then removed from the des-
sicator and weighed on the balance. The swatches were
then removed from the bottles and the empty bottles
weighed.

The percent solvent extractables were calculated
as follows: The weight of the bottle and original swatch
minus the weight of the bottle and extracted swatch equals
the weight of solvent extractables. The weight of the
bottle and original swatch minus the weight of the empty
bottle equals the weight of the original swatch. The
weight of the solvent extractables divided by the weight
of the original swatch multiplied by 100 equals the per-
cent solvent extractables.

The average solvent extractables of the three
swatches was found to be 1.99 percent.

The fabric was cut into swatches weighing ten grams
each for use in the dyeing tests. The fabric was initially
cut into swatches weighing slightly more than ten grams
each by use of the electric rotary shears. These swatches
were then placed individually on the balance, and trimmed
with shears until the ten gram weight was attained.

The swatches were marked for identification with
a yellow dye resisting marker. For example, the marking
on a swatch might read: red 60-4-4.0-45 min. This in-
dicated that that particular swatch was to be dyed with
C. I. Disperse Red 60 at pH 4.0, at the 4.0 percent on

weight of goods level, for 45 minutes. This marking
allowed easy identification of the swatches during the
evaluation procedure.

Dyeing Conditions

The dyeing conditions were chosen to approximate
those encountered in a commercial dyeing operation as
closely as possible. However, the conditions selected
here were somewhat more rigorous than those used in a
commercial dyehouse, since it was hoped that some of the
dyes would be adversely affected by these conditions,
as reported by Leube and Uhrig.[21]

All of the dyeings were carried out under the
following general conditions: One hundred milliliters
of the stock solution of the appropriate dye was added
to each dyeing machine beaker at room temperature. This
was the correct amount of the solution to attain a 4.0
percent on weight of the goods dyeing on the ten gram
swatches used. It was hoped that this concentration would
be sufficiently high to promote agglomeration as indicated
by Biedermann.[22]

The volume of the bath was adjusted to 400 milli-
liters with softened water at room temperature. The pH
was adjusted to the desired level with either a stock

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[21]Leube and Uhrig, loc. cit.

[22]Biedermann, loc. cit.

solution (5 milliliters per liter) of 56 percent acetic acid or tetrasodium pyrophosphate. The fabric was added at this point and the container securely closed. When twenty containers were filled in this manner, they were placed into the Laundrometer, which had been previously adjusted to a temperature of 100º F (38º C).

Each dyeing cycle was run at a rate of rise in temperature of eight degrees F per minute and a rate of cooling of five degrees F per minute.

A summary of the experimental factors is given in Table 2.

TABLE 2

Experimental Factors

Dyes: See Table 1 for dye names and numbers used in study.

Dyeing Conditions

        Condition 1:   4.0% owg dye, pH 4.0

        Condition 2:   4.0% owg dye, pH 8.5

        Condition 3:   4.0% owg dye, pH 5.0, 4.0% owg
                       nonionic surfactant

        condition 4:   4.0% owg dye, pH 5.0, 4.0% owg
                       anionic surfactant

Times

        Time 1:   45 minutes at top temperature (265º F
                  (130º C))

        Time 2:   3 hours at top temperature

        Time 3:   3 hours at top temperature with no fabric,
                  then 45 minutes at top temperature with fabric

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The first dye cycle was run as follows: The beakers containing the filter tests and the beakers containing the Time 2 and 3 dyeings were placed into the machine. The temperature was raised from the starting temperature of 100° F to 265° F (38° to 130° C). This top temperature was maintained for three hours. The machine was then cooled to 100° F (38° C). The beakers containing the filter tests and the beakers containing the Time 2 dyeings were removed from the machine and set aside. The swatches of fabric were added to the beakers containing the Time 3 dyeings and were placed back into the Laundrometer.

The beakers containing the Time 1 dyeings were added to the machine. The temperature of the machine was again raised from 100° F to 265° F (38° to 130° C) and the top temperature maintained for 45 minutes. The machine was again cooled to 100° F (38° C), and the remaining beakers removed from the machine.

When the beakers containing fabric were removed from the machine, the swatches were removed from the beakers and rinsed thoroughly using softened 100° F (38° C) water. The rinsing was continued until no more dye was evident in the rinse water. The swatches were then extracted in the Bock extractor for thirty seconds. The swatches were then hung on a drying rack using paper clips for holding devices and subsequently dried in the Despatch oven for two minutes at 300° F (150° C). The swatches

were removed from the rack and pressed on a steam press for thirty seconds. The swatches were then ready for further preparation.

The beakers containing dye for the filter tests were set aside briefly for the filter test, to be described later.

The first set of conditions used for dyeing was the following: Each dye was used at the 4.0 percent on the weight of goods level. The pH of each bath was adjusted to 4.0 with the stock solution of 56 percent acetic acid. The dyeing was carried out as in the general dyeing conditions above.

The second set of conditions used for dyeing was identical to the first set of conditions, except that the pH was set at 8.5 either with acetic acid or tetrasodium pyrophosphate. In most cases, a very small amount of acetic acid was required to attain this pH.

The third set of conditions used for dyeing was the following: This dyeing was to be carried out in the presence of a nonionic surfactant. This surfactant was made into a stock solution prepared by adding 16 grams of the surfactant to 300 milliters of 160° F (71° C) water in a 400 milliliter glass beaker. This was stirred until a clear solution was obtained. This solution was diluted to 100 milliliters in a volumetric flask with room temperature water.

The 100 milliliters of dye solution (equal to 4.0 percent on the weight of the goods) was added to each dye beaker. Twenty-five milliliters of the stock solution of nonionic surfactant (equal to 4.0 percent on the weight of goods) was added to each beaker, and the volume of the bath increased to 400 milliliters. The pH of the dyebath was adjusted to 5.0 with the stock solution of acetic acid, and the dye cycle was run as in the general set of conditions above.

The fourth set of conditions was conducted identically to the third set of conditions above, except that a anionic surfactant was used instead of the nonionic surfactant.

All dyeings were run in duplicate to insure accuracy.

The filter tests were run as follows: a standard 11 centimeter Buchner suction funnel was placed on a 2000 milliliter suction flask, using a standard faucet water aspirator as a source of vacuum. The filter paper used was number 2 Whatman, in a single layer.

The solutions to be filtered were allowed to cool to room temperature before filtering. The filtering was conducted as rapidly as possible. If the solution was not completely filtered at the end of five minutes, the excess solution was decanted off the funnel and the suction applied again. After filtering, the filter papers were allowed to air dry.

In order to prepare the dyed swatches for evaluation, the surface dye had to be removed from the swatches, because this surface dye would interfere with visual and spectrophotometric examination of the swatches. To accomplish this, each of the swatches was cut into two equal pieces, and each piece identified with the dye resisting marker. Half of each swatch was retained in its original form, and half treated to remove the surface dye. The half to be treated was placed into a 400 milliliter beaker containing 300 milliliters of perchloroethylene. This was agitated with a stirring rod for five minutes. The swatch was then transferred to a fresh beaker of solvent, and the agitation continued for another five minutes. This process was repeated a total of five times. After five of these repetitions, sufficient surface dye was removed from the swatch so that little if any coloration of the final beaker of solvent occurred. The swatch was then extracted in the Bock extractor for thirty seconds, then dried in the Despatch oven at 300° F (150° C) for two minutes. The swatches were then pressed on the steam press for fifteen seconds.

Rating of Results

After the swatches were cleaned of surface dye and dried properly, they were rated visually for any variations under the Macbeth light in illuminant C. This was accomplished as follows: All of the swatches dyed

with each individual dye were compared to the swatch dyed with that particular dye at Time 1, Condition 1. That swatch and the swatch to be compared to it were placed side by side in the Macbeth light, and a judgment made as to the difference, if any, between the two swatches. For example, one swatch might be noted as being darker or lighter than the standard swatch.

In the comparison of these swatches, it was very important that all the swatches be oriented exactly the same in relation to each other, since the swatches could appear quite different due to stitch differences if they were not oriented properly.

After the swatches had been evaluated visually, they were subjected to evaluation by spectrophotometer to verify the visual rating, as well as to provide a numerical rating of the appearance of the swatches for statistical analysis.

The spectrophotometric evaluation was done as follows: The swatches representing each dyeing condition were examined individually. The swatches were folded into four thicknesses, with the dye resist markings away from the portion of the swatches to be examined. The swatch was placed in the viewing port of the Diano-Hardy Spectrophotometer, with the lengthwise direction of the swatch in a verical position.

A barium sulphate reference standard was used in all the evaluations.

Although it was not to be used in this study, a graph was made of each group of three dyeings, i. e., the dyeings made with C. I. Disperse Red 60, at Times 1, 2 and 3. Thus, each group of three dyeings in a given set of conditions would be on the same graph, allowing easy comparison of the curves.

The color computer was set so that the teletypewriter gave a printout on the swatches of K/S values for the interval 380 nm to 700 nm at 10 nm intervals. The computer also printed the readings for the reflectance values of the tristimulus values for X, Y and Z. It was decided to use the Y values for statistical analysis, since this value is closely related to the depth of shade of the swatch.

The ratings of the filter tests were done visually only. The tests were rated according to the system used in the AATCC Test Method 146-1976, Dispersibility of Disperse Dyes: Filter Test.[23] The scale for this test consists of a numerical rating from one to five with five being excellent, very little residue on the filter paper, and one being the worst, with a completely clogged filter paper.

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[23]American Association of Textile Chemists and Colorists, AATCC Technical Manual (Research Triangle Park, N. C.: American Association of Textile Chemists and Colorists, 1976), p. 49.

CHAPTER IV

Discussion of Results and Data Analysis

This chapter will present the data gathered on each experimental factor in the study. A general overview of each factor will be discussed including essential data and the statistical significance of this data.

Ratings of the Dyed Swatches

The method of dyeing used in this study resulted in twelve swatches (plus duplicates) and four filter tests (plus duplicates) for each dye evaluated. For statistical analysis, the shades of the swatches were measured with a spectrophotometer and the filter tests were examined visually.

The measurements of the Y K/S values of these swatches for each dye were averaged to get an overall mean for each dye. A comparison of these means revealed varied values for the Y values (see Appendix B), which was to be expected since the dyes consisted of reds, yellows, blues and oranges which absorb light differently from each other in the Y region of the spectrum.

The means of the fifteen dyes were examined statistically and were found to differ significantly (significant at the .05 level) as visual examination of the means would indicate.

A preliminary visual examination of the twelve swatches dyed with each dye revealed that in most cases, the swatches dyed under Condition 1 were generally deeper in shade than the swatches dyed under the other conditions. This was expected since the general practice in the textile trade is to dye with these disperse dyes at a pH between 4 and 5. This practice is accepted in the trade as providing the best color yield from disperse dyes on polyester.

The results of the visual examination of the swatches were confirmed by statistical comparison of the means for the Y values for all the dyed swatches. This comparison showed that these swatches, dyed at pH 4.0 under Condition 1, were significantly darker in shade than those dyed under other conditions (significant at the .05 level).

Most of the swatches dyed at pH 5.0 using the nonionic surfactant, Condition 3, were visually rated as being lighter in shade than those dyed under the other conditions. The statistical analysis of the Y means for the Y values of these swatches confirmed that, at the .05 level, these results were statistically significant. These results confirmed the statement by Murray and Mortimer that some nonionic surfactants could increase the solubility of disperse dyes in water.[23] The lightness in

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[23]Murray and Mortimer, loc. cit.

shade of the swatches dyed under Condition 3 could by explained by the fact that the increased solubility of the dyes under this condition prevented them from exhausting onto the fiber. The dyes were being kept in solution throughout the dyeing cycle.

The swatches dyed at pH 8.5 (Condition 2), and the swatches dyed at pH 5 with anionic surfactant (Condition 4), were judged visually as being approximately equal in depth of shades. The Y values for these swatches were examined statistically and were found to be not significantly different (not significant at the .05 level) from each other. Visually, the swatches dyed under Conditions 2 and 4 were rated as being intermediate in depth of shade between those dyed under Conditions 1 and 3. This was again confirmed statistically and found to be significant at the .05 level. This slight loss in color yield for these two conditions indicates that these dyeings did not exhaust as well as those made under Condition 1, which is our optimum condition for application of these dyes.

The swatches were regrouped after the above examinations and again evaluated visually. In this comparison, the swatches were grouped by the time period in which they were dyed. This arrangement produced groups of swatches which were dyed for 45 minutes (Time 1), three hours (Time 2), and three hours without fabric, then 45

minutes with fabric (Time 3). The visual ratings indicated that the swatches dyed under Time 1 and Time 3 were roughly equal in depth of shades. The swatches dyed under Time 2 were darker in shade in general than those dyed under the other time periods. Statistical analysis of the Y values for all the swatches again confirmed that the visual examination was correct, and indicated a difference significant at the .05 level.

The probable cause for the difference in shade between the swatches dyed in Times 1, 2 and 3 is that the dyeings made at three hours at top temperature (Time 2) had sufficient time for the dyebath to come to equilibrium. That is, at this length of time more dye was transferred to the fiber from the bath than at the other time periods. The increased energy input into the dyeing system at this length of time forced the equilibrium to shift towards more dye on the fiber. At 45 minutes at the top temperature of 265° F (130° C), there was not sufficient time for the high concentration of dye to exhaust as completely as it did in three hours, thus producing lighter dyeings.

The original reason for running some of the dyeings for three hours without fabric, then 45 minutes with fabric (Time 3) was to determine if any permanent decomposition or particle size change of the dyestuff could be detected by examination of the final shades of the

swatches as compared to the swatches dyed for 45 minutes (Time 1). Since there was no significant differences between the means of the Y values for these two time periods, it can be concluded that either no change occurred in the dyestuffs during the three hours, or that if changes did occur, they could not be detected by this method.

Ratings of the Filter Tests

The four filter tests from each dye were evaluated visually to determine if any particle size change of the dyes occurred during the dye cycle used for this part of the test. This dye cycle consisted of running the dyes for three hours at the top temperature of 265° F (130° C) without fabric under each of the four conditions, a dye cycle which should promote degradation of the dye-stuff or cause a change in particle size. According to the Palanil Handbook, the combination of high temperatures, lengthly holding time at the top temperature, and high concentrations of dyestuff in the bath should promote degradation or agglomeration of the dye.[24]

Visual examination of the filter tests revealed large differences in results among the various dyes. Some left a large amount of residue on the filter paper, while others left very little residue, no matter what the condition used. There were many dyes intermediate

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[24]BASF, Palanil Handbook, loc. cit.

in amount of residue between these two extremes. When analysed statistically, the filter test results showed that some dyes were significantly better or worse than others, while some others were not significantly different at the .05 level. This was to be expected, since the range of ratings for the filter tests could have values only between 1 and 5, a very small range in which all the dyes could not differ by a significant amount.

It was expected that the results of the filter tests would differ considerably, due to the differences in chemical structures of the dyes, as well as differences in manufacturing techniques among the manufacturers of the dyes used in these tests.

Despite the large differences in ratings of the filter tests among the dyes, statistical examination of the ratings of the filter tests grouped by dyeing conditions showed that there was no significant differences at the .05 level among the four dyeing conditions. The mean of the tests run under Condition 3, using the nonionic surfactant, was slightly higher numerically than those of the other tests, which would indicate that the nonionic surfactant was retaining more of the dye in solution and not allowing the deposition of the dye on the filter paper. However, this difference was not significantly different.

Conclusions

The conclusions made in this section are discussed in relation to the null hypotheses presented in Chapter 1.

Hypothesis 1. That there are no significant differences in the behavior of dyes due to differences in time at the dyeing temperature. This hypothesis is rejected on the basis of observed differences between the 45 minute (Time 1) and the three hour (Time 2) dyeings. The shades of the swatches dyed for three hours at the top temperature of 265° F (130° C) were darker than those dyed for 45 minutes at top temperature. This difference was found to be statistically significant at the .05 level.

Hypothesis 2. That there are no differences in dye behavior as a function of pH. This hypothesis is rejected on the basis of observed differences in the shades of the swatches dyed at pH 4.0 (Condition 1) and pH 8.5 (Condition 2). The shades of the swatches dyed at pH 4.0 were darker than those dyed at pH 8.5. This difference was found to be statistically significant at the .05 level.

Hypothesis 3. That there are no differences in dye behavior as a function of surfactants. This hypothesis is rejected on the basis of observed differences between the shades of the swatches dyed at pH 5.0 with nonionic

surfactant (Condition 3) and those dyed at pH 5.0 with anionic surfactant (Condition 4). The shades of the swatches dyed at pH 5.0 with nonionic surfactant were lighter than those of the swatches dyed at pH 5.0 with anionic surfactant. This difference was found to be statistically significant at the .05 level of confidence.

Hypothesis 4. That there are no differences in dye behavior as shown by the variations in particle size caused by variations in pH or in surfactants used which are detectable by filter tests. This hypothesis was confirmed due to a lack of significant differences in the ratings of the filter tests made at pH 4.0, pH 8.5, pH 5.0 with nonionic surfactant, or pH 5.0 with anionic surfactant.

Hypothesis 5. That there are no differences in dye behavior as a function of delayed introduction of fabric into the dyeing system. This hypothesis was confirmed because there were no significant differences observed between the dyeings made for 45 minutes at top temperature (Time 1) and those dyeings made for three hours without fabric, then 45 minutes with fabric (Time 3).

CHAPTER VI

Summary

The major purpose of this study was to investi-
gate the changes in dyeing behavior of selected
disperse dyes resulting from variations in the condi-
tions under which the dyes were used. The variations
introduced into the dyeing system were: variations
in pH; variations in dyeing times; and variations
in sufactants used.

The procedure used to make the dyeings was chosen
because it would subject the dyes to severe conditions
which would aid in differentiating among the various dyes
and dyeing conditions by magnifying any potential insta-
bility of a given dyeing system. The dyeing procedure
consisted of conducting dyeings of each of fifteen dyes
under four main conditions. These four conditions were:
Condition 1, dyeings made at pH 4.0, with 4.0 percent
on the weight of the goods of dye; Condition 2, dyeings
made at pH 8.5 with 4.0 percent on the weight of the
goods of dye; Condition 3, dyeings made at pH 5.0, with
4.0 percent on the weight of the goods of dye and a non-
ionic surfactant; and Condition 4, dyeings made a pH
5.0, with 4.0 percent on the weight of goods of dye and
an anionic surfactant. Under each of these conditions,

dyeings were made with 265° F (130° C) as the top dyeing temperature. The pH of the solutions was adjusted with acetic acid in most cases.

The dyeings were held for various times at the dyeing temperature. One set of dyeings under each condition was held for 45 minutes (Time 1), another for three hours (Time 2), and the third was run for three hours without fabric, then 45 minutes after the addition of fabric (Time 3).

Filter tests were conducted on blank dyebaths which were held at 265° F (130° C) for three hours. Ratings of the filter tests were done according to AATCC Test Method 146-1976.

Ratings of the dyed swatches were done visually and by spectrophotometer. The value used for statistical analysis was the K/S value of the Y coordinate.

The results of the study showed that the dyeings made at pH 4.0 (Condition 1) were the darkest in shade, and the dyeings made with the nonionic surfactant at pH 5.0 (Condition 3) were the lightest. The dyeings made at pH 8.5 (Condition 2) and those made with the anionic surfactant at pH 5.0 (Condition 4) were intermediate in depth between these extremes, and were approximately in depth of shade.

The ratings of the filter tests did not show any significant differences among the four conditions. The

only significant differences noted were among the individual dyes. These conditions could be caused by chemical or physical differences among the dyes tested.

Conclusions

1. The filter test was an ineffective tool for use in differentiating among the effects of the various conditions upon the stability of the dyes in question.

2. Differing pH values can affect the equilibrium of the dyes examined in this study. An alkaline pH produced dyeings lighter in shade.

3. The surfactants used can affect the equilibrium of the dyes used in this study. The nonionic surfactant produced dyeings lighter in shade than the anionic surfactant.

4. Time at the top dyeing temperature can affect the depth of shade of dyeings made with the dyes in this study. Swatches dyed at the longer holding time at top temperature were darker in shade than those dyed for the shorter time.

5. The time at which the fabric is introduced into the system has no significant effect on the depth of shades of dyeings made with the dyes used in this study.

Recommendations

It is recommended that further research be done in the field covered in this study. Some specific areas to be covered are:

1. A larger selection of dyes could be examined to determine if the conditions used in this study affect other dyes as they did the dyes used in this study.

2. More conditions could be used to determine the effect of other surfactants, differing pH values, or different holding times at top temperature on the stability of the dyes.

3. A new rating scale with finer graduations could be devised for the filter test to determine if a better rating system would make the results of the filter tests more usable.

4. The filter tests could be examined for degree of crystallinity or agglomeration, instead of examining only the amount of residue on the paper.

5. The dyes could be examined for any signs of chemical decomposition under the conditions used in this investigation. This could be done by analyzing dye residue on the filter paper for any chemical changes from the original state.

6. The dyes could also be examined for changes in shade other than changes in depth, such as changes in tone or hue of the shade.

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APPENDICES

APPENDIX A

K/S VALUES FOR Y FOR EACH DYE, CONDITION AND TIME

DYE NUMBER	CONDITION NUMBER	TIME NUMBER	Y VALUE (K/S)
1	1	1	7.19
1	1	2	6.70
1	1	3	7.11
1	2	1	6.57
1	2	2	6.93
1	2	3	7.72
1	3	1	8.44
1	3	2	7.35
1	3	3	8.73
1	4	1	7.82
1	4	2	7.34
1	4	3	8.01
2	1	1	15.64
2	1	2	13.00
2	1	3	16.49
2	2	1	15.56
2	2	2	13.24
2	2	3	16.84
2	3	1	18.77
2	3	2	16.28
2	3	3	18.79

APPENDIX A-CONTINUED

DYE NUMBER	CONDITION NUMBER	TIME NUMBER	Y VALUE (K/S)
2	4	1	15.34
2	4	2	13.09
2	4	3	15.85
3	1	1	3.88
3	1	2	3.05
3	1	3	3.58
3	2	1	4.02
3	2	2	3.72
3	2	3	3.79
3	3	1	3.89
3	3	2	3.77
3	3	3	3.85
3	4	1	4.18
3	4	2	3.73
3	4	3	3.78
4	1	1	6.82
4	1	2	4.87
4	1	3	4.94
4	2	1	6.64
4	2	2	5.15
4	2	3	5.70
4	3	1	7.24
4	3	2	5.70
4	3	3	6.31

APPENDIX A-CONTINUED

DYE NUMBER	CONDITION NUMBER	TIME NUMBER	Y VALUE (K/S)
4	4	1	7.10
4	4	2	5.64
4	4	3	6.19
5	1	1	5.55
5	1	2	5.41
5	1	3	5.70
5	2	1	5.49
5	2	2	5.14
5	2	3	5.77
5	3	1	5.95
5	3	2	5.72
5	3	3	6.31
5	4	1	5.56
5	4	2	5.34
5	4	3	5.86
6	1	1	3.32
6	1	2	3.19
6	1	3	4.13
6	2	1	3.48
6	2	2	3.10
6	2	3	3.90
6	3	1	3.27
6	3	2	3.20

APPENDIX A-CONTINUED

DYE NUMBER	CONDITION NUMBER	TIME NUMBER	Y VALUE (K/S)
6	3	3	3.80
6	4	1	3.10
6	4	2	2.96
6	4	3	3.37
7	1	1	57.32
7	1	2	55.54
7	1	3	54.83
7	2	1	59.17
7	2	2	59.00
7	2	3	57.96
7	3	1	64.51
7	3	2	62.51
7	3	3	63.85
7	4	1	60.33
7	4	2	58.81
7	4	3	60.60
8	1	1	21.31
8	1	2	20.73
8	1	3	21.11
8	2	1	21.10
8	2	2	20.79
8	2	3	21.09
8	3	1	23.31
8	3	2	22.62

APPENDIX A-CONTINUED

DYE NUMBER	CONDITION NUMBER	TIME NUMBER	Y VALUE (K/S)
8	3	3	22.97
8	4	1	21.55
8	4	2	21.23
8	4	3	21.15
9	1	1	3.32
9	1	2	3.25
9	1	3	3.48
9	2	1	3.36
9	2	2	3.19
9	2	3	3.38
9	3	1	4.17
9	3	2	3.69
9	3	3	4.25
9	4	1	3.55
9	4	2	3.36
9	4	3	3.17
10	1	1	6.34
10	1	2	6.13
10	1	3	7.00
10	2	1	5.96
10	2	2	6.28
10	2	3	6.50
10	3	1	6.63
10	3	2	7.47

APPENDIX A-CONTINUED

DYE NUMBER	CONDITION NUMBER	TIME NUMBER	Y VALUE (K/S)
10	3	3	7.66
10	4	1	7.02
10	4	2	6.35
10	4	3	7.46
11	1	1	12.27
11	1	2	11.97
11	1	3	12.71
11	2	1	12.08
11	2	2	11.61
11	2	3	12.41
11	3	1	12.18
11	3	2	12.24
11	3	3	12.33
11	4	1	11.66
11	4	2	11.63
11	4	3	11.84
12	1	1	43.65
12	1	2	42.18
12	1	3	43.96
12	2	1	45.07
12	2	2	43.92
12	2	3	45.95
12	3	1	50.61

APPENDIX A-CONTINUED

DYE NUMBER	CONDITION NUMBER	TIME NUMBER	Y VALUE (K/S)
12	3	2	50.44
12	3	3	51.49
12	4	1	46.96
12	4	2	45.35
12	4	3	47.16
13	1	1	65.14
13	1	2	63.26
13	1	3	64.62
13	2	1	66.17
13	2	2	64.29
13	2	3	66.20
13	3	1	68.27
13	3	2	67.43
13	3	3	66.82
13	4	1	63.15
13	4	2	62.58
13	4	3	63.96
14	1	1	13.47
14	1	2	13.66
14	1	3	13.37
14	2	1	13.40
14	2	2	13.41
14	2	3	13.90

APPENDIX A-CONTINUED

DYE NUMBER	CONDITION NUMBER	TIME NUMBER	Y VALUE (K/S)
14	3	1	15.08
14	3	2	14.46
14	3	3	15.40
14	4	1	14.35
14	4	2	13.89
14	4	3	14.55
15	1	1	12.60
15	1	2	11.08
15	1	3	13.42
15	2	1	12.88
15	2	2	11.18
15	2	3	13.39
15	3	1	14.75
15	3	2	12.16
15	3	3	14.15
15	4	1	13.55
15	4	2	11.20
15	4	3	14.11

APPENDIX B

MEANS OF K/S VALUES OF Y OF ALL TIMES FOR DYES AND CONDITIONS

DYE	CONDITION	MEAN OF Y VALUES (K/S)
1	1	7.00
1	2	7.07
1	3	8.17
1	4	7.72
2	1	15.04
2	2	15.21
2	3	17.95
2	4	14.76
3	1	3.50
3	2	3.84
3	3	3.84
3	4	3.90
4	1	5.54
4	2	5.83
4	3	6.42
4	4	6.31
5	1	5.55
5	2	5.47
5	3	5.99
5	4	5.59
6	1	3.55

APPENDIX B-CONTINUED

DYE	CONDITION	MEAN OF Y VALUES (K/S)
6	2	3.49
6	3	3.42
6	4	3.14
7	1	55.90
7	2	58.71
7	3	63.62
7	4	59.91
8	1	21.05
8	2	20.99
8	3	22.97
8	4	21.31
9	1	3.35
9	2	3.31
9	3	4.04
9	4	3.54
10	1	6.49
10	2	6.25
10	3	7.25
10	4	6.94
11	1	12.32
11	2	12.03
11	3	12.25
11	4	11.71

APPENDIX B-CONTINUED

DYE	CONDITION	MEAN OF Y VALUES (K/S)
12	1	43.26
12	2	44.98
12	3	50.85
12	4	46.49
13	1	64.34
13	2	65.55
13	3	67.51
13	4	63.23
14	1	13.50
14	2	13.57
14	3	14.98
14	4	14.23
15	1	12.37
15	2	12.48
15	3	13.69
15	4	12.95

MEANS OF K/S VALUES OF Y OF ALL CONDITIONS FOR DYES AND TIMES

DYE	TIME	MEAN OF Y VALUE (K/S)
1	1	7.51
1	2	7.08
1	3	7.89
2	1	16.33

APPENDIX B-CONTINUED

DYE	TIME	MEAN OF Y VALUE (K/S)
2	2	13.90
2	3	16.99
3	1	3.99
3	2	3.57
3	3	3.75
4	1	6.95
4	2	5.34
4	3	5.79
5	1	5.64
5	2	5.40
5	3	5.91
6	1	3.29
6	2	3.11
6	3	3.80
7	1	58.97
7	2	59.31
7	3	59.31
8	1	21.82
8	2	21.34
8	3	21.58
9	1	3.60
9	2	3.37
9	3	3.71

APPENDIX B-CONTINUED

DYE	TIME	MEAN OF Y VALUE (K/S)
10	1	6.49
10	2	6.56
10	3	7.16
11	1	12.05
11	2	11.86
11	3	12.32
12	1	46.57
12	2	45.47
12	3	47.14
13	1	65.68
13	2	64.39
13	3	65.40
14	1	14.08
14	2	13.86
14	3	14.31
15	1	13.45
15	2	11.41
15	3	13.77

MEANS OF K/S VALUES OF Y OF ALL DYES FOR CONDITIONS AND TIMES

CONDITION	TIME	MEAN OF Y VALUE (K/S)
1	1	18.52
1	2	17.60
1	3	18.43

APPENDIX B-CONTINUED

CONDITION	TIME	MEAN OF Y VALUE (K/S)
2	1	18.73
2	2	18.06
2	3	18.96
3	1	20.47
3	2	19.67
3	3	20.45
4	1	19.01
4	2	18.17
4	3	19.17

MEANS OF K/S VALUES OF Y OF ALL DYES AND CONDITIONS FOR TIMES

TIME	MEAN OF Y VALUES (K/S)
1	19.19
2	18.37
3	19.25

MEANS OF K/S VALUES OF Y OF ALL DYES AND TIMES FOR CONDITIONS

CONDITION	MEAN OF Y VALUES (K/S)
1	18.18
2	18.58
3	20.20
4	18.78

APPENDIX B-CONTINUED

MEANS OF K/S VALUES OF Y OF ALL TIMES AND CONDITIONS FOR DYES

DYE	MEAN OF Y VALUES (K/S)
1	7.49
2	15.74
3	3.77
4	6.03
5	5.65
6	3.40
7	59.54
8	21.58
9	3.56
10	6.73
11	12.08
12	46.40
13	65.16
14	14.08
15	12.87

APPENDIX C

ANALYSIS OF VARIANCE FOR VARIABLE Y FOR K/S VALUES

Mean = 18.94

SOURCE	DEGREES OF FREEDOM	SUM OF SQUARES	MEAN SQUARE	F RATIO
Dyes	14	71930.55	5137.90	38567.00
Conditions	3	103.39	34.46	258.69
Times	2	28.65	14.33	107.57
Dyes X Conditions	42	158.11	3.76	28.26
Dyes X Times	28	30.47	1.09	8.17
Conditions X Times	6	0.67	0.11	0.84
Dyes X Times X Conditions	84	11.19	0.13	
Corrected Total	79	72263.04	403.70	

APPENDIX D

RATINGS OF FILTER TEST RESULTS FOR DYES AND CONDITIONS

DYE NUMBER	CONDITION NUMBER	RATING
1	1	1
1	2	1
1	3	1
1	4	1
2	1	4
2	2	4
2	3	3
2	4	5
3	1	4
3	2	2
3	3	1
3	4	3
4	1	1
4	2	1
4	3	1
4	4	1
5	1	1
5	2	1
5	3	1
5	4	1
6	1	4

APPENDIX D-CONTINUED

DYE NUMBER	CONDITION NUMBER	RATING
6	2	4
6	3	3
6	4	4
7	1	1
7	2	7
7	3	5
7	4	3
8	1	3
8	2	2
8	3	5
8	4	3
9	1	1
9	2	2
9	3	1
9	4	2
10	1	1
10	2	1
10	3	1
10	4	1
11	1	4
11	2	4
11	3	5
11	4	5

APPENDIX D-CONTINUED

DYE NUMBER	CONDITION NUMBER	RATING
12	1	1
12	2	2
12	3	3
12	4	2
13	1	1
13	2	1
13	3	5
13	4	1
14	1	4
14	2	4
14	3	5
14	4	4
15	1	2
15	2	2
15	3	5
15	4	4

MEANS

CONDITION	MEAN OF RATINGS OF ALL DYES
1	2.20
2	2.13
3	3.00
4	2.67

APPENDIX D-CONTINUED

DYE NUMBER	MEAN OF RATINGS FOR EACH DYE
1	1.00
2	4.00
3	2.50
4	1.00
5	1.00
6	3.75
7	2.50
8	3.25
9	1.50
10	1.00
11	4.50
12	2.00
13	2.00
14	4.25
15	3.25

APPENDIX E

ANALYSIS OF VARIANCE FOR VARIABLE RATINGS FOR FILTER TESTS

Mean = 2.50

SOURCE	DEGREES OF FREEDOM	SUM OF SQUARES	MEAN SQUARE	F RATIO
Dyes	14	90.00	6.43	6.84
Conditions	3	7.53	2.51	2.67
Dyes X Conditions	42	39.47	0.94	
Corrected Total	59	137.00	2.32	