EXAMINATION OF VARIABLES THAT

AFFECT THE DYEING OF ACETATE

IN A THREE DYE SYSTEM

by

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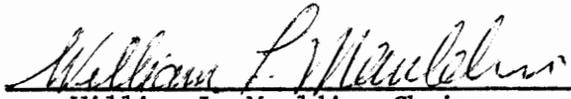
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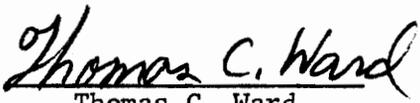
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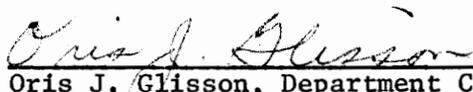
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CHAPTER I

INTRODUCTION

In a year of natural resource shortages, concern for their con-
servation, and emphasis on innovative progress for technology, there
exists a rising volume of man-made goods replacing natural products
and a pressing demand for quality and care in production of those
items that are available for consumption. This trend has been apparent
in the textile industry for several years. In 1967 United States man-
made textile fiber consumption, that includes all fibers put into pro-
duction at spinning mills, surpassed cotton consumption of 170 million
kilograms (375 million pounds) and exceeded wool consumption of 13.5
million kilograms (30 million pounds)(1).

Commercial production of the first man-made fibers, which were
derivatives of cellulose, the chemical basis for cotton, began in the
United States in 1910. The Celanese Corporation in 1924 developed a
fiber, cellulose acetate, that was relatively economical (2) but had
dyeing difficulties unlike cotton and rayon, the first fiber derived
from cellulose. Special dyes called acetate or disperse dyes were
developed and incorporated into the dyeing of the fiber to provide
colorfastness to sunlight, perspiration, air contaminants, and washing.
Improvement, refinement, and current acceptance of cellulose acetate
has resulted in many uses. These include satin, faille, crepe and
tricot fabrics for dresses, blouses and lingerie, fabric for foundation

1

garments, and cigarette filters.

Throughout the decades of this century, acetate dyeing procedures have been developed within company laboratories that are satisfactory for adaptation and use on industrial dyeing equipment (2)(3)(4)(5)(6). Textbooks have relied on these procedures for instruction and information (7), although no company guarantees reliable results (8). The American Association of Textile Chemists and Colorists[*], likewise, has no standard procedure to dye acetate or any other fiber (9). In 1973 Manay worked on a dyeing procedure for polyester knit that would give acceptable reproducibility from dyeing to dyeing, to provide industry "opportunity for exchanging and correlating dye results, both on a laboratory scale and an industrial scale"(10). The AATCC, in conjunction with several industrial participants and a state university, worked on a standard dyeing method for the same fiber; results of that study incorporated Manay's data and showed minimal and insignificant differences in color measurements from laboratory to laboratory (11). Cook conducted a similar study for nylon knit (8) in relation to a second AATCC research project in 1974 that used five laboratories to investigate variables that affected dyeing of four synthetic fibers, one of which was acetate. The dyeings of acetate "demonstrated the poorest reproducibility of any of the fibers studied"(12). The Association prescribed further work on factors that influence the color uptake of the fiber.

_____

[*]AATCC is a commonly accepted reference to the association.

Continuing the investigation of acetate dyeing, this research project implemented the following three objectives:

1. Examination of three variables within a dyeing procedure, using a three dye system, to determine their effects upon the dyeing of an acetate knit fabric:

   a) pH of the dyebath (2)(6),

   b) Maximum dyeing temperature (7)(13),

   c) Length of time at the maximum temperature (14).

2. Evaluation of final dyebath and dyed samples to determine optimum results that give acceptable color reproducibility and maximum dyebath exhaustion and that could be incorporated into a standard dyeing procedure for acetate.

3. Investigation of the nature of the test dyes as they relate to the absorption spectra and as they influence each other in spectrophotometric concentration determinations.

REVIEW OF LITERATURE

ACETATE

Acetate is derived from cellulose, an abundant natural polymer, the monomer unit of which is beta-glucose; the regenerated fiber has a high degree of polymerization. The fiber is formed by heating cellulose with acetic anhydride, initially producing triacetate; all three hydroxyl groups on the repeating unit, are acetylated. Partial hydrolysis or 2-1/2 acetylation of this primary acetate forms the secondary acetate, which is soluble in acetone (14).

triacetate (15) ————————————> cellulose acetate (16)
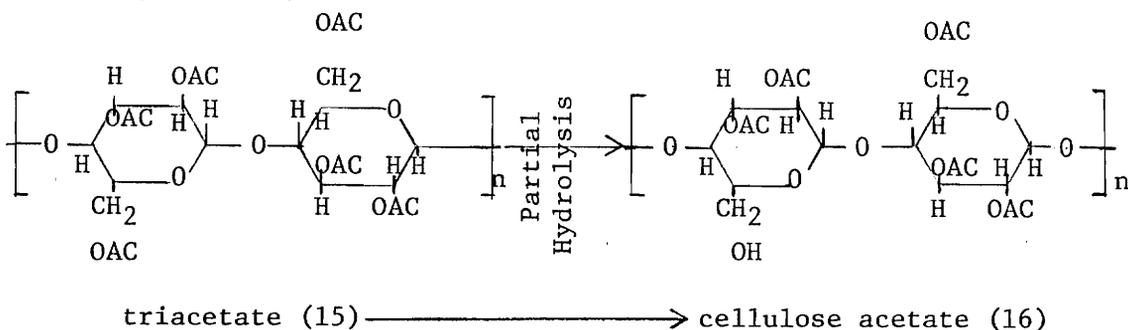
This procedure, termed regressive acetylation, allows hydrolysis of the most easily accessible groups and, in return, those sites are available for reaction with dye molecules (14). The viscous solution is then extruded through spinnerettes, drawn for molecular orientation, and spun into yarns.

Trademarked as Celanese[R], Estron[R], Acetate[R], and Seraceta[R] by its four producers in the United States, the fiber has a higher

moisture regain and is stronger than triacetate. High strength is accompanied by a high modulus of elasticity due to the chain structure orientation and morphology (16). The acetone solubility of the secondary acetate made it initially preferable to the primary acetate (14). Properties of the fiber (17) include a silk-like appearance, good dimensional stability, drapability, crisp or soft hand, and excellent resistance to moths and mildew. It has, however, poor absorbency, colorfastness, resistance to abrasion and to shrinkage, poor wash-and-wear qualities, and must be protected from products containing acetone.

The difficulty of dyeing fast shades on the fiber is connected with the chemistry of the macromolecules (14)(16).

1. There are very few free hydroxyl groups present to react with the dyes,

2. Acetate is sensitive to boiling (100°C) liquors. It is either completely delustered or luster is impaired. Temperatures must not exceed 85°C,

3. Saponification by caustic alkalies will hydrolyze the acetyl groups and regenerate cellulose,

4. The fiber swells only slightly when wet; dye liquors will not readily penetrate it,

5. The crystalline morphology obtained by drawing filament fiber lengths, provides well defined chain conformations and modes of chain packing; thus, even fewer reactive sites are easily accessible.

After initial fiber development, no existing dyes were suitable for acetate and experimentation for new dyes placed emphasis on water

solubility and insolubility. Acetate dyes, later called disperse dyes, colored the fiber in aqueous disperions of water insoluble dyes, held in solution by surface active wetting agents that also swelled the fiber (15). The dye mechanism depended on the degree of solubility of disperse particles in the fiber (14).

THE DYEING THEORY

The dyeing process is defined by Rys and Zollinger (18) as the distribution of a dye between at least two phases, dyebath and fiber, that can be described by dyeing statics, the sorption and desorption processes. There is no exact explanation for dyeing but characteristics can be based on simple models. Their equilibrium theory explains that dyestuffs form monomolecular layers on the acetate (substrate) surface, although the amount of dye used exceeds the value for monomolecular layer. There also is no interaction between dye molecules and no competition for adsorption sites. Only in rare cases, however, is there equal accessibility of sites; the energy varies at each position. The hydrophobic portion of the dye molecule is soluble in acetate while the hydrophilic portion does not penetrate the fiber. The more soluble the dye is in water, the smaller its partition ratio between water and acetate and the less affinity it has for acetate. Mullin (7) reports that physically solubilized dyestuffs are practically completely exhausted or removed from dyebath, while equilibrium means partitioning and non-complete exhaustion. The degree of solubility is affected by the competition between the two phases and the partitioning ratio. The size of the dye particle is important only to the rate of diffusion

into the fiber (7). As temperature increases, dye agglomerates and the shade deepens until equilibrium is reached. In combinations individual dyes exhaust nearly independently of each other; there is no general influence of one dyestuff on the exhaustion of another (19).

The exact position of dye within the polymer fiber has not been established with certainty; the dye must diffuse through a network of polymer chains in the amorphous area and over and around surfaces of the crystalline areas, although these regions are not penetrated by the dye. Peters and Ingamells (20) made a comparison between the 'Pore Theory' and the 'Free Volume' explanation as applied to the diffusion of dyes. The pore model represents a mechanical network of channels or pores, containing water, through which the dye diffuses. The theory is supported by the fact that there are different rates of uptake for different size dye molecules (21). Coming from thermal motion of atoms, the 'free volume' increases with rising temperature; this theory is based on the glass transition temperature of the fiber, at which a marked change of physical properties occur, allowing easier movement of polymer chains to speed dye movement.

Disperse dyes are successful in coloring cellulose acetate despite difficulty within the fiber.

> The rapid development that has taken place in cellulose acetate dyes has resulted from the observation that both water-soluble and water-insoluble dyes are applicable to cellulose acetate, provided the dyes can be offered as aqueous dispersions and solubility in water is produced without loss of solubility in (or affinity for) the cellulose ester (acetate)(22).

If fiber-soluble and water-insoluble dyes are fast to washing (14), they may be of several chemical structures. Anthraquinone dyes are derivatives of anthracene (23) and are truly water insoluble (14). Azo dyes

are insoluble dyes prepared on the fiber "in situ"; the color and
properties depend on intermediate compounds and final compound linkages
(22). Additives to disperse dyeings are wetting agents that can assist
dyeing in several ways:

1. Coupling with dye to form a compound which is more soluble
   in or has more affinity for acetate than the dyestuff
   alone (7),

2. Swelling the fiber so it can absorb the dye more readily (22),

3. Lowering the glass transition temperature (20).

LABORATORY DYEING PROCEDURE

Important to dye development evaluation and procedural develop-
ment, the dyehouse laboratory has several functions (24):

1. To produce satisfactory recipes and procedures to yield
   accurate bulk and production,

2. To maximize economy and customer satisfaction with maximum
   reproducibility,

3. To give satisfactory dyeings in the shortest length of time,

4. To test and control individual functions or variables.

Methods and procedures chosen follow applicability to mass production.
The exhaust procedure for dyeing cellulose acetate is defined as ex-
traction of dyestuff from dyebath into the substrate, the fibers or
the fabric. In dispersion the solid dyestuff impregnates the fabric
or yarn (14). This exhaustion method is used industrially for piece
dyeing on becks, for package dyeing of wound yarns, or for piece dyeing
on beam machines (2).

There are numerous variables for consideration in an acetate

dyeing procedure, including liquor ratios, pre- and post-treatment methods, pH of the dyebath, dyeing time and temperatures, equipment used for dyeing, methods of evaluation, and dye selection and combination. Soft water is essential for successful application for some dyes:

> . . .colloidal solutions are inclined to be effected to a considerable extent by what we are sometimes inclined to consider minor factors (7).

The amounts and types of assistants used determine mobility and solubility of the dye in the acetate fiber. Mullin (7) reports time and temperature are important due to the fact that when acetate is boiled for a long period of time, the dye migrates to the surface of the fiber in well defined crystals, which can be removed by washing and pressing. Very close temperature control is important in matching shades of disperse dyes on acetate, especially compound shades and, therefore, an indication of individual dye sensitivity is necessary.

> Each dyestuff apparently has a definite temperature at which it exhibits a maximum reactivity for, or solubility in the fiber (7).

Dye combination is also affected by individual striking rates and temperatures (25). In caution, acetate fibers are saponified by strong alkalies and cannot be exposed to conditions above pH of 9.5 (6). Pre- and post-scour are important as they remove dirt, grease, and excess dyestuff. The AATCC experimental procedure lists the following test variables and considerations:

1. Fabric type and weight,

2. Liquor or bath ratio of fabric weight to total liquid weight,

3. Dyebath: amount of dye assistant, pH, water, and percent
   dye on weight of fabric (owf),

4. Temperatures: beginning and maximum dyeing,

5. Rate of temperature increase and decrease: degrees per
   minute,

6. Time at the maximum temperature,

7. Weight loss.

METHODS FOR EVALUATION

Color is an important characteristic of a textile item and deter-
mines its acceptability and pleasing quality. In development of a
laboratory dyeing procedure, a chief aim is to obtain a color that
can be reproduced with minimal color differences (26). Two ways for
analyzing dyeing results are (1) determination of dyebath exhaustion,
using a transmission spectrophotometer and (2) measurement of color
values of dyed samples on a reflectance spectrophotometer (10).

Transmission Spectrophotometry

The Spec 20[R] measures light transmittance through the dye solution
as a function of a selected wavelength (10) which is determined for
each dye as the point of maximum absorption and minimum transmission.
This is where the greatest sensitivity and photometric accuracy is
obtainable (27). The absorption maximum is obtained by plotting trans-
mittance or absorbance versus wavelength; the wavelength desired is
the peak of the resulting graph (28). A general trend categorizes
absorption maxima of yellow dyes in the 400 nanometers range, red dyes
about 500 nanometers, and blue dyes in the 600 nanometers area (29)(26).

Sample concentrations that give transmittance readings between 20% and 80% are used to derive a calibration curve of concentration versus transmittance or absorbance (27); thus dye concentrations may be determined from transmittance readings. Concentration is related to exhaustion as 100% - (final concentration/initial concentration) equals the percent exhaustion.

The Spec 20 has a monochromatic light source. Although the absorption behavior of many substances are not adequately described by it, the Beer-Lambert law states that when a ray of monochromatic light enters an absorbing medium, e.g., the dyebath, its intensity decreases exponentially with an increase in medium thickness traversed and decreases exponentially as concentration of absorbing material increases (13). Problems that negate this description include interaction between dyes, to be considered later, additives, hydrogen ion activity, hydrolysis, interfering ions, and ionic strength (30).

In a dye mixture, wavelengths selected to correspond with peak absorptions for each single dye and concentrations are individually determined (28). Interference may occur as single transmission readings are affected by all of the single dyes in the dyebath. At the red dye wavelength, for example, transmission may be a result of red, blue, and yellow dyes. This interference may or may not be linearly related to transmission concentration determinations. An analytical method is available to resolve actual concentrations of a dye at its wavelength, subtracting the influence of other dyes present (31).

Color-Color Difference Measurements

The Color Eye[R] Spectrophotometer measures light reflected from a solid medium. The original energy source contains the entire visible spectrum of light. The textile fabric has the ability to alter this spectral distribution, portions of which are reflected as color, luster, and fluorescence, and absorbed, disappearing into the fabric (26). The instrument performing this function is a reflectance spectrophotometer, which will in textile applications, measure the amount of dye on the fabric.

The Color Eye is based on the CIE (Commission International de l'Esclairage or International Commission on Light) X Y Z System. The three imaginary primary lights, X, Y, and Z, are a measure of the red, green, and blue components, respectively (32); Y is also a measure of lightness. The instrument equates the color received from the fabric to the tristimulus and reports values in terms of the standards. The light source is adjusted to daylight conditions. Other reflectance spectrophotometers employ their own standards to evaluate color. The Hunter Color Difference Meter[R] values, L, a, and b, are an example. Coates, et al. found that

> . . .different types of instruments vary in their abilities
> to measure high precision according to the conditions of use
> and the nature of the specimens measured (33).

Each instrument has unique requirements and procedures that must be met.

Color differences, determined by evaluation of textile samples in comparison to the X Y Z Standards are important in determining how well two fabrics match (26). Although the AATCC does not recommend

instrumental evaluation of color differences, one reason being that

a criterion of a good test is simplicity, many textile laboratories

are successfully using it for color difference measurements (26).

> In general, it is of course, very difficult to exactly
> match a color. . .there is a need for a tolerance, and in color
> matching the magnitude of this tolerance depends on many
> things (32).

Setting tolerance depends on what colors one perceives, and what

adjacent colors he will accept and, furthermore, the end use of the

fabric, the customer, and how critical he is as far as color match-

ing is concerned. The state of the market, its supplies and demands

also play an important role.

CHAPTER III

PROCEDURE

Three variables within the AATCC experimental dyeing procedure for acetate (12) were examined. All other conditions were held constant, including a pre-scour method established by the researcher. All combinations of the variables were carried out for evaluation.

PRE-SCOUR METHOD

Pre-scour using the Nadjort Washer[R]:
  600 grams of acetate knit (102 grams/square meter)
   90 grams of AATCC detergent (approximately 15% owf)
Wash 10 minutes at (140°F) 60°C
Rinse 10 minutes at (120°F) 49°C
Repeat the wash and rinse
Centrifuge
Air dry

Fabric was then cut into 5 gram samples for dyeing, weighed on a direct reading balance. All test fabric was identical, pre-scoured in two washings.

AATCC EXPERIMENTAL PROCEDURE

1. Dyes: (equal to assistants listed later in this section)
        Three dyes chosen by the AATCC Research Committee
        were used.[*]

    a. Palanil Pink RF[R] is a Disperse Red 4 dye, color index
        number 60755 (34). The anthraquinone compound pro-
        duces a bright pink hue on acetate. The dye is

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[*]The researcher's use of trademarked dyes, chemicals or equipment does not imply that they are the only effective ones, excluding all other possible choices.

synthesized completely before application to the fiber.

b. Eastman Blue GBN[R] is a Disperse Blue 3 dye, color index
    number 61505 (34). The anthraquinone structure dyes
    acetate a bright blue. It is soluble in acetone,
    alcohol, benzene and cellosolve.

c. Intrasperse Yellow GBA[R] is a Disperse Yellow 3 class
    dye, color index number 11855 (34). The Intrasperse
    colorant is chemically an azo dye; it joins with a
    coupling agent to produce a colored compound on the
    fiber (22). It is soluble in ethanol, acetone,
    and benzene.

Stock solutions of each dye were prepared volumetrically: 1 gram

of each compound was weighed on an analytical balance and then trans-

ferred to 100 ml volumetric flask (hereafter 1%) and brought to

volume with distilled water.

Red - 1% solution or $7.8 \times 10^{-7}M$

Blue - 1% solution or $6.6 \times 10^{-7}M$

Yellow - 1% solution or $6.8 \times 10^{-7}M$

Assistants: Igepon T-51[R] is described by AATCC Products
1974 (35) as a biodegradable, general purpose, anionic
detergent and wetting agent: an assistant in bleach-
ing, dyeing, wetting, and finishing. Added in very
small amounts, it wets out the surface of the acetate
fibers to increase dye receptivity.

2. Dyebath Preparation: Dye(s) and wetting agent were combined
    in beakers, made up to volume, and the pH was adjusted.

    Each dyestuff was added to equal .2% on weight of
    fabric. 2 ml of 1% solution.

    1% Igepon T-51 was added: 10 ml of 1 gram/100 ml
    solution

    Bath Ratio--20:1 or 20 grams of total liquid per 1
    gram of fabric: 100 ml dyebath for 5 gram sample.

    pH of dyebath was adjusted to 6.0 with monosodium
    phosphate.

3. **Dyeing Procedure:** The laboratory dyeing apparatus used was the Atlas Laundry-O-meter[R] that permitted use of a maximum of 12 AATCC II A Wash Test sized cups. Each container was sealed with a Ball[R] jar lid, in addition to the neoprene gasket and metal lid. The procedure was as follows:

Add pH adjusted dyebath to wash cups and place on heating table with neoprene gaskets and metal lids.

Step laundry-O-meter to (100°F) 38°C.

Add fabric sample and seal with Ball lid, neoprene gasket, and metal lid.

Weigh cups on direct reading balance.

Place wash cups in constant temperature rotary drum and step to (200°F) 82°C, approximately (20°F) .7°C every 10 minutes.

Run at (200°F) 82°C for 45 minutes.

Cool to (140°F) 60°C, approximately (30°F) 1°C every 10 minutes.

Reweigh cup to determine weight loss or gain.

4. **Post Treatment:**

Rinse dyed samples in clean laundry-O-meter cup, using distilled water at approximate pH of 6. Dip fabric into cup 10 times.

Air dry samples on flat, clean paper towels.

EXAMINATION OF VARIABLES

Preliminary dyeings met the AATCC procedural requirements. All variables--temperature, time, pre- and post-treatment, pH, and dyeing assistant were constant. The purpose of this phase was to investigate individual dye exhaustion and measurement of that exhaustion for each of the three single dyes in the mixture. There were, therefore, four dyeings and each contained twelve individual wash cups, three with red dye--.2% owf, three with blue dye--.2% owf, three with yellow dye--.2% owf,

and three cups with a mixture of all three dyes in equal amounts,
each at .2% owf. The researcher wanted to investigate and compare
the rate and degree of color uptake on acetate, and to perfect her
skill in instrument operation, analytical preparation of the dyebath,
dyeing, and evaluation.

Experimental dyeings involved actual manipulation of only
three variables within the three dye system to evaluate their impor-
tance to and effect upon the dyeing of acetate. All other conditions
are identical to the AATCC procedure. Some factors under consideration
in relation to the variables included:

1. Temperature – the degree of temperature needed to produce
    and reproduce colored samples. Perhaps results obtained
    at (200°F) 82°C could be improved or equaled at lower
    temperatures, thus reducing cost, time, and energy for
    dyeing.

2. Time – the number of minutes at the maximum dyeing tempera-
    ture needed for acceptable results. Although time for
    temperature rise (number of degrees per minute) was not
    investigated, dyeing occurs all along the temperature
    scale. Elimination of dead time reduces cost and con-
    serves energy.

3. pH – the measure of acidity or basicity of the dyebath.
    Time consuming pH adjustment may not be necessary for
    reproducible color. Determination of pH range or exact
    value necessary, taking into consideration the sensi-
    tivities of acetate, would determine if pH accuracy is
    needed.

The six dyeings involved all possible combinations of the three
variables and they were drawn by random number to determine ordering--
a, b, c, d, e, f. The set of six dyeings was repeated three times
and final data included three replications of combinations of condi-
tions within each dyeing.

TABLE 3:1

Experimental Design for Combination of

Three Variables under Investigation

Dyeing Number	pH	Time at high temperature	High temperature	Number of wash cups	Total wash cups
1	4[a] 6[b] 8[c]	30 minutes	(150°F) 66°C	3 3 3	9
2	4 6 8	45 minutes	(150°F) 66°C	3 3 3	9
3	4 6 8	30 minutes	(175°F) 79°C	3 3 3	9
4	4 6 8	45 minutes	(175°F) 79°C	3 3 3	9
5	4 6 8	30 minutes	(200°F) 82°C	3 3 3	9
6	4 6 8	45 minutes	(200°F) 82°C	3 3 3	9

[a]adjusted with acetic acid.
[b]adjusted with monosodium phosphate.
[c]adjusted with ammonium hydroxide.

EVALUATION

Dyebath Exhaustion

After the wavelengths of maximum absorption for each of the dyes

used were determined (Yellow--370nm, Blue--525nm, Red--640nm)(see Graph

3.1), curves showing concentration versus transmission were prepared
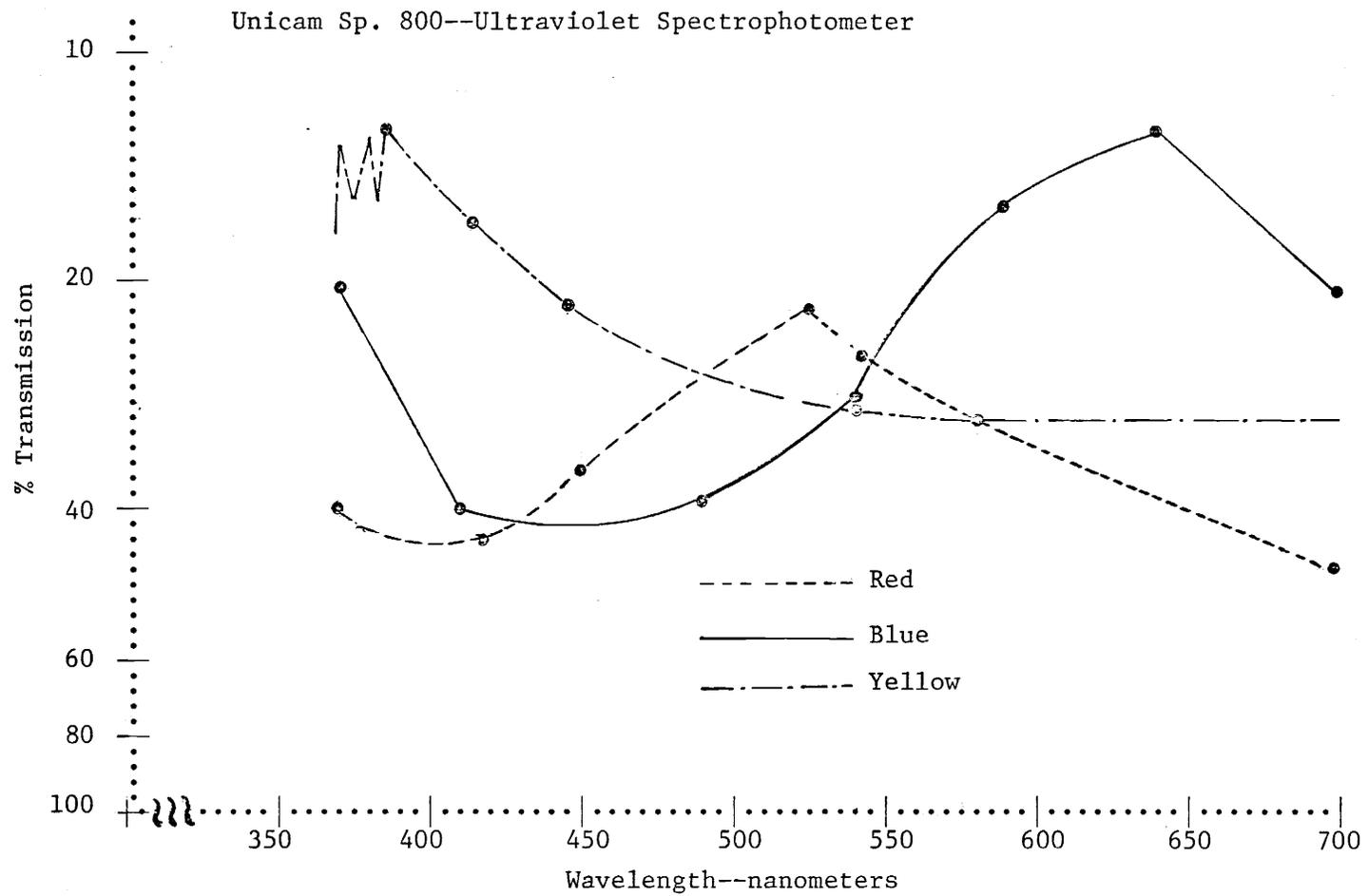
Graph 3.1.   Absorption Maxima for Red, Blue, and Yellow Dyes

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for each of the three dyes as described in Chapter II, Methods of
Evaluation (see Graphs 3.2, 3.3, and 3.4). The Spec 20 was zeroed
and calibrated to give 100% transmittance with distilled water. Con-
centration was expressed as "weight percent--grams of dry dye/grams
of total liquid" in the dyebath. One hundred weight percent equaled
0% exhaustion and all of the original dye present and zero weight per-
cent equaled 100% exhaustion with none of the original dye present.
The Beer-Lambert curve, plotting absorbance, -log transmission/100,
versus concentration in molarity produced a straight line that began
to show curvature at low transmission and high concentration. For
single dyes, therefore, concentration can be determined by trans-
mission readings.

Standard curves were also drawn for a mixture of the three test
dyes. A dyebath containing equal volumes of each of the 1% stock dye
solutions was prepared. Concentrations of each dye in the mixture
dilutions were equal to the concentration of each in the corresponding
single dyebath solutions. Transmission readings were then taken at
each of the three wavelengths. The resulting curves did not cor-
respond with the single dye graphs. Dyes in the mixture interfered
with individual readings and, therefore, concentrations could not be
determined in this manner. Correction calculations were made as fol-
lows to determine actual dye concentrations within each wash cup.
Assumptions were that absorptions of each of the three dyes add
linearly and could thus be separated. Simultaneous equations solved
for each dyebath are as follows (31):

$$-\log (I/I_o)_R = (al)_{RR} \, C_R + (al)_{RB} \, C_B + (al)_{RY} \, C_Y$$

$$-\log (I/I_o)_Y = (al)_{YR} \, C_R + (al)_{YB} \, C_B + (al)_{YY} \, C_Y$$
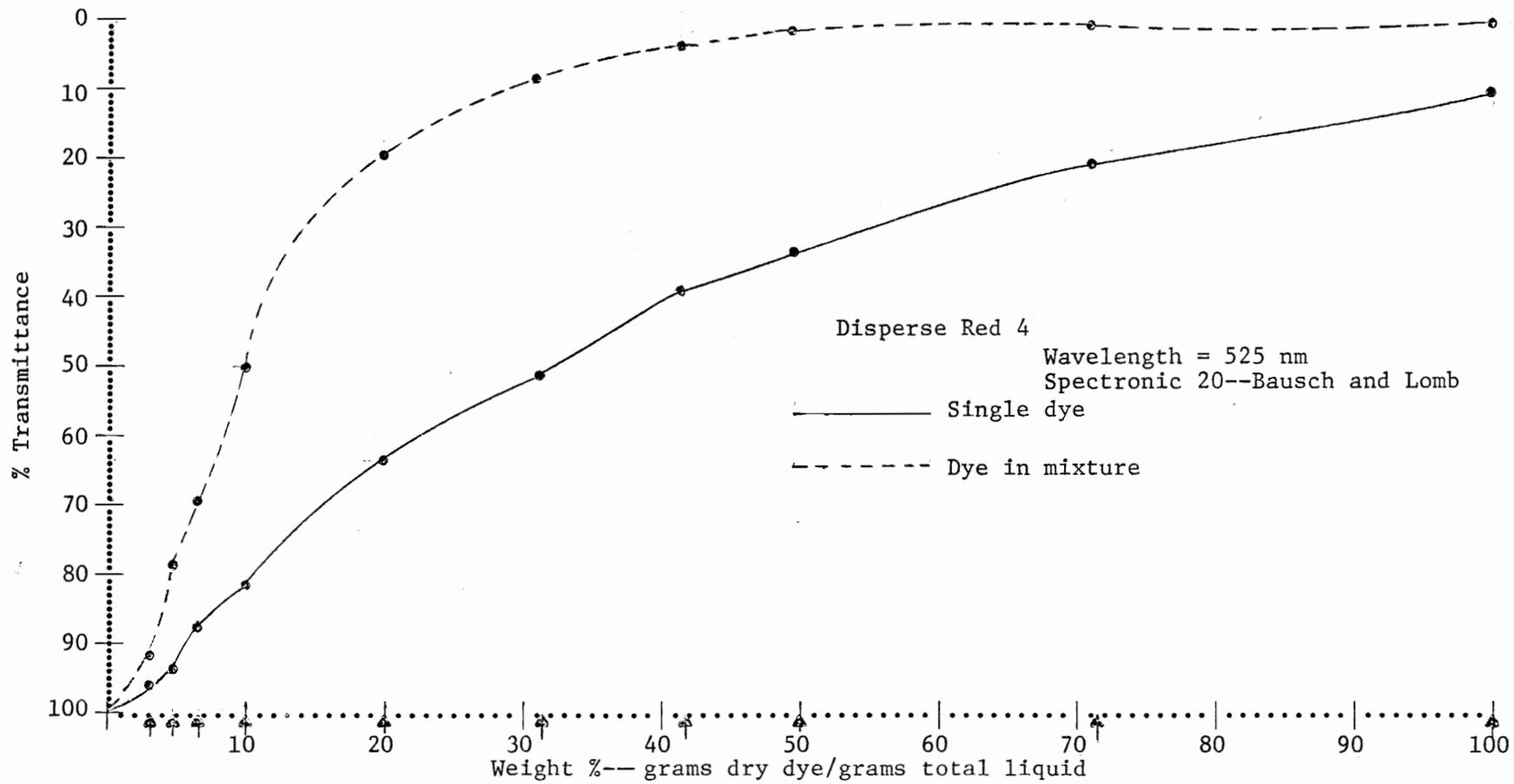
Graph 3.2.   Transmission vs. Weight percent—Red
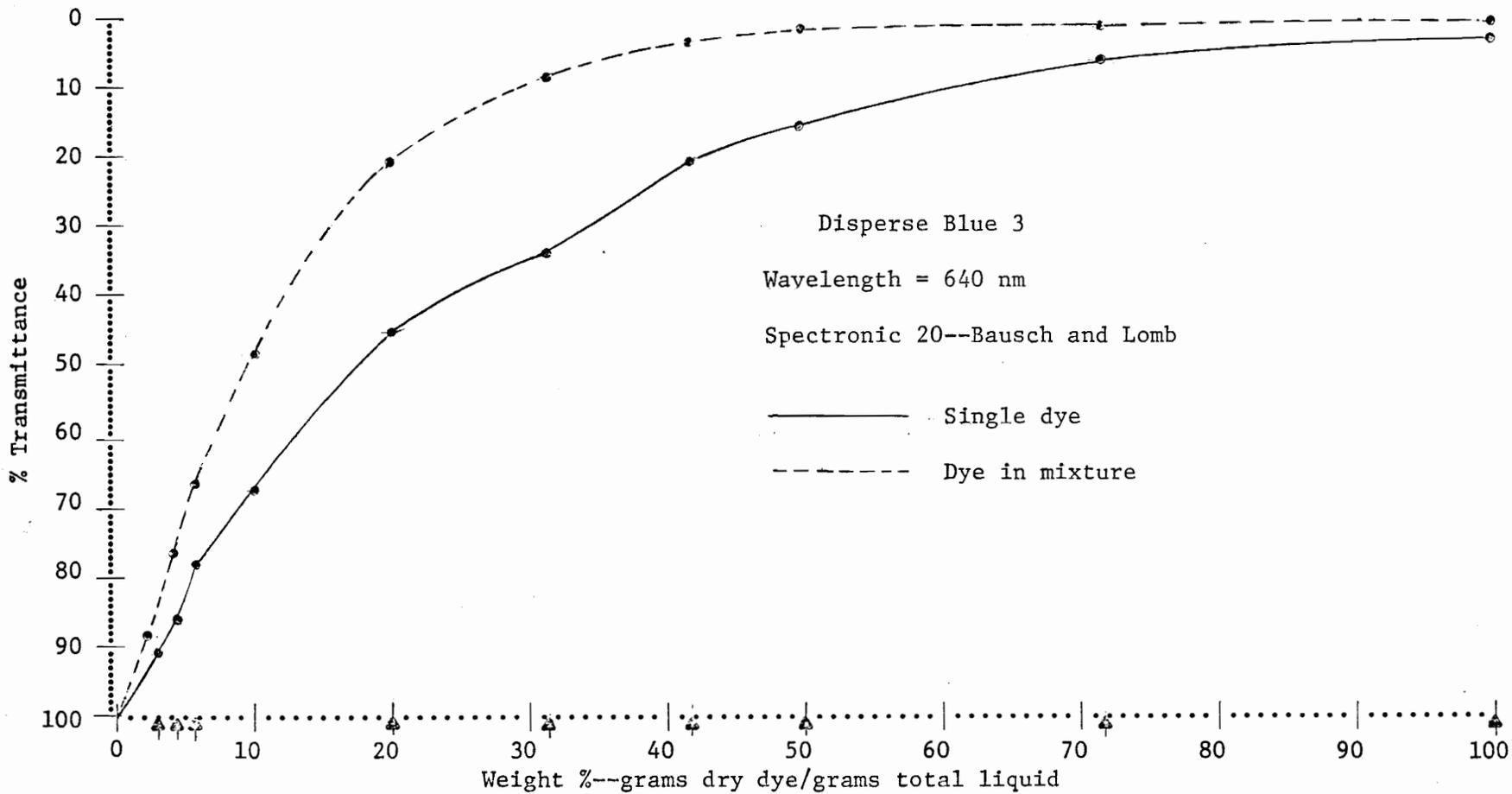
Graph 3.3.   Transmission vs. Weight percent—Blue
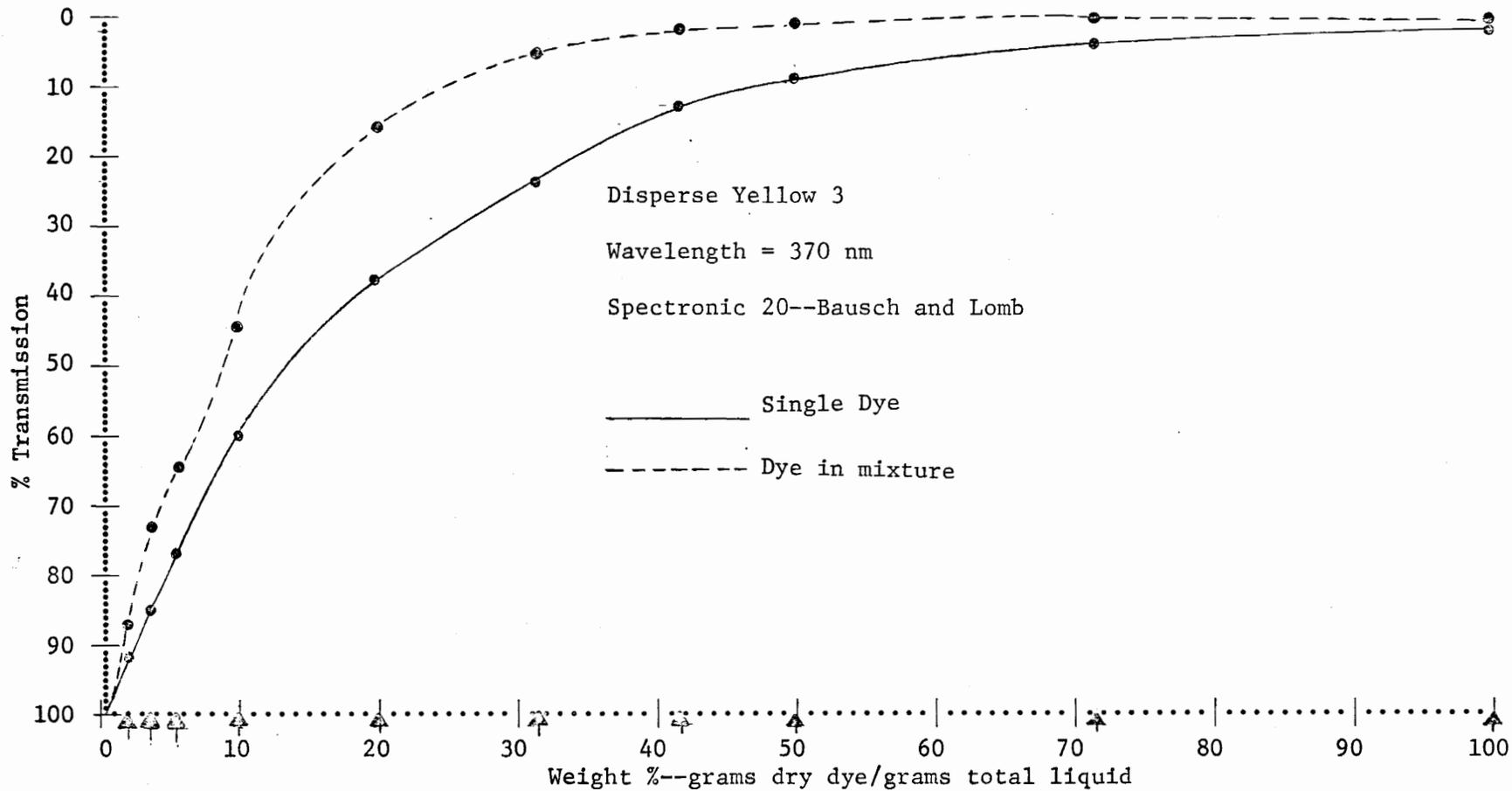
Graph 3.4.   Transmission vs. Weight percent—Yellow

$$-\log (I/I_0)_B = (a1)_{BR} C_R + (a1)_{BB} C_B + (a1)_{BY} C_Y$$

I/I_0 = % Transmission/100
(a1) = constant determined by the researcher from serial dilutions of single dyes, includes (1) length of cell containing sample and (2) a constant for the particular dye compound.

    Subscript 1--indicates dye wavelength used
    Subscript 2--indicates dye sample used to obtain that transmission
  Nine constants for 3 dyes and 3 wavelengths
C_R, C_B, C_Y = unknown concentrations of the 3 dyes:
    R = red
    B = blue
    Y = yellow

Color Measurement

Dyed fabric folded in four layers was placed one sample at a time in the Color Eye with warp in vertical direction; one reading was taken from each sample under daylight illumination. Corrections were made in the data to account for variance within the instrument as indicated by the Instrument Development Laboratories (36).

PROCEDURE FOR DATA ANALYSIS

Data was collected and grouped according to each set of conditions determined by combination of effects or variables. Evaluation information included transmission readings for each of the three dyes within each dye cup, and X, Y, Z Color Values from each sample. Data was processed by Analysis of Variance and means of the significant effects were subjected to the New Multiple Range Test. Interpretation resulted from significant differences in data. Concentration determination and evaluation of use of transmission were calculated as mean and standard deviation for each of the three dye concentrations in each of the eighteen sets of dyeing conditions.

CHAPTER IV

DISCUSSION AND INTERPRETATION OF RESULTS

Coloration of the acetate test fabric assumed penetration of dye particles throughout the fiber rather than ring dyeing or coating of just the fiber surface. Observation of fiber cross sections from each set of dye conditions determined by time, temperature, and pH, verified complete penetration of the three dyes into the fiber, formed by packing of the macromolecules. All results and data are, therefore, based on this factor.

OBSERVATIONS DURING DATA COLLECTION

A major area of concern for the researcher was leakage and subsequent loss or gain of total weight for each wash cup in which the dyeing was carried out. Manay (10) had found that leakage was totally prevented with the Ball lids as an additional sealant for the AATCC IIA Wash Test cups. Cook (8) observed that with the identical seal combination, some containers had dyebath to leak out while others had water from the LaundryOmeter tank to leak into the containers. She further concluded that some reused lids did not leak. After leakages of five to fifteen grams in preliminary dye experiments, the researcher carried out several runs of the LaundryOmeter with and without the Ball lids on the wash cups, each of which contained one hundred grams of water only. Weight changes were decreased to less than one gram with

the lids, which were used only one time. These differences were judged as actual weight change when above a one gram level, attributing some change to accumulation of drops of water (1 drop equals 1/20 gram) within the lid and clamp. The fabric holds the liquid in the cup proportional to the liquor ratio, the total liquid weight to the fabric weight.

There were only two leaks throughout the dyeings of this research and effects of them could not be detected visually or through color measurement. Cook (8) reported similar findings. Examination of residual dyebath, however, found high concentrations of colorant remaining. Dye concentration may have been large enough so that small leakages, the exact amount of which would have to be determined, did not affect the fabric color, although loss or gain could be detected in measurement of final dyebath concentration. The researcher further concluded that leakage may have occurred after dyeing was complete, or nearly complete. On the other hand, the neoprene gaskets enlarged with increasing temperature and if snugly fit to the metal lids, they could only allow transfer of liquid at lower temperatures. Yet, in this study, no weight changes affected the dye results.

In the preliminary dyeings, evaluation of final concentrations of each dye showed that the acetate fiber's uptake of each dye (red, yellow, and blue) was different. Table 4.1 reports the mean values of the final concentrations of each single dye, expressed as a percentage of the original concentration, individually obtained from Graphs 3.2, 3.3, and 3.4. Red exhausts to the highest percentage and blue and yellow exhaust about equally. Exhaustion refers to the amount

of dye that has left the bath and dispersed into the fiber. As a result color obtained by a mixture of dyes is affected by the varying individual degrees of uptake, although the dyes themselves do not influence each dyes' absorption into the fiber (19).

CONCENTRATION CALCULATIONS

Table 4.1 illustrates that the numerical ordering of transmission averages for the three dyes does not correspond with the ordering of weight percent dye remaining in the bath. A dyebath was made with equal volumes of each dye from the stock solutions. The molar concentrations of these 1% solutions vary as the gram molecular weights of each dye differ.

The dyebath was diluted to several concentrations to determine relationships between transmission, molar concentrations, and weight percent dye. Table 4.2 reports representative data from one dilution selected from the most accurate range of the Spec 20 (27). Several observations can be made from this information.

1. Concentrations calculated for each of the three test dyes from the three transmission readings do not equal known concentrations of those dyes in the same test solutions.

2. At equal exhaustion percentage levels for each of the three dyes in the test mixture, transmission readings do not increase or decrease with known concentrations for each of the three coloring compounds or with those calculated from transmission readings.

TABLE 4.1

Varying Uptake of the 3 Single Dyes

by the Test Fabric

Item	Red	Blue	Yellow
Weight Percent Dye Remaining	11.4%	19.8%	20.4%
Exhaustion	88.6%	80.2%	79.6%
Transmission	78.1%	45.5%	33.9%

TABLE 4.2

Differing Abilities of 3 Dyes in the Mixture to Alter

Light Transmission through the Dyebath

Item	Red	Blue	Yellow
Transmission	50%	48.3%	44.8%
Weight Percent Dye	10%	10%	10%
Exhaustion	90%	90%	90%
Known molar concentration	$.78 \times 10^{-7} M$	$.66 \times 10^{-7} M$	$.68 \times 10^{-7} M$
Calculated concentration	$.58 \times 10^{-7} M$	$.96 \times 10^{-7} M$	$.38 \times 10^{-7} M$

3. Evaluation of Graphs 3.2, 3.3, and 3.4 verifies the inter-
    ference of dyes in the mixture with individual transmission
    readings, thereby prohibiting their use to determine indi-
    vidual dye concentrations in a dye mixture.

Within the limits of this research, the effects of interference
are minimal; 2 to 3 percent. Although transmission readings may not
be used to quantitatively determine dye concentration and exhaustion,
they may be used qualitatively as an indication of increasing or de-
creasing exhaustion. Calculations made for each set of the dyeings
conditions--determined by pH, maximum dyeing temperature, and time at
that temperature--produced concentrations indicating exhausting per-
centages that increased or decreased with Color Eye color measurements.
Transmission was used, therefore, as an exhaustion level indication and
this research concluded that concentration calculations were not neces-
sary to evaluation of exhaustion percentage of the test mixture.

STATISTICAL ANALYSIS

Data obtained from reflectance and transmission readings, con-
tained three duplicates for each of the three pH levels, within the
six sets of temperature-time conditions, which were replicated three
times. The dyeing conditions yielding maximum transmission and re-
flectance values for the experimental system were determined by the
results of the data. The procedural design varied pH within each
dyeing of constant time and temperature.

Reflectance–Color Measurement

Analysis of variance for reflectance data is reported in Tables 4.3, 4.4, and 4.5. Mean differences of significant variables are recorded in Table 4.6. Results indicate that:

1. There are no significant differences in X (red), Y (green and lightness), and Z (blue) mean values as affected by maximum temperature of dyeing (T) or length of time at maximum dyeing temperature (L).

2. The pH level (P) is related to time and temperature of dyeing. Significant differences at the .01 level for the X and Y values and at the .05 level for the Z value indicate that a pH of 4 gives the highest X, Y, and Z values with the least deviation within that acidity level. In addition, Z values at pH 6 are significantly higher than pH 8. The higher the color tristimulus value, the more color is on the fabric (32).

3. Reflectance readings showed that time and temperature readings had no interaction with color reproducibility.

Correlation between the Color Eye Spectrophotometer and the Hunter Color Difference Meter used by Manay (10) and Cook (8) shows that the Lightness values L and Y have a +.9937 correlation and the –b and Z blueness readings have an inverse correlation, –.8191. Data from either color measurement instrument would, therefore, produce the same results.

Transmission--Exhaustion

Analysis of variance using transmission values for each of the three dyes is reported in Tables 4.7, 4.8, and 4.9. Mean differences

TABLE 4.3

Analysis of Variance--Reflectance Readings

X Value

(red)

Source	Degrees of freedom	Sum of Squares	Mean Square	F-statistic
Replications	2	2.9998	1.4999	1.3485
Treatment combinations	5	4.2822	.8564	--
T (temperature)	2	3.3703	1.6852	1.5151
L (time)	1	.0386	.0386	--
(TxL)	2	.8733	.4367	--
Error a	10	11.1229	1.1123	
P's (pH)	2	6.0244	3.0122	6.6290[a]
(PxT)	10	4.2984	.4298	--
Error b	24	10.9064	.4544	
Sample error	107	19.4047	.1814	
Total	160	59.0388		

[a]Significant at the .01 level.

TABLE 4.4

Analysis of Variance--Reflectance Readings

Y Value

(green and lightness)

Source	Degrees of freedom	Sum of Squares	Mean Square	F-statistic
Replications	2	1.4357	.7179	--
Treatment combinations	5	3.0672	.6134	--
T (temperature)	2	1.9766	.9883	1.1707
L (time)	1	.0968	.0968	--
(TxL)	2	.9938	.4969	--
Error a	10	8.4418	.8442	
P's (pH)	2	3.9793	1.9897	6.6147[a]
(PxT)	10	2.9083	.2408	--
Error b	24	7.2196	.3008	
Sample error	107	16.9689	.1586	
Total	160	44.0208		

[a]Significant at the .01 level.

TABLE 4.5

Analysis of Variance--Reflectance Readings

Z Value

(blue)

Source	Degrees of freedom	Sum of Squares	Mean Squares	F-statistic
Replications	2	1.3865	.6933	1.1805
Treatment combinations	5	1.7247	.3449	--
T (temperature)	2	.8183	.0492	--
L (time)	1	.0850	.0850	--
(TxL)	2	.8214	.4107	--
Error a	10	5.8725	.5873	
P's (pH)	2	1.5884	.7942	3.5266[b]
(PxT)	10	2.3866	.2387	1.0599
Error b	24	5.4043	.2252	
Sample error	107	14.7922	.1382	
Total	160	33.1552		

[b]Significant at the .05 level.

TABLE 4.6

Means of Reflectance Values

for Varying pH

Reflectance Values	pH		
	4	6	8
X (red value)	[c] 12.93	12.60	12.48
Y (green and light value)	[c] 12.45	12.19	12.08
Z (blue value)	[c] 11.54	[c] 11.49	11.31

[c]The difference between two means not underscored by the same line is significant at the .05 level.

TABLE 4.7

Analysis of Variance--Transmission Values

Disperse Red 4

(Palanil Pink RF)

Source	Degrees of freedom	Sum of Squares	Mean Squares	F-statistic
Replications	2	50.42	25.21	6.26^b
Treatment combinations	5	94.21	18.84	4.67^b
T (temperature)	2	88.33	44.17	10.96^a
L (time)	1	5.51	5.51	1.37
(TxL)	2	.37	.19	--
Error a	10	40.27	4.03	
P's (pH)	2	14.03	7.02	10.97^a
(PxT)	10	14.25	1.43	2.23
Error b	24	15.33	.64	
Sample error	106	60.83	.57	
Total	159	289.34		

[a]Significant at the .01 level.

[b]Significant at the .05 level.

TABLE 4.8

Analysis of Variance--Transmission Values

Disperse Blue 3

(Eastman Blue GBN)

Source	Degrees of freedom	Sum of Squares	Mean Square	F-statistic
Replications	2	70.34	35.17	7.60[a]
Treatment combinations	5	81.56	16.31	3.52[b]
T (temperature)	2	77.84	38.92	8.41[a]
L (time)	1	1.31	1.31	--
(TxL)	2	2.41	1.21	--
Error a	10	46.34	4.63	
P's (pH)	2	24.31	12.16	9.43[a]
(PxT)	10	19.74	1.97	1.53
Error b	24	30.91	1.29	
Sample error	106	143.40	14.34	
Total	159	416.60		

[a]Significant at the .01 level.

[b]Significant at the .05 level.

TABLE 4.9

Analysis of Variance--Transmission Values

Disperse Yellow 3

(Intrasperse Yellow GBA)

Source	Degrees of freedom	Sum of Squares	Mean Square	F-statistic
Replications	2	17.60	8.80	1.66
Treatment combinations	5	10.44	2.09	--
T (temperature)	2	9.03	4.52	--
L (time)	1	.01	.01	--
(TxL)	2	1.40	.70	--
Error a	10	52.86	5.29	
P's (pH)	2	6.55	3.28	54.67[a]
(PxT)	10	1.52	.15	2.50[b]
Error b	24	1.33	.06	
Sample error	106	5.22	.05	
Total	159	95.52		

[a]Significant at the .01 level.

[b]Significant at the .05 level.

of significant variables are recorded in Tables 4.10 and 4.11. Results indicate that:

1. pH of 4 gives the highest transmission readings for all three dyes with significant differences at the .05 level between readings for pH of 6 and 8. The higher the transmission reading, the lower the dye concentration in the final dye-bath, and the higher the exhaustion level. This corresponds with conclusions from reflectance data.

2. Time differences, as with reflectance, do not significantly affect the red, blue, and yellow transmission values obtained. The lower time for dyeing was, therefore, selected.

3. Mean values of transmission as indicated by temperature are significantly different at the .01 level for the red and blue dyes, although not for the yellow dye. Temperatures of (150°F) 65.5°C and (175°F) 79.4°C produced the highest trans-mission values for red and blue and equally good results for yellow as at any other temperature.

Confidence in transmission as a valid indicator is derived from observation of its value as a qualitative measure for dye concentration and exhaustion.

TABLE 4.10

Means of Transmission Values

for Varying pH

Transmission Values	pH		
	4	6	8
Red	[c] 26.36	25.77	25.71
Blue	[c] 37.29	36.52	36.43
Yellow	[c] 4.55	[c] 4.34	[c] 4.06

[c]The difference between two means not underscored by the same line is significant at the .05 level.

TABLE 4.11

Means of Transmission Values

for Varying Temperature

Transmission Value	Temperature		
	(150°F) 65.6°C	(175°F) 79.4°C	(200°F) 82°C
Red	26.74	26.15	c 24.96
Blue	c 35.80	36.99	37.45
Yellow	4.33	4.60	4.02

[c]The difference between two means not underscored by the same line is significant at the .05 level.

CHAPTER V

SUMMARY AND CONCLUSIONS

Within the limits of this investigation, the following procedure gives the highest levels of dye exhaustion and reproducibility of color for the acetate test fabric.

RECOMMENDED CONDITIONS

1. Pre-scour using Nadjort Washer:

    600 grams acetate knit (102g/sq. meter)
    90 grams AATCC detergent (15% owf)
    Wash 10 minutes at (140°F) 60°C
    Rinse 10 minutes at (120°F) 49°C
    Repeat the Wash and Rinse
    Centrifuge
    Air dry

2. Stock dye solutions:

    Prepare stock solutions of each dye volumetrically: 1 gram
      of each dye is weighed on an analytical balance and
      transferred to 100 ml flask and made up to volume (re-
      ferred to as a 1% solution).
    Red--1% solution--Palanil Pink RF
    Blue--1% solution--Eastman Blue GBN
    Yellow--1% solution--Intrasperse Yellow GBA

3. Dyebath Preparation:

    Add 2 ml of each stock dye solution (12% owf)
    Add 10 ml of 1% Igepon T-51 solution (1 gram/100 ml)
    Make up to 100 ml total liquid volume. Bath Ratio--20:1
    Adjust pH of dyebath to 4

4. Dyeing Procedure using Atlas LaundryOmeter:

    Add pH adjusted dyebath to wash cups and place on heating
      table with neoprene gaskets and metal lids.

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Step LaundryOmeter to (100°F) 38°C
Add 5 gram fabric samples and seal with Ball jar lid, neo-
    prene gasket, and metal lid.
Weigh cup on direct reading balance.
Place wash cups in rotary constant bath drum and step to
    (175°F) 74.9°C, approximately (20°F) .7°C every 10 minutes.
Run at (175°F) 74.9°C for 30 minutes.
Cool to (140°F) 60°C, approximately (30°F) 1°C every 10
    minutes.
Reweigh cup to determine weight loss or gain.

5. Post treatment:

Rinse dyed samples in clean LaundryOmeter cups, using dis-
    tilled water at approximately pH of 6.
Dip fabric into cup 10 times.
Air dry samples on flat, clean paper towels.

MAJOR FINDINGS

Interpretation of data corresponding to experimentation with the
three variables--time, temperature, pH--that affect dyeing of acetate
test fabric leads to the following conclusions about this research.
All results are valid within the specified conditions--instrumentation,
test fabric, chemicals, and other constant test procedures and methods.

1. The time variables, as evaluated, had no effect upon repro-

    ducibility of color for or the exhaustion of dye into the

    acetate test fabric. Further extension of the time at the

    maximum dyeing temperature might reveal the two time lengths,

    30 and 45 minutes, to represent a plateau area on a curve

    plotting temperature versus exhaustion or color measurement.

    Length at maximum dyeing temperature of 30 minutes reduced

    cost, time, and energy for dyeing.

2. The temperature indicating highest exhaustion levels for dye-

    ing the test fabric was clearly determined by transmission

values for each of the three dyes in the test dyebaths--
Mean values of transmission for the yellow dye at the three
test temperatures--(150°F) 65.6°C, (175°F) 79.4°C, (200°F)
82°C--were not significantly different. (175°F) 79.4°C
did not conflict with the other data and was, therefore, con-
cluded to be the best temperature.

3. Reflectance and transmission data reported pH to signifi-
cantly affect the dyeing of the test fabric. A pH of 4 pro-
duced the highest X, Y, and Z tristimulus color values and
the highest transmission values, indicating exhaustion, for
the red, blue, and yellow dyes.

4. Transmission for each dyestuff in the mixture read directly
from the Spec 20 is a valid qualitative measurement for
increasing or decreasing dye concentration and, thus, dye
exhaustion. Interference of the individual dyes used in
the mixture was minimal.

5. Leakage is controlled through use of Ball Jar Lids as an
additional sealant for the neoprene gasket and metal lids
on the AATCC Wash Cups IIA.

SUGGESTIONS FOR FURTHER RESEARCH

Several new areas and possibilities for further research are
recommended.

1. Evaluation of test results by additional testing: physical
    and chemical testing, other methods of color measurement,
    and dye exhaustion.

2. Calculation of McAdam Color Units for the data to determine
    the tolerance of color matching for the test conditions.
    A commercial color match is 2-1/2 McAdam Units (32). This
    method of evaluation was used by the AATCC Research Com-
    mittee in the initial experiment with a standard laboratory
    dyeing procedure for dyeing acetate (12).

3. Extension of the three variables' limits, especially time,
    to obtain additional data for the optimal procedural condi-
    tions. Examination of additional variables, such as dye
    concentration, to determine their effect on dyeing

4. Translation of these laboratory results into information
    related to industrial equipment and mass volume.

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APPENDIX

The following instruments and chemicals with Registered Trademarks^R have been cited in this research.

Acetate--Registered Trademark for E. I. du Pont de Nemours.

Atlas LaundryOmeter--Registered Trademark for Atlas Electric Device Co.

Ball jar lid--Registered Trademark for Ball Brothers Company.

Celanese--Registered Trademark for Celanese Corporation.

Color Eye--Registered Trademark for Instrumental Development Laboratories, Kollmorgen Corporation.

Eastman Blue GBN--Registered Trademark for Eastman Chemical Products, Inc.

Estron--Registered Trademark for Tennessee Eastman.

Hunter Color Difference Meter--Registered Trademark for Hunter-lab Associates

Igepon T-51--Registered Trademark for GAF Corporation, Chemical Division.

Intrasperse Yellow GBA--Registered Trademark for Crompton and Knowles Corporation

Nadjort Washer--Registered Trademark for Robert Ewing and Sons, Inc.

Palanil Pink RF--Registered Trademark for BASF Wyandotte Corporation.

Seraceta--Registered Trademark for American Viscose.

Spec 20--Registered Trademark for Bausch and Lomb.

VITA

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Elizabeth Lea Winfrey

EXAMINATION OF VARIABLES THAT

AFFECT THE DYEING OF ACETATE

IN A THREE DYE SYSTEM

by

Elizabeth Lea Winfrey

(ABSTRACT)

This research evaluated three variables within an AATCC experimental laboratory procedure for dyeing acetate. Evaluation of color measurement from test samples and transmission, an indicator of dye exhaustion, from final dyebath was focused on reproducibility of color from dyeing to dyeing and maximum dye exhaustion. Experimental effects were 1) pH of dyebath, 2) high dyeing temperature for the test fabric, an acetate tricot, and 3) time at the high dyeing temperature. Major findings included:

1. pH of 4 produces significantly higher X and Y color reflectance values, measured by the Color Eye[R] at the .05 level. pH of 4 and 6 produce significantly higher Z color values at the .05 level. Transmission readings, measured by the Spec 20[R], indicate exhaustion for the Disperse Red 4, Disperse Blue 3, and Disperse Yellow 3 dyes and are significantly higher for pH of 4 at the .05 level.

2. Temperature of (150°F) 65.6°C and (175°F) 79.4°C give significantly higher transmission readings for Disperse Red 4 at

the .05 level. (175°F) 79.4°C and (200°F) 82°C give signi-
ficantly higher transmission readings for Disperse Blue 3
at the .05 level. Disperse Yellow 3 is unaffected by tempera-
ture.

3. Time at the high dyeing temperature had no effect on color
measurements or transmission.

4. Transmission is a qualitative indicator of dye concentra-
tion and exhaustion.

5. Leakage was prevented with one time use of Ball Jar Lids[R]
on the AATCC Wash Cups IIA used on the Atlas LaundryOmeter.[R]