THE EFFECT OF TEMPERATURE, TIME, AND HYDROGEN ION CONCENTRATION ON THE COLORFASTNESS OF A HOUSEHOLD DYE ON SELECTED COTTON FABRICS

by

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Chapter I

INTRODUCTION

The arts of dyeing and creating designs with dye are very ancient. As early as 3,000 B.C., China, Persia, and Egypt had flourishing textile crafts, including coloring and decorating the fabric (1). The art of dyeing continues to be widely practiced today, but with a more varied choice of dyes and more simplified methods.

Although most home dyes on the market are relatively easy to use, there are some unsolved problems that accompany the use of these dyes. It is important that the consumer be aware of the limitations that exist with the use of a household dye. The contemporary craftsman and home dyer working with household dyeing procedures have neither the time nor desire to exert the effort required to dye textiles, unless they can be reasonably certain that their efforts will not be lost within a short time (2).

The fastness or permanence of a dye is an important consideration when selecting the dyestuff for home usage. It is stated that no household dye is completely colorfast and that "bleeding" will occur (3). However, very little information is available regarding research in which there was an attempt to improve colorfastness properties of these dyes.

There is evidence indicating the necessity for research in the area of consumer-applied products, including home dyes, on fabrics (4). A research project which will examine problems concerning colorfastness

of home dyed fabrics would provide useful information not only for the home dyer, but also decorators, textile designers, theatrical costumers who often use household dyes (5), and perhaps, household dye manufacturers.

An interest in crafts, textile design, and the consumer problems of using household dye as a decorating tool led this researcher to attempt to develop a laboratory procedure, reproducible in the home, for improving the colorfastness of a household dye.

The objectives of this research are as follows:

1. To develop a laboratory procedure which is reproducible in the home for improving the colorfastness of a red household dye at one concentration using cotton drill and crease resistant cotton muslin.
2. To evaluate the effects of the pH (the hydrogen ion concentration) of the dyebath, the temperature of the dyebath, and the time of dyeing.
3. To evaluate the effects of the colorfastness of the dye by using afterwashes of acetic acid and copper sulfate.
4. To evaluate the treated samples for lightfastness, washfastness, and crocking.

Chapter II

REVIEW OF LITERATURE

At the time of this research, literature on dyeing using household dyes and home dyeing procedures was very limited. No research was found which dealt with consumer problems that often occur in home dyeing situations. With a growing interest in home dyeing, information based on research is needed in this area. The literature reviewed is, therefore, supportive to this research topic.

Historical Review of Dyeing

Unlike sculpture, painting, or architecture, the art of dyeing is perhaps the one art in which not only the ancient world, but the world of comparatively few years ago was distinctly inferior to that of the present day. With each century, dyeing has been changing and developing (6).

The earliest dyes of Ancient Egypt, China, and Persia were probably of vegetable origin: flowers, leaves, berries, and roots. These were found to give exquisite color (1)(6)(7). The Chinese, around 3,000 B.C., were dyeing fabrics a deep orange by using water of iron springs, and often crushed or burnt lime, gypsum, clay, and coal were the sources of color. Perhaps the most celebrated dye in history was Tyrian Purple made by the Phoenicians about 2,000 B.C. with a fluid from a shellfish found along the coast of Phoenicia (8).

Although these natural dyes produced beautiful colors, they were not colorfast. While some were fast to light, none were fast to water. Many attempts were made to bind the dye to the fabric. The pigment was sometimes mixed with either a resin obtained from trees, with egg albumen, blood, or saliva, or it was roasted with turpentine, glues, or wax. The demand for a permanent fixation of color on fabric resulted in experiments with "magic properties" found in certain rivers, sea water, saliva, and urine (1).

The art and practice of dyeing was completely revolutionized in 1856 with the discovery of the artificial dyestuff named mauveine, or more commonly, mauve. William Henry Perkin found that aniline derived from coal tar could be chemically treated to produce beautiful colors. With this discovery began the close association of dyeing and chemistry (6)(8).

A serious difficulty with early dyestuffs called "Basic Dyes" was their characteristic of being far less colorfast than the best of vegetable dyes. They also did not fade true. A piece of cloth dyed bright red and then exposed to sunlight would have portions that would appear yellow, white, and even a darker red. In 1868 with the artificial manufacturing of alizarine, a whole new class of dyestuffs was developed that proved to be an improvement in both fastness to light and washing (6).

Color and General Dye Characteristics

Color is an important aspect of textiles. The color we see when examining a dyed textile is formed by subtractive mixing. The dyestuff

used absorbs a range of colors emitting only the one that is seen, whether a pure hue or an optical mix. Optical mixing is done by our eyes. The color in the dyestuff is created by the presence of chemical groups called chromophores. The intensity of the color created by the chromophores depends on the presence of one or more chemical groups called auxochromes. The auxochromes can give water solubility to the dye and form associative bonds with the fiber (9)(10).

In order to be successful, dyestuffs must possess all of the following properties:

1. "intense color
2. solubility or the ability to be dispersed into an emulsion
3. a small enough size to be either reacted with, or absorbed into, the subject fiber
4. colorfastness to external phenomena such as light, laundering, perspiration, gases, or other color-attacking factors" (9).

The dyer must choose a dye suited to the fiber content and the end use of the fiber, and the color should be applied so that it can penetrate and be held within the fiber. "A knowledge of fiber-dye affinity, method of dyeing, and equipment will give the consumer a better understanding of color behavior" (7).

According to Adrosko (2), one key to successful dyeing is the use of soft water. Writers of early nineteenth century dye manuals never failed to mention this point due to the fact that very few dyestuffs will work effectively in hard water. "Although the calcium, magnesium, and iron salts and other minerals which make water hard may not change color radically, they can cause spotting and irregular distribution of dyes in textiles" (2).

Household Dyes

The modern household dye industry did not begin until after Perkin's discovery of synthetic dyestuff in 1856 (8), although, records of books telling the housewife how to dye things at home date back as early as 1817 with "The Domestic Manufacturer's Assistant and Family Directory in the Arts of Weaving and Dyeing" by J. and J. Bronson.

The first United States patent (US40, 263) referring to household fabric dye was issued to Howe and Stevens on October 13, 1863. In 1864, Howe and Stevens were not only actively selling their "Family Dye Colors," but were offering some of the new synthetic dyes made from coal tar derivatives. This contributed a breakthrough in two major areas: 1) it signaled the utilization of synthetic dyes in preference to animal and vegetable dyes, and 2) it was probably the first significant attempt to market dyes for home use (11).

In an effort to provide convenience and satisfaction for the consumer, packaged dyes for home use have taken many forms through the years. Some of these were powdered forms in pouches or envelopes, flakes of dye and soap, pressed cakes of dye and salt, effervescent dye cakes and tablets, dyes in solution of water or water and acetic acid, dyes in water soluble packets, and dyes in aerosol form (11).

In a study by Farris (4), it was found that several types of dyestuffs are combined in packaged household dyes. Acid, direct, and disperse dyes are used in varying percentages depending on the shade and strength of dye required. A high percentage of the total weight of a

household dye is sodium chloride (salt). Salt acts to give better dye absorption and provides uniformity in weight (4)(10).

Household dyes will dye most common fabrics, with the exception of polyester, some acrylics, glass or mineral fibers; therefore, a combination of dyestuffs is necessary to produce a household dye which will work to some degree of satisfaction for a variety of fabrics. According to Johnson (12), however, no one dye could possibly be successful in dyeing all types of fibers or react in the same way to all aftertreatments.

> Basically the problem of perfecting a home package dye involved the selection and blending of the dyes that gave good all around fastness; were level-dyeing on various fibers; that could be applied successfully by the inexperienced as well as the skilled home-dyer with simple utensils found in the kitchen or home; responded well over a wide range of temperature; as well as the blending of selected dyes so as to take well on a wide range of fibers or mixtures. (11)

Labeling of Household Dyes

In 1947, the Federal Trade Commission issued a trade practice rule for the household dye industry regulating the labeling of household dyes (13). The rules which pertain to this research are briefly discussed below.

Rule 1 states that the terms "all fabric," "all purpose," or similar terms may not be used if the dye or tint will not color to the shade specified for "all types and kinds of fabric commercially produced and used in germents, household furnishings or home decoration," unless accompanied by a statement which informs the consumer of the limitations or exceptions. Rule 2 states the reasonable approximations

of the size of the article or quantity of fabric which the dye will satisfactorily cover must be truthfully and nondeceptively stated (13). Rule 4 restricts or prohibits the use of the term "fast," "fadeproof," "washfast," "sunfast," or similar terms unless the color, when applied as directed, shows no substantial change or deterioration during the life of the article under normal wear and laundering conditions. Rule 7 of the FTC practice rules prohibits the use of guarantees which mislead the consumer in respect to the serviceability, utility or effectiveness of the household dye (13).

Testing for Colorfastness

Because the need for a particular kind of fastness depends on the nature of the color change and the fabric end use, methods for testing color permanence in fabric exposed to numerous wearing and laundering conditions are important if satisfactory dyeing results are to be achieved. The Standard Methods for the Determination of the Colour Fastness of Textiles defines colorfastness as "the resistance of the color of textiles to the different agencies to which [they] may be exposed . . . " (14). Only in recent years has standardized testing for colorfastness been carried out on scientific lines, though there are records showing interest in the fastness of dyestuffs to light and washing, even as early as the first century A.D. The guilds in medieval times had very strict control over the dyers who were either "dyers of fast colors" or "dyers of fugitive colors" (14).

Modern researchers attribute some of the insistence on forbidding

the printing of cotton in eighteenth century France to the realization that standards of dye fastness "left a lot to be desired" (14). This is sometimes a complaint of the dyer using household dyes. The dyer, therefore, often must sacrifice one attribute of the dye for another, i.e., the brilliance of the color desired may be given up to ensure washfastness (12).

Lightfastness Testing

Adrosko (2) tested color permanence of natural dyes for fastness to light by two methods. Samples of the dyed fabric for the first method were exposed for forty hours to the rays of a carbon-arc lamp. During the testing, half of each piece was shielded while the rays of the lamp shone directly on the uncovered part. The second method for testing lightfastness could be performed in the home without the use of a fading apparatus. Instead, the samples are exposed to sunlight. Each sample is fastened in a cardboard frame with a two inch opening. Samples are then placed outdoors in direct sunlight and tilted toward the sun. After a few days exposure, the frame is removed and differences in covered and uncovered portions are evaluated. Although not an absolutely conclusive test, it would suggest lightfastness properties of a dyed fabric that would be exposed to sunlight, such as a drapery fabric (2).

Washfastness Testing

Adrosko's (2) test for washfastness consists of placing samples in half-pint containers partially filled with a neutral soap solution (1.0% for cotton) at 120°F and agitating these containers for thirty

minutes. Samples are removed and rinsed by agitating in lukewarm water for ten minutes. Samples are dried and compared to original unwashed fabric by a rating system used for both lightfastness and washfastness.

Evaluating Colorfastness

After colorfastness tests are carried out, the samples subjected to the particular treatment are assessed on the basis of a visual comparison between original material and the test specimen. In order to give a rating to the amount of change that has occurred, the Gray Scale for Color Change (15) is used. "The fastness rating of the specimen is that number of the gray scale which corresponds to the contrast between the original and the treated specimen" (14).

Cotton and Its Dyeing Characteristics

Unlike most fibers, the origin of cotton is unknown. Most authorities will agree, however, that cotton was produced and utilized in India about 3000 B.C. (10). Cotton is considered the most universally used fiber and is used for a multitude of purposes; being both durable and easy to care for. It is also characterized by high absorbency, easy dyeability, and a high degree of pliability and flexibility (10).

The basic unit of the cellulose molecule is formed from glucose, made up of the chemical elements carbon, hydrogen, and oxygen (7)(9)(10). In the natural cellulose molecule, estimates of the degree of polymerization are as high as 10,000 glucose monomers per molecule. The glucose monomer has an abundance of hydroxyl (-OH) groups which aid in

water attraction making cotton a readily absorbent fiber (7)(9). Fibers that are easily dyed are those which contain absorption sites that will react with the dye molecule (7).

For processes such as dyeing and finishing, water penetration is a key property. Major factors that influence the effect of water-borne processes on textiles are:

1. the degree of polymerization of the fiber
2. the crystallinity of the polymer group
3. presence or absence of fiber-reactive groups (9)

The degree of polymerization, the total number of monomer units within a single polymer chain, is responsible, in part, for the degree of crystallinity within a fiber. The crystalline areas consist of highly oriented molecules. Molecules in an amorphous region exhibit low orientation, which means that the molecules lie in random arrangements, allowing high absorbency. The cotton fiber has been thought to contain as much as 25% amorphous polymer which allows for significant water penetration and ease in dyeing (9)(10)(16).

Crease Resistant Cotton

The crease resisting property is one which is somewhat difficult to define. "It is generally agreed that the term includes both resistance to and recovery from creasing, but with much greater emphasis on recovery" (16). Cellulosics are extremely susceptible to creasing, and the application of a resin not only retards wrinkling, but can bring improvements in hand, draping, and overall textile character (16).

Crease resistant cotton has approximately 3% resin. Resins are applied to cotton as a monomer solution. The monomers penetrate the fibers, and heat curing forms cross-links between the molecular chains of the cellulose (7).

Crease resisting resins are applied after the dyeing process in manufacturing as the dye molecules cannot adequately penetrate the fibers once a resin treatment has been applied. The resin protects the cellulose against degradation by light and can actually improve fastness properties of dyed fabric (16). However, when the crease resistant finish is applied before dyeing, the dye results are often unsatisfactory. The cotton fiber with the resin treatment is unable to swell as fast as the untreated cotton and, therefore, the dye cannot penetrate as rapidly or as evenly. No literature advising the home dyer of this problem with resin treated fabrics could be found.

Conditions Influencing a Dyeing Procedure

In preparing for a dyeing procedure, many factors must be considered, such as: dye selection, weight of the sample, liquor ratio (fabric to dye liquor), pH of dyebath, temperature of dyeing, and time of dyeing (17)(18).

The pH of the dyebath can influence the strike rate of the dye, the transfer of the dye, and also the final color value obtained (18). Temperature affects the rate of dye penetration, and both temperature and time control the total shade and fastness obtained (17)(18). These conditions need to be carefully examined in order to work towards the improvement of colorfastness properties of household dyes.

Chapter III

PROCEDURE

For this research, attempts to improve colorfastness properties of a liquid household dye, using two selected cotton fabrics, were made. This research considered important variables such as: (1) dyeing time, (2) dyeing temperature, (3) pH of the dyebath, and (4) aftertreatments that could affect colorfastness to light, washing, and crocking. From all variables considered, 36 different dyeing combinations were investigated. This included two dyeing times, two dyebath temperatures, three pH levels, and three treatments after dyeing.

Description of Test Fabrics

The two cotton fabrics used for this research were selected because of their easy dyeability and availability to the consumer. The first fabric was cotton drill weighing 7.2 oz. per square yard. The second fabric was cotton muslin having a crease resistant finish and weighing 3.3 oz. per square yard. The crease resistant muslin was chosen to determine if any differences would occur in dyeing and colorfastness performance because of the crease resistant finish.

Sampling Procedure

The fabrics used were unwashed, and from each of the fabrics, specimens were cut 6" in the warp direction x 4-1/2" in the filling

direction. Four test samples for each of the 36 individual dyeing conditions for each of the cotton fabrics were cut. The test specimen size was determined in order to accommodate the three physical tests for colorfastness: to light, to washing, and to crocking.

Code numbers were assigned for each of the dyeing times, temperatures, and pH levels. Fabric samples were then notched according to the code numbers assigned for distinguishing samples. The sectioning of the samples for testing will be described later in this chapter.

Preparation of the Dyebath

For this research, a red liquid household dye was chosen. Liquid dye was decided upon rather than a powdered dye because of convenience and easier mixing. Also, it was found that more stores surveyed carried household dye in the liquid form, which may be an indication of consumer preference.

The laboratory dyeing procedure was developed in order to simulate a home dyeing situation. The water to dye ratio was calculated for a 16 gallon capacity washing machine using an 8 oz. bottle of liquid dye. According to the manufacturer's directions, an 8 oz. bottle will dye up to 2 lbs. of dry fabric. Using this ratio as a guideline, the fabric-to-dye liquor was determined. Two samples per 400 ml of dye liquor, consisting of 1.6 ml of dye to 400 ml of water, were within the limit suggested by the manufacturer.

Because the pH of the dyebath affects the transfer of the dye and the total color value obtained (18), steps were taken to adjust

the pH using means available to the household dyer. For lowering the pH of the dyebath to 4.4 - 4.5, ordinary white vinegar (5% acetic acid) was used. A 10% solution of sodium bicarbonate was used to raise the pH of the dyebath to 8.9 - 9.0.

Dyeing Procedure

Prior to dyeing, a code sheet for distinguishing fabric samples was developed. Numbers were also assigned to various dyebath formulations for marking the Launder-Ometer^{R*} containers.

1. The dye solution for one set of dyeings was prepared.

2. Fabric specimens were notched according to code numbers. Notches were used rather than permanent ink marks, which may have been illegible after dyeing.

3. Each Launder-Ometer container was filled with 400 ml of prepared dyebath. Four containers were set aside to be dyed at a pH of 7.6, the pH level of the dye from the manufacturer. For lowering the pH to 4.4 - 4.5, 32 drops (1.7 ml) of white vinegar were added to four more containers. To raise the pH of the dyebath to 8.9 - 9.0, 10 ml of 10% solution of sodium bicarbonate (ordinary baking soda) were added to the last four containers.

4. Five stainless steel balls were added to each of the Launder-Ometer containers to aid agitation.

5. The dyebath in the Launder-Ometer containers was preheated to designated temperatures using the Launder-Ometer preheating tray. The

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*Launder-Ometer^{R} is a trademark for Atlas Electric Device Co.

first temperature setting of 140°F was chosen as the approximate temperature for the hot water setting of a washing machine. Because the home dyer or craftsman may prefer to "pan dye" articles where the dyebath would be heated to the boil, a temperature of 210°F was also selected as a variable.

6. The samples to be dyed were wet completely and hand squeezed to remove excess water. Samples were then added to the containers; two samples per container based on liquor ratio mentioned under Preparation of Dyebath, and immersed completely using a glass stirring rod.

7. A Ball[R]* jar lid was placed on the top of each container, making certain that no nicks or dents were present which might result in leakage.

8. The Launder-Ometer cap, with a neoprene gasket inside, was clamped on tightly, and the container was turned upside down to check for leaks.

9. The containers were loaded on the Launder-Ometer racks and fastened securely.

10. The containers were rotated in the Launder-Ometer for 10 minutes at the temperatures previously mentioned. Six of the containers were removed, and the remaining six containers continued dyeing for an additional 20 minutes.

11. Samples were immediately removed from the containers after dyeing and rinsed thoroughly in cold water until the water was clear.

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*Ball[R] is a registered trademark of Ball Brothers Co.

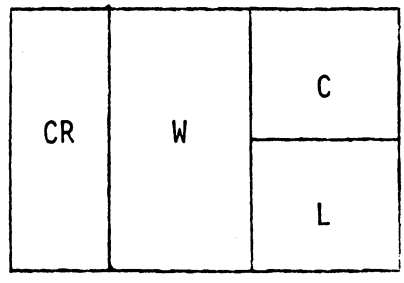
The samples were hand squeezed to remove excess water and laid flat to air dry.

Procedure for Application of Afterwashes

Two afterwashes were applied to the fabric samples after dyeing. The first solution was made by combining 10 ml of 5% acetic acid (white vinegar) with one liter of water (approximately 4-1/8 cups). The second afterwash was a 1% solution of copper sulfate. Enough of each solution was made to afterwash all specimens of each fabric type. However, the two fabrics were not treated in the same container. Forty-eight specimens for each of the fabrics were thoroughly washed, one at a time, in the acetic acid. This was repeated with the copper sulfate solution. When completely soaked, the samples were removed and squeezed with a hand wringer, and laid flat to air dry.

Preparation of Samples for Colorfastness Testing

After dyeing and aftertreating, samples were marked with code numbers in indelible ink. Samples were then sectioned as illustrated below for testing of lightfastness, washfastness, and crocking.



CR = crocking
W = washfastness
L = lightfastness
C = control

Code numbers were marked each of the four sections.

Procedure for Testing Colorfastness to Light

1. Using the procedure listed in the AATCC Test Method 16A-1977 (15), samples were prepared and mounted in specimen holders. Samples were then exposed for a period of 20 hours using the Carbon-Arc Lamp Fade-Ometer^{R*}.

2. Samples were rated after exposure using the Gray Scale for Color Change (15).

Procedure for Testing Colorfastness to Washing

1. Washings were done in a Maytag^{R} agitator type washing machine. The equipment used for washfastness testing was chosen in order to simulate home laundering practices.

2. The machine was set for full water level, warm water wash, and cold water rinse.

3. Ninety grams of AATCC Standard Detergent were added along with the test specimens and enough dummy pieces to make a four lb. load (AATCC Test Method 124-1975).

4. The fabric samples were agitated for a 10 minute washing cycle on the normal setting and allowed to proceed automatically through the final spin cycle.

5. Washings were repeated for three additional cycles before samples were removed and air drived at room temperature.

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*Fade-Ometer^{R}--Trademark of the Atlas Electric Device Co.

6. The samples were then rated after four washings according to the Gray Scale for Color Change (15).

Procedure for Testing Colorfastness to Crocking

1. Samples were tested for their colorfastness to both wet and dry crocking. This would determine the degree of color which might be transferred from the dyed sample to other surfaces by rubbing.

2. Samples were crocked according to the AATCC Crockmeter Test, AATCC Test Method 8-1977 (15).

3. The crocked specimens were evaluated by the use of the AATCC Chromatic Transference Scale (15).

Standards for Evaluation of Color Change

For tests of colorfastness to light and washing, the Gray Scale for Color Change was used for evaluation. The scale consists of standard gray chips, each pair representing a difference in color or contrast corresponding to a numerical fastness rating. The test samples were rated by comparing the difference in color of the tested specimen and the original textile with the differences represented by the Scale (15). Samples were rated from one to five indicating the degree of color change as follows:

5 = Negligible or no change

4 = Slightly changed

3 = Noticeably changed

2 = Considerably changed

1 = Much changed

For the crocking tests, specimens were evaluated using the AATCC Chromatic Transference Scale (15). The rating of 5 depicts no color transference, with all other ratings showing a similar graduation in depth of color down to a rating of 1 or the heaviest color transference.

All ratings were done in a period of two days. The samples were rated by random selection under the MacBeth Light^{R} set for daylight. After ratings were completed, samples were sorted, and ratings numbers were recorded on a data sheet.

Chapter IV

RESULTS AND DISCUSSION

Data concerned with the effects of dyeing time, dyeing temperature, pH of the dyebath, and aftertreatments on the colorfastness of a liquid household dye were obtained according to the procedure described in Chapter III. Ratings for colorfastness to light, washing, and crocking were recorded. The ratings for lightfastness and washfastness were statistically analyzed using the analysis of variance procedure.

Observations During Data Collection

The following section describes observations recorded prior to and during testing.

1. Because of the size of the sample and the large number of specimens to be dyed, the use of a washing machine for dyeing was impractical. Therefore, the Launder-Ometer was the dyeing machine chosen.

2. The temperatures of the dyebath were based on: (a) the water temperature of a washing machine set for hot water, which was approximately 140°F, and (b) the temperature for pan dyeing on a stove top at the boil, approximately 210°F. The Launder-Ometer enabled dyeings at these temperatures to be achieved with relative ease and consistency.

3. In the initial testing with the household dye, some of the samples were washed in mild detergent and rinsed thoroughly before dyeing. However, after examining dyed samples, no difference was found

in the washed and unwashed samples. It was then decided to only wet samples before placing them in the dyebath.

4. In preparing the dyebath, the amount of liquid dye per Launder-Ometer container was 1.6 ml. In order to make measuring easier and more accurate, a solution consisting of 120 ml of dye and 300 ml of water was mixed and poured into a flask that was sealed with a rubber stopper to prevent evaporation. This dye solution was used for one set of dyeings, which consisted of 36 different dyeing conditions.

5. It was observed that the dyestuff would begin to separate in the flask after sitting overnight. Therefore, it was important to shake the dye well before use.

6. Ball^{R} jar lids were used to minimize leakage in Launder-Ometer containers. During the dyeing procedure, leakage occurred in 5 out of 144 containers used. It was important to check the lids after each use to make certain no damage was present that might prevent lids from fitting tightly. The lids were discarded after 10 - 12 dyeings.

7. The cotton drill fabric dyed a darker shade of red for all dyeing conditions investigated. It was noted that the crease resistant muslin showed signs of unlevelness in dyeing. The problem of dye penetration due to a crease resistant finish prior to dyeing was evident in this research.

8. It was thought that an extended period of dyeing would produce more satisfactory results. However, fabrics dyed for 30 minutes showed very little, if any, difference from those samples dyed for 10 minutes. With the household dye used in this research, it can be assumed that

most of the dyeing took place within the first 10 minutes.

9. With the first experimental dyeings, it was noticed that the crease resistant cotton muslin had a color difference from one side of the fabric to the other. In order to make certain that the same side of the sample would be evaluated and that ratings would be consistent, samples were marked with a dot of permanent ink in the upper left hand corner before dyeing. The color difference could be attributed to the resin treated finish.

10. A decision was made to wear rubber gloves during the dyeing procedure to prevent prolonged periods of skin contact with the dye solution.

Analysis of Data

Four replications for each of the 36 dyeing conditions were made for each of the fabrics tested. Analysis of the data showed no significant interactions. The means of treatments for lightfastness were grouped by Duncan's New Multiple Range Test. There were significant main effects which will be discussed below.

Colorfastness to Light

1. Results of the rating tests for lightfastness showed a significant difference between fabrics. In Table 1, the results show the cotton drill had better lightfastness properties. The crease resistant cotton muslin had very poor colorfastness to light with a considerable loss of color after 20 hours of exposure in the Fade-Ometer.

Table 1

Analysis of Variance for Varying Fabrics,

Dyeing Times and Temperatures

Source	Means			
	Lightfastness		Washfastness	
Fabric	F_1	F_2	F_1	F_2
	1.72	3.42*	1.29	2.97*
Time	T_1	T_2	T_1	T_2
	2.49	2.65	2.02	2.25
Temperature	TP_1	TP_2	TP_1	TP_2
	2.22	2.92*	1.77	2.50*

*The difference between the two means not underscored by the same line are significant at the 0.05 level.

F_1 = crease resistant muslin

F_2 = cotton drill

T_1 = 10 minutes

T_2 = 20 minutes

TP_1 = 140°F

TP_2 = 210°F

2. No significant difference was found in the dyeing times of 10 minutes and 30 minutes.

3. The samples dyed at 210°F had significantly higher ratings for lightfastness than samples dyed at 140°F.

4. There was no significant difference in colorfastness to light due to the variations in pH of the dyebath, as shown in Table 2.

5. Table 2 shows the results of the aftertreatments on lightfastness. There was a significant difference found in samples treated with the copper sulfate solution. These samples showed less color loss after exposure in the Fade-Ometer than either the acetic acid treatment or samples without aftertreatment.

6. Although there were no significant interactions among the factors, rating averages are given in Tables 3, 4, 5, and 6. From these tables, conclusions can be drawn as to the dyeing condition which would yield the best results for lightfastness. The combination of a dyebath temperature of 210°F, a dyeing time of 30 minutes, and an aftertreatment with a copper sulfate solution showed the highest lightfastness ratings. The pH of the dyebath had little influence on the final result, therefore, no alteration of the pH, as it is sold to the consumer, is recommended.

Colorfastness to Washing

1. The results in Table 1 show a significant difference between the two fabrics in colorfastness to washing, with the 100% cotton drill having the highest ratings.

Table 2

Analysis of Variance for Varying pH Levels and Aftertreatments

Source	Means					
	Lightfastness			Washfastness		
pH Levels	P_1	P_2	P_3	P_1	P_2	P_3
	2.49	2.49	2.73	2.09	2.15	2.15
Treatments	TR_1	TR_2	TR_3	TR_1	TR_2	TR_3
	2.17	2.28	3.26*	2.04	2.15	2.22

*The difference between the two means not underscored by the same line are significant at the 0.05 level.

P_1 = 7.6

P_2 = 4.4-4.5

P_3 = 8.9-9.0

TR_1 = No Treatment

TR_2 = Acetic Acid

TR_3 = Copper Sulfate

Table 3

Rating Averages for Interaction of

Varying Times and Temperature

Source	Means			
	Lightfastness		Washfastness	
Muslin	TP_1	TP_2	TP_1	TP_2
T_1	1.31	1.94	1.00	1.39
T_2	1.33	2.31	1.03	1.75
Drill	TP_1	TP_2	TP_1	TP_2
T_1	3.06	3.64	2.36	3.34
T_2	3.19	3.78	2.70	3.53

Time

T_1 = 10 minutes

T_2 = 30 minutes

Temperature

TP_1 = 140°F

TP_2 = 210°F

Table 4

Rating Averages for Interaction of Varying Temperatures and Aftertreatments

	Means			
Source	Lightfastness		Washfastness	
Muslin	TP_1	TP_2	TP_1	TP_2
TR_1	1.00	1.17	1.04	1.50
TR_2	1.04	1.79	1.00	1.54
TR_3	1.92	2.88	1.00	1.54
Drill	TP_1	TP_2	TP_1	TP_2
TR_1	2.54	3.42	2.17	3.46
TR_2	2.75	3.54	2.54	3.38
TR_3	4.08	4.17	2.88	3.46

Temperature

TP_1 = 140°F

TP_2 = 210°F

Treatments

TR_1 = No Treatment

TR_2 = Acetic Acid

TR_3 = Copper Sulfate

Table 5

Rating Averages for Interaction of Varying Times and Aftertreatments

Source	Means			
	Lightfastness		Washfastness	
Muslin	T_1	T_2	T_1	T_2
TR_1	1.25	1.46	1.17	1.38
TR_2	1.38	1.46	1.21	1.46
TR_3	2.25	2.54	1.21	1.33
Drill	T_1	T_2	T_1	T_2
TR_1	2.92	3.04	2.63	3.00
TR_2	2.96	3.33	2.79	3.13
TR_3	4.17	4.09	3.13	3.21

Times

T_1 = 10 minutes

T_2 = 30 minutes

Treatments

TR_1 = No Treatment

TR_2 = Acetic Acid

TR_3 = Copper Sulfate

Table 6

Rating Averages for Interaction of

Varying pH and Aftertreatments

Source	Lightfastness			Washfastness		
Muslin	P_1	P_2	P_3	P_1	P_2	P_3
TR_1	1.25	1.31	1.50	1.13	1.44	1.25
TR_2	1.50	1.31	1.44	1.25	1.50	1.25
TR_3	2.19	2.38	2.63	1.13	1.50	1.19
Drill	P_1	P_2	P_3	P_1	P_2	P_3
TR_1	2.88	2.75	3.13	2.69	2.75	3.00
TR_2	3.13	2.94	3.38	3.13	2.69	3.06
TR_3	4.00	4.25	4.13	3.25	3.06	3.19

pH

P_1 = 7.6

P_2 = 4.4-4.5

P_3 = 8.9-9.0

Treatments

TR_1 = No Treatment

TR_2 = Acetic Acid

TR_3 = Copper Sulfate

2. The samples dyed for 30 minutes had no significant differences from those dyed for 10 minutes.

3. The samples dyed at 140°F showed significantly lower ratings than those dyed at 210°F.

4. Table 2 shows no significant difference in washfastness ratings due to variations in the pH of the dyebath.

5. The effects of aftertreatments on overall washfastness ratings, presented in Table 2, show no significant difference in treatments.

6. Referring to Tables 3, 4, 5, and 6, there was no significant interaction among the factors investigated. From the data given, the variables that produced the most satisfactory dyeing combination for colorfastness to washing are as follows: (1) a dyebath temperature of 210°F, (2) a dyeing time of 30 minutes, (3) no pH alteration of the dye as it is manufactured, and (4) an aftertreatment of 1% copper sulfate solution.

Colorfastness to Crocking

Tests for wet and dry crocking were performed as described in Chapter III. Samples were not analyzed statistically because visual ratings showed no differences for any of the 36 dyeing conditions tested. The only difference in the crocking test was between the two cotton fabrics themselves. Both fabrics showed good to excellent colorfastness to dry crocking, with the cotton drill having a rating of 5--depicting no color, and the crease resistant cotton muslin showing only slight color transference with a rating of 4.

For wet crocking, the muslin fabric showed a noticeable amount of color transference with a rating of 3. The cotton drill, regardless of dyeing treatment, also was given a rating of 3.

Chapter V

SUMMARY AND CONCLUSIONS

The craftsman and home dyer want some assurance that their efforts required in home dyeing will produce satisfactory results. Because some problems still exist for consumers using household dye, the objectives of this research were to develop a dyeing procedure for improving the colorfastness of a household dye using two cotton fabrics; to evaluate the effects of time, temperature, and pH of the dyebath and determine the dye condition which would give optimum colorfastness; to evaluate the effects of aftertreatments; and to evaluate the dyed samples for lightfastness, washfastness, and crocking.

Dyeings were carried out using a red liquid household dye obtained from a single manufacturer. The Launder-Ometer was used as the dye machine. Tests for lightfastness, washfastness, and crocking were run on a total of 288 specimens.

Data were obtained by rating samples according to the degree of color change after testing. Ratings were recorded and an analysis of variance was conducted to determine any significant differences between dyeing conditions and interactions.

Results of the analysis indicated the following major findings:

1. The aftertreatment of 1% copper sulfate solution resulted in significantly higher lightfastness ratings. The same aftertreatment, however, showed no significant difference for washfastness or crocking.

2. The temperature of the dyebath had a significant influence on the results of colorfastness ratings for both fading and washing. A temperature of 210°F produced higher ratings than a temperature of 140°F. This would, however, limit the use of a washing machine for dyeing as the water setting would not reach the boil. If a main concern of the dyer is colorfastness, then pan dyeing, where dyebath temperatures could be heated above 140°F, would be the better method.

3. In evaluating the overall results of tests for lightfastness and washfastness, the cotton drill fabric received higher ratings as compared to the crease resistant cotton muslin. From the data analyzed and the literature reviewed, it can be concluded that the crease resistant finish has a somewhat negative effect on household dyes. In both colorfastness tests, the muslin fabric was given ratings between 1 and 2, indicating a considerable change in color from the control.

4. Although colorfastness ratings tended to be slightly higher for a dyeing time of 30 minutes than 10 minutes, the difference was not significant. Therefore, it can be concluded that leaving the fabric in the dyebath for a period of time longer than that indicated by the dye manufacturer would not increase colorfastness.

5. Although not analyzed statistically, the household dye performed well in crocking tests for both fabrics with all dyeing combinations considered. A rating of 5 (no change) was given for the cotton drill fabric, and the crease resistant muslin showed only a slight color transference with a rating of 4. For wet crocking, both fabrics

tested, regardless of treatment, received a rating of 3 (noticeable color transference).

6. From the data analyzed, no interaction was found among variables investigated. Any significant difference in samples could be attributed to an added effect of variables, such as an increase in the temperature of the dyebath.

In developing a research project, certain limitations must be recognized. This research was concerned with one red household dye and its colorfastness properties. However, it must be understood that all household dyes, especially those of a different color, may not give the same results found in this research. Further study using different dye brands and a wider range of colors is needed. Because of the many variables investigated and the large number of dyeing combinations, it was not feasible to study more than two fabrics. Therefore, a recommendation for further research would involve a dye project using other fabric types, particularly blends and synthetics.

Recommended Dyeing Procedure for Optimum Colorfastness

1. Follow manufacturer's directions for determining water to dye and fabric to dye ratios.

2. Heat the prepared dyebath to the boil, approximately 210°F in a container large enough to accommodate the fabric.

3. Thoroughly wet fabric and hand squeeze to remove excess water.

4. Place fabric in preheated dyebath, stirring constantly to make certain dye is covering fabric completely.

5. Dye fabric at the boil for 30 minutes, or until desired shade is reached. Stir fabric continuously to prevent unevenness.

6. After 30 minutes, remove fabric and rinse in cold water until water is clear. Let fabric air dry or tumble dry at low heat.

7. Prepare a 1% solution of copper sulfate. Copper sulfate, sometimes called Blue Stone, can be purchased at most drug stores. An example of a 1% solution would be 3 liters of water to 30 grams of copper sulfate crystals. Warm water can be used to aid the dissolving of the crystals in the solution.

8. After the copper sulfate crystals are completely dissolved in the water, soak the fabric in the solution for 1 - 2 minutes. Remove, squeeze out excess liquid, and let air dry. This aftertreatment helps to "set" the color.

One limitation of the copper sulfate solution is that it may affect the final dye shade, depending on the color used. Copper sulfate will dull the shade slightly, and the dyer may have to compromise the color desired if optimum colorfastness is a major objective. Copper sulfate significantly improved colorfastness to light. It is recommended that this aftertreatment be used for articles, such as draperies, that would be exposed to long periods of light.

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THE EFFECT OF TEMPERATURE, TIME, AND HYDROGEN ION CONCENTRATION ON THE COLORFASTNESS OF A HOUSEHOLD DYE ON SELECTED COTTON FABRICS

by

Susan Elaine Green

(ABSTRACT)

A liquid household dye and two fabrics, a cotton drill and a crease resistant cotton muslin, were used to pursue the following objectives: (1) to develop a dyeing procedure for improving the colorfastness of a household dye; (2) to evaluate the effects of temperature, time, and pH (hydrogen ion concentration) of the dyebath on the colorfastness properties of the household dye selected; (3) to evaluate the effects of aftertreatments of acetic acid and copper sulfate; and (4) to evaluate the dyed samples for colorfastness to light, washing, and crocking.

Dye results were evaluated by the use of the Gray Scale for Color Change and the AATCC Chromatic Transference Scale. Colorfastness ratings were statistically analyzed. Major findings of the research included the following:

1. The cotton drill fabric had higher colorfastness ratings for lightfastness, washfastness, and dry crocking than did the crease resistant cotton muslin. For wet crocking, both fabrics had considerable color transference.

2. A dyebath temperature of 210°F produced significantly higher ratings for lightfastness and washfastness in both fabrics tested.
3. The aftertreatment of 1% copper sulfate solution resulted in significantly higher lightfastness ratings for both fabrics tested.
4. There was no significant difference in the dyeing times of 10 minutes and 30 minutes, although the time of 30 minutes produced slightly higher ratings.