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Development of a Dyed Verification Standard

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DEVELOPMENT OF A DYED VERIFICATION

STANDARD

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ABSTRACT

Color fastness is important for satisfactory performance of textiles in use. Successful color fastness testing relies on accurate interpretation of published test methods, and control of laboratory conditions. The accuracy of testing may be checked by the use of a verification standard, which should produce known results if the test is carried out correctly. The AATCC does not have a verification standard for two of its most often-used colorfastness tests: Method 15 (Colorfastness to Perspiration) and Method 61 (Colorfastness to Laundering). A survey of dyes in the Colour Index was used to select candidate disperse, direct and acid dyes on the basis of their fiber suitability, fastness, availability, and hue. The dyes were applied individually to cotton or nylon as appropriate, and their fastness to perspiration and laundering assessed. The results were used to plan combinations of dye, which were applied to a nylon-cotton blend. Once again, fastnesses of the dyeings were assessed.

In a final step, the combination dyeings showing the most appropriate (i.e. borderline pass/fail) levels of fastness were subjected to the variations in the test method conditions to see which responded most strongly to such variations The combination comprising of Direct Blue 80 (0.2% owf), Disperse Red 60 (0.5% owf) and Acid Red 299 (0.2% owf) performed well as verification fabric for colorfastness to laundering. This combination did not perform well as a verification for colorfastness to perspiration; such a fabric will need to be developed separately.

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CHAPTER 1

INTRODUCTION

The coloration of textile products has been practiced since ancient times. Today, the majority of textile items are colored by dyeing or printing. Colorfastness is the resistance of a material to changes in any of its color characteristics, transfer of its colorant(s) to adjacent materials, or both as a result of exposure to any environment that might be encountered during the processing, testing, storage or use of the material. Hence, colorfastness is an important deciding factor in evaluating the quality of a colored textile. (AATCC, 2015) Colored textiles should exhibit satisfactory colorfastness to agents such as light, laundry, crocking, chlorine, and perspiration based on their intended end use. Agents such as acids/alkalis, oxidizing/reducing agents, dry-cleaning solvents, burnt gas fumes, heat, oxides of nitrogen, ozone, and chlorinated water, may cause color changes in textiles, and fastness to those agencies also may be required and determined by testing.

When consumers put these colored textile products to use they are expected to be fast to common agents such as laundering, perspiration, light, and crocking. A product that fails to perform satisfactorily results in degrading the brand image as well as a huge loss for the company and the manufacturer. Hence, rather than wait for poor fastness to reveal itself during use by the ultimate consumer, its likely occurrence may be predicted by standardized test methods. Precision and accuracy of the test results rely on the test being performed according to the test protocol.

Retailers and manufacturers need to be certain that an item is serviceable and ensure its colorfastness performance using standardized tests; if not done correctly they face the risk of monetary loss, reputation, and consumer loyalty. (Bide, 2010) It might not be obvious to a laboratory performing the tests that the protocol is being followed correctly, producing test results that are not be an accurate representation of the performance of the product. A dyed fabric of known colorfastness can be used as a verification fabric to validate the results of colorfastness tests. Such materials are available for some colorfastness tests, but not in the US for two of the most commonly used colorfastness tests, i.e. colorfastness to laundering and perspiration. This study aimed to develop a dyed verification standard for those tests.

CHAPTER 2

REVIEW OF LITERATURE

Despite the fact that ad-hoc testing of colorfastness has long been practiced, the formation of a committee by the British Association for the Advancement of Science in the 1890s was one of the early attempts to formalize the evaluation of colorfastness. (Tordoff, 1984) Dyed fabrics were included in early test methods as the comparisons that directly provided test results, but these were provided as recipes (a specific fabric dyed with a certain amount of a particular dye) rather than dyed fabrics being supplied directly. Fastness was an early topic of interest for the American Association of Textile Chemists and Colorists (AATCC) founded in late 1921. The seventh meeting of its Research Committee held on June 9, 1922, included discussion of fastness to washing, soaping, and laundering of dyed and printed cotton against white cotton. (American Dyestuff Reporter, 1922) A provisional test method specified four classifications and the types of dyes belonging to each classification based on their fastness properties. See Table 1 (American Dyestuff Reporter, vol. 10, p. 449)

Table 1. Classification of Fastness to Laundry

Classification	Type
1. When treated the shade is unaffected and white cotton is untinged.	Alizarine- old method. Indanthrene Yellow G paste (849) 6% dyeing. Indanthrene Violet RR Ex. Paste (767) 16-ounce printing color. Naphthol AS developed with Fast Red G Base.
2. When treated the shade is somewhat reduced but white cotton is untinged.	Brilliant Indigo B (885) 5% dyeing or 4 ounces to gallon printed shade.
3. When treated the shade is considerably affected.	Alizarine Blue S (804) printed shades. Sulphur Black (720).
4. When treated the shade is badly affected.	Methylene Blue B (659) dyed or printed shade.

The October 1934 Report of the Society of Dyers and Colourists' (SDC) Fastness Committee on the Standards for light perspiration and washing described four series of washing tests based on the severity of laundering. Two series of standards, one in blue and one in red, were selected to provide a pass/fail result. (Tordoff, 1984) In 1942, tests showed that the dyed standards were not satisfactory. In fact, they were developed only to show the permissible loss in color under a particular test only to facilitate the judgment of results. (Tordoff, 1984)

The colorfastness test to laundering for dyed or printed cotton followed by AATCC until 1940 similarly suggested four testing methods with varying temperatures, times and amounts of sodium carbonate, chlorine; and five classes of fastness to washing from 0-4. The test results were determined by comparison with a corresponding standard dyed sample for each class. Standard dyed samples were derived from the following dyed on full bleached 64 x 60 cotton print cloth:

Class I – 2% dyeing of Diamine Fast Blue FFB Pr. 71.

Class II – 4% deying of Primuline Conc. C.I. 812. Diazotized and developed with Beta Naphthol.

Class III – 2% dyeing of Vat Blue 2BD 100% powd. C.I. 1184.

Class IV – 3-1/3% dyeing of a Vat Violet BN 100% powd. C.I. 1163. (AATCC, 1940)

Unsatisfactory results from dyed standards led to the idea of using fixed standards as the only rational solution to the problem of assessment of colorfastness. The first proposal to use set standards had been made in 1939 by a member of the SDC's Washing Fastness Sub-Committee. At the time, the test methods were under revision and thus the fixed standards were not accepted. (Fastness Test Co-ordination Committee, 1953) Unlike dyed standards, these are not produced each time the test is carried out, but produced only once for the assessment of staining; a series of five patterns ranging from white to medium depth would be used. The idea of using fixed standards was later conceived by in the USA in 1945 by the U.S. Quartermaster Corps. At about the same time, similar gray scales were developed in Europe by Imperial Chemical Industries Ltd. (Fastness Test Co-ordination Committee, 1953). Later in 1954, AATCC accepted gray scales as the evaluation process for colorfastness. (AATCC, 2015)

The current use of gray scales is described in AATCC's Evaluation Procedures 1 (EP1) and 2 (EP2) (AATCC, 2015). Unlike versions developed in Europe in 1943, these include half-step ratings. Unsatisfactory colorfastness may show itself as a loss of color on an item or in the staining of an adjacent material. Therefore, the evaluation of colorfastness focuses separately on change in color (EP1) and staining (EP2) with a separate gray scale for each. The evaluation of color change or staining may be through visual or instrumental assessments, both based on the gray scales originally developed for visual evaluation. For changes of color, AATCC Evaluation Procedure 1-2012 describes the use of a gray scale that consists of a series of pairs of standard gray chips. One of the pairs is constant, and the other increasingly different with the differences starting with zero (a grade of 5) and increasing to a grade of 1. (AATCC, 2015) The difference in the original and the test specimen is compared to these pairs. The gray scale pair with the color difference equal to that of the original/test specimen is given as the colorfastness grade, where 5 represents no change in color and 1 represents a significant change in color. Staining is evaluated in a parallel manner, except that the constant chip is white: the use of this scale is described in AATCC Evaluation Procedure 2-2012. An original unstained white "adjacent fabric" is placed next to the fabric from the test procedure, and again, the gray-scale pair with the same difference is given as the staining rating of the test. The grade is assigned ranging from 5, i.e. no stain, to 1, which is highly stained. (AATCC, 2015) Despite using precisely made gray scales for visual assessment it can be difficult to get precise results, and well-trained observers are required. The evaluation of color change or staining can be also assessed instrumentally, as explained in AATCC EP 7-2009, and EP 12-2012 respectively. These procedures assess color difference in a way unique to fastness testing and were developed to correspond directly to the grey scales. The calculations turn spectrophotometric lightness (L*), chroma (C*), and hue (H*) values to gray scale readings that correlate directly with the non-linear scale used for visual evaluation developed before the instrumental measurement was available. (AATCC,

2015) The color difference (ΔEF) is calculated using L*, C* and H* values, ΔEF is then converted to gray scale grade using following formulas:

$GSc = 5 - \Delta EF / 1.7$ (when $\Delta EF \leq 3.4$) or

$GSc = 5 - \text{Log}(\Delta EF / 0.85) / \text{Log}(2)$ (when $\Delta EF > 3.4$).

Hence, ΔEF can be used to provide the gray scale value for the color change. (Bide, 2010) Similarly, for stained material, data is collected from spectrophotometer of reflectance of stained and unstained materials to calculate CIELAB color difference (ΔE^*) and magnitude of lightness difference (ΔL^*) which is used further to calculate Gray Scale difference (ΔEGS). The staining scale grade is computed using the equation:

$SSG = 6.1 - 1.45 * \ln(\Delta EGS)$

If SSG results in more than 4, then it is recalculated using following formula:

$SSG = 5 - 0.23 * (\Delta EGS)$

Hence, ΔEGS can be used as a determinant for staining. (Bide, 2010) AATCC Evaluation procedures should evaluate test results as precisely and accurately as possible.

With the introduction of gray scales, the use of dyed standards declined, although verification or control fabrics continued to be included in some tests. In 1982, an AATCC subcommittee focused on developing a standard dyed-comparison fabric to reduce variance in the colorfastness to hypochlorite bleach test. (Clark, 2001) Standards for colorfastness to chlorinated swimming pool water, and atmospheric contaminants also were developed, but the use of these standards was dropped as the results were not satisfactory.

Various organizations and associations publish standard procedures to test colorfastness, notably the American Association of Textile Chemists and Colorists (AATCC) and the International Organization for Standardization (ISO). Test procedures and standards set by AATCC are widely accepted in the US and internationally for evaluating the performance of a textile material in various areas including colorfastness. Most of the colorfastness tests are either based on the real conditions that the textile will undergo during use, or they provide an accelerated version of the real situations. Acceleration may be achieved by changing test conditions, such as temperature or light intensity. (AATCC, 2015) Hence, the test conditions are not always representative of real situations and may require specific testing equipment to run a test as specified in the procedure. AATCC TM 61-2013: Colorfastness to Laundering: Accelerated, defines in detail the apparatus, reagents and materials for six tests with varying temperature, detergent solution concentration and volume, the number of steel or rubber balls and time according to the desired severity of the test ranging from hand laundering to machine laundering under different conditions. The test is designed to give results that approximate the color change effects of five launderings in a time frame of 20 to 45 minutes. (AATCC, 2015) Hence, test conditions, reagents, and materials like temperature, percentage detergent and agitation agents (steel/rubber balls) are higher when compared to a single laundering under normal conditions. One of the commonly used tests for colorfastness to perspiration, AATCC TM 15-2013, is not an accelerated test method and involves wetting the sample in simulated acid perspiration solution (pH 4.3 ± 2), which is prepared using a standard reagent recipe. The wet sample with a controlled solution amount is heated in an oven at 38˚C ± 1˚C (slightly elevated relative to normal body temperature) for 6 hours ± 5 minutes and evaluated for colorfastness using the appropriate evaluation method. (AATCC, 2015)

Despite such detailed test procedures and evaluation methods, some degree of variation in the test and its results is inevitable when carried out by different testers and/or in different laboratories, and the extent of that variation is (or should be) determined as part of the test development. The degree of variation expected in different circumstances (within a lab, between lab and number of determinations) is expressed in the test method's "Precision and Bias" statement, which gives critical differences (the difference between test results required for there to be statistical significance at 95% significance level). Such critical differences are, in some tests, surprisingly high. The critical difference for AATCC TM 61-2013 (Colorfastness to Laundering: Accelerated) in rating color change ranges from 0.36 to 1.37, and for staining it ranges from 0.33 to 1.39. Whereas, for visual assessment of the results, the critical difference as accepted by the procedure ranges from 0.46 to 1.03 in the case of a single operator, 0.91 to 1.29 within a laboratory and 1.17 to 1.49 grading for between laboratories. (AATCC, 2015) Such high critical differences, this might cause disagreements between laboratories, or between customer and supplier, causing confusion, delays, and monetary loss.

Furthermore, operators may be untrained, and the test method language may be difficult to interpret accurately. These factors increase both the likelihood of tests being mis-performed, and the number of disagreements between laboratories, or between customer and supplier. Results that are not reliable may lead to failure of the product at the consumer level, degrading the brand image as well as creating a huge loss for the vendor on shipment rejection.

All tests require control of the many variables in the test procedure. Any lack of control may affect the results of the test. The tests are written for completeness and not for easy reading, and unintended variations of procedure are not uncommon. With so many factors driving the results of the test, it is important to check if the test is being run as directed under standard procedures. Unlike their earlier role as essentially "built-in" gray scales, today, standard verification fabrics can play a significant role in deciding the reliability of the test results of each test.

Most notably among current verification fabrics, AATCC TM 16 uses AATCC blue wool lightfastness standards to verify the light exposure provided by the instrument. (AATCC, 2015) A standard dyed fabric with 0.4% C.I. Disperse Blue 3 on cellulose acetate satin, and standard of fading dyed with 0.3% C.I. Direct Blue 8 and 0.015% Direct Violet 47 based on the weight of the viscose satin fabric is used for colorfastness to burnt gas fumes (AATCC TM 23). These dyes are used because they tend to exhibit different fading characteristics with change in variables. (AATCC, 2015) A dyed standard of fading and control sample ribbon is currently in use for colorfastness to ozone in the atmosphere under high humidities (AATCC TM 129). Also, for AATCC TM 164 colorfastness to oxides of nitrogen in the atmosphere under high humidities, a similar standard of fading and control sample are being used. Both the control sample and the standard for fading are dyed with a combination of dyes. The principle of verification fabric works as it fades at a different pace when variables such as temperature and humidity fluctuate; this is achieved by the combination of dyes used. Similarly, the color change of the control will reflect the combined effects of atmospheric contaminants and temperature and humidity variations. (AATCC, 2015)

A standard crockmeter verification cloth was developed by AATCC Committee RA38 using a direct navy dye and was tested for use with Crockmeters in 2006 for verification of AATCC TM 8, i.e. colorfastness to crocking. The test procedure also allows the use of an in-house fabric with known crock fastness in place of a verification fabric. Similarly, the test method for colorfastness to perspiration (AATCC TM 15) permits the use of an in-house fabric with mid-range staining on one of the contents of multi-fiber strips for verification. (AATCC, 2006) However, the use of an in-house colored fabric with known behavior is not a reliable source to verify the test procedures more broadly, between labs, because in-house fabrics are not standardized and may exhibit fastness outside critical pass-fail levels. Moreover, the supply and the quality control of such fabric may be uncertain.

Colorfastness to Laundering: Accelerated (AATCC TM 61) uses no verification fabric to validate the test results. Laundering is an important process of care and maintenance of textile products. In the US during a lifetime of one year of a textile product, 52 wash cycles are considered as average, making colorfastness to laundering a significant factor in the performance of a colored textile. (Muthu, 2015) AATCC test method 61 developed in 1950 by AATCC Committee RA60 has been continually revised to reflect changes in laundry practices and detergent formulation. The test has 5 test conditions to mimic a range of laundry conditions.

Most apparel is subject to perspiration when worn, making colorfastness to perspiration another important aspect to determine the quality of clothing. In AATCC test method 15, colorfastness to perspiration, a colored fabric is wetted with a simulated acidic perspiration solution, subjected to fixed pressure of 8 lbs and allowed to dry at slightly elevated temperature. The test method suggests the use of an in-house colored textile of known colorfastness producing mid-range of staining as verification. However, as discussed earlier, using an in-house fabric may not give the exact idea of the accuracy of the test procedure.

Globalization of the textile industry has meant that standard test procedures are being performed all over the world for commercial use, as well as research and development. Fabrics are tested in different labs at various stages of the supply chain, from manufacturing to retailer, to ensure good colorfastness. With high critical differences, possibilities of human error and incorrect test conditions, it is critical to verify the test results to avoid risks of product failure regarding its performance. There are presently no standard verification fabrics for AATCC TM61: Colorfastness to Laundering: Accelerated and AATCC TM15: Colorfastness to Perspiration in use. There is thus an opportunity for the development and use of verification fabrics for these widely used test methods.

With encouragement from AATCC, this work was aimed at developing a verification fabric that would provide a gray scale staining with one or more stains in the range of 3 to 4 on the multi-fiber fabric used in the test. Such values are typically those that represent a pass or fail level in industrial practice. A mid-range staining would also offer more scope for change (lesser or more staining), with altered variables.

Before conducting experimental work, initial selection of dyes and fabrics was required. Dyes may be classified chemically, or, more usefully, by their application 'type', when they are divided into groups based on their suitability for certain fiber types. While these groups may tend to have generally similar fastness properties, the fastness of dyes within a group can vary. Dyes with excellent colorfastness will achieve less or no color change and staining irrespective of the test conditions, on the other hand dyes with poor colorfastness properties will result in low reading for stains and color change which will be difficult to see changes when the test conditions are altered. For this study, dyes with moderate to poor fastness properties were considered essential.

Many cellulosic fibers are in use, cotton being one of the important ones. Cotton is dyed with direct dyes, sulfur dyes, azoic dyes, reactive dyes and vat dyes. (Christie, 2001) Sulfur dyes are used widely on cotton because they are economical, but tend to have good wash fastness: further, their chemical compositions are uncertain, making the supply of a consistent product less certain. Sulfur dyes were thus not considered further. Azoic dyes are classified as "ingrain dyes" since as the colorant is formed in the fiber by a reaction between intermediate compounds. (Perkins, 1996) As the colorant formed in the fiber is extremely insoluble, it is fast to laundering, making this class unsuitable for the development of verification standard. Similarly, vat dyes are water-insoluble pigments that are applied after conversion to a water-soluble salt using an alkaline reducing agent, which after application are re-oxidized to insoluble pigments, imparting exceptional colorfastness properties. (Perkins, 1996) Hence, vat dyes were ruled out from the study. Reactive dyes, as the name suggests, react with the fiber and become fixed, they have excellent fastness to washing because of the high strength of the covalent bond. (Perkins, 1996) The reaction is not 100% efficient, however, and achieving such fastness requires extensive rinsing to remove the dye that has not reacted with the fiber: if this does not happen, the unfixed dye will bleed in use and fastness might be poor. However, for this project, controlling the level of fixation to generate a known degree of fastness would be difficult. Direct dyes are economical, simple to apply and provide bright shades but with typically moderate to poor fastness to laundering. Of all the dye classes for cotton, direct dyes represent the most suitable class for this study. (Perkins, 1996)

Acid dyes are anionic and applicable to fibers containing free amino groups that protonate under acid conditions, hence their name. Nylon is a polyamide, has such free amino groups and is routinely dyed with acid dyes. The color fastness of acid dyes ranges from good to excellent, giving a selection of dyes that can generate the required levels of staining for this project. Disperse dyes are useful for hydrophobic thermoplastic fibers. Their primary application is on polyester, but nylon, acetate, and acrylics can also be dyed using disperse dyes. The wash fastness of disperse dyes varies with the fiber to which they are applied. For the project, it is useful that their fastness on nylon is low. (Perkins, 1996) Therefore, after considering various dye classes and the fibers for which they are suitable, cotton and nylon were chosen as fibers, and a combination of direct, acid and disperse dyes would be most appropriate.

A verification fabric is used to validate test results on a regular basis by various testing firms, and results over time might need to be compared. Ensuring dye availability in the long run therefore is of utmost importance for a successful verification fabric. The availability of dyes was determined by examination of the 1991 AATCC Buyer's Guide, to find dyes listed generically that were produced at that time by ten or more manufacturers. Dyes with questionable environmental or health effects were eliminated from consideration on the basis of possibility of future restricted. Yellow colors are difficult for visual assessment and were avoided. Additionally, the same generic dye produced by different manufacturers or different lots can perform differently in terms of colorfastness, hence, the dyes used in the study were used from same brand and lots. The six dyes found to fit these criteria are the following along their commercial names:

C.I. Acid Orange 116 (Nylosan Orange SL)

C.I. Acid Red 299 (Nylosan Rubine S5B)

C.I. Disperse Blue 56 (Dispersol Blue B-R)

C.I. Disperse Red 60 (Resolin Red FB)

C.I. Direct Blue 8 (Pyrazol Fast Blue BGUL)

C.I. Direct Red 80 (Pyrazol Fast Red 7BSW)

CHAPTER 3

METHODOLOGY

The study was conducted in three phases. The first stage involved application of the selected dyes individually to cotton and nylon fabrics to determine the staining of each of the dyes in fastness testing. Based on the results, in the second stage combinations of dye were applied to a nylon-cotton blend fabric. In the final stage, the best combinations were tested for staining ratings resulting from the use of standard and modified conditions of AATCC TM 61. The methodology for each phase is explained in detail below.

Materials and Equipment

Materials:

- Direct dyes
- Disperse dyes
- Acid dyes
- Sodium chloride
- Sodium carbonate
- Acetic acid
- Ammonium sulphate
- Monosodium phosphate
- Dispersing Agent – StarLev WWB

- Cotton plain weave fabric with thread count 77X66 and fabric weight of 3.0oz/sq yd
- Texturized knit nylon fabric with fabric weight of 6.5oz/sq yd
- 1x2 twill weave 50- 50 nylon-cotton blend with 54X86 thread count, and fabric weight of 6.6 oz/sq yd
- Lactic acid
- l-histidine monohydrochloride
- AATCC 1993 Standard Reference Detergent without optical brighteners

Equipment:

- Ahiba Polymat sample dyeing machine
- Atlas Launder-Ometer
- Horizontal Perspiration Tester
- Spectrophotometer X-Rite and Color iControl Software

Phase 1: Staining evaluation of selected dyes

The selected dyes, C.I. Acid Orange 116, C.I. Acid Red 299, C.I. Disperse Blue 56, C.I. Disperse Red 60, C.I. Direct Blue 80, and C.I. Direct Red 80 were applied individually to cotton and nylon fabrics in shades of 0.1%, 0.2%, 0.5%, 1%, 2%, and 4% on weight of the fabric (owf) using a batch dyeing process. Dyeing was carried out with material to liquid ratio (MLR) of 1:25 on samples weighing 5gms each. Dyes and chemicals were pre-dissolved and the volumes required were calculated using the following formula.

Millileters required = %owf X Weight of fiber / % solution strength

The dyebath for direct dyes was prepared with dye solution, and 10% owf sodium chloride. The bath was then transferred to a canister with the pre-wetted cotton cloth was run in Ahiba Polymat dye machine with temperature rising at 4˚F per minute and is maintained at 180˚F for 60 minutes. The bath was then cooled to 120˚F, the samples removed, rinsed and dried.

Acid dyes were applied to texturized nylon in a bath containing the required amount of dye solution, ammonium sulfate (5% owf) and acetic acid (1g/l). The dye bath was made up to 125mL with water, the pH was measured, and adjusted if necessary to pH 6 with acetic acid or ammonia. Pre-wetted nylon samples were added and dyeing carried with temperature increased to 212˚F at 5˚F per minute, maintained at 212˚F for 60 minutes, with subsequent cooling to 120˚F. The dyed samples were then removed, rinsed, and dried.

Disperse dyes were applied to same texturized nylon fabric used for acid dyes using a bath containing 1g/l StarLev WWB, 2g/l monosodium phosphate and the required amount of dye dispersion. The samples were dyed with the same temperature profile as used for the application of acid dyes, after which the samples were rinsed and dried.

The various dyed samples were then tested for colorfastness to laundering using AATCC TM 61 1A and 2A conditions. They were also tested for colorfastness to perspiration using AATCC TM 15. The stains on the multifiber strip present in these tests were evaluated using the spectrophotometer. The dyeings which produced stains on one or more fibers on a multi-fiber strip in desired range of 3-4 gray scale reading were selected for use in combination in the next phase.

Phase 2: Selection and application of combination of dyes

Combinations included one of each class of dyes selected in the previous stage. The dyes in the combinations were selected so that the dyes from each class were of different hues. The depth of shade of these dyes was selected based on Phase 1 results. These combinations were applied to the 50-50 nylon-cotton (ny-co) blend. Two additional combinations were dyed using depths of shade (0.3%owf) that were not included in the first phase. Table 2 gives two series of combinations, the first containing Direct Red 80, Disperse Blue 56, and Acid Orange 116, and the second with Direct Blue 80, Disperse Red 60, and Acid Red 299, along with their commercial names and the depths of shades applied:

Table 2: Combinations of dyes

Combination	Dyes	Commercial Names	Shade %
1A	C.I. Direct Red 80	Pyrazol Fast Red 7BSW	0.2
	C.I. Disperse Blue 56	Dispersol Blue B-R	0.2
	C.I. Acid Orange 116	Nylosan Orange SL	0.2
1B	C.I. Direct Red 80	Pyrazol Fast Red 7BSW	0.1
	C.I. Disperse Blue 56	Dispersol Blue B-R	0.2
	C.I. Acid Orange 116	Nylosan Orange SL	0.2
1C	C.I. Direct Red 80	Pyrazol Fast Red 7BSW	0.2
	C.I. Disperse Blue 56	Dispersol Blue B-R	0.2
	C.I. Acid Orange 116	Nylosan Orange SL	0.5
1D	C.I. Direct Red 80	Pyrazol Fast Red 7BSW	0.1
	C.I. Disperse Blue 56	Dispersol Blue B-R	0.2
	C.I. Acid Orange 116	Nylosan Orange SL	0.5
2A	C.I. Direct Blue 80	Pyrazol Fast Blue BGUL	0.2
	C.I. Disperse Red 60	Resolin Red FB	0.2
	C.I. Acid Red 299	Nylosan Rubine S5B	0.2
2B	C.I. Direct Blue 80	Pyrazol Fast Blue BGUL	0.1
	C.I. Disperse Red 60	Resolin Red FB	0.2
	C.I. Acid Red 299	Nylosan Rubine S5B	0.5
2C	C.I. Direct Blue 80	Pyrazol Fast Blue BGUL	0.2
	C.I. Disperse Red 60	Resolin Red FB	0.1
	C.I. Acid Red 299	Nylosan Rubine S5B	0.2
2D	C.I. Direct Blue 80	Pyrazol Fast Blue BGUL	0.2
	C.I. Disperse Red 60	Resolin Red FB	0.5

	C.I. Acid Red 299	Nylosan Rubine S5B	0.2
2E	C.I. Direct Blue 80	Pyrazol Fast Blue BGUL	0.2
	C.I. Disperse Red 60	Resolin Red FB	0.3
	C.I. Acid Red 299	Nylosan Rubine S5B	0.2
2F	C.I. Direct Blue 80	Pyrazol Fast Blue BGUL	0.1
	C.I. Disperse Red 60	Resolin Red FB	0.2
	C.I. Acid Red 299	Nylosan Rubine S5B	0.3

The three-dye combinations were applied to the 5 grams of 50- 50 nylon-cotton in a single dye bath using batch dyeing. The required amount of each dye solution was measured into a dyebath prepared with 1g/l StarLev WWB, 1g/l monosodium phosphate, 5% owf ammonium sulfate, and 10% owf sodium chloride. The pre-wetted fabric was added and dyed using the procedure used for disperse dyes in phase 1.

The dyed samples were then tested for colorfastness to laundering and perspiration using AATCC TM 61 (1A and 2A), and AATCC TM 15 and data for staining and color change was collected using a spectrophotometer. The data was used to select one or more suitable combination with wide range staining on two or more multi-fiber components with two mid-range staining for further testing.

Phase 3: Effect of varying fastness test conditions

New batches were dyed for selected combinations 2D, 2E and 2F to make more samples for further testing for AATCC TM 61 1A and 2A. The combinations were tested through standard conditions again in this stage to confirm the stability of the results derived from combinations selected. The variables involved in 1A and 2A conditions were switched one at a time as the following table describes.

Table 3: AATCC TM 61 1A and 2A– Standard and Altered Variables

Variables	AATCC TM 61 1A		AATCC TM 61 2A	
	Standard	Altered	Standard	Altered
Temperature (˚F)	105	120	120	105
Liquor Volume (mL)	200	150	150	200
% powder detergent of total volume	0.37	0.15	0.15	0.37
Number of steel balls	10	50	50	10
Time (+10 minutes)	45	55	45	55
Time (-10 minutes)	45	35	45	35
Sample size (inches)	2X4	2X6	2X6	2X4

The change of color of the test specimen and the stain on the multifiber was measured in each case.

CHAPTER 4

RESULTS AND DISCUSSIONS

Results of Phase 1

The gray scale readings (staining and color change denoted as "CC") for each of the dyes applied in Phase 1 collected using spectrophotometer for AATCC TM 61 1A, AATCC TM 61 2A and AATCC TM 15 are listed in Table 4, 5, and 6 respectively.

Table 4: Gray Scale Readings for AATCC TM61 1A

Dye	Fiber	Shade	Staining						CC
			Acetate	Cotton	Nylon	Polyester	Acrylic	Wool	
Acid Orange 116	Nylon	0.1%	5	4.5	5	5	5	4.5	5
		0.2%	5	4.5	5	5	5	4.5	4.5
		0.5%	5	4.5	5	5	5	4.5	4.5
		1%	5	4.5	4.5	5	5	4.5	4.5
		2%	5	5	4	5	5	4.5	4.5
		4%	5	4.5	3.5	5	5	4.5	4.5
Acid Red 299	Nylon	0.1%	5	5	5	5	5	4.5	4.5
		0.2%	5	5	5	5	5	4.5	4
		0.5%	5	4.5	4.5	5	5	5	4.5
		1%	5	4	4	5	5	4.5	5
		2%	4.5	3	3	5	4.5	4.5	3
		4%	4.5	2.5	2	4.5	4	4.5	5
Disperse Blue 56	Nylon	0.1%	5	4.5	5	5	5	4.5	4
		0.2%	4.5	4.5	4.5	4.5	5	4.5	4
		0.5%	4	4.5	4	4.5	5	4.5	4
		1%	4.5	4.5	4	4.5	5	4.5	4.5
		2%	3.5	4.5	3	4	4.5	4.5	4.5
		4%	3.5	4.5	3	3.5	4.5	4	4.5
Disperse Red 60	Nylon	0.1%	4.5	4.5	4.5	5	5	4.5	4
		0.2%	4.5	4.5	4.5	5	5	4.5	4
		0.5%	3.5	4.5	3	4.5	5	4.5	4

		1%	3	4.5	2.5	4.5	5	4.5	4.5
		2%	3.5	4.5	3	4.5	5	4	4
		4%	3	4.5	2.5	4.5	5	4	4.5
Direct Blue 80	Cotton	0.1%	5	4.5	5	5	5	4.5	3
		0.2%	5	4.5	5	5	5	4.5	3.5
		0.5%	5	3	5	5	5	4.5	2
		1%	5	2.5	5	5	5	4.5	2.5
		2%	5	2.5	5	5	5	4.5	2.5
		3%	5	2	5	5	5	4.5	2
		4%	5	2	5	5	5	4.5	2
Direct Red 80	Cotton	0.1%	5	4	5	5	5	4.5	4
		0.2%	5	3.5	5	5	5	4.5	3.5
		0.5%	5	3	4.5	5	5	4.5	2.5
		1%	5	2	5	5	5	4.5	2.5
		2%	5	1.5	4.5	5	4.5	4.5	3
		3%	5	1.5	5	5	5	4.5	3
		4%	5	1.5	4.5	5	4.5	4.5	2.5

Acid dyes yielded mid-range staining on mostly nylon and cotton fibers in shades 1%, 2%, and 4%. The disperse dyes exhibited mid-range stains on acetate, nylon, and wool in shades 0.5% and higher. Direct dyes produced stains mostly on cotton and wool: 3-4 gray scale reading were achieved mainly with lower shades of 0.1%, 0.2%, and 0.5%. Acid and disperse dyes, with color change readings varying from 5 to 4, did not fade as much the direct dyes for which the color change rating was from 4 to as low as 2.

Table 5: Gray Scale Readings for AATCC TM61 2A

Dye	Fiber	Shade	Staining						CC
			Acetate	Cotton	Nylon	Polyester	Acrylic	Wool	
Acid Orange 116	Nylon	0.1%	4.5	5	4.5	5	5	4.5	4.5
		0.2%	4.5	5	4.5	5	5	4.5	5
		0.5%	4	4.5	3.5	4.5	5	4.5	5
		1%	3.5	4	2.5	4.5	4.5	4.5	4.5
		2%	2.5	3	2	4	4.5	4.5	4.5
		4%	2	2.5	1	4	4.5	3.5	4.5

Acid Red 299	Nylon	0.1%	5	4.5	4.5	5	5	4.5	4.5
		0.2%	5	4	4	5	5	4.5	4
		0.5%	5	3	2.5	5	5	4.5	4
		1%	4.5	2	2	5	5	3.5	5
		2%	3.5	1.5	1.5	4.5	4.5	2.5	3
		4%	2.5	1	1	3.5	4	1.5	4.5
Disperse Blue 56	Nylon	0.1%	4.5	4.5	4	4.5	5	4.5	4.5
		0.2%	4	4.5	3.5	4.5	5	4.5	4.5
		0.5%	3	4	2	3	4.5	3.5	4.5
		1%	2.5	3.5	2	2.5	4	3.5	4.5
		2%	2.5	3.5	2	2.5	4	3	4.5
		4%	2.5	3.5	2	2.5	4	3	4.5
Disperse Red 60	Nylon	0.1%	3.5	4.5	3	4.5	5	4.5	4
		0.2%	3	4.5	2.5	4	4.5	4	4
		0.5%	2	4	1.5	3	4.5	3	4
		1%	2	4	1.5	2.5	4.5	3	4.5
		2%	2	4	1.5	3	4.5	3	4.5
		4%	2	4	1.5	3	4.5	3	4.5
Direct Blue 80	Cotton	0.1%	5	3	5	5	5	4.5	3.5
		0.2%	5	3	5	5	5	4.5	3
		0.5%	5	2	4.5	5	5	4.5	1.5
		1%	5	2	5	5	5	4.5	2
		2%	5	1.5	5	5	5	4.5	2
		3%	5	1.5	4.5	5	5	4.5	2
		4%	5	1.5	5	5	5	4.5	1.5
Direct Red 80	Cotton	0.1%	5	3	5	5	5	4.5	3.5
		0.2%	5	3	5	5	5	4.5	3
		0.5%	5	2	4.5	5	5	4.5	4
		1%	5	2	4.5	5	5	4.5	3
		2%	5	1.5	4.5	5	5	4.5	3
		3%	5	1.5	4.5	5	5	4.5	2.5
		4%	5	1.5	4.5	5	5	4.5	2

The more severe test conditions of AATCC TM61 2A resulted in heavier stains than were found from AATCC TM61 1A. The required staining range occurred in three lower shades in case of disperse dyes on all fibers except acrylic, with one or two readings below the mid-range staining. Direct dyes drew mid-range staining mainly on cotton in shades 0.1% and 0.2%. The acid dyes performed differently: C.I. Acid Orange 116 in shades 1% and up resulted in stains mainly on acetate, cotton, and nylon ranging from as low as 1 to 3.5 gray scale reading. C.I. Acid Red 299 at 0.2% and 0.5%owf produced a desirable range of staining, with darker shades producing heavier stains on one two or more fibers. Acid and disperse dye did not show severe color change (a minimum reading of 4) while direct dyes exhibited poor fastness regarding color change as they did in AATCC TM61 1A.

Table 6: Gray Scale Readings for AATCC TM15

Dye	Fiber	Shade	Staining						CC
			Acetate	Cotton	Nylon	Polyester	Acrylic	Wool	
Acid Orange 116	Nylon	0.1%	5	5	5	5	4.5	5	4.5
		0.2%	5	5	5	5	4	5	4.5
		0.5%	5	4.5	5	5	4	5	4.5
		1%	5	4.5	5	5	4	5	4.5
		2%	5	3.5	3.5	5	3.5	5	4.5
		4%	4.5	2.5	2	5	3	5	4.5
Acid Red 299	Nylon	0.1%	5	5	5	5	5	5	4.5
		0.2%	5	5	5	5	5	5	4
		0.5%	5	5	5	5	5	5	4.5
		1%	5	4.5	4.5	5	5	5	5
		2%	4	3	3	4.5	4	4.5	4.5
		4%	2.5	1.5	2	4	4.5	2.5	5
Disperse Blue 56	Nylon	0.1%	5	5	5	4.5	4.5	5	4.5
		0.2%	5	4.5	5	4.5	4	5	4.5
		0.5%	4	4	4	5	4	4.5	4.5
		1%	3	4	3	3.5	4	4	4.5
		2%	3.5	4	3.5	3.5	4	5	4.5
		4%	4	3.5	4	3.5	4	4	4.5
Disperse Red 60	Nylon	0.1%	5	5	5	5	5	5	4
		0.2%	4	5	5	5	5	5	4
		0.5%	4	5	4	5	5	5	4

		1%	4	5	4	5	5	5	4.5
		2%	5	5	4.5	5	5	5	4.5
		4%	4	5	4	4.5	5	5	5
Direct Blue 80	Cotton	0.1%	5	3	5	5	4	4	4.5
		0.2%	5	3	4	5	4	4	4.5
		0.5%	5	1.5	3	5	4	3.5	4
		1%	5	2	2.5	4	4	3	3.5
		2%	5	1	2.5	4	3.5	1.5	4
		3%	5	0	2.5	4	3	1.5	4
		4%	5	0	2.5	4	3.5	1.5	3
Direct Red 80	Cotton	0.1%	5	3.5	5	5	4.5	4.5	3
		0.2%	5	2.5	4	5	4.5	5	2.5
		0.5%	5	1.5	3.5	4	4	3.5	2
		1%	5	1.5	4	4.5	4	3	2.5
		2%	5	0	2.5	4	4	3	2.5
		3%	5	0	2.5	4	3.5	2	2.5
		4%	4.5	0	2	3.5	3	2	2.5

In AATCC TM15 (Colorfastness to Perspiration) the disperse dyes on nylon displayed mid-range staining on two or more fibers, notably in the case of Disperse Blue 56 in shades 0.5% owf and higher. Disperse Red 60 at 0.2% owf and higher stained acetate and nylon in the desired mid-range. Both direct dyes at 0.5% owf and higher stained the cotton on the multi-fiber strip severely, with gray scale ratings ranging from 2 to 0. However, lower shades resulted in a range of staining on all fibers except acetate and polyester which had no staining. All shades except 0.1% of Acid Orange 116 yielded stains primarily on acrylic and; also, resulted in staining on cotton and nylon in case of higher shades of 2% and 4%. Acid Red 299 demonstrated good fastness to perspiration and only stained at higher concentrations of 2% and 4%. The color change readings resembled the previous two tests results; only direct dyes exhibited evident color change ranging from 4.5 to 2 gray scale reading.

Results of Phase 2

Phase 1 showed that shades of 0.1%, 0.2%, and 0.5%owf of all six dyes were suitable for testing in combination to produce candidate verification standards. Ten combinations planned with selected dyes in varied shades listed in Table 2 were planned so that each contained all three dye types, the hues of which were different. The results of the colorfastness tests (AATCC TM 61 1A, AATCC TM 61 2A, and AATCC TM 15) on the nylon-cotton samples dyed with the planned combinations are listed in Tables 7, 8 and 9 respectively.

Table 7. Colorfastness of Combinations to AATCC TM 61 1A

Comb.	Staining						CC
	Acetate	Cotton	Nylon	Polyester	Acrylic	Wool	
1A	4.5	3.5	4	5	5	4.5	3
1B	4.5	3	4	5	5	4.5	2.5
1C	4.5	3.5	4	5	5	4.5	2.5
1D	4.5	3	4	5	5	4.5	2.5
2A	4	3.5	3.5	4.5	5	4.5	2.5
2B	4	3.5	3	4.5	5	4.5	2.5
2C	4.5	3.5	4	4.5	5	5	2.5
2D	3	3	2.5	4	4.5	4	2.5
2E	3.5	3.5	3	4.5	5	4.5	3
2F	4.5	3.5	3.5	4.5	5	4.5	3

The four combinations containing Direct Red 80, Disperse Blue 56, and Acid Orange 116 (together listed as “Series 1”) performed quite similarly in AATCC TM61 1A, with mid-ranged gray scale staining on cotton and nylon and no or little staining on rest of the fibers on the multi-fiber strip. The combinations of Direct Blue 80, Disperse Red 60, and Acid Red 299 (“Series 2”) elicited a wider range of staining than Series 1 combinations, with stains mostly on acetate, cotton, and nylon. Combination 2D specifically stained all five fibers of the multi-fiber strip; the staining rating ranged from 2.5 to 4.5. Combinations 2E and 2F produced staining similar to that of 2A and 2B. The staining of combination 2C did not offer a wide range of gray scale reading for staining.

Table 8. Colorfastness of Combinations to AATCC TM 61 2A

Comb.		Staining					CC
	Acetate	Cotton	Nylon	Polyester	Acrylic	Wool	
1A	4.5	3	3.5	4.5	4.5	4.5	3.5
1B	4.5	3.5	3.5	4.5	4.5	4.5	3
1C	4.5	3	3	4.5	4.5	4.5	3
1D	4.5	3.5	3	4.5	4.5	4.5	3.5
2A	4.5	4	3	4.5	4.5	3	4.5
2B	3	3	2	4	4.5	4.5	4.5
2C	4	3	3	4.5	4.5	4	4.5
2D	2	3	1.5	3	4	3	3.5
2E	2.5	2.5	2	3.5	4.5	3.5	4
2F	3	3	2	4	4.5	4	4.5

As in the 1A tests, the combinations performed very similarly in 2A test, with Series 2 combinations exhibiting a wider range of staining than Series 1. Specifically, combinations 2D, 2E, and 2F produced stains with gray scale ratings that ranged over lower, mid and higher gray scale values.

The staining and color change ratings from the AATCC TM 15 test of the combinations is presented in Table 9. Neither the first or second series of combinations produced a range of staining on fibers. Only combinations 1C, 2A, 2D and 2E generated stains of 4 to 4.5 gray scale rating on two to three fibers. Although mid- range staining was achieved by the combinations mentioned above, the staining achieved did not provided sufficient range of staining which might provide variation if the test conditions are altered.

Table 9. Colorfastness of Combinations to AATCC TM 15

Comb.		Staining					CC
	Acetate	Cotton	Nylon	Polyester	Acrylic	Wool	
1A	5	4	5	5	5	5	3.5
1B	5	5	5	5	5	5	4
1C	5	4	5	5	4	5	4.5
1D	5	4.5	5	5	4	5	4
2A	4.5	4	5	5	5	4	4
2B	5	4.5	5	5	5	5	4
2C	5	4.5	5	5	5	5	4
2D	4	4	4.5	5	5	4	4
2E	4	4	4	5	5	4	4.5
2F	5	5	5	5	5	4.5	4.5

The color change of the original specimen in all three test results (Table7, 8 and 9), were inconsistent, with results varying by 0.5 to 1 points on the gray scale. The data presented are a mean of 3 gray scale readings irrespective of face and back of the fabric. AATCC TM 1A resulted in a greater color change in dyed fabric than AATCC TM 2A, in contrast to the staining found in the same tests. The color change in AATCC TM 15 was ranging between mid to higher gray scale rating.

Results of Phase 3

The results from Phase 2 indicated that combinations 2D, 2E, and 2F provided a wide range of staining in tests AATCC TM 61 1A, and 2A, but no combination in AATCC TM 15 produced diverse stain ratings. It was determined that further testing would be continued with only AATCC TM 61 1A and 2A.

Gray scale ratings only provide discrete whole-number or half-step values. To get a better impression of changes in staining caused by variations in variables in the tests, ΔE^* and ΔL^* values were recorded which were further converted to determine ΔEGS values ("EGS"). Also, to get a better understanding of color change under standard and altered test variables, gray scale readings for color change of both the face and back of the fabric were recorded.

Tables 10 through 15 display results collected from standard test conditions, and seven altered test conditions of AATCC TM 61 1A for staining and color change values of combinations 2D, 2E, and 2F.

Table 10. Staining with standard and altered test conditions of Combination 2D to AATCC TM 61 1A.

S. No	Staining											
	Acetate		Cotton		Nylon		Polyester		Acrylic		Wool	
	GS	EGS	GS	EGS	GS	EGS	GS	EGS	GS	EGS	GS	EGS
Std	3	8.00	3	7.18	2.5	11.87	4.5	2.32	4.5	1.36	4	3.36
I	2.5	11.13	3	7.74	2	15.37	3.5	5.21	4.5	1.68	3.5	5.74
II	3	9.15	3	7.27	2.5	11.38	4.5	2.71	4.5	2.58	4	4.05
III	3.5	6.59	3.5	6.34	2.5	11.09	4.5	2.77	4.5	1.15	4	3.80
IV	3	7.36	3	7.38	2.5	11.69	4.5	2.86	4.5	1.43	4	3.52
V	3.5	5.83	3.5	6.25	3	9.95	4.5	2.05	4.5	1.14	4.5	2.85
VI	4	4.90	3.5	5.71	3	8.24	4.5	1.94	4.5	1.11	4.5	2.12
VII	3	8.94	3	8.24	2.5	13.30	4.5	3.04	4.5	1.46	4	4.66

Table 11. Color change with standard and altered test conditions of Combination 2D to AATCC TM 61 1A.

S. No	Color Change	
	Face	Back
Std	3.5	3.5
I	3	3
II	3.5	4
III	4	4
IV	3.5	4
V	3.5	4
VI	3	3
VII	3	4

Table 12. Staining with standard and altered test conditions of Combination 2E to AATCC TM 61 1A.

S. No	Staining											
	Acetate		Cotton		Nylon		Polyester		Acrylic		Wool	
	GS	EGS	GS	EGS	GS	EGS	GS	EGS	GS	EGS	GS	EGS
Std	3.5	5.27	3.5	6.22	3	7.61	4.5	1.91	4.5	1.34	4.5	1.96
I	3	7.76	3	7.62	2.5	10.87	4.5	2.41	4.5	1.23	4	1.23
II	3.5	5.30	3.5	6.81	3.5	6.90	4.5	1.94	4.5	1.19	4.5	2.18
III	4	3.56	4	5.64	3.5	5.74	4.5	2.16	4.5	1.18	4.5	1.38
IV	4	4.77	3.5	6.68	3	7.51	4.5	2.24	4.5	1.23	4.5	1.52
V	4	4.52	3.5	6.45	3	7.22	4.5	2.04	4.5	1.09	4.5	1.61
VI	4	3.53	4	5.51	3.5	5.53	4.5	1.38	4.5	1.14	4.5	1.13
VII	3.5	6.29	3.5	7.55	3	8.22	4.5	2.32	4.5	1.50	4.5	2.55

Table 13. Color change with standard and altered test conditions of Combination 2E to AATCC TM 61 1A.

S. No	Color Change	
	Face	Back
Std	4.5	3.5
I	4	2.5
II	3.5	3
III	3	3.5
IV	3	3.5
V	3	2.5
VI	4	3.5
VII	3	2.5

Table 14. Staining with standard and altered test conditions of Combination 2F to AATCC TM 61 1A.

S. No	Staining											
	Acetate		Cotton		Nylon		Polyester		Acrylic		Wool	
	GS	EGS	GS	EGS	GS	EGS	GS	EGS	GS	EGS	GS	EGS
Std	4.5	3.07	4	5.07	4	4.95	4.5	1.54	4.5	1.20	5	0.85
I	4	4.67	3.5	6.07	3	7.60	4.5	2.34	4.5	1.24	4.5	1.70
II	4	3.53	3.5	6.20	3.5	6.05	4.5	1.75	4.5	1.25	4.5	1.56
III	4.5	3.16	4	5.10	3.5	5.69	4.5	1.61	4.5	1.19	4.5	1.23
IV	4	3.42	4	5.38	3.5	5.44	4.5	1.55	4.5	1.23	5	0.94
V	4	3.58	4	5.34	3.5	5.22	4.5	1.40	4.5	1.17	5	1.08
VI	4.5	2.36	4	5.02	4	3.88	4.5	1.23	4.5	1.13	5	0.82
VII	4	4.49	4	5.89	3.5	6.80	4.5	1.51	4.5	1.32	4.5	1.47

Table 15. Color change with standard and altered test conditions of Combination 2F to AATCC TM 61 1A.

S. No	Color Change	
	Face	Back
Std	2.5	2
I	2.5	2
II	3.5	2
III	3	3
IV	3	2.5
V	3.5	3.5
VI	4	4
VII	3	2.5

When care was taken to differentiate the measurement of front and back of sample the color change values for front and back gave more stable results than in Phase 2, but variations of 0.5 to 1 gray scale were still noted within the three readings recorded for individual samples. Overall results for color change did not show a clear relation between the change in gray scale reading and variation in a test condition.

The combination 2E presented the widest change of result when the conditions of the test were altered. It was also noticed that the Gray Scale staining values for cotton, nylon, acrylic and wool under standard test condition varied from results in Phase 2. Similarly, the difference in staining of acrylic and polyester caused under standard test conditions by combination 2E and 2D respectively differ from staining observed in Phase 2. This variation is explained by "borderline" staining reading where a slight change in staining (and in ΔEGS) results in a change in step-wise gray scale reading. The gray scale readings for staining recorded under altered variables for combination 2D showed more variations compared to 2E from that of the readings recorded under standard conditions. Additionally, combination 2D shows more clear difference regarding ΔEGS values with any change in a variable than that recorded for combination 2E. Although there is no change recorded for the 2D combination in altered conditions II, IV and VII, ΔEGS values show a definite increase in staining for condition II and VII on most of the fibers.

The test staining and color change results from AATCC TM 61 2A collected for 2D, 2E and 2F dye combinations for standard and varied test conditions are shown in Tables 16 through 21.

Table 16. Staining with standard and altered test conditions of Combination 2D to AATCC TM 61 2A.

S. No	Staining											
	Acetate		Cotton		Nylon		Polyester		Acrylic		Wool	
	GS	EGS	GS	EGS	GS	EGS	GS	EGS	GS	EGS	GS	EGS
Std	2	14.42	3	11.24	1.5	20.90	3	8.62	4.5	2.78	3	7.83
I	3	9.86	3.5	8.40	2	15.08	4	4.93	4.5	1.73	3.5	5.32
II	2.5	13.03	3	10.25	1.5	21.39	3.5	6.74	4.5	2.09	3	7.63
III	2	15.59	3	11.16	1.5	20.87	3.5	6.70	4.5	2.69	3	8.27
IV	2.5	14.21	3	9.80	2	19.38	3.5	5.15	4.5	2.57	3	7.23
V	2	16.72	2.5	11.87	1.5	22.23	3	8.92	4	3.29	3	8.08
VI	2.5	12.67	3	9.88	2	18.76	3	7.34	4.5	2.88	3.5	7.04
VII	3	9.71	3.5	8.27	2	14.92	4	4.40	4.5	1.86	3.5	5.59

Table 17. Color change with standard and altered test conditions of Combination 2D to AATCC TM 61 2A.

S. No	Color Change	
	Face	Back
Std	4	3.5
I	4	3
II	3.5	3.5
III	3.5	2.5
IV	3	3.5
V	3	3
VI	3	3.5
VII	2.5	2.5

Table 18. Staining with standard and altered test conditions of Combination 2E to AATCC TM 61 2A.

S. No	Staining											
	Acetate		Cotton		Nylon		Polyester		Acrylic		Wool	
	GS	EGS	GS	EGS	GS	EGS	GS	EGS	GS	EGS	GS	EGS
Std	2.5	11.33	3	10.21	2	18.28	3.5	6.63	4.5	1.68	3.5	6.14
I	3	9.06	3	9.09	2.5	13.10	4	4.64	4.5	2.21	4	4.50
II	3	8.51	3	9.18	2	14.26	4	4.71	4.5	1.54	4	4.13
III	2	14.65	2.5	12.58	2	18.42	3.5	5.37	4.5	2.67	3.5	7.00
IV	2.5	11.37	3	10.67	2	17.08	3.5	5.06	4.5	2.30	3.5	5.79
V	2.5	11.20	3	10.80	2	16.34	3.5	5.68	4.5	2.05	3.5	5.17
VI	2.5	10.30	3	10.07	2	16.16	3.5	5.62	4.5	2.67	3.5	5.62
VII	3.5	6.98	3.5	9.29	2.5	12.50	4	4.19	4.5	2.04	4	4.33

Table 19. Color change with standard and altered test conditions of Combination 2E to AATCC TM 61 2A.

S. No	Color Change	
	Face	Back
Std	4.5	3.5
I	3.5	3.5
II	3	2.5
III	4.5	2.5
IV	4.5	2.5
V	3.5	2.5
VI	4.5	3.5
VII	3.5	3

Table 20. Staining with standard and altered test conditions of Combination 2F to AATCC TM 61 2A.

S. No	Staining											
	Acetate		Cotton		Nylon		Polyester		Acrylic		Wool	
	GS	EGS	GS	EGS	GS	EGS	GS	EGS	GS	EGS	GS	EGS
Std	3	9.17	2.5	11.69	2	16.42	3.5	5.44	4.5	1.79	4	5.16
I	3	5.73	3	7.40	2.5	10.36	4	2.74	4.5	1.33	4.5	2.89
II	3	7.85	3	9.17	2	15.19	4	3.33	4.5	1.50	4	4.82
III	2	10.26	3	9.48	2	14.07	4	3.80	4.5	1.88	4	4.89
IV	2.5	7.31	3	8.59	2.5	13.96	4	3.65	4.5	1.42	4	4.30
V	2.5	10.76	3	9.89	2	15.64	3.5	5.12	4.5	1.94	4	4.77
VI	2.5	9.67	3	9.65	2	16.03	3.5	5.09	4.5	2.16	4	5.51
VII	3.5	8.31	3	9.40	2.5	13.74	4	3.23	4.5	2.02	4	4.54

Table 21. Color change with standard and altered test conditions of Combination 2F to AATCC TM 61 2A.

S. No	Color Change	
	Face	Back
Std	4.5	3.5
I	4.5	3.5
II	3	2.5
III	4.5	3
IV	4.5	2.5
V	3.5	2.5
VI	4.5	3.5
VII	3.5	3

Modifications in the test condition produced changes in staining readings compared to the readings collected under standard conditions. The staining rating of acrylic in the third phase was 4.5, versus 4 in Phase 2. Combinations 2E and 2F also showed variation in one and two fibers staining respectively. As explained above, it is clear from the ΔEGS values that this variation derives from stains that are borderline between two gray scale points.

The greatest variations in staining were found in combinations 2D and 2F. Combination 2F displayed most variation in the stains with a change in test conditions on cotton and polyester, the staining readings for cotton and polyester collected under standard test conditions vary from the readings observed in Phase 2. Hence, the fluctuations caused are questionable. The fluctuation in staining caused by variation of test conditions for combination 2D occurs in several fibers of the multifiber strip. Additionally, combination 2D shows a clearer increase or decrease in ΔEGS values than was found from the 2E dye combination.

The findings from Phase 3 shows that the combination 2D performed as a verification fabric is expected. Whereas not all the modifications in variables result in a change in gray scale reading, but in the test AATCC TM 61 1A, the most of the ΔEGS values increased in the case of I, II, IV, and VII. While for the rest of the conditions the ΔEGS values clearly decreased. Similarly, when tested for AATCC 61 2A, there was an increase in ΔEGS values compared to the standard value for V, and the values decreased for rest of the conditions. Test condition III did not show a clear decrease or increase overall, but fluctuations were noticed from that of the standard readings. While combination 2D displayed a clear relation between fluctuations and variations in testing conditions, combination 2E failed to exhibit such relation for conditions II and IV for AATCC TM 61 1A and conditions IV and V for AATCC TM 61 2A. On the other hand, a clear relation was noted in the case of combination 2E, but as discussed in results of Phase 2 with borderline staining these variations are questionable.

CHAPTER 5

CONCLUSION

This study represents initial research into the development of a dyed verification standard for AATCC Test Method 61, 1A and 2A and Test Method 15. A preliminary study of dyes was carried out to select dye classes that offer medium to poor fastness to laundering and perspiration. Considering the criteria discussed, the study produced acid, direct and disperse dyes as candidates for the development of dyed verification fabric. The selection of dyes was further narrowed down to C.I. Acid Orange 116, C.I. Acid Red 299, C.I. Disperse Blue 56, C.I. Disperse Red 60, C.I. Direct Blue 80, and C.I. Direct Red 80 based on the continuous availability of the dyes. A consideration of fibers suggested that cotton and nylon would be appropriate substrates for application of the selected dye class.

In the first phase, the dyes were applied separately to cotton and nylon fabrics at a range of concentrations. The fastness of those individual dyeings was tested. Those results identified appropriate concentrations of the dyes for subsequent phases of the work.

In the second phase, these concentrations were applied to a nylon-cotton blend fabric, and the fastness of the resulting dyeings to the three tests was assessed. Three combinations, 2D, 2E, and 2F, provided a wide range of staining in AATCC TM 61 1A and 2A, while none did in AATCC TM 15. Further research hence was determined to be conducted on 2D, 2E and 2F combinations for AATCC TM 61 1A and 2A tests.

In the third phase, the three selected combinations were subject to variations of temperature, liquor volume, percentage powder detergent of total volume, the number of steel balls, time and sample size in the test method. Combination 2D comprising Direct Blue 80 (0.2% owf), Disperse Red 60 (0.5% owf) and Acid Red 299 (0.2% owf) produced staining results that displayed most fluctuations from those of the standard conditions as test conditions were changed ΔE_{GS} values changed clearly when each of the variables was altered. This combination is thus suggested as a suitable verification fabric for TM 61 in its staining of multifiber adjacent materials.

The data collected for color change of the test specimen in TM 61through 2 were inconsistent. Similarly, in Phase 3, the gray scale rating for color change did not show an apparent increase or decrease with the change in test conditions. Further work is suggested to determine the reasons for the inconsistency before color change can be a factor in the use of this dye combination on a verification fabric. None of the combinations gave a broad range of stains when subjected to AATCC TM 15. As suggested by the results from Phase 2, the test AATCC TM 15 would require a separate dyed verification standard.

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