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# The Future of Dye House Quality Control with the Introduction of Right-First Dyeing Technologies

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## 1. Introduction

The manufacturing of a textile begins with the fiber input, whereby each processing step results in an added cost to the final product. As dyeing of a textile is often the last step in the manufacturing of a fabric, it requires extra caution to get it right by avoiding waste and maintaining cost control. Only under favourable conditions is it possible to get it right the first time. In the past, it was not unusual for a dyer to re-dye until the target shade was reached. A typical strategy was to start with a base recipe that undershot the target shade. After each dyeing the missing dye component was added to the bath until the shade was matched. The smaller the number of reformulation, the more skilful the dyer was considered.

The Right-First-Time (RFT) dyeing concept was introduced in 1970 and became a desired feature of textile dyeing. This concept meant that at each dyeing the target shade was achieved the first time, hence not requiring re-dyeing. However, the successful evolution of the concept depended on work carried out over many years by a relatively small number of organizations. Many application research and development projects were carried out mainly by the laboratory, pilot-plant and bulk-scale equipment of the major users and manufacturers of dyes and equipment, as well as by universities.

Since the end of 20th century, with the increased competition, dye houses are asked to meet more exact requirements while they are under pressure to reduce the cost of manufacturing. In order to stay competitive and be in business, they were required to exercise tighter quality control and seek ways to optimize dyeings. This necessitated the understanding of a) dyes, chemicals and substrates and their compatibilities; and b) the parameters that influence the rate and extent of dye uptake by the substrate (Park & Shore, 2007).

The variables that are objectively measured and monitored during a dyeing for quality control purposes have traditionally been limited to time, temperature, pH, and conductivity. Measurement of the fabric shade reflectance is only utilized in the development of new recipes/procedures and the verification of the dyed fabric shade. The development of recipes/procedures and the debugging of dyeing problems continue to rely on indirect information obtained by ad-hoc trial and errors, subjective observations, and visual assessments of dyers.

The first prototype system (Beck et al., 1990; Keaton & Glover, 1985) to measure the dye build up directly in real-time during a dyeing was developed in 1990's. However, due to technological limitations such as; a) the high cost of spectrophotometers, b) the low computation speed that is not sufficient to process the data generated by the

spectrophotometer, and c) the insufficient computer memory restricting the amount of data that can be manipulated for the accurate determination of dye amounts, the adaptation of these prototype systems from the industry was delayed. For example, to determine the dye concentrations of a 3-dye combination precisely in real time (2+ decimal points), it is obligatory to have a computer with more than 500 MBs of memory. About eight years ago, computers with these configurations became available at low cost.

With advances in computation and electronics by the mid 2000's, the limitations listed above were overcome. As a result, two groups, one in England and the other in the US, began adapting dyebath monitoring systems (Dixon & Farrell, 2009; Ferus-Comelo et al., 2005). These systems became the back-bone of commercial RFT dyeing in select plants. The historical progress of these technologies were discussed in detail by Smith (Smith, 2007) in his acceptance of the AATCC Olney Award at the 2007 AATCC conference.

It was recognized that the successful practical dyer must achieve the following objectives:

- the material must match the target color (usually being color constant or isometric in different illuminants)
- the color must be applied uniformly and the processed material must have satisfactory fastness and physical properties
- the cost of production must be within the allowed budget
- the material must be delivered on time.

Important factors to achieve successful dyeing at RFT include dye standardization, compatibility of dyes and chemicals, catching dyeing problems earlier in the dyeing cycle, reducing or eliminating wasted textile and optimizing dyeing and quantifying end product quality. Early adapters of the RFT technologies reported reduction in time and cost while increasing overall product quality.

Another important aim of dyeing research was the optimization of dyeing recipe and procedures by:

- characterizing the performance of dyes, auxiliaries and substrates by identifying physio-chemical parameters,
- cutting extra time from the procedure,
- using compatible dyes and chemicals,
- avoiding unlevelness by controlling temperature and pH and limiting rate of exhaustion,
- eliminating chemicals that are not necessary,
- evaluating the performance of dyeing equipment and optimizing dyeing machine parameters such as flow rate or circulation time,
- developing procedures that reduce dyeing time with increased rate of exhaustion at under utilized dyeing steps

It is not the purpose of this chapter to discuss optimization strategies, although some of that optimization occurs with the quality control improvements discussed here.

It is perhaps somewhat surprising that no formal research and development program has yet to be undertaken to systematically develop RFT quality control and optimization procedures in practical dyeing processes.

In order to be consistent in communicating the analysis and interpretation of the dye exhaustion data, several new terms were introduced over the past several years by the

industry. With this chapter, the author defines and shares this emerging terminology with the academic researchers to facilitate communication.

New plots that are used regularly in the analysis of dye absorbance / concentration data are also introduced and discussed in this chapter for the first time.

## 2. Typical configuration and the setup of the dyebath monitoring system

A typical dyebath monitoring system such as shown in Figure 1 enables dyers to monitor each dye concentration in the dyebath while measuring the temperature, pH, and conductivity of the dyebath simultaneously.

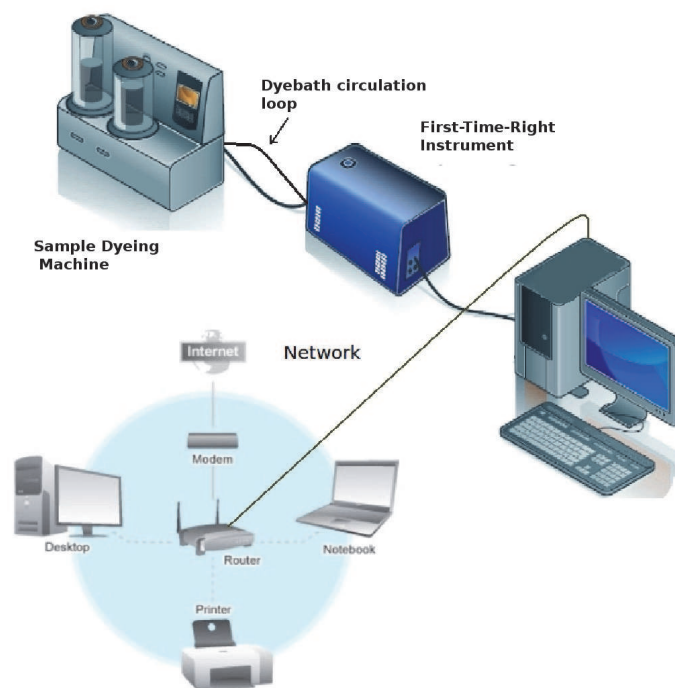


Fig. 1. HueMetrix Dye-It-Right Monitor.

The determination of dye concentrations in real-time works as follows once the dyes are calibrated:

1. Approximately every 90 seconds during the dyeing process, the instrument takes a micro sample of dyebath via a small dyebath circulation loop connected to the dye machine.
2. The sample is conditioned, both physically and chemically for optimum spectral analysis.
3. Each micro sample is read with the state-of-the art spectrophotometer and analyzed.
4. The resulting data set (includes dye concentration, exhaustion, temperature, pH, and conductivity) is recorded by the software.
5. The instruments post-processing software provides certain analysis features to aid dyers in determining dyeing performance.

The system determines individual dye concentrations according to the Beer-Lambert law (McDonald, 1997):

$$A(\lambda) = l\epsilon(\lambda)c \quad (1)$$

and the additivity of individual dye spectrums when they are mixed (Johnson, 1989).

In Equation 1,  $\epsilon$  : molar absorbance [mol/L],  $\lambda$  : wavelength,  $A$  : absorbance [Au], and  $c$  : dye concentration [g/L].

Existing systems (Dixon & Farrell, 2009) are now capable of measuring dye concentrations of almost any dye class, including water insoluble disperse dyes. They can even measure multiple dyes of the same colors.

The determination of dye amounts may be done *manually* or *automatically*.

- Manual mode;** requires the injection of dye solution via syringe from the luer-lock syringe port of the instrument. Following injection, the technician may choose to re-sample or inject a new sample.
- Automatic mode;** is selected during the dyeing process where, every ninety seconds or so, a dyebath sample circulating in the circulation loop connected to the dyebath is automatically taken from the dyebath by the opening and closing of the solenoid valve.

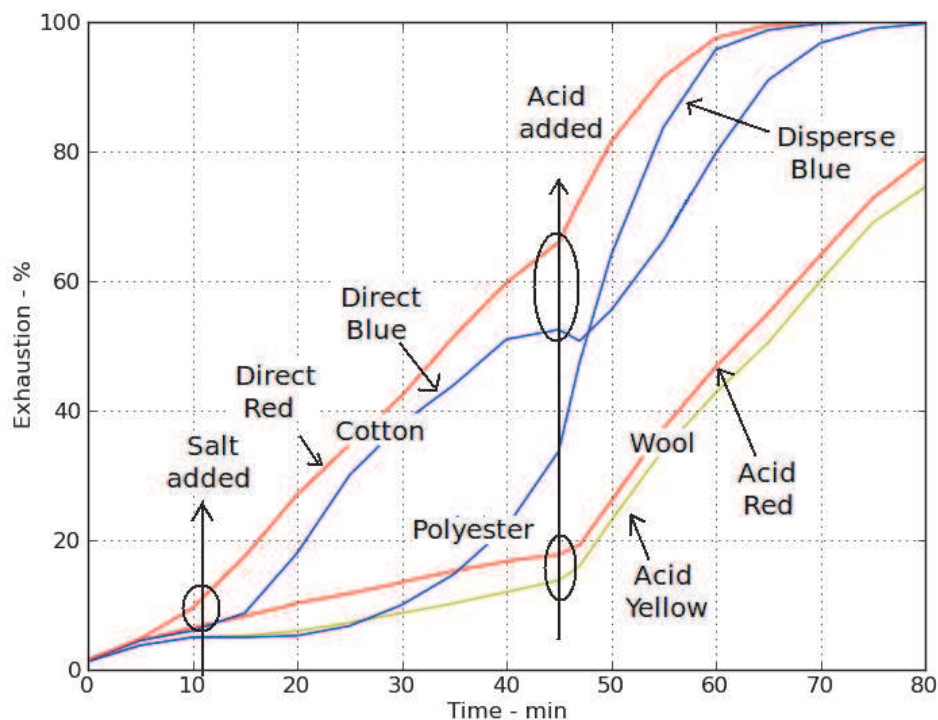


Fig. 2. The monitoring of dye uptake by the fabric for individual dyes during a dyeing cycle.

Regardless of the choice to run in manual or automatic mode, a micro sample is enough to calculate the individual dye amounts in the solution. This data may then be correlated with the temperature, pH, and conductivity sensor measurements if they are attached. The results are then presented to the dyer in real-time at various overlay graphs.

When the dyeing is completed, the technician may save the results in a database. The data stored may then be analyzed by an engineer following the methodology suggested in this paper.

3. Data processing and tools to analyze and interpret real-time dye monitoring data

Establishing a standard for quality attributes against which assessments will be made is critical in dyeing quality control.



The quality attributes that have practical importance are:

- concentration
- exhaustion
- temperature
- pH
- conductivity
- strike rate [maximum rate of exhaustion]
- strike temperature [the temperature at which exhaustion accelerates]

Among these attributes, concentration, temperature, pH, and conductivity are measured directly, and the exhaustion, maximum rate of exhaustion (strike rate) and the temperature at which the exhaustion accelerates (strike temperature) are the derived attributes of those measured.

Figure 2 shows the % exhaustion of red, yellow and blue color dyes on a fabric made of polyester, cotton and wool blended fibers versus time. On this substrate, the acid dyes color the wool, the direct dyes color the cotton, and the disperse dyes color the polyester fibers. The chart plotted in Figure 2 shows that, during the dyeing, the addition of salt triggers the cotton fibers to absorb the direct dyes, the addition of acid triggers wool fibers to absorb acid dyes, and the rise of temperature eventually starts the dyeing of polyester with heat driven disperse dyeing.

Furthermore, Figure 2 shows that the citric acid also interacts with direct dyes, resulting in the exhaustion of these dyes by the cotton fibers. The capability to capture such detailed information on dyes and their interactions with chemicals, fabric, and process variables enables the dyer to develop the proposed quality control schema below for a dye-house.

In the evaluation of quality attributes one has to be aware of the limitations of the technology which are discussed by Günay and Jasper (Günay & Jasper, 2010). However, note that the effect of these limitations on the conclusions drawn is often insignificant.

In addition, note that using equipment for solution and dispersion preparation in conjunction with the low-cost electronic dispensing pipette for dyebath preparation increases confidence in results and improves repeatability of tests.

Definitions of the critical attributes and their determination methodology are given below:

**Exhaustion:** is the percent amount of dye that has migrated onto the substrate. % Exhaustion is a function of the initial and current concentrations and calculated as follows;

$$\%Exhaustion = 1 - \frac{C(t)}{C(t_s)} * 100$$

**Final exhaustion:** is the value of the exhaustion at the end of a dyeing.

**Strike time -  $t_s$  and temperature -  $T(t_s)$ :** are the time and the corresponding dyeing temperature at which the dye starts to go onto fabric with an increasing rate. Especially in synthetic fibers at glass transition temperature, fiber relaxes and expands and eventually allows dye molecules to penetrate into amorphous regions. Knowing the time and temperature of this point may permit fiber manufacturers to compare different fibers for blending and quality control purposes. It may also provide data on the comparative information on fiber morphologies and crystallinity.

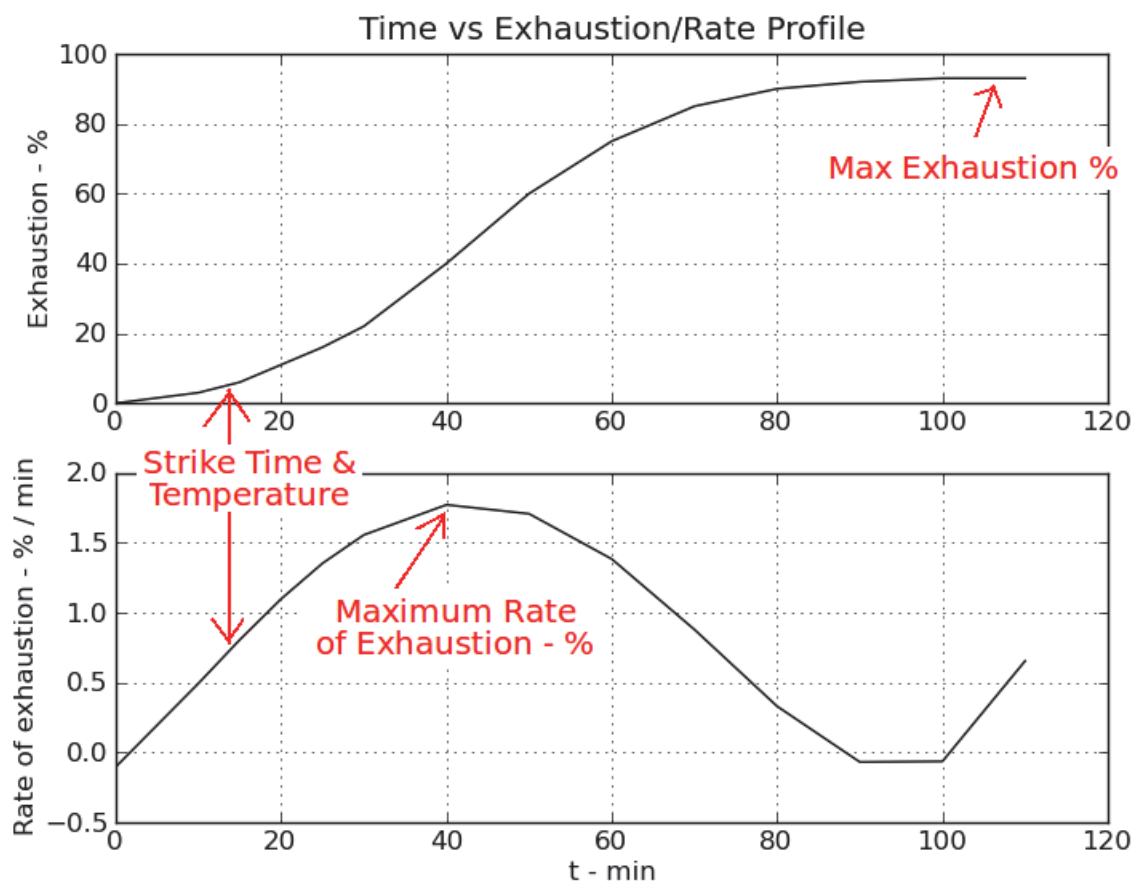


Fig. 3. Display of critical attributes on exhaust profile for a typical dyeing.

**Equilibrium time -  $t_e$ :** is the time when exhaustion of dye slows down and begins to level of as it reaches its ultimate exhaustion,  $E(t_e)$ .

**% Rate of exhaustion -  $\frac{\Delta E}{\Delta t}$ :** is determined by calculating the average exhaustion rate between the strike and equilibrium times as follows:

$$\%Rate \text{ of Exhaustion} = \frac{E(t_e) - E(t_s)}{t_e - t_s}$$

**Maximum exhaustion % rate:** is the value of the maximum rate of % exhaustion. This is a critical value often effects the levelness of the dyeing.

**Temperature for maximum exhaustion % rate:** is the temperature at which the maximum rate of % exhaustion occurs. Near this temperature the exhaustion rate is decreased to prevent unlevelness.

Figure 3 demonstrates the definitions of critical attributes on exhaustion versus time and the rate of exhaustion versus time plots. For this dyeing, the final exhaustion reached was about % 90. The % strike time is around 16<sup>th</sup> minutes.

4. Comprehensive quality control strategy

Several factors which must be monitored or controlled to achieve RFT dyeing were identified with the combined efforts of researchers at universities and the technical staff of dyers and their suppliers (Koksai & Smith, 1995; Park, 1987).

It was concluded that the shade variation in a dye-house may be minimized by a comprehensive quality control strategy that adapts:

- tight dye-house input quality control,
- last checks right prior to a dyeing,
- monitoring of dye uptake during the dyeing, and
- post dyeing quality confirmation.

Of note in this chapter is the author's proposal and discussion of new testing methods emerging with the introduction and advances in right-first-time dyebath monitoring technologies.

#### 4.1 Variables that needs to be monitored for RFT dyeing

The principal factors in obtaining dyeing reproducibility are listed in Table 1.

These factors are grouped and ordered accordingly with respect to the stages of dyeing. A typical dyeing is considered to begin in the laboratory with shade matching and sample dyeing, continues with the final checks before dyeing, and reaches completion with the post dyeing quality confirmation.

The remainder of this chapter is a detailed analysis of tests to monitor and control the factors listed in Table 1. It is vital to apply these tests into routine testing and operating procedures in both in the laboratory and in production.

The failure to monitor these factors, due to fatigue or a lack of standard operating procedure (SOP), prevents dye houses from achieving a high level of right-first-time production.

#### 4.2 Quality control in the laboratory

Even though dye manufacturers provide services for determining a matching recipe for a given shade, these can only be used as a starting point for in-house laboratory work by the dye user. The reason for this is due to the fact that the dyeings carried out in different locations are not comparable unless the same dyes, chemicals, substrate and water supplies have been used (Park & Shore, 2009).

##### 4.2.1 Color matching

Even when visual assessment of color is carried out by standardized methods, the results obtained can be subjective and unreliable, as decisions may be affected by the age, sex, and sensitivity in color discrimination as well as the current physiology of the observer (McLaren, 1970). To make visual assessment of color more consistent, a set of observers may be asked to evaluate samples.

An improvement in the quality of color matching in terms of accuracy, consistency, and reproducibility can only be achieved by the use of instrumental methods based on spectrophotometry (Park & Shore, 2009). Color-difference equations such as  $DE_{CMC(2:1)}$  and  $CIEDE_{2000}$  provide a single number while effectively compensating for the visual non-uniformity of the color space. The numbers obtained from these equations, especially the CMC equation, have been widely adopted in setting tolerance limits for color-differences between the reference and manufactured fabrics.

Many dye houses rely on external color matching and recipe formulation laboratories. However, quick response, just-in-time, and right-first-time dyeing can only be achieved by a basic in house dye house laboratory.



In the laboratory	<ul style="list-style-type: none"><li>• Color matching<ul style="list-style-type: none"><li>– Determination of metamerism index</li></ul></li><li>• Dyes:<ul style="list-style-type: none"><li>– Hue of dyes</li><li>– Strength of dyes</li><li>– Moisture content of dye when powder</li><li>– Physical form of dyes, liquid at national have greatly improved accuracy</li><li>– Fastness properties</li><li>– Ease of dissolving or dispersing dye</li><li>– Dye compatibility (Heat, blocking effect)</li></ul></li><li>• Dyeability of substrate:<ul style="list-style-type: none"><li>– Evaluation of heat-setting conditions; (polyester): time, temperature, tension</li><li>– Substrate contraction evaluation</li><li>– Evaluation of yarns for blending</li><li>– Construction</li><li>– Substrate content variations</li><li>– Substrate preparation: a) Degree of bleaching, Degree of mercerization, b) Cleanness c) existence of chemicals, Size content, etc</li></ul></li><li>• Evaluation of water quality<ul style="list-style-type: none"><li>– Hardness, pH, purity, chlorine amount, etc.</li></ul></li><li>• Evaluation of recipe/procedure in dyeing:<ul style="list-style-type: none"><li>– Maximum exhaustion determination</li><li>– Dye compatibility evaluation for heat and blocking effects</li><li>– Dye instability and Dye-Dye interactions</li><li>– Evaluation of chemicals and auxiliaries pre dyeing</li></ul></li></ul>
Pre-dyeing	<ul style="list-style-type: none"><li>• Verify dry weight of substrate / Moisture content of substrate may vary</li><li>• Verify weight of dyes / dyebath color (dye check)</li><li>• Liquor-to-ratio</li><li>• Salt amount (cotton dyeing)</li><li>• Weight of chemicals and auxiliary product</li></ul>
During dyeing	<ul style="list-style-type: none"><li>• Dyebath temperature</li><li>• Dye concentration</li><li>• Dyebath pH</li><li>• Conductivity</li><li>• Exhaustion profile vs. standard</li><li>• Time of dyeing</li></ul>
After dyeing	<ul style="list-style-type: none"><li>• Shade match confirmation and levelness evaluation</li><li>• Color fastness confirmation (Wash/Light) and waste water examination</li></ul>

Table 1. Important factors that need to be monitored for RFT dyeing

Currently, both Datacolor and Hunter Labs provide customer tailored turnkey systems that integrate computer color matching with the plant dye and substrate inventory. Today, even dye houses with a few dyeing machines are equipped with low-cost hand-held color measurement spectrophotometers. It was essential for the automotive industry to adapt these technologies where tolerance limits are low and tight, while the visual limit of the trained observer is only  $0.6 DE_{CMC}$ .

The ability to measure reflectance of a substrate and correlate it with the visual assessment of trained color observers was the critical step in achieving recipe/ procedure prediction. Sophisticated computer programs based on non-linear equations were developed so that close recipe predictions may be achieved. These predicted recipes were often modified to obtain the final product shade and quality attributes that satisfy customer requirements. Afterwards, the modified recipe can be adjusted for the bulk production as an RFT dyeing.

Two specimens having identical tristimulus values for a given reference illuminant and reference observer are metameric if their spectral radiance distribution differ within the visible spectrum (Schanda, 2007). As a result, a sample may look different at different lighting conditions. This could be an issue especially for a manufacturer where a garment is assembled from various parts that are supplied by different manufacturers. The metamerism index is a single number which indicates how well two materials that match one illuminant will match under another illuminant. Using a spectrophotometer determining L, a, b values, one may calculate the metamerism index as follows:

$$\text{Metamerism Index} = \sqrt{(\Delta L_{i1} - \Delta L_{i2})^2 + (\Delta a_{i1} - \Delta a_{i2})^2 + (\Delta b_{i1} - \Delta b_{i2})^2} \quad (2)$$

where  $\Delta = X_s - X_r$ ,  $i1$  is illuminant 1,  $i2$  is illuminant,  $s$  is standard and  $r$  is reference.

#### 4.2.2 Incoming dye shipment test

Dye standardization makes a major contribution to dye selection and the ability to achieve RFT dyeing. Researchers (Park & Shore, 2007) listed the parameters for dye selection and their responses to environment and behavior in the dye-fiber system. Even though all parameters listed in the publication (Park & Shore, 2007) are important, only a number of factors are considered critical and are included in the routine evaluation of dyes, either in the assessment of deliveries or in the evaluation of competitive products at the plant. The parameters that may be monitored practically are:

- Dye hue test
- Dye solution strength test : a) moisture content of dye when powder, b) Physical form of dyes; liquid versus powder.
- Dye stability and sensitivity to chemicals in particular pH
- Determination of strike temperature for dye database
- Dispersion performance for disperse dyes
- Fixation for reactive dyes
- Fastness

Among all the variables, perhaps hue and strength are the most important control parameters that need to be evaluated for each incoming dye (Park & Shore, 2007).

Commercially, absorption spectrophotometry is frequently used in assessing dye strength. Despite time and experience, there are significant variations in measuring dye strength even

within the same lab. These variations are often caused by differences in weighing methods such as a) errors in weighing, b) variability in the solution making, and c) variability in wait time before absorbance measurement, and d) evaporation of the dye solution. To ensure accurate and comparable results between laboratories, it is essential to have standard test procedures to estimate hue and the relative strengths of dyes (Günay & Jasper, 2010; Park & Shore, 2007).

4.2.2.1 Dye hue test

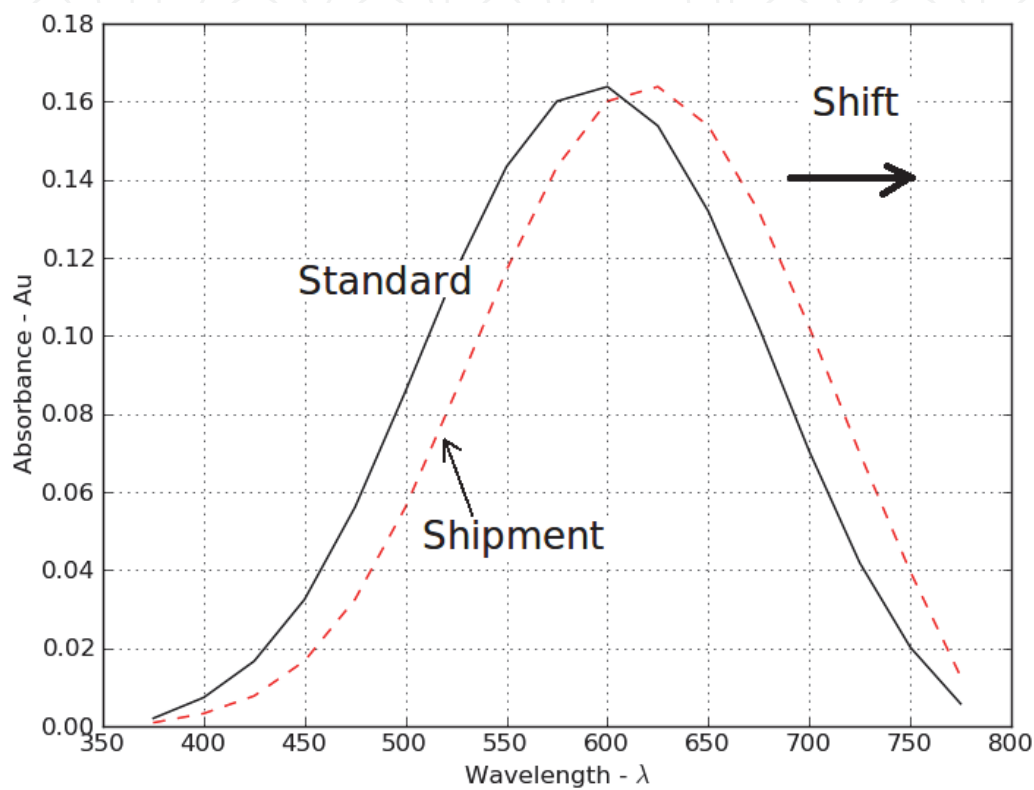


Fig. 4. Significant spectral differences between the standard and shipment dyes.

The dyes which determine the shade of the fabrics are shipped to dye houses lot-by-lot, sometimes from different plants, and occasionally from different countries. After being stored in a warehouse for some time, they are used in production as needed. Even though attempts are made to manufacture dyes that are exactly the same, this is often not an easy task to achieve. There may be spectral differences among dye shipments. These may be caused by either color chemistry differences between the dye shipments or unfavorable stocking conditions such as heat and humidity which modify the chemistry of the dye at the warehouse.

Spectral shifts such as shown in Figure 4 cannot be easily corrected. In such cases, it is highly recommended to avoid those dyes that exhibit these spectral shifts in manufacturing. To quantify the spectral shifts, a dyer may measure the absorbance of the dyes in the inventory and graph them as a function of time. Later using Equation 3, the dyer can determine the storage stability of plant dyes.

$$= \frac{\sum_{380}^{780} |A(t)_{\lambda} - A(0)_{\lambda}|}{\sum_{380}^{780} A(0)_{\lambda}} \tag{3}$$

4.2.2.2 Dye solution strength test

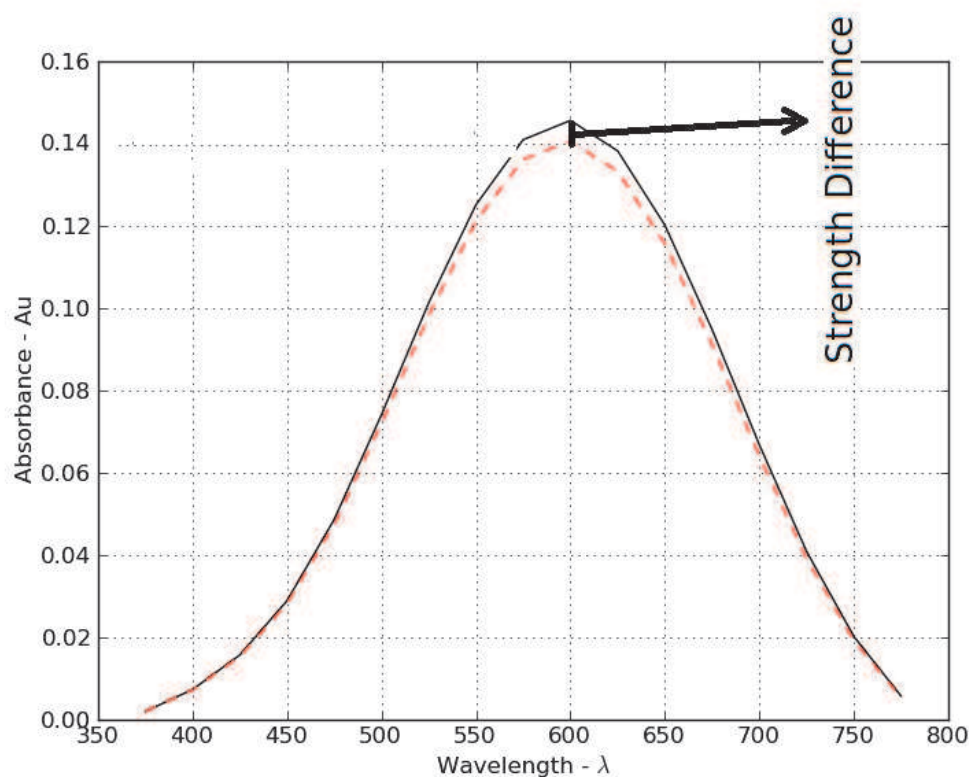


Fig. 5. Strength differences in dyes.

When a partnership is established between dyer and dye supplier, the latter provides the customer with a certificate of conformity to the standard for individual deliveries. However, it is still good practice to verify dye strength differences between the shipments of dyes and their standards.

The storage conditions of dyes in use must be carefully controlled, as the effect of ambient conditions on moisture content can significantly impact dye strength (Gaunt & Moffitt, 1991) which in turn may cause severe color differences in depth and hue in dyeing. Consequently, it may be good practice for a dyer to check dye strength prior to each dyeing.

This could be especially important for disperse dyes, as these contain different diluents that vary in hygroscopicity.

Figure 5 shows the strength differences in spectra between the standard and shipment dye. This difference may be quantified by determining the maximum absorbance at  $\lambda_{max}$  for each dye and calculating the ratio between them as given in Equation 4.

$$\%Strength_{dye} = \frac{A_{\lambda_{max}}^{shipment}}{A_{\lambda_{max}}^{standard}} \times 100 \quad (4)$$

Figure 5 demonstrates that the strength of the shipment dye is only % 96 of the standard one. If the result obtained in Equation 4 is less than % 100, the result is due to a weaker dye. If the shipment dye is above % 100, the dye is stronger than the standard one. To match the same shade using the new shipment dyes, the recipes have to be corrected accordingly. For example, if one of the shipment dye's strength is 96%, instead of simply adding 4% of that dye, the proper correction would be calculated as follows:

$$= \frac{100}{\%Strength_{dye}} \times Amount_{dye} \quad (5)$$

Knowing the spectral strength deviations from the standards increases one's chances of successful dyeing.

Using the method suggested above, it is possible to achieve dye standardization to within +/- 0.05% tolerance. With currently available dye strength determination based on dyed sample reflectance measurement technology, tighter tolerances less than +/- 3% can not be reached (Park & Shore, 2009). Hence, the proposed method has clear advantages over the existing dye strength determination based on reflectance measurements.

Note that as moisture content and dye solubility are key parameters in determining the dye strength accurately, it is particularly important to establish SOP (Standard Operating Procedures) for storage and handling of the dyes.

Furthermore, some dyes are available in both liquid and powder form. Liquid dyes may be preferable for accuracy reasons and for health and safety due to reduced exposure of operators to dye dust.

#### 4.2.2.3 Determination of strike temperature for dye database

Heat sensitive dyes such as acid and disperse class dyes do not begin exhausting significantly until the temperature reaches a certain level. When this level is reached, the dye starts migrating into fiber as polymer expands and allows dye molecules to penetrate. While dyes with high molecular volume may require higher temperatures to begin exhaustion, the dyes that are small in molecular size are likely to exhaust earlier.

A database of dye and strike temperature may be populated for each dye in the inventory, and when a recipe is formulated, dyes close in strike temperature may be selected for efficient and level dyeing. Refer to section 3 for the determination of strike temperatures.

#### 4.2.2.4 Dispersion breakage of disperse dyes

Dispersions are not stable and over time dye particles coagulate and settle in the machine and/or on the fiber (Ferus-Comelo, 2008). Even though dispersion breakage increases with liquor flow velocity in the machine, it is still dye specific. Once the dye is no longer dispersed, it deposits on any available surface; fiber, metal or glass, for example, leading to uneven dye distribution in the machine and on the substrate. Dispersion breakage is therefore undesirable but, to some extent, unfortunately also unavoidable. It is possible that deposited dye is re-dispersed or dissolved during later stages of the dyeing so that dispersion breakage might not always be noticeable on the final product (Ferus-Comelo, 2009).

A quantitative indication of the extent of dispersion breakage can be obtained by comparing exhaustion values calculated by dye concentration measurements in the dyebath with those

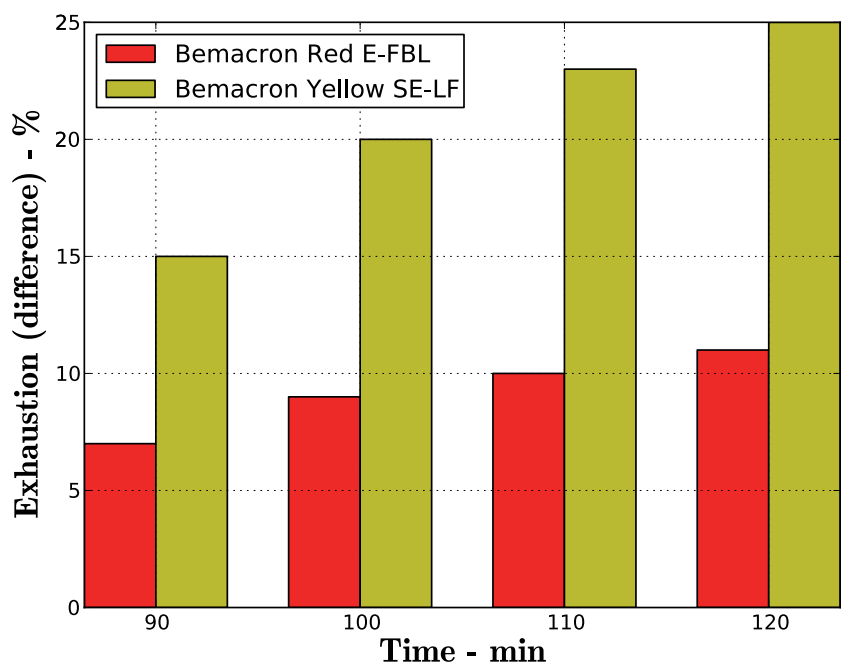


Fig. 6. Dispersion breakage for 2 dyes calculated from exhaustion differences.

calculated from fiber reflectance measurements. When the exhaustion values obtained from dyebath measurements are significantly higher than those obtained from the washed substrate, the difference is probably caused by dispersion breakage since the deposited dye is neither taken up by the fiber nor present in the dyebath. Exhaustion values calculated from the solution measurement are in this situation, therefore systematically too high (Ferus-Comelo, 2009).

Figure 6 shows dispersion breakage for 2 sample dyes calculated as the differences between the measured exhaustions using a real-time dyebath monitoring system and the calculated exhaustions from the reflectance measurement of the substrate. There are significant differences in dispersion breakage for given dyes. The exhaustion measured is on the average 12% more for the Bemacron Yellow SE-LF than that of Bemacron Red E-FBL. Furthermore, the dispersion breakage increases for Bemacron Yellow SE-LF more rapidly than that of Bemacron Red E-FBL. Consequently, the dispersion breakage of former dye is less than the latter, hence has a higher tendency to produce uneven dyeing.

4.2.2.5 Fixation efficiency for reactive dyes

Since fixation efficiency of reactive dyes varies, it is recommended that dyes with higher fixation are identified and preferred in recipe formulation in plants. The methodology to determine fixation efficiency of reactive dyes using RFT monitoring technologies is rather straightforward. Determination could be achieved by dyeing a few different kinds of substrates with the same dye and recording their final exhaustion when dyeing is completed. Since fixation is the amount of dye covalently bonded to the substrate, it is critical to remove excess dye from the substrate upon completion of the dyeing by means of successive rinses. Successive rinses should be performed until no significant dye is bleeding into the rinse bath. Following each rinse the amount of dye flushed from the substrate should be accounted for and deducted from the final dye exhaustion by means of basic book keeping using Equation



6.

$$\%Fixation = \%Final \text{ Dye Exhaustion} - \sum_i^n \%R_i \quad (6)$$

where  $R_i$  is the percentage of dye flushed at the  $i$ th step of the rinse and  $n$  is the number of rinses.

#### 4.2.2.6 Fastness

Fastness is the ability of fabric to retain color in use. There are three types of fastness tests : a) wash fastness, b) rubbing fastness, and c) light fastness. These test methods are discussed in detail at AATCC test methods 61, 8, and 16, respectively. Briefly,

**wash test:** is done by stitching the shaded fabric between the two white fabrics and laundering the specimen. After drying the degree, the stained white fabrics are evaluated against a standard stain scale.

**rubbing test:** is performed by rubbing the shaded fabric under pressure to a white fabric specimen. The degree to which the white specimen is stained determines the rubbing fastness of the fabric.

**light fastness:** Light fastness is the degree to which a dye resists fading due to light exposure. Different dyes have different degrees of resistance to fading by light. The test is often conducted under Xenon light and standard condition for a determined duration. After the test, the level the sample fades is compared against a standard scale to quantify light fastness.

In order to grade and quantify loss of color, reflectance measurements may be employed. This section provides several quality attributes about dyes. Due to complexity in dye quality attributes, simple product substitution using the so-called equivalent dyes is not an easy option. It requires a great deal of study to assess all the factors which impact dyeing, some of which are given in this section.

#### 4.2.3 Dyeability of substrate - substrate evaluation

Both natural and synthetic fiber properties may vary between different shipments of bales or lots. Variations in fiber properties caused by changes in manufacturing inputs/ parameters for synthetic fibers or environmental factors for natural fibers may produce textile products that do not satisfy customer requirements such as strain/strength, shade consistency, or levelness. The variation in textile physical properties may affect the performance of dyeing and the final product significantly. Hence, it is important to develop tests to determine, evaluate and control:

- substrate content variations,
- yarns for blending,
- variations caused by heat-setting conditions; (polyester): time, temperature, tension,
- variation in substrate preparations such as a) degree of bleaching, b) degree of mercerization (size content), c) existing of chemicals and d) cleanness,
- effect of construction in dyeability,
- expected contraction after dyeing.

This section provides examples to demonstrate that monitoring the performance of dyeing may lead one to determine the dyeing quality attributes of fibers and in turn: a) improve fiber properties, b) develop new fibers, and c) verify fiber quality for new crop and merge.

4.2.3.1 Substrate content variations

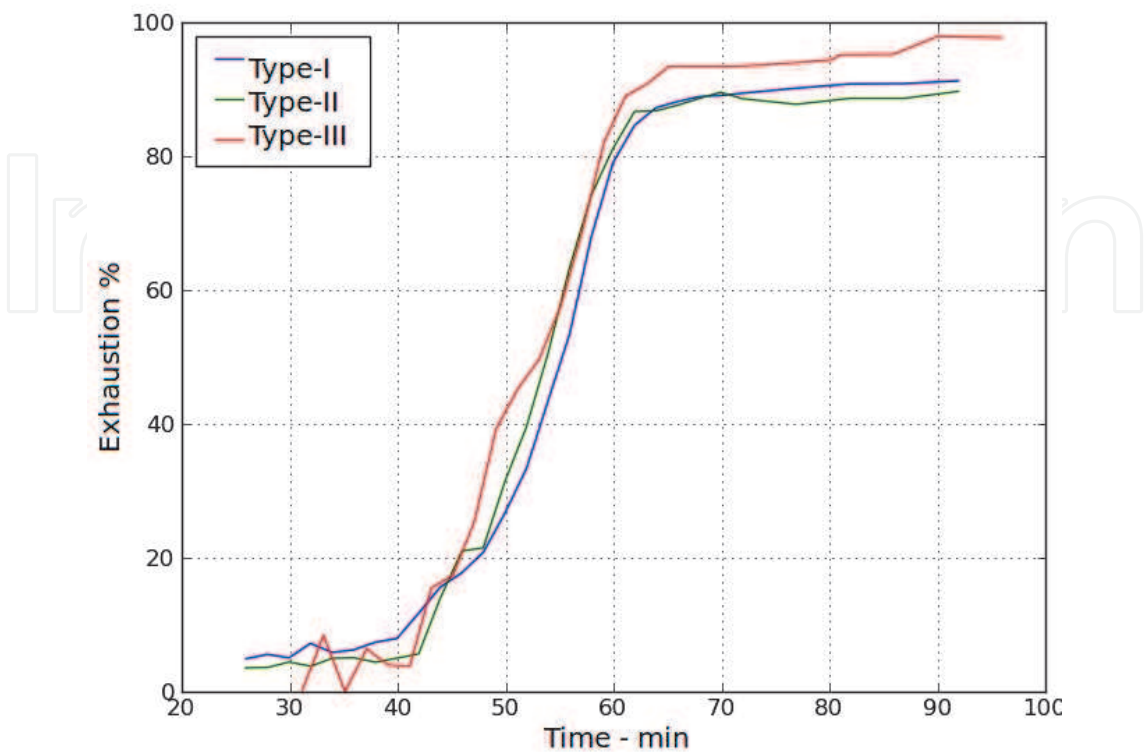


Fig. 7. The exhaustion curves of three shipments of the polyester fiber.

Figure 7 shows the exhaustion performance of disperse dyeing of Red 167 on three different polyester fibers. In Type-I and Type-II, recycled polyester fibers were blended into yarn. The pure polyester fiber, Type-III, has a 10% greater final exhaustion than the others. As a result, the foreign matters present in the recycled polyester fiber blends negatively affect the dyeing performance of the yarn. If Types I, II and III fibers were combined in a fabric, it would come as no surprise to observe barre effect in the constructed fabric, as some parts of the fabric would uptake more dye than others.

4.2.3.2 Evaluation of yarns for blending

A common source of frustration and blame is barre in blended fabrics. The dyer will usually blame the barre on micronaire or yarn texturing differences, and the fiber producer then blames the dyer for bad procedure or faulty dyeing.

Figure 8 shows three different exhaustion profiles of two different polyester fibers blended into a final fabric. The single fibers and blended fabric were dyed individually with a single disperse dye. The key to the graph is as follows: 767 is a Drawn Textured Yarn (DTY), 769 is a Semi Drawn Yarn (SDY), and 771 is the blended fabric produced from both yarns. Ideally, the exhaustion profile for each yarn would overlay and dye exactly the same, and so would the blend. This is not the case however, as the base fibers dye differently. And when the fibers are blended, the overall exhaustion profile for the dyeing of the combined yarns is different than either fiber. Consequently, when there are three or more total dyes for a shade, the shading between the yarns can become quite significant because of the differences in the fibers dyeability.

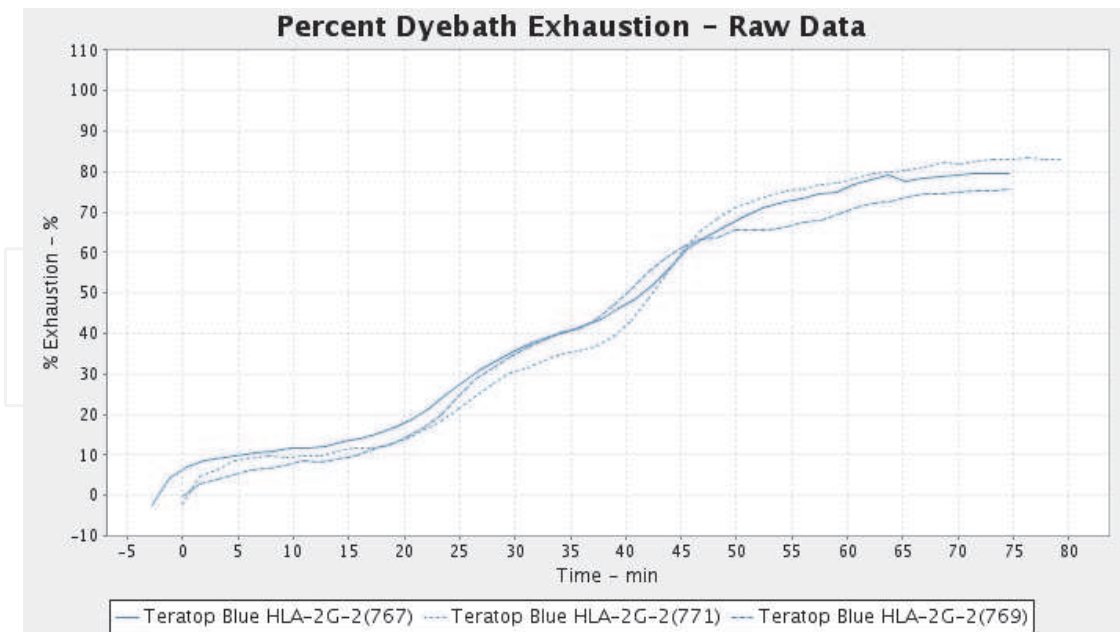


Fig. 8. Dyeings of individual and blended polyester yarns with different exhaustion profiles.

4.2.3.3 Evaluation of fiber properties on dye uptake

Fiber characteristics may be impacted by heat treatment conditions such as temperature and tension at which drawing is applied, as well as the heat treatment history.

Figure 9 shows dye exhaustion performance of the same substrates of different yarns. As seen from the graph, the matching characteristic exhaustion curves indicate that the absorption and/or migration of dyes to substrate behave the same way. The morphological differences due to different yarn cross-sections contributed to strike temperature differences between these two dyeings.

4.2.3.4 Evaluation of substrate construction on dye uptake

Figure 10 shows dye exhaustion of different substrates that use the same yarns. As seen in the picture, both dyeings have the same strike temperatures, indicating the yarns have the same physical and chemical properties. However, the time of dyeing,  $t_e - t_s$ , for both substrates varies significantly. This is perhaps due to differences in surface area affecting dye absorption and migration.

4.2.3.5 Evaluation of pre dyeing treatment of the substrate

Many coloration problems arise as a result of inadequate preparation of the substrate. Substrate preparations such as a) degree of bleaching, b) degree of mercerization (size content), c) existence of chemicals, and d) cleanness may not only significantly affect the accuracy of concentration measurement but also dyeing performance. Therefore, in order to reduce the shade variation between lots that may have been caused by inconsistent substrate treatments in pre dyeing steps, it is highly recommended to wash off the substrate before the dyeing.

4.2.3.6 Determination of contraction after dyeing

It is not uncommon to observe fabric contraction after a dyeing. In order to meet customer requirements, it is highly recommended to do a lab dyeing prior to batch dyeing to estimate %

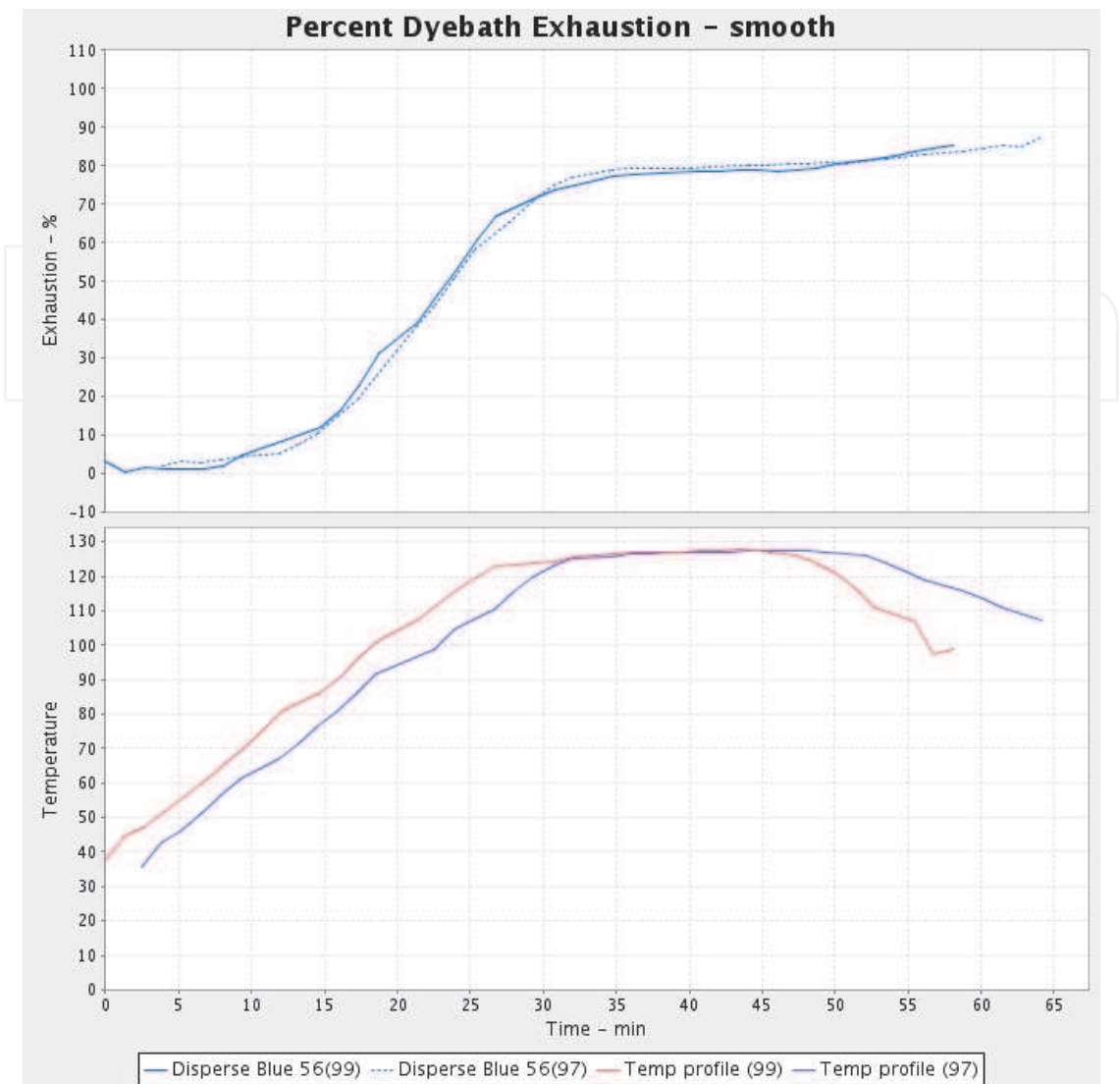


Fig. 9. Different yarn used in same fabric structures.

contraction. Final fabric dimensions may be set by adjusting tenter frame width and overfeed to accommodate % contraction.

4.2.4 Dye house water quality control

The quality of textiles produced by any manufacturing operation which employs wet processes, such as preparation, dyeing and finishing, is profoundly affected by the water quality (Smith & Rucker, 1987a).

Dye house water may be supplied from;

**collected rain water** filtered by sand bed. It is pure, but limited in supply, availability, and varies with weather conditions and contents deviate depending on air pollution.

**collected surface water** from lakes and rivers. May contain dissolved organic and inorganic matters such as chloride, sulphate, carbonate, bicarbonate of sodium, potassium, calcium an iron. Often dependable and consistent supply. Variation may occur gradually.

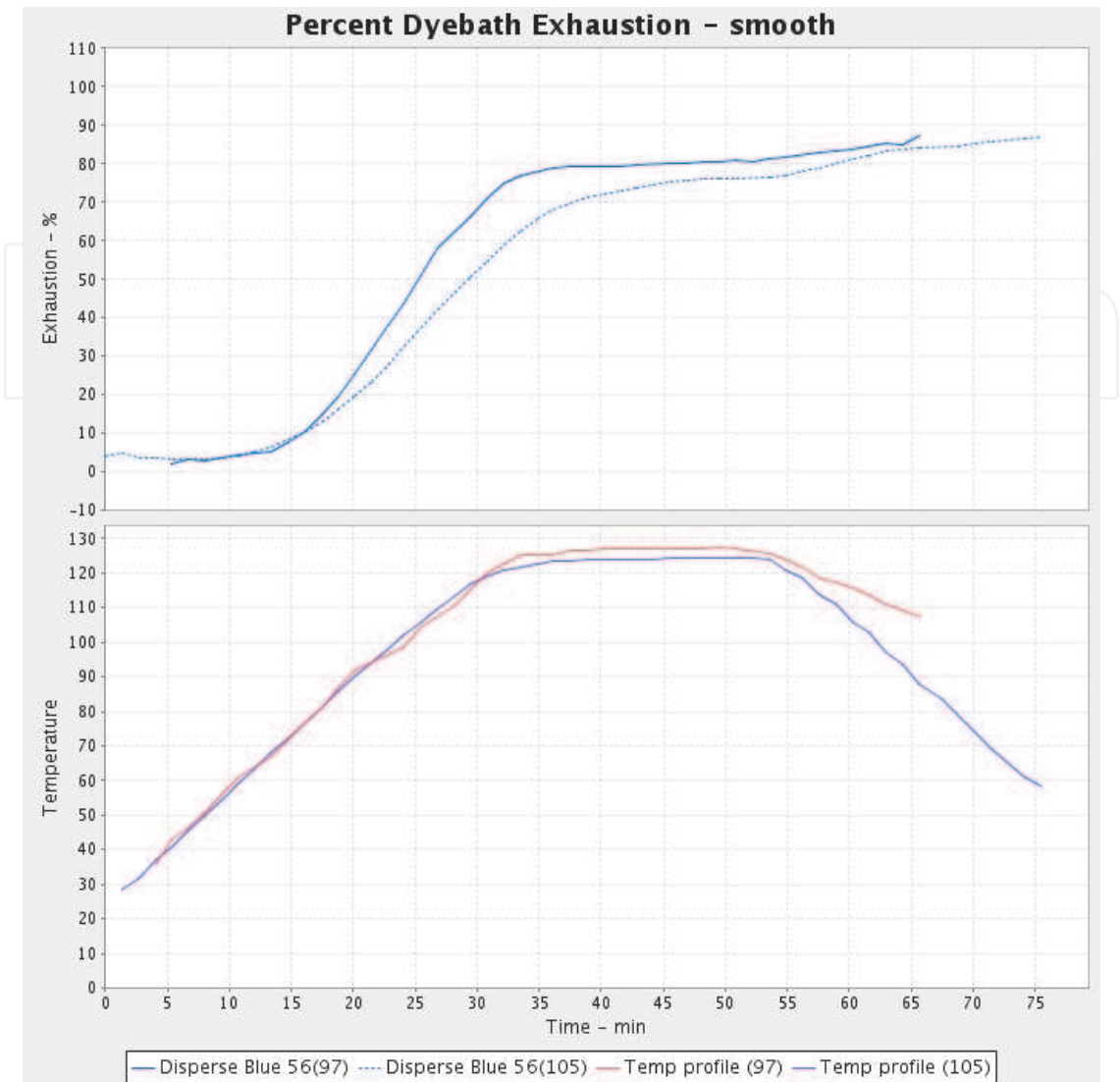


Fig. 10. Same yarn used in different fabric structures.

**well water** below the surface. May contain soluble organic compounds, ammonium salts, nitrates. Often dependable and consistent supply with gradual variation over time possible.

**city water.** Consistent supply but not necessarily dependable composition. Chemicals may vary with season and occasionally even by day.

Choosing the correct water supply consistency is critical in RFT dyeing. For the ease of the dyeing process, dye plants are located accordingly. For example, if plant water has hardness, scale could form in boilers and pipes and could result in increased energy use due to reduced thermal conductivity. High CO<sub>2</sub> concentration may cause boiler corrosion when CO<sub>2</sub> reacts with iron in the presence of O<sub>2</sub>.

Using hard water has often produced serious negative results on dyeing performance, such as:

**Wastage of Soap & Detergent:** Ca and Mg salts react with soap and lather is not formed easily.



**Reaction with Dyestuff:** The reactive groups present in dyes react with Na, Ca, Mg, and Fe in hard water. This causes wastage of dye, and produces duller shade and faulty dyeing. A sequestering agent is added to prevent this.

**Deposition on Textile Material:** Soap reacts with hard water and produces insoluble salts which deposit on the fabric during scouring. These salts harden and reduce flexibility in the fabric.

Ideal water has the following properties (Heetjans & Tindall, 1995):

1. Color: Water must be colorless by which the dyeing will be carried out.
2. Odor/ Smell: Water must be odorless.
3. pH: Value would be 7 to 8.
4. Water Hardness: Less than 90 ppm (90 milligrams of  $\text{CaCO}_3$  per litre of water).
5. Dissolved Solids : <1 ML/L
6. Solid Deposits: < 50 Mg/l.
7. Organic Substance: < 20 Mg/ L.
8. Inorganic Salt: < 500mg/L
9. Iron : < 0.1 Mg/l.
10. Copper: < 0.005 Mg/L.
11. Nitrate: < 50 Mg/ L.
12. Nitrite: < 5 Mg/L.

Water contaminants, especially metals, can have a substantial effect on many textile wet processes. The effects are not always adverse, but it is not desirable to have variance in processes and product quality due to water quality changes. Because these variations in the quality of water make process and machinery optimization and control difficult (Smith & Rucker, 1987b).

There are many quick qualitative tests for detection of trace quantities of ions and elements in water. There are also quantitative tests for determining the exact concentration of cations such as calcium, magnesium, iron, copper, and manganese in water (Smith & Rucker, 1987b). In addition to regular testing for pH and the hardness of the incoming water, a standard sample dyeing with single dye without chemicals may be chosen and monitored using RFT technologies every so often, and exhaustion profiles may be compared with the standard ones. Although not conclusive, the deviation from the standard exhaustion profile may indicate variation in the incoming plant water.

#### 4.2.5 Evaluation of recipe/procedure in laboratory dyeing

Even though researchers developed techniques and sophisticated algorithms to predict recipes and procedures using color matching instruments and historic dyeing databases, it is still cost-effective to repeat bulk production dyeing in the laboratory. It has been shown that a bulk correction of a 300-kg production batch may cost as much as 25 laboratory dyeings depending on the process and dye house infrastructure (Hildebrand & Hoffmann, 1993).

It is important for staff to follow SOP to minimize errors caused by inconsistent procedures within the lab between operators.



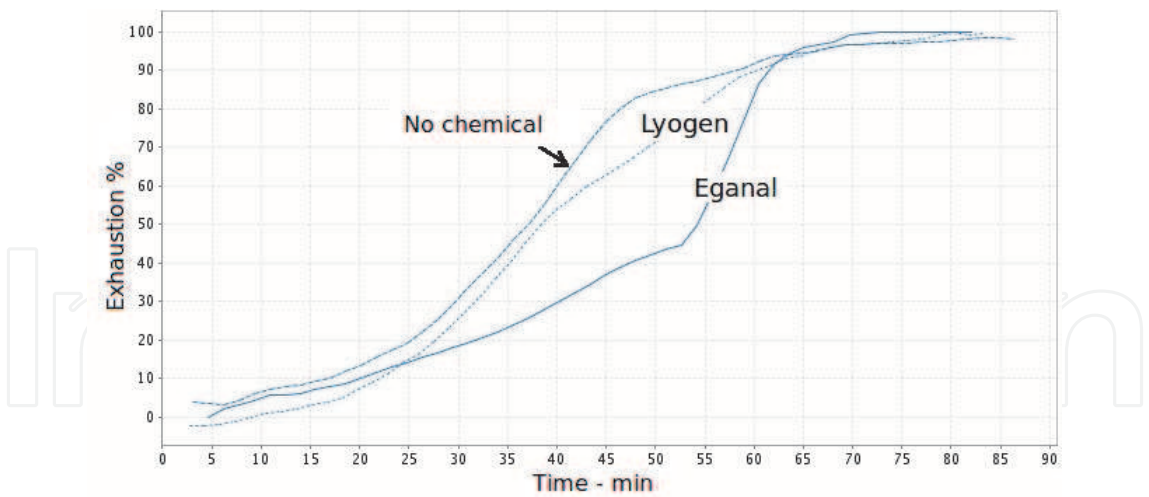


Fig. 11. Effect of leveling agent on dyeing

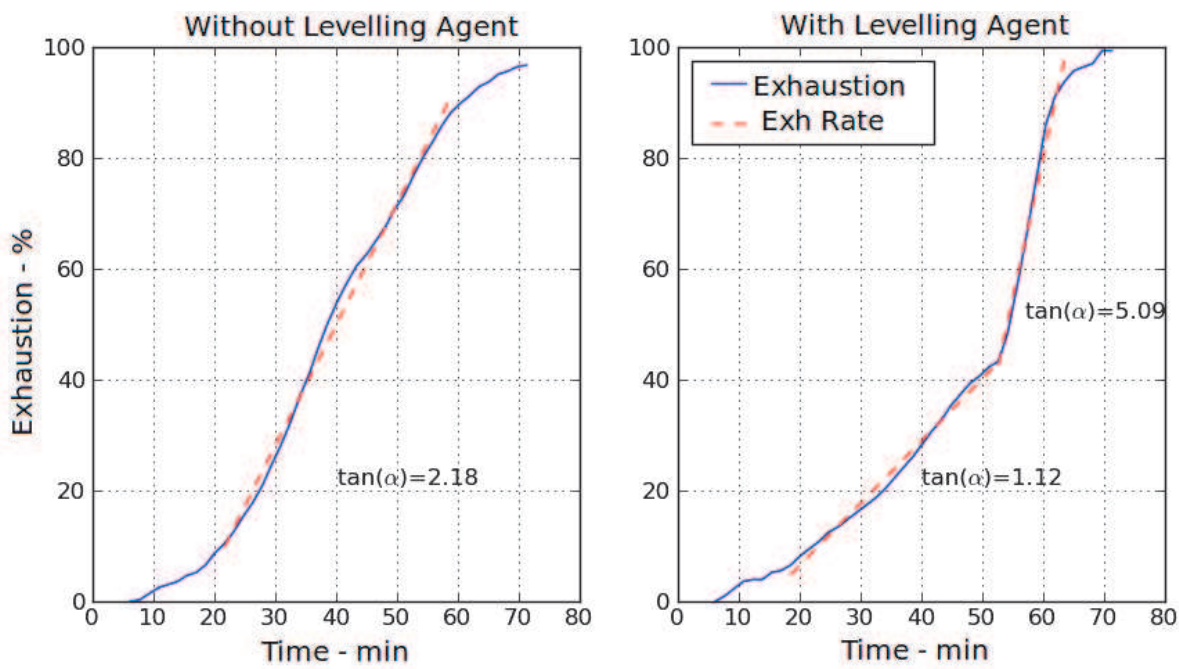


Fig. 12. Effect of leveling agent on dye strike rate

4.2.5.1 Evaluation of chemicals and auxiliaries pre dyeing

Different chemicals may produce different results on the same dyeing. Sometimes the same chemical may act differently in another dyeing. To determine the effect of chemicals on dyeing performance, it is best to monitor the dye exhaustion profile and determine strike temperatures and rates.

Figure 11 demonstrates Lyogen and Eganal leveling agent’s effect on two different dyeings using the same disperse dye *Terasil Blue RBS*. Both leveling agents delay the dye exhaustion differently as demonstrated in their dye exhaustion profiles shown in Figure 11. For this particular dyeing, the absence of a leveling agent did not cause any dyeing unlevelness. On the contrary, the leveling agent was the cause of shade levelness observed on the fabric. This was explained by the increased rate of exhaustion in the presence of levelness agents at the exhaustion phase.

Figure 12 shows the exhaustion trends during the exhaustion phase. In the absence of chemicals, the average rate of exhaustion was calculated to be at 2.18 % / min using Equation 7.

$$\tan(\alpha) = \frac{\%Dye \ uptake}{Time} \quad (7)$$

In the presence of a leveling agent, the chemical Eganal blocks the dye uptake until the 53<sup>th</sup> minute. Up to that point the average dye uptake is around 1.12 % / min. Once the blocking effect of the chemical disappears, 50 % of the exhaustion occurs in the next 10 minutes with an average exhaustion rate of 5.09 % / min. If the fabric in the dye machine circulates at a slower pace, then observation of shade unevenness would come as no surprise.

Consequently, different leveling agents produce different results on dyeing performance. In some cases, it may be even better to avoid using a leveling agent.

#### 4.2.5.2 Dyebath stability and sensitivity to chemicals

Dye instability may occur when:

- dyes in dyebath interact with each other,
- one or more dyes interact with chemicals in the dyebath,
- dyes change with temperature or time.

When two or more dyes are mixed together with no chemical interaction among them, the sum of the spectra of the individual dyes should equal the spectrum of the mixture. Dyes with this property obey the law of spectral additivity (Günay, 2009). A number of investigations with direct and VAT classes of dyes in mixture have proven this statement invalid. Neale and Stringfellow (Neale & Stringfellow, 1943) concluded that for certain direct dyes, the spectra of the mixtures in water were not additive. In an aqueous solution, pairs of dyes interact with each other, possibly through the operation of resonance bonds or residual valency forces similar to those responsible for anchoring the dye onto the hydroxyl groups of cellulose.

Using a dyebath monitoring system, one may determine the absorbance profile of each dye and the dyebath. Figure 13 shows the absorbance spectrums of Solophenyl Bordeaux 3BLE, Solophenyl Blue FGLE 220% and Solophenyl Scarlet BNLE 200% shade direct dyes at concentrations of 2.60 g/L, 1.00 g/L and 0.90 g/L respectively. When these three dyes are mixed together, the dye interaction between the Blue FGLE dye and the other two dyes shifts the spectrum compared to a spectrum comprised of the linear sum of the absorbance spectra, as shown in Figure 14. Such shifts due to spectral additivity (or super position) may cause concentration estimation and dyeing problems.

A pair test of the dye mixture indicated that the Blue FGLE of the mixture interacts with both the Bordeaux and Scarlet dyes. It was therefore recommended that the Solophenyl Blue FGLE 220% dye be substituted with another blue dye (DF Blue FGL) to reduce or eliminate dyeing problems.

#### 4.2.5.3 Dye compatibility evaluation for heat and blocking effects

It is not uncommon for certain dyes to block each other when combined and consequently lower the exhaustion of the other dye. This is often caused by differences in dye molecular sizes. In other words, when a smaller molecular sized dye is combined with a larger molecular sized dye, the dye with the smaller molecular size may penetrate into the structure of the substrate faster than the larger one, consequently exhausting faster. However,

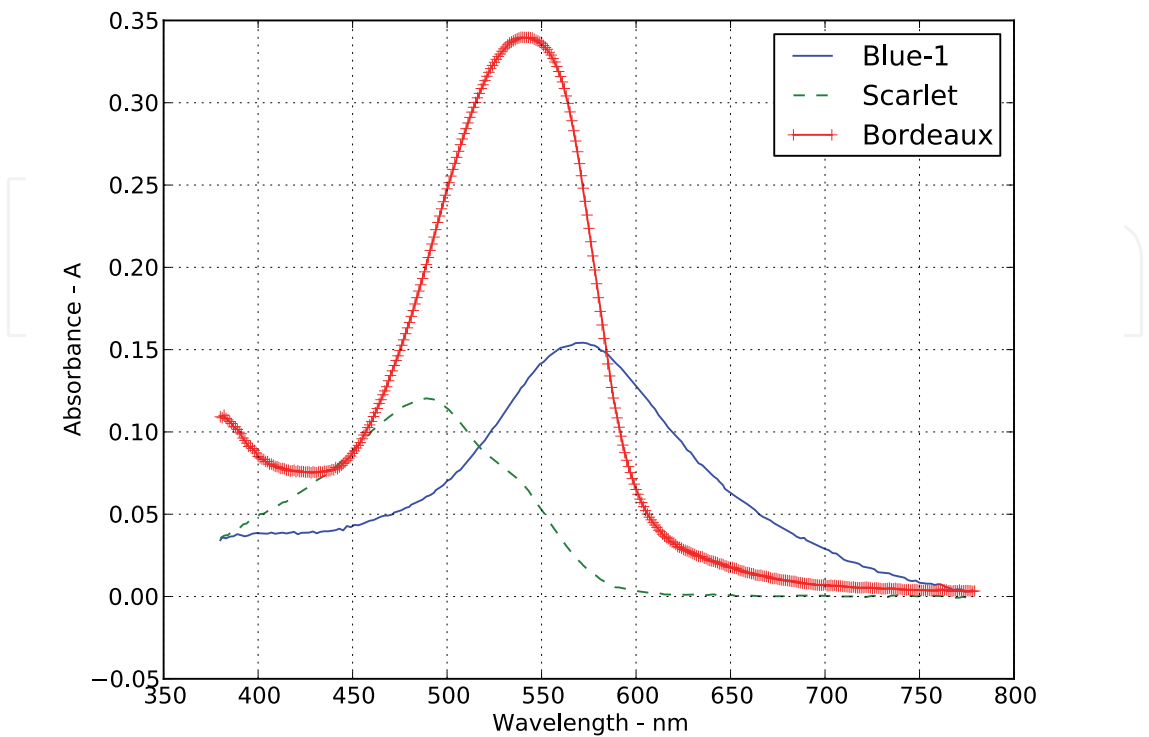


Fig. 13. Spectra of 3 different direct dyes

another possible scenario could have the larger molecular sized dye block entry points of the substrate and block the smaller molecular sized dye’s penetration into the structure. Nevertheless, whether the former or latter phenomenon occurs, the dye incompatibility may create differences in strike temperatures and exhaustion rates. Level dyeing and high exhaustion may often be achieved only with compatible dyes.

Figure 15 demonstrates dyes that are not considered compatible when used together. As Orca Navy NRBL has a lower strike temperature than both the Lanaset Yellow S-L and Lanaset Red, it begins exhaustion before the others. Consequently, the substrate will first be dyed with the Navy shade dye, followed by the Red and Yellow shade dyes. As a result, even if the same amount of dye were exhausted onto the fabric, the shade obtained would be different when the dyes are incompatible, as opposed to when they are not.

4.2.5.4 Maximum exhaustion determination

One of the goals of optimum dyeing is reaching the maximum exhaustion efficiency possible. This may not be possible when dyes and/or chemicals are not compatible, or the recipe or procedure is not appropriate for the target dyeing. Nevertheless, it is still good practice to determine the maximum exhaustion of a dyeing for process optimization to lower cost and environmental waste.

4.2.6 Considerations to achieve lab-to-dye house correlation

In the past, nonreproducibility was so common that dyeings frequently needed follow up corrective dyeing. Consequently, the dyers subtracted 10-15% from the quantities given in

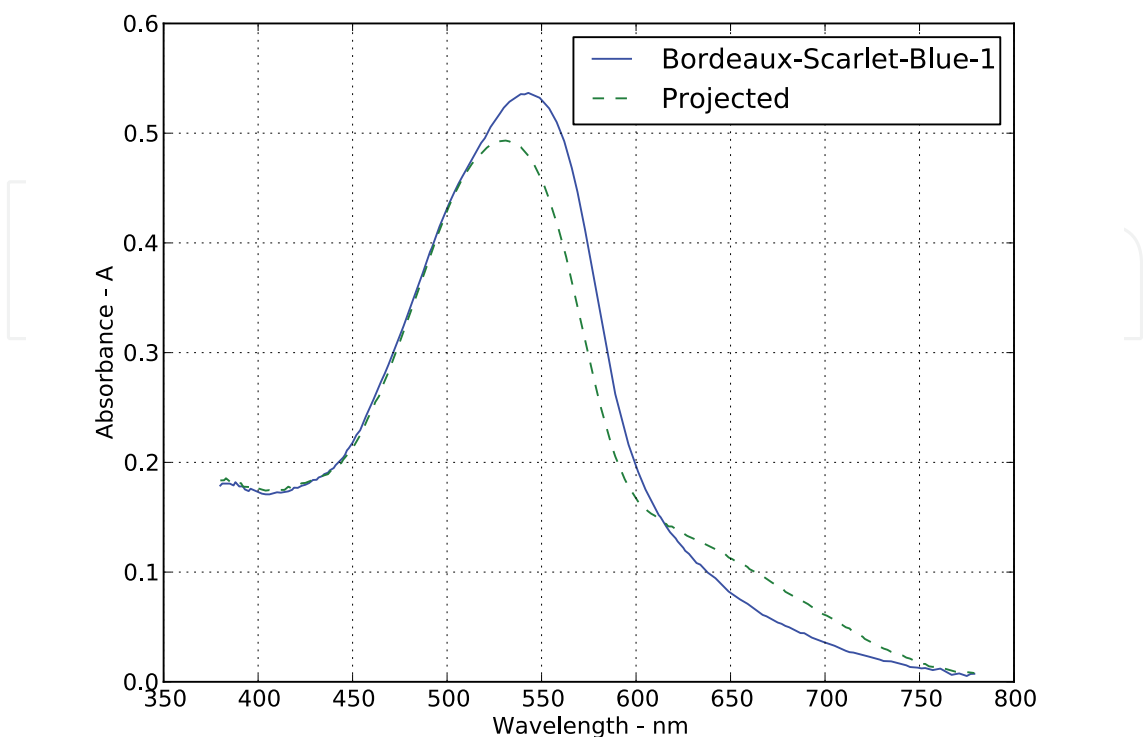


Fig. 14. Combined Spectrum of Bordeaux, Scarlet and Blue-1 Direct Dyes

the laboratory recipe before proceeding to mass production. This caused at least one color addition and removed any possibility of RFT dyeing.

As a result of numerous independent worldwide audits, it was found that many important factors varied between the laboratory and production. The dyeings carried out in the laboratory may not be comparable unless the same dyes, chemicals, substrate, and water supplies are used (Park & Shore, 2007). Since, high-accuracy laboratory dyeing is essential for dye evaluation and database preparation for shade matching. It is therefore critical for dyers to achieve Lab-to-Dye house correlation by replicating the conditions of the dye house in the laboratory. Achieving this would permit dyers to simulate dyeing and prevent production loss.

4.3 Pre-dyeing last controls

- Verify dry weight of substrate as moisture content of substrate may vary the weight
- Verify weight of dyes / dyebath color (dye check)
- Liquor-ratio
- Weight of chemicals and auxiliaries. Verify pH and conductivity

It was demonstrated by Sumner (Summer, 1976) that inaccuracies in weighing and measurement of volume in the drug room or dye dispensary were two of the principal causes of poor reproducibility in the dyeing process. It is therefore critical to verify the % amount of dye per unit fabric weight using RFT technologies described below.

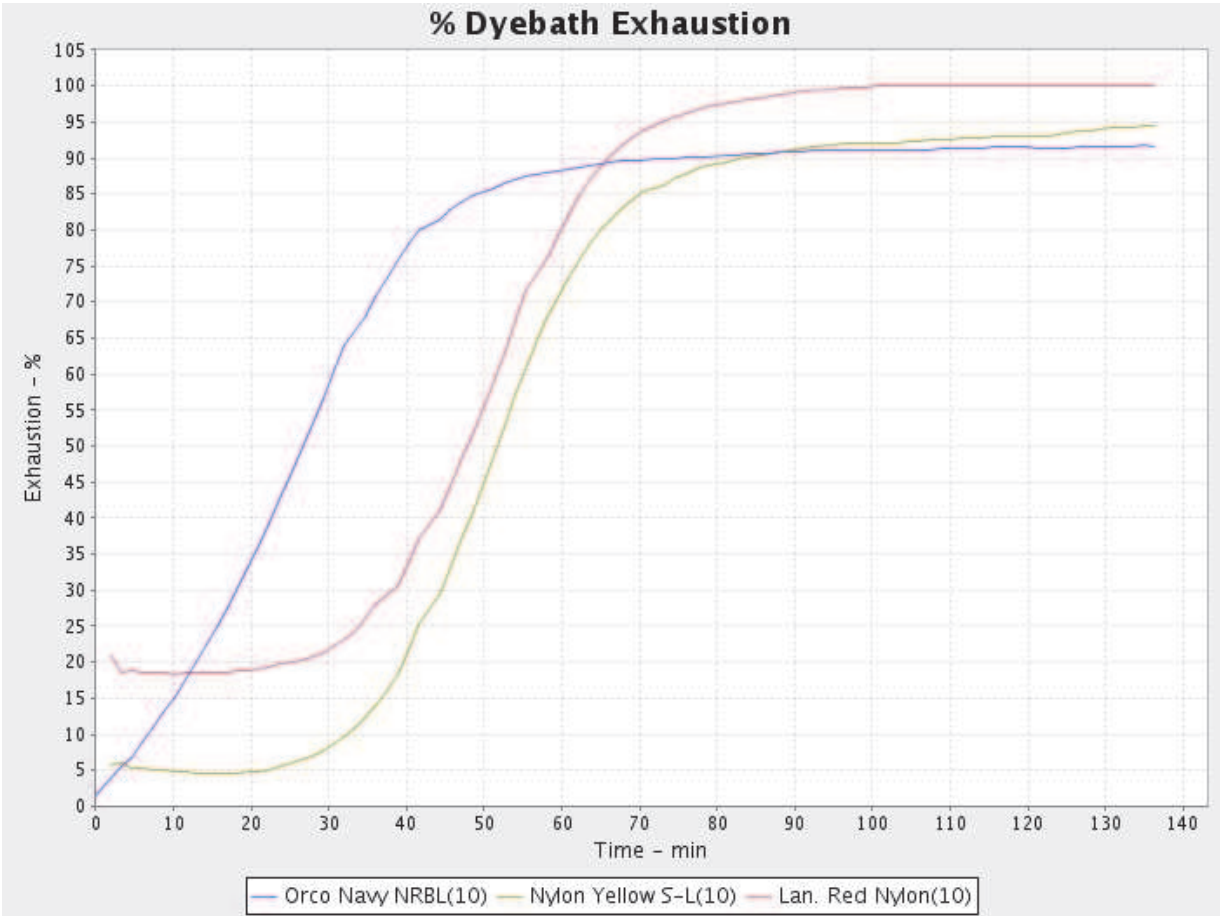


Fig. 15. Incompatibilities between dyes.

4.3.1 Dye amount check in the prepared dyebath

Even though instrumentation errors are rare in automatic dye/chemical dispensing, human weighing errors often cause varying dye amounts in the recipe. To prevent these errors, it is recommended to check dye concentrations in the dyebath mixture before they are transferred to the dyeing machine. This task can be achieved easily by injecting a sample of the dyebath to the RFT monitoring system and comparing the individual dye concentrations with the specified amount in the recipe. For this task, it is best to inject the dyebath sample up to 5 times and determine the average dye concentration. Once the average dye concentrations are determined, they are compared with the expected dye concentrations, and the absolute error for each dye is calculated.

$$E_s = |C_e - \bar{C}_m| \tag{8}$$

$$\%E_s = \frac{E_s}{C_e} \tag{9}$$

When evaluating solution errors -  $E_s$ :

- use Equation 8 at low concentrations (< 0.01 g/L) due to instrumental limitations
- use Equation 9 at high concentrations (> 1 g/L) for percent error.

where  $C_e$  and  $C_m$  are the expected and measured concentrations in g/L, respectively. This is due to limitations in dye concentration predictions discussed by the author in an earlier paper (Günay & Jasper, 2010).



#### **4.4 Quality control during dyeing**

##### **4.4.1 Dyeing monitoring and procedure confirmation**

During a repeat dyeing, pH, conductivity, temperature, and dye bath concentrations may be monitored and compared with the monitoring of the standard dyeing. The deviations from the standard profiles may be observed in real-time and corrected during the process. If it is not possible to take the corrective action during the dyeing procedure, the monitoring data collected may enable the dyer to determine the root causes of the problem and take the right corrective actions for the following steps and dyeings. Quantification of the departure from the standard would also allow one to employ statistical control charts to trigger alarms for out-of-control procedures.

##### **4.4.2 Use of control systems**

It has been demonstrated that RFT production can be achieved in dye houses where dye bath monitoring systems were deployed. As expected, major financial savings and other benefits have been observed from RFT processing. Further savings in labor may be obtained by introducing control systems at various levels of sophistication using semi-automated (SAC) or fully- automated (FAC) systems and by increasing accuracy and consistency. The installation of robotic systems can convert blind dyeing into a lights-out operation; however, a slight increase in labor qualifications may be necessary (Park & Shore, 2004). Commercially available control systems have been available since 1979 (Ziv et al., 1979), and these control systems, including dispensing, have recently been discussed (Jahmeerbacus et al., 2004; Jasper & Joshi, 2001).

#### **4.5 Post dyeing checks**

##### **4.5.1 Shade confirmation**

The difficulties associated with visual assessment were eliminated with the availability of optimized color-difference equations which allowed for the development of standards and tolerance limits. The total color difference formulas such as DE and DE(CMC) enable dyers and customers to objectively communicate closeness of color match by a single number. Whether visual or instrumental methods are used, a standard operating procedure (SOP) is required which must include factors such as preparation, handling, and measurement of samples. Conditioning of samples for illuminant, humidity, and temperature is essential, and the variations obtained as a result of differences in moisture content alone have been reported. Inappropriate storage conditions can also lead to significant color changes (Park, 1991).

The small color differences [within the range of DE(CMC) 0.3-0.5] demanded by automotive manufacturers and textile retailers cannot be achieved consistently by dyeing processes based on visual matching and correction; these small color differences can only be measured using the instrumental color assessment methods.

##### **4.5.2 Levelness**

Uniformity of fabric appearance is critical in the manufacture of high quality products. Irregularity is normally not acceptable, with the exception of stone-washed, acid- washed or pigment-dyed textiles, where an effect is sought to comply with a fashion trend (Chong et al., 1992). Uniform distribution of a shade within a dyed fabric is affected by several factors (Shivramkrishnan, 1983). The causes of shade variation may be the result of materials, equipment, controls, procedures, human factors, and logistics. These factors affect the distribution of dye in the dyebath and/or the ability of a dye to migrate on the fabric for



uniform fabric appearance. For example, the mass variation of yarn could create non-random periodic irregularities that cause visible streaks on the woven fabric appearance (Günay et al., 2007). Furthermore, the high rate in dye uptake could lead to unlevel dyeing of the textile (Shamey & Hussein, 2005; Smith, 2007). It is therefore critical to monitor and control the dyeing and to evaluate the resulting textile appearance as objectively as possible to achieve optimum dyeing.

The common practice for evaluating color levelness today is based on the visual assessment of one or more observers. While in some cases the grading is simply a pass or failure, in others it is finer tuned by assigning a number such as 5 for the most level and 1 for the least level (Chong et al., 1992; Yang & Li, 1993). Visual assessments are highly dependent on the judgment of the observer; therefore, significant deviations occur in human assessment estimates. In addition, visual assessments often fail to quantify levelness. Communication of levelness data is therefore difficult to assess among suppliers, laboratories, and manufacturers.

Due to the shortcoming of visual assessments, several colorists have suggested objective methods to measure color levelness (Cardamone et al., 1995; Chong et al., 1992; Yang & Li, 1993). Scientists have previously used the colorimetric data of fabric obtained from a spectrophotometer. While some have calculated the standard deviation of the reflectance measurements of the spectrophotometer (Chong et al., 1992), others have instead suggested using K/S values (Yang & Li, 1993). In both approaches, levelness is derived from the variation of reflectance after the spectrum is adjusted with the spectral luminous efficiency function (Wyszecki & Stiles, 2000). Cardamone et al. demonstrated that colorimetric analysis with reflectance and K/S offers limited descriptions of color uniformity. They additionally suggested levelness evaluation based on digital image analysis (Cardamone et al., 1995). Tavanai and et al. furthermore confirmed that the common defects of a dyed fabric are better detected by using the gray scale format over the colored format (Tavanai et al., 1997). Cardamone et al. (Cardamone et al., 1995) obtained the histogram of the digitized image at a particular gray scale. They then calculated the levelness index as the standard deviation of the gray scale histograms.

While the above methods are useful in quantifying levelness objectively, they do not take into account;

- the 2-dimensional aspects of the levelness,
- the scale or extend (micro to macro) of levelness.

#### 4.5.2.1 Measurement of levelness

In an earlier study by the authors, it was demonstrated that the between-area variance curves may be employed to analyze and quantify fabric appearance irregularities where the signal contains periodic irregularities (Günay et al., 2007; Suh et al., 2007).

The first task in determining the levelness of a dyed fabric using between-area variance curves is the identification of representative areas for imaging. This is critical, as the confidence level of the conclusions will depend on the number and nature of the sampling.

Next, the dyed sample images may be captured using an instrumentation setup that consists of a light-box (Gretag Macbeth Spectra Light III), a digital camera, and a tripod. While the illuminant  $D_{65}$  is incident upon the surface of the specimen by an angle of  $45^{\circ}$ , the camera should be perpendicular to the plane of the specimen, as suggested by the AATCC Technical Manual (Evaluation Procedure 9-2007 Option C).

The distance between the camera and the sample and the size of the sample may be adjusted so that the effect of lighting variation is minimized. This relatively simple and inexpensive setup allows one to capture sample images without the shadow of the camera on the sample. Each image is converted into gray scale as it is captured. Depending on the size of the fabric and the desired scale of levelness (micro or macro), users may adjust the sample size and/or resolution.

Finally, each captured image is uploaded into custom developed software to determine the levelness of the sample in an automated fashion. The software auto partitions the fabric image into different grid sizes, calculates the coefficient of variation at different scales, plots coefficient of variation as a function of grid size, and reports a surface irregularity index. The approach summarized above is subjected to a detailed analysis in the sections that follow.

#### 4.5.2.2 Between-area variance curves

Simply put, the between-area variance curves are obtained by calculating the variances of gray scale pixel values, while varying rectangular unit areas within the 2-D gray scale matrix. This 2-D gray scale matrix is constructed from the actual fabric image that is captured using a standard digital camera as described above.

Although there are many ways to choose rectangular unit areas having equal area sizes (Günay, 2005), these unit areas may be considered squares for the purpose of levelness.

Let  $m$  be the pixel length and  $n$  be the pixel width of the entire virtual fabric matrix. The fabric is first partitioned into unit areas of size  $A_{1 \times 1}$  as shown in Figure 16. The between-area variance curve,  $CB(A_k)$ , is the plot of the coefficients of variation between the varying unit areas  $A_{k \times k}$ . This relationship is mathematically formulated with Equation 10.

$$CB(A_k) = \frac{100}{\bar{F}_k} \sqrt{\frac{1}{m_k n_k} \sum_{i=1}^{m_k} \sum_{j=1}^{n_k} [F_{i,j} - \bar{F}_k]^2} \quad (10)$$

where  $CB(A_k)$  is the between-area variance among the unit areas of size  $A_{k \times k}$ ,

$m_k$  is the number of segments in the x direction, that

is  $m_k = m/k$ ,

$n_k$  is the number of segments in the y direction, that is  $n_k = n/k$ ,

$F_{i,j}$  is the value of the property at the cell of at row  $i$  and column  $j$   
(ex; the gray scale pixel value at the cell),

$\bar{F}_k$  is the mean value of the property for all unit areas;

$$\bar{F}_k = \frac{\sum_{i=1}^{m_k} \sum_{j=1}^{n_k} F_{i,j}}{m_k n_k}$$

$m_k n_k$  is the total number of unit areas within a designated fabric matrix.

When the size of each unit area ( $A_k$ ) increases, the variance within the unit areas increases. Consequently, the coefficient of variation between areas,  $CB(A_k)$ , decreases asymptotically to zero. While the initial values of the  $CB(A)$  curve indicate the level of overall irregularity, the rate with which it drops asymptotically is determined by the correlation among the neighboring unit areas.

Non random appearance defects will exhibit patterns that are easily identifiable by the observer. Defining and measuring levelness as the departure of visual appearance of a dyed fabric from randomness will produce results in agreement with the user's expectation.

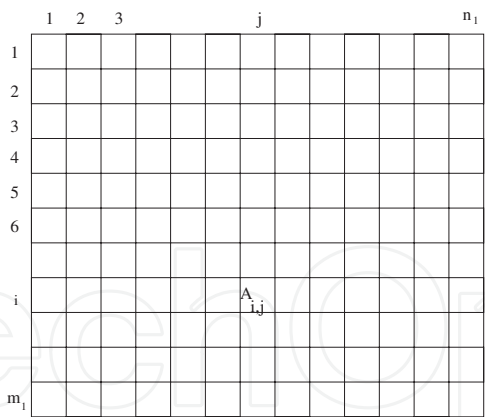


Fig. 16. Fabric matrix unit area 1x1

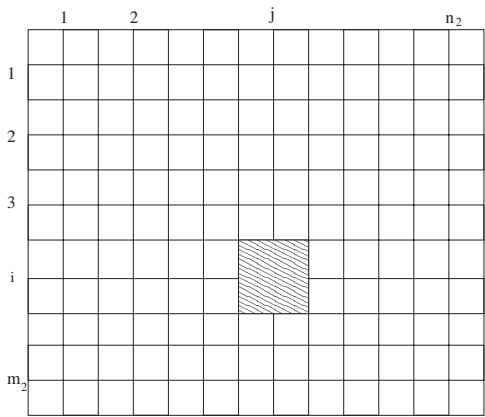


Fig. 17. Unit area 2x2

As a first step, the dyed RGB fabric image is converted into gray scale using the standard RGB to gray scale conversion protocol [GSV = 0.3\*Red + 0.59\*Green + 0.11\*Blue]. It must be noted however that variation in chroma may occasionally be lost during this conversion. The resultant image is then mapped to a fabric matrix as shown in Figure 16. The  $CB(A_1)$  is then calculated using the Equation 10. Let  $F_{i,j}$  be the gray scale value of a cell located at row  $i$  and column  $j$  where  $i = 1, 2, \dots, m_1$  and  $j = 1, 2, \dots, n_1$ . If  $\mu$  is the expected value and  $\sigma^2$  is the variance of the unit cells whose size is  $1 \times 1$  and value  $F_{i,j}$ , then the  $CB(A_1)$  is simply:

$$= 100 \frac{\sigma}{\mu} \tag{11}$$

If the gray scale values of the neighboring unit cells are independent from one another, when they are merged to obtain unit cells of size  $k \times k$  as demonstrated in Figure 17 for  $2 \times 2$ ,  $CB(A_k)$  equals to:

$$= 100 \frac{\sqrt{k^2 \cdot \sigma^2}}{k^2 \cdot \mu} \tag{12}$$

$$= 100 \frac{\sigma}{k \cdot \mu} \tag{13}$$

$$= \frac{CB(A_1)}{k} \tag{14}$$

The coefficient of variation between areas will decrease as the unit area size increases for a completely random and isotropic irregular fabric. This is also the theoretical limit when  $\sigma > 0$ .

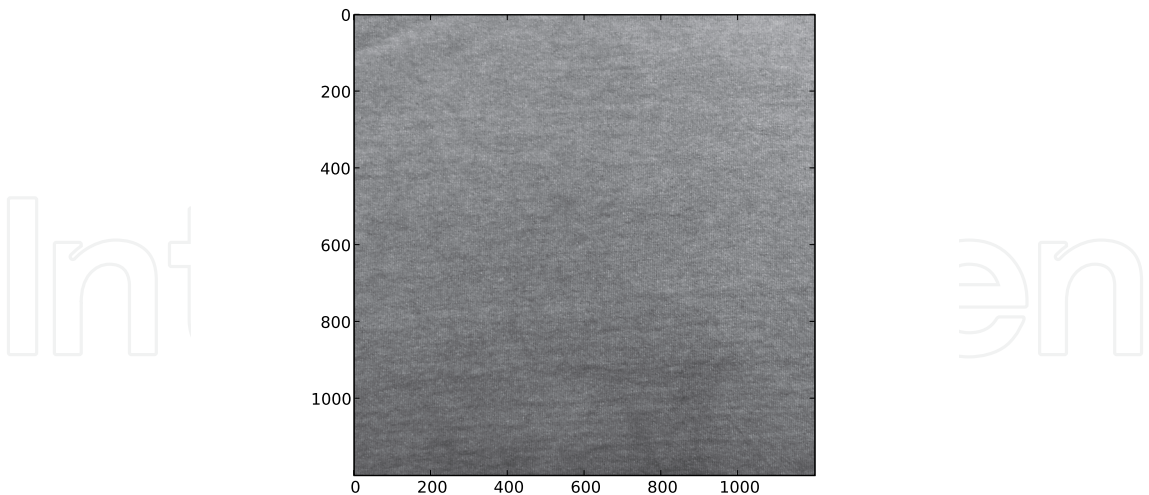


Fig. 18. Gray scale image of fabric sample 8

On the other hand, the  $CB(A_k)$  of a real fabric gray scale image matrix would likely produce a  $CB(A_k)$  curve above the theoretical limit.

Since levelness is considered a departure from randomness, one can obtain surface irregularity by calculating the ratio of the  $CB(A)$  curves of the actual fabric appearance and its theoretical limit as follows:

- 1. take  $k$  as  $1, 2, 3, \dots, N$  where  $N \leq \text{floor}(\text{minimum}(m, n)/2)$
- 2. calculate  $CB(A_k)$  for each  $k$
- 3. define surface irregularity (SI) as:

$$SI = \frac{\sum_{k=1}^{k=N} (100 \frac{\sigma}{k\mu})}{\sum_{k=1}^N CB(A_k)} \tag{15}$$

Note that unless the fabric is completely random  $SI > 1$ . Equation 15,  $k$  is regarded as the scale factor and  $N$  depends on the resolution of the image. Even though  $k$  was chosen to be one of  $1, 2, 3, \dots, N$  in this study, another set of scales may be chosen for optimum results as needed.

Figure 18 shows the gray scale image of a brown colored fabric sample 8. As seen from the image, the levelness information of the dyeing was not lost with the gray scale transformation. Actual and theoretical  $CB(A)$  curves corresponding to this fabric sample are overlaid as shown in Figure 19. Due to appearance irregularities that span over several pixels, the actual  $CB(A)$  curve does not decrease rapidly despite the increase in the measurement area. The surface irregularity suggested as the ratio of the area underneath the theoretical  $CB(A)$  curve over the actual one accounts for the nature of the irregularity demonstrated in Figure 18.

Figures 20 and 21 are the gray scale images of fabric samples 3 and 5. >From the gray scale image of fabric sample 3, it is clear that the dye was not evenly distributed. Overlaid  $CB(A)$  curves for these fabrics are shown in Figures 23 and 22. The theoretically projected

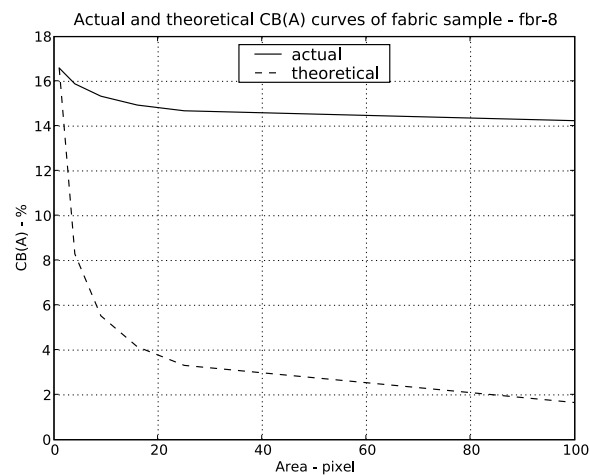


Fig. 19. CB(A) of fabric sample 8

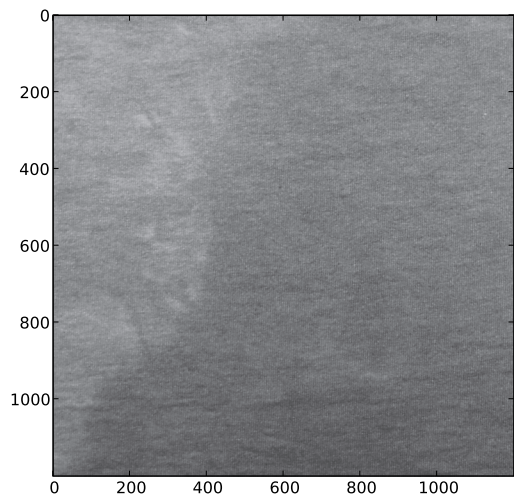


Fig. 20. Gray scale image of fabric sample 3

CB(A) curves do overlay perfectly on top of each other as shown in Figure 22; however, the calculated actual CB(A) curves shown in Figure 23 differ significantly due to practically the same levelness or irregularity of both fabrics at the micro level. But as grid size or scale increases, the levelness becomes more pronounced for fabric sample 3 shown in Figure 20 than fabric sample 5 shown in Figure 21. In other words, fabric sample 3 has irregularity that spans over many more pixels. The increase in the measurement area does not drop the corresponding CB(A) as rapidly as for fabric sample 5. If only the coefficient of variation for unit area size CB(1), 39.97 and 39.74 were used in determining the levelness of fabrics samples 3 and 5 respectively, the conclusions drawn would likely be erroneous.

In order to compare the performance of the proposed levelness measurement with the visual assessment of experts, 8 dyeing samples are identified. Five experienced observers in the field are asked to rate the quality of dyeing for levelness. Currently, there is no standard to visually assess levelness of dyeing. Considering the need to be consistent with the earlier studies and

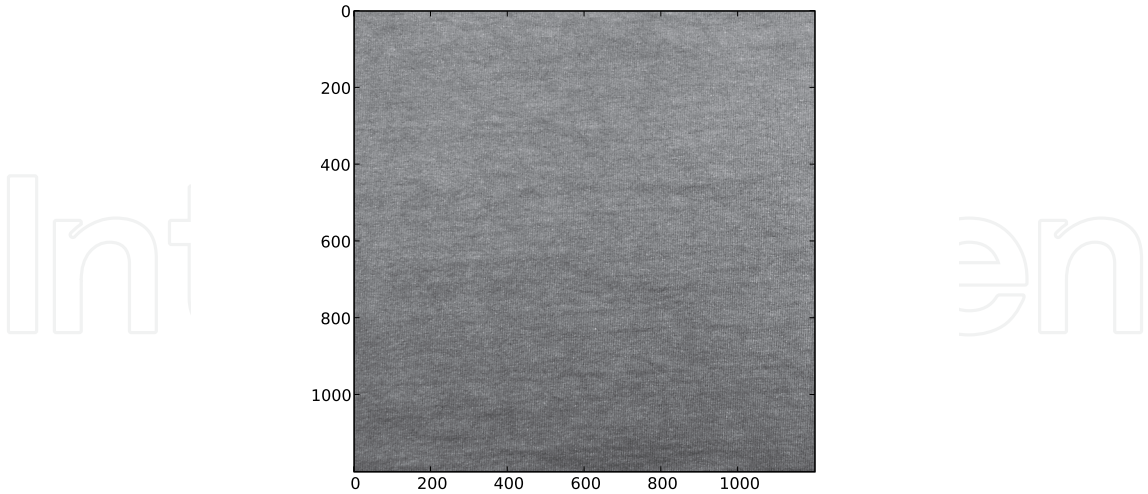


Fig. 21. Gray scale image of fabric sample 5

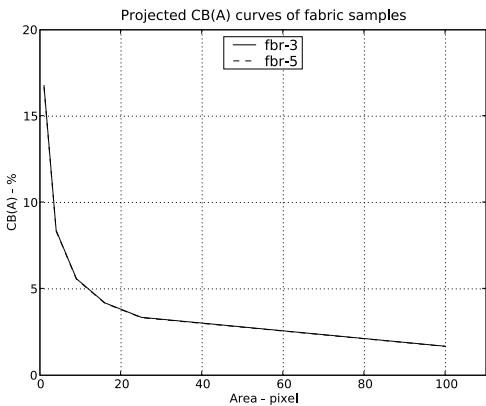


Fig. 22. Theoretically projected CB(A) curves

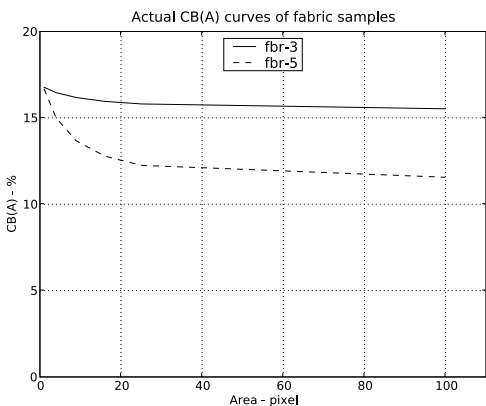


Fig. 23. Calculated CB(A) curves



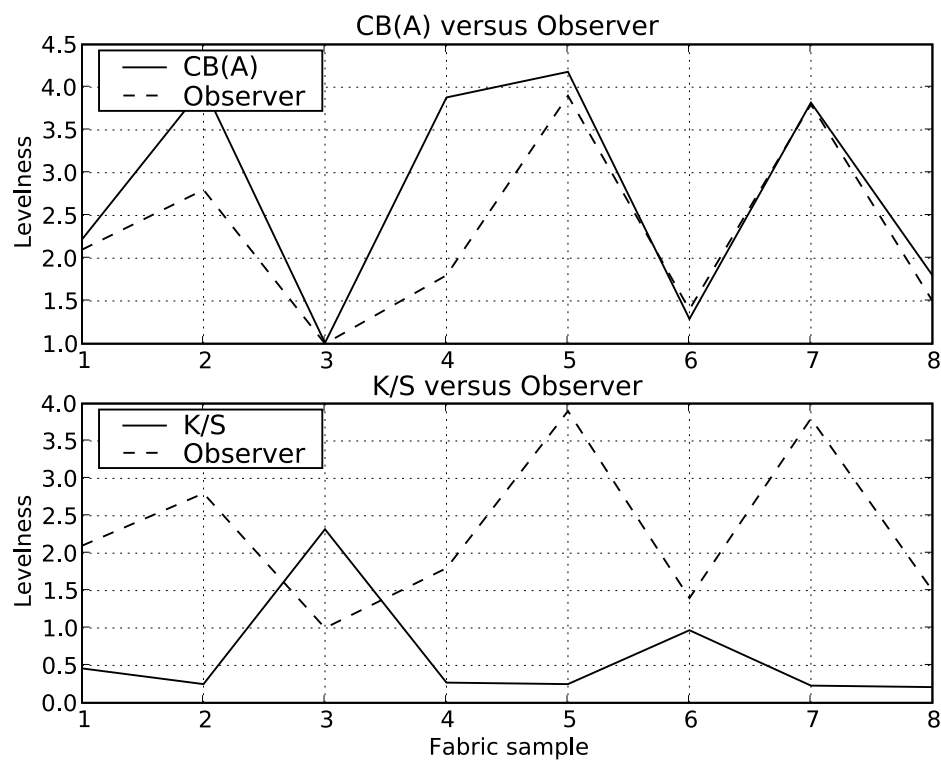


Fig. 24. Observer rating versus levelness calculated by CB(A) and K/S overlaid

to develop a method that is familiar to expert observers, it was decided that a grading system based on gray scale format similar to color difference evaluation would be appropriate. Meanwhile, using a custom transformation function  $44.12 * SI - 17.22$ , the surface irregularity calculated using Equation 15 is mapped between 1 and 5 in order to approximate the visual assessment of the observers.

To evaluate performance of the K/S, reflectance measurement is made using a Datacolor spectrophotometer. Fabrics are read at 4 random locations over the visible spectrum ( $\lambda = 400 - 700nm$ ). Levelness index (U) is calculated as follows:

$$U = \sum_{\lambda=400}^{700} S_r(\lambda)V(\lambda)$$

where  $V(\lambda)$  is the luminous efficiency at  $\lambda$ ,  $S_r(\lambda)$  is

$$= \frac{\sqrt{\sum_{\lambda=1}^n [(K/S)_{\lambda} - (K\bar{S})_{\lambda}]^2}}{n - 1}$$

(17)

and  $K\bar{S}$  is the sample mean of K/S at  $\lambda$ .

The results are given in Table 2 and demonstrated in Figure 24. The results show that the observer’s gradings agree more with the surface irregularity calculated based on the CB(A) than with the levelness index (U) determined based on the K/S values suggested by (Yang & Li, 1993). This is mainly because; 1) reflectance measurement with a spectrophotometer is highly sensitive to small changes in color, 2) the representative number

Sample #	Observer's Average	CB(1)%	U - K/S	Surface Irregularity
1	2.1	27.05	0.46	2.22
2	2.8	30.40	0.25	4.00
3	1.0	39.97	2.32	1.01
4	1.8	24.61	0.27	3.88
5	3.9	39.74	0.25	4.18
6	1.4	33.11	0.97	1.29
7	3.8	31.81	0.23	3.82
8	1.5	39.54	0.21	1.80

Table 2. Comparison of levelness obtained from the observer, K/S, CB(1) and SI

of sampling using a spectrophotometer is not as convenient as simply taking a picture of a fabric. Vast differences between the observer and the surface irregularity for samples 2 and 4 are due to outlier observer responses.

It is critical to note that the levelness for a large fabric could appear at different scales. Using the proposed methodology, technicians could tackle this issue in the following ways:

1. imaging of a much larger area of fabric using a higher resolution camera,
2. imaging of fabric at distant locations and merging them to form the fabric matrix shown in Figure 16,
3. imaging of fabric at distant locations, determining the levelness of these individual cells, calculating the mean and variance of the quantified levelness.

The methodology developed is practical and resistant to human subjectivity and fatigue since it involves:

1. capturing fabric images using a digital camera under standard lighting conditions,
2. analysis of the fabric sample images with software developed using the surface irregularity function suggested.

Unlike previous methods, the methodology suggested by the author (Günay, 2009) also deals with the issue of levelness at different scales; that is, at micro or macro levels. The superiority of the proposed approach is that the variation is plotted as a function of grid size. Varying grid size acts like varying resolutions or scales. The surface irregularity function proposed adds all these variations at different resolutions to produce a single number. Nevertheless, there will occasionally be cases where the surface irregularity obtained by adding variations at different resolutions will not be sufficient. This is because it is theoretically possible to arrive at the same surface irregularity index by adding a different set of numbers. Although this would seldom occur, researchers are encouraged to investigate and propose solutions to handle such cases.

4.5.3 Waste water check

After the dyeing is completed, collecting the waste water of the dyeing and rinses and measuring the dye amounts flushed allows one to determine the actual dye uptake of the fabric, or in dyers terminology, *the fixation*.

Table 3 shows the cumulative percent amount of dye flushed after each dyeing process for specific reactive dyeing. As given in Table, following the 5th rinse, 56% of the Yellow dye is wasted. By calculating fixation for each stage and analyzing it, one may be able to notice any significant deviation from the expected results and identify potential problems in dyeing.

Stage	Remazol Navy RGB	Remazol Red RB	Remazol Yellow RNL
Exhaustion	13.459	33.553	39.637
Rinse-1	18.513	44.042	51.709
Rinse-2	19.891	46.431	54.38
Rinse-3	21.388	48.215	56.41
Rinse-4	21.848	49.012	56.517
Rinse-5	22.001	49.451	56.09

Table 3. Cumulative dye wasted after each reactive dyeing process step.

5. Benefits of RFT quality control

The batch dyer incurs severe financial penalties if a proportion of batches are not dyed correctly the first time. A color correction added between 24% and 36% to the total dyeing cost of the initial dyed batch, depending on the dye substrate system and the stage of manufacture at which dyeing was carried out. Stripping and re-dyeing increased the initial dyeing cost by between 170% and 200%; however, these are direct costs that do not include indirect costs such as loss in revenue and profit (Park & Shore, 2007). Only a few dye houses worldwide have successfully achieved RFT production by implementing some of the quality control strategies mentioned in this chapter. Hence, there have been relatively few case studies published to describe these projects or to document the success achieved. A number of these dye houses in the US obtained more than 98% first-time-right dyeing by adapting the RFT technologies introduced here while achieving significant cost reductions. In addition to the cost savings obtained by RFT processing, there are several other benefits, including improved quality through shorter processing times, increased productivity from the same equipment and time frame, improved production planning, and less capital expenditure on processing equipment (Park & Shore, 2004). RFT processing is an essential prerequisite to the introduction of automation and robotics.

6. Conclusions

With the recent advances in spectrophotometer and computing technologies, it became feasible to practically and cost effectively measure the dyeing characteristics of dyes in a solution. In this chapter, methodologies are suggested to systematically monitor the quality of incoming materials, including dyes, chemicals, and fibers throughout the dyeing process. Being able to monitor quality control attributes during the dyeing cycle also increases profitability by reducing reworks. Furthermore, by measuring and quantifying quality attributes, the dyer-customer relationships are likely to improve by fast troubleshooting, correct dispute resolution and accurate technical information exchange. Although the concept of RFT was never the subject of formalized major R&D projects, it evolved through the foresight, determination, and ultimate success of a relatively few organizations over a considerable span of time in the dye- and machinery-making and - using industries. The author hopes this chapter will lead textile researchers and process engineers to develop additional quality control methodologies in the future, in addition to the ones suggested here.

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