### Real-Time Analysis and Control of Batch Dyeing Processes

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#### Abstract:

Work summarized in this final report covering three years of research includes: 1) A method for real-time monitoring of disperse dye exhaustion on polyester and acetate; 2) A method for real-time monitoring of both reactive dye exhaustion and hydrolysis; 3) A comparison of a newly developed method for real-time monitoring of indigo with the conventional titrametric method; 4) Models of direct dye exhaustion developed with information from flow injection analysis; 5) A novel color matching system based on a neural network; 6) Closed-loop control of acid dyeing of nylon with two dyes and with three dyes to give on-tone buildup; 7) Work to date on closed-loop control of a laboratory jet dyeing machine. We believe that this work has redefined the manner in which the industry will look at monitoring and controlling dyeing processes.

### Introduction:

The Dye Applications Research Group (DARG) at North Carolina State University was formed in 1988 and has been funded by several sources. For this report we have chosen to highlight DARG results from research sponsored by the National Textile Center since 1995. During this period, we have graduated one Ph.D. student (Crompton and Knowles) and three MS students (Burlington Industries and DyStar). Two more MS students will finish their theses this fall. These students have contributed to 18 publications, 14 technical presentations and one patent pending. This report will summarize the results of this work in the areas of dyebath monitoring, modeling and control.

Previously, our group has taken several elements from various disciplines to redefine the state of the art in textile process control. These include

a systems approach to manufacturing process control real-time, multi-channel control concepts from aerospace parametric dyeing models from theoretical kinetics and thermodynamics neural network models, modified to use sigmoid rather than 0,1 outputs fuzzy logic rule-based control strategies robust, real-time analytical methods

We brought these together to create a new thinking in textile control systems, while discarding the previously used concept of simply automating the manual process steps. This has been embraced by many in the industry as the way to go, and there is now a new way of thinking about textile process control. Using these ideas, it is possible to greatly reduce product variation with associated cost benefits. These methods are broadly applicable to many textile processes.

## **Progress in Monitoring Batch Dyeing**

In the course of this project, we have improved and expanded the capabilities of flow injection (FIA) analysis as a means of monitoring batch dyeing processes. FIA has been used to monitor exhaustion of reactive dyes, exhaustion of direct dyes, exhaustion of disperse dyes on cellulose acetate and polyester, and, in a modified version, to simultaneously monitor reactive dye exhaustion and hydrolysis (by high performance liquid chromatography, HPLC). The original

FIA system has also been modified so that it is portable. This mobile system has been attached to a 70-lb. pilot-scale jet dyeing machine and used to monitor exhaustion of direct dyes on that machine. To harden the portable system, it has been encased in a NEMA enclosure to protect it from the harsh environment of a production dyehouse floor. The following figures illustrate some of these uses of the FIA monitoring system. Figure 1 illustrates exhaustion of three direct dyes in a mixture. From the differences between this mixture exhaustion profile and a similar one for the individual dyes, it is obvious that the dyes behave very differently in a mixture. It is also apparent that the blue dye reproducibly desorbs to a small extent before salt is dosed into the bath.

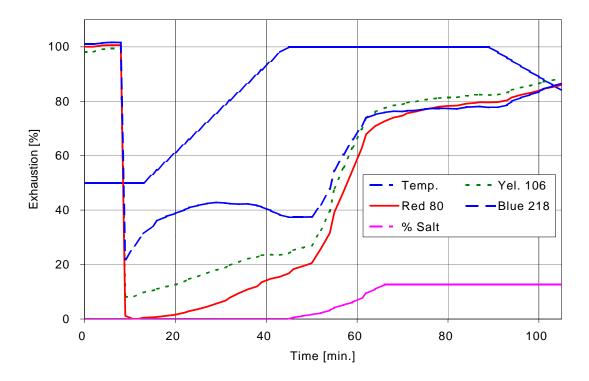


Figure 1. FIA Monitored Exhaustion of Mixture of Three Direct Dyes

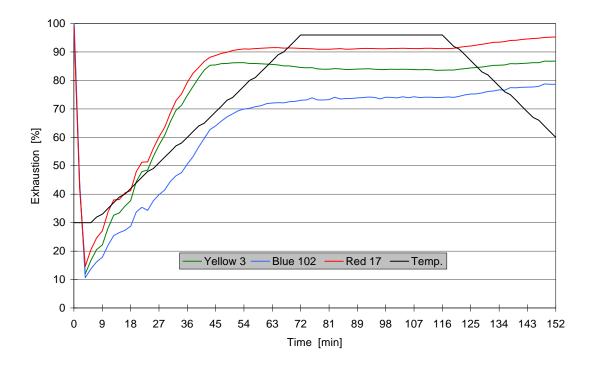


Figure 2. FIA Monitored Exhaustion of Mixture of Three Disperse Dyes on Acetate

Figure 2 shows the FIA monitored exhaustion of three disperse dyes on cellulose acetate. The yellow and red dyes follow similar exhaustion profiles and exhaust more completely than the blue dye. Exhaustion of all three dyes is slightly increased by the decrease in bath temperature.

The combination of FIA and HPLC creates a powerful tool for studying the behavior of reactive dyes in real-time (see Figure 3). To our knowledge, the system (Figure 4) we have developed is the only one in the world capable of giving on-line exhaustion and hydrolysis data during a reactive dyeing process. An example of typical data from a dyeing with a heterobifunctional (monochlorotriazine and vinyl sulfone) blue reactive dye is shown in Figure 3, which shows addition of dye to the bath followed by exhaustion to 80%. The chromatographic data show that the concentration of all of the reactive dye species decreases with time and the addition of alkali. It also shows that the dye remaining in the bath after exhaustion is primarily dihydrolyzed.

Using a modified FIA system, a real-time method for monitoring indigo concentration has been developed. This system has been used to monitor a simulated indigo dyeing process. As shown in Figure 5, the results are both more accurate and more precise than the standard titrametric method for determining indigo concentrations.

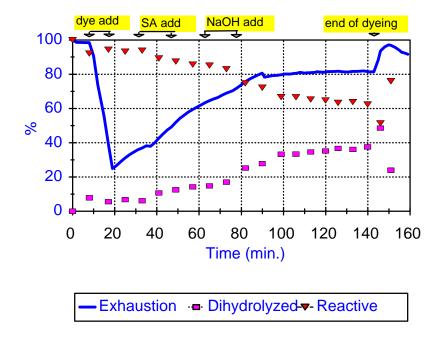


Figure 3. Exhaustion and hydrolysis of a blue reactive dye by FIA/HPLC at 140°F

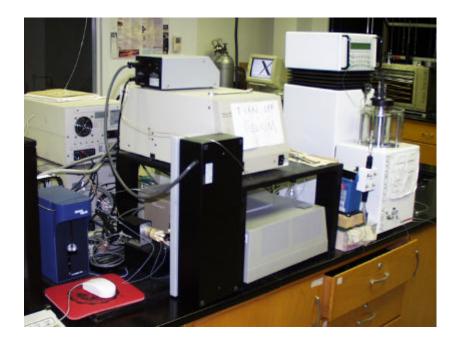


Figure 4. FIA/HPLC apparatus

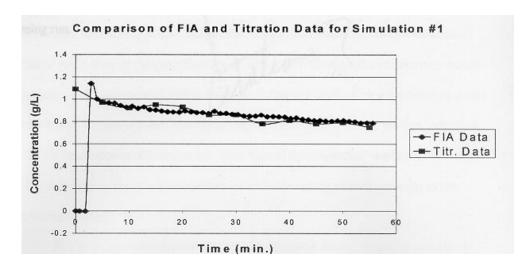


Figure 5. Indigo concentration in a simulated continuous dyeing

# **Progress in Modeling Batch Dyeing Processes**

We have worked with process models and control models to advance the state of the art in textile process control. Our methods are theoretical and also practical. As a development tool, they have been applied in many situations, but these are only examples, and the methods we have developed are broadly applicable to many processing situations including:

Dyes: Acid, Basic, Direct, Fiber Reactive, Indigo Machines: Lab and pilot plant, Batch and continuous

Substrates: Woven and knit fabrics, yarns

Fibers: Cotton, Polyester, Acetate, Nylon, Acrylic

We have evaluated and used many process and control modeling strategies, including advanced control models from aerospace, such as, neural networks, fuzzy logic, rule-based control models, and parametric models based on process chemistry and physics. Our approach to textile process control depends on several fundamentals, i.e. defining product attributes desired at the end of the process, sensing the system state at all times during the process, predicting the outcome from current state, and adjusting future process parameters to get the desired product. Integral to this is the need to model processes for several purposes, i.e., accurately forecasting the process outcomes, evaluating effects of possible process adjustments, evaluating control models in various situations, including normal process variations and major process excursions.

Our models have been developed with certain important factors in mind, such as, conformance to standards, flexibility, robustness, simplicity, accuracy and usefulness. Several major areas have been investigated with respect to modeling. These include models of the entire manufacturing process, the dyeing process, color and its relation to dyes and dye interactions control systems

Perhaps the most innovative and widely known models we have developed are engineering models of processes and information flow, such as the "fishbone diagram". This approach provides a solid foundation for the design of control and process models, and for loss function (cost) minimization by multichannel optimizing control strategies. For further information on

these models see references in our annual reports prior to 1995.

We have developed advanced models which describe and predict the kinetics, reactivity, affinity, exhaustion, fixation, and solution interactions of various dyes. These are integrated into our controllers and united with control models in several ways. First, our parametric models are useful to predict the outcome of processes, and to define physical and chemical parameters related to the control of product attributes. Second they are critical in defining loss functions (costs) for real-time process adjustments which are necessary to arrive at the lowest cost solution to a control problem. Third, they are useful in training the nonparametric models, such as, neural net, fuzzy logic and rulebased controllers. Some of our models are based on dimensionless groups, a technique, which recognizes the equivalence of various options for the control of processes. This is also important in determining the minimum cost solution to the control problem. Our most useful parametric models include the Nernst and Langmuir kinetic models, modified for dye-dye solution interactions, as well as a modified Donnan models for ionic dye equilibrium. The latter are implemented in our dosing control systems. For further information see references 5, 10, 14, 23, and 25.

Extensive work has been done on models of optical transmission, as related to dye concentration, as well as color matching using neural network models. This entirely novel approach to modelling is many times more accurate than the previously used Beer-Lambert Law models based on a linear matrix algebra approach. Our innovation in this area was twofold. First, we applied neural nets to the color matching and dye concentration determination problem successfully. Second, we developed a new approach to neural net modelling, which is based on non-linear sigmoid functionality. This takes dye interactions in fiber and in solution into account in a totally unique and very accurate way. Extensive investigation into these interactions by several researchers has led to color matching programs and dye concentration prediction algorithms of unprecedented accuracy. These are closely allied to our control models in the sense that the kinetics of the dye exhaustion, which is our control target, is intimately related to the solution interaction of the dyes, thus, the kinetic and optical data are highly correlated. For further information see references 8, 9, and 22.

Several control models have been investigated. These are described more fully under the "control" section of this report. In many cases these are advanced models of control strategies, which are imported from aerospace and other technologically advanced areas. For further information see the "control" section of this report.

### **Control Strategies for Batch Dyeing Processes**

Control of dyeing processes is a complicated function because of the variability in system parameters, the number of uncontrollable variables and the nonlinear effects of the system dynamics. We have studied several parametric and non-parametric control approaches for batch dyeing processes. Two approaches are discussed here. One approach uses output measurements of dye concentrations to generate an error for a proportional-integral control law. In the second approach, a neural network model is developed for color matching; back propagation with shocking improves the performance of the classifier, which may be used as a model for dyeing process control.

# **Closed-Loop Control of Nylon 66 Acid Dyeing**

One of the stated goals of the project was to develop a process to control in real-time the amount of dye transferred to the cloth. Our emphasis was to achieve an on-tone build-up of

shade in dyeings of polyamide fabrics with a three-dye mixture of C.I Acid Blue 25, C.I. Acid Yellow 49, and Nylantherene Red. The advantages of such a system are:

- 1. The ability to achieve shade repeatability between dye lots
- 2. The ability to prescribe and control the on-tone build-up of dye on the cloth
- 3. Obviate the need for a drug room or premixing of dyes
- 4. Reduction in the amount of auxiliaries in the dyebath
- 5. Optimization of the wash and rise cycles in batch dyeing to reduce water usage
- 6. Dyebath reuse and reduction of color and chemical effluent
- 7. Reduction of energy usage through isothermal dyeings
- 8. Improved color matching for "right first-time" dyeings

In the dyeing process, there are variables over which the dyer has control, such as the amount of dye in the dyebath, the time and temperature profiles, the mass of the cloth to be dyed, etc. We call those variables controllable variables. There are other variables over which the dver has little or no control, such as the water quality (amount of minerals, pH,) and the fabric (merge, variability, weave tightness). We call these variables uncontrollable variables. One of the advantages of a properly designed feedback control system is the ability to maximize the effects of the controllable variables on the dyeing process while minimizing the affect s of the uncontrollable variables. It is through the ability to monitor and control the batch dyeing process, described in the previous sections, that we accomplish this goal. Another important feature of our controller is that we do not dye to a predefined process specification, rather, we dye to a predefined product specification. The difference, although subtle, is a major departure from conventional dyeing. Normally, a dye recipe will prescribe a temperature profile, as well as volumes and times to add water and chemicals; but the most important factor of all, the amount of dye being diffused onto the fabric, is nether measured nor prescribed. In our controller, we predefine three desired exhaustion trajectories or exhaustion profiles, which describes how each dye should exhaust onto the fabric. These desired trajectories could be almost any smooth monotonically increasing function. For example, in Figure 6., we prescribed a linear trajectory for all three dyes to achieve an even on-tone 1% owg build-up in 80 minutes.

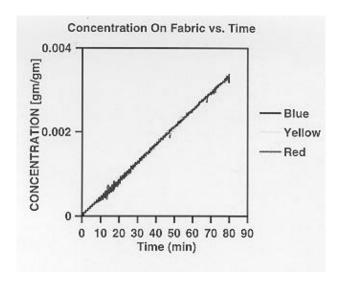


Figure 6. Controlled Acid Dye Buildup on Nylon

If all the dye were initially added to the dyebath, it would be quite difficult to control the exhaustion rate. A common approach is to add retarders to slow the initial strike rate. A more elegant approach is to dose each dye separately into the dyebath in the proper amount to insure that the exhaustion profile follows the desired one. Additionally, the pH is set 8.4 and decreased to 5.5 over the course of the dyeing to slow down the initial strike rate and increase the affinity of the dyes during the end of the dyeing. All the dyeings are done isothermally at 82° C.

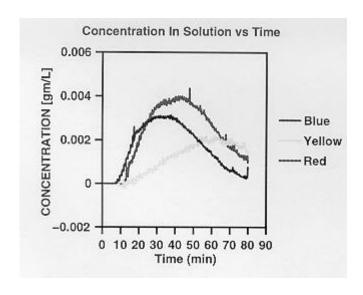
Using our direct dyebath monitoring system (shown in Figure 7), individual dye concentrations are determined every 20 seconds. From these measures, and

knowledge of how much dye was dosed into the system, the amount of dye on the fabric is determined. An error term is formed from the deviation of the actual dye concentration on the fabric from the desired one. This error term is fed into a PI (proportional integral) control law, which determines the amount of dye to be dosed to the system. Figure 8 shows a graph of the concentration of each dye in solution during a dyeing. Notice that although the dye uptake is linear, as shown in Figure 6, the amount of dye in solution is very non-linear, and changes depending upon the affinity of the dyes, which change with pH and dye concentration.



Initial results have been quite promising. A series of experiments are underway to determine the reproducibility of our system. With proper pretreatment and scouring, we have achieved variations of less than 1 ΔE\* between dyeings. Further experiments are being performed to determine dye penetration in fiber cross section, effects of variability in pretreatment on the dyeing process, and the effects of variability of the stock solutions on final shade.

Figure 7. Direct dyebath monitoring system



### **Color Matching Neural Network**

We have created an adaptive neural network that determines the dye concentrations needed to reproduce the reflectance spectrum of a fabric or dyed yarn sample. Our neural network technique is more accurate than conventional color matching techniques at consistently approximating the actual dye concentrations for a tertiary dye mixture. This is essentially the same task that is done by present color matching software, but obviates the need for tedious single dyeings.

Figure 8. Concentration of Individual Dyes in Solution During Dyeing

Up to now, we have studied only disperse dyes on polyester yarn samples, although our program can be modified to predict other dye classes and substrates, as well as recipe predictions for prints and digital printing using 4 to 8 dyes. We have developed a completely standalone neural network system which interfaces a Minolta or Data Color spectrophotometer with a Pentium PC running Linux, and will perform all the color matching, neural network training and maintenance needed to support the system.

One of the major reasons for deviations or poor correlation between lab, production and computer simulation is lack of control of the dyeing process itself. With our new dyeing method, we can control and determine how much dye is on the cloth at the end of a dyeing and use that information for color recipe formulation, instead of trying to calibrate the color matching system to the initial amount of dye.

### Acknowledgement

The members of DARG wish to express their appreciation to the Department of Commerce and the National Technical Center for supporting this multifaceted interdisciplinary project.

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