Abstract. Textile wet processes, particularly batch dyeing and finishing, consume vast quantities of water, posing significant demands on the water resources in regions where the industry is concentrated. Some dyebaths offer the potential for reuse, if appropriate adjustments are made for the residual dyes and their effect on product shades. If manufacturers successfully implement reuse, both the textile firm and the community will draw substantial benefits, including reduced consumption of water, energy, and chemicals and reduced wastewater effluent to be treated.

Current efforts at Georgia Tech involve development and demonstration of an automated system to analyze the spent dyebaths in a nylon carpet manufacturing process, permitting their reconstitution and reuse without additional labor or expertise. Process water requirements may be reduced by about 7,300 gallons per batch (> 55% savings) while reuse of chemicals and energy is estimated to provide a substantial savings of –4.3¢ per pound of carpet. The basic technology is applicable to a wide range of textile products.

INTRODUCTION

The textile industry uses more than 100 billion gallons of water per year, primarily due to dyeing and finishing processes for yarn, fabric, and carpet. The high concentration of carpet manufacturing plants in the Dalton, Georgia area imposes a significant demand on the water resources of the region.

In the conventional batch dyeing processes, the dyebaths are used only once, then discharged to the sewer. Single use is an inherently wasteful technique, and it results in the dyeing process being a major consumer of both water and energy. In addition, valuable auxiliary chemicals are lost with each batch of water, and significant loads are placed on the waste treatment system.

For some combinations of fibers and dyes, reuse of the dyebaths is possible and offers several potential benefits; however, there is a significant impediment. Although most of the dyes are taken up from the bath by the textile fiber, there is a small and variable amount of residual dye left in each bath. This residual dye is sufficient to lead to off-shade dyeing in subsequent cycles if the bath were reused in place of fresh water. The solution is to determine the residual dye quantity and to adjust the recipe to give the proper dye concentration for reuse.

BACKGROUND

Batch dyeing of nylon broadloom carpets is typically performed in an atmospheric beck (Figure 1). Water, chemicals, dyes, and the carpet are loaded in the beck, with the carpet in a loop about a rotating reel which provides the necessary agitation. A water recirculation loop maintains proper distribution of the chemicals and dyes in the bath and provides a convenient point for introducing water, chemicals, and dyes to the system. The bath is heated by direct injection of steam. A perforated baffle protects the moving carpet from the piping and from direct contact with the injected steam.

The standard dyeing process begins at the water supply temperature, with the bath being heated at a rate of 2-3°F per minute to a temperature in the range of 200-208°F, depending on the product. As the bath is heated, the dyes penetrate the fiber and form chemical bonds. The elevated bath temperature is held for a period to permit dyes to migrate to a uniform distribution over the carpet, giving a level dyeing. The bath is cooled by dilution with water before the carpet is removed and the bath drained.

Nylon fiber is generally dyed with acid dyes. Typically, three dyes are used in a dye recipe — a red, a blue, and either a yellow or an orange — with different concentrations and ratios of the dyes providing the various colors desired. Techniques have been developed through research projects at Georgia Tech over the past several decades which permit a spent dyebath to be analyzed for the residual concentration of each component dye. With this information, it is possible to...
calculate the necessary makeup amount of each dye to reconstitute the bath for the next batch, even if it is a different color, provided the same component dyes are used. In this manner, the water may be used numerous times while achieving on-shade dyeing of each batch.

Dyebath reuse has been successfully demonstrated on a production scale for several textile products (Cook et al., 1979; Tincher et al., 1980; Tincher et al., 1985). The process has not achieved commercial acceptance, however, because of human factors. Economic competitiveness requires that each dye cycle be closely followed by another in the same beck. A trained chemist must be immediately available to collect a sample of the spent dyebath, conduct a quantitative analysis, and perform the necessary calculations to adjust the recipe in time for the next dyebath.

While the direct expenses associated with adding chemists to the staff are justified by the associated process savings, the need to manage these employees' activities in minute detail in order to support the production schedule presents an undesirable administrative burden. In addition, having employees work under such tight time restrictions can lead to costly errors in either the chemical analyses or the recipe calculations. In the textile plants which implemented dyebath reuse, personnel difficulties and other administrative issues were sufficiently troubling that the process was discontinued in spite of favorable technical and economic results.

**AUTOMATED DYEBATH ANALYSIS**

Georgia Tech is conducting a program in conjunction with Shaw Industries and both state and federal sponsors to develop and demonstrate an automated dyebath reuse system for carpet manufacturing. By automating the process, the adverse human factors are eliminated. The automated system is interfaced with the plant's existing process control system and incorporates all of the required chemistry expertise in the hardware and software.

The technique for analysis of the spent dyebath is absorbance spectrophotometry, and the analysis system configuration is illustrated in Figure 2. A dual flow cell is used and permits a single light source to illuminate a sample of the dyebath and a sample of a reference solution which consists of water and all of the auxiliary chemicals in the same concentration as in the dyebath — everything except the dyes.

The light passing through the two samples is captured in optical fibers and carried to a dual-beam spectrophotometer which measures the light absorbance for wavelengths covering the visible spectrum. The absorbance spectrum for the reference is subtracted from the spectrum for the dyebath, providing the absorbance spectrum of just the residual dyes.

Absorbance is (theoretically) a linear and additive function of concentration of the component dyes (Beer's law), so the concentration of each of the dyes in the bath may be determined using calibration curves developed for the specific set of dyes. Causes of non-linearity and methods for responding to it in the analysis have been addressed by White et al. (1996).

Calibration curves have been developed for the acid dyes used at Shaw Industries plant #2 in Dalton, Georgia, where the demonstration is being performed. Data for the curves were obtained from analysis of laboratory-prepared solutions with concentrations ranging from 0.3 mg/L to 20 mg/L, covering the range expected in the spent dyebaths. Analysis of prepared mixtures of dyes used as “unknowns” indicates concentration measurement accuracy comparable to the accuracy obtained in weighing out the dye recipes in a production environment. Thus, the spent dyebath may be reconstituted and reused with satisfactory results in matching the specified product shades.

Application of the analysis system to the manufacturing process requires that the spent dyebath be captured at the end of the dye cycle, held for analysis and reconstitution, then returned to the beck. To support demonstration of the technology, a production beck at the Shaw plant was adapted as illustrated in Figure 3 to include a holding tank, transfer piping, and a sampling point. The wet-processing portion of the analysis system is contained in a wall-mounted enclosure next to the beck and holding tank. The optical fibers extend 400 ft. to the spectrophotometer and computer located in the beck control room.

**MODIFYING THE DYEING PROCESS**

Objectives of the development and demonstration effort include reduced water consumption, reduced environmental pollution, and energy and chemical conservation through efficient reuse of the dyebaths. These objectives should be achieved in an economically-attractive manner.
For reuse, the energy and reusable chemicals must be recovered in as concentrated a form as possible. This precludes the conventional process of cooling the dyebath by dilution at the end of the cycle. The dyebath must be transferred to the holding tank while it is still concentrated and hot, although the carpet itself must then be cooled with a rinse to permit handling. The subsequent batch which reuses the dyebath will start at an elevated temperature.

Both the hot start and the hot drop aspects of the new dye cycle present potential product quality problems, including unlevel dyeing, spot damage, and pile deformation. These potential difficulties must be addressed in order to achieve the desired objectives.

Through a series of in-plant tests, procedures were developed which were applicable to Shaw's nylon carpets and which incorporate hot startup and a minimum of dilution before the hot drop, without introducing quality defects. As automated dyebath reuse is introduced to other textile processes, similar product-specific development efforts will be required to assure that high product quality is maintained.

**RESOURCE RECOVERY AND REUSE**

The conventional process cycle consumes approximately 5,900 gallons of water and steam for dyeing and about 7,000 gallons of water for the dilution cooling. The modified cycle requires 5,100 gallons for rinsing (total for pre- and post-dyeing rinses) and reuses much of the water from the dyebath, giving a substantial reduction in total water usage.

Only about 5,100 gallons of the 6,600 gallons (~77%) of dyebath available at the end of the modified cycle can be recovered and reused productively. Because the bath is heated by steam injection, the condensate increases the water volume in the bath. In addition, there is some dilution, though greatly-reduced, in the revised cool-down procedure. Thus, there is more spent dyebath available for recovery than there is a use for in the subsequent batch.

For each dye cycle which processes 1,500 to 1,800 pounds of carpet, the projected water savings is about 7,300 gallons, including a combination of reused dyebath and a reduction of cool-down/rinse water. This represents more than 55% savings in total water consumption in the dyeing process.

Shaw plant #2 operates 16 of these becks with each beck processing about six batches per day, and plant #2 has just one of the many carpet dye houses in the Dalton area. The impact which extensive implementation of automated dyebath reuse could have on the water resources in the Dalton region is substantial.

Economic benefit to each carpet company will be most heavily realized in chemical and energy reuse. An anticipated 79% of both the auxiliary chemicals and the thermal energy in the bath will be recovered. This is slightly higher than the 77% total bath water recovery noted above, since a portion of the bath is recovered before the limited cool-down dilution. Initial estimates of heat losses in storage and transfer suggest only about 60% of the energy can actually be reused. These figures translate to savings of approximately 2.3¢ per pound of carpet for chemical reuse and 2.0¢ per pound of carpet for energy reuse. These represent substantial cost savings for this manufacturing process. These estimates will be updated based on results obtained in the demonstration.

**STATUS OF THE DEMONSTRATION**

At the time of this writing, the actual demonstration of automated dyebath reuse has not been completed. The system is installed and calibrated, and final software is being prepared for the plant's central computer to receive the data on dye concentrations in the recovered dyebath and calculate the required recipe adjustments for reconstituting the bath for the next scheduled carpet. Dyebath reuse demonstrations are planned for carpets of a variety of colors and styles, using both nylon 6 and nylon 6,6 fibers. If the demonstration runs are completed prior to the 1997 Georgia Water Resources Conference, the results will be discussed in the oral presentation.

Following the demonstration of automated dyebath reuse, the Pollution Prevention Assistance Division of the Georgia Department of Natural Resources will be leading a technology transfer effort to assure that the technology is disseminated to the carpet industry in Georgia and elsewhere. Plans for the transfer mechanism have not been finalized.

**FUTURE WORK**

After the completion of the current development and demonstration program, several additional phases of work...
are anticipated. First, it will be necessary to develop a commercial version of the automated analysis system so that the technology may be implemented on a wide-spread basis. Obstacles to commercialization include the need to solve issues specific to each plant, such as compatibility with hot startup and hot termination, and the need for the automated analysis system to interface with the variety of existing process control systems at different plants. Discussions are underway with a potential commercialization partner.

Second, the automated analysis technology should be developed and extended to other types of dyes and fibers, particularly disperse dyes for polyester, and to other textile products. Third, the automated analysis system offers potential applications beyond dyebath reuse. The system may be adapted to monitor dye concentrations during the dye cycle, providing a process control parameter not previously available (White et al., 1997).

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LITERATURE CITED


