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Full-Scale Demonstration of Textile Dye Wastewater Reuse

Sverdrup and Parcel and Associates, Inc. St. Louis, MO

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#### FULL-SCALE DEMONSTRATION OF TEXTILE DYE WASTEWATER REUSE

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#### NOTICE

This document has been reviewed in accordance with U.S. Environmental Protection Agency policy and approved for publication. Mention of trade names or commercial products does not constitute endorsement or recommendation for use. The finishing of textile products in the United States is estimated to result in the discharge of over  $3.8 \times 10^{11}$  liters of wastewater annually. Discharges from textile dyeing operations constitute a large fraction of this total.<sup>(1)</sup> In 1980, approximately  $3.5 \times 10^{9}$ kilograms of textile fibers were dyed, consuming  $7.8 \times 10^{7}$  kilograms of dyestuffs, and  $5.8 \times 10^{8}$  kilograms of auxiliary chemicals.<sup>(2)</sup> Most of the auxiliary chemicals remain in the dye liquor and are subsequently discharged with the spent dyebath. Although most of the dyestuffs are taken up by the product being dyed, typically 5 percent remain in the dye liquor and are also discharged.

Textile dyeing wastewater is therefore a high volume discharge, containing large amounts of inorganic and organic auxiliary chemicals, and is typically highly colored from the residual dyestuffs. About 80 percent of textile finishing mills discharge their wastewaters to publicly owned treatment works (POTWs). The remaining mills largely employ biological treatment prior to direct discharge.<sup>(1)</sup> Biological treatment, either by POTWs or by textile mills, is only partially effective in removing certain dyeing chemicals, especially dyestuffs.

A recent study by the U.S. Environmental Protection Agency's Air and Energy Engineering Research Laboratory examined technologies by which textile processing wastewaters could be recycled or reused, thereby reducing the amounts discharged. <sup>(3)</sup> One of these technologies, dyebath reconstitution and reuse, was investigated in detail and was found to be an environmentally beneficial and cost-effective technology. This paper presents some of the results of that investigation.

#### TEXTILE DYEING

Textile dyeing can be performed using either continuous dye ranges or batch dye machines. Continuous dye ranges are relatively expensive but are more efficient than batch machines in their usage of water, energy, and chemicals. Continuous dyeing is used primarily in the broadwoven and carpet segments of the industry where high volume, long color runs are more common. About half of all textile goods are dyed on continuous ranges. The balance of dyeing is performed in batch dye machines. Batch dyeing offers flexibility, short-run capability,

and ease of control. It is widely used in dyeing knit fabrics, carpet, stock, and yarn. Batch dyeing, however, is relatively inefficient in its usage of water, energy, and chemicals. By reconstituting and reusing the dyebath instead of discharging it after one dyeing, these efficiencies can be increased and, as a result, cause a reduction in the quantity of wastewater and pollutants discharged.

In a typical batch dyeing operation, water usage as dye liquor ranges from 8 to 40 liters for each kilogram of fiber dyed. Auxiliary chemicals and dyes are added to this dye liquor. Auxiliary chemicals can include buffers and pH control chemicals, wetting and dispersing agents, softeners, lubricants, and chemicals that affect the way the dye is taken up by the textile fibers. The total amount of auxiliary chemicals added will vary depending on the fiber and dyestuff types, but will generally range from a few percent to as much as 50 percent of the fiber weight. Dyestuff quantities are generally a few (less than 4) percent of the fiber weight. Following the addition of auxiliary chemicals and dyes, the dyebath temperature is raised to the desired dyeing temperature and held until dyeing is complete and a level dyeing is achieved The exhausted dyebath, now containing only a few percent of the original quantity of dyestuff but still most of the auxiliary chemicals, is discharged, and the dyed product is rinsed with fresh water.

#### DYEBATH RECONSTITUTION AND REUSE

Instead of the normal procedure of discharging the exhausted dyebath, a process modification can be made wherein the dyebath is reconstituted by adding the appropriate imounts of makeup dyes and auxiliary chemicals. The reconstituted bath can then be reused for dyeing a second batch of textile goods, resulting in significant auxiliary chemical, energy, and water savings. The reuse cycle can be repeated many times before the dyebath is finally discharged. See Figure 1.

An essential step in implementing dyebath reuse is to select product styles and shades that can be incorporated into a dyebath reuse scheme, since the residual dyes in the dyebath from the just-completed

dyeing must be the same ones that are to be used in dyeing the next shade. In many cases, this may mean that the plant would ref coulate dye recipes to utilize a small number of dyestuffs to optimize use of dyebath reuse.

Textile mills using batch dyeing operations are regarded as the major potential users of dyebath reuse. Dyebath reuse has been examined with a wide variety of textile products, fibers, and dyestuffs, so a large number of mills can potentially adopt this recycle technology. At many mills, not all production will be amenable to dyeing by dyebath reuse. A mill that is ideally suited to employ dyebath reuse will generally only dedicate half of its dye machines to reuse dyeing.

Dyebath reconstitution and reuse can be thought of as consisting of four parts:

- 1. Saving the just-exhausted dye bath.
- 2. Analyzing the bath for residual dyes.
- Reconstituting the bath by calculating and adding makeup quantities.
- 4. Reusing the bath for subsequent processing.

These four items are discussed in more detail below.

#### Saving the Exhausted Bath

Two basic alternatives are available for saving the exhausted dyebath The dyebath can be pumped to a holding tank while the product is rinsed in the same machine in which it was dyed. Then as soon as the rinsed product is removed, the dyebath can be returned to the dye machine for the next dyeing cycle. Alternatively, the dyed product can be pulled from the exhausted dyebath and moved to another machine for rinsing. This alternative eliminates the need for holding tanks and pumps but requires additional product handling and a spare machine or other equipment for rinsing.

#### Dyebath Analysis

The exhausted dyebath consists of water, and unused dyestuffs and auxiliary chemicals. The volume of water can be measured easily. The quantities of unused auxiliary chemicals can be estimated with sufficient accuracy by calculating simple mass balances. The quantities of unused dyestuffs, however, must be measured precisely to ensure that the proper shade is achieved in the next dyeing cycle. Analyses are performed using a technique developed by the School of Textile Engineering at the Georgia Institute of Technology. A spectrophotometer is used to measure visible light absorbance at predetermined wavelengths. These measurements are then used in conjunction with previously developed absorbance coefficients for each of the dyes to calculate the dye quantities.

#### Reconstituting the Bath

Reconstituting the bath consists of adding back the quantities of water, auxiliary chemicals, and dyestuffs needed for the next dyeing cycle. Water is added to replace any lost through evaporation, in the product, or by other means. Auxiliary chemicals are also replenished, generally in proportion to the amount of water added. Any auxiliary chemicals that exhaust during dyeing are added back to make up for such losses. Dyestuff makeup quantities are determined by subtracting the quantities present in the exhausted dyebath, as determined above, from the recipe quantities for the next shade to be dyed.

#### Reusing the Bath

Once the bath has been reconstituted and is in place with the product for the next dyeing, conventional dyeing procedures are used to complete the dyeing. Once the dyeing cycle is completed, the four items described above are repeated. Generally, the bath is reused for 4 to 10 dyeings prior to discharge.

#### FULL-SCALE DEMONSTRATION

To develop information on full-scale implementation, costs, and environmental benefits of dyebath reconstitution and reuse, a demonstration of the technology was performed at a carpet mill owned by Bigelow-Sanford, Inc. This mill performs batch dyeing primarily of nylon carpet.

Because of the risks involved in testing a new technology and the high dollar value of carpet dyed in a typical batch, bench- and

pilot-scale tests of this reuse technology were first conducted at Bigelow's laboratories in Greenville, SC. Two popular, large-volume carpet styles were selected for dyeing using dyebath reuse procedures. For the bench- and pilot-scale tests, six shades from each of the styles were used. Twenty-three series of laboratory dyeings were performed with dyebath reuse; each series consisted of 5 to 10 dyeings using the same dyebath.

For the first 12 bench-scale series, 5 dyeings of each shade were performed by first performing a conventional dyeing and then reusing the dyebath for the latter 4 dyeings. The latter four dyeings were then analytically compared to the conventional first dyeing. Results showed that acceptable shade matching had been achieved with dyebath reuse.

Following the success of these single shade dyeings, three additional bench-scale series were run in which the batches dyed from the same bath gradually progressed from light to dark shades, again with successful results. The value of these bench-scale tests was that they provided opportunities to become familiar with dyebath reuse concepts and procedures, to test the shade matching capability of reuse, and to resolve problems while still on a small scale.

Following the bench-scale dyeings, eight additional dyebath reuse series were conducted using a pilot-scale dye machine. The pilot-scale equipment and dyeing procedures provided an opportunity to test dyebath reuse under conditions that approximated full-scale dyeing. Both single- and multi-shade series were dyed for each carpet style. The shade matching, levelness, and colorfastness results (primary measures of product quality) of the dyed samples were very good.

Based on the success of these laboratory dyeings, the decision was made to conduct a week-long, full-scale demonstration of dyebath reuse at Bigelow's Summerville, GA, plant. Reuse was achieved by using a temporary pump and piping arrangement set up for the demonstration. At the end of a dyeing, the exhausted 20,800-liter dyebath was pumped to an adjacent dye machine already loaded with carpet for the next dyeing. The dyed carpet in the first machine was then rinsed and pulled in the normal fashion. Meanwhile, a sample of the exhausted dyebath was

analyzed, and the amounts of dyes and chemicals to add for the next dyeing were calculated.

The same two grades of nylon carpet that were used in the bench- and pilot-scale experiments were selected for the full-scale dyeings. Following some initial difficulties caused by an unanticipated change in the carpet yarn lot being dyed, 2 multi-shade dyeing series consisting of 6 and 10 dyeings, respectively, were conducted. All the dyeings were of first quality and were done without any processing above normal requirements.

During the full-scale dyeings, the process was monitored carefully to permit calculation of the savings in water, energy, dye, and chemicals attributable to dyebath reuse.

The calculated savings averaged \$23.85 and \$28.60 per dye cycle for the two carpet styles, or about \$0.025/kilogram of carpet. About 65 mercent of these savings were due to reduced auxiliary chemical requirements. Energy savings accounted for another 20 percent. Water and sewer use savings accounted for the remaining 15 percent. Based on these data, annual savings of \$30,000 are projected for each use machine converted to dyebath reuse. Future optimization of the reuse dyeing procedure could easily increase the realized cost savings even further.

Capital costs for equipping two dye machines at this mill for dyebath reuse were estimated to be \$70,500. This cost includes a pump, an elevated 22,700-liter storage tank, piping, valves, controls, and analytical equipment (including a spectrophotometer and computer). With an allowance of \$10,000 for developmental costs, and estimated operating and maintenance costs of \$5,000 per year, the net payback period for instituting dyebath reuse at this mill is estimated to be 1.5 years. For other mills wishing to use this technology, typical payback periods will range from 0.5 to 2 years.

#### USER MATERIALS

To assist other mills in implementing dyebath reconstitution and reuse, a user's manual and a dyebath reuse computer program were developed as a part of this study.<sup>(4)</sup> The objective of the user's guide is to present in a logical sequence the information needed by mill

personnel to confidently evaluate and implement dyebath reuse at their dyebouse. A straight-forward explanation of the principles of dyebath reuse is developed. The factors to be considered in determining the applicability of dyebath reuse to a given mill are discussed, and the elements of an evaluation/implementation program are described. Detailed procedures and descriptions of necessary equipment and their use are presented.

A number of mathematical exercises, such as solving simultaneous linear equations, performing data conversions, and computing makeup quantities, are required in dyebath reuse. These calculations must be performed quickly and accurately to maintain dyehouse production schedules. As part of this study, an automatic data processing system, based on a desk-top computer, was developed. The dyebath reuse computer program, written in BASIC, is capable of performing the various computations necessary for dyebath reuse. It is designed to guide the operator through the various steps, and it gives the operator an opportunity to verify all the information he has input. This program, as well as a discussion of the required pieces of computer equipment, is presented in the user's guide noted above.

#### ENVIRONMENTAL DATA AND RESULTS

The environmental benefits of dyebath reconstitution and reuse were quantified by taking samples of the dyebaths during each series of pilot- and full-scale dyeings. These samples were analyzed for BOD and COD, as measures of the organic content of the dyebath; for alkalinity and total phosphorus, as measures of the buffering capacity of the dyebath (MSP and TSP buffering system); and for suspended (TSS) and dissolved solids (TDS). Samples were taken both during the pilot-scale experiments and the full-scale demonstration.

#### Pilot Scale Results

Several important observations were made in reviewing the data from the pilot-scale dyeings. First, the concentration of organics (as measured by BOD or COD) increased as the dyebath was reused. Additionally, the concentration of dissolved solids showed a similar increase. Figure 2 shows the concentration increase in a typical series of dyeings – Several possible causes of these increases were postulated:

- o buildup of auxiliary chemical concentrations
- o evaporation losses from the dyebeck
- o buildup of carpet fiber impurities

The concentration of total phosphorus (measuring MSP and TSP) remained about constant as the dyebath was reused. This suggests that the increase in organic and dissolved solids was not caused by a buildup in the levels of auxiliary chemicals in the dyebath. Evaporative losses were largely made up with steam condensate, again suggesting that this was not the cause. It was concluded that impurities from the carpet fibers were building up in the dyebath as it was reused.

Even though the concentrations of various pollutants did increase as the dyebath was reused, the total amounts of pollutants discharged as a result of incorporating dyebath reconstitution and reuse were less than the amounts from a similar number of conventional dyeings. This is shown in Table 1. Reductions of 33 to 36 percent were noted for organics (BOD and COD), 30 percent for TSS, 48 percent for TDS, and 74 percent for total phosphorus.

#### Full-Scale Data

It was recognized that several factors will affect the actual reduction in discharge under full-scale conditions. These include the length of the reuse series (number of dyeings before discharge), the amount of "blowdown" from each cycle in a series, and the addition of cooldown water to the dyebath following dyeing. Therefore, data were collected after each dyeing in the full-scale demonstration to confirm the findings noted above.

The full-scale data, see Figure 3, show a general, but smaller increase in the concentrations of organics (COD) and TDS as compared to the pilot-scale data. Again, the sampling data for alkalinity and total phosphorus showed that these concentrations remained relatively constant.

The actual reductions in wastewater and pollutant discharges experienced in the full-scale dyeings, as shown in Table 2, were also

smaller than those observed in the pilot-scale dyeings. Reductions c about one-third were again noted for organics, but somewhat smalle. reductions in TDS and total phosphorus were seen compared to the pilot-scale data.

The smaller buildup in pollutant concentrations and the smaller reductions in pollutant discharge for the full-scale dyeings can both be explained by the use of more cooling water in the full-scale dyeings than in the pilot-scale dyeings. This resulted in more dilution of the dyebath before reuse, and consequently less recycle (in a given volume of dyebath) of auxiliary chemicals.

The amount of TSS discharged, as shown in Table 2, actually increased as a result of dyebath reuse with Carpet Style 2. Such an increase did not occur in other full-scale reuse dyeings or in the pilot-scale dyeings. At present, the cause of this increase remains unexplained.

Finally, an analysis was made of the pollutant reduction after a given number of reuse dyeings as compared to the same number of conventional dyeings. Figure 4 shows the results. A series length of at least five dyeings appears necessary to maximize the environmental benefits of dyebath reuse. Any length series, however, offers reductions in the discharge of wastewater and pollutants.

#### SUMMARY AND CONCLUSIONS

The full-scale demonstration of dyebath reuse showed that up to 10 dyeings could be performed with recycled dye liquor without affecting product quality. All carpet produced was first quality and required no additional processing above carpet dyed conventionally.

Dyebath reuse resulted in significant operating cost savings. The savings were primarily a result of lower auxiliary dyeing chemical usage. Smaller savings resulted from water and energy use reductions. Because this reuse technology has a relatively low capital cost, the savings result in a short payback period, typically less than 2 years. To assist the large number of mills that could potentially use this technology, computer software and a dyebath reuse operations manual were developed.

Significant environmental benefits of dyebath reuse were documented Although pollutant concentrations in the dye liquor did increase as the dyebath was reused, the overall result of dyebath reuse was to decrease both the volume of wastewater discharged and the mass of pollutants discharged. Long dyebath reuse series are not needed to maximize the environmental benefits of reuse. In general, a series of five or more dyeings results in maximum environmental benefits.

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# TABLE 1POLLUTANT LOADINGS FROM DYEBATH REUSEPILOT-SCALE DATA FOR CARPET STYLE 2

Pollutant (g/kg)	Pollutant Loading			
	Conventional	Dyebath Reuse	Reduction, %	
BOD	22	14	36	
COD	80	54	33	
TSS	0.2	0.14	30	
TDS	40	21	48	
Total Phosphorus	1.9	0.5	74	

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#### TABLE 2

### POLLUTANT LOADINGS FROM DYEBATH REUSE FULL-SCALE DATA FOR CARPET STYLE 2

Pollutant	Pollutant Loading			
	Conventional	Dyebath Reuse	Reduction, %	
Flow (1/kg)	54.9	36.0	34	
BOD (g/kg)	36	24	33	
COD (g/kg)	96	64	33	
TSS (g/kg)	0.23	0.78	0	
TDS (g/kg)	49	28	43	
Total Phosphorus (g/kg)	1.8	1.0	44	

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## Typical Batch Dyeing Sequence

- 1. Load Product
- 2. Add Water
- 3. Add Chemicals
- 4. Add Dyes
- 5. Heat to Dyeing Temperature
- 6. Hold at Dyeing Temperature
- 7. Drop Dyebath to Sewer
- 8. Rinse Product and Remove

## Dyebath Reuse Sequence

- 1. Load Product
- 2. Add Water (Make up)
- 3. Add Chemicals (Make up)
- 4. Add Dyes (Make up)
- 5. Heat to Dyeing Temperature
- 6. Hold at Dyeing Temperature

Analyze

- 7. Save Dyebath-----
- 8. Rinse Product and Remove

# Figure 1 Comparison of Conventional Dyeing with Dyebath Reuse



FIGURE 2 POLLUTANT CONCENTRATION vs DYEING CYCLE PILOT - SCALE DATA







# FIGURE 4





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