RECOVERY OF HOT WATER, DYES AND AUXILIARY CHEMICALS FROM TEXTILE WASTESTREAMS*

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SUMMARY

The recovery of hot water, dyes and auxiliary chemicals from a continuous dye range has been demonstrated using a full-scale dynamic membrane hyperfiltration system. The recovered hot water has been suitable for reuse greater than 99% of the time. The reuse of recovered dyes and chemicals depends on the compatibility of the dyes and chemicals in the concentrate with the dyes and chemicals required for various production formulations.

INTRODUCTION

Hyperfiltration is one of the most promising methods developed in recent years to treat hot water and recover the heat value in the water. The development work leading to the hyperfiltration (HF) demonstration project described in this paper has been reported in previous publications [1–3]. The system has been in full-scale operation for more than a year. The purpose of this paper is to illustrate the HF system, its function in the recycle process, and the initial effect the recycled water has on the finished fabric.

To provide a critical test for the hyperfiltration system, LaFrance Industries, located in LaFrance, SC, was selected as the installation site. This plant is a dyeing and finishing plant processing almost exclusively automotive and upholstery fabrics. Because of this, the standards for color shade and light-

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fastness are very demanding and therefore provide a critical test for a reuse system. In addition to conserving energy and water, the recovery system would reduce the quantity of wastewater which would need to be treated for disposal to the receiving stream.

RANGE PROCESS AND EFFLUENT CHARACTERISTICS

The finishing range consists of a dye applicator pad, spiral atmospheric steamer, and a washing section, shown schematically in Fig. 1. It is used for dyeing, bleaching, and scouring a variety of velour fabrics. The washing sections contain, in sequence, a jet washer, a dip box, and two rotojet washers. The range operates three shifts per day, five to seven days per week, at speeds of 9 to 36 m/min selected as required by the process. Cotton, acrylic nylon, rayon, and polyester fabrics and their blends are processed. Although several classes of dyes are used (direct, disperse, acid, basic, and reactive) the

Fig. 1. Schematic of recovery system and hyperfiltration system.
types of auxiliary components in the wash water effluents are common to most dye formulations. The dye baths contain dyes, a thickener, surfactants and in some cases, dye solvents. The concentration of these components in the wash water will depend on the component concentration in the dye pad formulation. While about 85% of the dyes are exhausted on the fabric, the remaining dye and most of the auxiliary components are removed by the washing process. Analyses of composite effluents from the washing section are presented in Table I.

### TABLE I
CHEMICAL ANALYSIS OF DYE RANGE RINSEWATERS, LaFRANCE INDUSTRIES

<table>
<thead>
<tr>
<th></th>
<th>Minimum</th>
<th>Maximum</th>
<th>Average</th>
</tr>
</thead>
<tbody>
<tr>
<td>Alkalinity, mg/l</td>
<td>50</td>
<td>1950</td>
<td>390</td>
</tr>
<tr>
<td>BOD₅, mg/l</td>
<td>45</td>
<td>820</td>
<td>280</td>
</tr>
<tr>
<td>COD, mg/l</td>
<td>330</td>
<td>4530</td>
<td>1350</td>
</tr>
<tr>
<td>Conductivity, µmho/cm</td>
<td>150</td>
<td>7700</td>
<td>1570</td>
</tr>
<tr>
<td>Color, ADMI</td>
<td>200</td>
<td>9890</td>
<td>1710</td>
</tr>
<tr>
<td>Hardness, mg/l</td>
<td>5.6</td>
<td>320</td>
<td>50</td>
</tr>
<tr>
<td>pH</td>
<td>5.1</td>
<td>11.8</td>
<td>7.5</td>
</tr>
<tr>
<td>Phenols, mg/l</td>
<td>&lt; 0.005</td>
<td>0.547</td>
<td>0.103</td>
</tr>
<tr>
<td>TOC, mg/l</td>
<td>72</td>
<td>1490</td>
<td>340</td>
</tr>
<tr>
<td>Total solids, mg/l</td>
<td>450</td>
<td>6570</td>
<td>1860</td>
</tr>
<tr>
<td>Suspended solids, mg/l</td>
<td>9</td>
<td>350</td>
<td>65</td>
</tr>
<tr>
<td>Volatile solids, mg/l</td>
<td>240</td>
<td>2650</td>
<td>960</td>
</tr>
<tr>
<td>Calcium, mg/l</td>
<td>0.7</td>
<td>7.4</td>
<td>3.5</td>
</tr>
<tr>
<td>Copper, mg/l</td>
<td>&lt; 0.3</td>
<td>&lt; 0.5</td>
<td>0</td>
</tr>
<tr>
<td>Iron, mg/l</td>
<td>&lt; 0.1</td>
<td>9.16</td>
<td>1.0</td>
</tr>
<tr>
<td>Magnesium, mg/l</td>
<td>&lt; 0.4</td>
<td>24.9</td>
<td>5.3</td>
</tr>
<tr>
<td>Manganese, mg/l</td>
<td>&lt; 0.05</td>
<td>0.44</td>
<td>0.08</td>
</tr>
<tr>
<td>Nickel, mg/l</td>
<td>&lt; 0.2</td>
<td>0.4</td>
<td>0.08</td>
</tr>
<tr>
<td>Zinc, mg/l</td>
<td>&lt; 0.01</td>
<td>3.9</td>
<td>0.53</td>
</tr>
</tbody>
</table>

**RECOVERY PROCESS**

The system used to recover chemicals and renovate water for reuse in the washing section is shown in Fig. 1. The hyperfiltration unit produces a clean permeate water for reuse in the washing section and a concentrated aqueous residue. The chemicals recovered in the concentrate may be reused in pad formulations or disposed of in the existing wastewater treatment plant.
During normal operation there is a period of time between each run for cleaning the equipment and reloading the dye pad. Even though the fabric flow stops between production runs, the recycle rinsewater flow is continued so that additional purification of the water by the hyperfiltration unit is obtained. When the run is long enough, the concentrate has the characteristics of the specific dye formulation being run and can be treated in the hyperfiltration system as a batch containing components from a single dye run. The residue remaining in the dye pad can be combined with the HF concentrate mixture. If laboratory tests show that it is compatible with subsequent runs it may be used for pad bath formulation.

To be suitable for reuse, an average of 97% of the dye must be removed from the wastewater by the hyperfiltration (HF) system. In rare instances when light shades must follow dark shades in a normal production schedule, it may be necessary to use fresh water in the washing section. This will remove residual color from the wastewater reservoir and flush out the recovery system. In addition, the build-up of non-colored auxiliaries must not be sufficient to impair fabric performance standards set by the customer.

The HF concentrate contains dyes at concentrations much lower than those in the dye pad solution, but comparable concentrations of the auxiliary chemicals. Based on pilot studies, reuse of the concentrates in dye formulation is feasible with about 75% savings in auxiliary chemicals and about 10–20% in dyes depending on the dye class [1–2]. Possible reuse of the residual pad dye liquor and HF concentrates depends on the results of fabric performance such as crock, light and washfastness test. When experience shows good reuse of the recovered concentrate, significant savings can be obtained. Experience has shown that the reuse of dyes and chemicals will be determined by run length and compatibility of dyes and chemicals from different fabric styles. This can be optimized by careful scheduling of production dye runs and experience gained with reuse.

HF UNIT DESCRIPTION

Range wash water effluent is collected at a rate of 250 m³/d (46 gpm) during the dyeing runs in the 23 m³ (6,000 gals) accumulator tank. This tank serves two purposes. The first purpose is to allow two hours of wastewater retention while changes or maintenance is performed on the HF unit. Secondly, fluctuations in wastewater flow can be accommodated without changing flow to the HF unit.

The permeate storage (rinse water) tank is also 23 m³ to accommodate fluctuations in operating conditions and fresh hot water requirements. In normal operation, the rinse tank is nearly full of clean hot water and the accumulator tank is nearly empty.

The HF unit, shown in Fig. 2, is a Single-Pass (patented) [4] system con-
sisting of zirconium oxide-polyacrylate (ZOPA) membranes dynamically formed [5] on the interior of sintered stainless steel support tubes, arranged in ten modules. Characteristics of ZOPA membranes have been reported previously [2]. The total membrane area is 139 m\(^3\) (1,500 ft\(^2\)) pressurized by a positive displacement pump fed from the accumulator tank. The positive displacement pump forces a constant feed flow rate into the membranes. The concentrate flow rate is controlled so the inlet pressure varies to a set upper limit at which an automatic bypass valve bleeds feed to the accumulator tank reducing the feed flow to the HF unit.

There is also a 1 m\(^3\) (250 gal) tank, not shown in the basic schematic diagram (Fig. 1), following the accumulator tank which permits recycle of a solution for cleaning the membranes.

The interaction between the dye range and the recovery system occurs through a control panel located near the existing range control panel. The range operator selects the appropriate range operating mode; normal wash, high flow, or water off. In the normal wash mode, the water flow rate to the range is controlled from the control panel. Water is delivered from the accumulator tank or from the plant process water source as required. Switching to or from the plant process water source occurs automatically with the rinse tank designated as the preferred source. The high flow mode allows the flow controller to be bypassed to deliver a maximum flow rate to the washing sec-
tion. The water off mode stops water flow to the range. An interrupt can be used with any of the modes to discontinue an operation.

Pretreatment of the HF feed consists of a 40 mesh screen to remove link and other large particulate matter. A pH control is incorporated to permit recycle of the bleaching wash water as dye rinse water.

HF PERFORMANCE

The size of membrane surface area for the HF unit was designed from pilot test data to treat wash water at a process temperature of 85°C with color removal of at least 97% at high volumetric recycle [1]. The performance characteristics of ZOPA membranes in pilot testing of range effluent have been reported [1, 2].

Installation of the HF unit was completed on May 1, 1981. For the past year the HF unit performance, recycle efficiency, range product quality, and economic evaluation have been carefully monitored. The HF unit is used routinely for full-scale permeate cycle. Performance experiments have occupied some of the operating time. Regenerative heat exchangers have been installed to allow operation of the HF at 85–95°C while the range effluent temperature is varied from 55–85°C. A maximum inlet pressure of about 8.5 mPa (1,230 psig) is being used. Water recoveries of up to 90% are being used, depending on range speed, to return concentrate at dye pad strength. There has been no effect on fabric quality as determined by the normal product quality control. Some permeate from very concentrated dye formulations are considered unsuitable for reuse by the range operator and rejected. A full-scale reuse of the concentrate has been conducted on the range. Fig. 3 presents representative performance characteristics of the HF unit.

![Fig. 3. LaFrance single pass hyperfiltration system pressure profile modeled vs measured.](image-url)
REUSE TESTS

Laboratory reuse tests were used to determine the suitability of the product water for reuse prior to the implementation of full-scale reuse. The laboratory test consisted of stirring four 3 x 3 inch fabric samples in 400 mls of product water for 5 mins at 60°C. The fabric samples utilized were representative of fabric samples finished at LaFrance and included the following compositions: (1) 100% cotton, (2) 65% cotton, 35% rayon, (3) 41% cotton, 59% acrylic, and (4) 68% cotton, 15% rayon, 17% polyester. After the 'rinsing' test, the fabrics were dried and evaluated for staining on an AATCC chromatic transference scale against untreated fabric samples. On this scale, a value of 5.0 is assigned to an unstained sample and 1.0 is assigned to a heavily-stained fabric sample. A value of 4.0 was arbitrarily set as an acceptable level for product reuse. All product samples, with the exception of a few product samples generated from dark shade direct dye/basic dye formulations were judged to be suitable for reuse.

Using the laboratory data as a reuse guide, full-scale product water reuse was initiated on the dye range. Some dark shade direct/basic formulation rinsewaters were rejected for reuse since the laboratory tests indicated that these products would stain light shade fabrics. This problem could be partially solved by scheduling production runs of light to dark shades.

Additional laboratory testing was conducted to determine if color measurement could be used as a basis for rejection of product water for reuse. In these tests, 'synthetic' product waters were prepared by adding various amounts of direct and basic dyes to LaFrance process water. These product waters were then used to rinse light shade fabrics dyed on the LaFrance laboratory dye range. Each fabric sample was rinsed in a series of synthetic product waters consisting of various concentrations and combinations of direct and basic dyes. Both the fabric samples and the rinsing solutions were colorimetrically evaluated on a scanning spectrophotometer. The objectives of this testing program were to determine the maximum amount of residual color that could be tolerated for product reuse on critical (light) shades, and to develop a fast and accurate colorimetric test for rejecting product waters for reuse. This test would have to consider both the residual color in the reuse water as well as the shade depth of the fabric to be rinsed.

The results of this test program were mixed. While it was possible to determine at what color level certain dyes interfere with product reuse on light shades, it was not possible to apply the color matching procedures to all shades and dye classes. At this time it appears that additional experience with rinsewater reuse will be required to develop more refined and exact methods of determining if specific product waters can be reused on specific light shades.
CONCLUSION

Pilot studies and preliminary results of the full-scale HF recycle system continue to indicate high potential for recycle operation with reuse of water and chemicals. The membrane stability, flux, and rejections are satisfactory. Recycle of 88—96% of the wash water has occurred thus far and the remainder is suitable for normal waste treatment and discharge. Reuse of the dye pad drops and HF concentrates is possible; the eventual amount will depend on the type of dyes and run length. Plant operator experience and color matching problems will also determine practical limits of concentrate reuse. A means has been evaluated on a pilot scale whereby any residual dye liquor and concentrate can be reduced to an officially innocuous solid suitable for landfill application or perhaps agricultural use. By this means it is possible to approach zero discharge.

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REFERENCES