Closed-Cycle Textile Dyeing
Full-Scale Hyperfiltration Demonstration

La France Industries, SC

Prepared for

Industrial Environmental Research Lab.
Research Triangle Park, NC

Apr 83

U.S. DEPARTMENT OF COMMERCE
National Technical Information Service
NTIS®

PB83-193219

EPA-600/2-83-027
April 1983

CLOSED-CYCLE TEXTILE DYEING:
FULL-SCALE HYPERFILTRATION DEMONSTRATION

BY

Riegel Textiles
La France Division
La France, South Carolina 29656

CARRE, Inc.
Seneca, South Carolina 29678

EPA Cooperative Agreement No. S805182
EPA Program Element No. 1BB610

DOE Project Officer: William Sonnett
DOI Project Officer: Frank Coley
EPA/IERL-Cin Technical Advisor: Robert Mournighan

EPA/IERL-RTP Project Officer: Robert Hendriks

Industrial Environmental Research Laboratory
Office of Environmental Engineering and Technology
Research Triangle Park, North Carolina 27711

Prepared for:

U. S. Department of Energy
Industrial Programs
Washington, D.C. 20585

U. S. Department of the Interior
Office of Water Research & Technology
Washington, D.C. 20240

U. S. Enviromental Protection Agency
Office of Research and Development
Washington, D.C. 20460

TECHNICAL REPORT DATA

(Please read Instructions on the reverse before completing)

1. REPORT NO. EPA-600/2-83-027	2.	3. RECIPIENT'S ACCESSION NO. PB83 193219
4. TITLE AND SUBTITLE Closed-Cycle Textile Dyeing: Full-Scale Hyperfiltration Demonstration		5. REPORT DATE April 1983
		6. PERFORMING ORGANIZATION CODE
7. AUTHOR(S)		8. PERFORMING ORGANIZATION REPORT NO.
9. PERFORMING ORGANIZATION NAME AND ADDRESS Riegel Textiles, LaFrance Division LaFrance, South Carolina 29656 and CARRE, Inc., Seneca, South Carolina 29678		10. PROGRAM ELEMENT NO.
		11. CONTRACT/GRANT NO. CR805182
12. SPONSORING AGENCY NAME AND ADDRESS EPA, Office of Research and Development * Industrial Environmental Research Laboratory Research Triangle Park, NC 27711		13. TYPE OF REPORT AND PERIOD COVERED Final: 9/80-3/82
		14. SPONSORING AGENCY CODE EPA/600/13

15. SUPPLEMENTARY NOTES IERL-RTP project officer is Robert V. Hendriks, Mail Drop 63, 919/541-2547. (*) Also supported by U.S. DoE and DoI under EPA IAG-R701203.

16. ABSTRACT
The report gives results of a project of joining a full-scale dynamic-membrane hyperfiltration (HF) system with an operating dye range. (HF is a membrane separation technique that has been used successfully to desalinate natural water. The dye range is a multi-purpose unit with a variety of effluents from preparation and dyeing of textile fabrics.) The project follows a series of government-sponsored investigations of recycling of the large quantities of energy, process chemicals, and water discharged from industrial processes. On-site pilot-scale tests of three membrane types led to selection of the dynamic membranes on porous sintered stainless-steel tubular supports. The tests also led to conversion of the washing to counterflow, thereby reducing water use from 400 cu m/d (75 gpm) to 190 cu m/d (35 gpm) without any loss in effectiveness. Water recycle, up to 95 percent of waste water, is now used routinely. Over 1 million m of fabric has been produced with recycled water. Two 4000-m lots of fabric have been produced with recycled chemical concentrate.

17. KEY WORDS AND DOCUMENT ANALYSIS

a. DESCRIPTORS		b. IDENTIFIERS/OPEN ENDED TERMS	c. COSATI Field/Group	
Pollution	Waste Water	Pollution Control	13B	
Filtration	Water Treatment	Stationary Sources	07D	
Fabrics	Fluid Filters	Hyperfiltration	11E	13K
Dyeing	Membranes	Recycling	13H	
Circulation	Chemical Engineering	Industrial Chemicals	14G	07A
Industrial Processes		Reverse Osmosis		

18. DISTRIBUTION STATEMENT Release to Public	19. SECURITY CLASS *(This Report)* Unclassified	21. NO. OF PAGES 109
	20. SECURITY CLASS *(This page)* Unclassified	22. PRICE

EPA Form 2220-1 (9-73)

NOTICE

ABSTRACT

Hyperfiltration (HF) is a membrane separation technique that has been used successfully in desalination of natural water. Because energy, process chemicals and water are discharged from industrial processes in large quantities, recycle has been studied in a series of government sponsored research projects. The results of the research led to the current project of joining a full scale dynamic membrane HF system with an operating dye range into an integrated production unit. The dye range is a multi-purpose unit having a variety of effluents from preparation and dyeing of textile fabric.

On site pilot scale tests were conducted with three membrane types. High temperature membranes of hydrous zirconium oxide and polyacrylic acid dynamically formed on porous sintered stainless steel tubular supports were selected for the demonstration unit. Also the testing led to conversion of the washing to counterflow thereby reducing the water use from 400 m^3/d (75 gpm) to 190 m^3/d (35 gpm) without any loss in effectiveness. Water recycle, up to 95% of waste water, is now routinely used. Over a million meters of fabric have been produced with recycle water. Two 4000 meters lots of fabric have been produced with the recycled chemical concentrate.

The project developed capital and operating costs and savings for one year of operation. A payout time of 3.3 years will be realized when chemical recovery is fully implemented and membrane washing procedures are better developed. Both these areas will be further developed during an extension of this project to September, 1983.

The results of this demonstration have shown that an HF recovery system will yield a payout time in the range of one to five years in situations where there are simultaneous benefits for water, energy, and chemical recovery and/or where significant waste treatment costs can be abated.

This report describes the hyperfiltration equipment; presents and evaluates data from one year of operation; gives costs for equipment, installation and operation, and credits for savings due to recycle; and describes the objectives of an 18 month project continuation.

TABLE OF CONTENTS

FIGURES

TABLES

ENGLISH-METRIC CONVERSION TABLE*

To Convert From	To	Multiply by
Inch	Meter	2.54×10^{-2}
Feet	Meter	3.05×10^{-2}
Square inch	Square meter	6.45×10^{-4}
Square feet	Square meter	9.29×10^{-2}
Cubic feet	Cubic meter	2.83×10^{-2}
Gallon	Cubic meter	3.79×10^{-3}
Pound	Kilogram	4.54×10^{-3}
Pound per sq. inch (psi)	Atmosphere	6.80×10^{-2}
Horsepower (Hp)	Watt	7.46×10^{2}
Gallon per day	Cubic meter per day	3.79×10^{-3}
Gallon per minute (GPM)	Cubic meter per day	5.45
Gallon per sq. ft.-day (GFD)	Cubic meter per sq. meter-day	4.10×10^{-2}
Gallon per minute per sq. ft.	Cubic meter per sq. meter-day	5.87×10^{1}

*The units most familiar to the projected readership of this report have been maintained.

ACKNOWLEGEMENTS

This study was conducted by a team and major contributions were made by a number of people. The cooperation and assistance of the La France staff members is particularly acknowledged: Mike Drummond, Perry Lockridge, Charles Smith, Al Whitney and many machine operators and laboratory technicians. Jim Bostic, Jr., Ted Meyer and Ernie Freeman of Riegel Corporate staff provided valuable advice.

This demonstration is an interagency program and thus benefited from the guidance of Max Samfield and Robert Hendriks, U. S. Environmental Protection Agency, as Principal Project Officers; John Rossmeissl and William Sonnett, Department of Energy, and Frank Coley, Department of the Interior, as Project Officers; and Robert Mournighan, U. S. Environmental Protection Agency, as Technical Advisor. J. S. Johnson, Jr., of the Oak Ridge National Laboratory has served as membrane technology consultant on this and all the previous related research and development projects.

CARRE, Inc. provided overall program management and engineering design of the recovery system. The contributions of staff members at CARRE, Inc. are acknowledged. Staff members making major contributions are C. A. Brandon, J. L. Gaddis and H. G. Spencer; Donald K. Todd and Daniel A. Jernigan, engineers; and Roger Hunt, Don King and Cindy Cochran, technicians.

J. J. Porter and Grant Goodman of Texidyne, Inc. made significant contributions in providing chemical analyses and textile process consultation. E. Harrison served as a consultant on control and instrumentation.

The detailed design and bid specifications were provided by the J. E. Sirrine Company. The membrane equipment vendors made significant contributions in technical comments and advice.

GLOSSARY

CONCENTRATE- the fluid that does not permeate the membrane and thus contains the solute species that do not pass through the membrane.

CONTINUOUS DYE RANGE- the equipment components arranged sequentially such that fabric is dyed as it moves continuously through the range components. Other preparation and finishing operations can be performed on the dye range such as bleaching and scouring.

DRUG ROOM- the room where the dye formulations are mixed in 100 liter to 4,000 liter batches for transfer to the dye pad during the dyeing production.

DYE FORMULATION- a solution of dyes and chemicals applied to fabric as it moves through the continuous dye range. The chemicals include a thickener (guar gum), surfactants, and in some cases, dye solvents.

DYE PAD- the 50 liter tank in which the dye formulation is applied - "padded" to the fabric.

FLUX- the volume of permeate generated per unit of membrane area per unit of time.

HYPERFILTRATION - a membrane separation process operating on the principle of selective diffusion through a semi-permeable membrane achieved by pressure differential.

HYPERFILTRATION (HF) UNIT - a component of the recovery system consisting of ZOPA membranes arranged in a Single Pass configuration.

MEMBRANE FOULING- a decrease in flux that is not an instantaneously reversible effect of controlled operating variables. Fouling is usually categorized as (1) an alteration in the membrane due to a chemical reaction with the waste stream components or (2) an accumulation of a deposited material.

MODULE- a group of membrane segments contained in a housing suitable for collecting permeate. Membrane segment connections are external to the housing.

PERMEABILITY- the volume of permeate generated per unit of membrane area, time and pressure across the membrane. (Flux per unit of pressure across the membrane.)

PERMEATE- the fluid that passes through the membrane and thus contains the dissolved materials that pass through the membrane.

RECOVERY- the fraction of fluid exposed to the membranes that becomes permeate.

RECOVERY SYSTEM- the equipment that collects waste fluid from the dye range, processes this fluid with a membrane unit, and makes available hot clean permeate for use as washwater on the range and concentrate for use in dye formulations or for disposal. This system includes tanks for the wastewater, recycle water and concentrate; pumps; hyperfiltration unit and controls.

REGENERATIVE HEAT EXCHANGERS- a unit used to exchange thermal energy between incoming wastewater and outgoing permeate. Net effect is to operate the membrane unit at a higher temperature than the dye range washers when operated with a small steam heater applied to the wastewater after this heat exchanger.

REJECTION- the fraction of a particular material specie that does not pass through the membrane.

SEGMENT- the porous tube lengths connected serpentinely with small radius u-bends. The two ends of the segment protrude through one end of a module for connecting with other segments in the desired system configuration.

SINGLE PASS- a patented (U.S. Patent No. 4200533 of CARRE, Inc.) membrane arrangement such that entering fluid is separated into permeate and concentrate streams without the concentrated fluid ever being routed upstream of a segment of membrane area through which it has passed.

ZOPA MEMBRANE- a high temperature dynamic membrane formed of zirconium oxide/polyacrylic acid membrane (U.S. Patent Nos. 3,431,201; 3,449,245; and 3,503,789).

SECTION 1

INTRODUCTION

The technical feasibility of using hyperfiltration to renovate textile wastewater for direct recycle has been shown, at pilot scale, in a series of research projects conducted as part of a cooperative program between the textile industry and the U. S. Environmental Protection Agency which began in 1972.

The current project to demonstrate, at full scale, the use of hyperfiltration with a production dye range establishes the practicality of hyperfiltration. This project is funded by a cooperative agreement between the Department of Energy, the Department of the Interior, the Environmental Protection Agency and La France Industries, a Division of Riegel Textile Corporation. This report summarizes the results of the demonstration program.

The wide scale application of hot process effluent recycle/reuse has a large potential impact on pollution abatement. The cost of achieving this pollution abatement with hyperfiltration will be offset by a combination of savings from the simultaneous recovery of energy, water, and chemicals. If subsequent waste treatment is required of all or a portion of the chemicals, the cost of this treatment will probably be less because of the volume reduction achieved by hyperfiltration.

HYPERFILTRATION

Hyperfiltration is a membrane separation process operating on the principle of selective diffusion through a semi-permeable membrane, achieved by pressure differential. Since the separation is achieved without a change of phase, membranes are inherently energy efficient. The optimized Single Pass arrangement (U. S. Patent No. 4200533 of CARRE, Inc.) which requires no recirculation of any concentrated material, utilizes approximately four BTU's per pound of water passing through the membrane. The energy used is generally electrical energy to operate the pumping system. Converted to the equivalent thermal basis of 10,500 BTU/kilowatt hour, this would be approximately 12 BTU's per pound of permeate produced. Change of phase technologies, such as freezing and evaporation, require four to forty times as much energy per pound of water separated.

Initial interest in membrane separation was largely directed to reverse osmosis of sea and brackish water. Attempts to utilize the technology in industrial situations encountered limitations dictated by temperature and composition of the typical individual waste streams. The innovation of zirconium oxide/polyacrylic acid (ZOPA) membranes (U. S. Department of Energy Patent No: 3,431,201, 3,449,245, and 3,503,789) dynamically formed on sintered stainless steel tubes, relaxed many of the limitations. The ZOPA membrane can operate under a wide range of corrosive conditions at high pressures and temperatures, are able to withstand high suspended and dissolved solids and are not subject to bacteriological attack. These high temperature membranes are utilized in the hyperfiltration system being demonstrated.

PREVIOUS STUDIES

Three previous studies led to this demonstration. The first study (1) began in 1972 involved the pilot-scale separation of composite wastewater from a beck dyeing process and a full-scale reuse of hyperfiltration permeate and concentrate at this site. Polyamide (hollow-fine fibers), cellulose acetate (spiral and tubular), and hydrous Zr(IV) oxide-polyacrylate membranes were used. Eighteen production dyeings involving a total of 1348 m of cloth were carried out in a production dye beck.

The purified permeate water was a satisfactory substitute for normal process water in all production dyeings for water recoveries ranging from 75 to 90%. Membranes used in the renovation of the wastewater had conductivity rejections of 65 to 95% and color rejections from 86 to > 99%.

It is also technically feasible to reuse all the concentrate. In 11 production dyeings over a 700 m of cotton velour fabric was produced, graded as first quality, and sold commercially. In 10 tests standard shades were produced with an average dyestuff savings of 16%.

The second study (2) involved composite wastewaters obtained from the several processes occurring in a dyeing and finishing plants were separated by hyperfiltration, and the cumulative permeate and concentrate of a 90% recovery run was tested for reuse as process water in laboratory dyeing. Precast and dynamically formed membranes were used at eight dyeing and finishing plants. The processes encountered were: dyeing of nylon using premetallized dyes, dyeing of acrylic fabric using basic dyes, and the scouring, desizing, and dyeing of cotton and polyester. In all cases the product water was acceptable for replacement of process water as determined in laboratory dyeings using standard production evaluations. Analyses indicated higher COD and dissolved solids and lower concentrations of metals in the permeate water than in the fresh plant-process water. The concentrate from the premetallized dye process was suitable for dyeing very deep shades upon addition of appropriate dyes. Laboratory dyeing tests using concentrates from the other processes were unsuccessful. Perhaps this result is not surprising because the concentrates were obtained from a feed containing a composite of effluents from the plant processes and not from a single process.

The first two studies dealt with renovation of the composite wastewater from the dyeing and finishing plants. Because of the obvious advantages for reclamation and energy conservation, a third study (3) evaluated hyperfiltration for direct recycle of unit process effluents. Five major water and energy consuming preparation and dyeing processes were studied with high temperature hyperfiltration membranes. The permeate produced by the membranes was again found to be universally usable as process water. In some cases the reuse of the concentrate from the individual process effluent streams was estimated to be practical.

ENERGY RELATED PROBLEMS

Two trillion gallons of hot water are discharged by industry each year. Literally about 6% of all the energy consumed by industry goes down the drain. Much of this hot water is "contaminated" with chemicals and other dissolved or suspended material which not only constitute a hazard to the environment, but represent an additional "waste" of materials which requires substantial energy to produce or replace. Additionally, much energy is expended by industry to remove water from the industrial waste stream to achieve desired levels of chemical concentration to permit reuse, or to reduce the volume of materials to be stored, processed, or transported.

Many, if not most, industrial situations in which the manufacturing process involves industrial waste water have similar requirements:

1. A contaminate level that requires reduction to permit hot water recycle; and/or

2. A high content of the dissolved chemicals or suspended solids in the water that requires reduction to permit the simultaneous recycle or recovery of both the water and the solids.

Often, the simple separation between the water and solids in the typical industrial waste stream will permit the simultaneous recycle or recovery of both the water and the solids.

Despite this similarity, each industrial waste stream contains unique features which must be considered not only as to the type of separation but the economic soundness of a program of recycle and recovery. The uniqueness may be in the type of corrosiveness (i.e., acid, base, oxidant, etc.); the specific separation required; the type of foulants which may lead to reduction of the performance of the separation equipment; and other characteristics of the particular industrial waste stream. The ultimate impact of using HF on industry could approach 5% savings of present energy consumption just in the single area of hot water recycling. Additional energy savings could probably be effected by reducing the required amount of evaporation, as well as by recovering of the chemicals presently being literally thrown away. Additional energy savings will result from reduced pollution and the reduced energy consumed in pollution treatment resulting from the greatly reduced volume. The estimated potential in energy and material resource conservation by recycle in the U. S. textile industry in 1978 is shown in Table 1 (4). The energy conservation for the textile industry is equivalent to approximately fifteen million barrels of oil per year. The total value of energy and material conservation is estimated to be nearly one billion dollars per year.

PURPOSE AND SCOPE

The purpose of this demonstration project was to design, install, and operate a full scale commercially available hyperfiltration system to show that dye range waste water can be recycled and materials reused.

The scope of the project included the following activities divided among four phases (the phases are identified in parentheses):

1. Engineering design and quotations for a recovery system including installation (Phase I).
2. Installation and start-up of a full scale hyperfiltration unit (Phase II).
3. Evaluation of effects, if any, on the manufacture, quality control, and productivity of the dye range (Phase III).
4. Determination of operating costs (Phase IV). Evaluation of the economics and technical feasibility of hyperfiltration as a means to approach zero discharge.

METHOD OF STUDY

This demonstration project was conducted with the assistance of five major subcontractors. The Oak Ridge National Laboratory provided a hyperfiltration mobile laboratory and consultation on membrane technology. Clemson University conducted some fundamental experiments on membrane behavior with key wastewater components. CARRE, Inc. provided the overall program management, the process design of the recovery system, the evaluation of the hyperfiltration equipment and the effects of recycle/reuse on production and product quality. Texidyne, Inc. performed chemical analyses and provided consultation in the areas of process modification and recycle. J. E. Sirrine Company detailed the recovery system, including preparation of the equipment and installation specifications in requests for bids for the equipment and installation. They also received and reviewed the quotations. The method of study is illustrated in the List of Milestones, Table 2. Initially the period of the project was September 23, 1977 to April 22, 1982. (Currently the project is being continued through September, 1983). There was no project activity from September, 1978 through March, 1979 while the decision was being made about continuation following Phase I, the design phase.

TABLE 1. ENERGY AND MATERIAL RECOVERY FOR THE U. S. TEXTILE INDUSTRY (4)

	Water Discharge (10^3 m^3/d)	Dyes (10^3 kg/d)	Auxiliary Chemicals (10^3 kg/d)	Salt (10^3 kg/d)	Process Thermal Energy (10^9 BTU/d)
1978 Study Total[a]	120	27	13[b]	12	38
Industry Total	2700[c]	1000	270	264	784
Recycle Potential	2400	60	222	220	352
Estimated Annual Savings[d] ($ x 10^6/yr)	120	158	36	2.6	616

[a] EPA Report No. EPA-600/2-78-047

[b] Exclusive of 80,000 kg. of NaOH used daily at these plants.

[c] 1972 census of manufacturers, assuming 250 days/year.

[d] Unit costs: water @ $0.2 m^3; dye @ $2.3/kg; auxiliary chemicals @ $0.66/kg; salt @ $50/1,000 kg; process steam @ $7/$10^6$ BTU.

TABLE 2. PROJECT MILESTONES

1.	Detailed Work Plan Completed	11 November 1977
2.	Process Selected	31 May 1978
3.	Recovery System Design Completed	31 July 1978
4.	Equipment and Installation Bids Received	31 August 1978
5.	Project Continuation Authorized	6 April 1979
6.	Auxiliary System Installed	30 November 1980
7.	Hyperfiltration Unit Installed	1 May 1981
8.	Permeate Recycle on Production Basis	12 May 1981
9.	Interim Final Report	15 September 1981

OVERALL RESULTS

A Phase I design report was published (5). The two major results of the study and design activities of Phase I were:

1. The selection of the continuous dye range instead of the dye becks for the full scale demonstration.
2. The determination that hyperfiltration, based on the quotations for installed costs, had the potential to achieve a practical approach to zero discharge with a positive rate of return on the investment when energy and chemical conservation were fully realized.

The continuous dye range was selected because it is the more modern dyeing equipment technology and is representative of the trend in the industry due to the lower production costs associated with this method. At the demonstration site, the dye range has largely replaced the becks as the standard production equipment.

During Phase II and Phase III, the Single Pass hyperfiltration unit was installed beginning in January, 1981, and is producing water for recycle. Over a million meters of standard width (1.4 meters) velour fabrics have been washed with recycle water during routine production. The hyperfiltration unit is being operated by production personnel. The procedures for reuse of the dyes and auxiliary chemicals have been developed on a laboratory basis. Initial tests with production lots of 4,000 yards of velour have been achieved in the first quarter of 1982. This project, initially scheduled to be concluded in April, 1982, is being extended through September, 1983 to better establish the reuse of chemicals in production. During this extended period the performance of the membrane system will continue to be monitored to further establish membrane life and operating cost. The initial membranes installed in January, 1981 are still in operation as of March 1, 1982. The study of membrane fouling will be a major effort during the extended period with the goal of increasing average unit capacity.

The capital costs, including installation, were $484,000. The operating costs including membrane maintenance are $58,800/year. The savings have not been fully realized because procedures for chemical reuse in production are still being developed and the capacity of the HF unit is limited by fouling to a time average of about 60% of design capacity. When the potential savings are realized, the payout time will be 3.3 years. At current levels of reuse and HF performance there is a net savings of approximately $16,000 per year.

SECTION 2

CONCLUSIONS

For twelve months a production size HF unit has been integrated with a manufacturing dye range resulting in the full scale recycle of hot wash water from a dynamic ZOPA membrane hyperfiltration system. The results have demonstrated satisfactory use of permeate recovered from all types of effluents from this multi-purpose range. More than one million meters of fabric have been produced with recycled wash water with no effect on fabric quality.

Full scale use of the HF concentrate to formulate solutions for dyeing has been demonstrated in selected cases. Two lots of 4,000 meters each have been dyed with HF concentrate plus dye additives. The eventual extent of such reuse of HF concentrate will depend on the ability of the plant to work out the problems of production scheduling by dye class and shade.

Throughout the twelve months of demonstration, the membranes have remained stable with respect to rejection. It has been demonstrated that the stainless tubing can be cleaned and that new membranes can be formed even after several months of operation with waste water. The availability of permeate has been limited to about 60% of the production during this demonstration because of membrane fouling. Membrane cleaning and foulant removal procedures will be developed during the extended evaluation period.

No build up of solute components was observed in the permeate during a continuous recycle run of four hours, thus the expected normal continuous recycle period of eight to twenty-four hours should not be limited by component build up.

The capital and operating costs of HF were found to be about as projected in 1977. The payout time for the capital cost of this demonstration will be 3.3 years (after taxes) when the full potential for reuse is achieved.

The most favorable situation for the application of HF is the one where there are simultaneous and significant benefits for water, energy, and chemical recovery and where significant waste treatment costs can be abated by reuse or volume reduction. For example a new plant in a city in a water-short region dyeing nylon velour might find HF economically attractive. The value of water and the charges for sewage could be 2 to 5 times that at La France. The value of pre-metalized dyes could be 5 to 10 times those normally in the wash water during this demonstration.

A new plant considering installing a pre-treatment or waste treatment plant could build a smaller facility if recycle and reuse were being considered. At La France the capital cost amortization schedule on the existing treatment facility could not be reduced by recycle and chemical reuse. In specific situations of chemical recycle, e.g. polyvinyl alcohol (PVA) recovery and caustic recycle payout times as short as 12-15 months (after taxes) are realizeable.

Disposal of HF concentrate was studied. The technical feasibility of incineration after further concentration and drying was shown. Thus a method of complete on-site disposal was studied. Further study is required to determine the practicality of incineration because of the cost of commercial units which are not designed for the small capacities required. At La France, the HF concentrate is treated in a biological treatment system with no apparent problems.

SECTION 3

RECOMMENDATIONS

The results of the demonstration project can be enhanced by further study to increase chemical reuse and to improve performance of the hyperfiltration unit.

It is recommended that this project be extended so that this full-scale installation can be used to:

(1) continue full scale reuse testing of HF concentrates;
(2) continue documentation of the operation, including savings, for an additional 12 months;
(3) study membrane fouling and to develop better cleaning procedures.

The goal of the further study of reuse should be demonstration through full scale implementation with selected production lots. The study of reuse of HF concentrate should include all these aspects:

(1) production scheduling,
(2) color matching techniques, and
(3) reuse of HF concentrate consisting of mixtures from several production dye lots.

It is improbable that reuse of 100% the HF concentrate will ever be practical. Thus it is recommended that this full-scale production unit be used to further study HF concentrate disposal. HF concentrate constitutes a new form of industrial effluent. The disposal of HF concentrate because of the small volume and high concentration may be amenable to disposal by chemical processing not normally considered for waste treatment.

The applicability of the results of this demonstration of high temperature dynamically formed HF membranes on reuseable porous stainless steel tubes should be extended to many industrial situations. Sintered metal tubing can be widely applied to hot, corrosive, and dirt-laden industrial effluents. The dynamic technique of membrane formation is inherently versatile permitting in-situ membrane replacement and the use of a wide variety of membrane materials selected for specific separation requirements. It is recommended that research in membrane tailoring for selected important industrial categories be undertaken.

SECTION 4

TEXTILE PROCESS DESCRIPTION

The textile processes involved in this project are conducted on a continuous dye range. The range and its operation are described in this section. The production history beginning in January, 1980, forms the baseline for evaluating this demonstration project. The chemical characteristics of the range effluent, i.e., the supply to the hyperfiltration unit, are also presented.

DYE RANGE

Most of the dyeing production is done on the continuous range. The range is also used for bleaching and scouring and consists of a dye applicator, a spiral atmospheric steamer, and a washing section shown in Figure 1. Most of the production is velour upholstery fabric with stringent light fastness and crocking (color wipe off) specifications. The types of fibers processed include cotton, acrylic, nylon, rayon, and polyester as well as their blends. The range is fully automated to control cloth speed, process temperature and water flow rate. The range processes fabric from 9 to 36 meters per minute depending on the fabric type and the process details. The range is operated three shifts per day, five to seven days per week.

Fabric moves sequentially through the range components beginning with the dye pad. Dye formulations are mixed in the drug room and pumped to the pad where they are applied to the fabric. The applicator is a 50 liter tank in which the fabric is saturated. Excess dye and auxiliary chemicals are removed from the fabric by squeeze rollers as it leaves the pad and before entering the steamer. The temperature is maintained at 100°C in the steamer. The steamer holds approximately 150 meters of fabric so range speed is a function of residence time required for the particular fabric and dye combinations.

Fabric moves from the steamer to a series of washers where excess dyes and auxiliary chemicals are removed. The washing train consists of jet washer, dip box, and two rotojet washers. Squeeze rollers follow the dip box and each rotojet washer. The jet washers and the two rotojet washers incorporate large recirculation flow rates which pass through 100 mesh lint filters. The majority of range wash water enters the second rotojet which overflows to the first rotojet which in turn overflows into the jet washer. A smaller amount

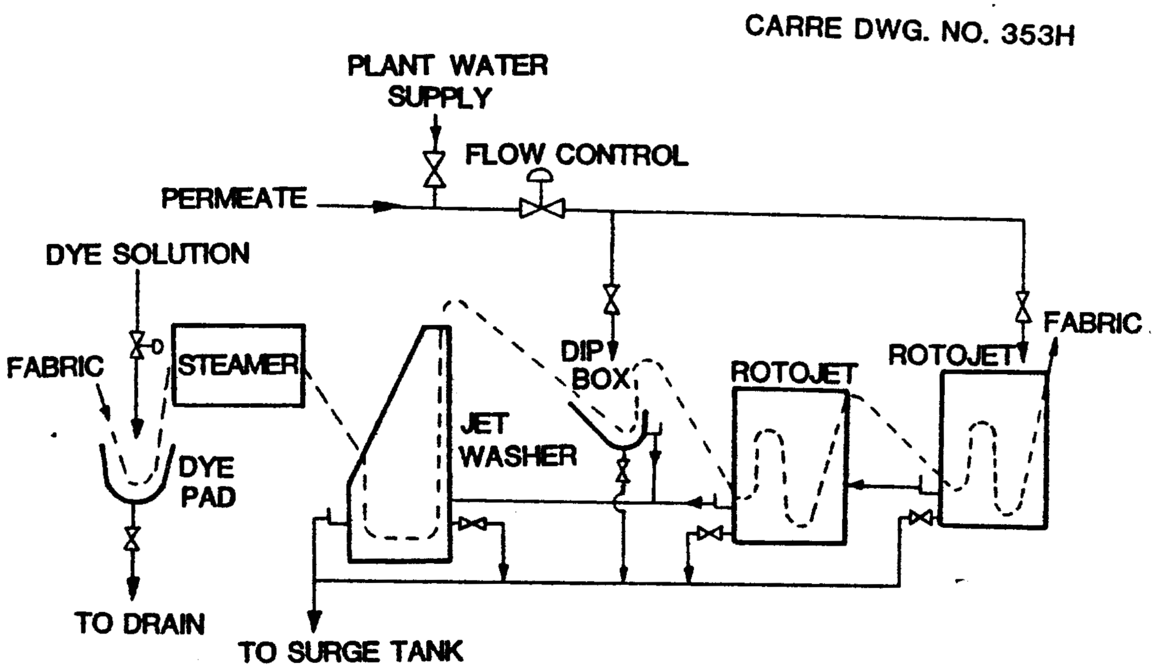


Figure 1. Continuous Dye Range Schematic

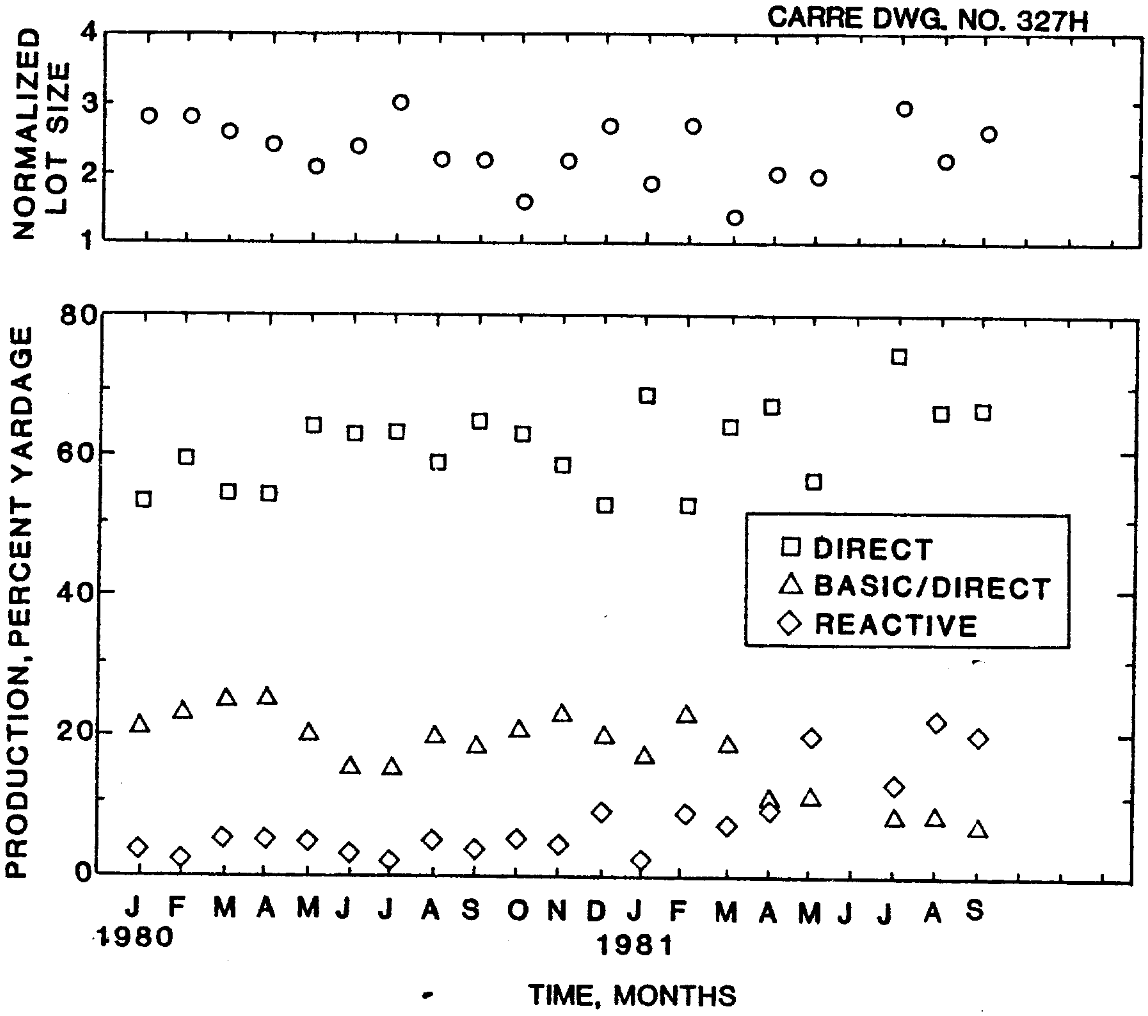


Figure 3. Averaged Weekly Production Characteristics for Lot Size and Dye Class. (Lot Size is Normalized by a Proprietary Constant. Percent of Total Production for the Predominant Dye Classes at La France are Shown on a Weekly Basis.)

EFFLUENT CHARACTERISTICS

Several classes of dyes are used at different times: direct, basic, disperse, acid and reactive. The wash water effluent components vary in dye class and concentration but the types of components are common to all the wash water from the dyeing operations. The dye formulations contain dyes, a thickener (guar gum), surfactants, and in some cases, dye solvents. While about 85% of the dyes are exhausted on the fabric, the remaining dyes and most of the auxiliary components are removed from the fabric by the washing process. Analysis of composite effluents from the dye pad applicator, and the plant tap water, are presented in Table 3. Table 3 also shows representative concentrations and flow from the washing section. Water flow to the range has been reduced to as low as 113 liters per minute (30 gpm) for long periods of time without any observed affect on quality of produced goods. However, the standard operation currently uses 138 liters per minute (35 gpm) and ranges up to 280 liters/min. as additional evaluations are being concluded.

The wash water is characterized by being highly colored and quite "hard." The hardness is the result of chemicals added in the dye formulations because both the plant water and HF permeate are relatively soft. The wide pH range is the result of including the scouring and bleaching effluents along with the dye wash water. The organic content of the wash water is not large, as indicated by the TOC and COD parameters. Most the dyes are exhausted onto the fabric and do not appear in the wash water. The fraction of the dye exhaustion onto the fabric is indicated, qualitatively, by a comparison of the color measured in the dye pad and in the wash water taking into account a dilution factor of about 8 for the wash water. The exhaustion implied by the data in Table 3 is about 85 percent.

of water enters the dip box and overflows to the jet washer. The jet washer overflow becomes the supply to the recovery system. Water flow to the range is automatically controlled at the operator control panel. Steam is injected into the dip box and rotojet washers to maintain the controlled process temperature.

When the range is used for bleaching, the operation is essentially the same as with dyeing. The bleach formulation is mixed in the drug room and pumped to the pad where it is applied to the fabric. The active bleaching agent is reacted in the steamer and essentially only auxiliary chemicals are removed in the washing. The scouring operation does not use the pad. The fabric is simply treated in the steamer and washed.

PRODUCTION HISTORY BASELINE

Records of range production were kept beginning in 1980 before process modifications and the installation of the recovery system. These records comprise a baseline of data prior to the changes in range operation, i.e., counterflow and reduced wash water flow and recycle.

Figure 2 shows specific consumption of water, steam, and dyes and chemicals beginning in January, 1980. Normalized production numbers are included to show relative rates of production during each week. The baseline portion of the data extends from January 1, 1980, to April 1, 1981 (63 weeks) after which the rate of water flow delivered to the range washers was reduced from about 400 m^3/d (75 gpm) to about 200 m^3/d (35 gpm). (The reduced water flow rate was shown to be acceptable by experiments during Phase 1 (5) of this project.) Energy use dropped with the reduction of water use as expected.

Phase 1 (5) experiments also indicated that improving washing could be obtained at elevated temperatures. The wash water temperature was set to be 85°C. However changes in production has resulted in the current practice of setting a specific wash water temperature for each fabric, dye class, and process (dyeing, bleaching and scouring). The washing temperature for dyeing is less than 60°C. For bleaching and scouring the standard operating temperatures are 70°C and 60°C respectively.

The production characteristics which could influence recycle of concentrate and permeate is included on a monthly basis in Figure 3. The figure includes the normalized average dye lot size and a breakdown of production type as a fraction of total production. Larger production lots result in larger volumes of a concentrate facilitating chemical reuse. The membrane color rejection is different for the various production types so a significant shift in production trends could influence permeate recycle. However, no significant variations in production have occurred.

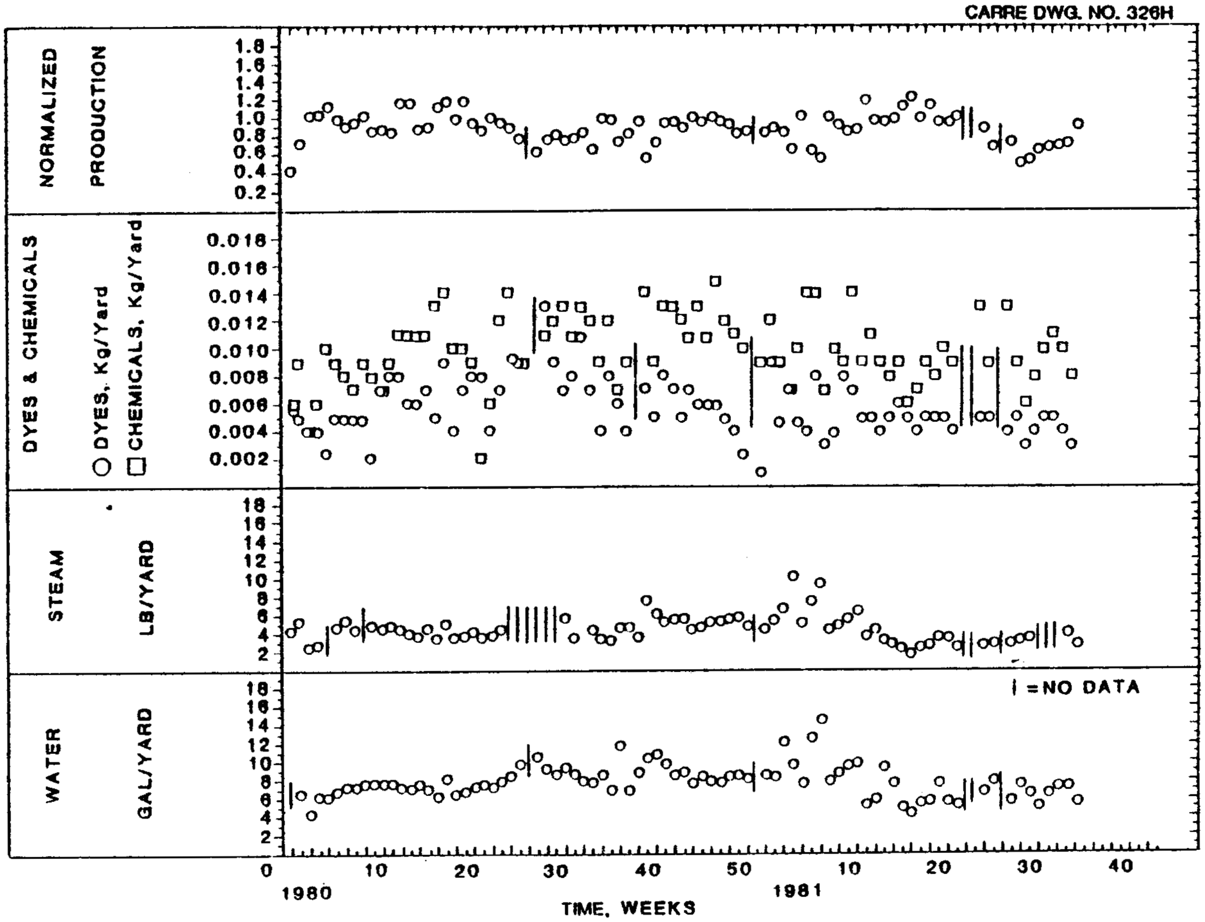


Figure 2. Weekly Normalized Dye Range Production and Consumption Characteristics (Production Yardage is Normalized by a Proprietary Constant. Water, Steam, Dyes and Chemicals are Normalized by the Total Production Yardage for Each Week.)

TABLE 3. CHEMICAL CHARACTERISTICS OF THE DYE RANGE EFFLUENT

ASSAY	Average concentration or flow Dye Pad	 Composite[a] Effluent	 Tap Water
Flow, ℓ/min.	12-35[b]	138	-
BOD, mg/ℓ	5,400	200	-
COD, mg/ℓ	23,900	1,200	9
Conductivity, µS/cm	1580-28,000[c]	200-2,000	90
Alkalinity, mg/ℓ	4,150	180	-
Color, ADMI	98,800	1,750	-
Hardness	-[c]	30	9
pH	3.6-10.9[c]	5.0-10.5	7.05
Phenols, mg/ℓ	0.84	-[d]	-
TOC, mg/ℓ	6,250	325	-
Total Solids, mg/ℓ	20,900	1,140	60
Suspended Solids, mg/ℓ	1,730	45	3
Dissolved Solids, mg/ℓ	19,200	1,100	57
Chromium, mg/ℓ	5.3	0.2	0.002
Copper, mg/ℓ	19.2	0.2	-
Iron, mg/ℓ	2.8	0.63	0.022
Manganese, mg/ℓ	0.2	0.1	-
Nickel, mg/ℓ	0.1	0.007	-
Zinc, mg/ℓ	2.7	0.25	-
Magnesium, mg/ℓ	10.4	8.5	1.00
Calcium, mg/ℓ	7.4	3.5	2.36

---

[a] These are representative values based on measurements at various flow rates of wash water.

[b] Dye pad flow depends on cloth pickup. Pad drops are added directly to the HF concentrate without going through the HF unit.

[c] These values were estimated without averaging.

[d] Sample color interferes with analytical procedure.

SECTION 5

RECOVERY SYSTEM DESIGN AND INSTALLATION

The recovery system is designed to collect all water from the dye range and supplies the fluid to the hyperfiltration (HF) unit. The permeate from the HF unit is returned to the range as hot wash water and the concentrate is collected for reuse or disposal. Operation of the range is independent of the operation of the recovery system. Automatic controls allow for continuous operation. Additional instrumentation is installed to record operating parameters of economic importance.

A design report has been issued separately (5). In the discussion which follows, only a portion of the detailed design specifications are mentioned. (Detailed specifications used as a basis for the quotation are available from the EPA Project Officer.) Emphasis is given to the aspects of design which affect the system operation.

DESIGN

Wash Water Balance

The design wash water flow rate, as shown in Figure 4, was 174 liters per minute (46 gpm)(5). The assumed fabric speed was 18 meters per minute. At a fabric speed of 18 meters per minute, 159 liters per minute of water was designed to be supplied to the washers and 18 liters per minute of dye formulation is applied to the fabric. The moisture added in the steamer less the amount of drag out from the last washer and the vapor loss from the hot fabric comprise the total mass balance of water.

The average lot size was taken as 10 pieces of fabric (one piece = 50 meters of 1.4 m wide fabric). Each lot is separated by approximately 20 minutes from the next lot. During this 20 minutes time the wash water flow is continued. (In the original design it was assumed that the washers would be drained.) The total volume drained during this average cycle resulted in an average flow rate of 174 liters per minute.

The waste water and the recycle water tanks were sized to accommodate surges representing maximum expected deviations in water use and also provide for about two hours of HF unit down time. The maximum deviation envisioned was two consecutive shifts with consecutive 80 piece lots. The two large tanks were sized at 23 m^3.

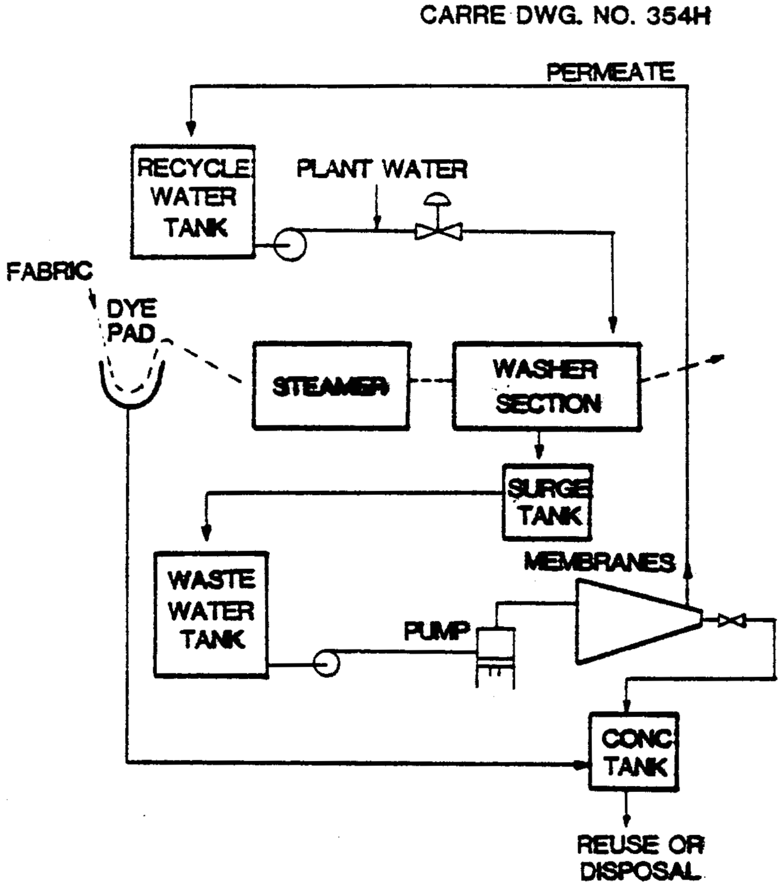


Figure 4. Recovery System Interaction with the Continuous Dye Range

The concentrate tank was sized to accept the concentrate from one 80 piece dye run, 3000 liters (800 gallons). The HF unit was designed to concentrate to 75% of the dye liquor volume to allow for the addition of make-up chemicals for reuse.

The range was modified for counter-flow washing with the overflow to drain from the jet washer. The flow scheme is also shown in Figure 4. The drains and overflows are collected in a sump and pumped to the wastewater tank. The drain piping and sump pump are designed to accept short term surges of up to 1,514 liters per minute (400 gpm) during system draining. Of the total flow supplied to the range, a substantial fraction was used to remove lint by reverse spray of three rotating screen filters. Approximately 23 liters per minute (6 gpm) is used at each filter with about one third escaping from the system with the lint cake. Rather than use permeate for this backwash function, water is now diverted from the washer circulation within each washer. Lint baskets of screen wire are provided to catch the lint cake. The baskets are fitted with a fluid collection pan so that the fluid draining from the lint is routed to the sump. This detail of the drain system is omitted from Figure 4 for clarity.

The dye formulation remaining in the applicator pad and in the 100 meter long pipe (approximately 100 liters) supplying the pad from the mixing room is transferred directly to the concentrate tank.

The equipment provides for nearly complete capture of all fluids emanating from the range. Spillage still occurs primarily with foam overflow from the washers. Adjustments to the internal circulating flows of the washers have resulted in a reduction in the amount of foam spillage. (Study of the proper internal flow and use of antifoam chemicals is continuing during the extension of this project.)

The recovery system is designed to provide a balance of the wash water. The flow rates outlined above are based on assumed demands. The variations are accommodated by control of the flow to range, the HF concentrate flow control and automatic provisions for use of plant water as required. For continuous HF system operation the design concentrate flow was 11.4 liters per minute (3 gpm). The flow rate to the dye applicator from the drug room depends on the range speed. The design assumed 1 liter of dye solution is added for each meter of fabric. The average flow rate of dye solution, however, depends on lot size because the 140 meters of leader on each end of each lot does not require dye solution. Also there is no dye solution flow for 20 minutes between each lot. The average drug room flow is thus quite small for small lot sizes and asymptotically approaches 1 liter per meter of fabric. For an average lot size of 20 pieces, about 4 liter/minute of water is required in addition to concentrate recycle. This design provision allows water to be added with the chemicals necessary for dye reuse.

Operational Mode

Conceptually, the cycle of operation begins with washers empty and no flow. The operator commands "high flow" and the flow valve opens completely. The rinse system pump is energized to provide permeate to fill the washers. If the permeate tank is empty, plant water automatically enters the system. When the washers are full, the range operator stops the flow by the command "interrupt." The operator signals for dye solution, sets the temperature controls (independent system) and fabric speed (also an independent system). The fabric leading edge exits the steamer several minutes after operation starts. The operator commands "controlled flow" which admits flow from the recycle water tank or the plant water supply. The counterflow overflow enters the drain system and is pumped to the wastewater tank (unless a decision has been made not to process the particular wash water in which case the water overflows the sump and enters the plant waste system.)

The HF unit starts automatically when the fluid level in the wastewater tank reaches a set point. The wastewater is pumped through a regenerative heat exchanger and a steam heater where it is heated to the design temperature for the membranes, 85°C, before it enters the positive displacement pump. The permeate passes through the regenerative heat exchanger to the recycle water tank. When the liquid level set point is satisfied, permeate is used as the wash water on the range. The hot concentrate (85°C) is delivered to the concentrate tank if reuse is scheduled, if not the concentrate enters the plant waste system directly.

When the fabric trailing edge passes the washers, the operator may elect to drain the wash boxes or allow wash water to continue to flow, or stop the wash water depending on the next color to be dyed.

Instrumentation and Controls

Automatic control is provided for the operating temperature of the HF unit and the flow of wash water to the range. The system automatically switches to plant water when the level in the recycle water drops to a set point. An array of flow meters, liquid level indicators, temperature elements, steam meters, electric power meters and on time indicators were selected to allow evaluation and documentation of the recovery system performance. A more complete description and a list of controls and instruments may be found in Appendix A.

HF Unit Configuration

The HF unit is a Single Pass system consisting of zirconium oxide-poly(acrylic acid)(ZOPA) membranes dynamically formed on the interior of 70 segments of sintered stainless steel support tubes. The 70 segments are contained in ten modules. The total membrane area is 139 m^2 (1,500 ft^2).

The high pressure flow from the positive displacement pump enters a manifold which distributes the flow into an initial section comprised of seven parallel flow paths of 1.6 cm diameter tubing. The seven streams from this section are collected in a manifold and divided into four parallel paths in the second section of 1.6 cm tubes. Similarly the third section has three parallel paths; the fourth section, two; and the last section has only one. The last two sections contain 1.3 cm diameter tubes. Each path has several segments of tubing connected in series. The flow path lengths are indicated in Figure 5(a) where the tapered arrangement of the segments is depicted.

The flow arrangement was designed to yield a low velocity, low pressure drop system through the 1.6 cm diameter tubing. At the point where concentration becomes high enough to produce a significant velocity effect, 1.3 cm diameter tubing has been used to increase the fluid velocity and minimize the concentration effect. The velocity range in the 1.6 cm diameter tubing was designed to be 1.5 - 2 m/sec and in the 1.3 cm tubing was 2 - 3 m/sec. A photograph of the HF unit is shown in Figure 5(b).

The key components of the recovery system are shown in Figure 6. The piping is 304 stainless steel. All tanks are fiberglass except for wash tank for the HF unit. These materials are corrosion resistant over a wide range of pH and temperature consistent with the dye range equipment. The transfer pumps are stainless steel single-stage centrifugal pumps. The line and pumps sizes are chosen to permit transfer not more restrictive of process time than were already were the production standard. For example, the transfer pump and line for recycle of permeate provides the same maximum flow rate as available from the plant water supply.

INSTALLATION

The recovery system installation began during the July, 1979 plant shutdown concurrent with the replacement of three existing washers with two rotojet washers (which utilize internal circulating flows and lint filters). Recovery system installation included piping to incorporate counterflow washing on the range and piping to connect washer and lint filter drains to the recovery system. A design modification was made in the recovery system at this time to incorporate a surge tank and pump to collect the wash water from the range and to pump it to the recovery system. This surge tank replaced a diverter valve on the jet washer circulating stream. A mechanical totalizing water meter was installed at this time to measure total water used on the range.

Work continued during the December, 1979 plant shutdown on the piping to deliver recycle water to the range and on the foundation work for the recovery system building. Foundation problems were discovered resulting in a project delay while civil engineering studies were made that resulted in an alternate location being proposed. It was decided to locate the recovery system inside the plant near the range. Preparation of the new site included cutting floor drains, installing guard rails, and fabricating pump and tank concrete pads. The 23 m^3 (6,000 gallon) wastewater and recycle water tanks had to be cut and field fabricated because the new location had limited access doors. A 0.75 m^3 (200 gallon) stainless steel wash tank was added to facilitate membrane cleaning. Equipment installation and associated piping

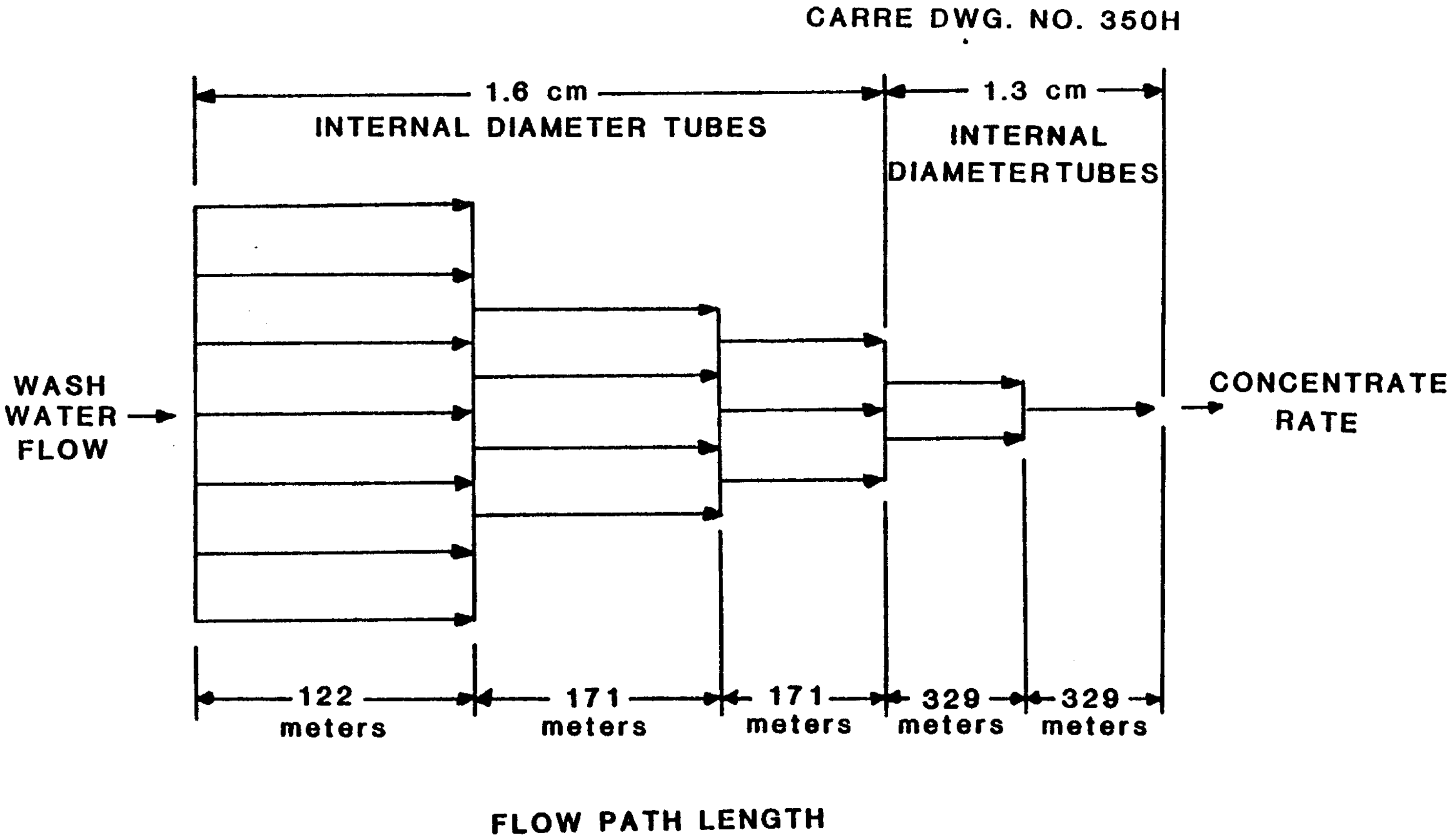


Figure 5(a). Single Pass Membrane Unit Flow Configuration. (Each arrow represents a flow channel. The tapered configuration is 7 to 4 to 3 to 2 to 1 parallel flow channels.)

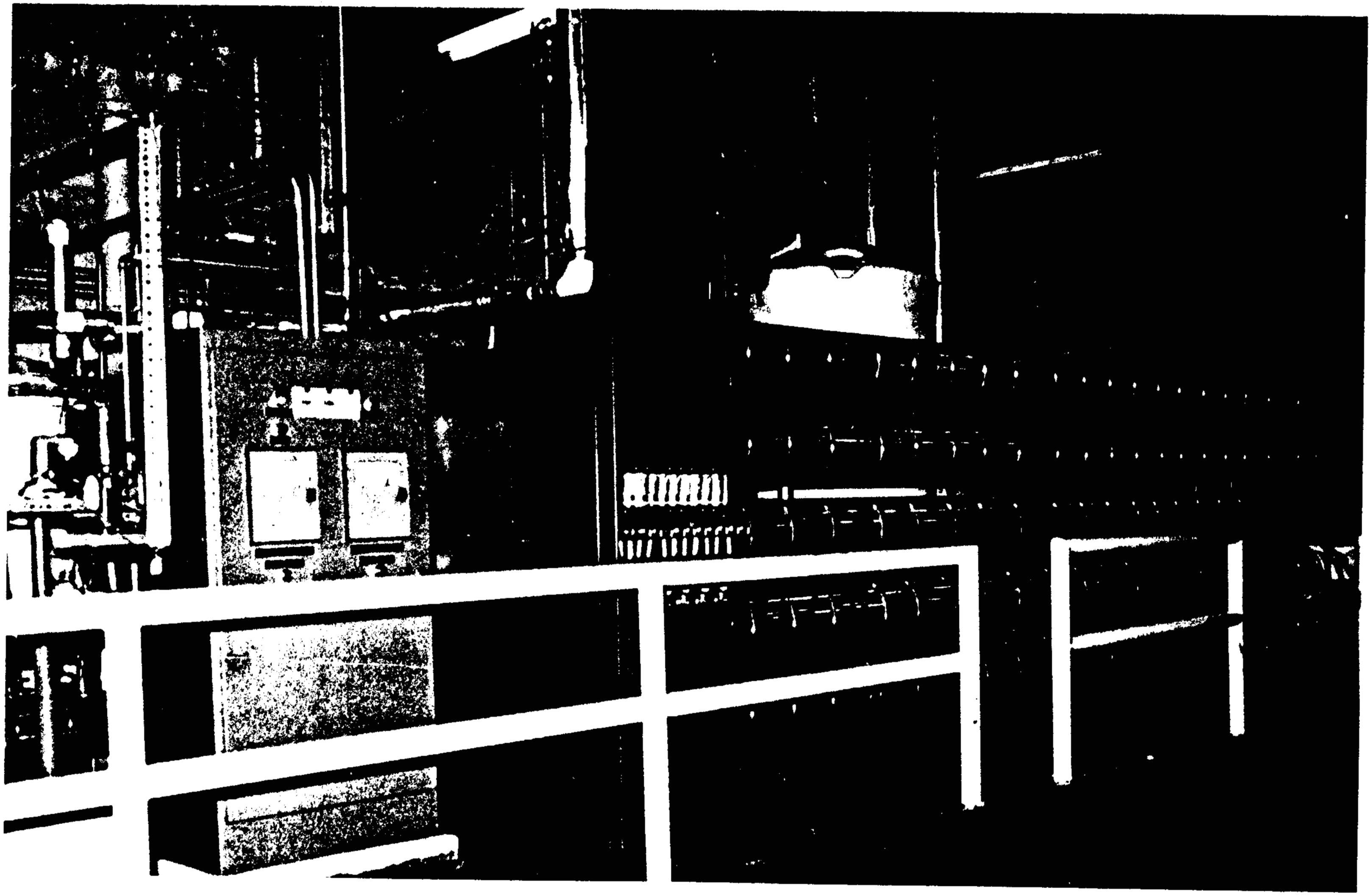


Figure 5 (b). Photograph of Hyperfiltration Unit at La France.

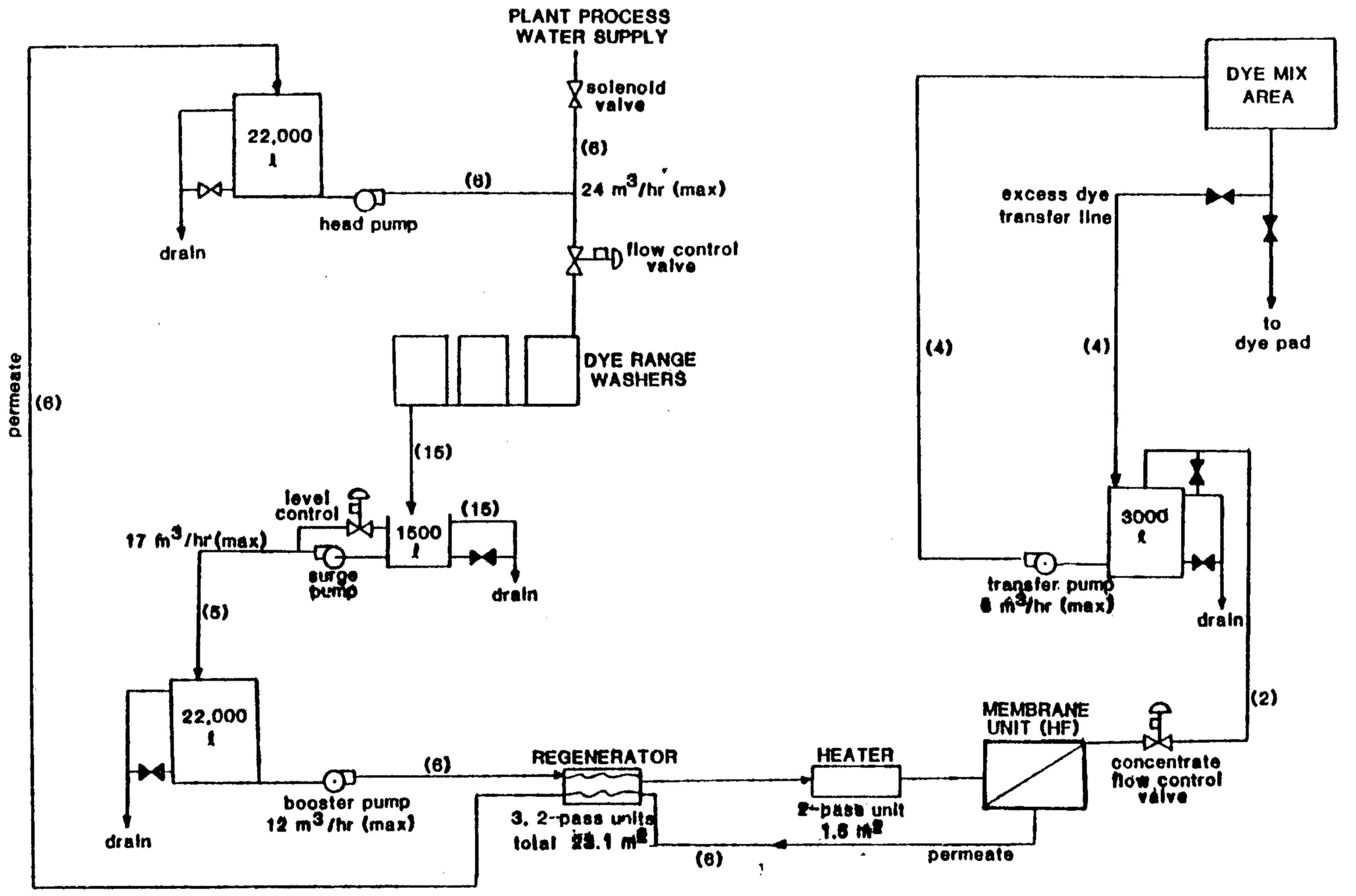


Figure 6. Recovery System Schematic. (Line sizes are indicated in paratheses in cm. All tanks are insulated fiberglass. All lines are stainless steel.)

and electrical work continued through November, 1980. At this time membranes had not been installed, however, the recovery system controls affecting range operation, particularly the flow rate to the washers, were operational.

Hyperfilter modules were delivered at an approximate rate of one per week starting January 20, 1981. Before the recovery system was completed on May 1, 1981, several membrane support tubes were broken due to a control characteristic subjecting the HF system to a pressure shock during automatic start-up. This control characteristic was changed to avoid pressure surges on start-up. Broken tubes were repaired in September, 1981.

Another significant system modification resulted after initial permeate production rates from the HF unit were below design levels primarily because the membrane operating temperatures were below the design level of 85°C. The original recovery system design (5) had no provisions for control of the temperature of the wastewater supplied to the HF unit. The HF unit thus operated at the temperature of the wash water as controlled at the range. The operating temperature was to be 85°C determined by range testing (5) but process changes since have reduced this temperature to less than 60°C.

Tests with the entire system showed that at design temperatures the HF unit would deliver the rated permeate flow so regenerative heat exchangers and a heater were installed during August, 1981. Three regenerative shell and tube heat exchangers are piped in series with permeate flowing countercurrent to the wastewater flow heating the wastewater for HF unit processing and cooling the permeate for use on the range. Because the heat exchangers are not 100% effective and the permeate flow is less than the wastewater flow by the amount of HF concentrate, a heater consisting of a shell and tube heat exchanger uses steam to raise the wastewater temperature about 5°C after it leaves the regenerative heat exchangers.

SECTION 6

HF UNIT PERFORMANCE

The membrane performance of the HF unit is judged in terms of quantity of wash water filtered for recycle and the quality of the water, i.e., the removal of chemicals (dyes, gums, etc.) from the filtered water permeate. The performance of the HF membranes is a function of the operating variables that produce instantaneous effects, e.g., chemical concentrations, temperature, etc., and long term effects such as foulant accumulation and membrane stability. The effective life of the HF unit is defined as the time interval that component failure does not impair the utility of the total unit. The effective life of a membrane is defined as the time interval that the levels of quantity and quality of water are acceptable for recycle.

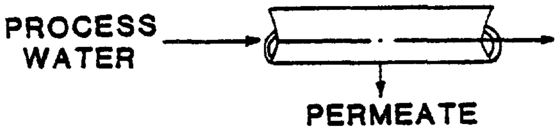
In this section, membrane performance is discussed in light of a mathematical model developed to describe (predict) the quantity of water produced as a function of the wash water characteristics: chemical concentrations, temperature, pressure, and fluid velocity. The experience with foulant accumulation and membrane washing is also presented. The build-up of chemicals in the wash water during a period of closed cycle operation of the dye range is presented. The replacement history of membranes and porous tubes is also presented in the discussion of membrane and HF unit life.

The plans for study and improvement of membrane performance during the continuation of this project through September, 1983 are mentioned.

PERMEATE QUALITY (MEMBRANE REJECTION)

The separation achieved by a membrane is defined as "rejection." Rejection is the fraction of a particular material that does not pass through the membrane. This definition is illustrated in Figure 7(a) for a differential membrane element. The Single Pass membrane system arrangement is illustrated in Figure 7(b). In the Single Pass system the fluid progresses continuously from the feed pump to the final discharge through the concentrate control valve. The water that permeates the membrane is removed from modules, segments of the system, or as a composite whichever is most convenient. The demonstration HF unit has provisions for sampling the water permeate from each of ten modules. For recycle the water from all modules is pumped into a common collection tank thus forming a composite. The rejection in terms of the composite permeate quality is related to the rejection of each membrane element integrated over the total membrane area of the system. This relationship is presented in Figure 7(b). The degree of conversion from wash water

CARRE DWG. NO. 313H

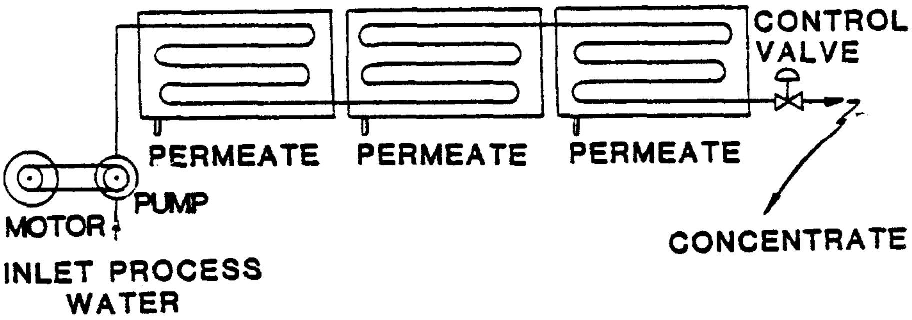


$$\text{REJECTION, } \gamma = 1 - \frac{C_p}{C}$$

$C =$ Process Water Specie Concentration

$C_p =$ Permeate Specie Concentration

Figure 7(a). Differential Membrane Element.



$$\text{RECOVERY, } R = \frac{\text{Volume Flow of Permeate}}{\text{Volume Flow of Inlet Process Water}}$$

$$\frac{\text{Composite Permeate Specie Concentration}}{\text{Inlet Process Water Specie Concentration}}, \frac{C_p}{C_f} = \frac{1-(1-R)^{1-\gamma}}{R}$$

$$\frac{\text{Concentrate Specie Concentration}}{\text{Inlet Process Water Specie Concentration}}, \frac{C_c}{C_f} = (1-R)^{-\gamma}$$

Figure 7(b). General Single Pass Membrane System Arrangement to permeate is called "recovery," (R). Recovery is simply the ratio of the volume of the permeate to the volume of wash water being supplied to the HF unit, Figure 7(b).

The material rejected by the membrane is concentrated as the wash water passes down the Single Pass system. The degree of concentration depends on the recovery as well as the rejection. This relationship is also shown in Figure 7(b).

Permeate Quality Versus Time

Samples of the wash water feed to the hyperfiltration unit and the composite permeate are checked for color periodically. Color is the parameter of primary interest for reuse of the permeate. A list of typical data, one set for each month of the demonstration, are presented in Table 4 to illustrate that color removal has been maintained. The color in the permeate has ranged from undetectable to about 5% of the color in the wash water. The data in Table 4 are for the composite permeate from all membranes in service at the time. No data are included in this listing for periods when any module is malfunctioning. As discussed later in this section, a malfunctioning module is immediately isolated from the collection of composite permeate. The history of individual module availability is tabulated later.

Build Up of Chemicals

Closed cycle operation of the dye range raises the question of build up of chemicals in the wash water. Build up can occur because rejection is less than 100%. The build up is limited, however, by the introduction into the system of fresh water. Fresh water enters with the dye pad solutions that are mixed in the drug room with plant water. This flow of water varies from about 9 liters per minute to 35 liters per minute depending on the details of a particular production lot. Of course, a corresponding amount of water is removed from the washers since the fabric is initially dry but leaves the last washer saturated containing about one kilogram of water for each kilogram of fabric.

To date, complete closed operation of the dye range has occurred only for periods of 2-4 hours duration, after which no recycle occurs for a corresponding time period while permeate is accumulated. A detailed evaluation of the chemicals introduced in the dye pad, the composition of wash water from the range, and the quality of permeate generated by the hyperfiltration unit were, however, monitored during a controlled period of complete recycle. The analysis of the results are presented in Appendix B. Taking into account the time lag involved between when the dye solution is deposited on the fabric and when wastewater is pumped into the hyperfiltration unit, the permeate concentration follows the concentration of material applied in the pad. Any accumulation of material would cause a deviation from the simple relationship between pad composition and the permeate composition. For example, during the course of the controlled recycle experiment, the concentration of dyes being used varied by 50 to 1. The concentration of dissolved solids in the

TABLE 4.

Selected Examples of Membrane Color Rejection Throughout the Course of the Demonstration

Date	Wastewater (absorbance)	Permeate Color (absorbance)
6-2-81	1.1	0.058
7-30-81	0.65	0.0
8-18-81	0.75	0.01
9-30-81	0.20	0.015
10-9-81	0.76	0.02
11-24-81	0.96	0.021
12-15-81	1.0	0.015
1-20-82	0.23	0.012
2-19-82	2.7	0.031
3-2-82	0.68	0.014

permeate varied in a way predicted simply by the rejection of the membrane system and the time for the dye chemicals to pass through the washers and the recovery system (Appendix B). Consequently it is concluded that there is no significant build up of chemicals in the wash water.

A quasi-steady state condition of permeate quality with dye solution is established in less than 2 hours from the moment permeate is used in place of plant waters. This quasi-steady state is essentially in equilibrium with the concentration of material having been introduced into the dye pad. There is a wide variation in permeate quality because of the wide variation in dye pad. Thus, it is possible for the fabric production to be sequenced in such a manner that the permeate would be unsatisfactory for recycle if a very heavy production shade were to be followed by a very light shade. The effects of production on the reuse of permeate are discussed more completely in Section 7, Recovery Process.

PERMEATE QUANTITY (MEMBRANE FLUX)

The quantity of permeate generated by the recovery system is a function of the operating conditions at any given moment and a function of longer term effects such as fouling and possibly membrane modification including deterioration and chemical reactions.

The quantity of permeate generated is expressed as membrane flux (i.e., the volume of permeate per unit of membrane area per unit of time). Common units are m^3/m^2/day or m/d. This measure of flux may further be normalized by dividing by pressure to give permeability (i.e., flux per unit of pressure, m/d/Pa.)

Effects of Operating Conditions

The principle operating conditions that are known from the design phase of this project (5) to have essentially an instantaneous effect on membrane flux are temperature, pressure, and concentration of gum. The flux increases rapidly with increases in temperature. Approximately the flux doubles with an increase in temperature of 25°C. The flux increases linearly with pressure up to a limiting value that depends on the presence of macromolecules, e.g. gum. Depending on the concentration of gum, there may also be an effect of fluid velocity. A mathematical model describing the effects of these gum on membrane flux has been developed. This model is discussed in Appendix C.

The measured pressure distribution is compared in Figure 8 with the calculations using the model. The model requires as input the inlet pressure and the flow rate. Experience has indicated that the membrane performance is insensitive to instantaneous values of chemical concentration but may be diminished by long term accumulative effects, e.g., fouling.

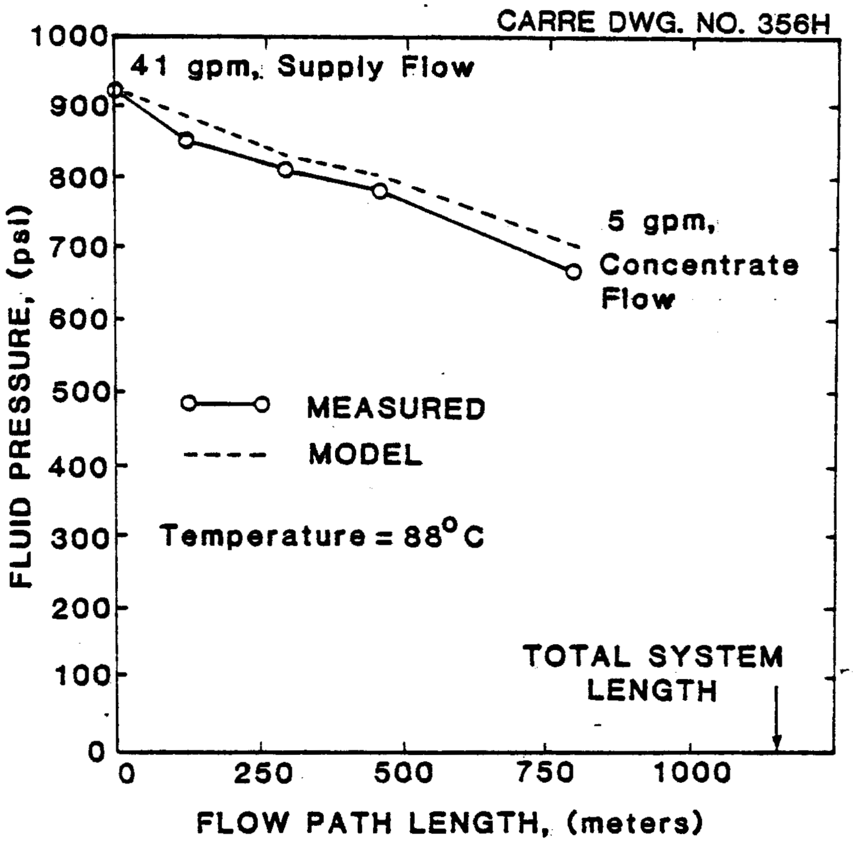


Figure 8. Single Pass Membrane Unit Pressure Profile as a Function of Membrane Length.

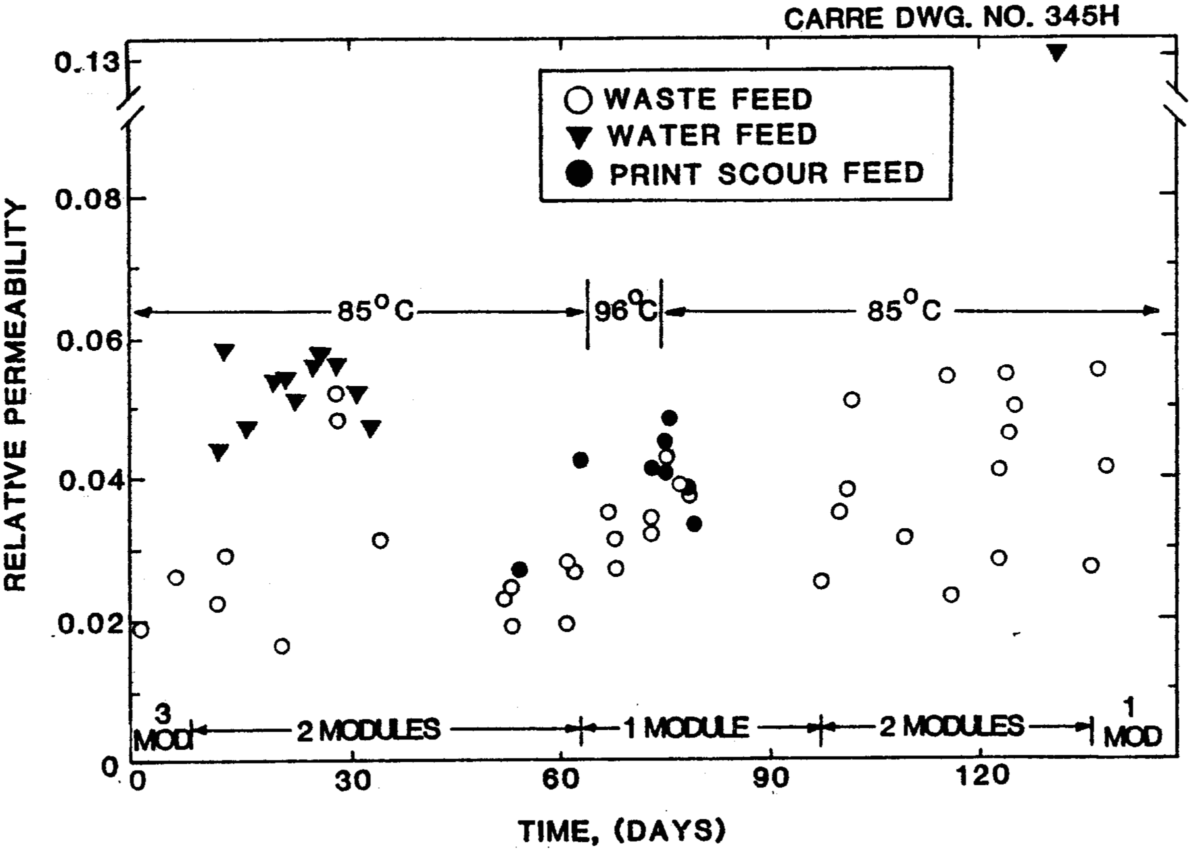


Figure 10. Relative Membrane Permeability Versus Time for the Downstream Three Modules.

The system was designed to have lower velocities in the initial sections because pilot plant test data indicated limited velocity effects in the regions where the concentrations had not progressed extensively. Consequently, the initial sections of the membrane system were designed to have velocities in the range of 1.5 to 2 m/sec. The outlet section of the membrane unit where the concentration of gum is highest, was designed to have velocities ranging up to 3 m/sec. However, as fouling progresses to the point that the membrane system is not capable of handling the full pump output, the velocity in the inlet section of the membrane system actually decreases while the velocity in the outlet section is maintained constant. The outlet velocity is constant because the volume of concentrate leaving the system is controlled. Thus any fouling effects that are associated with velocity will become progressively worse in the inlet section. At the flux levels indicated in January and February, 1982, the maximum velocity entering the first portion of the membrane system had decreased to about 1 m/sec.

In the outlet section of the membrane system the velocity is dependent on the number of modules installed. With three modules in place, the velocity varies from 2.5 m/sec. at the entrance of the section to about 3 m/sec at the exit of the membrane system (when controlled at 5 gallons per minute of concentrate). With two modules in place, the velocities are about 3.5 m/sec to 3 m/sec. And with one module in place, the inlet velocity goes as high as 5 m/sec. While there is a lot of scatter in the data presented in Figure 10, there is clearly an increased unit output associated with the period starting 63 hours (December, 1981) when there was only one module in place.

Following the extensive cleaning that took place in October that resulted in high membrane permeability while operating on water, there was a decline in membrane performance in the outlet section corresponding very closely to that occurring in the inlet section of the system. However, in early December the performance of the outlet section of the membrane system began to improve. And even though there is a large scatter in the data, the membrane performance has remained high (more than 50% higher than the permeability on December 1) throughout the period when two modules were again in place.

In summary, the flux performance of the membrane system is not well understood. The differences in the performance of the inlet section and outlet section of the membrane system is also not well understood. The effects of washing are discussed in the next paragraph. The role of foulants and the effects of velocity and membrane position in the single pass system will be studied further during the extension period of this demonstration project.

Membranes are subject to fouling due to both dissolved and undissolved components in the membrane system feed stream. In many cases particular feed constituents can be identified as foulants and can be washed from the membrane surfaces at time intervals such that the time averaged membrane performance is increased over what it would be without washing. Membrane washing during this project has been complicated by the large number of industrial chemicals used in the dye range processes, particularly when new chemicals

Effects of Membrane Fouling

Membrane fouling is defined as a decrease in flux that is not an instantaneous reversible effect of pressure, temperature, velocity, concentration, or other controlled operating variable. Fouling is usually categorized as (a) an alteration in the membrane due to chemical reaction with the waste stream or (b) an accumulation of a deposited layer. An example of alteration in the membrane is reactions between the charged membrane and metal ions Cu++, Mg++, Ca++, Fe+++, etc. that commonly occur in the wash water. Reactions can also occur with polymers. The ZOPA membranes are particularly sensitive to cationic polymers. Examples of the type of deposits that may occur are:

(1) undissolved dye particles
(2) calcium carbonate, or similar insoluble compounds of the metals
(3) undissolved fraction of the guar gum which could form a carbohydrate film
(4) fine lint and other suspended solids, perhaps in combination with the gum,
(5) the insoluble reaction products of basic and direct dyes (the two major classes of dyes in use on the dye range),
(6) silicone and other oils used as anti-foam agents, and
(7) biological material that may form in the storage tanks.

Because the removal of foulants depends on the nature of the foulant, the washing procedures tested have been selected to attack one or more of the types of fouling potentially present. The sequence of cleaning is also important, for example an oil coating will interfere with the acid cleaning for calcium carbonate and will also interfere with enzyme removal of a carbohydrate film.

In Figures 9 and 10, apparent membrane permeability, based on the inlet pressure, is plotted for the period of October, 1981 through February, 1982. Figure 9 shows this information for the first seven modules of the ten module membrane system. Figure 10 shows similar information for the outlet section of the membrane recovery system. This outlet section contained at various times three modules, two modules, and one module. The exact configuration at any time is indicated in Figure 10. Examination of the two figures indicates that perhaps different phenomena are being experienced in the two portions of the membrane system. The fouling rate seems to have progressed to a greater extent and in a somewhat different manner in the first seven modules of the membrane system. The reason for the difference in the performance of the two sections of the membrane system is not well understood. There are some obvious differences in operation that may be contributing factors. Possibly the seven modules which are upstream in the system provide a large surface area for the absorption or deposition of foulants. It is also true that the fluid velocity in this first section is lower than fluid velocity in the outlet sections of the system.

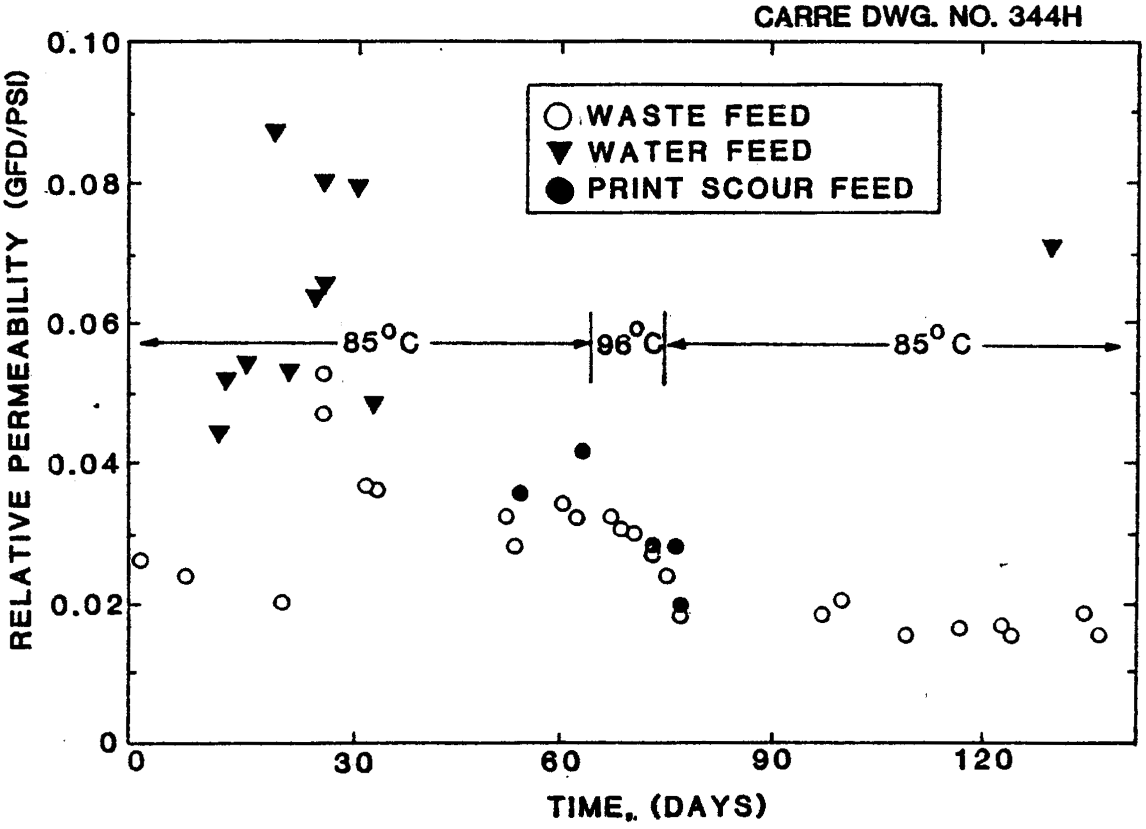


Figure 9. Relative Membrane Permeability Versus Time for the Upstream Seven Modules.

are introduced with no knowledge of their potential as foulants. The following paragraphs describe washing conducted through February, 1982. Membrane washing studies will continue as the project continues. (On May 15, 1982 it was learned that during October 1981 the use of a cationic polymer as a dye fixing agent was greatly increased. The role of this cationic material on flux decline is being studied).

Washing with hot water, at high temperatures (up to 100°C), and at high velocity over the membrane surface, can be effective and has been used in this project. High temperatures increase solubility and diffusion rates of most substances and high flow rates cause increased shear stress at the membrane surface. While the recovery system membranes were being installed over a ten-week period, water was run at frequent intervals when range effluent from direct dye lots was not available (range effluents from other dye types were introduced later). Soon after the final membrane segment was installed a steam coil was added to the wash tank and hot water (up to 95°C) could be used to wash the membranes. To compare the results of washing, membrane performance data were recorded while operating on water and used to calculate a system average membrane permeability.

A list of membrane system permeabilities on water following various washes are listed in Table 5. Before April 24, 1981, the system operating temperature was limited to 60°C. The membrane permeability increase between April 24 and May 11 could have resulted from increased washing temperatures.

Between May and October, 1981, the recovery system operating time increased to 3 shifts per day on all range processes except bleach. An intensive effort to wash the membranes was made in October, 1981, a summary of which is listed in Table 5. Water washing over a weekend (approximately 30 hours) resulted in lower membrane permeability than previously obtained after water washing (October 12, 1981, versus May 11, 1981). Waste was then run on the system for 8 hours followed by an overnight water wash (approximately 16.5 hours) resulting in a membrane permeability on October 13, 1981, of about half that obtained in May, 1981. Washing with a water and acetic acid solution at a pH of 4.0 was conducted to remove hardness components. Analysis of the cleaning solution after washing indicated that nearly a kilogram of hardness, as $CaCO_3$, was removed from the membrane surfaces and the membrane permeability was increased to within 10% of the previous best system permeability in a fraction of the washing time required to wash with water alone, Table 5. Results of a second pH 4 wash after operating with waste resulted in the same membrane permeability as after the first pH 4 wash.

The washing sequence continued with another pH 4 wash followed by an enzyme wash aimed at removing organic foulants such as gums. The combination of washes resulted in the highest permeability thus far on October 20, 1981, about 250% of design capacity.

By February, 1982, the membranes had been exposed to more waste without washing than at any previous time. Also, an oily substance was found in the

TABLE 5. MEMBRANE PERMEABILITY ON WATER AFTER WASHING

Date	Washing Procedure	Relative Permeability*
4-24-81	Water at 60^0C	1.4
5-11-81	Water at 85^0C	1.8
10-12-81	Water at 85^0C, 30 hours	1.4
10-13-81	Water at 85^0C, 16.5 hours	0.9
10-13-81	Acetic acid solution, pH = 4	1.6
10-16-81	Acetic acid solution, pH = 4	1.6
10-20-81	Acetic acid solution plus enzyme	2.5
2-10-82	Sodium bicarb. + detergent Acetic acid solution, pH = 4 plus enzyme water at 85^0C, 16.5 hours	No Data
2-11-82	Acetic acid solution, pH = 4 Sodium hydroxide + detergent Acetic acid solution Citric acid solution Water at 85^0C, 16.5 hours Acetic acid solution	2.2

*Relative permeability is defined as the capacity of the unit compared to the design capacity.

wash tank. The oily substance was suspected to be the residual from antifoam chemicals used on the range. A sodium bicarbonate (pH = 9) and anionic detergent solution was used to wash as recommended by the antifoam supplier. A series of washes over a 3-day period including water, solutions of acetic acid, citric acid, sodium bicarbonate and detergent, and sodium hydroxide and detergent resulted in a membrane permeability of within 12% of the maximum permeability obtained in October. No one washing procedure caused a substantial flux increase which could indicate none were primarily effective in removing the foulant. However, if the foulants are layered on the membrane surface and the various washing procedures remove particular foulants, the sequential washing may be necessary. For example, the oil coating would prevent a pH = 4 wash or an enzyme from being effective on hardness scale or a gum layer.

Membrane washing procedures appear to restore membrane permeability even after prolonged operation with effluents from the many range processes and variations within given processes. The time required to wash may depend on the amount of effluent processed since the last washing and/or the type of foulants in the fluid. Membrane washing in this complex and varying fluid is not fully understood at this time and studies will continue as the project continues. The study will include investigation of possible substitution for production chemicals found to be particularly strong foulants.

Because of the severe fouling problem discussed in this section, the development of effective washing procedures is important. Only a test section exposed to the history of the recovery system can provide credible results in the study of washing procedures. Two modules have been kept out of service for periods of time to study membrane washing. During the project extension period modules or membrane segments may be removed from the unit for use in washing studies.

MEMBRANE STABILITY

Membrane stability is judged by the color rejection because color (indicative of dyes) in the permeate is the parameter of primary concern in recycle for this application. The expected mode of failure is a loss of dye rejection that may be accompanied by a rapid increase in flux if in fact the failure mode is an attrition of the membrane material. The experience with the 70 individual membrane segments in the ten modules of the hyperfiltration unit are included as data on membrane stability.

Of the 70 membrane segments in the recovery system, 46 segments have remained in continuous service since their installation, see Table 6. The oldest has been in place for 16 months (from January 17, 1981, through May 17, 1982). Of the remaining 24, 11 have been re-membraned after replacing a broken tube (each segment contains 12 or 16 tubes); four segments have been re-membraned after being used in washing studies, four are currently bypassed with (suspected) tube breaks, and 6 are being re-membraned after being used in washing studies. One of the segments currently bypassed is in Module #8 that was re-membraned, thus this accounting totals 71, instead of 70. No membrane has been replaced due to loss of color rejection.

TABLE 6. RECOVERY SYSTEM AVAILABILITY

Module	Installed	Removed from Service	Returned to Service	Comments
10	1/17/81			Continuous Service
9	1/29/81	12/3/81 - 7 segments		Replace 1 broken tube; Reform membrane. Module being used in washing studies.
8	3/11/81	6/16/81 - 3 segments	1/8/82	Replace 3 broken tubes; Reform membranes. Module used in washing studies.
7	2/24/81			Continuous Service
6	3/3/81	7/17/81 - 1 segment	9/24/81	Replace 1 broken tube.
5	3/16/81	6/3/81 - 1 segment		Replace 3 broken tubes.
		6/9/81 - 1 segment		
		6/18/81 - 1 segment	9/18/81	
		10/5/81 - 1 segment		
4	3/20/81			Continuous Service
3	3/27/81	5/19/81 - 1 segment	9/3/81	Replace 1 broken tube.
		1/25/82 - 1 segment		
2	4/8/81			Continuous Service
1	4/10/81	4/14/81 - 1 segment		Replaced 2 broken tubes.
		4/22/81 - 1 segment		
		1/25/81 - 1 segment		

The membranes in Module #9 were originally thought to have "failed" because the color rejection was observed to decrease quickly without a corresponding major increase in permeate flow rate - as would be expected from a broken tube. Only after dismantling the module following washing experiments was a tube crack found - tightly plugged by fine lint - thus explaining the lack of high flux which would normally be indicative of a broken tube.

There has been no indication of membrane deterioration during the more than one year of operation. And even allowing for Module #9, the membranes have performed without loss of rejection to date. Four of 10 modules have been in continuous service.

The color rejection for Module #9 and Module #10 are presented in Figure 11. These were the first two modules installed. Module #10 is the outlet module of the system. Module #9 is next to the outlet and is operated in parallel with Module #8. These modules are operating with concentrated wash water.

The rejections presented in Figure 11 are based on the mixed permeate from the entire module compared to the fluid entering the module. This averaging provides a slightly conservative estimate of rejection. The scatter in the data may be partially explained by the range in absolute values of the absorbance measurements. The fluid entering the modules had absorbances in the range of 0.36 to 12.0 and the permeates had absorbance readings in the range of 0.005 to 0.17 (except for the low rejection noted for Module #9 just prior to its removal from the system). The rejection of direct dyes is higher than the rejection of basic dyes but the rejection of each is stable.

HF UNIT AVAILABILITY

The hyperfiltration unit availability is determined by the reliability of the components and the time required for replacement. The availability of a particular segment of the membrane area is determined by the membrane itself and by the integrity of the support structure. The availability of the hyperfiltration unit is enhanced by the design that:

(1) incorporates only 1.5% of the membrane area within one segment,
(2) provides for quick identification and location of a membrane or tube failure and includes the ability to bypass the single segment containing the failure, and
(3) provides for rapid replacement of the failed segment at a time scheduled for convenience.

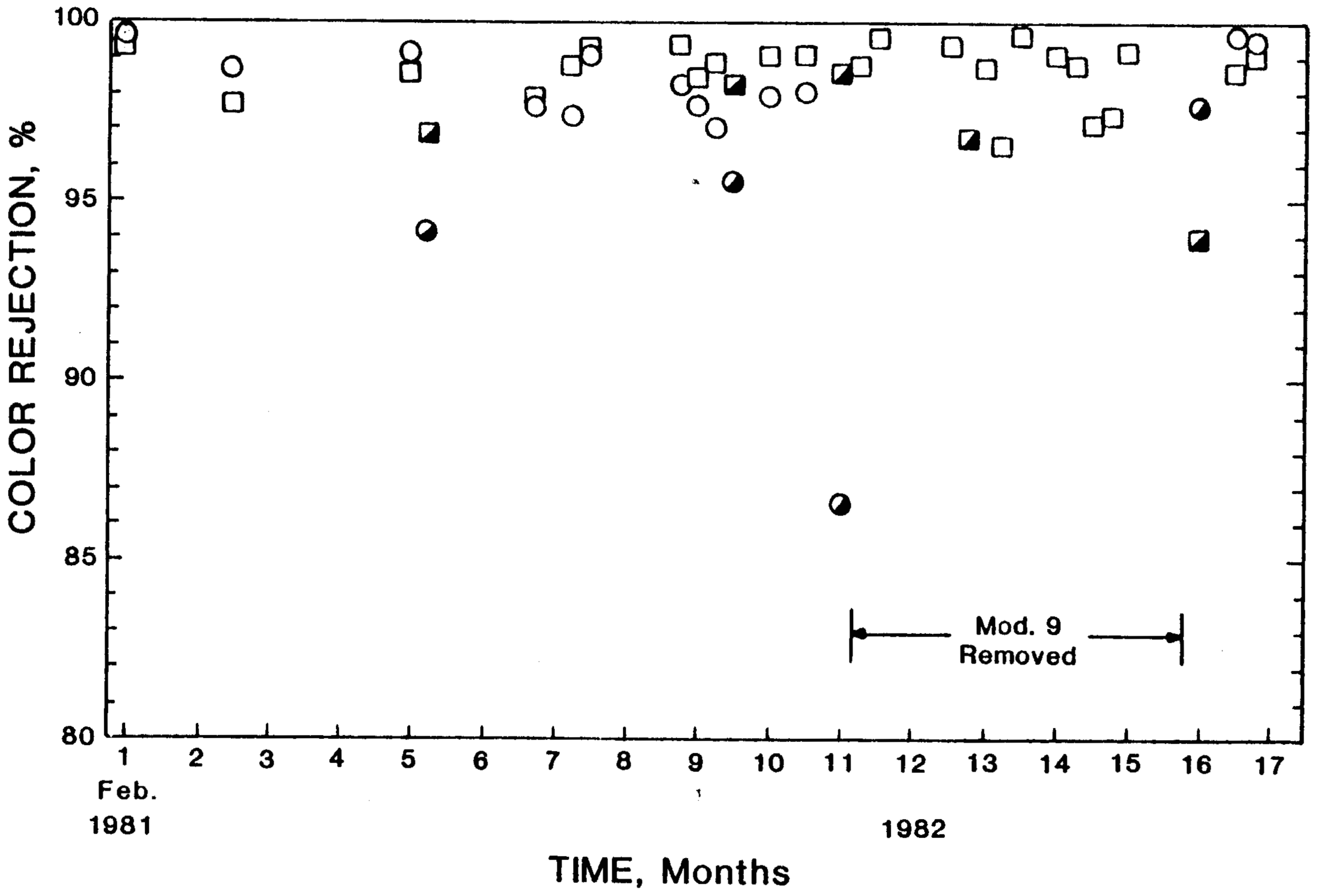


Figure 11. Color Rejection for Module #9 and Module #10 (Module #10□; Module #9○; Open symbols are for direct dyes; shaded symbols are for basic dyes).

The composite permeate from each of 10 modules is monitored in terms of flow rate and quality (qualitative color) using sight glass flow meters (rotometers). In the event of a tube failure, the module can be immediately identified and bypassed (requiring the entire unit to be off-line for less than 30 minutes). By individually checking in turn each of three groups of membrane segments within a module and then each segment of the group containing the failure, the single segment can be bypassed and the full system (minus the segment containing the failure) can be returned to service within 2 hours. (The 23 m^3 capacity of the wastewater and recycle water tanks allow for over 2 hours of normal range operation with the HF unit off.)

Three membrane segments are stored as spare parts. A module can be bypassed, removed from the system, dismantled, and reassembled with a spare membrane segment and returned to the recovery system within four hours.

These procedures have been demonstrated during the course of this project. The experience with membrane availability is summarized in Table 6. As delineated in the discussion of membrane stability, a total of 11 tubes, of the 1,000 in the unit, have been replaced. Four additional segments require replacement at this time. We believe that the failure rate of tubes will approach zero as the initial flaws in the tubes are eliminated.

SECTION 7

RECOVERY AND REUSE EXPERIENCE

The recovery system is shown in Figure 4 with the dye range. The recovery system consists of a wastewater tank, pumps, hyperfiltration modules and concentrate and recycle water tanks. The wastewater flows by gravity from the dye range to a surge tank and is pumped to the wastewater tank. There is a period of time between each production lot for cleaning the range equipment and filling the dye pad. However, the water flow is continued during the down period to reduce the color in the water in the washers by about 30%. A production lot can be treated in the hyperfiltration system as a batch containing chemicals from a single dye formulation when this knowledge of the composition is important in reuse or disposal of the chemicals.

The wastewater (HF supply) is usually highly colored and removal of at least 97% of the dyes is considered necessary to avoid possible staining of fabric during reuse. The auxiliary components must also be removed sufficiently to provide recycle water with concentration differences suitable for effective washing of the fabric.

The HF concentrate contains dyes at concentrations much lower than those in the dye pad solution, but with comparable concentrations of the auxiliary chemicals. Based on pilot studies, reuse of the concentrates in the dye formulations is feasible with about 75% savings in auxiliary chemicals and an average of about 10-20% in dyes depending on the dye class (5). Effective reuse of the HF concentrate depends on the ability to add dyes to achieve hue and crocking characteristics needed in production. HF concentrate reuse can be enhanced by judicious scheduling of dyeing lots by shade and dye class and by using the experimentally determined guideline of using only 25% of auxiliary components in every reuse dye formulation. Appropriate scheduling has been initiated but is often interrupted by production demands.

Laboratory tests were conducted to evaluate reuse in preparation for the full scale demonstration. The results of these laboratory tests have been updated. These results are included in Appendix D for the convenience of the reader.

PERMEATE REUSE

Wash water from all of the production processes, except bleaching, have been reused after hyperfiltration. The only parameter used to judge permeate quality for reuse is the color. (Selected values of color in the permeate were presented in Table 5.) Bleaching effluents are not hyperfiltered until the pH adjustment can be accomplished to permit its reuse. The permeate has been used in all classes of processes performed on the range; washing of all dye classes, bleaching, and scouring.

Over a million meters of velour upholstery fabric have been washed or scoured with permeate. All of this fabric was produced and supplied to customers as standard quality. Permeate is intermittently supplied to the range during all full scale operations. In normal operation permeate is automatically used when it is available. Plant water is automatically provided as the alternative source.

In recent weeks, with the unit operating with 80% or more of the membrane area in place, full-strength permeate has been used on 50-60% of the fabric produced. Although the HF unit is capable of matching the wash water requirement of the range for a period following cleaning of the membranes, its average supply of permeate has been sufficient to wash only about 60% of production.

Interruptions in the HF unit operation occur during membrane cleaning and evaluations of the HF unit performance characteristics. In addition, the range operator discontinues the supply of wastewater to the HF unit whenever the range wash boxes are normally emptied and refilled, i.e. when a dark shade is followed by a light shade or a difficult process crossover occurs. These judgements have been included in the standard operating procedures to utilize operator experience.

This operator experience rule is conservative. In one observation period covering two weeks, the wash boxes were emptied and refilled 43 times. Despite the operating instructions (stated above), the wash water was supplied to the HF unit during the production of 27 of these lots. Only one was not successfully reused after hyperfiltration. The single case of staining occurred when wash water from a lot dyed a dark shade (the formulation contained 32.5 grams per liter of basic and direct dyes) was reused on a lot dyed a light shade (the formulation contained only 0.3 grams per liter of direct dyes). This 108:1 ratio in dye formulation concentration normally produces an even greater ratio in the dye concentrations in the wash boxes because the dye exhaustion is relatively less for the dark shades of basic/direct dyes than for the light direct shades. Such a large ratio in the dye concentration for consecutive lots is a severe test for the hyperfiltration unit where basic dye overall rejections of about 90% and direct dye overall rejections of 97% are routinely obtained. This single result combined with the successful hyperfiltration and reuse of effluents from hundreds of production lots suggests very few redyes would result if all range effluents were hyperfiltered without operator selection (See Appendix E).

CONCENTRATE REUSE

The full scale use of HF concentrates in production dye formulations has been initiated in a manner designed to minimize complications by utilizing hue and dye class in the reuse.

Reuse Concept

About 30% of the production is dyed to one of three hues of tan to brown. These print base dyeings normally occur in 80 piece (4,000 meter) lots. Because there are only three shades involved, concentrate reuse between these shades can be standardized using formulas developed in the dye laboratory. The initial efforts at full scale reuse of HF concentrate is emphasizing these three shades.

Laboratory Formulations

Formulations were developed in the dye laboratory with HF concentrate generated from washing the medium and the dark shades. The laboratory procedure began.by striking a fabric sample using as received HF concentrate. A strike consists of running a fabric sample in a prototype of the dye range dye pad and steamer. Striking with as received HF concentrate indicates the dye content available for reuse. HF concentrate from the medium and dark shades contain too much dye to allow formulation to the lightest shade. They could, however, be reformulated to their original shades respectively and to each other. HF concentrate from the lightest shade contains so little dye that it can be reformulated to itself and to any production shade by adding, without altering, the dye in the standard formulations. The reuse formulations developed in the laboratory (5) indicated that only 25% of the auxiliary chemicals are needed when HF concentrate is used on a production basis.

Full Scale Reuse

HF concentrate from the darkest of the three brown shades was collected and reformulated to dye a 4,000 meter lot of the medium shade. Only 2,400 liters of HF concentrate was returned to the drug room so 1,600 liters of water were added to the concentrate to make the 4,000 liter volume necessary.

After the initial 4,000 liter of formulation was mixed, based on the laboratory formula, three dye additions were necessary. The initial formulation produced a strike that was too light (probably because of the 1,600 liters of water added to achieve required volume) so the first addition was made. Results after the first addition of dye were still slightly too light so the second dye addition was made. This resulted in too much orange so an additional add was required resulting in the proper shade. When the 4,000 meter lot was run on the dye range it came out slightly dark indicating too much dyes were used. No auxiliary chemicals were added to the formulation. The total amount of dyes used was 25% more than would have been required in a standard formulation (starting with plant water only). However, assuming that after the first dye add the shade would have been acceptable (based on dye house supervisor comments), a dye savings of 5% would have resulted. HF concentrate reuse will continue on a production scale among these three tan to brown shades before expanding the effort to other shades.

SECTION 8

DISPOSAL OF HF CONCENTRATE

The HF concentrate stream will not be reuseable in every instance because of production schedules. The guar gum pad thickener is biodegradable and causes the HF concentrate to have a useful storage life of about 24 hours. Also since an average of 6 different dye lots are produced each 8-hour shift, storage for reuse for more than one or two shifts would be complicated. Consequently the disposal and/or treatment of some HF concentrate will probably be required. Because of the high concentration of chemicals and corresponding small volume of the HF concentrate, treatment methods not generally applicable to textile wastewater may be considered. Several treatment methods that may be applicable due to the smaller volume include application to agricultural land, additional concentration by membranes followed by drying for land fill or incineration and oxidation by exposure to ozone. A cost estimate for direct land application is presented in the Section 9, Cost Analysis.

Discharge from the range occurs in two forms: the wash water discharged continuously and the drop from the dye pad following each production lot. The dye pad drop occurs in a time expected to allow direct addition to the corresponding HF concentrate. The combined concentrate and pad drop is of known composition and its reuse or disposal can be planned with that knowledge. This combined concentrated fluid becomes feed for a disposal system when the mixture cannot be reused in dye formulations. The waste stream is still too dilute at this point to go directly into a dryer so further concentration by membranes was studied. Permeate from this membrane separation may not be suitable for wash water because of the dye content but it can be reprocessed through the full scale recovery system. Overhead vapors from the dryer can be condensed in a feed preheater to recover the energy and the water then recycled. Solids from the dryer can be incinerated or disposed of by some other method.

A thin-film dryer was tested with various solutions of HF concentrate and dye pad drop mixtures in 200 liter batches. Two concentrations were tested, 4 and 6 percent total solids. A third solution contained 1.5 percent total solids and was too dilute for successful processing with the dryer test equipment. The 4 and 6 percent solutions were obtained by concentrating dye pad drops with HF membranes. Dye pad drops were used instead of range rinse water because limited membrane area made it impractical to process the 10,000 and 14,000 liters needed to generate the 4 and 6 percent solutions respectively. It was possible to obtain steady-state data for various flow rates and dryer operating conditions for each batch of feed tested. The dryer operating temperature was selected to equal that of standard process steam (175°C). Data were also obtained for a steam temperature of 185°C. Flow rates were limited by the quality of bottoms and amount of entrainment in the overhead vapors.

Low feed rates yield bottom products that had properties like tar in appearance and handling characteristics. The highest flow rates resulted in entrainment of feed solution into the overhead vapor flow. The point of entrainment provides an upper limit to feed flow rate. Preheating feed material to greater than 100C also caused entrainment due to flashing. Between these operating limits, bottom product varies in quality from friable powder to powder plus "tar-like" material.

The results of total solids analysis for bottom and condensed overhead vapors are listed in Table 7. Bottoms characterized as "tar-like" contained between 1 and 2 percent moisture by weight. Bottoms consisting of powder plus "tar-like" material contained between 5 and 10 percent moisture by weight. Bottoms samples consisting of only friable powder contained between 16 and 17 percent moisture by weight. This powder appears damp but retains friability even after being compressed by hand. Because the powder was easier to handle for disposal by incineration, flow rates for various sized dryers were determined to deliver bottoms with approximately 15 percent moisture. Test results indicate a optimum feed flow rate of 0.013 liters per minute per square meter for steam temperature of 175°C with a preheat temperature of 95°C for 8 percent total solids feed to the dryer.

MEMBRANE TESTING

Membrane performance data was obtained with the unused concentrate from the dryer testing. For this test a combination of 1.5 and 4 percent total solids solution was concentrated with membrane equipment to 7 percent total solids. Samples were taken at specified recoveries to determine the concentration of solids in the feed and to evaluate total solids rejection.

Flux versus total solids concentration is plotted in Figure 12. The flux declines with feed concentration. Flux versus pressure is shown in Figure 13 for several levels of concentration. The curves are typical of hyperfiltration of high molecular weight solutes. Flux becomes increasingly less dependent on pressure as the pressure increases. (Velocity variation effects were not studied because the highly colored solution obscured the float in the flow meter.) Color rejection was constant at about 97%.

MEMBRANE/DRYER INTERFACE

Membrane sizing can be based on an average flux for concentrating the waste stream from 2 percent to 10 percent total solids in a single pass system. With an average membrane flux of 1 m/d, the area required to deliver 2 liters per minute at 10 percent total solids from 10 liters per minute of 2 percent is approximately 12 square meters. The membrane cost is $24,000 with about $10,000 for pumping and controls if the additional area cannot be

TABLE 7. TOTAL SOLIDS ANALYSIS RESULTS FOR CONCENTRATE DISPOSAL TESTING

	Sample	Total Solids (%)	Description
Distillate:	1D	.072	
	2D	.054	
	3D	.011	
	4D	.060	
	5D	.050	
	6D	.030	
	7D	.020	
	8D	.035	
	9D	.020	
	10D	.055	
Bottoms:	2B	91.86	(b)
	3B	85.00	(a)
	4B	99.07	(c)
	5B	98.64	(c)
	6B	82.59	(a)
	7B	82.32	(a)
	8B	91.59	(b)
	9B	94.25	(b)
	10B	93.95	(b)

(a) powder
(b) powder plus "tar-like" material
(c) "tar-like material

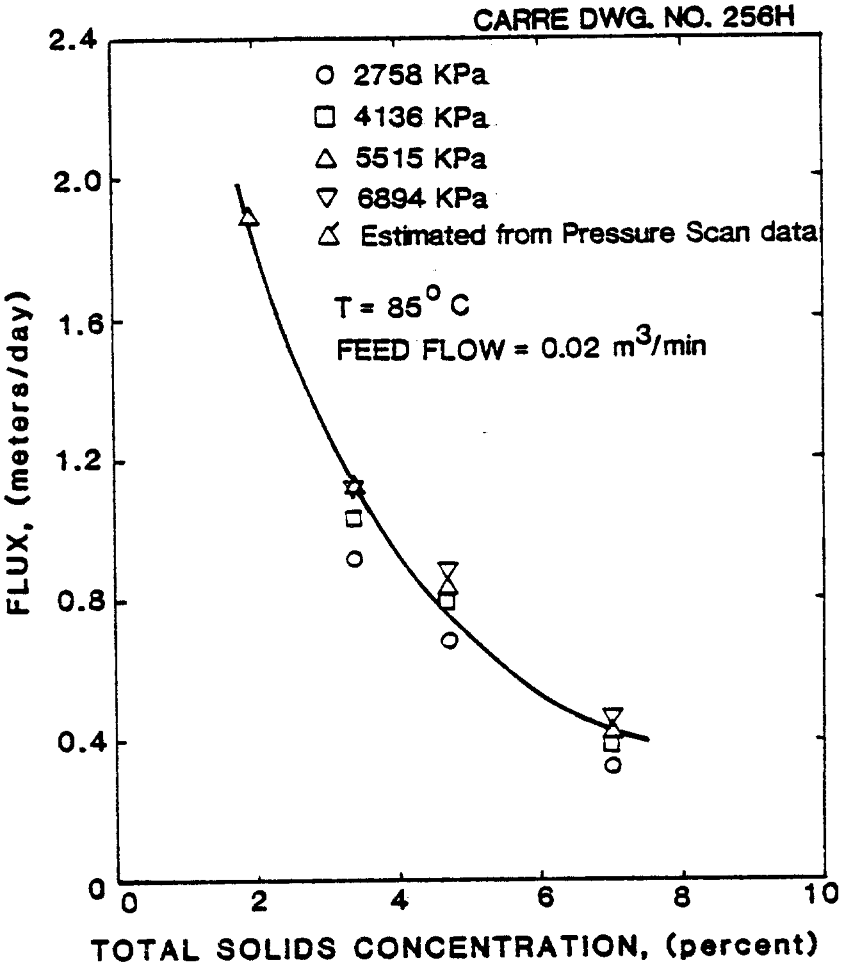


Figure 12. Flux Versus Total Solids Concentration: Concentrate Disposal Membrane Performance.

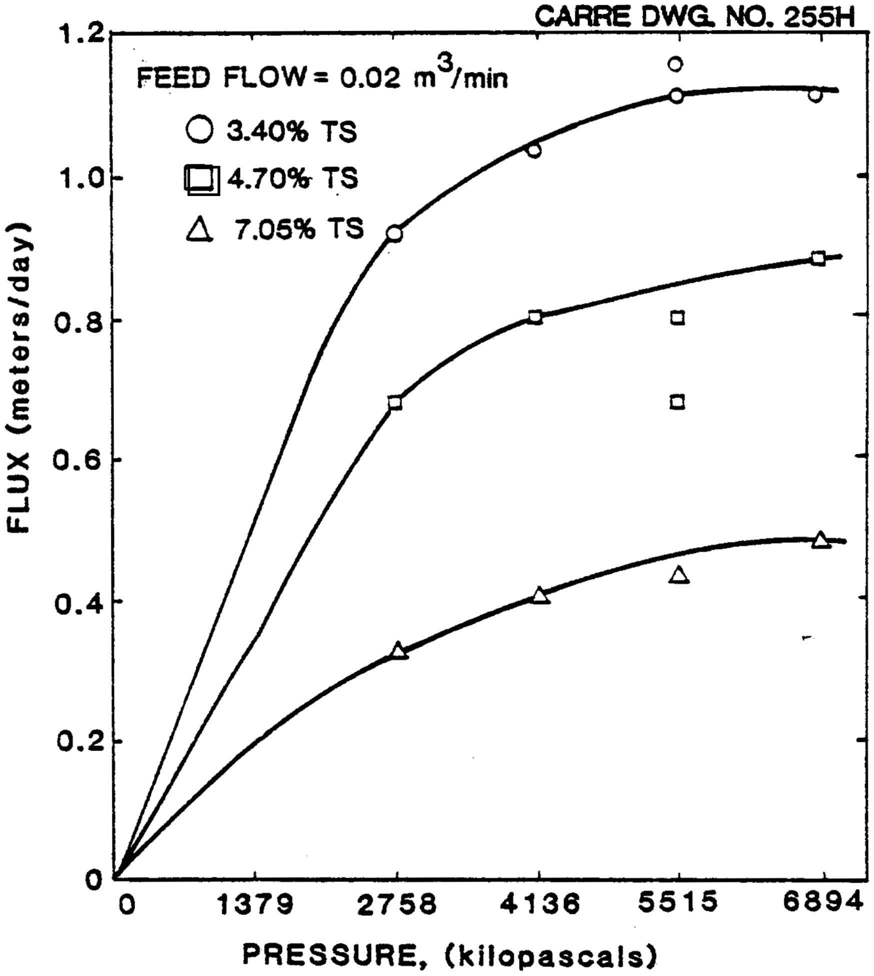


Figure 13. Flux Versus Pressure: Concentrate Disposal Membrane Performance

attached to the existing hyperfiltration unit. The dryer area required to achieve 85 percent solids is 1.4 square meters. Standard dryer sizes are 1 square meter and 2 square meters. The smaller dryer should be adequate because the system can be operated as a batch process. The equipment cost (commercial estimates) for the dryer is about $73,000. An entrainment separator is estimated to cost $7,500.

INCINERATION TEST

Disposal of sludge generated from the testing with the dryer and the hyperfiltration pilot unit was investigated. A laboratory study was conducted to evaluate the incineration characteristics of the sludge, the ash volume generated by incineration, and the toxic characteristics of the sludge and the incinerated ash.

A mixture of powder and "tar-like" material was evaluated. A sample of this material was dried at 105°C for 24 hours to determine the moisture content, and a sample of the residue was ignited in a muffle furnace at 815°C to determine the ash content. These analyses are presented in Table 8.

The sludge ignited readily and generated a yellowish white smoke that was slightly irritating to the nose and throat. Burning the residue in open air over a flame generated a black soot probably due to incomplete combustion. The residue was fluid at incineration temperature, but solidified on cooling to a hard black solid that crumbled easily and had a sulfur-like odor. The ash was scraped from the ceramic dishes, weighed, and subjected to the extraction procedure required to compare with allowable EPA concentrations (6). A sample of the residue before incineration was also subjected to the extraction procedure. The results of the chemical analysis of the extracts are presented in Table 8. Since for every specie analysis the measure of value is below the allowable concentration both the sludge and the ash are classified as non-toxic. It should be noted that dried residue from the membrane/dryer equipment remains water soluble and contains substantial amounts of dyes. Leachant from this residue would be highly colored.

It was noted that the initial pH of the sludge extract was 5.5; the initial pH of the ash extract was 12.5. The sludge extract was adjusted to pH of 5.0 by the addition of 5 mls of 0.5N acetic acid. The addition of the maximum amount of acid recommended by the extraction procedure, 4 mls acid/gram of solid, reduced the pH of the ash extract from 12.5 to pH 9.8. During the extraction procedure, a sulfide odor was generated by the incinerated ash sample; the sulfide concentration in the extract at the conclusion of the extraction was 850 mg/liter.

LAND DISPOSAL

The disposal of the HF concentrate on agricultural land is economically attractive compared to further processing by evaporation and incineration. Based on information provided by representatives of a company engaged in sludge disposal by direct land application (7), disposal of HF concentrate would cost about $15,000/year. This cost is based on a predicted disposal requirement of 4000 cubic meters per year with little or no reuse. Significant reuse of HF concentrate would reduce disposal costs.

TABLE 8. RESULTS OF THE ANALYSES OF HYPERFILTRATION (HF)/ EVAPORATION (E) SLUDGE AND INCINERATION ASH

Contaminant	Allowable (3) Concentration, mg/ℓ	HF/E Sludge (As received), mg/ℓ	Incineration Ash, mg/ℓ
Arsenic	5.0	<0.03	<0.1
Barium	100.0	0.2	0.2
Cadmium	1.0	0.03	0.10
Chromium	5.0	0.28	0.07
Lead	5.0	0.35	0.77
Mercury	0.2	0.0008	<0.0002
Selenium	1.0	<0.01	<0.01
Silver	5.0	0.08	0.09
Endrin	0.02	<0.001	<0.001
Lindane	0.4	<0.001	<0.001
Methoxychlor	10.0	<0.010	<0.01
Toxaphene	0.5	<0.01	<0.01
2,4-D	10.0	<0.01	<0.010
2,4,5-TP	1.0	<0.01	<0.01

SECTION 9

COST ANALYSIS

Economics of this hyperfiltration demonstration project and additional potential hyperfiltration applications in the textile industry are considered in this chapter.

There are four elements of purpose for this chapter: (1) to present the cost and savings of this project, (2) to estimate the cost and savings for future hyperfiltration applications in the textile industry based on the data of this demonstration, (3) to evaluate the sensitivity of costs to major process and operational variables, and (4) to discuss the economic aspects of hyperfiltration as applied to other industrial processes. The actual capital expended for the installation of the demonstration unit is reported and examined in some detail. Because this is a demonstration project, certain costs were incurred that are greater than would normally be expected for a non-demonstration application. These include such items as recording instrumentation to allow reporting of results and engineering costs incurred through changes of scope in the demonstration project. The experience of this demonstration is used as the basis for capital cost projections for other applications.

Hyperfiltration economics are sensitive to a number of parameters; major ones of those will be examined. In this demonstration, the hyperfiltration recovery system was a retrofit to an existing textile plant. Therefore, waste treatment facilities were in place and only savings in waste treatment due to reduced operating cost could be counted. In the case of a new plant where a recovery system would result in a smaller waste treatment system, the reduced capital cost could be taken as a savings against the hyperfiltration system capital cost. Savings through recycle result from reduced consumption of water and reduced water treatment costs, energy conservation, and chemical recovery. Other applications may be more or less conducive to recycling of any of these constituents.

All analyses for this report are done in 1981 dollars with no direct effects of inflation.

All major emphasis for this chapter is on establishing capital and operating costs and savings information based on the demonstration. Examples of measures of merit are given: internal rate of return, payout time, and return on original investment are calculated.

Elements of capital costs and operating expenses are those recommended by the United States Environmental Protection Agency and their standard procedure for cost analysis of pollution control operations (8).

COST DATA FOR THE DEMONSTRATION

Capital Costs Data

Table 9 lists elements of capital costs for the recovery system. The hyperfiltration membrane modules, their support structures, and the high pressure pumping and its associated controls including a pH control system were provided by a single equipment vendor. Both the control panel and a monitoring or display panel were provided independently by another vendor. Engineering was provided by a local architectural/engineering firm. All other items were provided by a second contractor. All other direct costs were taken from invoices submitted by the prime contractor. Freight, sales tax and construction overhead were also taken from these invoices as was the contractor fee.

The total recovery system plant cost $547,000. This is the total depreciable investment since neither interest nor start-up costs were capitalized or provided for. No value was placed on the space required for the hyperfiltration unit and recovery system. Surplus space was available and costs for installation and site preparation are included in the "Other Items" purchased and "Piping."

Because this is a demonstration unit, certain of these capital costs were incurred because of special reporting requirements, delays and changes of scope in the project, and engineering design changes. Table 10 is a revision of Table 9 in which some of the capital costs have been changed to remove the demonstration project effects and to reflect, in the case of the hyperfiltration unit, a 1981 price.

The following are comments on Table 10 and are listed by number (superscript) shown in the table.

1. The revised hyperfiltration unit price reflects an updated 1981 quotation from the vendor. The 1981 price also includes a feed booster pump and a 0.75 m^3 stainless steel wash tank.
2. While the control panel requirements did not change, the monitoring (display) panel was eliminated from Table 10. The panel housed instruments (not included in the panel price) that are necessary only for the special data collection and reporting of the demonstration project.
3. Because the decision to locate the recovery system inside the plant was a mid-project change, two major cost items can be removed from the "Other Items" catagory. These are $17,000 for the external structure and $6,000 required to modify existing tanks so that they would fit through the plant doors.
4. Two items were eliminated from the piping cost. One was a $6,000 wash tank now included in the cost of the hyperfiltration unit; the other was $8,000 in piping changes associated with the decision to locate inside the plant.
5. $18,000 for instruments in the monitoring (display) panel were eliminated. Additionally the $4,000 cost incurred as restocking charges for instruments returned when the design was changed to eliminate a recovery system for the fix chemicals was eliminated.
6. A $12,000 cost to install the monitoring (display) panel was eliminated.
7. Excavation and foundation rework costs of $12,000 are eliminated. (The decision to move the hyperfiltration unit inside the plant was made when, following excavation, it was discovered that the soil foundation would not support the recovery system).
8. The indirect cost reductions associated with the direct cost items are estimated.
9. $4,000 in construction overhead is deleted based on 6 1/2% of $58,000 in direct cost deletions.
10. Additional engineering costs totaling $15,000 above the bid price were deleted. These costs were incurred due to the project changes previously mentioned.
11. The $5,000 reduction in the contractor fee is due to the previously summarized reduction in direct and indirect cost.

TABLE 9. ACTUAL CAPITAL COST DATA FOR HYPERFILTRATION DEMONSTRATION

1.	Purchased Equipment Cost for each major plant item				
	a. Hyperfiltration	$244,000			
	b. Control Panel	12,000			
	c. Monitoring Panel	10,000			
	d. Heat Exchanger	10,000			
	e. Other Items (pumps, tanks, etc.)	37,000			
			TOTAL	$313,000	
2.	Direct Field Materials				
	a. Piping	$ 42,000			
	b. Instrumentation	32,000			
	c. Electrical	37,000			
	d. Excavation	12,000			
	e. Insulation	10,000			
	f. Equipment Rental	5,000			
			TOTAL	$138,000	
3.	Direct Field Labor			$ 30,000	
			TOTAL DIRECT COSTS		$481,000
4.	Indirect Costs				
	a. Freight	$ 2,000			
	b. Insurance	2,000			
	c. Sales tax	4,000			
	d. Construction O. H.	11,000			
	e. Engineering	30,000			
			TOTAL	$ 49,000	
5.	Contractor's Fee	$ 17,000			
			TOTAL PLANT COSTS		$547,000
6.	Interest during construction if capitalized	-0-			
7.	Start-up costs, if capitalized	-0-			
			TOTAL DEPRECIABLE INVESTMENT		$547,000
8.	Land	-0-			
9.	Work Capital	-0-			
			TOTAL CAPITAL INVESTMENT		$547,000

TABLE 10. CAPITAL COSTS FOR SUBSEQUENT HYPERFILTRATION APPLICATIONS BASED ON THIS DEMONSTRATION

1. Purchased Equipment Costs for Major Plant Item				
a. Hyperfiltration Unit[1]	$300,000			
b. Control Panel	12,000			
c. Monitoring Panel[2]	-0-			
d. Heat Exchanger	10,000			
e. Other Items[3] (pumps, tanks, etc.)	14,000			
		TOTAL	$336,000	
2. Direct Field Materials				
a. Piping[3,4]	28,000			
b. Instrumentation[5]	10,000			
c. Electrical[6]	25,000			
d. Excavation[7]	-0-			
e. Insulation	10,000			
f. Equipmental Rental	5,000			
		TOTAL	$ 78,000	
3. Direct Field Labor			$ 30,000	
		TOTAL DIRECT COSTS		$444,000
4. Indirect Costs				
a. Freight[8]	1,500			
b. Insurance[8]	1,500			
c. Sales tax[8]	3,000			
d. Construction O. H.[9]	7,000			
e. Engineering[10]	15,000			
		TOTAL	$ 28,000	
5. Contractor's Fee[11]			$ 12,000	
		TOTAL PLANT COSTS		$484,000
6. Interest during construction, if capitalized	-0-			
7. Start-up Costs, if capitalized	-0-			
		TOTAL DEPRECIABLE INVESTMENT		$484,000
8. Land	-0-			
9. Working Capital	-0-			
		TOTAL CAPITAL INVESTMENT		$484,000

The revised total capital cost, Table 10, is $484,000. The cost for auxiliary equipment (other than the hyperfiltration unit) and installation is $184,000 or 60% of the cost of the hyperfiltration unit. This percentage is consistent with CPI estimates for these costs. Table 10 will be used as the capital cost basis for the remainder of this report.

Annual Operating Expenses and Savings

The demonstration phase of the project is presently in progress. While capital costs are firmly established, data on annual operating expenses and savings are presently being accumulated with preliminary results available. In particular, reuse of chemicals will be further developed during the extension of this project to September 1983. The operating costs for maintenance and membrane replacement will also continue to be documented during this extension project period. At this writing the hyperfiltration system operates automatically on a full time basis. Daytime operation is frequently interrupted to carry out studies on membrane cleaning and other aspects of operation aimed at optimizing the recovery. Studies are also carried out on a longer term at other than standard operating conditions; this adversely affects the impact of recycle. Concentrate reuse techniques have been developed in the laboratory. Full scale techniques are presently being developed. Concentrate reuse trials have been successfully run but none in sufficient quantities to significantly affect savings.

The demonstration period has been extended for 18 months during which additional data on savings and operating expenses will be collected. Annual expense and savings data presented here incorporate all data available to date. All data to date are extrapolated from weekly or monthly figures to obtain an annual figure.

Operating Costs--

Table 11 summarizes the annual operating expenses for the demonstration. The cost categories set forth by the Environmental Protection Agency Standard Procedures for Cost Analysis are used.

There are no raw materials required for this operation. Direct labor has been provided under the demonstration grant through one part-time technician at the site. One-half of his time was devoted to unit operation and data collection. Additional recovery unit operation was done by the dye range operators via the recovery unit control panel which is adjacent to their work station. This easily became a part of the existing operator's routine and no additional manpower was required for the recovery system operation. Maintenance material costs are taken from a maintenance log book kept on-site. The majority of the maintenance labor was provided by the project on-site technician. One quarter of his time was devoted to maintenance. Sixteen percent of the direct labor cost are labor additives. Electricity

TABLE 11. ANNUAL OPERATING EXPENSE FOR DEMONSTRATION

1. Raw Materials	-0-	
2. Processing Expenses		
a. Operating		
b. Direct Labor	$ 8,000	
c. Direct Supervision	-0-	
d. Maintenance Labor	4,000	
e. Maintenance Material	2,000	
f. Operating Supplies	3,100	
g. Labor Additives at 16% (of direct labor costs)	1,900	
h. Electricity	6,000	
i. Membrane Maintenance	20,000	$ 45,000
3. Plant Overhead		
a. Control Laboratory	-0-	
b. Engineering	-0-	
c. Plant Overhead at 88% of labor	$ 8,800	
d. Other	-0-	$ 8,800
4. Fixed Charges		
a. Insurance	$ 2,500	
b. Property Tax	2,500	
c. Royalty	-0-	5,000
NET ANNUAL OPERATING EXPENSES		$ 58,800
5. Annual Depreciation[1]	$ 48,400	48,400
TOTAL ANNUAL OPERATING EXPENSES		$107,200

---

[1] Straight Line 10 years

required to run the hyperfiltration system is metered. The primary user of electricity is the high pressure pump for the hyperfiltration system. Additional electrical power is used for small centrifugal pumps in the pipe and tank system and in the power control panels. Electricity consumption costs are based on the week ending 12/21/81 during which 4200 kilowatts of power were used. At 50 weeks/year and $0.028 per KWH, the annual cost is $5,880. At La France, concentrate not used is routed to the existing waste treatment pond and no additional costs are incurred. Plant overhead is taken as 17% of direct costs. Insurance, property tax, and overhead costs figures are based on capital costs and reflect the current percentages.

Concentrate reuse techniques developed and tested to date for concentrate reuse indicate that reformulating the concentrate for subsequent dyeings involves no more labor than the standard color matching technique now employed for new and standard dyeing. Therefore, no additional laboratory expenses are included.

A concentrate disposal cost item may be required to acquire zero discharge since 100% reuse will probably not be reached. Based on estimates received in interviews with representatives of a national company engaged in sludge disposal by land application (7), disposal of La France concentrate requires short hauling distances (10-20 miles) because La France Industries is located in an agriculturally intensive area. A cost of $15,000/yr. is based on disposal of 4.4 cubic meters of concentrate at $3.75 per cubic meter. This assumes that approximately 14% of the concentrate is reused and 86% is disposed. Significant reuse of concentrate would lower the concentrate disposal costs.

Annual depreciation is calculated using a ten year, straight line method.

Savings--

Table 12 presents a summary of annual savings. Potential savings occur in three main areas: savings of dyes and chemicals occur through concentrate reuse; water and waste treatment costs are reduced when process water is returned to the dye range; the energy is saved when hot water from the dye range is recycled at full process temperature thus avoiding the need to heat plant water. Boiler feed chemicals are saved with energy savings because make-up water to the boiler is reduced. Plant overhead savings are calculated as seventeen percent of the materials savings and reflect reductions in indirect costs (purchasing, inventory control, etc.) associated with these direct savings.

Savings possible through reuse of dyes and chemicals is $130,000 a year. At present, the concentrate reuse program is just getting under way. Full scale trials have been run with three shades which make up about 20% of production (based on an analysis of production between October and December of 1981). Actual savings are calculated by comparing dye and auxiliary chemical

TABLE 12. CURRENT ANNUAL PROCESSING SAVINGS DATA FOR DEMONSTRATION

1.	Processing Savings		
	a. Direct Labor	-0-	
	b. Direct Supervision	-0-	
	c. Maintenance Labor	-0-	
	d. Maintenance Material	-0-	
	e. Dyes and Chemicals	$ 24,000	
	f. Labor Additives	-0-	
	g. Steam	45,400	
	h. Water	3,400	
	i. Effluent Treatment & Disposal	3,500	
	j. Boiler Chemicals	1,000	$ 77,300
2.	Plant Overhead		
	a. Control Laboratory	-0-	
	b. Engineering	-0-	
	c. Plant Overhead	$ 13,000	
	d. Other	-0-	$ 90,300
3.	Fixed Charges		
	a. Insurance	-0-	
	b. Property Tax	-0-	
	c. Royalty	-0-	$ 90,300
	TOTAL ANNUAL PROCESSING SAVINGS		$ 90,300

formula sheets, with the derived formula sheets using HF concentrate. Chemical costs are based on actual invoice cost for 1981. The per meter of fabric savings is then applied to the projected production of the three shades in the year of 1982. Trials to date have shown that approximately 30% of the concentrate derived from these dye lots will not be able to be reused because of scheduling and other production difficulties. When this is taken into account the savings from these three shades is $24,000 per year. Energy savings are taken from the data from instruments provided to monitor flows and temperatures throughout the recovery systefor experimental reasons. Table 13 is the computer output showing energy savings for the week ending December 21, 1981. This week represents a typical weekly energy saving at this time. Using the 1981 average energy cost for La France of $7.15 per million BTUs (as steam), and projecting 50 weeks per year operation, the annual savings in energy is 6300 million BTUs at a savings of $45,000 year. The potential energy savings from the dye range, based on range flow rates and average plant water temperature, is approximately 18,000 million BTUs per year. As the demonstration continues the energy savings will more nearly approach the maximum of $125,000 per year. Because less steam is used, boiler feed chemical savings of $3,000 per year can also be approached.

Table 13 also presents the total water savings. The operating savings for water from the on-site filter plant is $0.44 per 1,000 gallons. The indicated water recycle results in a savings of $3,400 per year. The operating savings for the reduced flow to the water treatment is $0.45 per 1,000 gallon which results in an annual savings of $3,500. As the amount of use increases the two savings will approach $18,000/yr.

It is assumed that all fixed charges are unaffected by the savings. There is no change in annual depreciation. Therefore, the total annual savings for demonstration, at present, is $90,000. This represents only 30% of the total projected savings possible through recovery. As the demonstration progresses, and concentrate reuse is more fully implemented, the savings will more nearly approach the potential of $288,000/yr.

Data Quality

The costs and savings data used in this report represent the present situation as closely as possible. Since actual invoices are used, capital cost information is correct. Because expenses were incurred in this demonstration project that would not normally exist in subsequent applications, a more representative capital cost is established in Table 10. Annual operating expenses were collected from plant records and operator logs. Operating techniques and procedures are still being developed so these expenses are subject to revision as more data is compiled during the extension of this project. Indirect costs are based on current plant procedures.

Savings are estimated from operating logs and data from strip charts. As operating techniques develop further, equipment on-stream time will increase and savings will accrue at a faster rate.

TABLE 13. HYPERFILTRATION/DYE RANGE WASHERS

ENERGY AND WATER USE SUMMARY FOR THE WEEK ENDING MIDNIGHT 12/21/81

ENERGY AUDIT FOR WASHERS	
BOILER STEAM SUPPLIED TO WASHER	135 MILLION BTU 14274 MEGA JOULES
BOILER STEAM SUPPLIED TO HEAT EXCHANGER	37 MILLION BTU 3897 MEGA JOULES
ENERGY RECYCLED BY HF SYSTEM	127 MILLION BTU 13482 MEGA JOULES
ENERGY SUPPLIED	299 MILLION BTU 316530 MEGA JOULES
WATER AUDIT FOR WASHERS	
TAP WATER SUPPLIED TO WASHERS	278208 GALLONS 1054 CUBIC METERS
WATER RECYCLED BY HF SYSTEM	154032 GALLONS 583 CUBIC METERS
GROSS WATER SUPPLIED TO WASHERS	432240 GALLONS 1637 CUBIC METERS

FACTORS WHICH INFLUENCE COSTS AND SAVINGS

Economics of recovery depend on specific situations which vary widely from plant to plant. Table 14 summarizes the major factors which influence hyperfiltration costs and savings.

Hyperfiltration membrane costs vary inversely with membrane flux (volume productivity per unit area). Since flux values are specific for each process stream and depend on the characteristics of the process water being filtered individual tests performed for each application are required to determine membrane flux. In this demonstration, membrane flux is largely controlled by the concentration of a thickening agent used in the dye pad formula and the membrane fouling.

While membrane system costs are largely determined by membrae flux, a significant portion of the capital cost is represented by auxiliary equipment. The auxiliary system and installation represented 40% of the total capital cost of the demonstration. Each recovery application will have its own specific auxiliary system requirements and the complexity can very widely from plant to plant. The dyeing operation is intermittant with significant down time between production lots. It was, therefore, desirable to incorporate large holding tanks and a complex control system to allow the hyperfiltration unit to operate in a steady fashion. In a more continuous operation the auxiliary system complexity can be reduced according to the economics and consequences of down time.

In areas where water is in short supply or plant discharges are restricted recycle may permit plant expansion. If discharge is limited because of hazardous materials, recycle of the hazardous material becomes particularly attractive.

Energy savings are proportional to the difference between the process and the supply water temperatures. While for hot process effluents, the savings due to energy recovery are significant, hyperfiltration is not a good alternative to heat exchangers for heat recovery alone. Hyperfiltration is an economically attractive technology when water, waste treatment, and materials recovery are combined with energy recovery. Particularly since the cost of hyperfiltration is reduced by higher fluxes achieved at higher temperatures, hyperfiltration is best suited for recycle of hot water. Figure 14 shows the influence of process temperature on energy savings for a system operating similarly to the demonstration unit. It is assumed that regenerative heat exchangers are used to allow the hyperfiltration unit to operate at 85°C. It is also assumed that energy added to the concentrate is lost. Savings depend on the plant water temperature since this influences the increment of heating that would have to be added without recovery. The plant water temperatures shown represent extremes of summer and winter operation. Potential savings vary by a factor of five between 40°C process temperature and 85°C process temperature.

TABLE 14. SUMMARY OF FACTORS AFFECTING COSTS AND SAVINGS

Costs

Capital Cost

Basis: $300,000 HF System, $184,000 installation and auxiliary system

Comments:

1. Hyperfiltration system cost varies inversely with membrane flux.
2. Complexity of auxiliary system affects cost.
3. In areas with limitations on water supply or discharges, recycle can enable plant expansion.
4. Scale factor* for the HF system is near 0.9.
5. Scale factor* for auxiliary system and installation is 0.6.

$$\text{*New cost} = \text{Old Cost} \times \left[\frac{\text{New capacity}}{\text{Old capacity}}\right]^{\text{Scale factor}}$$

Operating Cost

Basis: $58,000/yr. excluding depreciation.

Comments:

1. Concentrate disposal costs can vary from plant to plant.

Savings

Energy Savings

Basis: $125,000/yr. - 75,000 m^3 water/yr. 55^{0}C warmer than tap water.

Comments:

1. Hyperfiltration is not a good alternative to heat exchangers for heat recovery only.
2. Savings are proportional to the difference between process and tap water temperatures.

Water Savings

Basis: 8,700/yr. - 75,000 m^3 water/year at $0.116/$m^3$.

Comments:

1. No significant problem encountered in reuse of 95% of water.
2. Savings vary with local water costs.

Water Treatment Savings

Basis: $9,000/yr. - 75,000 m^3 water/yr. at $0.12/$m^3$.

Comments:

1. Only treatment plant operating costs are saved for existing direct dischargers. For indirect dischargers, monthly user charges may be reduced.
2. New plant, direct dischargers can save full BAT costs.
3. Existing direct dischargers save proportional equipment upgrade costs.
4. Indirect dischargers may or may not save direct treatment charges.
5. If the chemical reused is classified as hazardous, disposal costs are saved.

TABLE 14. (continued)

Dyes and Chemicals Savings
  Basis: $130,000/yr. - reuse of 2/3 of concentrate at $0.033/liter.
  Comments:
1. Has been done on limited production basis at La France.
2. Cannot be done at all plants. The frequency of process change may complicate scheduling. Chemical reaction may prohibit or complicate direct reuse (1,2).

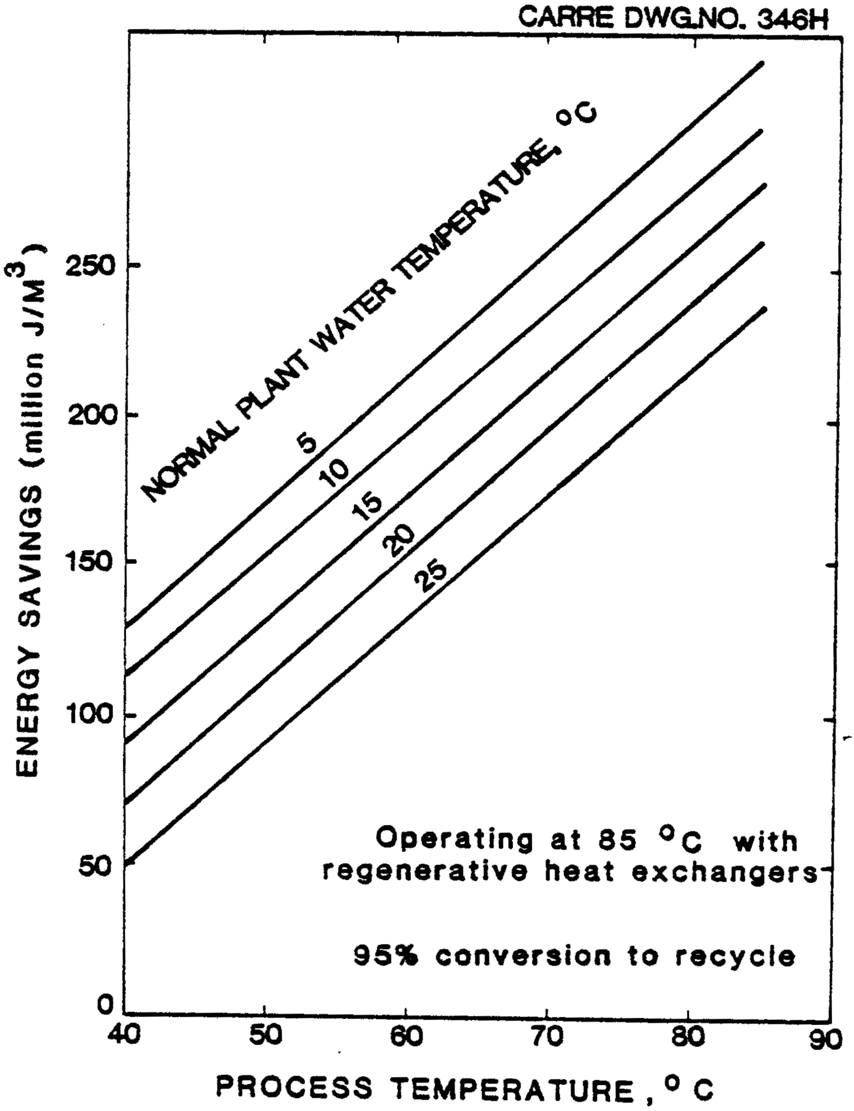


Figure 14. Energy Savings per Volume Wastewater Processed Versus Process Temperature for High Temperature Recovery of Hot Water.

Savings through reduced water and waste treatment can very widely depending on specific situations. At La France the costs of conventional water and waste treatment are small, therefore, savings are small. Because the demonstration is a retrofit of recovery to an existing direct discharger, only treatment plant operational savings (excluding labor) are to be counted. If treatment plant upgrading is necessary, then capital savings can be taken for the reduced upgrade cost. New plants who must construct new, best available technology (BAT), treatment facilities can credit the reduced treatment facility cost against the cost of the recovery system. Indirect dischargers may or may not save direct treatment cost. If fair-share costs are assessed against indirect dischargers for upgrade of municipal or regional treatment systems, then these charges are reduced by chemical recovery and water recycle.

Material recovery varies widely from process to process. To implement material recovery, a development program may have to be undertaken for new recovery applications. The economics of the development program depend on the value of the recovered material. While the potential value of the recovered chemicals at the demonstration site is about $0.005/liter, the value of other applications (for instance textile size recovery, or caustic recovery) may approach $0.025/liter.

Since the hyperfiltration membrane system is modular, capital cost varies with system size with a scale factor 0.9. (See Table 13 footnotes). The installation and auxiliary system costs can be scaled up using a scale factor of 0.6 to 0.7. Since savings are directly proportional to flow rates, economics improve with larger systems.

MEASURES OF MERIT

Internal rate of return, payout time, and return on original investment are calculated in this section using standard techniques (8). Example calculations are given in Appendix F. For these calculations an income tax of 46% is assumed. While more rapid depreciation techniques are available, a ten-year straight line depreciation method is used.

Figure 15 shows the variation for the three measures of merit with savings for the demonstration system. Potential annual savings total approximately $288,000 which would represent a payout time between 3 and 4 years, a return on original investment of 15-20%, and an internal rate of return on 20-30%. Presently the system is operating at approximately the break even point. (The internal rate of return approximately equals zero).

Figure 16 shows the effect of changes in flux on the overall measures of merit for the recovery system. The savings are assumed constant and the effects of capital cost changes on the measures of merit are given.

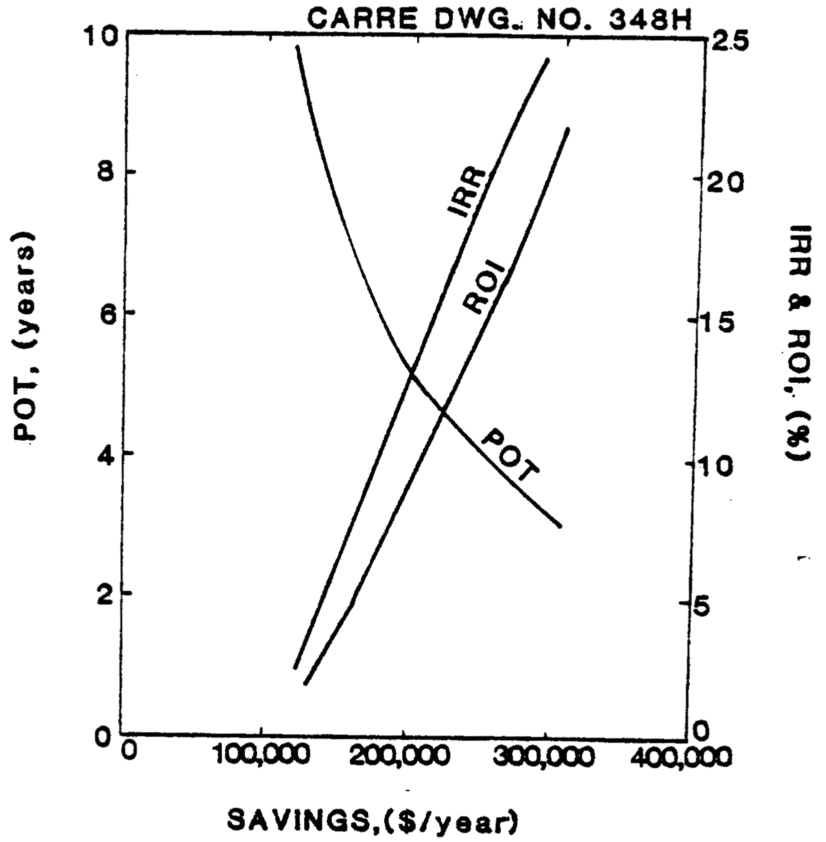


Figure 15. Payout Time (POT), Internal Rate of Return (IRR), and Return on Original Investment (ROI) Versus Savings for the Demonstration.

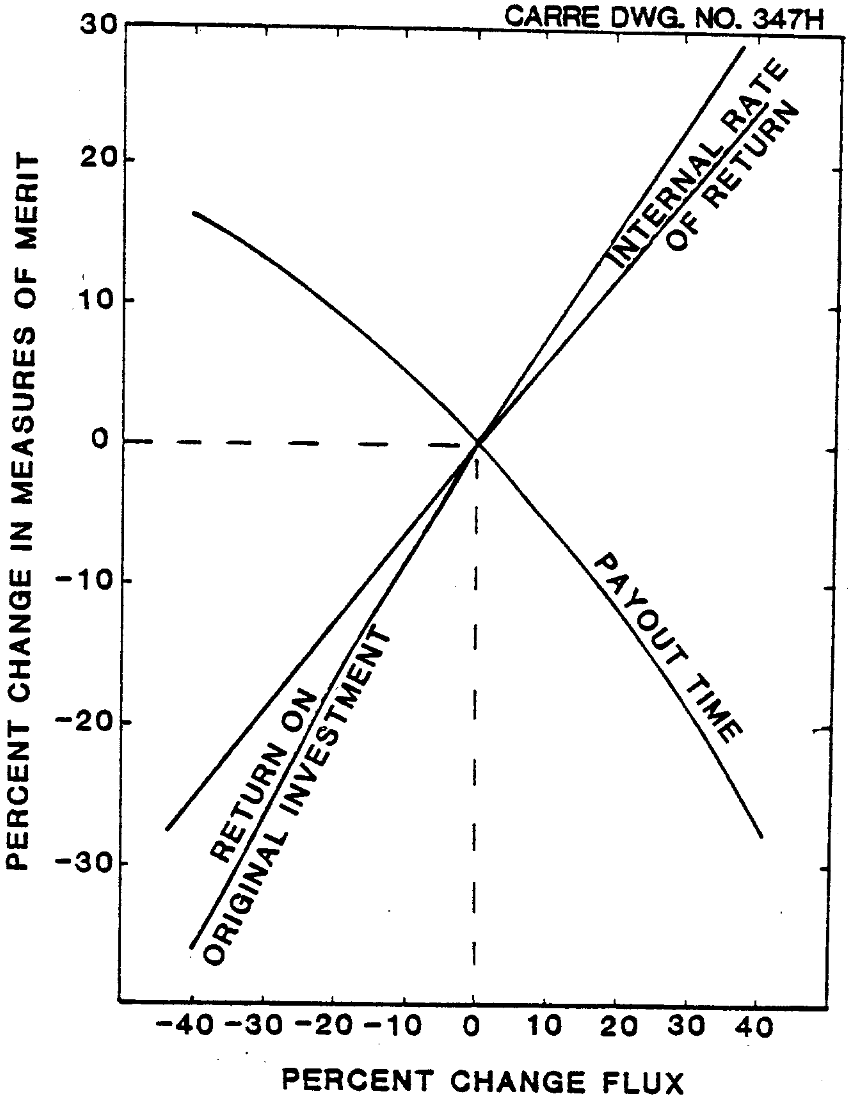


Figure 16. Effect on Flux on Measures of Merit for the La France Recovery System. Reference point represents: 3.7 year payout time; 22% Internal Rate of Return; 17% Return on Original Investment.

REFERENCES

1. C. A. Brandon and J. J. Porter. Hyperfiltration of Textile Finishing Plant Wastewater. EPA-600/2-76-060, U. S. Environmental Protection Agency, Research Triangle Park, North Carolina, 1976. 147 pp.

2. C. A. Brandon, J. J. Porter and D. K. Todd. Hyperfiltration for Renovation of Composite Waste Water at Eight Textile Finishing Plants. EPA-600/2-78-047, U. S. Environmental Protection Agency, Research Triangle Park, North Carolina, 1978. 237 pp.

3. J. L. Gaddis, C. A. Brandon and J. J. Porter. Energy Conservation Through Point Source Recycle with High Temperature Hyperfiltration, U. S. Environmental Protection Agency, EPA-600/7-79-131 (June 1979).

4. Brandon, C. A. and J. L. Gaddis. Full-scale Demonstration of Hyperfiltration for Closed-Cycle Textile Dyeing Facility. Desalination, 23: 19-28, 1977.

5. Brandon, C. A. Closed-Cycle Textile Dyeing: Full-scale Hyperfiltration Demonstration (Design). EPA-600/2-80-055, U. S. Environmental Protection Agency, Washington, D. C. 1980. 91 pp.

6. Federal Register, Vol. 45, No. 98, Monday, May 19, 1980, p. 33122.

7. TEXTILE INDUSTRIES (October 1979), Staff Report article entitled "Use dye waste sludge to grow corn."

8. Uhl, Vincent W. A Standard Procedure for Cost Analysis of Pollution Control Operations, Vol 1 and 2, EPA-600/8-79-018a and b, U. S. Environmental Protection Agency, Research Triangle Park, N. C., 62 pp. and 150 pp.

9. A. S. Michaels. New Separation Technique for CPI. Chemical Engineering Progress. 64:31, 1968.

10. W. F. Blatt, A. Dravid, A. S. Michaels, and L. Nelson. Solute Polarization and Cake Formation in Membrane Ultrafiltration: Causes, Consequences, and Control Techniques. Membrane Science and Technology. J. E. Flinn, Editor. New York: Plenum Press, 1970, p. 47.

APPENDIX A

LIST OF CONTROLS AND INSTRUMENTATION

The automatic controls included in the recovery system assure that there will be no interruption of range operation due to the HF system.

1. The waste water tank is fitted with a low level cut-off switch (on/off) which controls the HF unit.
2. The recycle water tank is fitted with a level control switch (on/off) which at low level causes the plant water supply to be used.
3. The recycle water head pump will shut off when the plant water supply solenoid valve opens.
4. Under command "Fill," the Flow Control Valve will open fully.
5. Under command "Rinse," flow rate (from rinse tank or plant water) will be controlled according to operator setting.
6. Under command "Drain," flow to the range will cease.

Meters

The following list of meters, by function, is required to monitor the performance of the HF system. Figure A-1 shows the location of meters in the recovery system. Codes "read at meter location" (L), "read at booth" (B) and "used primarily for reporting purposes" (*) are affixed.

1. Cumulative clock for rinse solenoid operation (B)*
2. Rinse make up solenoid (B)*
3. Cumulative clock for HF unit operation (B)*
4. Cumulative clock for range (B)*
5. Fluid level of wastewater tank (LT 115)(B)
6. Fluid level of recycle water tank (LT 102)(B)
7. Low level of recycle water tank alarm (B)

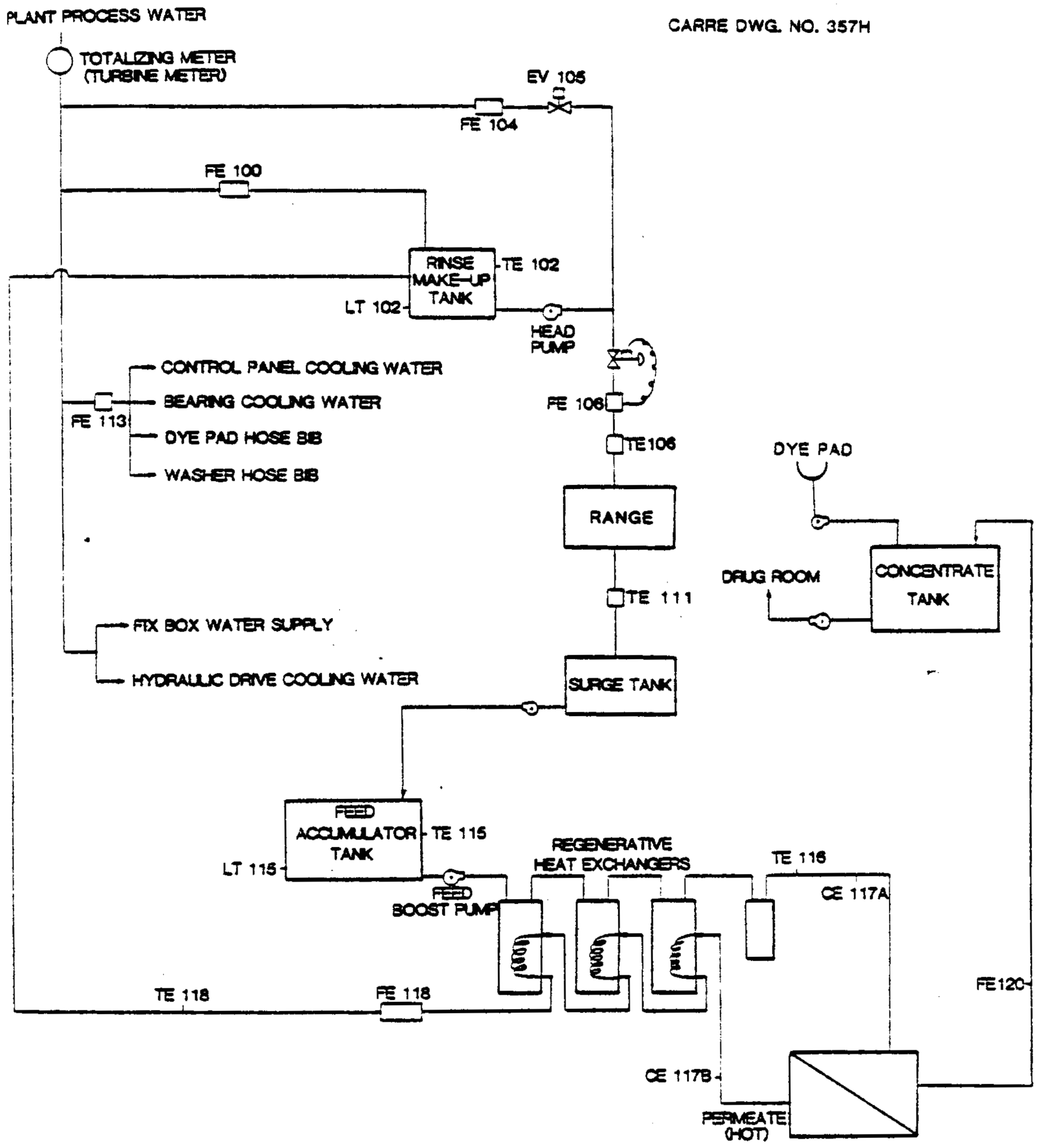


Figure A-1. Schematic of Recovery System Instrumentation.

8. Flow rate of permeate (FE 118)(L)
9. Flow rate of concentrate (FE 120)(L)
10. Flow rate to wash down hoses and bearing cooling water (FE 113)(B)
11. Flow rate of steam to wash boxes (B)*
12. Flow total of steam to wash boxes (B)*
13. Temperature of collected drains (TE 111)
14. Temperature of HF unit feed (TE 116)
15. Temperature of HF unit permeate (TE 118)
16. Temperature of waste water tank (TE 115)
17. Temperature of recycle water tank (TE 102)
18. Temperature of wash feed (TE 106)
19. Conductivity of permeate (CE 117B)(B)
20. Conductivity of HF feed (CE 117A)(B)
21. Comparison of 19 to 20, with alarm above set sum (B)

The HF system is equipped with glass tube and float rotometers for each of the 10 membrane modules. These also serve to enable an operator to estimate optical clarity of the fluid. Pressures within the system are indicated and concentrate sample ports are provided at 6 header locations in the single pass configuration. These meters provide detailed membrane performance data.

APPENDIX B

CHEMICAL BUILD UP TEST DATA AND RESULTS

The hyperfiltration system including tanks and range has been modeled to determine the response of the water conductivity in the recycle water tank. In the model, referring to Figure B-1, wash water and dye pad fluids are added to the range and mixed in the volume characteristic of the range. The mixed range fluid is added to the waste water tank and allowed to mix uniformly. Fluid from the waste water tank is processed in the HF system to provide a permeate with conductivity estimated to be 35 percent of the waste water conductivity. This permeate is mixed with the recycle water tank fluid and the mixed average is the prediction of the model.

Input data for water flow rates, tank levels, and fabric speeds were obtained from operating records. During the period analyzed, nine different dye pad formulations were introduced. Assuming a 140 yard delay from the operator start to the entrance of the fabric to the washer, the start/stop times of fabric to washers is recorded in Table B-1. One gram per liter solutions of each dye used were analyzed for conductivity and the expected dye pad conductivities were calculated and are shown in Table B-1. This assumes that other dye pad materials contribute negligibly to conductivity. Finally the fabric liquid uptake rates (pad flow) expected to have occurred are also shown in Table B-1. The conductivity is modeled and treated as a solute which can be passed, in solution, to other tanks and diluted or concentrated. Simple, lumped-parameter concentration models of the range (800 liter volume), the waste water tank (22,700 liter full volume), and the recycle water tank (22,700 liter full volume) are included.

The flow rates are observed values, so are not results of the model. However, the conductivities of feed and of the rinse tank are shown in Figure B-2. Clearly dye batches 1 and 2 produced low feed conductivity for the first two hours. Then the dye pad conductivity increased dramatically causing the conductivity level in the waste water and recycled water tanks to follow. Finally dye batches 8 and 9 were comparatively low in conductivity producing a slight decline near the end of the test period.

Measured values of conductivity are shown to agree well with the model prediction. Since the wash flow conductivity is modeled as constant, the recycle water tank conductivity only reflects passage of salts from the wastewater and, in turn, from the dye pad. Build up from recirculation effects are not necessarily zero, but evidently are small in the period of time studied.

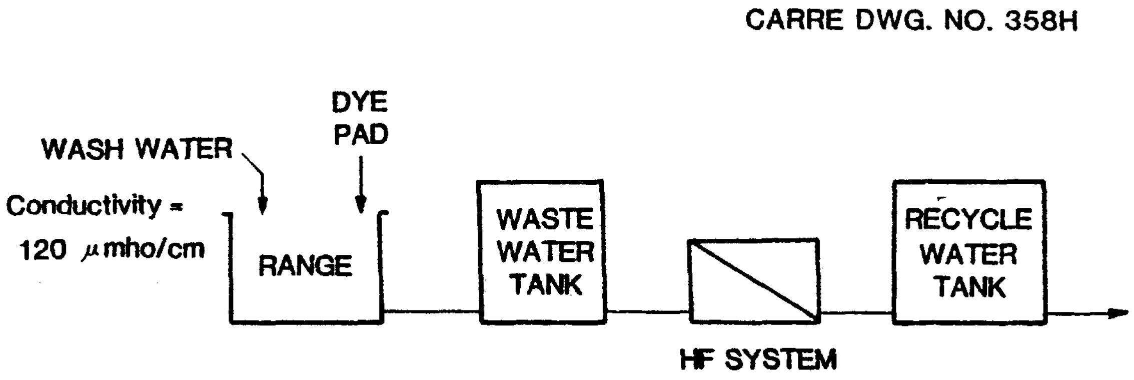


Figure B-1. Simplified System Schematic Assumed for Calculation of Material Build-up During Total Closed Cycle Operation of the Dye Range.

TABLE B-1.

INPUT DATA FOR MATHEMATICAL MODEL OF CLOSED CYCLE OPERATION OF DYE RANGE

Dye Batch	Time Start	Time Finish	Pad Flow (ℓ/min)	(Dyes Only) Pad Conductivity
1	-	3:16	32.4	657
2	3:27	3:43	12.3	4357
3	3:48	4:29	4.88	15722
4	4:42	5:29	5.19	18466
5	5:39	6:37	9.63	23840
6	6:47	7:48	6.35	10856
7	8:01	9:40	10.69	36084
8	10:04	10:32	12.69	4266
9	10:40	11:20	7.0	6000 est.

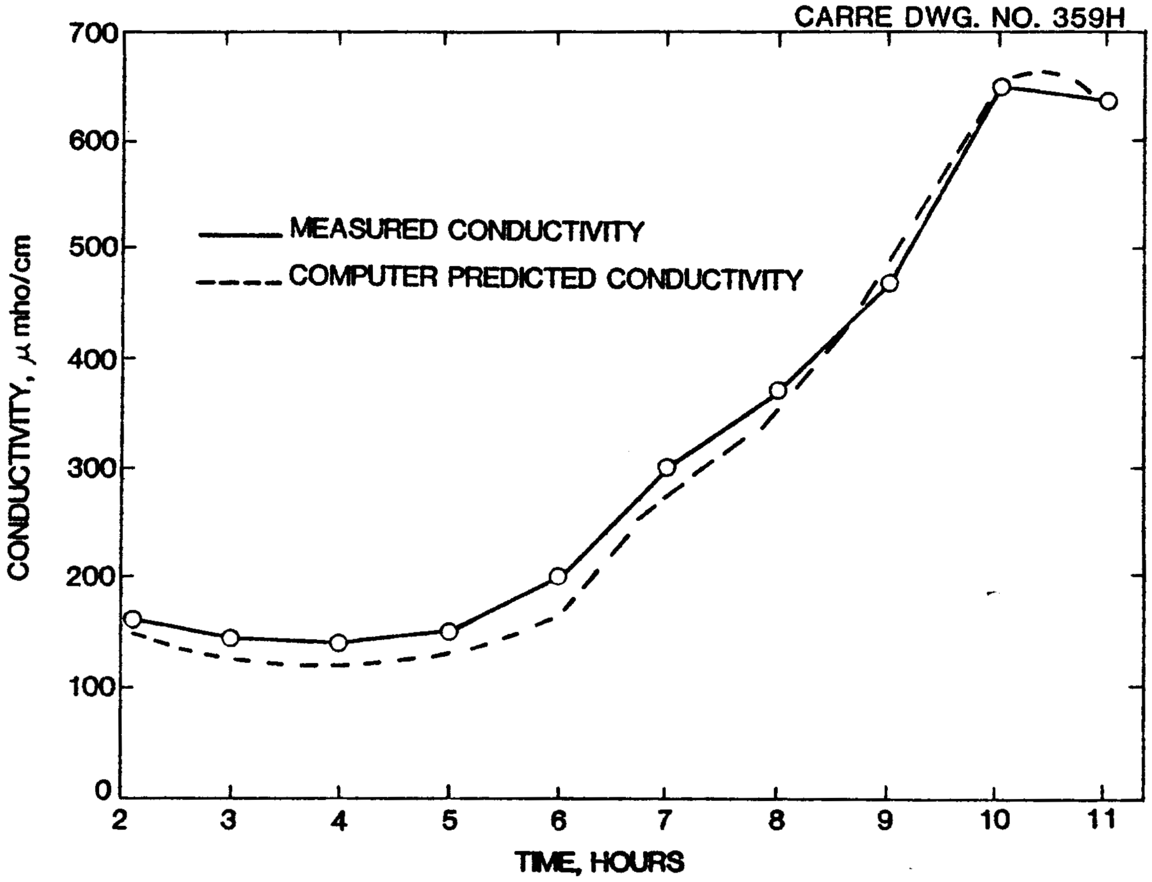


Figure B-2. Conductivity of Permeate During Test of Total Closed Cycle Operation of the Dye Range.

APPENDIX C

MEMBRANE SYSTEM FLUX MODEL

The flux of the hyperfiltration system is currently being analyzed by a mathematical model of flux behavior. The model estimates the effect of the natural polymer, guar gum, which is used as a dye pad thickener in quantities sufficient to reduce the membrane flux. The model follows the theory (9,10) that slow diffusing molecules rejected at the membrane cause an accumulation which retards the permeate flow. This accumulation forms a "gel layer" and has predictable characteristics. (The particular expression of the theory along with the details of its predictive capability will be published separately). The use of the theory in this project is described in this appendix.

Figure C-1 shows a theoretical curve of non-dimensional flux J/¶ P* versus non-dimensional pressure P/P*.

$$\frac{J}{\P P^*} = \frac{1}{2}\left(3 - \frac{P^*}{P}\right)^2, \quad \frac{P}{P^*} > 1$$

Where:
J is flux.
¶ is water permeability
P is pressure
P* is a theoretical variable with units of pressure.

Shown on the curve is a group of data taken with guar gum at several pH levels and one concentration. Data on other large molecules correlate as well with the same curve. This part of the theory is well supported.

The theory predicts the dependence of the variable P* on wall shear stress and concentration. The predicted form depends on the diffusion coefficient and viscosity of the fluid in addition to the shear stress and concentration. The form predicted by theory for P* is:

$$P^* = B\ (Tw/C)^{1/3}/\mu\P$$

Where:
B is a constant
Tw is the wall friction
μ is viscosity
¶ is water permeability
c is gum concentration.

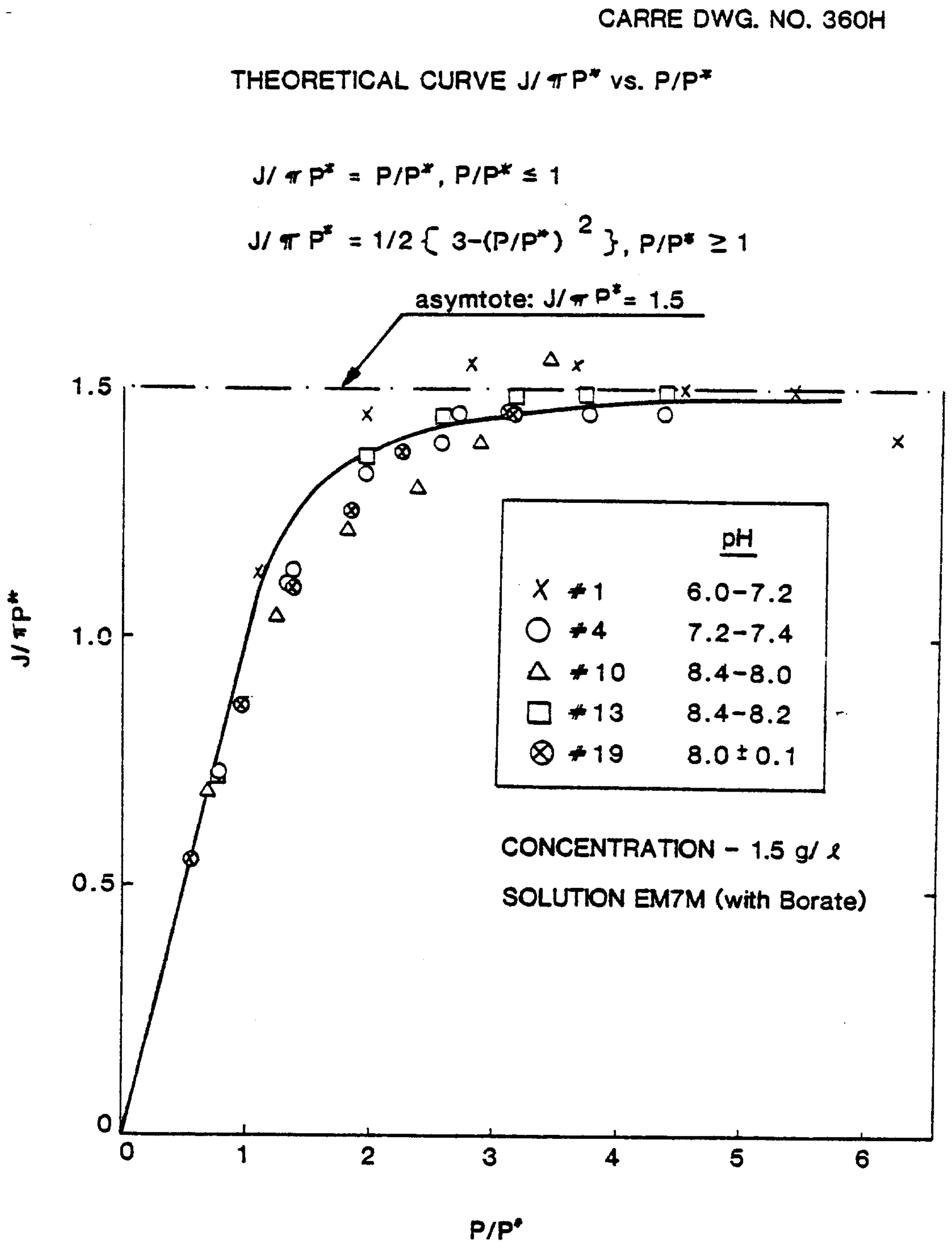


Figure C-1. Correlation of Flux and Pressure for Gum Solutions Shown with a Theoretical Prediction.

Certain assumptions have been evoked which may be valid only for the present case. The constant B in the above contains information on the diffusion coefficient while the viscosity influence accounts for the effect of concentration on both viscosity and diffusion. Considerable effort has been made to determine a satisfactory value for B based on the Single Pass data and on laboratory scale recirculation testing. Laboratory tests are known to degrade the polymer by repeated recirculation and result in a value of "B" of 400 (in the units of the computer program). The Single Pass system viscosity is higher (non degrading) so a projected value of 235 was felt to be reasonable. However, the system data prediction is really superior with B = 400 and even higher in a few cases.

The value of input concentration has a substantial effect on P* largely through the viscosity which increases exponentially with concentration. The input concentration ranges from 0.2 or 0.3 weight percent so that variations of 150 percent in P* is expected. This 150 percent variation in P* corresponds to inlet pressure variations of up to 6 MPa. For this reason results are difficult to obtain with precision.

The module configuration is input to the computer program. The inlet pressure, flow, and concentration are also inputs together with the controlled outlet flow rate and assumed permeability. The program balances the flow between parallel paths, computes permeate flow and pressure losses and computes the concentrations for each parallel flow section. When the flow or pressure becomes zero or when the end of the system is reached, the program will adjust inlet conditions to provide the measured outlet flow.

One alternative method is to successfully guess the permeability required to satisfy a set of inlet conditions. This method may be used but is sensitive to changes in membrane operating conditions or to slight performance variations in time.

The model as described assumes that a single value of permeability can characterize the entire system. This assumption is only an approximation as is indicated by observation of the identical specified system segments (Modules 1, 2, 3, 4; Modules 5, 6, 7 and Modules 8, 9 and 10). The only independent analyses that can be made of the various system segments is for the downstream segments (Modules 8, 9, and 10) and the remaining modules as a group.

APPENDIX D

HYPERFILTRATION OF TEXTILE PROCESS WATER FOR REUSE

Summary of Previous Studies

Three previous studies provide experience leading to the work reported in this appendix. The first study (1) involved the pilot-scale separation of composite wastewater from a beck dyeing process and a full-scale reuse of the hyperfiltration products and concentrates. Polyamide (hollow-fine fibers), cellulose acetate (spiral and tubular), and hydrous Zr(IV) oxide-polyacrylate membranes were used. Eighteen production dyeings involving a total of 1348 m of cloth were carried out in a two-piece dye beck.

The purified product water was a satisfactory substitute for normal tap water in all production dyeings for water recoveries ranging from 75 to 90%. Membranes used in the renovation of the wastewater had conductivity rejections of 65 to 95% and color rejections from 86 to > 99%.

It is also technically feasible to reuse all the concentrates. In 11 production dyeings over 700 m of cotton velour fabric was produced, graded as first quality, and sold commercially. In 10 tests standard shades were produced with an average dyestuff savings of 16%.

In the second study (2) composite wastewaters obtained from the several processes occurring in a dyeing and finishing plant were separated by hyperfiltration, and the cumulative product and concentrate of a 90% recovery run was tested for reuse as process water in laboratory dyeing. Precast and dynamically formed membranes were used in eight dyeing and finishing plants. The processes encountered were: dyeing of nylon using premetallized dyes, dyeing of acrylic fabric using basic dyes, and the scouring, desizing, and dyeing of cotton and polyester. In all cases the product water was acceptable for replacement of fresh water as determined in a laboratory dyeing using standard production evaluations. Analyses indicated higher COD and dissolved solids and lower concentrations of metals in the product water than in the fresh plant-process water. The concentrate from the premetallized dye process was suitable for dyeing very deep shades upon addition of appropriate dyes. Laboratory dyeing tests using concentrates from the other processes were unsuccessful. Perhaps this result is not surprising because the concentrates were obtained from a feed containing a composite of effluents from the plant processes and not from a single process.

The first two studies dealt with renovation of the composite wastewater from the dyeing and finishing plants. Because of the obvious advantages for reclamation and energy conservation, a third study (3) evaluated hyperfiltration for direct recycle of unit process effluents. The five major water and energy consuming preparation and dyeing processes were studied with high temperature hyperfiltration membranes.

The product water produced by the membranes was again found to be universally usable. In some cases the reuse of the concentrate from the individual process effluent streams was estimated to be practical.

In Phase I of the current study (5), three hyperfiltration membranes were evaluated in a pilot unit under test conditions consistent with the membrane manufacturer recommendations and the anticipated operating conditions at La France Industries. The technical feasibility for using hyperfiltration in closed-cycle operation of a dye range involves the performance of the membrane equipment and the reuse of the product (95% of the range effluent volume) and the concentrate (the remaining 5% of the effluent volume). The products were judged suitable for recycle as process water. The reuse and/or disposal of the concentrates from a multiple-process range requires detailed studies of each type of process and the compatibility of the concentrate with subsequent processes.

Recovery Concept

Discharge from the range occurs in two forms; the wash water discharged continuously during the dyeing run and the dye drop from the dye pad following each run. There is a time period between each run needed for cleaning and reloading the dye pad and/or rinsing out the washing train. This permits each dye run to be treated in the hyperfiltration system as a batch containing the components washed from a single dye run. The dye drop is of known composition and its reuse in a subsequent dye pad preparation or its disposal can be planned with that knowledge. The wash water effluent from the run constitutes the feed for the pilot unit and eventually the full-scale hyperfiltration unit. Most feeds are highly colored and the dyes must be removed to avoid possible staining during the reuse of the product as wash water. The auxiliary components must also be removed to provide wash water with the concentration differences suitable for effective removal of these components from the fabric during the washing step. The concentrate contains the dyes at concentrations much lower than those in the dye pad solution but concentrations of the auxiliary components are comparable. Reuse of the concentrates as a dye pad solution depends on the ability to add dyes and auxiliary components to develop a needed production shade that possesses the necessary shade, hue and crocking characterization. Generally this reconstitution must be accomplished by trial and error in the dye laboratory. Even beginning with pure materials each dye bath requires an adjustment based on the routine tests accomplished in the dye laboratory. Eventually, optimum reuse of concentrate may involve judicious scheduling of dye runs and the use of guidelines based on the properties of dye classes. Some sequences are expected to be favorable for reuse, e.g., those using the same dye class and those of increasingly darker shades in the same general hue. Other sequences are expected to be unfavorable for reuse resulting in disposal of the concentrate. Although some crossovers between dye classes are feasible it could be difficult, for example, to cross over from a basic dye to an acid dye. Crossovers between dye classes will generally require addition of auxiliary components particular to the dyes being added to the concentrate.

Product Reuse

Product samples were generated by hyperfiltration of range wash water effluents to 95% recovery using the pilot test with ZOPA membrane modules. The samples were evaluated in the laboratory for reuse as wash water by: (1) simulated rinsing using product water on greige fabric samples, (2) chemical analyses, and (3) foam tests.

The suitability of the product samples for reuse was determined primarily from the results of rinsing tests. This test consisted of stirring the four fabric samples (3" x 3") in 400 mls of product water for 10 minutes at 60°C. The fabric patterns used were: (F-1) 100% cotton, (F-2) 65% cotton, 35% rayon, (F-3) 41% cotton, 59% acrylic and (F-4) 68% cotton, 15% rayon, 17% polyester. The dried test fabric samples were evaluated for staining on an AATCC Chromatic Transference Scale against untreated fabric samples. The scale assigns 5.0 to practically unstained fabric and 1.0 to the fabric stained heaviest. A stain index (SI) will be used in the paper, calculated by subtracting AATCC scale reading from 5.0 to provide an index which increases from zero as the extent of staining increases.

Chemical analyses were conducted on the initial feed, final concentrate, and cumulative product obtained from a 95% recovery hyperfiltration. Analytical parameters included chemical oxygen demand (COD), total solids (TS), suspended solids (SS), volatile solids (VS), and pH. The absorbance at λ = 460 nm and the conductivity of the feed and the concentrate and product at the final recovery were also determined. Color rejections refer to rejections at 95% recovery and not the cumulative samples.

Selected samples were evaluated for foaming. A 50 ml sample was added to a 100 ml graduated cylinder, and the cylinder was stoppered and agitated vigorously. The foam volume five minutes after agitation was recorded and interpreted as a qualitative indication of the presence of foaming agent or surfactant. Generally, the higher the concentration of surfactant the greater the foam volume. However, the relationship is not linear and cannot be interpreted as a quantitative estimate of surfactant concentration.

Some results from these tests are tabulated in Table D-1. Only one sample exhibited a color rejection below the expected acceptable value of 0.97. However, if a stain index of 1.0 is arbitrarily set as an acceptable level six samples fail to meet this criterion; mostly very dark shades of basic/direct formulation. Generally, the color rejections of the basic/direct formulation rinse water are less than those of the direct formulation rinse water, and the stain indexes are greater when compared at the

TABLE D-1. REUSE SAMPLE CHARACTERISTICS

Product Sample Number	Type Dye Formulation	Absorbance, Feed	Absorbance, Product[1]	COD Units mg/ℓ	DS Units mg/ℓ	VS Units mg/ℓ	Stain Index[2]	Color Rejection[1]
3196	Bleach	0.06	0.00	354	920	467	0.0	1.00
3127	Bleach	0.20	0.00	596	1380	744	0.0	1.00
3175	Direct	0.03	0.00	71	104	50	0.0	1.00
3178	Direct	0.06	0.00	122	154	75	0.0	1.00
3157	Direct	0.23	0.00	141	270	114	0.0	1.00
3166	Direct	0.24	0.00	158	273	201	0.0	1.00
3139	Direct	0.79	0.02	157	373	169	0.0	0.99
3148	Direct	0.79	0.04	29	101	40	0.0	0.99
3205	Direct	0.92	0.08	88	500	105	0.4	0.99
3130	Direct	1.50	0.22	140	454	195	0.6	0.99
3172	Direct	4.00	0.22[3]	94	448	169	0.4	0.99[3]
3211	Direct	7.70	0.84	404	1340	357	2.7	0.99
3181	Acid/Direct	0.15	0.01	193	148	113	0.0	0.99
3163	Acid/Direct	1.90	0.22	167	116	65	0.0	0.98
3160	Basic/Direct	0.07	0.01	1030	291	123	1.1	0.99
3193	Basic/Direct	0.16	0.01	1020	307	207	0.0	1.00
3169	Basic/Direct	0.38	0.03	1430	363	220	1.2	0.99
3133	Basic/Direct	0.39	0.10	834	376	239	0.0	0.98
3154	Basic/Direct	0.74	0.08	730	249	148	0.6	0.99
3151	Basic/Direct	0.85	0.13	807	310	175	1.1	0.99
3184	Basic/Direct	1.10	0.34	981	547	264	1.6	0.96
3142	Basic/Direct	2.00	0.35	845	243	136	2.2	0.98
3145	Disperse	1.10	0.00	148	42	26	0.0	1.00
3136	Disperse	1.40	0.27	128	172	111	0.6	0.98
	(Plant Water)			9	57	18		

[1] At 95% recovery

[2] Average for the four fabrics.

[3] At 89.4% recovery.

same product absorbance. The stain index, of course, varies with the fabric composition. The direct formulation wastewater products stain fabrics high in cotton content more readily than the samples with a high acrylic content. The relative staining of these two classes of fabrics by the basic/direct formulation wastewater products depends on the weight ratio of the basic-to-direct dye content in the dye bath. (See Figures D-1 and D-2.)

Staining is not the only possible criterion for qualifying product water for reuse in washing. The concentration of the auxiliary components should also be low. The ratios (P/F) of COD, DS, and VS in the cumulative 95% recovery product to their values in the corresponding feed is given in Table D-2. The P/F ratios for COD, DS, and VS are <0.3 for direct formulations. It is greater for COD than for DS and VS for basic/direct and acid/direct where a low molecular weight (volatile) dye solvent is used in the formulation, which would contribute to COD but not to DS or VS. The ratio is $\leq$0.1 for the disperse dye formulations.

Concentrate Reuse

Laboratory tests are in progress to determine the reuse potential of the cumulative 95% recovery concentrates. The concentrate was provided as the starting solution to prepare a production dye bath, dye a test fabric on a pilot range, and compare the product with production standards for hue, shade, and crock. Three types of tests were involved:

1. Reformulation of the original dye bath.
2. Preparation of the dye bath of the same dye class but different hue and deeper shade.
3. Preparation of the dye bath in a different designated dye class with different hue and deeper shade.

The quantities of dyes and auxiliary components required to prepare the production dye bath are then compared to the standard production formulation and reported as reduced or excess quantities.

Some results are available for type-1 reformulation tests. A series of tests were run using direct, basic/direct, and disperse formulations. In the initial tests 100% of the quantities of the auxiliary components prescribed in the production pad were first added to the concentrate, a test dyeing was performed, additional dyes were added to prepare the production hue and shade, and the test fabric was compared to the production standard. The average dye reduction was 23% for direct, 10% for basic/direct, and 0% for disperse dye classes. Based on the production levels 67% direct, 24% basic/direct, and 1% disperse, the weighted dye reduction (savings) is approximately 19%. This procedure involves no savings in auxiliary components.

A second set of type-1 reformulation tests involved the preparation of the test dye formulation with 0, 25, 50, and 75% of the specified auxiliary

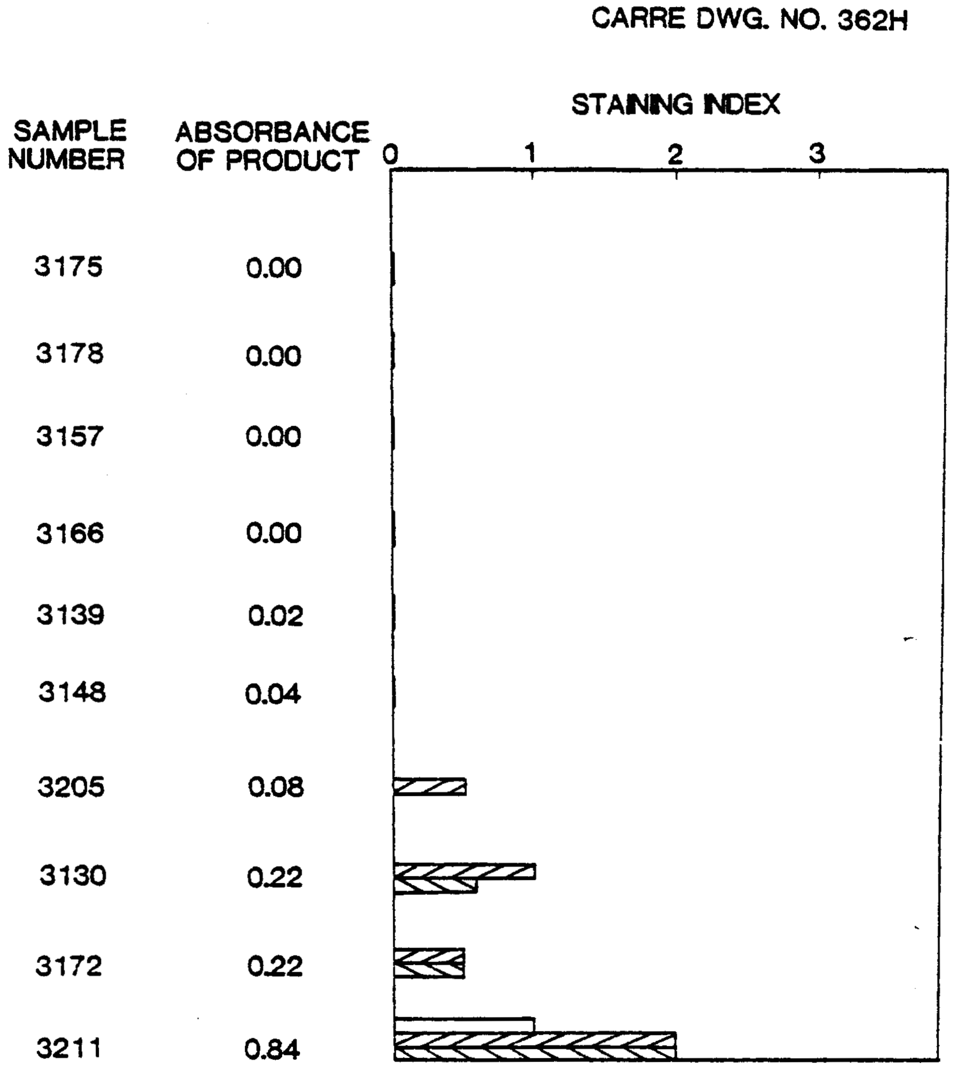


Figure D-1. Staining Index on Fabrics by Hyperfiltration Products from Direct Dye Range Effluents: ☐ 41% Cotton, 59% Acrylic; ▨ 100% Cotton and 65% Cotton, 35% Rayon; ▧ 68% Cotton, 15% Rayon, 17% Polyester.

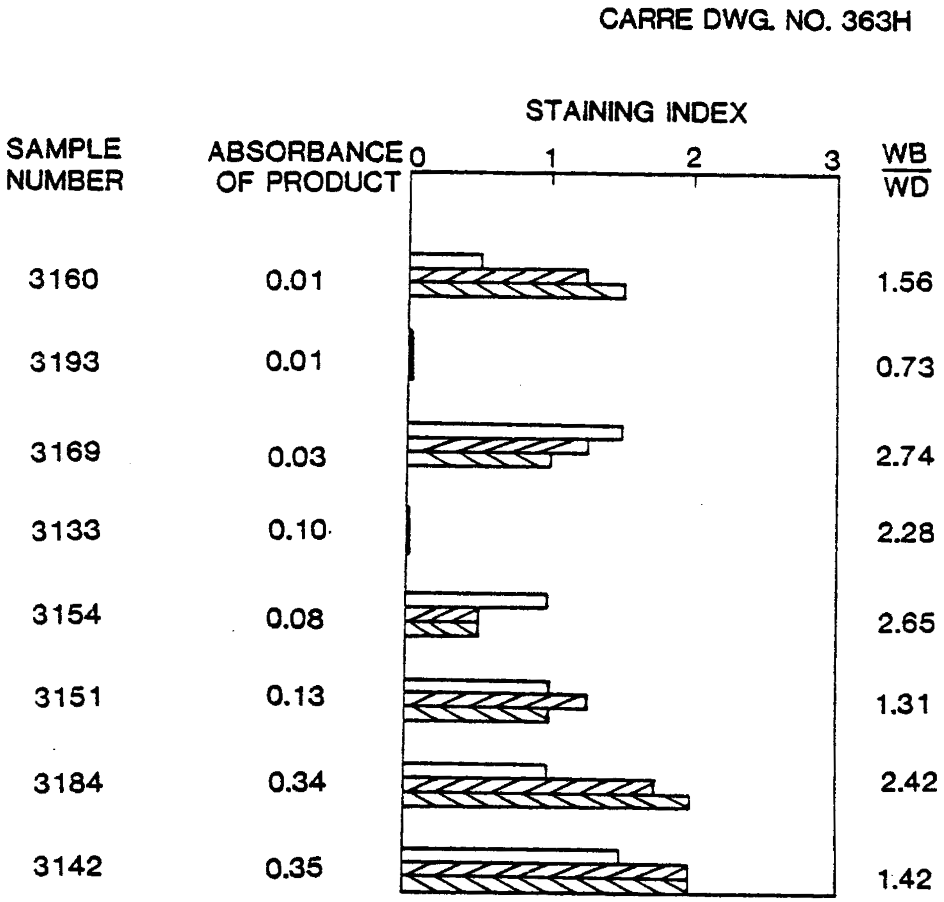


Figure D-2. Staining Index on Fabrics by Hyperfiltration Products from Basic/Direct Dye Range Effluents: □ 41% Cotton, 59% Acrylic; ▨ 100% Cotton and 65% Cotton, 35% Rayon; ▧ 68% Cotton, 15% Rayon, 17% Polyester.

TABLE D-2.

RATIOS OF CHEMICAL OXYGEN DEMAND, DISSOLVED SOLIDS, VOLATILE SOLIDS, AND FOAM VOLUME FOR THE CUMULATIVE PRODUCT TO FEED

Product Sample Number	Type Dye Formulation	$\overline{P}/F$			
		COD	DS	VS	Foam Volume
3175	Direct	.23	.33	.23	
3178	Direct	.26	.36	.26	
3157	Direct	.21	.27	.21	
3166	Direct	.20	.23	.32	
3139	Direct	.22	.31	.25	.71
3148	Direct	.15	.37	.17	.2
3199	Direct	.08	.38	.22	
3130	Direct	.15	.29	.28	.2
3172	Direct	.09	.34	.23	
3211	Direct	.40	.87	.50	
3181	Acid/Direct	.66	.42	.48	
3163	Acid/Direct	.46	.35	.29	
3160	Basic/Direct	.47	.24	.21	
3193	Basic/Direct	.52	.38	.33	
3169	Basic/Direct	.33	.18	.14	
3133	Basic/Direct	.33	.27	.22	.45
3154	Basic/Direct	.49	.25	.20	.3
3151	Basic/Direct	.49	.31	.25	.3
3184	Basic/Direct	.46	.29	.30	
3142	Basic/Direct	.33	.16	.13	.6
3145	Disperse	.07	.07	.04	.7
3136	Disperse	.09	.09	.10	.0

components. The results for a direct dye and a basic/direct dye mixture are provided in Table D-3. The dye requirement was reduced by 5 to 9% for the direct dye and increased by approximately 4% for the basic/direct dye. The production tests indicated a reduction of 50 to 75% of the auxiliary components gave satisfactory dyeings.

These preliminary tests indicate that a modest savings in dye and at least a 50% savings in auxiliary components may be possible. Extensive research at this pilot/laboratory scale and full-scale reuse is required to determine the practicality of concentrate reuse.

Bleaching Process

Fabric bleaching in preparation for dyeing is also carried out in the range. This process occurs for several continuous hours each week providing an opportunity for recycle for the same process. The bleach pad contains sodium hydroxide, a surfactant wetting agent and hydrogen peroxide. The wash water effluent contains diluted sodium hydroxide and wetting agent; the peroxide is barely detectable. Hyperfiltration to 95% recovery, after reducing the pH from approximately 11 to 9-10, reduced the COD and VS to about 30% of the feed, indicating good rejection of the wetting agent. The pH values of the product and concentrate remain high and the reduction of DS is about 40%. Although some sodium hydroxide is rejected, the product (the new rinse water) is now at a pH about two units higher than fresh plant water. Reuse of the concentrate was carried out using product water with only hydrogen peroxide additions of 0, 25, 50, 75, and 100% of the production formula. The addition of 100% of the production formula produced excellent bleaching. The 50 and 75% additions were also satisfactory.

Summary

This pilot study of renovation of wastewater from a production dye range by high temperature hyperfiltration using dynamic zirconium oxide-poly(acrylic acid) membranes indicates the potential for closed cycle operation by reuse of water and chemicals. The membrane stability, flux and rejection are satisfactory. Simple laboratory reuse tests indicate only a small percentage of the hyperfiltration products may be unsuitable for general reuse as wash water. Judicious planning of the sequence of dyeing processes should permit reconstitution of many hyperfiltration concentrates for reuse in the dye pad. Significant savings (50 to 70%) of auxiliary components and minor savings of dyes in the concentrate reuse are indicated by the laboratory tests. Reuse of the hyperfiltration products and concentrate obtained with bleach wastewater appear suitable for reuse with savings of wetting agent in the pad formulation.

TABLE D-3. TYPE-1 REFORMULATION RESULTS

Dye Class	Weight of Dyes in Production Formula (g/ℓ)	Weight of Dyes in Test Formulas; Designated % of Auxiliary Components 0%	25%	50%	75%
Direct	16.35	15.7	15.5	15.2	14.9
Basic/ Direct	0.82	0.87	0.85	0.83	0.84
Direct	5.26	5.72	4.58	4.35	4.58

APPENDIX E

WASH WATER REUSE QUALITY

The data presented in this appendix describes the water quality required for washing fabric. The data provide the empirical base obtained from observing the standard procedure of the production operators. Following each production dye lot the range operator judges the suitability of the wash water remaining in the boxes for rinsing the next dye lot. When judged unsuitable the operator drains the washers and refills with clean water before dyeing the next lot.

Even if draining is not required, the water remaining in the washers is diluted by approximately 30% as the cloth leader passes through the dye range.

The required quality of the wash water in the wash boxes depends on the relative shades of the consecutively dyed lots. Dyeing of a light shade followed by a darker shade would reduce the need to drain wash boxes between dye lots. Also, since eight pieces (400 meters) are required to establish equilibrium concentrations in the wash boxes, one piece lots may generate low wash box concentrations, even though the dye formula concentrations are high. An attempt has been made to quantify the operator's perceived concentrations in terms of the dye concentration in the lot just dyed (D_i), the number of pieces in that lot (P) and the dye concentration in the formula for the next lot (D_{i+1}). Since there is no further increase in wash water concentration after 8 pieces of fabric are washed, in this analysis concentration in the just-dyed lot is weighted by the multiplier, P/8 (limited to a maximum value of 1), to yield a weighted concentration, D_i. Several consecutive lots were analyzed to establish the criterion used by the dye range operators.

Figure E-1 is a graph of a weighted dye concentration, D_i versus the dye concentration, D_{i+1}. Using this empirically information it is evident that the operator normally drains and refills the washers when D_i is greater than 15 g/L or when lot (i + 1) is a significantly lighter shade than lot (i). The only documented instance of staining with permeate occurred when D_i/D_{i+1} was greater than 100.

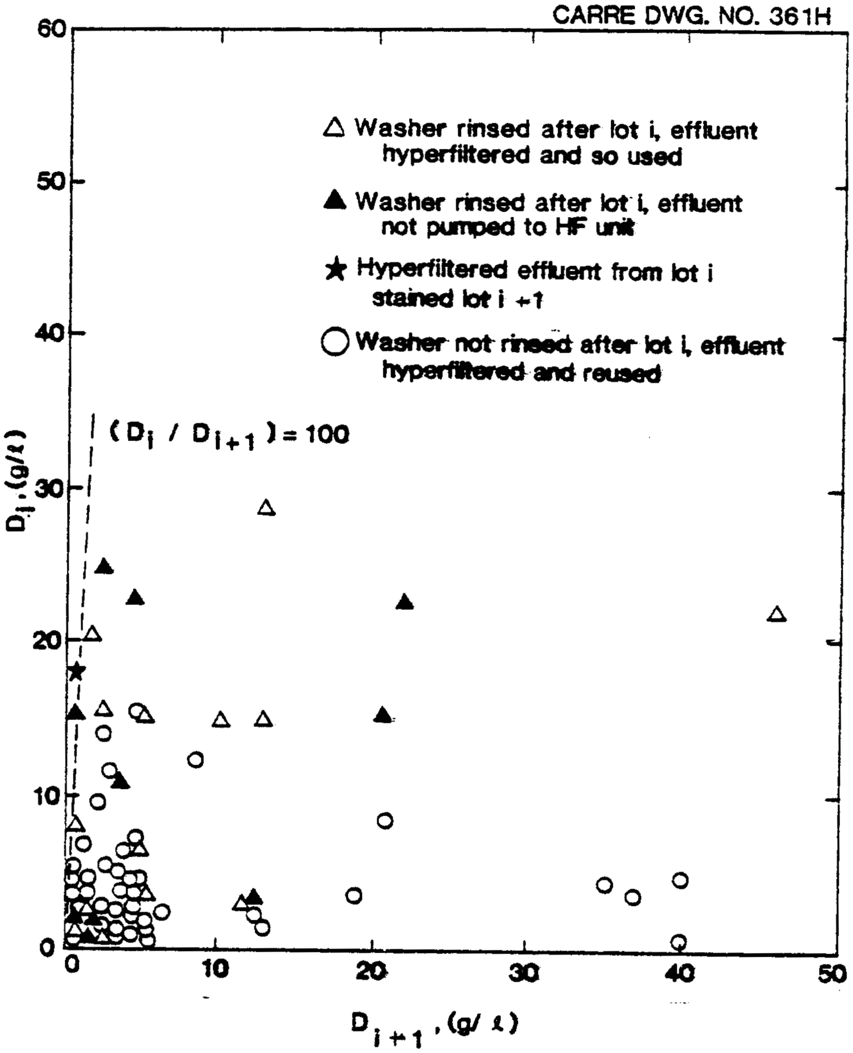


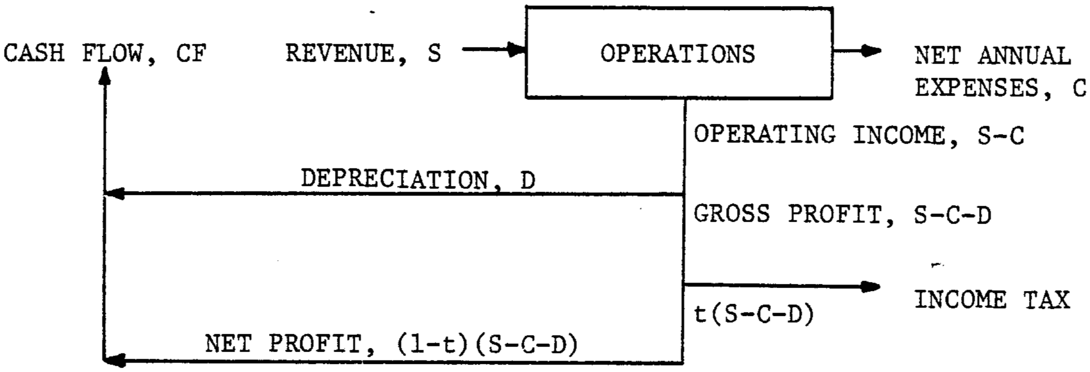
Figure E-1. Weighted Dye Concentration in the Dye Bath for Lot i versus Dye Concentration in the Dye Bath for the Following Lot, i + 1.

APPENDIX F

SAMPLE CALCULATION OF ECONOMIC MEASURES OF MERIT

The methods shown here conform to those recommended by "A Standard Procedure for Cost Analysis of Pollution Control Operations," (8).

The following diagram represents the money flow for a general operation:



In the following example case for La France Industries a differential money flow is generated by subtracting money flows for the operation with and without hyperfiltration recovery. The following are hyperfiltration costs savings and assumptions:

Capital Cost:	$484,000
Annual Savings:	$288,000
Annual Operating Costs (exclusive of depreciation)	$58,800
Depreciation period (straight line):	10 years
Equipment life	10 years
Tax rate	46%

The resultant money flows are as follows:

Effect on operating income	$229,200/year
Effect on gross profit	$180,800/year
Effect on net profit	$ 97,632/year
Effect on cash flow	$146,032/year

Based on these money flows measures of merit can be calculated as follows:

$$\text{Return on Original Investment (ROI)} = \frac{\text{Net Profit}}{\text{Capital Cost}} = 20.2\%$$

$$\text{Payout Time (POT)} = \frac{\text{Capital Cost}}{\text{Cash flow}} = 3.3 \text{ years}$$

Internal Rate of Return (IRR) is the discount rate at which the sum of the time-discounted cash flow equals the capital cost present worth. For this case IRR = 24.4%. Continuous interest factors were used per Ref. (8).