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James C. Watters[a]; Emmanuel Biagtan[ab]; Oya Senler[ac]
[a] Chemical Engineering Department, University Of Louisville, Louisville, Kentucky [b] Materials Science and Engineering Department, University of Florida, Gainesville, Florida [c] Eli Lilly & Company, Indianapolis, Indiana

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Ultrafiltration of a Textile Plant Effluent

JAMES C. WATTERS,* EMMANUEL BIAGTAN,† and OYA SENLER‡

CHEMICAL ENGINEERING DEPARTMENT
UNIVERSITY OF LOUISVILLE
LOUISVILLE, KENTUCKY 40292

Abstract

This preliminary study was initiated to determine the feasibility of using ultrafiltration to remove dyes and other contaminants from industrial textile plant waste streams. Various runs were conducted on samples of the waste stream by using a lab-scale UF unit fitted with a polysulfone XM50 hollow fiber membrane. The effects of temperature and pressure on permeate flow rate and rejection coefficient were investigated. Spectrophotometric analysis was used to determine the rejection coefficients. The average rejection coefficients ranged from 30 to 90%. The permeate-to-feed ratios ranged from 1.4 to 15.2%. Increasing the pressure increased the permeate flow rate, but also decreased the rejection coefficient. The effect of temperature was inconclusive. Fouling varied with the waste solutions, but could be enough to clog the whole unit. The pH remained at the same value of 10 for the permeate, retentate, and feed in all the runs.

INTRODUCTION

A local textile facility currently uses a municipal waste treatment plant to treat its wastewater containing dyes and other contaminants. The treated wastewater then enters the local waterway. At present, the waste treatment plant effectively reduces the dye concentration in the waste stream, but it will have difficulties in treating larger inflows. The textile facility is interested in alternative methods of treating or separating the dyes in the waste stream. This study investigated the feasibility of dye removal by ultrafiltration.

*To whom correspondence should be addressed.
†Present address: Materials Science and Engineering Department, University of Florida, Gainesville, Florida.
‡Present address: Eli Lilly & Company, Indianapolis, Indiana.

Ultrafiltration has been successfully used in other industries, but it has not been widely accepted by the textile industry. A literature search indicated that one textile facility is using a pilot plant ultrafiltration unit to recover and reuse a sizing agent, and is also using a reverse osmosis unit to separate and reuse water from a waste dye stream (*1*). The authors of that study concluded that both methods were economical and effective, but they pointed out that the reverse osmosis unit had problems with fouling, resulting in low fluxes and poor separation. Two other studies investigated the ability of UF membranes made of polysulfone to separate dyes from aqueous solutions (*2*, *3*). Polysulfone membranes were used because they can withstand a wide range of pH and high temperatures. The studies concluded that rejection coefficients greater than 90% are possible for certain dyes such as "direct red" and "direct green." Unfortunately, the previous studies used "model" aqueous dye solutions and did not indicate if additives other than the dyes were present.

The present study used a laboratory-scale ultrafiltration unit, incorporating a polysulfone membrane, to separate dissolved contaminants from samples of the plant waste stream. An inherent complication in measuring the effectiveness of the ultrafiltration was that proper determination of the sample compositions was not feasible. Each waste dye sample contained not only the organic dyes, but additives including surfactants and salts. The concentration and nature of some of the additives are proprietary. Furthermore, the dyeing process is a batch process, and the spent dye solutions from each batch are combined into one waste stream. This waste dye stream is further combined with a waste bleaching stream before it flows to screening filters and to the municipal waste treatment plant. Consequently, the color, type of dyes, and additives in the waste dye stream vary from day to day and even from hour to hour.

Spectrophotometric analysis proved to be the most practical method to determine the effectiveness of ultrafiltration in removing dissolved colored species.

THEORY

The ability of a membrane to retain a particular molecular component of a solution is characterized by its rejection coefficient, R. This coefficient is defined as

$$R = 1 - C_p/C_r \tag{1}$$

where C_p is the concentration of the component in the permeate and C_r is the concentration of the component in the retentate (*4*). The ratio C_p/C_r is termed the "sieving coefficient."

For each run in the present study, a spectrophotometer detected the amount of light that passed through samples of the feed, retentate, and permeate. For a given wavelength and solution component, the transmittance, T, is related to the absorbance, A, by

$$A = -\log_{10}(T) \tag{2}$$

Furthermore, the absorbance is directly proportional to the concentration, c, by the Beer–Lambert law:

$$A = a*b*c \tag{3}$$

where a is the absorptivity of the component and b is the solution thickness through which the light has to pass (5). The sieving coefficient can then be determined by a combination of Eqs. (1), (2), and (3). The result is

$$R = 1 - [\log_{10}(T_p)/\log_{10}(T_r)] \tag{4}$$

Since the waste solutions had unknown quantities of different solutes which could differ in their absorption of light at a particular wavelength, the percent light transmittance through the three different samples was determined for wavelengths between 350 and 800 nm at 50 nm intervals. The rejection coefficient at each wavelength and the average of these coefficients were then calculated.

MATERIALS AND METHODS

Experimental System

The experimental system is depicted in Fig. 1. The ultrafiltration unit (ROMICON HFXS-MKII) consisted of the process pump, the hollow fiber unit, and a backflush unit all mounted on an aluminum frame. A temperature gauge detected the temperature of the incoming fluid. Pressure gauges P1 and P2 measured the inlet and outlet gauge pressures exerted on the bore side of the hollow fibers. The average of P1 and P2 is the absolute pressure exerted across the membrane walls, since the permeate side was open to the atmosphere.

A Romicon HF 1.1-45-XM50 hollow fiber membrane cartridge was used in all the runs. It consisted of a 63.5-cm long by 2.54-cm wide cylindrical clear plastic shell containing 50 hollow fibers, each with a 1.1-mm inside diameter. The nominal molecular weight cutoff of the membrane is 50,000.

The percent light transmittance through the feed, retentate, and permeate samples was measured by a Milton Roy Spectronic 20D spectro-

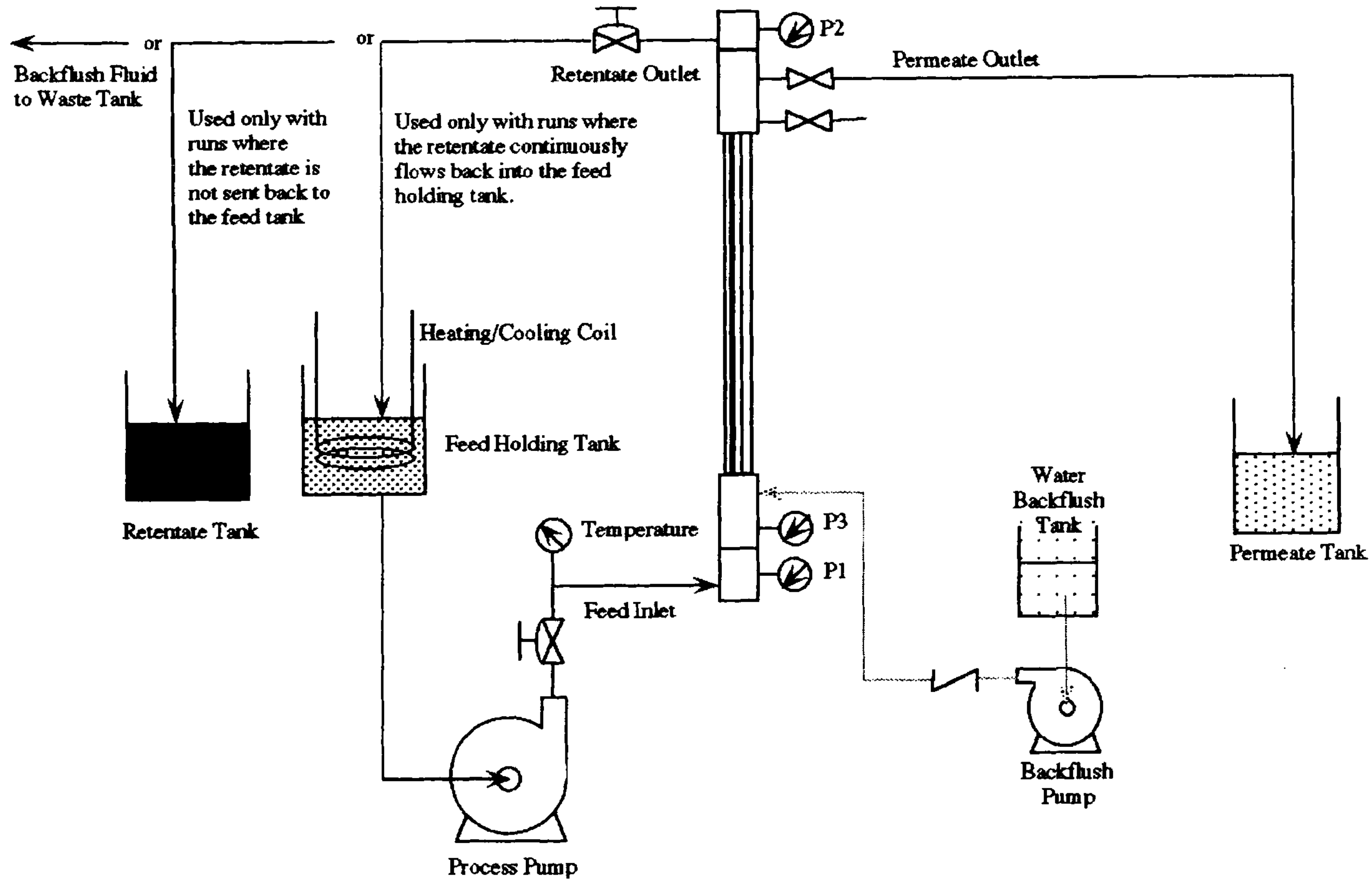

FIG. 1. Romicon HFXS-MKII ultrafiltration system.

photometer. Deionized water was used as the reference. The pH of the three samples was measured by an Orion Research 601D pH-meter. Buffer solutions with pH levels of 4.0 and 10.0 were used to calibrate this instrument.

Procedure

Samples of the wastewater were collected in 20-L buckets at the textile plant and transported to the lab. The ultrafiltration experiments were performed on the day of collection or on the days soon after so that the samples would not have time to degrade.

The general procedure was as follows:

1. Waste dye solution was poured into the feed holding tank.
2. Samples of the feed solution were retained for spectrophotometric and pH analysis.
3. The UF unit was activated, and the inlet and outlet pressures were adjusted to the desired values. The operating conditions were recorded.
4. The run was stopped after a specific time had elapsed, or when the feed tank was almost empty, or when the UF unit became clogged.
5. Changes in the operating conditions during a run were recorded. The volumes of permeate and retentate collected were noted.
6. Samples of the retentate and permeate solutions were obtained for spectrophotometric and pH analysis.
7. The percent light transmittance through the samples was measured and recorded for wavelengths between 350 and 800 nm at 50 nm intervals. The pH of the samples was measured.
8. The UF unit was backflushed, and the amount and nature of fouling material, if any, was noted.

The rejection coefficients at each wavelength were calculated from the transmittance values using Eq. (4). These rejection coefficients and the percent light transmittance of the feed, retentate, and permeate were plotted versus the wavelengths. The permeate flow rate, permeate flux, and the average value of the rejection coefficients were calculated as well.

Batch Runs

Runs 2 to 12.1 and Run 23 were batch runs. Table 1 lists their operating data. In the batch runs, the retentate was fed back into the holding tank to mix with the feed solution. The UF unit was thus filtering a feed solution that had an increasing concentration of the dissolved rejected species. The runs were stopped after 10 to 15 min, and the solution left in the feed tank was collected as the retentate.

TABLE 1
Operating Data for Batch Runs

Run	Color	Average pressure (psia)	Operating temperature range (°F)	Duration (min)	Backflushed after run	Other
2	Wine-red	20	76–82	30	No	
3	Brown-yellow	18	100 (final)	24	Yes	
4	Red-brown	21	70–90	23	Yes	
5	"	19	70–90	23	Yes	
6	"	19	70	3	Yes	Feed is Runs 4 and 5 permeates
7	Ash black	24	100–110	15	Yes	
8	"	24	86–98	15	Yes	
9	"	24	78–90	15	Yes	
10	Ash black	9	76–80	15	No	
10.1	"	9	76–84	15	Yes	Continuation of Run 10
11	"	13	76–86	15	No	
11.1	"	13	78–90	15	Yes	Continuation of Run 11
12	"	19	76–88	15	No	
12.1	"	19	80–90	15	Yes	Continuation of Run 12
23	Ash black	9	72	10	Yes	Feed is Runs 13–22 permeates

For the batch runs, the likelihood of fouling increased as the feed solution became more concentrated. After each of these runs, except for Runs 2, 10, 11, and 12, the UF unit and tank were backflushed and cleaned with tap water to remove any fouling material.

Continuous Runs

Runs 13 to 22 were continuous runs. Their operating parameters are listed in Table 2. In the continuous runs, the retentate was fed into a separate collecting tank. The feed concentration therefore remained the same during the runs. The UF unit was operated until the feed tank was nearly empty. In addition to the permeate flow rate and flux, the retentate flow rate and the ratio of the permeate to feed were calculated.

The continuous runs generally lasted less than 5 min because there was a limited volume of feed material available in each case. The likelihood of fouling was less than in the batch runs, but in order to see if fouling would occur, the unit was not backflushed between Runs 13 to 19.

Runs 2 to 6 were preliminary runs using samples collected in March. The rest of the samples were collected in May. Runs 7 to 9 were operated over different temperature ranges. Run pairs 10 to 12.1 were operated at different pressure differentials across the membrane; 9, 13, and 19 psia, respectively.

TABLE 2
Operating Data for Continuous Runs

Run	Color	Average pressure (psia)	Operating temperature range (°F)	Duration (min)	Backflushed after run	Other
13	Ash black	19	72	4	No	
14	"	20	72	5	"	
15	"	20	72–74	8	"	
16	"	20	72–74	7	"	Feed is Run 15 retentate
17	"	20	74	6	"	Feed is Run 16 retentate
18	"	25	76	14	"	Feed is Run 17 retentate
19	"	21	80	10	"	Feed is Run 18 retentate
13	Ash black	19	72	4	No	
20	"	9	80	8	See below	Feed is Run 13 retentate
22	"	9	72	1	No	Feed is Run 20 retentate
14	Ash black	20	72	5	No	
21	"	20	72	1	No	Feed is Run 14 retentate

Run 20 was backflushed and redone. Afterwards the unit was taken apart and discovered obstructions were removed.

Runs 13 to 15 used fresh identical waste solutions as their feed. Run 16 used the retentate from Run 15 as its feed solution. Subsequently, Run 17 used the retentate from Run 16 as its feed solution, and so on through Run 20.

Run 20 used the retentate from Run 13 as its feed. Since Run 20 had an unusually low permeate flow rate, the unit was dismantled to determine the cause. Balls of cotton fiber were discovered obstructing several of the hollow fibers. They must have influenced the permeate rate in Run 20 and possibly even in earlier runs. Runs 21 and 22 were operated using the same pressure settings and temperatures as earlier runs to determine how much the cotton balls had influenced the earlier results.

Runs 6 and 23 used the combined permeates from earlier runs as their feed solutions.

DISCUSSION OF RESULTS

Scaling

Tables 3 and 4 list the calculated data for the batch and continuous runs, respectively. The tables indicate that scaling was not a problem with the

TABLE 3
Calculated Data for Batch Runs

Run	Average pressure (psia)	Operating Temperature range (°F)	Permeate flow rate (L/h)	Permeate flux (L/m²*h)	Average % rejection coefficient	Amount of scaling
2	20	76–82	7.6	76.0	83	[a]
3	18	100	2.8	27.5	34	Large/clogged
4	21	70–90	7.8	78.0	79	Medium
5	19	70–90	10.7	106.9	90	Medium
6	19	70	20.0	200.0	40	Not observed
7	24	100–110	16.0	160.0	82	Small
8	24	86–98	13.7	137.0	71	Small
9	24	78–90	12.6	126.0	76	Small
10	9	76–80	8.0	80.0	91	[a]
10.1	9	76–84	6.3	63.0	93	Miniscule
11	13	76–86	8.0	80.0	78	[a]
11.1	13	78–90	7.4	74.0	85	Miniscule
12	19	76–88	9.6	96.0	70	[a]
12.1	19	80–90	8.0	80.0	77	Small
6	19	70	20	200	40	Not observed
23	9	72	9.2	92	88	Not observed

[a]Not applicable.

TABLE 4
Calculated Data for Continuous Runs

Run	Average pressure (psia)	Operating Temperature range (°F)	Permeate flow rate (L/h)	Permeate flux (L/m²*h)	Permeate/ feed flow ratio	Average % rejection coefficient	Amount of scaling
13	19	72	18	180	7.8%	77	[a]
14	20	72	14.8	148	7.5%	76	[a]
15	20	72–74	13.9	139	9.8%	77	[a]
15	20	72–74	13.9	139	9.8%	77	[a]
16	20	72–74	13.2	132	9.3%	76	[a]
17	20	72	12.3	123	7.8%	79	[a]
18	25	76	9.2	92	15.2%	64	[a]
19	21	80	9.2	92	11.6%	73	[a]
13	19	72	18	180	7.8%	77	[a]
20	9	80	4.6	46	5.1%	71	None
22	9	72	9	90	1.4%	90	[a]
14	20	72	14.8	148	7.5%	76	[a]
21	20	72	37	370	7.7%	78	[a]

[a]Not applicable.

samples collected in May, but was a problem with the earlier samples collected in March. The March samples were reddish brown in color, while the May samples were ash black. Different dyes were present in the two sets of samples, but it is not certain if the differences in the dyes or other components in the solutions caused the fouling. The tendency to foul varies with the stream solution and could be large enough to cause clogging. Prefiltering of the waste dye stream is advised.

Effect of UF on pH

Tables 5 and 6 are the data sheets for Runs 2 and 3, respectively. The pH of the feed, retentate, and permeate stayed the same for both of the runs, and in fact stayed at the same value of 10 for all of the runs. The largest component by far in the waste solution is bleach. Bleach is not separated by ultrafiltration, so the quantity in the feed, retentate, and permeate remained the same, and hence so did the pH.

Spectrophotometric Analysis

Figure 2 is the plot of the percent light transmittance for the feed, retentate, and permeate, and of the rejection coefficient versus the wavelength for Run 2. The plot reveals an important anomaly in using light

TABLE 5
Run 2: Preliminary Run Data[a]

Run	2
Color	Wine red
Collected	3/30
Run	3/30, 8:30 PM
Apparatus	Romicon
Membrane	XM50 hollow fibers
Method	Batch
P1	22 psi
P2	18 psi
T	76–82°F
Duration	30 min
Backflushed	Before run
Scaling	No scaling
Other	observed

Wavelength (nm)	Percent transmittance			Percent rejection coefficient
	Feed	Retentate	Permeate	
350	3	0	30	100
400	4	1	30	74
450	2	0	21	100
500	2	0	18	100
550	8	2	38	75
600	22	9	70	85
650	31	16	73	83
700	18	11	44	63
750	6	2	34	72
800	4	1	31	75
pH	9.73	9.73	9.7	
Volume (mL)	[b]	4000	3800	

[a]Permeate flow was not constant; it went up and down during the run.
[b]Not recorded.

TABLE 6
Run 3: Preliminary Run Data[a]

Run	3
Color	Brownish yellow
Collected	3/30
Run	3/30, 10:18 PM
Apparatus	Romicon
Membrane	XM50 hollow fibers
Method	Batch
P1	20 psi
P2	15 psi
T	100°F
Duration	24 min
Backflushed	After run
Scaling	Clogged
Other	

Wavelength (nm)	Percent transmittance			Percent rejection coefficient
	Feed	Retentate	Permeate	
350	8	7	22	43
400	5	4	13	37
450	2	2	6	28
500	3	3	7	24
550	11	11	23	33
600	14	13	27	36
650	24	23	40	38
700	23	22	37	34
750	9	9	21	35
800	5	5	14	34
pH	9.94	9.94	9.91	
Volume (mL)	[b]	3800	1100	

[a]P2 decreased steadily after 16 min. P2 decreased to 0 psi after 24 min. Run stopped due to no flow. Sample of fouling material collected.
[b]Not recorded.

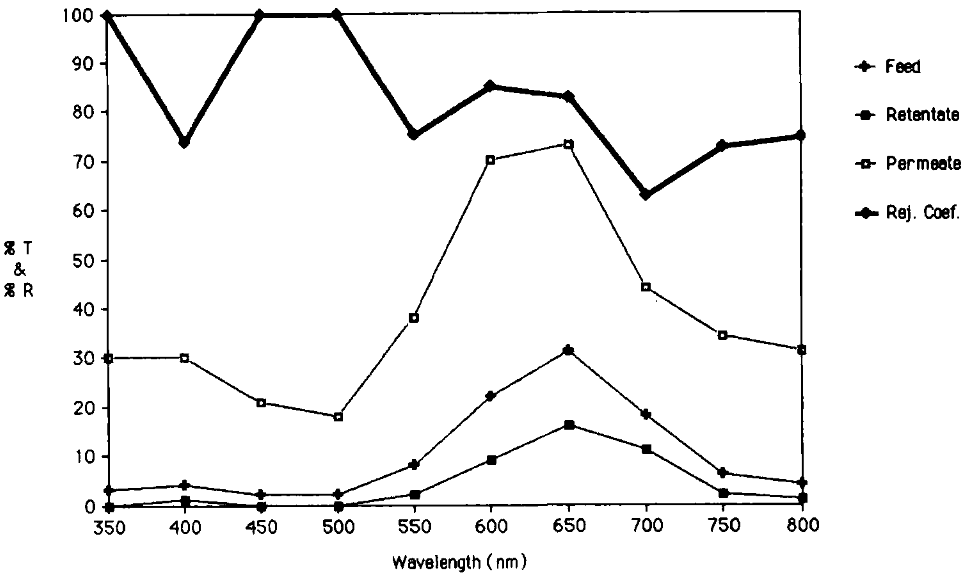

FIG. 2. Run 2: Preliminary run graph.

transmission to determine the rejection coefficient. When the concentration of the dyes in the retentate is high enough to prevent any passage of light, it does not matter what the concentration of dyes in the permeate is. The rejection coefficient always becomes 100%. It is therefore important to realize that the rejection coefficient is a function of both permeate and retentate concentrations, and that a 100% rejection coefficient does not necessarily mean a pure permeate. Figure 3 is the plot for Run 3, where the rejection coefficient value was more constant over the given wavelength range.

Spectrophotometric analysis was practical and simple, but some of the components in the waste solutions may not absorb wavelengths of light in the visible range. Any further spectrophotometric analysis should also include wavelengths in the ultraviolet and infrared range in order to detect the presence of these other components.

Effect of Temperature

Figure 4 is a plot of the average rejection coefficients and permeate flow rate for all of the batch runs. Due to temperature overlap and the limited set of data, any relationships between permeate flow rate or average percent rejection coefficient and temperature cannot be determined.

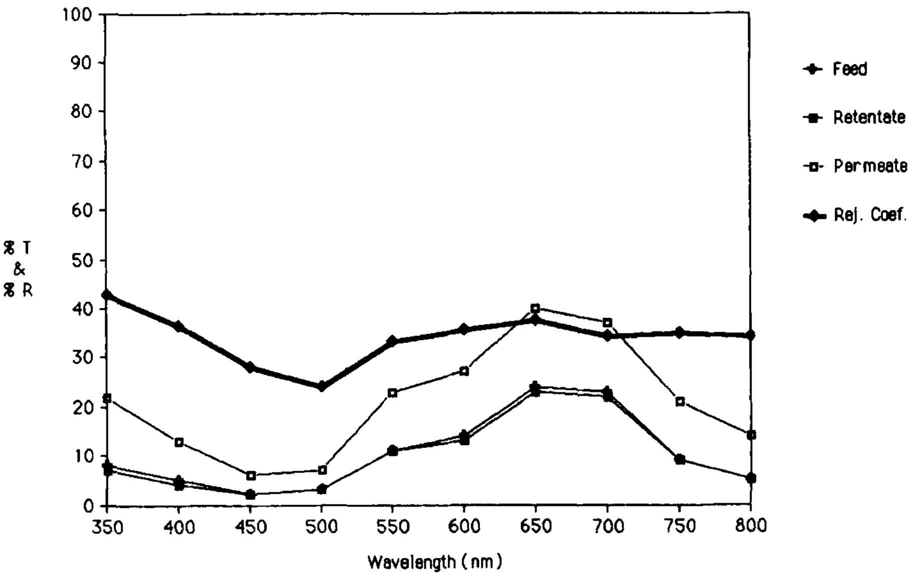

FIG. 3. Run 3: Preliminary run graph.

Effect of Pressure

Run pairs 10–10.1, 11–11.1, and 12–12.1 were operated over different average operating pressures of 9, 13, and 19 psia, respectively. The permeate flow rate increased with increasing operating pressure (Fig. 5). The average rejection coefficient decreased with increasing operating pressure (Fig. 6). The figures also indicate that the permeate flow rate decreased, while the average rejection coefficient increased with increasing time. These results are consistent with the concept of a build up of rejected material at the membrane wall. This thin layer acts as a prefilter. It allows only the smaller molecules to pass through; therefore the clarity of the permeate improves, but at the same time the permeate flow is reduced. The differences in the permeate flow rates are less than the measurement errors, but there is a consistent trend with time and with pressure.

Batch versus Continuous

The batch runs were not as revealing as the continuous runs. The duration of a batch run has a direct effect on the rejection coefficient. The longer the unit is allowed to operate, the more permeate is collected, and the more concentrated the feed becomes in the rejected components. Any desired amount of permeate can be collected by operating the unit for a

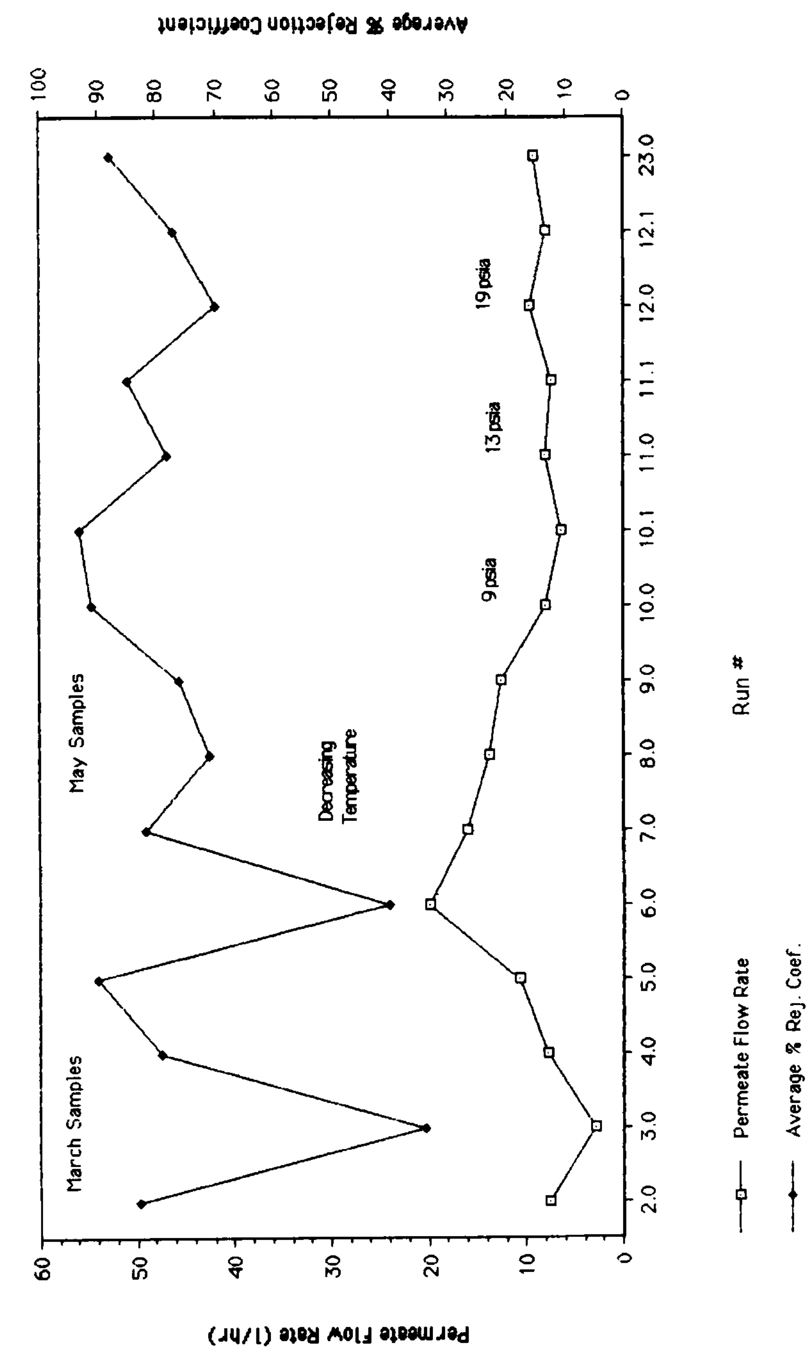

FIG. 4. Permeate flow rate and average percent rejection coefficients for batch runs.

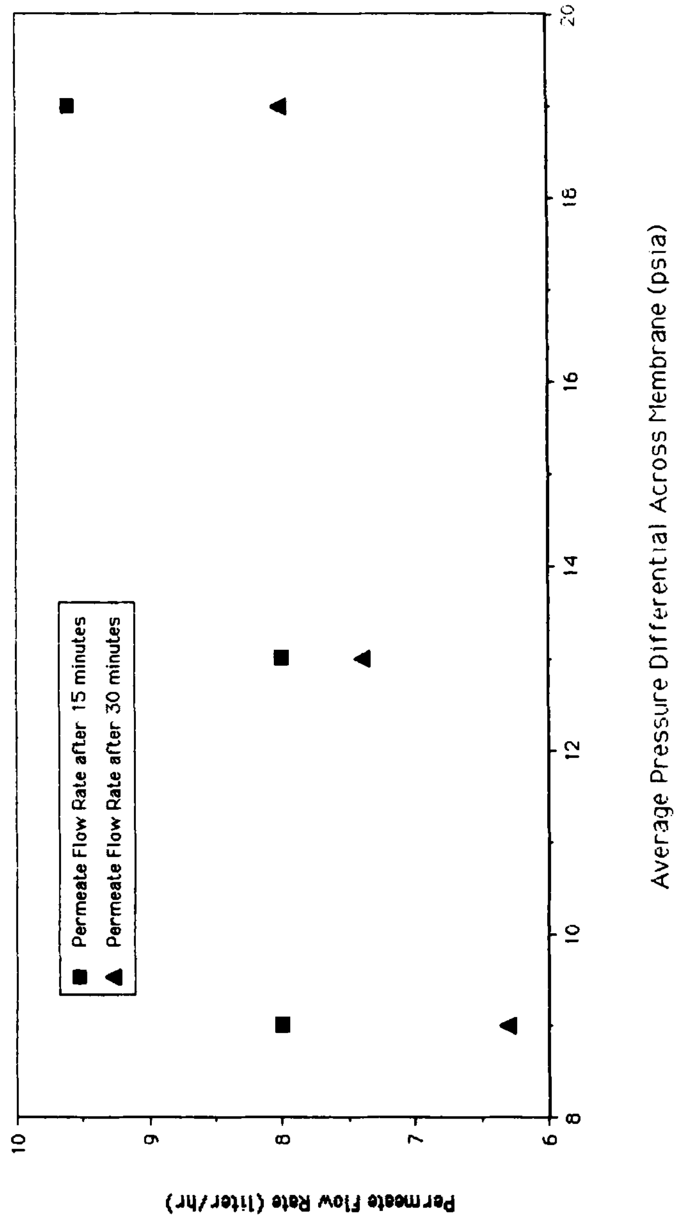

FIG. 5. Permeate flow rate as a function of pressure and run time.

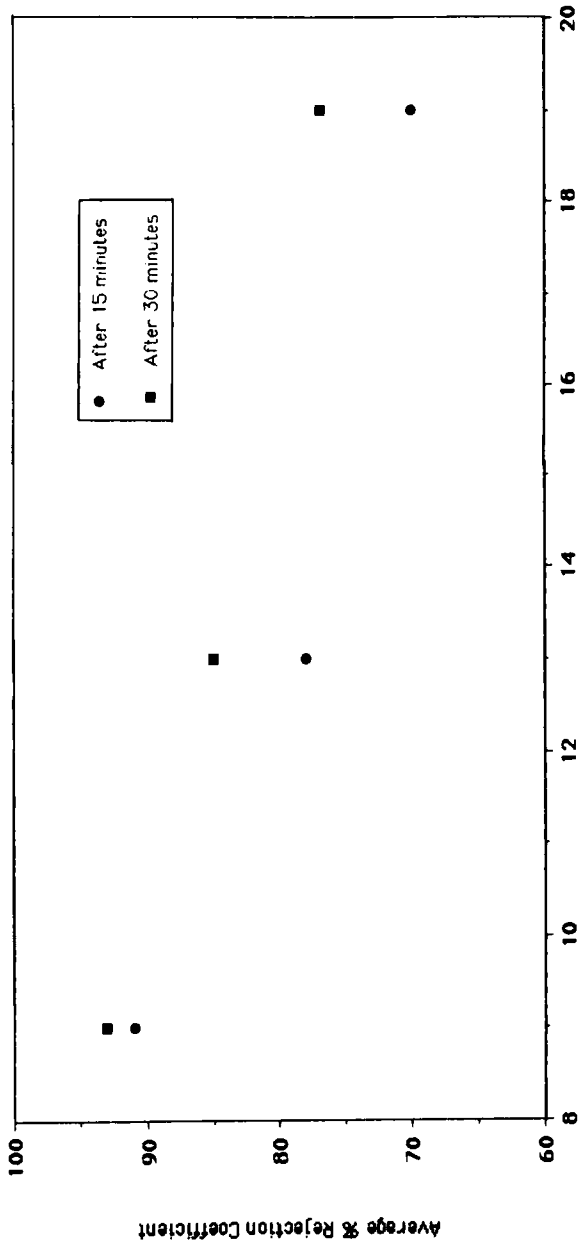

FIG. 6. Average percent rejection coefficient as a function of pressure and run time.

long enough time. Therefore, the resultant rejection coefficients and ratio of the permeate to feed volumes are functions of the run time.

The continuous runs were independent of the run time. Whatever volume of permeate is collected from that one pass is more a function of the pressure, temperature, flow rate, and dye composition.

Continuous Runs

Runs 13 to 19 were operated at approximately the same temperature and pressure. The permeate flow rates and the average percent rejection coefficients were similar for Runs 13 to 17 (Fig. 7). Discrepancies observed for Runs 18 to 20 were likely due to the cotton ball obstructions removed after Run 20.

Runs 21 and 22, performed after the cotton ball obstructions had been removed, showed much higher permeate flow rates than Run 20 and earlier. This indicates that the obstructions decreased the permeate flow rate. However, the effect of the obstructions on the rejection coefficients is not apparent from the data obtained.

Permeate-to-Feed Ratio

The permeate/feed ratios range from 1.4 to 15.2% (see Fig. 8). For Runs 13 to 22, the average value is 8.3%. The permeate flow therefore is

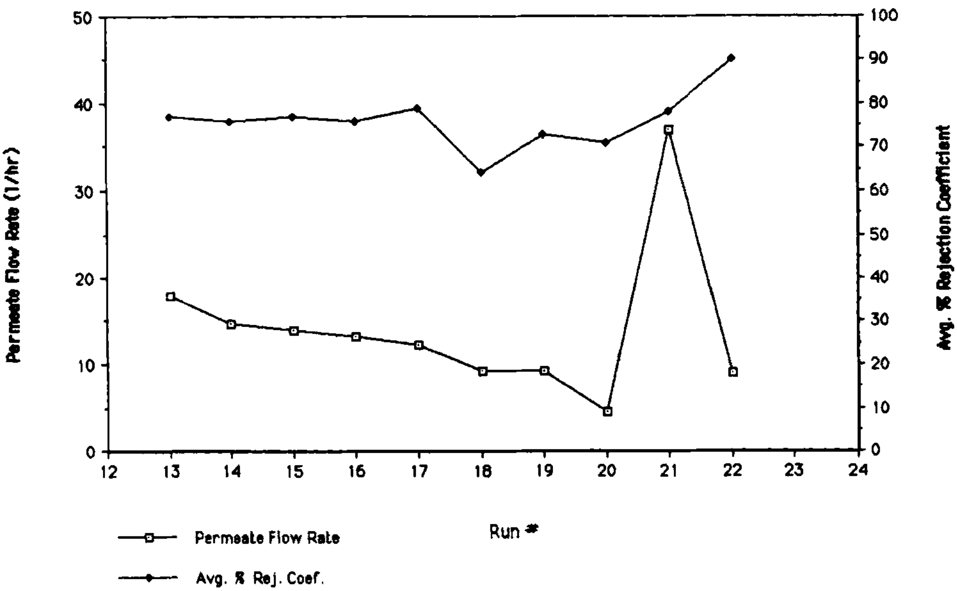

FIG. 7. Permeate flow rate and average percent rejection coefficient for continuous runs.

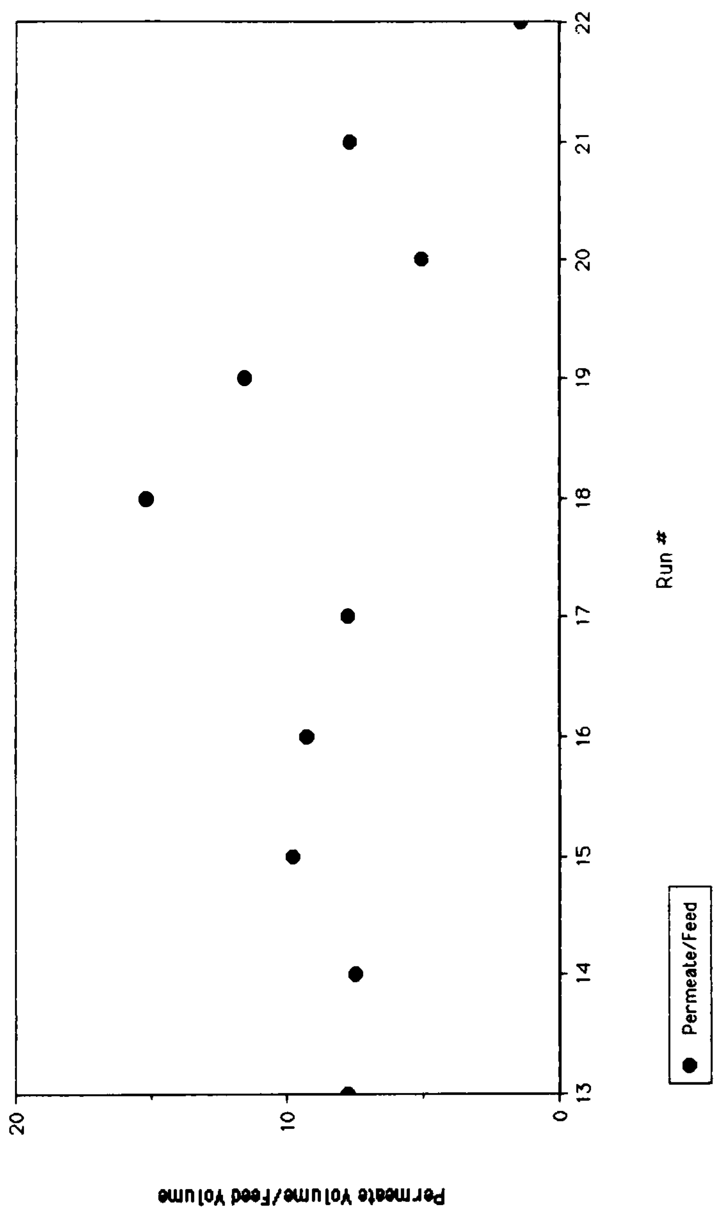

FIG. 8. Permeate-to-feed-volume ratios for continuous runs.

the smaller of the two outflows, and it must be increased considerably for UF to be a viable option in the treatment of this waste water.

Refiltering the Permeate

Runs 6 and 23 used the permeates from earlier runs as their feed solution (Fig. 4). The permeate can be further cleaned by passing it through the filter again. The additional clarification will depend on the operating parameters and the initial clarity of the permeate solution.

Difficulties in Extrapolating Results to a Larger Unit

Although 22 runs were done, possible errors in volume measurement, difficulties with scaling and clogging, difficulties in maintaining temperature, and the limited variety of the waste solutions make it difficult to extrapolate numerical results from this study to a working unit for the textile plant. More studies are necessary to determine the practicality of ultrafiltration in separating the dyes from the textile waste stream. However, some general conclusions are summarized below.

CONCLUSIONS

Ultrafiltration can remove some of the dyes from the waste feed solutions. The separation depends on the solution properties and the UF unit operating parameters. In the present study, rejection coefficients ranged from 30 to 90%. The effect of temperature was inconclusive. Increasing the pressure increased the permeate flux but decreased the average rejection coefficient. Permeate-to-feed ratios ranged from 1.4 to 15.2%. For the process to be industrially viable, considerably higher permeate flow rates are necessary. However, the permeate fluxes were generally 50% or greater of the pure water flux rate quoted by Romicon in their literature for the XM50 membrane. Scaling and eventual clogging of the UF membrane may also occur, and did in some of the runs described herein. The tendency to foul varied with the waste solution, and some level of prefiltering may be necessary.

On-site studies should be done to better determine the practicality for ultrafiltration of textile waste streams. On-site studies will be more insightful since the unit will be operating under more real conditions and for longer run times.

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