Microfiltration for Cleaner Bath Life Extension

An EPA Environmental Technology Verification of a Metal Finishing Pollution Prevention Technology

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The U.S. Environmental Protection Agency (EPA) Environmental Technology Verification for Metal Finishing Pollution Prevention Technologies (ETV-MF) Program, in association with the National Metal Finishing Strategic Goals Program, is a pilot for verifying the performance of innovative, commercial-ready technologies designed to improve industry performance and achieve cost effective pollution prevention solutions. Test plans are developed cooperatively between Concurrent Technologies Corporation, EPA and the technology supplier. Verification testing is conducted under strict EPA quality guidelines in metal finishing shops, where possible, under actual operating conditions.

This paper will discuss verification test results of a cross flow microfiltration technology for an alkaline cleaner bath. The test methods, data analysis, and conclusions will be presented, including the environmental and economic benefits of this technology. Preliminary data indicate that this technology is effective in removing organic soils from the bath while preserving the chemical constituents. The presentation will include an update of the EPA ETV-MF program and the status of other verification test projects.

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Introduction

The U.S. Environmental Protection Agency (EPA) has instituted a program, the Environmental Technology Verification Program, or ETV, to substantially accelerate the entrance of new environmental technologies into the domestic and international marketplace. ETV conducts performance verification of commercial-ready, technologies through 12 centers that cover a range of industry sectors and environmental areas. For one of these 12 centers, EPA has partnered with *CTC* to establish the Environmental Technology Verification for Metal Finishing Pollution Prevention Technologies (ETV-MF) Center.[1] *CTC* is currently testing pollution prevention technologies that are used for water reuse, bath maintenance, chemical recovery, sludge reduction, and energy conservation.

This paper describes the test results of a microfiltration technology used for recycling used alkaline cleaner.[2] The test was conducted according to a verification test plan prepared by the ETV-MF Center [Ref 1]. This system removes suspended solids and oil and produces a permeate that is reused in the cleaning process. The equipment was tested under actual production conditions at Gates Rubber Company in Versailles, MO, during May and June 2000.

A summary of key findings from the verification test is presented in this paper. A complete summary of this project can be found in the verification test report [Ref. 2].

System Description

Alkaline cleaning is performed at various points in the Gates Rubber Company plant. There are 12 in-process cleaning tanks present in areas such as machining. Eleven of these units hold 40 gallons of alkaline cleaner and one holds 75 gallons. The largest cleaning operation is located on the barrel plating (zinc) line, where there is a 1,800 gallon soak cleaning tank and a 1,800 gallon electrocleaning tank. The 12 in-process cleaning tanks and the soak cleaning tank are plumbed into the cleaner recycling system that was tested during this project. The electrocleaning tank is serviced by a separate recycling system that was not tested.

A diagram of the alkaline cleaner recycling system at Gates Rubber Company is shown in **Figure 1**. In operation, the contaminated cleaner enters a two-compartment, type 304 stainless steel tank through a filter (polypropylene sock and stainless steel basket) that removes large particulate material from the feed stream. The level in the tank is maintained by a level switch, which controls the tank inlet valve and also acts as a low-level cutoff for the system pump. Oils may accumulate in the initial compartment (referred to as the settling tank) and can be removed on a periodic basis through a drain port located on the upper part of the tank. The liquid then moves to a second tank compartment through a sub-surface passage, leaving any floating oils in the first compartment. The liquid in the second compartment (referred to as the recirculation tank) is pumped through the ceramic membrane located in the microfiltration module. A portion of the water and cleaner chemicals are forced through the ceramic membrane and exit the system to a permeate holding tank, while a portion of the water and cleaner chemicals are retained, along with oil and suspended solids and recycled back to the recirculation tank. Once a week at Gates Rubber Company the liquid in the recirculation tank is discarded and the tank and ceramic membrane are cleaned.

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[1] Additional information on EPA's ETV program can be found at www.epa.gov/etv. Information specific to the ETV-MF program can be found at www.etv-mf.org.
[2] USFilter Membralox® Silverback™ Model 900 Alkaline Cleaner Recycling System.

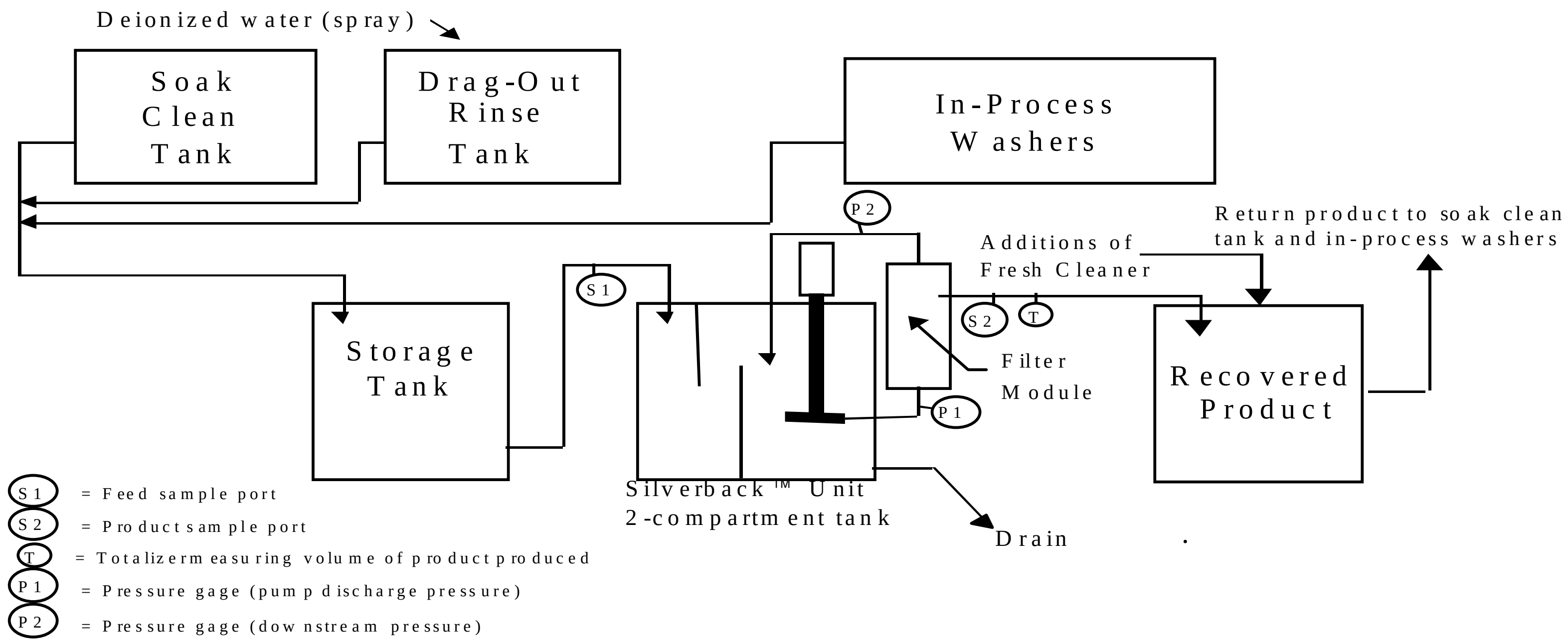

Figure 1. Alkaline Cleaner Recycling at Gates Rubber Company

Verification Testing

The alkaline cleaner recycling unit was tested in accordance with the verification test plan [Ref. 1]. The primary objectives of the test were:

- Evaluate the ability of the recycling unit to process used alkaline cleaner solution and separate usable cleaner solution chemistry from bath contaminants.
- Evaluate the impact of the recycling unit on waste generation at this particular site.
- Determine the cost of operating the alkaline cleaning recovery system for the specific conditions encountered during testing.

Testing was conducted during two distinct, five-day test periods:

- During the first test period (Run 1), the unit was operated under the normal production conditions at Gates Rubber Company. The unit processed 7,123 gallons (26,953 L) of used alkaline cleaner. At the completion of Run 1, the recirculation tank, which holds the soil removed from the alkaline cleaning solution, was drained from the recovery unit and stored for later use in Run 2.
- During the second test period (Run 2), the recovery unit was operated under normal production conditions, with one exception. To evaluate the operation of the recovery unit under a high soil loading condition, the recirculation tank solution that was removed and stored during Run 1 was introduced into the storage tank that feeds the cleaner recovery system at a uniform rate during the entire second test period. This procedure significantly increased the soil loading on the recovery unit during Run 2. During Run 2, the unit processed 7,028 gallons (26,601 L) of used alkaline cleaner.

A complete summary of analytical data is presented in **Table 1**. The samples coded "IN" are 24-hour composite samples of the feed to the recovery unit, and those coded "EFF" are 24-hour composite samples of the recovered permeate. Average values calculated for both the IN and EFF samples are also shown. The R-1 and R-2 samples are grab samples from the recovery tank, collected at the end of Runs 1 and 2. The "CLEANER" sample is a grab sample of the unused concentrated cleaner. The values for "5% of CLEANER" were calculated by multiplying the CLEANER results by 5%. These values approximate the concentration of these constituents in a freshly formulated alkaline cleaner bath (i.e., the alkaline cleaning solution at Gates Rubber Company is formulated with a 5% solution).

Recovery Efficiency of Alkaline Cleaner Components

Recovery efficiencies were calculated for four dissolved species: total alkalinity, carbonate, bicarbonate, and dipropylene glycol ether. These calculations were performed for each daily set of paired analytical results. The equation for the alkalinity recovery calculation is shown below (Eq. 1). The recovery efficiency for other parameters was calculated using a similar equation.

$$A_{eff}\,(\%) = [(A_{prod} \times Prod_{vol})/(A_{feed} \times Feed_{vol})] \times 100\% \qquad (1)$$

where:
- A_{eff} = alkalinity recovery efficiency;
- A_{prod} = permeate (EFF) stream alkalinity concentration (grams/liter);
- $Prod_{vol}$ = permeate volume collected during the cycle (liters);
- A_{feed} = feed (IN) solution alkalinity concentration (grams/liter); and
- $Feed_{vol}$ = feed solution volume processed during the cycle (liters).

Table 1. Summary of Analytical Results

	Total Alkalinity mg/l as CaCO3	Carbonate Alkalinity mg/l as CaCO3	Bicarbonate Alkalinity mg/l as CaCO3	Hydroxide Alkalinity mg/l as CaCO3	Dipropylene Glycol Ether mg/l	Ammonia Nitrogen mg/l	Total Nitrogen (TKN) mg/l	Total Phenol mg/l	Total Suspended Solids mg/l	Total Solids mg/l	Phosphate mg/l (as P)	Oil mg/l
Run 1												
IN-1	2,700	1,300	650	<1	6,200	2.8	2.8	0.59	100	9,600	400	100
EFF-1	2,500	1,300	640	<1	6,300	3.7	3.8	0.63	24	9,100	390	19
IN-2	2,600	1,400	680	<1	6,200	3.1	4.3	0.57	170	9,600	390	180
EFF-2	2,500	1,300	670	<1	6,100	4.1	4.5	0.54	30	7,600	380	16
IN-3	2,600	1,200	620	<1	6,400	0.97	3	0.58	180	9,600	390	76
EFF-3	2,600	1,400	680	<1	6,200	3.5	23	0.42	48	9,200	390	18
IN-4	2,600	1,300	670	<1	6100	1.1	7.2	0.05	160	9,500	390	200
EFF-4	2,600	1,400	680	<1	6,300	3.3	3.6	0.16	66	9,100	380	17
IN-5	2,400	1,100	550	<1	5,900	2.6	12	0.05	210	8,400	350	180
EFF-5	2,400	1,100	560	<1	6,300	2.7	3.2	0.062	92	8,500	350	51
IN-RUN 1 AVG	2,580	1,260	634	<1	6,160	2.1	5.9	0.4	164	9340	384	147
EFF-RUN 1 AVG	2520	1300	646	<1	6240	3.5	7.6	0.4	52	8700	378	24
R-1	3,300	1,200	620	<1	6,900	11	68	0.52	10,000	16,000	800	5,000
Run 2												
IN-6	2,200	1,000	560	<1	6,400	2.1	37	0.5	590	11,000	410	440
EFF-6	1,900	1,000	480	<1	6,400	0.023	37	0.57	1	10,000	320	21
IN-7	2,100	1,100	520	<1	5,700	1.9	43	0.7	910	11,000	490	1000
EFF-7	2,000	1,100	500	<1	4,650	0.088	35	0.63	23	10,000	310	17
IN-8	2,600	1,100	540	<1	5,100	1.4	36	0.61	400	9,600	420	620
EFF-8	2,600	1,300	630	<1	5,100	0.025	39	0.58	4	9,300	360	13
IN-9	2,500	1,200	600	<1	5,200	2	26	0.57	180	8,900	420	530
EFF-9	2,200	1,000	520	<1	4,850	0.032	33	0.54	6	8,300	400	18
IN-10	2,300	1,000	500	<1	4,500	1.3	6.7	0.47	170	10,000	320	710
EFF-10	2,300	940	470	<1	4,500	0.078	0.49	0.19	35	11,000	340	23
IN-RUN 2 AVG	2,340	1,080	544	<1	5,380	1.7	29.7	0.6	450	10,100	412	660
EFF-RUN 2 AVG	2,200	1,068	520	<1	5,100	0.049	28.9	0.5	14	9,720	346	18
R-2	3,900	1,000	520	<1	5,200	3.4	44	0.95	6700	85,000	990	16,000
CLEANER	23,000	3,000	15,000	6,400	118,000	0.024	0.25	1.5	100	80,000	310	470
5% of CLEANER	1,150	150	750	320	5,900	0.0012	0.0125	0.075	5	4,000	15.5	23.5

The calculated results for recovery efficiency are shown in **Table 2**. The average recovery percentages for alkalinity, carbonate, bicarbonate, and dipropylene glycol ether were high (93.9% to 103.5%), indicating that over the short time period of the verification test, there was little or no change in the concentration of these parameters. Calculated recoveries can be greater than 100% due to chemical reactions or measurement uncertainty.

Table 2. Cleaner Recovery Efficiency

Test Run and Sample Date	Total Alkalinity % Recovered	Carbonate % Recovered	Bicarbonate % Recovered	Dipropylene Glycol Ether % Recovered
Run 1				
5/22/00	92.6	100.0	98.5	101.6
5/23/00	96.2	92.9	98.5	98.4
5/24/00	100.0	116.7	109.7	96.9
5/25/00	100.0	107.7	101.5	103.3
5/26/00	100.0	100.0	101.5	106.8
Avg. Run 1	97.8	103.5	101.9	101.4
Standard Deviation	3.3	9.1	4.6	3.9
Run 2				
6/12/00	86.4	100.0	85.7	100.0
6/13/00	95.2	100.0	96.2	81.6
6/14/00	100.0	118.2	116.7	100.0
6/15/00	88.0	83.3	86.7	93.3
6/16/00	100.0	94.0	94.0	100.0
Avg. Run 2	93.9	99.1	95.9	95.0
Standard Deviation	6.5	12.7	12.5	8.0

Contaminant Removal Efficiency

Contaminant removal efficiencies were calculated for the primary contaminants of the alkaline cleaning bath: oil and total suspended solids (TSS). The equation for oil removal efficiency is shown below (Eq. 2). The TSS removal efficiency was calculated using a similar equation.

$$O_{eff}\ (\%) = 100\% - [[(O_{prod} \times Prod_{vol})/(O_{feed} \times Feed_{vol})] \times 100\%] \qquad (2)$$

where:
- O_{eff} = oil recovery efficiency;
- O_{prod} = permeate stream oil concentration (grams/liter);
- $Prod_{vol}$ = permeate volume collected during the cycle (liters);
- O_{feed} = feed solution oil concentration (grams/liter); and
- $Feed_{vol}$ = feed solution volume processed during the cycle (liters).

The calculated results are shown in **Table 3**. During Run 1, the recycling unit removed an average of 69.3% of the TSS (6.7 lb) and 82.3% of the oil (7.3 lb) from the feed solution, producing a permeate with average concentrations of 52 mg/l TSS and 24.2 mg/l of oil.

Table 3. Contaminant Removal Efficiency

Test Run and Sample Date	TSS % Removal	Oil % Removal
Run 1		
5/22/00	76.0	81.0
5/23/00	82.4	91.1
5/24/00	73.3	76.3
5/25/00	58.8	91.5
5/26/00	56.2	71.7
Avg. Run 1	**69.3**	**82.3**
Std. Dev. Run 1	**11.3**	**8.8**
Run 2		
6/12/00	99.8	95.2
6/13/00	97.5	98.3
6/14/00	99.0	97.9
6/15/00	96.7	96.6
6/16/00	79.4	96.8
Avg. Run 2	**94.5**	**97.0**
Std. Dev. Run 2	**8.5**	**1.2**

During Run 2, the recycling unit removed an average of 94.5% of the TSS (25.6 lb.) and 97.0% of the oil (37.6 lb.) from the feed solution, producing permeate with average concentrations of 13.8 mg/l TSS and 18.4 mg/l of oil.

During Run 1 there was a lower average concentration of TSS in the feed (164.0 mg/l) than during Run 2 (450.0 mg/l). This difference is due to the testing procedure, where adding a concentrated soiled solution to the feed stream during Run 2intentionally increased the concentration of these contaminants. Despite a higher TSS loading during Run 2, the permeate stream had a lower TSS concentration than in Run 1 (13.8 mg/l vs. 52 mg/l). The average TSS removal efficiency was 68.3% during Run 1 and 96.9% during Run 2. The higher removal efficiency during Run 2 was due to the combined effect of a higher average loading concentration and a lower average effluent concentration.

A similar, but less pronounced pattern was observed for the oil results. The average oil removal efficiencies were 83.6% for Run 1 and 97.2% for Run 2. The average feed (IN) and permeate (EFF) concentrations during Run 1 were 147.2 mg/l and 24.2 mg/l, respectively. During Run 2, the average feed and permeate concentrations were 660 mg/l and 18.4 mg/l, respectively.

Chemical Use Analysis

Prior to the installation of an alkaline cleaner recovery unit, Gates Rubber Company used 8,448 gal/yr of their concentrated cleaner, CLEAN-R-120GR (data from 1993).[3] Adjusted for changes to production volume, an

[3] In 1994 Gates Rubber Company implemented a polymer membrane, alkaline cleaner recovery unit and subsequently replaced that equipment with the ceramic membrane recycling unit. Therefore, 1993 is the most recent year that is representative of using the alkaline cleaning system without a recovery unit installed.

equivalent quantity for 1999 is 10,729 gal/yr.[4] During 1999, Gates Rubber Company actually used 5,390 gal/yr of the concentrated cleaner product. Therefore, the production-adjusted savings in cleaner use is 5,339 gal/yr (10,729 gal/yr - 5,390 gal/yr).

Waste Generation Analysis

Prior to the installation of the recovery unit, the alkaline cleaning bath was drained and fresh chemistry was added 15 times per year. During use, the alkaline cleaning system generated a discharge from the rinse tank following the alkaline cleaning tank. This discharge from rinsing was estimated to be 1 gpm. The used rinse water was treated on-site. This information could not be verified during the project. The treatment process generated a sludge that was sent off-site for disposal. The quantity of sludge generated prior to the installation of the recycling unit could not be quantified during this project. Overall, the bath replacement procedure generated the following wastes:

- Spent alkaline cleaning solution
- Dilute wastewater from tank washdown
- Rinse water following alkaline cleaning step (dilute wastewater)
- Wastewater treatment sludge

Skimming oil off of waste storage tanks in the waste treatment area generates waste oil. Waste oil was generated in similar quantities before and after the installation of the recycling unit. Gates sends about 500 gallons of waste oil off-site every two years. The cost of hauling/disposal is $1.00/gallon There is no waste reduction or cost savings that have resulted by installation of the recycling unit, with respect to waste oil.

Following installation of the recovery unit, the alkaline cleaning system is drained and replaced two times each year. The recovery unit is drained and cleaned weekly. These procedures generate a concentrated waste and dilute wastewater (from cleaning the unit's tank and filter module). These liquid wastes are combined with other wastewaters and treated on-site. The quantity of sludge generated could not be quantified during this project. Overall, this procedure generated the following wastes[5]:

- Spent alkaline cleaning solution
- Wastewater from tank washdown (dilute wastewater)
- Weekly draining of recovery unit
- Weekly cleanout of recovery unit (dilute wastewater)
- Wastewater treatment sludge

The results of the waste generation analysis (concentrated wastes only) are shown in **Table 4.**

[4] This adjustment was calculated using zinc anode purchases as a normalizing factor. Zinc anodes are used on the zinc plating line and are a good indicator of overall production volume at this site. Zinc anode purchases in 1993 and 1999 were 44,800 lb/yr and 56,700 lb/yr, respectively. Therefore, under the same conditions, if Gates Rubber Company used 8,448 gal. of cleaner in 1993, they would be expected to use 10,729 gal. in 1999.

[5] This recycling unit has a drain port located on the upper part of the settling tank that can be used to remove floating oil from the tank. This drain is not used at Gates Rubber Company and therefore a separate oil waste is not generated during the recovery process.

Table 4. Results of Waste Generation Analysis

Waste Type	Waste Volume gal/yr	Total Solids lb./yr[6]
Without recycling unit		
Spent alkaline cleaning solution	37,500	3,039
Totals without recycling unit	37,500	3,039
With recycling unit		
Spent alkaline cleaning solution	7,200	583
Weekly draining of recovery unit	5,000	665
Totals with recycling unit	12,200	1,248

Cost Analysis

The capital cost of the recycling unit was $43,000 (1999; includes $36,000 for the unit, $5,000 for storage tanks and plumbing, and $2,000 for installation costs).

Annual costs and savings associated with the alkaline cleaner recovery operation are shown in **Table 5.** The operating costs prior to installation of the recycling unit were $82,653, resulting in a net annual savings of $32,604. The operating costs of the alkaline cleaning/recycling process with the recycling unit are $50,049. The simple payback period is 1.3 years (capital cost/net annual savings).

Table 5. Annual Costs/Savings

Item	Prior to Installation of Recycling Unit			After Installation of Recycling Unit		
	Units	Unit Cost $/unit	Cost $	Units	Unit Cost $/unit	Cost $
Recycling unit O&M labor	0	N/A	0	187.5 hr	20.00	3,750
Alkaline Clean tank maintenance O&M labor	120 hr	20.00	2,400	16 hr	20	320
Alkaline cleaner	10,729 gal	7.48	80,253	5,390 gal	7.48	40,317
Tank/module cleaning chemicals	0	N/A	0	50	40.92	2,046
Electricity for recovery unit	0	-	0	47,005 kWh	0.07	3,290
Natural gas for recovery process	0	-	0	941.5 therms	0.35	326
Total Costs			**82,653**			**50,049**

[6] Total solids are calculated based on the analytical results from Runs 1 and 2.

SUMMARY

The test results show that the alkaline cleaner recycling system provides an environmental benefit by extending the bath life of the alkaline cleaner, thereby reducing the amount of liquid and solid wastes produced by the cleaning operation without removing the cleaning constituents of the bath. The economic benefit associated with this technology is low operating and maintenance labor and a payback period of approximately 1.3 years. As with any technology selection, the end user must select appropriate cleaning equipment and chemistry for a process that can meet their associated environmental restrictions, productivity, and cleaning requirement.

References

1. Concurrent Technologies Corporation, "*Environmental Technology Verification Program for Metal Finishing Pollution Prevention Technologies Verification Test Plan, Evaluation of USFilter Membralox® Silverback™ Model 900 Alkaline Cleaner Recycling System,*" April 4, 2000.

2. Concurrent Technologies Corporation, "*Environmental Technology Verification Program for Metal Finishing Pollution Prevention Technologies Verification Test Report, Evaluation of USFilter Membralox® Silverback™ Model 900 Alkaline Cleaner Recycling System,*" September 30, 2000.