EPA Environmental Technology Verification Experience

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The U.S. EPA's Environmental Technology Verification for Metal Finishing Pollution Prevention Technologies (ETV-MF) Program verifies 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 *CTC*, EPA and the technology supplier. Verification testing is conducted under strict EPA quality guidelines in metal finishing shops under actual operating conditions. This paper summarizes the verification test results for two alkaline cleaner recycling technologies: microbial digestion and microencapsulation. Following this summary, verification of a cross-flow microfiltration technology for an alkaline cleaner bath is discussed in detail. The test methods, data analysis, and conclusions are presented, including the environmental and economic benefits of this technology. The data indicate that this technology is effective in removing organic soils from the bath while preserving the chemical constituents.

For more information contact: James Totter, CEF Concurrent Technologies Corporation 7935 114th Avenue Largo, FL 33773-5026 Phone: (727) 549-7089 FAX: (727) 549-7010 The Environmental Technology Verification for Metal Finishing Pollution Prevention Technologies (ETV-MF) Program is a partnering program under a cooperative agreement between Concurrent Technologies Corporation (*CTC*) and EPA. *CTC* has teamed with CAI Resources, Inc.; CAMP, Inc.; Integrated Technologies, Inc.; and the Michigan Manufacturing Technology Center; they are collectively called the ETV-MF Team.

The purpose of the program is to perform verification testing of environmental technologies intended for reducing pollution in the metal finishing industry. The goal of the ETV-MF Program is to verify the performance characteristics of commercial-ready metal finishing pollution prevention technologies through unbiased third party testing under actual operating conditions in metal finishing shops and facilities nationwide. As defined by EPA, commercial-ready technologies are either in use or ready for full-scale production. This does not include technologies at the bench or pilot scale or those in the research and development stage. The benefits of the ETV-MF Program include:

- Providing high-quality, objective performance data to metal finishers, environmental permitting agencies, and consulting engineers
- Facilitating technology acceptance and expediting permitting at the state and local level
- Reducing risk for financial investors and insurance companies
- Facilitating domestic use and export of American products and their acceptance abroad
- Enhancing the use of pollution prevention technologies

This paper discusses the ETV-MF Program's experience in the verification of alkaline cleaner recycling technologies. As of now, three alkaline cleaner recycling technologies have been verified. The first two technologies are discussed briefly, and the third is discussed in detail. The three technologies are:

- Microbial Digestion
- Microencapsulation
- Cross-Flow Microfiltration

Microbial Digestion

The idea of using microbes to consume oil is not revolutionary. For over 40 years microbes have been utilized to consume oil from oil spills. The cleaning system combines this idea with a cleaner. Most conventional alkaline cleaning solutions would immediately kill the oil-consuming microbes, because of high operating temperatures or high pH. The cleaner chemistry was constructed around the characteristics of the microbe.

The cleaning system employs a mild alkaline bath or spray that operates at relatively low temperatures between 104°F and 131°F (40°C – 55°C) and in a pH range of 8.8 - 9.2, which is a viable habitat for oil-digesting microorganisms. The cleaning solution contains biodegradable compounds (nonylphenol-free) that help to keep the cleaner stable. The cleaning process takes place in two separate operations. When parts come in contact with the solution, the oil and impurities are emulsified into micro-particulates. The particulates then are consumed by microorganisms, which are present in the bath or spray. The microbial consumption of the oil results in the production of CO₂ as a by-product.

The primary equipment component of the cleaning system is the separator module, which is a selfcontained system that provides an environment conducive to microbial growth. Within the separator module, the solution temperature, pH level, and additions of biodegradable compounds are controlled. The cleaning solution is circulated continually between the cleaning tank and the separator module. The separator's automated control system constantly monitors the bath solution and maintains a preset concentration by adding chemical solution as needed.

The performance of the system was fairly similar throughout each test period, ranging from 49% to 64% for oil removal efficiency. However, if the test period had been extended from three to seven days, the oil removal efficiency may have approached 100 percent.

A waste generation analysis was performed on the system tested. Implementation of the system has reduced the disposal frequency of the cleaning process from 64 tank dumps and remakes per year to 20 per year. The overall volume of concentrated waste generated from alkaline cleaning has been reduced by 72 percent.

Operating and maintenance (O&M) labor requirements for the system were monitored during testing. The O&M labor requirement for the equipment was observed to be two hours each week.

A cost analysis of the system was performed using current cost factors and historical records the test site. With the purchase of the system, the test site experienced a payback period of less than a year (i.e., 0.6 yrs).

Microencapsulation

Microencapsulation is a process technology that chemically separates and clarifies the alkaline cleaner solution and encapsulates the waste for disposal. The process utilizes adsorption and electrostatic forces to encapsulate waste products. The chemical compound used in the encapsulating process tested is a non-hazardous proprietary product, which is formulated to treat a range of specific contaminants in the waste stream based on the desired disposition of the effluent; e.g., recycling or discharge to a Publicly Owned Treatment Works (POTW). The microencapsulation unit is equipped with two connecting tanks made of sheet steel: a mixing/reaction tank (upper reservoir tank) and a holding tank (lower reservoir tank). The upper tank is of a trapezoidal design; this is where the untreated alkaline cleaner is pumped and the treatment is added. Once the solution is thoroughly mixed, the encapsulated material is allowed to settle to the bottom of the upper tank. After encapsulation, the treated alkaline cleaner passed through a filtration medium (30-micron filter paper) into the lower tank. As the waste is collected on the filter paper, the paper is slowly pulled forward and wrapped around the encapsulated waste. As the encapsulated waste is rolled in the filter paper, the paper is squeezed to remove excess solution. This process is continued until all of the solution passes through the filter paper into the lower tank. The treated alkaline cleaner in the lower tank is transferred for further treatment or directly back into the parts washer. Further treatment includes a basic ion exchange system and a granular activated carbon filter.

The test results show that the microencapsulation system provides an environmental benefit by reducing the volume of hazardous waste by 93 percent. The treated alkaline cleaner was able to be recycled and reused since contaminants were sufficiently removed; yet the cleaner constituents were not significantly removed. The economic benefit associated with this technology is low O&M labor and a payback period of approximately 2.8 years.

Cross-Flow Microfiltration

The technology verified is a microfiltration system for recycling used alkaline cleaning solutions (cleaners). Alkaline cleaning is performed on metal parts at different times during the manufacturing process to remove oils, coolants and other metalworking fluids prior to electroplating. The verification test evaluated the ability of the technology to remove oils and recover the alkaline cleaning chemistry. It was tested by *CTC* under the U.S. Environmental Protection Agency (EPA) ETV-MF Program. The unit was tested to evaluate and characterize the operation of the microfiltration system through measurement of various process parameters. Testing was conducted at a hydraulic hose manufacturing facility.

A diagram of the unit is shown in **Figure 1**. In operation, the contaminated cleaner enters a twocompartment, 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 product 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. Periodically, the liquid in the recirculation tank is discarded and the tank and ceramic membrane are cleaned.

This technology contains a filtration module consisting of seven α -alumina elements, each with 19 lumens (channels) that are 4.0 mm in diameter. The inside of each channel is lined with an α -alumina or zirconia membrane having a total surface area of 1.68 m² (18.1 ft²) and a pore size of 0.2 μ m.

The unit is equipped with an air-driven, backpulsing device that is designed to clean the filtration modules during operation by periodically pushing solution, under pressure, in a direction opposite to that used for normal filtration. A timer controls the frequency and duration of the backpulse cycle. During each backpulse, a volume of approximately 700 mL of product is pushed through the ceramic membranes in about one-tenth of a second. The unit then returns to its normal flow pattern.

Utility requirements for operation of the equipment include:

- Instrument air: <1 scfm, 80 psi, dry, oil-free
- Electricity: 460 VAC, 60 Hz, three-phase
- Heat (optional): steam, 6 lb/hr or hot water 160°F



Figure 1. Alkaline Cleaner Recycling System

Test Objectives

The following is a summary of verification test objectives. Under normal system operating setpoints for the installation at the test site and varying contaminant-loading rates:

- Prepare a material balance for certain alkaline cleaner constituents and soils in order to:
 - 1. Evaluate the ability of the microfiltration unit to recover alkaline cleaner.
 - 2. Evaluate the ability of the microfiltration unit to remove contaminants from used cleaning solution.
- Determine the cost of operating the alkaline cleaning recovery system for the specific conditions encountered during testing by:
 - 1. Determining labor requirements needed to operate and maintain the microfiltration unit.
 - 2. Determining the quantity of energy consumed by the microfiltration unit during operation.
 - 3. Determining other costs associated with operation of the microfiltration unit.
- Quantify the environmental benefit by performing an analysis of waste generation, which compares the quantity of waste generated before and after the installation of the microfiltration unit.

Testing Procedures

Prior to testing, the recirculation tank was drained and cleaned and the membrane was cleaned according to the manufacturer's instructions [Ref. 1]. The recirculation tank was then filled with spent cleaner solution from the storage tank, and the microfiltration unit was started. Sampling proceeded, once the unit was operating, with a product flow rate of 1.0 gpm. This flow rate is the target operating rate used by the test site.

The microfiltration unit was tested in accordance with the verification test plan [Ref. 1]. 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 the test site. 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.

Test objectives and related test measurements are presented in Table 1.

The alkaline cleaning system is completely drained and recharged with fresh solution approximately every 180 days (two times per year). Runs 1 and 2 were initiated 98 days and 120 days, respectively, after the system was recharged with fresh solution. Therefore, during this project, all testing was conducted during the 3rd quarter of the semi-annual operating cycle.

Test	Test Objectives	<u>Test Measurements</u>
Run 1	Prepare a material balance for	Processing rate of solution through unit.
(baseline):	alkaline cleaner constituents and	Chemical characteristics of feed solution.
Normal soil	soils.	Chemical characteristics of recovered product.
loading rate.		Volume and chemical characteristics of wastes removed from
-		two-compartment tank.
		Quantity of concentrated cleaning solution added during testing.
		Chemical characteristics of concentrated cleaning solution.
	Evaluate the ability of the	Chemical characteristics of feed solution.
	microfiltration unit to process	Chemical characteristics of recovered product.
	used alkaline cleaner solution	Chemical characteristics of the waste products.
	and separate usable cleaner	1 I
	solution chemistry from bath	
	contaminants.	
	Determine the alkaline cleaner	Volume of product produced.
	recovery rate of the system,	Production throughput for all associated cleaning baths.
	normalized based on production	Chemical characteristics of feed solution.
	throughput and soil loading.	Chemical characteristics of recovered product.
	Determine labor requirements	O&M labor required during test period.
	needed to operate and maintain	
	the microfiltration unit.	
	Determine the quantity of energy	Quantity of energy used by pumps and filtration module.
	consumed by the microfiltration	Quantity of energy used to reheat solution after recovery.
	unit during operation.	
	Determine the cost of operating	Costs of O&M labor, materials, and energy required during test
	the alkaline cleaning recovery	period.
	system for the specific	Quantity and price of fresh cleaning chemicals added during
	conditions encountered during	testing.
	testing.	
	Quantify/identify the	Review historical waste disposal records and compare to current
	environmental benefit.	practices.
Run 2: High	Same objectives as Run 1.	Same measurements as Run 1.
soil loading		
rate		
(approximately		
two times the		
normal soil		
loading rate)		

Table 1. Test Objectives and Related Test Measurements Conducted During the Verification of the Microfiltration Unit

Sampling

Prior to the verification test, sampling ports were installed on the feed (IN) and product (EFF) lines of the mcrofiltration unit. Polyethylene tubes were connected to these two sampling ports and directed into 2.5-gallon, high-density polyethylene (HDPE) containers. During sampling, the sample collection containers were kept cool by placing them in a cooler containing ice.

The feed and product samples were collected in the HDPE containers at a continuous, uniform rate of approximately five to eight mL/min (controlled by valve on sample ports) for 24-hour. At the end of each 24-hour sampling period, the HDPE containers were labeled and stored in a cooler containing ice, awaiting shipment to the analytical laboratory.

A grab sample from the recirculation tank was collected at the end of each test run. A sample of the proprietary cleaner (CLEAN-R-120GR) was collected from its original shipping container. These samples were labeled and stored prior to shipment in a cooler containing ice.

Samples shipped to the analytical laboratory were packed in coolers containing "blue ice." A two-day express service was used. All shipments were accompanied with chain of custody forms.

Samples were analyzed using the methods found in **Table 2**.

Analyte	Units	Method Number	Method Reference	Method Detection Limit
Alkalinity	mg/L	2320B	SM	1.0
(as CaCO3)				
Carbonate-Alkalinity	mg/L	2320B	SM	1.0
(as CaCO3)	-			
Bicarbonate-Alkalinity (as CaCO3)	mg/L	2320B	SM	1.0
Hydroxide Alkalinity	mg/L	2320B	SM	1.0
Ammonia Nitrogen	mg/L	350.1	EPA	0.005
Total Nitrogen (TKN)	mg/L	351.3	EPA	0.25
Total Phenol	mg/L	420.2	EPA	0.005
Total Suspended Solids	mg/L	160.2	EPA	1.0
Total Solids	mg/L	160.3	EPA	1.0
Phosphate (as P)	mg/L	200.7	EPA	0.01
Dipropylene Glycol Ether	mg/L	GC/FID (see Appendix E)	N/A	1.0
Oil	mg/L	8015 modified	SW-846	0.2

Table 2. Laboratory Methodology Information

Notes for Table 2: SM = Standard Methods for the Examination of Water and Wastewater, 18^{th} ed.; EPA = Methods for Chemical Analysis of Water and Wastes, 1983; GC/FID = gas chromatography/flame ionization detector.

Analytical Results

A complete summary of analytical data is presented in **Table 3**. 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 product. 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 five percent. These values approximate the concentration of these constituents in a freshly formulated alkaline cleaner bath (since the alkaline cleaning solution at the test site is formulated with a five percent solution).

The primary contaminants of the alkaline cleaning solution are total suspended solids and oil. The values for these parameters during Run 1 represent normal production conditions. During Run 2, adding a concentrated, soiled solution to the feed stream intentionally increased the concentration of these. This procedure simulated a higher soil loading condition than during Run 1.

Table 3. Analytical Result

	Total Alkalinity	Carbonate Alkalinity	Bicarbonate Alkalinity	Hydroxide Alkalinity	Dipropylene	Ammonia	Total Nitrogen	Total	Total Suspended	Total		
Sample	(mg/L as	(mg/L as	(mg/L as	(mg/L as	Glycol Ether	Nitrogen	(TKN)	Phenol	Solids	Solids	Phosphate	Oil
Location	CaCO ₃)	CaCO ₃)	CaCO ₃)	CaCO ₃)	(mg/L)	(mg/L)	(mg/L)	(mg/L)	(mg/L)	(mg/L)	(mg/L as P)	(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	6,100	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	9.340	384	147
EFF-RUN	_,	-,_ • •			0,200					,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,		,
1 AVG	2,520	1,300	646	<1	6,240	3.5	7.6	0.4	52	8,700	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	1,000
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

Process Measurements

Certain process measurements were taken on a daily basis during verification testing. These data have been consolidated and are summarized in **Table 4**. Solution temperature measurements were taken using a hand-held digital thermometer. The pump discharge pressure was read from a gauge located on the Microfiltration Unit's manifold piping, and the product pressure was read from a gauge located on the membrane element.

Sample Date	Total Product Volume (gallons)	Temperature, Recirculation Tank (°F)	Temperature, Recovered Product Tank (°F)	Temperature, Soak Clean Tank (°F)	Pump Discharge Pressure (psi)	Product Pressure (psi)
			Run 1	· · · ·		
5-22-00	1,509	118.8	111.8	133.7	52	26
5-23-00	1,496	127.4	115.4	141.0	52	24
5-24-00	1,405	126.5	117.5	139.5	52	24
5-25-00	1,361	123.5	112.5	154.0	52	21
5-26-00	1,352	122.4	113.0	142.2	52	18
Total/Avg.*	7,123	123.7	114.0	142.1	52	23
			Run 2			
6-12-00	1,442	114.8	111.6	146.9	53	26
6-13-00	1,394	131.2	114.2	141.3	52	25
6-14-00	1,383	130.7	112.0	145.8	53	23
6-15-00	1,382	125.3	90.7	142.0	52	20
6-16-00	1,427	119.7	95.2	155.2	52	16
Total/Avg.*	7,028	124.3	104.7	146.2	52	22

Table 4	Summary	of Process	Measurements
	Summar	y 01 1 100033	wicasurements

*Average values for the test run, except for total product volume, which is the cumulative volume for the test run.

The target product flow rate used by the test site is 1.0 gpm. During operation of the unit, operators periodically check the flow rate. If the flow rate drops below 1.0 gpm, the operator opens the product outlet valve until the 1.0 gpm rate is achieved. When such an adjustment is made, a one to three psi drop in the product pressure is observed. During the first test run and the first four days of the second test run, the product outlet valve was never in the fully opened position. Only during the fifth day of the second test run was it necessary to fully open the product valve in order to maintain a 1.0 gpm product flow. Therefore, the unit could have operated at a higher flow rate than 1.0 gpm, except toward the end of the second test run. However, if the unit is operated above 1.0 gpm, the chemical characteristics of the product may be different than those measured during verification testing.

Production Data

The alkaline cleaning system connected to the recovery unit tested during this project includes a soak cleaning tank located on the zinc plating line, plus 12 in-process parts washers. The surface area of parts processed through these units during verification testing is summarized in **Table 5**. The number of loads of parts processed per day through the soak cleaning tank is shown in column two. Columns three and

four indicate the percentage of these loads that were transported by barrel or rack. Columns five and six indicate the surface area of parts processed, respectively, through the soak cleaning tank and small parts washers. Column seven indicates the total surface area of parts processed in the alkaline cleaning system.

Sample Date	Loads/ day	Rack %	Barrel %	Soak Cleaning ft ²	Part Washers ft ²	Total Area ft ²
			R	lun 1		
5/22/00	199	0	100	19,900	1,990	21,890
5/23/00	112	0	100	11,200	1,120	12,320
5/24/00	187	0	100	18,700	1,870	20,570
5/25/00	214	0	100	21,400	2,140	23,540
5/26/00	197	0	100	19,700	1,970	21,670
					Total Run 1	114,786
			R	tun 2		
6/12/00	142	33	67	9,795	980	10,775
6/13/00	205	0	100	20,500	2,050	22,550
6/14/00	219	0	100	21,900	2,190	24,090
6/15/00	155	0	100	15,500	1,550	17,050
6/16/00	156	0	100	15,600	1,560	17,160
					Total Run 2	91,625
					Total Runs 1 and 2	206,411

Table 5. Gates Rubber Company Production (Surface Area of Metal Parts Cleaned)

The surface area of parts processed through the soak cleaning tank was calculated based on the following estimates from the test site

- Average surface area of a loaded barrel is 100 ft²
- Average surface area of a loaded rack is 6 ft²
- Surface area of parts processed through the parts washers is 10 percent of that processed on the zinc plating line

The actual surface area of metal parts processed was 114,786 ft² during Run 1 and 91,625 ft² during Run 2. However, during Run 2, the soil removed from the cleaner during Run 1 was added to the feed solution during Run 2. Therefore, the effective surface area processed during Run 2 is the sum of the surface areas from both test runs (206,411 ft²).

Other Data

Other data collected during the course of the verification test are summarized in Table 6.

Description	Value
Cost of concentrated alkaline cleaning solution	\$7.48 per gallon
Zinc anode use in 1993*	44,800 lb
Zinc anode use in 1999*	56,700 lb
Alkaline cleaner use in 1993*	8,448 gallons
Alkaline cleaner additions during Run 1	19 gallons
Alkaline cleaner additions during Run 2	0 gallons
Electricity cost	0.07 \$/kWh
Natural gas cost	0.35 \$/therm
Labor cost (loaded rate)	\$20.00/hr
Initial cost of Microfiltration Unit	\$36,000 (1999)
Installation cost of Microfiltration Unit	\$7,000 (1999)

 Table 6. Other Data Collected During Verification

*Data from the test site historical records

Evaluation of Results

Comparison of Recovered Product and Unused Five Percent Cleaner

As shown in **Table 3**, there are both similarities and differences between the analytical results of the recovered product (EFF) (combined EFF-Run 1 average and EFF-Run2 average) and the calculated results for the five percent cleaner. Total alkalinity is approximately two times greater in the recovered product than in the fresh five percent solution. The difference in carbonate is even more significant (7.7 times higher in the recovered product). The hydroxide concentration of the five percent cleaner is 320 mg/L, while the hydroxide concentrations of the product samples were consistently below one mg/L. This could be caused by reactions of the solution with atmospheric carbon dioxide, since, as noted earlier, Runs 1 and 2 were initiated 98 days and 120 days, respectively, after the cleaning system was recharged with fresh solution. This may account for the fact that the total solids content of the recovered product is approximately two times greater than the five percent solution. The increased concentration of total solids is probably due to drag-in from parts, ambient dust, and an accumulation of byproducts from breakdown of organic ingredients of the cleaner. There is very little difference between the recovered product and the five percent cleaner with regard to dipropylene glycol ether, a key organic ingredient of the cleaner.

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. The recovery efficiencies for other parameters were calculated using similar equations.

(1)	A_{eff} (%)	= $[(A_{prod} \times Prod_{vol}) / (A_{feed} \times Feed_{vol})] \times 100\%$
where: A_{eff}	= alkal	linity recovery efficiency
A_{prod}	= produ	uct (EFF) stream alkalinity concentration (grams/liter)
$Prod_{vol}$	= produ	uct volume collected during the cycle (liters)
A_{feed}	= feed	(IN) solution alkalinity concentration (grams/liter)
$Feed_{vol}$	= feed	solution volume processed during the cycle (liters)

The calculated results for recovery efficiency are shown in Table 7.

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

 Table 7.
 Cleaner Recovery Efficiency

The average recovery percentages for alkalinity, carbonate, bicarbonate, and dipropylene glycol ether were high (93.9 to 103.5 percent), indicating that, over the short time period of the verification test, there was little or no change in the concentration of these parameters. Recoveries can be greater than 100 percent due to method error.

Contaminant Removal Efficiency

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

(2)
$$O_{eff}$$
 (%) = 100% – [[($O_{prod} x \operatorname{Prod}_{vol}$) / ($O_{feed} x \operatorname{Feed}_{vol}$)] x 100%]
 O_{eff} = oil recovery efficiency
 O_{prod} = product stream oil concentration (grams/liter)
 $\operatorname{Prod}_{vol}$ = product volume collected during the cycle (liters)
 O_{feed} = feed solution oil concentration (grams/liter); and
 $\operatorname{Feed}_{vol}$ = feed solution volume processed during the cycle (liters)

The calculated results are shown in Table 8.

where.

	TSS	Oil
Sample Date	% Removal	% 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 1, the microfiltration unit removed an average of 69.3 percent of the TSS (6.7 lb) and 82.3 percent of the oil (7.2 lb) from the feed solution, producing a product with average concentrations of 52 mg/L TSS and 24 mg/L of oil.

During Run 2, the microfiltration unit removed an average of 94.5 percent of the TSS (24.9 lb) and 97.0 percent of the oil (37.5 lb) from the feed solution, producing a product with average concentrations of 14 mg/L TSS and 18 mg/L of oil.

During Run 1, there was a lower average concentration of TSS in the feed (164 mg/L) than during Run 2 (450 mg/L). This difference is due to the testing procedure, where adding a concentrated soiled solution to the feed stream intentionally increased the concentration of these contaminants. Despite a higher TSS loading during Run 2, the product stream had a lower TSS concentration than in Run 1 (14 mg/L vs. 52 mg/L). The average TSS removal efficiency was 69.3 percent during Run 1 and 94.5 percent during Run 2. In most cases, the removal efficiency is higher when there is a higher concentration difference across the membrane.

A similar but less pronounced pattern was observed for the oil results. The average oil removal efficiencies were 82.3 percent for Run 1 and 97.0 percent for Run 2. The average feed (IN) (IN-Run 1 Average) and product (EFF) concentrations during Run 1 were 147 mg/L and 24 mg/L, respectively. During Run 2, the average feed and product concentrations were 660 mg/L and 18 mg/L, respectively.

Mass Balance

Mass balance calculations were performed to evaluate how effectively the sampling and analytical procedures account for certain key parameters. The equation for mass balance uses the equation for recovery efficiency and adds a term for the quantity of material contained in the recovery tank at the end of the test run (R-1 and R-2). A calculated result of 100 percent indicates that the quantity of a particular parameter found in the feed stream is fully accounted for in the product and waste tank. Mass balance values were calculated for the following parameters: total alkalinity, TSS, total solids, oil, and dipropylene glycol ether. The mass balance equation for alkalinity is shown below. The mass balances for the other constituents were calculated using similar equations.

(3) mass balance (%) =
$$[[(A_{prod} \times Prod_{vol}) + (A_{waste} \times Waste_{vol}) / (A_{feed} \times Feed_{vol})] \times 100\%$$

where:

A _{prod}	= product (EFF) stream alkalinity concentration (grams/liter)
Prod _{vol}	= product volume collected during the cycle (liters)
Awaste	= waste stream (R) alkalinity concentration (grams/liter)
Wastevol	= waste stream volume (liters)
Feed _{vol}	= feed (IN) solution volume processed during the cycle (liters)
A _{feed}	= feed solution alkalinity (grams/liter)

The mass balance results are shown in Table 9.

	Oil	TSS	Total Solids	Total Alkalinity	Dipropylene Glycol	
Test Run	%	%	%	%	Ether %	
Run 1	64.2	117.4	95.6	99.5	102.9	
Run 2	37.3	24.3	108.2	96.4	96.2	

Table 9. Mass Balance Results

The mass balance results for total solids, total alkalinity, and dipropylene glycol ether are close to 100 percent, indicating that quantities of these parameters found in the feed stream (IN) are accounted for in the product (EFF) and waste tank (R) samples. The results for oil and TSS are variable. The TSS value during Run 1 was high (117.4 percent), but within a reasonable percentage error of +/- 25. The low percentage results for oil (Runs 1 and 2) and TSS (Run 2) may be due to the difficulty of collecting a representative sample from the waste tank (R). These samples were grab samples collected at the end of each run. Although the recirculation pump was operating at the time of sampling, complete mixing may not have been achieved. The bottom of the recirculation tank is sloped, and it is suspected that a layer of sludge containing solids and possibly entrained oil accumulated on this sloped surface, and was not within the recirculated solution that was sampled. This situation would have been accentuated during Run 2, where the TSS and oil loading were significantly higher than Run 1. The parameters for which the mass

balance percentages were close to 100 percent were dissolved species and, therefore, would be less affected by the presence of a sludge layer. Recoveries higher than 100 percent are due to inherent method error.

Energy Use

The primary energy requirements for operating the Microfiltration Unit at the test site include electricity for the system pump and steam (from a natural gas fired boiler) for reheating the product solution when it is returned to service. Electricity is also used for instrumentation and compressed air; however, the energy requirements for these are less significant and were not evaluated during this project.

Electricity use was calculated by dividing the horsepower (HP) of the system pump (7.5 HP) by 1.341 HP-hr/kWh. The result is 134.3 kWh/day, based on continuous use of the pump.

The energy required to raise the solution temperature of the product solution was calculated using the following equation:

4) (BTUs/cycle) =
$$\operatorname{Prod}_{\operatorname{vol}} x \ 2.2 \ \frac{lb}{L} x \ \Delta T x \ 1 \ \frac{BTU}{lb^{\circ}F}$$

where:

 $Prod_{vol} = product volume collected during the cycle (liters)$ $\Delta T = average temperature difference between recovered product tank and soak cleaning tank (°F)$

The average volume of solution processed per day during the test period was 1,415 gal/day (5,356 liters/day). The average temperature of the product was 121.2°F and the average temperature of the soak cleaning tank was 144.1°F. Using the above equation, the resultant energy requirement is 271,000 BTUs/day. The amount of natural gas required to generate this quantity of energy is approximately 2.7 therms/day (based on 100,000 BTU/therm).

Operations and Maintenance Labor Analysis

Operations and maintenance (O&M) labor requirements for the recycling system were observed during testing. Prior to each test run, the unit was drained and cleaned. This process took two labor hours¹ to complete and was performed once each week. On a daily basis, operators periodically checked the product flow rate and made adjustments, if necessary. Also, on a daily basis, the operator removed the cover of the unit and observed the feed going into the bag filter. If the solution was overflowing the bag filter, instead of passing through it, the operator removed the bag filter, cleaned it, and returned it to the unit. These daily tasks took approximately 1.75 hours each week (i.e., 15 minutes per day) to perform. Therefore, the total O&M labor associated with the unit at the test site is 3.75 hours/week (50 weeks/year, 187.5 hours/year). No additional O&M tasks were performed during the test period.

Other O&M labor associated with the alkaline cleaning system, which is affected by the installation of the recycling unit, is associated with the disposal and makeup of bath chemistries. This process involves draining the alkaline cleaning tanks, in-process washers and soak cleaning tank) cleaning them, and

¹ The task took 3.0 hours to complete. However, during certain steps, operator labor was not necessary and the operator was free to perform other tasks; the actual labor hours applied to the microfiltration unit were estimated to be 2.0 hours.

refilling them with fresh chemicals. This process takes eight hours to complete. Prior to the installation of the recovery unit, the alkaline cleaning tanks were drained and filled 15 times per year (120 hours/year). After the recovery unit was installed, the frequency was reduced to twice per year (16 hours/year).

Chemical Use Analysis

Prior to the installation of an alkaline cleaner recovery unit, the test site used 8,448 gal/year of their concentrated cleaner, (data from 1993).² Adjusted for changes to production volume, an equivalent quantity for 1999 is 10,729 gal/year.³ During 1999, the test site actually used 5,390 gal/year of the concentrated cleaner product. Therefore, the production-adjusted savings in cleaner use is 5,339 gal/year (10,729 gal/year – 5,390 gal/year).

With the present operating practices at the test site, concentrated cleaner is used when formulating the alkaline cleaning bath two times per year, and it is added as needed to maintain the alkalinity of the alkaline cleaning bath. Alkaline constituents are consumed during the cleaning process, and are also lost from the bath due to drag-out. Concentrated cleaner is also used during the microfiltration unit cleaning cycle. The approximate volumes of cleaner used for these three purposes at the test site in 1999 were:

- Formulating fresh bath: 360 gal/year
- Added as makeup to maintain alkalinity: 4,822 gal/year
- Used to clean the microfiltration unit: 208 gal/year
- Total use in 1999: 5,390 gal.

Concentrated cleaner, peroxide, and hydrochloric acid are used to clean the microfiltration unit and filter module. The annual quantities of these chemicals used in 1999 were:

- Concentrated cleaner: 200 gal/year
- Peroxide: 12.5 gal/year
- Hydrochloric acid: 200 gal/year

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 one 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 microfiltration unit installation could not be quantified during this project. Overall, the bath replacement procedure generated the following wastes:

² In 1994 the test site implemented a polymer membrane, alkaline cleaner recovery unit and subsequently replaced that equipment with the microfiltration unit. Therefore, 1993 is the most recent year that is representative of using the alkaline cleaning system without a recovery unit installed.

³ 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/year and 56,700 lb/year, respectively. Therefore, under the same conditions, if the test site used 8,448 gal of cleaner in 1993, they would be expected to use 10,729 gal in 1999.

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

Waste oil is generated by skimming oil off of waste storage tanks in the waste treatment area. Waste oil was generated in similar quantities before and after the installation of the microfiltration unit. Gates sends about 500 gallons of waste oil off-site every two years. The cost of hauling/disposal is \$1.00/gal. There is no waste reduction or cost savings that have resulted by installation of the microfiltration 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. This procedure generates 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 wastes⁴:

- 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 10.

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

Table 10. Results of Waste Generation Analysis

The quantity of sludge generated from treatment of the liquid wastes could not be quantified during this project because liquid wastes from various sources are combined prior to treatment at the test site. However, the quantity of sludge generated with and without the recovery unit is expected to be in proportion to the total solids content of the wastes generated.

⁴ The microfiltration unit has a drain port located on the upper part of the settling tank that can be used to remove floating oil from that tank. This drain is not used at the test site, and therefore a separate oil waste is not generated during the recovery process.

Cost Analysis

The capital cost of the microfiltration 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 11**. The operating costs of the microfiltration unit are \$50,049. The operating costs prior to installation of the microfiltration unit were \$82,653, resulting in annual net savings of \$32,604. The simple payback period is 1.3 years (capital cost/net annual savings).

Waste treatment and sludge disposal costs could not be quantified and are not included in the above analysis.

	Prior to Installation of Microfiltration			After Installation of Microfiltration		
Item	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	20.00	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 units	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

Table 11. Annual Costs/Savings

References

All references are available by accessing the EPA's ETV Program Internet website at www.epa.gov/etv.

- 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 Metal Finishing Technologies Quality Management Plan." December 9, 1998.