Alkaline Cleaner Recycling Using Microbial Digestion
An Environmental Technology Verification-Metal Finishing (ETV-MF) Pollution Prevention Pilot Test Report

James Totter, Concurrent Technologies Corporation (CTC), Largo, Florida
Gus Eskamani, Ph.D., CAMP, Inc., Cleveland, Ohio
Karrie Jethrow, CAMP, Inc., Cleveland, Ohio

The ETV-MF Program, in association with EPA's Common Sense Initiative for the Metal Finishing Sector, is a pilot for verifying 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 is conducted under strict EPA quality guidelines in metal finishing shops, where possible, under actual operating conditions.

This paper will discuss the verification testing of a cleaner recycling technology that uses microbial digestion to remove organic soils from the bath. The test methods, data analysis, and conclusions will be presented, including the environmental and economic benefits of this technology. The presentation will conclude with an update of the EPA ETV-MF program and the status of other verification test projects.

For more information contact:

James Totter
Concurrent Technologies Corporation
7990 114th Avenue
Largo, FL 33773-5026
Phone: (727) 549-7089
FAX: (727) 549-7010

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 testing of commercial-ready, private sector technologies through 12 centers that cover a range of industry sectors and environmental areas. For one of these 12 centers, EPA has partnered with Concurrent Technologies Corporation (*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 an alkaline cleaning system that uses microbial digestion to remove oils from the alkaline cleaner and recycles the cleaner back to the cleaning tank. The cleaning system was tested to evaluate and characterize the operation of the system through measurement of various process parameters. Testing was conducted at The National Manufacturing Company according to a verification test plan prepared by the ETV-MF Center [Ref 1]. National Manufacturing has two facilities that utilize this system: Rock Falls, IL (704,000 ft^2), and Sterling, IL (550,000 ft^2). The Sterling facility was chosen for the verification test because it employs a Module I, which is the larger of the two units and also more automated. The cleaning system is being used to: (a) consume oil, coolants, and other metal working fluids that are removed from metal parts during the cleaning process, and (b) recycle the cleaner back to the cleaning tank.

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 report [Ref. 2].

TECHNOLOGY DESCRIPTION

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 104ºF and 131ºF (40ºC – 55ºC) 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 CO_2 as a by-product.

The primary equipment component of the cleaning system is the separator module, which is a self-contained system that provides an environment conducive to microbial growth (**Figure 1**). 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.

---

[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.

Figure 1. Separator Module

The chemical solutions include the cleaner, booster, and pH+/pH- buffer solutions. The cleaner is used to break the bond between the part and the oil and then forms a molecule around the oil particle. The booster is a surfactant that aids the cleaning process. The pH-/pH+ solutions are used to maintain the cleaning solution's pH, as well as supply nutrients for the microbes. The pH- contains phosphoric acid and nutrients for the microbes. The pH+ contains sodium hydroxide and nutrients for the microbes. The microbes ingest the oil first, but if the oil concentration in the cleaning solution is low, the microbes eat the nutrients in the buffer solutions as a supplementary food source.

The separator control system also uses a blower to aerate the solution to provide oxygen, which is needed by aerobic microorganisms. The microbial population is naturally occurring, and its living habitation is maintained in the separator. The microbes also are self-controlling. In theory, as the volume of oil increases, the organisms should multiply in direct proportion.

VERIFICATION TESTING

The test strategy, as outlined in the verification test plan [Ref. 1], was to evaluate the cleaning system performance at three different oil loads. This was accomplished as follows:

- To evaluate the effectiveness of the cleaning system at a high oil loading (HOL), three plating lines were monitored and sampled during an eight-hour shift over a three-day period. The plating processes at the Sterling facility were allowed to run normally during the HOL test, with three cleaning baths being fed into the cleaning system. A daily average part production of 750,000 pieces was run through the plating system. The system was monitored for eight hours every day over a three-day period.

- To evaluate the effectiveness of the cleaning system at a low oil loading (LOL), one plating line was monitored and sampled during an eight-hour shift over a three-day period. The plating processes also were allowed to run normally during the LOL test, with only one cleaning bath (Bath #3) being fed into the cleaning system. A daily average part production of 107,500 pieces was run through the plating system. The system was monitored for eight hours each day over a three-day period.
- To understand how the microbes react with a known increase in oil concentration over time, one cleaning bath was spiked with a known amount of oil, and was monitored and sampled during an eight-hour shift over a three-day period. This condition is referred to as spiked oil loading (SOL). The SOL test was conducted with no parts running through the zinc barrel Plating Line #3, and with its cleaning bath being fed into the cleaning system. The oil was introduced into the system in a short time frame through three aliquot additions. The system was spiked during the first hour of SOL Test Day #1. A total of 9,600 g of oil were added to Cleaning Bath #3. The system was monitored for eight hours each day over a three-day period. The other cleaning baths were isolated from the cleaning system during the SOL test.

Air sampling was performed at various points around the cleaning system and away from the system to determine bacteria and fungi concentrations. In the cleaning system bacteria digests the oil. However, conditions exist in the separator that can foster fungal growth. Therefore, samples for fungi were also collected and analyzed.

Samples were collected for chemical and microbial analysis at AMTest Laboratories and U.S. Micro-Solutions, respectively. Sample collection and analysis were performed according to the procedures outlined in the verification test plan [Ref 1]. The analytical methods used for analyzing the chemical samples are standard EPA methods and standard microbiological methods for analysis of the microorganisms.

Results

Oil Removal Efficiency

The goal of this project was to verify performance, and this can generally be measured in terms of the efficiency of the system in removing oil from the alkaline cleaner. The oil removal efficiency equation for the cleaning system is shown below.

$$\text{Oil Removal Efficiency (\%)} = \frac{A - \left| \Sigma V_i X_{i,s} - \Sigma V_i X_{i,f} \right|}{A} \times 100\% \tag{1}$$

where:

where:	V_i	=	Volume (l),
	I=1	=	Volume of Cleaning Bath #1
	I=2	=	Volume of Cleaning Bath #2
	I=3	=	Volume of Cleaning Bath #3
	I=4	=	Volume of Separator System
	$X_{i,s}$	=	Starting oil concentration at point I
	$X_{i,f}$	=	Final oil concentration at point I
	A	=	Mass of at test end

The calculated oil removal efficiencies for the system verification test during the three different oil loading rates (high, low and spiked oil loads) are shown in **Table 1**.

The oil concentrations at the beginning and end of each test run were multiplied by the sampling point specific volumes to determine the initial and final mass of oil within the system. The “oil added” refers to the oil coming into the system on the metal parts for HOL and LOL tests, and the oil that was added to the system in its neat form for the SOL test.

The oil removal efficiencies were calculated based on mass balances of the system. These calculations were performed for each oil load test.

For the HOL test, **Table 1** shows that 49% of the oil introduced into the system was consumed by the microbe population during the three days of sampling. The system during the HOL test consisted of the separator and holding tank, Cleaning Baths #1 – #3, and associated piping. The table includes each of the components that make up the system and their respective volumes, starting oil concentration, and the oil concentration at the end of the test.

For the LOL test, **Table 1** shows that 64% of the oil introduced into the system was consumed by the microbe population during the three days of sampling. The system during the LOL test consisted of the separator and holding tank, Cleaning Bath #3, and associated piping.

For the SOL test, **Table 1** shows that 51% of the oil introduced into the system was consumed by the microbe population during the three days of sampling. The system during the SOL test consisted of the separator and holding tank, Cleaning Bath #3, and associated piping.

Overall, the mass balance shows that the bacteria are consuming the oils being introduced into the system on the parts. An important observation is that sampling occurred on Tuesday through Thursday during the HOL and LOL test phases. Given that National Manufacturing does not dump and replace the cleaner baths, we can surmise that the microbial consumption of oil in the cleaner continues throughout the weekend, when no additional oil is being introduced. If the test period had been extended to include weekends, the calculated oil removal efficiency may have approached 100%.

Table 1. Oil Removal Efficiency

			Oil Concentration		*Oil Mass*				
	Location	**Volume (l)**	**Initial (g/l)**	**Final (g/l)**	**Initial (g)**	**Final (g)**	**Oil Added (g)**	**Oil Consumed by Microbes, (g)**	**Efficiency (%)**
High Oil Load	**Bath #1**	**2,080**	**3.9**	**12.0**	**8,100**	**25,000**			
	Bath #2	**3,410**	**4.8**	**16.0**	**16,400**	**54,600**			
	Bath #3	**2,080**	**6.1**	**29.0**	**12,700**	**60,300**			
	Separator	**2,840**	**5.9**	**5.9**	**16,800**	**16,800**			
	Total				**54,000**	**156,700**	**201,600**	**98,900**	**49**

			Oil Concentration		*Oil Mass*				
	Location	**Volume (l)**	**Initial (g/l)**	**Final (g/l)**	**Initial (g)**	**Final (g)**	**Oil Added (g)**	**Oil Consumed by Microbes, (g)**	**Efficiency (%)**
Low Oil Load	**Bath #3**	**2,080**	**7.0**	**17.0**	**14,600**	**35,400**			
	Separator	**2,840**	**4.2**	**8.6**	**11,900**	**24,400**			
	Total				**26,500**	**59,800**	**93,100**	**59,800**	**64**

			Oil Concentration		*Oil Mass*				
	Location	**Volume (l)**	**Initial (g/l)**	**Final (g/l)**	**Initial (g)**	**Final (g)**	**Oil Added (g)**	**Oil Consumed by Microbes, (g)**	**Efficiency (%)**
Spiked Oil Load	**Bath #3**	**2,080**	**10.0**	**15.0**	**20,800**	**31,200**			
	Separator	**2,840**	**18.0**	**16.0**	**51,100**	**45,400**			
	Total				**71,900**	**76,600**	**9,600**	**4,900**	**51**

Microbial Assessment

It was important to quantify the biological populations (bacteria and fungi) at selected locations within the cleaning system in order to determine their response to oil loading and the potential health and safety risk associated with the aerosolized microbes in various stages of the cleaning process. Air samples were collected and sent to a microbiological laboratory to quantify and identify the species of bacteria and fungi that were present. Aqueous samples from the cleaning system were sampled and analyzed to verify the relationship between the oil and microbial concentration. Sample results were reported in units of colony forming units per milliliter (CFU/ml).

Aqueous Samples

The following graphs show the bacteria and oil concentrations at the separator effluent for samples taken during the three test runs. Note that the bacteria concentrations are total bacteria in solution. No attempt was made to determine which particular species found were involved in oil digestion.

Figure 2 shows the bacterial concentration versus oil concentration at the separator effluent during the high oil load testing. This graph shows that it took several days for the bacteria population to grow in response to a relatively constant oil load. It is useful to note that this high oil load test most closely replicated the normal operating system used at the Sterling facility of National Manufacturing because three out of the four plating lines and their associated alkaline cleaning tanks were connected to the system during this test.

Figure 3 shows the separator effluent bacteria concentration versus oil concentration for the low oil load testing. The drop in bacteria concentration on day 2 cannot be explained by the available data. As shown in **Figure 2**, an increase in bacteria population lags an increase in oil concentration. The drop in bacteria concentration, during the low oil load, may be due to some stress applied to the bacteria population several days prior to the start of sampling. The bacteria concentration does show signs of recovery after this drop on the third day of sampling. While the bacteria population dropped during the low oil load test, the oil removal efficiency was the highest obtained (64%). This indicates that while the bacterial population may fluctuate, there are still enough bacteria to effectively digest the oil.

.

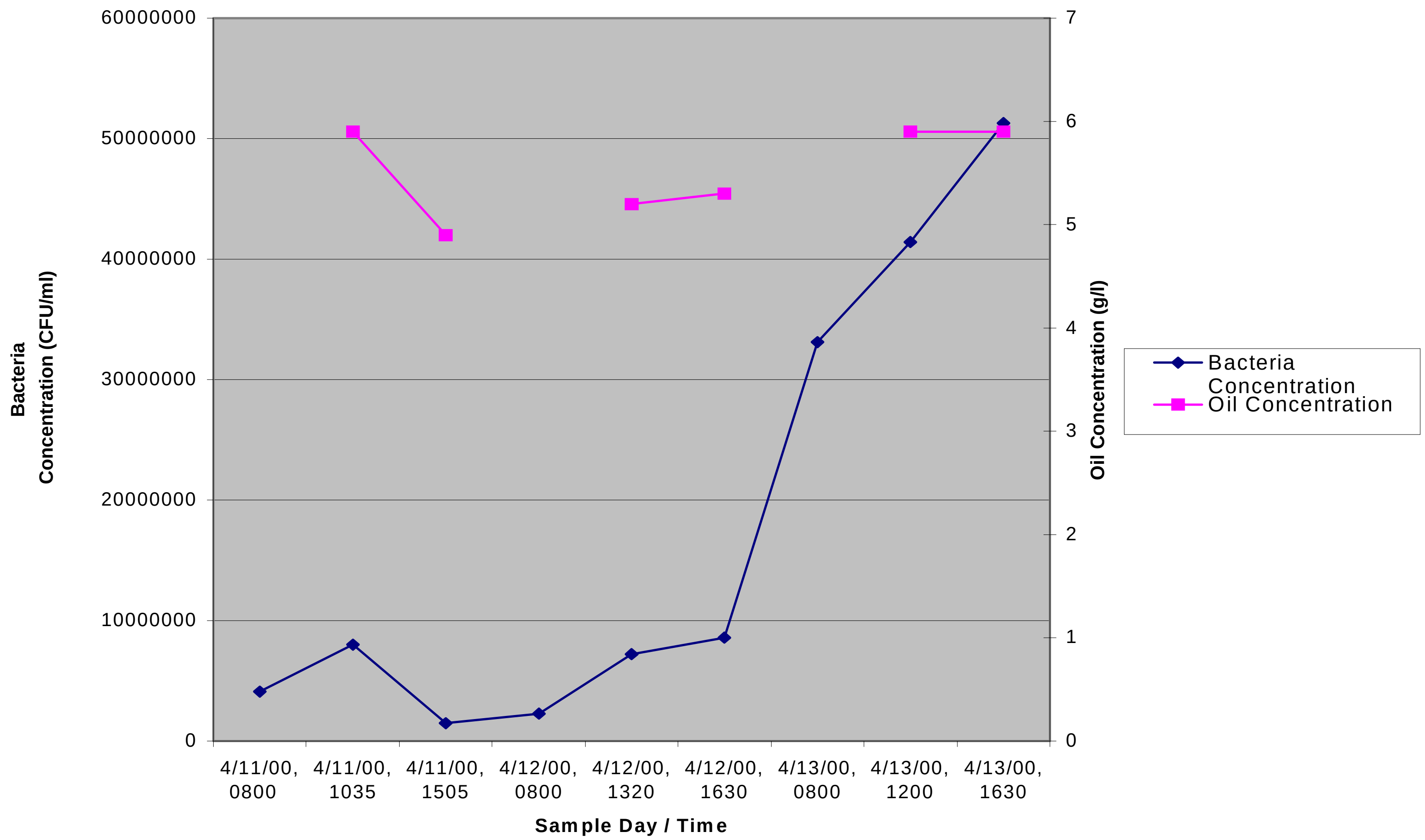

Figure 2: High Oil Load Separator Effluent Data

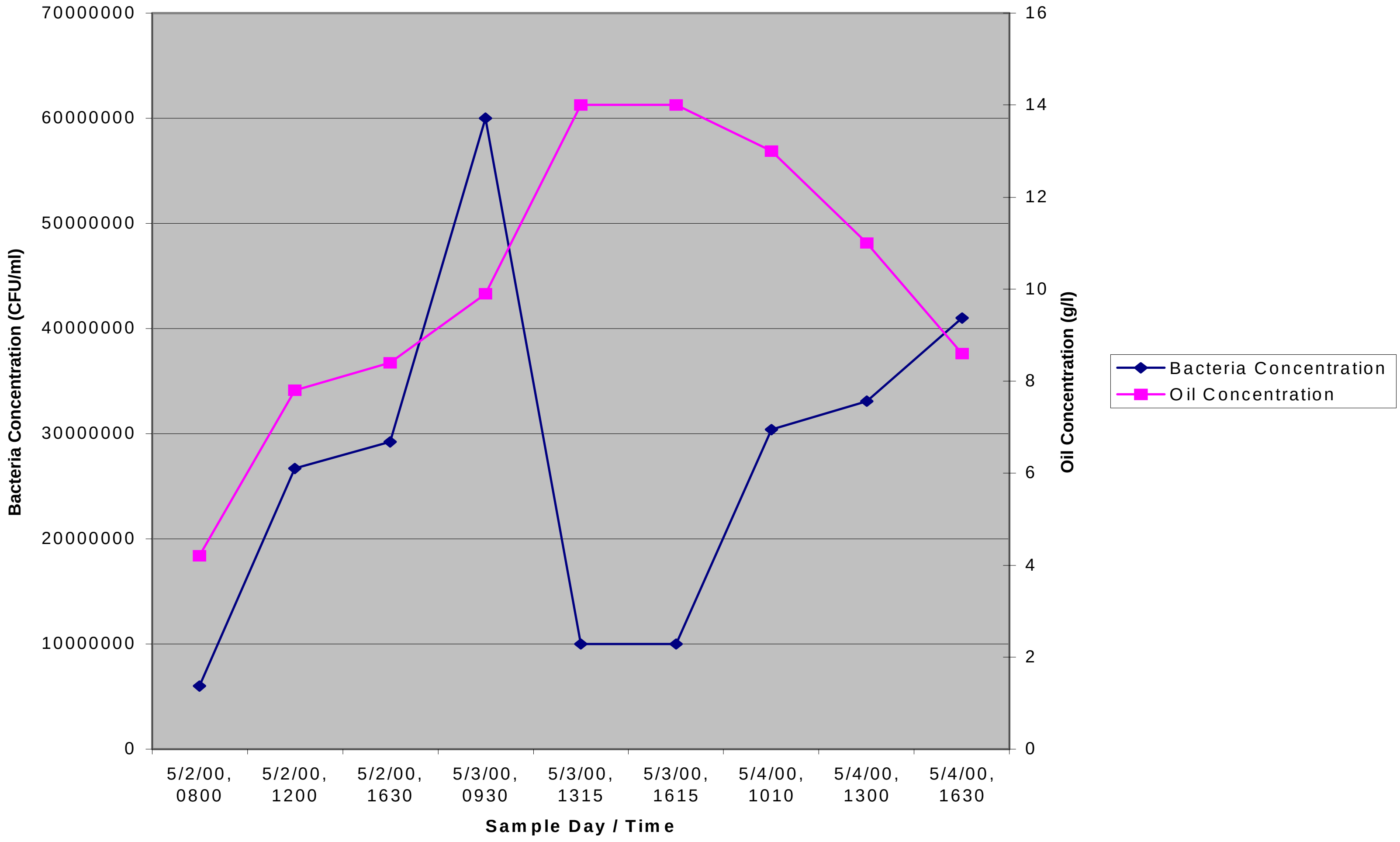

Figure 3: Low Oil Load Separator Effluent Data

Figure 4 shows the bacteria concentration versus oil concentration during the spiked oil load testing. Note the change in scale for the bacteria concentration (CFU/ml). The radical change in bacteria concentration (approximately two orders of magnitude lower than during the HOL and LOL) may be due to a stress occurring prior to the start of this test. Recovery in response to the oil load seems to be starting by the end of sampling. Note also that the oil for this phase of testing was added as a spike of neat oil, and some degree of emulsification could be required prior consumption by the bacteria.

In summary, the data throughout the experiment indicate that a majority of the digestion of oil occurs in the separator; however, some oil digestion does occur throughout the entire system. There is some delay between a change in oil concentration and a response by the bacteria population. The changes in bacteria population are driven by the available food supply, i.e., the oil concentration. Over a period where oil is not being introduced to the cleaner tanks (a weekend, for example), the bacteria continue to digest the oil until the supply is exhausted. As the supply of oil is depleted, the bacteria concentration can no longer be supported by the available food. The bacteria concentration then decreases. Once production resumes, the concentration of oil increases, but the bacteria concentration does not immediately increase. One reason for this is that the oil must first be emulsified. The emulsification increases the surface area of the oil in solution, making it more readily available to the bacteria. Second, the emulsified oil must be dispersed throughout the cleaning system, making the oil available to more bacteria. Once the oil is emulsified and dispersed, the bacteria population is seen to react to the increased oil concentration.

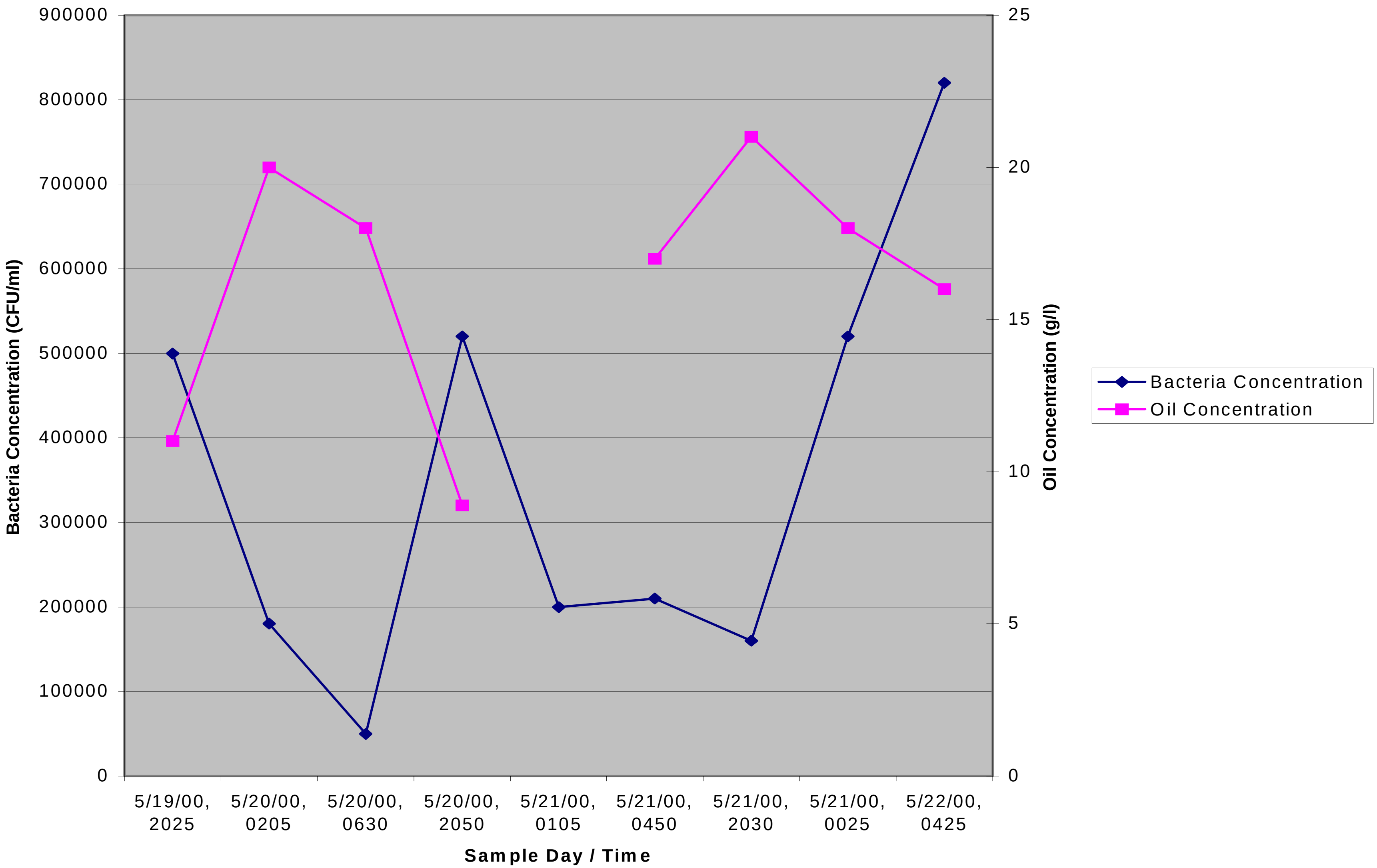

Figure 4: Spiked Oil Load Separator Effluent Data

Air Samples

The cleaning system is designed to provide an environment in which oil-digesting bacteria and other microorganisms thrive. The National Institute for Occupational Safety and Health has found that 5% of indoor air quality problems can be traced to microbial contamination [Ref. 3]. Microbial contamination can cause allergic reactions and infections. The U.S. Occupational Safety and Health Administration (OSHA) has issued guidance [Ref. 4] that concentrations of 1,000 colony forming units per cubic meter (CFU/cubic meter) of air may be an indicator of contamination. However, levels in excess of this amount do not necessarily imply that the conditions are unsafe or hazardous. The type and concentrations of the airborne microorganisms will determine the hazard.

Bacteria

During the high oil load, air samples were taken from the areas near cleaning baths 1, 2, and 3; the separator; and the holding tank. A sample of outside air measured 18 bacteria CFU/cubic meter. Results of the high oil load air samples are summarized in **Table 2**:

Table 2. HOL Bacteria Air Sampling Results

Sampling Point	Average Concentration (CFU/cubic meter)
Cleaning Tank 1	378
Cleaning Tank 2	477
Cleaning Tank 3	165
Separator	4,558
Holding Tank	140

Only the samples from air exiting the separator exceed 1,000 CFU/cubic meter indicating a potential for contamination. An evaluation may need to be conducted to determine appropriate personal protective equipment when performing maintenance activities if necessary inside the separator tank. Outside air samples during the LOL test averaged 700 CFU/cubic meter.

The samples were characterized also for the type of bacteria present, although no attempt was made to quantify the concentrations of any individual species. *Bacillus spp*, *Micrococcus spp*, *Corynebacterium spp*, and *Micrococcus luteus* were most often identified. These species are gram positive bacteria usually isolated from a variety of environmental sources. Although usually considered harmless, they may become a source of infection in immuno-compromised individuals [Ref. 5].

Fungi

During the high oil load testing, only one air sample (1,572 CFU/cubic meter) taken in the vicinity of cleaning tank 2 exceeded the OSHA indicative value of 1,000 CFU/cubic meter. The average results of testing are shown in **Table 3**:

Table 3. HOL Fungi Air Sampling Results

Sampling Point	Average Concentration (CFU/cubic meter)
Cleaning Tank 1	254
Cleaning Tank 2	842
Cleaning Tank 3	359
Separator	240
Holding Tank	319
Outside Air	9

During the low oil load testing, samples from outside air and the holding tank exceeded 1000 CFU/cubic meter. The average results are presented in **Table 4**:

Table 4. LOL Fungi Air Sampling Results

Sampling Point	Average Concentration (CFU/cubic meter)
Cleaning Tank 1	646
Cleaning Tank 2	650
Cleaning Tank 3	461
Separator	695
Holding Tank	1263
Outside Air	1360

The samples were characterized for the type of fungi present. The most common species found were *Cladosporium spp*, *Penicillium spp*, *Alternaria spp*, *Fusarium spp*, and *Epicoccum spp*.

Economic Evaluation

An economic evaluation was prepared comparing National Manufacturing's previous method of cleaning bath treatment and disposal against their current method, using the alkaline cleaner recycling system.

Chemical Costs

Prior to installing the cleaning system, National Manufacturing used an aqueous soak cleaner, which operated at 140-145°F, followed by an electrocleaner, on all four plating lines. These baths were maintained by analysis, and the baths were dumped and remade eight times per year. The associated raw chemical costs for the soak clean/electroclean system are presented below:

	Soak Cleaner	Electrocleaner	Total Annual Cleaner Costs
Annual make-up costs:	$12,460	$10,496	$22,956.00
Annual replenishment costs:	$52,470	$34,876	$87,346.00
Subtotals	$65,110	$45,372	$110,482.00

After installing the system the cleaner dump and remake frequency was set at once per year, and the electroclean bath dump frequency was reduced to four times a year with the following associated chemical costs:

	Cleaner	Electrocleaner
Annual make-up costs:	$6,687	$5,284
Annual replenishment costs:	$5,657	$25,584
Total	$12,344	$30,832
Total Annual Cleaner Costs		$43,176

The annual savings in direct cleaner costs is $67,306.

Energy Costs

Because the cleaner is maintained at 120-125°F, as opposed to the previous soak clean temperature of 140-145°F, there is a savings in the utility costs of the preplate cleaning cycle. The heating costs were calculated using the formulae found in the *Metal Finishing Guidebook and Directory* [Ref. 6] chapter on immersion heaters. System auxiliary equipment includes pumps and heaters for the separator and holding tank.

	Microbial Cleaner (kWh)	Soak Cleaner (kWh)
Heat Required for Startup	12,300	17,200 (4-hr cycle, 50 cycle/yr)
Heat Required for Surface Loss	35,900	88,100
Heat Required for Tank Wall Loss	13,500	20,300
Aux. Equipment.	34,100	0
Total	95,800	125,600
Savings	29,800	
Savings (at $.07/kWh)	$2,086/year	

Waste Disposal

Seven gallons of bottoms, with the following average composition, were collected from the separator during each test period.

Oil	20.6 g/L
Total Solids	130 g/l
Total Suspended Solids	117 g/l
Total Organic Carbon	43.4 g/l
Zinc	10.5 g/l
Copper	0.11 g/l

The amounts and concentrations of these materials are negligible with respect to the system mass and energy calculations.

The separator bottoms were disposed of in the verification test site's on-site waste treatment facility, and can be assumed to be negligible in terms of the total annual waste generation there. Waste disposal costs prior to the system installation for the combination soak clean/electroclean system from historical records were $8,800 per year, as compared to $4,000 per year for the microbial system/electroclean system, which corresponds to a savings of $4,800/year.

Labor

Daily preventative maintenance labor observed during testing included checking the function of the air blower, circulation of the cleaning baths through the separator, function of the metering pumps, chemical level in the replenishment pumps, pH value, and temperature value. Weekly maintenance tasks included checking the level probes, cleaning and calibration of the separator pH probe, and removing the sludge from the bottom of the separator. These tasks required a total of two labor-hours per week.

Regardless of tank size or content, a bath change in the preplate cleaning process requires eight labor-hours. Prior to the cleaning system installation, eight cleaning baths were changed eight times annually. The annual labor hours required to change the baths were:

2)

8 baths x 8 changes/year/bath x 8 hours/change = 512 labor-hours.

The microbial system, with its requirement for cleaning rather than changing, requires the following annual labor hours:

3)

4 microbial cleaner baths x 1 cleaning/year/bath x 8 hours/cleaning +
4 electroclean baths x 4 changes/year/bath x 8 hours/change = 160 labor-hours.

To the system labor requirements, the additional preventative maintenance burden of 104 man-hours/year must be added (2 labor-hour/week x 52 weeks/year). The total preventative maintenance burden for the microbial system, therefore, is 104 + 160 = 264 labor-hours/year.

National Manufacturing assumes labor costs (with burden) to be $25/labor-hour, so the total annual labor savings is:

4)

(512 - 264 labor-hours/year) x $25/labor-hour = $6,200/year.

Summary

Total savings seen in the operation of the system annually are:

	Savings
Chemical Usage	$67,306
Energy	$2,806
Waste Disposal	$4,800
Labor	$6,200
Total	$81,112/year

The installed cost of the system at National Manufacturing was $47,569; the simple return on the investment (payback) was 0.6 years.

CONCLUSIONS

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%.

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

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 from National Manufacturing. With the purchase of the system, National Manufacturing experienced a payback in less than a year (i.e., 0.6 yrs).

REFERENCES

Note: References 1 and 2 are available by accessing the ETV-MF Program Internet website at: www.etv-mf.org.

1. Concurrent Technologies Corporation, "*Environmental Technology Verification Program for Metal Finishing Pollution Prevention Technologies, Verification Test Plan, Evaluation of BioClean™ USA, LLC Biological Degreasing System for the Recycling of Alkaline Cleaners*", February 4, 2000.
2. Concurrent Technologies Corporation, "*Environmental Technology Verification Program, Metal Finishing Technologies Quality Management Plan*", December 9, 1998
3. National Institute for Occupational Safety and Health, "*Guidance for Indoor Air Quality Investigations*", 1987.
4. Occupational Safety and Health Administration (OSHA).*OSHA Technical Manual, Section 3, Chapter 2: Indoor Air Quality Investigation*, 1999.
5. Murray, P. R., E. J. Baron, M. A. Pfaller, F. C. Tenover, and R. H. Yolken. *Manual of Clinical Microbiology, 6th Edition, American Society for Microbiology*, Washington, D.C., 1995.
6. Richards, Tom, "*Immersion Heating*", Metal Finishing, Vol. 98, No. 1, pages 755 – 766, 2000.