OILY WASTEWATER TREATMENT BY ULTRAFILTRATION: PILOT-SCALE RESULTS AND FULL-SCALE DESIGN

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ABSTRACT: A West Virginia company that produces an oily waste decided to upgrade their wastewater treatment facilities to prevent possible ground and surface water contamination. The contaminant of concern was oil/grease. West Virginia University's Department of Civil and Environmental Engineering was asked to perform pilot-scale studies using the following treatment technologies: chemical-addition-dissolved air flotation, ultrafiltration, biological filter, constructed wetlands, and land application. Based on data collected over a 2-year period, the following treatment train was proposed: settling ponds → oil/water coalescer → tubular ultrafiltration → constructed wetlands. The ultrafiltration effluent had an oil/grease content that was consistently below 100 mg/L and very low total suspended solids. Ultrafiltration effluent will be sent to a 15-acre hybrid constmcted wetlands/land application system for tertiary treatment. The ultrafiltration residual (2,600 gal/day, >5% oil) will be concentrated by a factor of 10 (260 gal/day, >50% oil) using a high-shear rotary ultrafiltration system. The highly concentrated oil waste will be disposed of via an off-site oil recycler.

INTRODUCTION

In August 1993, a West Virginia company that produces a waste mixture of coolants and lubricants decided to upgrade their industrial wastewater treatment facilities to prevent possible ground and surface water contamination. Until this time, the company collected a mixture of coolants and lubricants from their milling operations and mixed them with more dilute waste in two lined ponds having a combined capacity of about 5 million gallons. A significant amount of the lubricant oils separated and were skimmed from the ponds. The resulting pond effluent, which contained approximately 0.2–0.5% oil/grease (O/G) and had a daily flow of 80,000 gallons, was then sprayed on a 36 acre site, where the remaining O/G was degraded naturally. In 1993, the West Virginia Department of Environmental Protection issued new ground water regulations that strongly discouraged direct land application of industrial wastes and, shortly thereafter, the company began to investigate alternative treatment methods. West Virginia University's Department of Civil and Environmental Engineering (WVU-CEE) was asked to perform pilot-scale studies for the company involving the following treatment technologies: chemical-addition-dissolved air flotation (CA-DAF), tubular ultrafiltration (UF), biological aerated filter, constructed wetlands, and land application. The primary pollutant of concern from a regulatory standpoint was (O/G).

CA-DAF

CA-DAF is a two-step process. In the first step, a chemical(s) is added to the system to break the oil emulsion and alter the surface chemistry of the particles so that smaller particles can agglomerate into larger particles. In the flotation chamber, particles are physically separated from the liquid using buoyant forces. Very fine air bubbles are introduced into the wastewater and are attached to the particle surface. The particle-air bubble system will rise to the surface where it can be efficiently removed using a skimming apparatus.

Experimental Design

In this study, chemicals from two companies were investigated. Calgon Corp. supplied a cationic polymer (W-2923) to break the emulsion and an anionic polymer (POL-E-Z 2706) to enhance coagulation. KLAR-AID 2400, a cationic polymer, was supplied by Grace Dearborn (GD). Jar tests were used to determine the required chemical dosage prior to CA-DAF unit operation (Zhu et al. 1997). In Fig. 1, a schematic of the CA-DAF system is presented. The cationic polymers were added directly to the chemical mix tank (CMT). The anionic polymer (Calgon only) was added at the transfer pump inlet and the turbulence from the pump provided the mixing energy. The CA-DAF unit was operated in either the semibatch or continuous mode. In the semibatch mode, the CMT was filled with wastewater and the appropriate amount of cationic polymer (determined by jar tests, see Zhu et al. 1997) and mixed for about 20 min. The contents of the CMT were then transferred to the DAF unit (Calgon anionic polymer added continuously). During the continuous operation the CMT was constantly being emptied/filled and all chemical additions were continuous. The turbidity of the DAF effluent were measured hourly. If the DAF effluent turbidity was greater than 30 nephelometric turbidity units (NTU), a jar test was conducted to determine if a change in chemical dosage was warranted. Influent and effluent O/G were measured periodically. The flow rate was 8.9 gal/min, recycle ratio = 0.7, and air pressure was 42 psi. The detention times of the CMT and DAF unit were 30 and 67 min, respectively.

Results

DAF effluent turbidity and O/G for a typical Calgon run are presented in Fig. 2. Turbiditys were highly variable and exceeded the maximum measurable value (200 NTU) on numerous occasions. When the DAF effluent turbidity exceeded 30 NTU a "quick" jar test was performed to determine if the chemical dosage required adjustment. A total of 16 of these tests were conducted during the 2/13–2/19 run. In six of the jar tests a chemical adjustment was required. The new chemical dosage is indicated in Fig. 2 by the vertically oriented numbers

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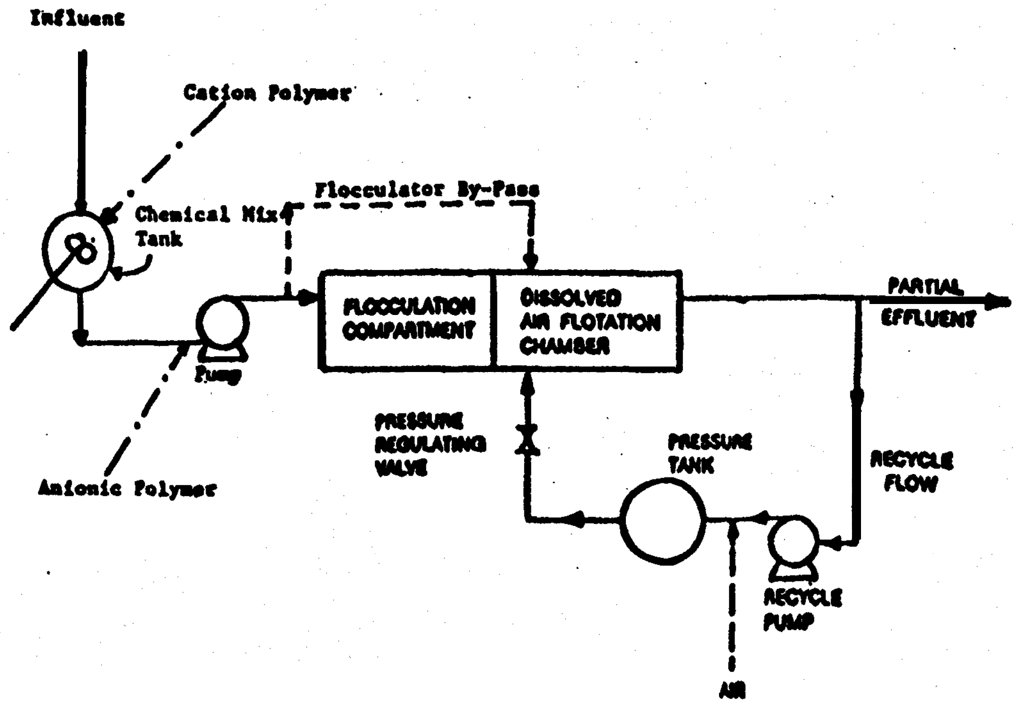


FIG. 1. Schematic of CA-OAF System

(e.g., 600/50 ≡ cationic polymer dose/anionic polymer dose). The vertical arrows with no number designation refer to the quick jar tests in which a change in chemical dosage was not warranted. It would typically take 2-3 h before the change in chemical dosage would take effect. Influent O/G ranged from 1,490 to 3,830 mg/L, with an average value of 3,130. Effluent O/G ranged from 11 mg/L to 278 mg/L (60 ± 80) in the semibatch mode and from 8 to 327 mg/L (205 ± 135) in the continuous mode. During the semibatch operation, between about 15 and 50 h, the DAF O/G ranged from 12 to 29 mg/L and averaged about 19 mg/L. Despite numerous chemical dosage adjustments, this was the only period of time where consistent and relatively low values of O/G were obtained

DAF effluent turbidity and O/G for a typical GD run are presented in Fig. 3. Turbiditys were lower and less variable compared with results from Calgon. For the DAF effluent, turbiditys were generally less than 20, except for a few spikes. The most noticeable turbidity spike occurred at about 70 h. At this time there was a power outage and the chemical *addition* pump failed. The units were idle for about 12 hours and the turbiditys were high following the restartup of the operation, but decreased to less than 30 NTU for the remainder of the run. Turbiditys were slightly higher during continuous operation. Only two jar tests were conducted during the GD run, and for both tests a change in chemical dosage was not warranted. Influent O/G ranged from 2,360 to 3,240 mg/L and averaged 2,950 ± 260 mg/L. Effluent O/G ranged from 15 mg/L to 37 mg/L (28 ± 8) in the semibatch mode and from 9 to 41 mg/L (30 ± 9) in the continuous mode.

Summery

In Table 1 a summary of CA-DAF results are presented. O/G concentrations were significantly better and frequent jar testing was not required for the GD chemical. However, depending on the mode of operation, there was between two and four times more sludge produced for the GD chemical compared with the Calgon chemicals. Assuming a sludge disposal cost of $0.26/gal. (supplied by company), the disposal cost per 1,000 gallons of wastewater treated ranged from $1.77 to $1.90 for the Calgon chemicals and $3.46 to $5.98 for the GD chemical.

UF

UF is a pressure driven membrane technique that uses porous membranes for the separation of material in the 1 nm–10 μm size range or compounds with molecular weights in excess of 5,000. Membranes that are used for UF are characterized by the molecular weight of a compound that is not able to pass through the membrane [molecular weight cutoff (MWCO)]. In theory, a significant number (90%) of compounds having a molecular weight greater than the MWCO would be retained by the membrane. Membranes having a surface charge equal to the charge of the contaminant can be used The repulsive force between the membrane surface and contaminant decreases membrane fouling. In this study, the pilot-scale system was operated first in the semibatch and then in the batch mode. In the semibatch mode, the permeate and raw waste flow rates were equalized by maintaining a constant volume in the feed tank. In the batch mode, no raw waste is added to the system and the concentrate remaining from semibatch operation is "batchdowned." Flow based concentration factors (CF) are calculated as follows:

Semibatch operation:

$$CF_{SB} = 1 + V_{perm}/V_{feed\ tank}$$

Batchdown operation:

$$CF_{BD} = CF_{SB} \times [V_{feed\ tank}/(V_{feed\ tank} - V_{perm})]$$

where CF_{SB} = CF during semibatch operation; V_{perm} = volume of permeate produced (gal); $V_{feed\ tank}$ = volume of the feed tank (gal); and CF_{BD} = CF during batch-down operation. CFs are expressed as 1 X, 2X, etc., and increase with treatment time.

Experimental Design

UF experiments were conducted during the summer (eight runs) and winter (four runs) months. Two membrane types, denoted "M" and "P", were investigated (Reed et al. 1997a). The M membrane had a MWCO of 100,006 and no net surface charge, whereas the P membrane had a MWCO of 120,000 and a net negative surface charge. Both membranes were constructed of polyvinylidane fluoride. Sixteen membrane tubes,

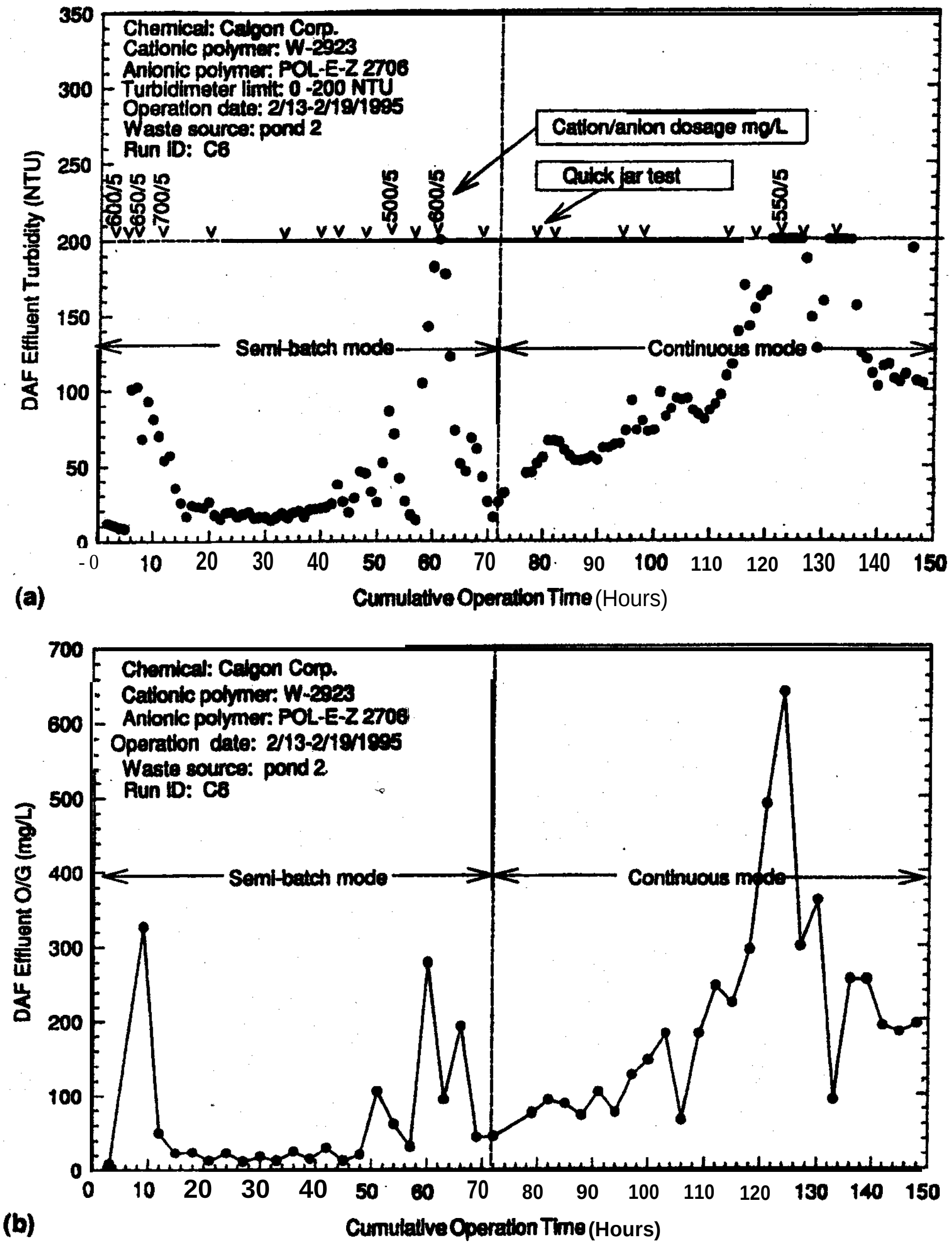
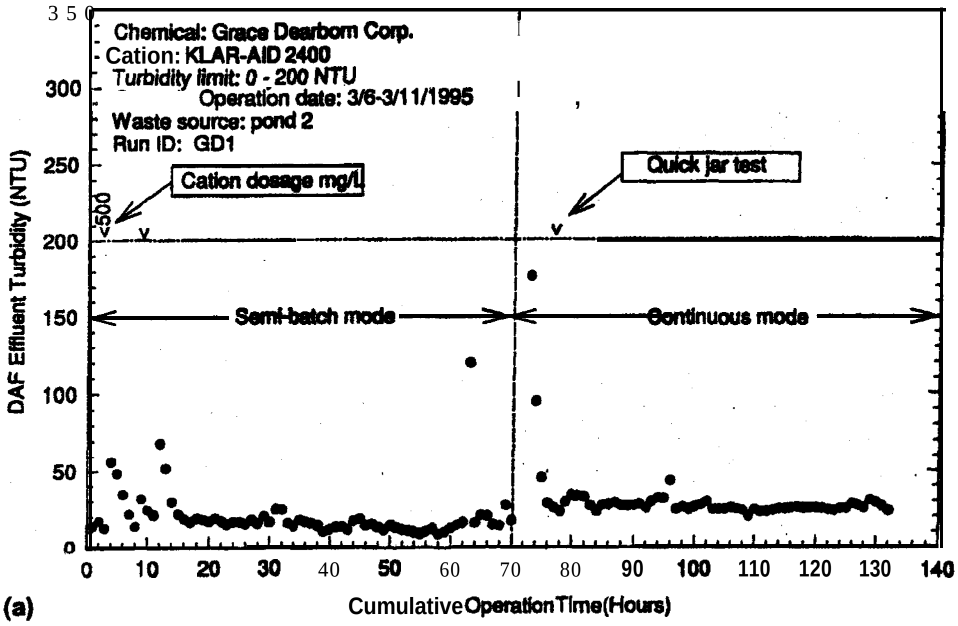


FIG. 2. DAF Effluent Turbidity (a) and O/G (b) for Typical Calgon Run

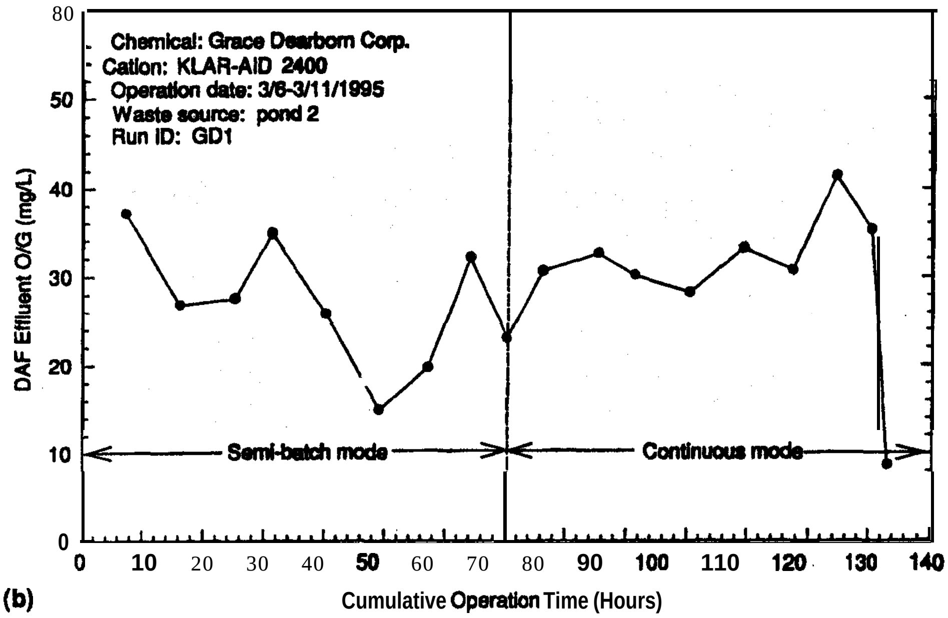
eight for each membrane type, were housed on the UP unit. The average transmembrane pressure was held constant at 42 psi. Samples of influent and UF concentrate and permeate were taken periodically and analyzed for turbidity, O/G, total suspended solids (TSS), and pH. Permeate flux was monitored using a graduated cylinder and stopwatch. At the conclusion of batch operation, membranes were cleaned for a total of 60 min with a proprietary cleaning solution.

Results

In Fig. 4, the permeate flux and concentration factor versus time during semibatch operation for summer run 1 (S1) and for winter run 3 (W3) are presented. The horizontal line represents the average permeate flux over the duration of the run The permeate flux versus time for the other UF runs were similar in shape except for summer run 4, which had to be ended early because of low fluxes for both membranes. For all runs, the permeate flux decreased dramatically during the first several hours of operation and then leveled off for the remainder of the semibatch operation. The decrease in flux with time can be attributed to concentration polarization. In Table 2, a summary of UF data is presented. Run S4 was omitted because the flux decreased to almost zero after only 2 h of operation. In a full-scale operation, waste that is processed at such a low flux would be returned to the facility's separation ponds for a longer opportunity to separate and homogenize. The overall average P membrane flux was significantly higher than the M membrane flux [38 versus 27 gal/ft^2 per day (gpd)], as well as the flux observed during both summer and winter operations. On an individual run basis, the P membrane flux was greater than or equal to the M membrane flux for all runs except W3 and W4. The higher flux for the P membrane can be attributed to its higher MWCO (120,000 compared with 100,000) and negative surface charge. The larger the MWCO the higher the membrane's permeability and the repulsion of the negatively charged O/G droplets by the P membrane surface decreased the fouling layer thickness. The average temperature of the UF influent and concentrate was about 10 degrees lower in the winter than in the summer. A portion of the difference between summer and winter fluxes is due to viscosity differences. Influent O/G ranged from 920 to 5,600 mg/L and averaged 2,460, whereas the TSS ranged from



(a)



(b)

FIG. 3. DAF Effluent Turbidity (a) and O/G (b) for Typical GD Run

TABLE 1. Summary of DAF Unit Operation Results for Calgon and GD Chemicals

	Effluent O/G (mg/L)		Sludge Produced (gal./1,000)		Chemical Costs ($US/1,000 gal.)		Sludge Disposal ($0.26/gal.)	
Chemicals (1)	Semibatch (2)	Continuous (3)	Semibatch (4)	Continuous (5)	Semibatch (6)	Continuous (7)	Semibatch (8)	Continuous (9)
Calgon	60 ± 80	205 ± 135	7.3	6.8	4.54	4.05	1.90	3.46
GD	28 ± 8	30 ± 9	13.3	23.0	10.80	10.80	3.46	5.98

150 to 2,100 mg/L and averaged 645. Waste variability over the course of entire project as well as within a given run was large and demonstrates the complex nature of the settling pond waste.

Although there were some effluent samples with high O/G and TSS concentrations, the vast majority of the samples had O/G concentrations of less than 50 mg/L and TSS levels of less than 25 mg/L. O/G removal efficiencies averaged 98% for the M membrane and 97% for the P membrane. TSS rejections were approximately 97% for both membranes. For all runs, the effluent O/G concentration and turbidity from the P membrane were higher than the M membrane. Effluent TSS was greater for the P membrane for all runs except W3, and S7 TSS for run S7 were almost equal. Given the larger MWCO *of the* P membrane (120,000 compared with 100,000) the effluent should be better for the M membrane. Effluent turbiditys were measured for all samples in which O/G analyses were performed to determine if there was a relationship between the two parameters. It was hoped that turbidity could be used as a real-time indicator of effluent quality, specifically O/G concentration. However, there was not a statistically significant relationship between O/G and turbidity.

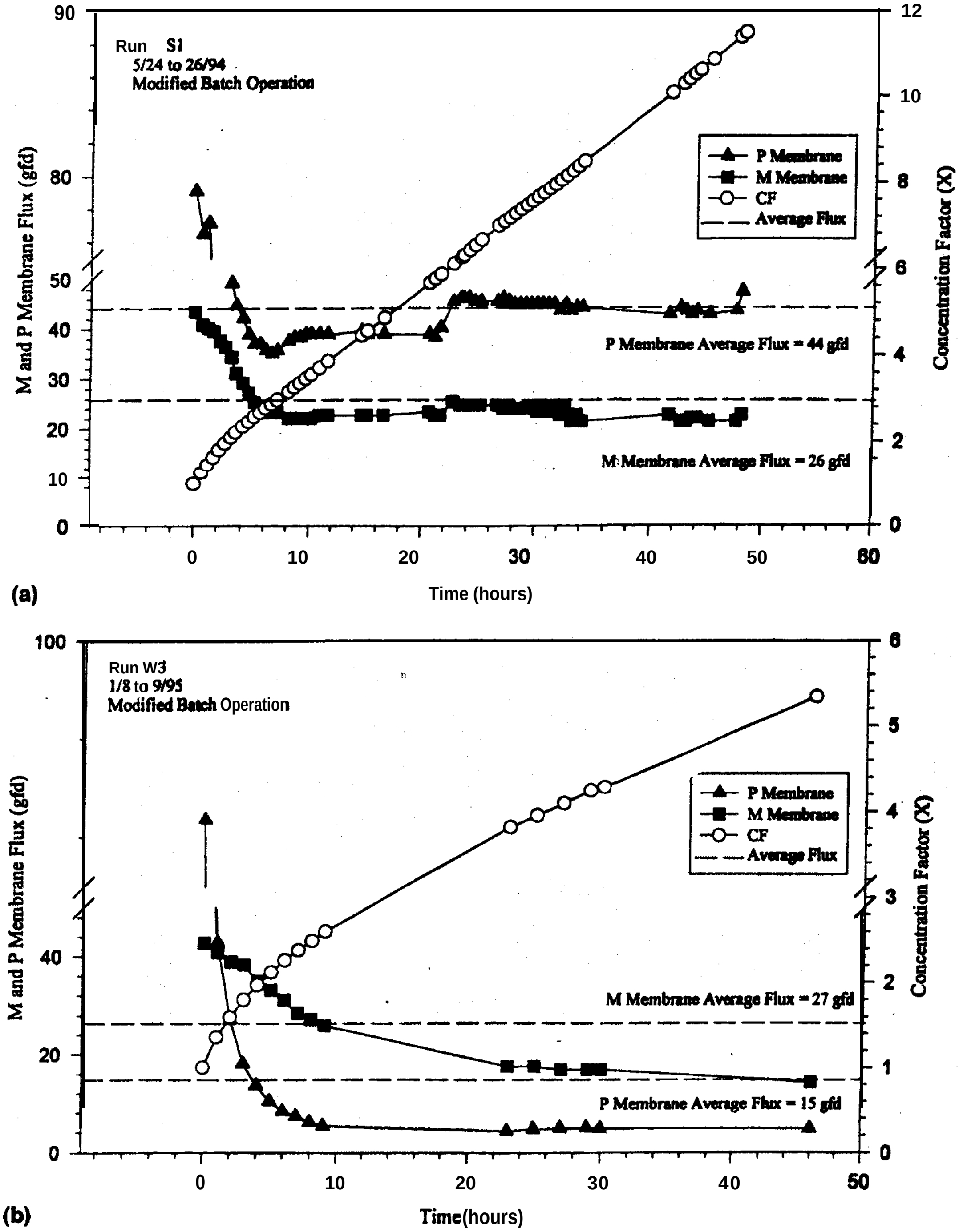


FIG. 4. Permeate Flux and CF for Typical Summer (a) and Winter (b) Run8

Batchdown was not conducted for runs S4, S5, S7, W1 and W2. For runs S7 and W2, the feed tank overflowed because of a faulty solenoid valve. High temperatures prevented batchdown during run S5, whereas low fluxes at the end of the semibatch portion of runs S4 and W1 necessitated membrane cleaning. If batchdown was not possible in the full-scale operation, the concentrate at the end of semibatch operation would be sent back into the separation ponds to allow for free oil separation and for a more homogenous mixture of wastes to be introduced to the UF unit. CF between 16 and 116X and volume reductions between approximately 94 and 99% were observed at the end of batchdown. Residual production ranged from 10 to 58 gal./1,000 gal. of wastewater treated. Batchdown for run S3 ended prematurely because of high temperatures producing the lowest CF and the highest residual production (16X and 58 gal/1,000 gal.). The average volume reduction and residual production for the entire project were 97% and 32 gal./1,000 gal., respectively. During batchdown for runs S7, S8, W1, W3, and W4, free oil appeared in the feed tank. The free oil was periodically removed and later analyzed for water content. Free oil production ranged from 2.5 to 7.3 gal./1,000 gal. of wastewater treated. All samples had a trace amount of water associated with the free oil (<5%). The facility in question conducted bumability tests and reported that the free oil was suitable for burning in on-site boiiers.

A preliminary design was conducted for the P membrane using winter conditions. The P membrane was chosen because during winter and summer testing the P membrane flux was consistently higher than that of the M membrane, whereas the effluent quality for the P membrane was not significantly dif-

TABLE 2. Summary of UF Results

Run number (1)	Average Flux (gal./ft² per day) M (2)	P (3)	Average O/G (mg/L) M (4)	P (5)	Average TSS (mg/L) M (6)	P (7)	CF (8)	Volume reduction (%) (9)	Residuals (gal./1,000) (10)
S1	26	43	39	45	NA	NA	27X	95.6	44
S2	30	63	9	11	NA	NA	23X	95.6	44
S3	58	58	9	15	10	11	16X	94.2	58
S5	16	25	245	470	39	43	—		—
S6	17	20	25	55	NA	NA	60X	96.6	30
S7	26	31	23	30	6.3	6.0	—	—	—
S8	32	69	14	15	NA	NA	116X	98.3.	18
W1	25	29	26	34	15	19	—	—	—
w 2	22	47	40	33	7 5	5.5	—	—	—
w 3	26	15	a7	115	93	43	42x	97.8	23
w 4	17	16	55	82	14	18	104x	99	10
Average summer	29	44	40	67	18	20	—	—	—
Average winter	22	26	52	66	32	21	—	—	—
Overall average	27	38	46	66	20	20	—	—	—

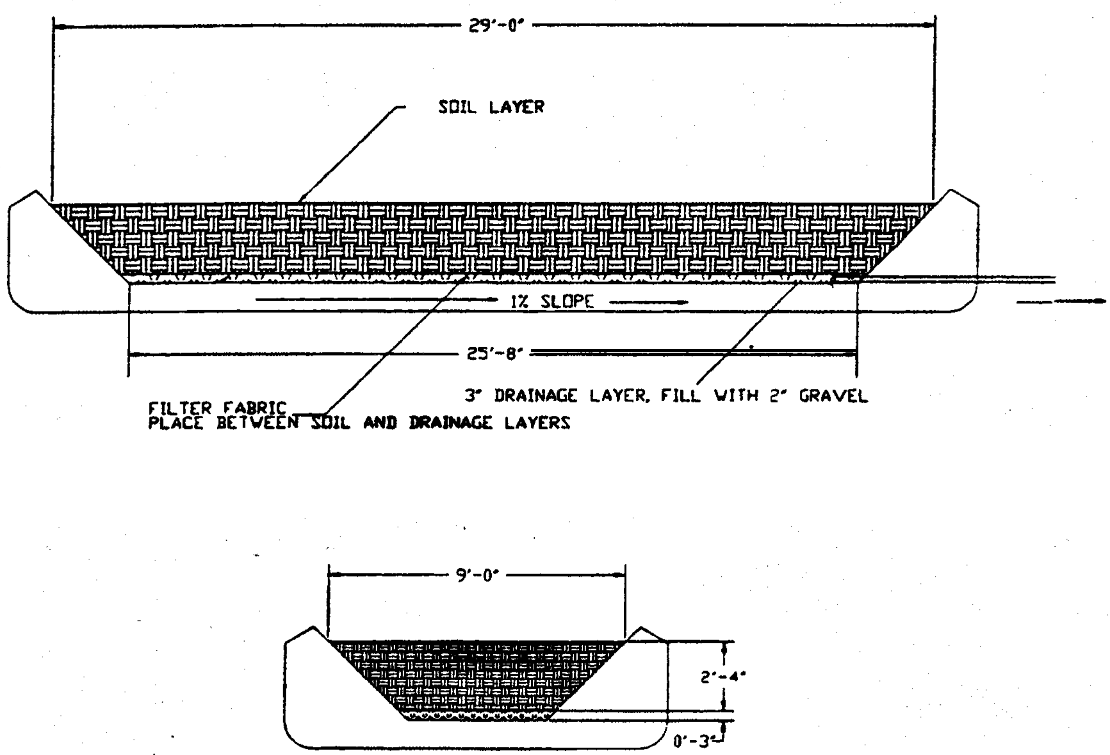


FIG. 5. Schematic of Land Application Cell (Adapted from Cooper and Hobson 1989)

ferent than that observed for the M membrane. The design flow rate is 80,000 gpd. The required membrane area for winter conditions, assuming a 5% expansion capability, is 3,240 ft². During summer months, the system will be able to process more waste than the daily flow, which is attractive for the facility because the settling ponds can be drawn down creating additional storage space for periods of inclement weather.

BIOLOGICAL AERATED FILTER

The pilot-scale biological aerated filter consisted of four cylindrical reactors operated in series. Each reactor was 6 ft high and 2.5 ft in diameter. The particle size of the media ranged from 1/4 inch to 1 inch, and was tapered because of the high solids loading. Air was applied through medium coarse diffusers at the bottom of each reactor. A nutrient solution, consisting of ammonium sulfate, nonbasic sodium phosphate, and dibasic potassium phosphate, was injected into the first reactor. The influent flow rate was varied from 0.5 to 3 gal./min. The hydraulic loading ranged from 294 to 880 gpd/ft², whereas the recirculation ratio ranged from 0 to 3. The system was back; washed based on head loss during the first 6 months of operation, whereas a time schedule was developed for the latter part of the study. The wastewater parameters chosen to evaluate the system's performance were O/G and TSS. These parameters were sampled on a daily basis because of the high variability of the waste stream. Effluent-dissolved oxygen, pH, and water temperature were measured during each sampling event. Other analytical testing included chemical oxygen demand, potassium, phosphate, and the various forms of nitrogen. Many difficulties were encountered during the operation of the system mostly due to high solids and O/G loading, insufficient water velocity during backwash events, and low dissolved oxygen. Modifications were made to the system increasing system performance. Removal efficiencies decreased with increasing hydraulic loading, whereas recirculation adversely affected system performance. Frequent clogging of the filter was problematic and the inability to effectively backwash the filter led to its abandonment as a treatment option.

LAND APPLICATION

A schematic of land treatment cell is presented in Fig. 5. To evaluate the effect of vegetation, two land treatment cells were constructed. One cell was vegetated with a mixture of tall fescue and orchard grass, chosen for their ability to evapotranspirate and hardiness. The seed was allowed to germinate and develop root and leaf structure prior to any wastewater application. This cell was referred to as the V cell. The other cell remained nonvegetated and was referred to as the NV cell. Each cell was 9 ft wide, 29 ft long, and contained soil to a depth of 20 in. Beneath this soil was a geotextile filter fabric and 3 in. of river gravel. The entire system was bounded on the bottom and sides by a 30-mil polyvinyl chloride (PVC) liner. The effluent from the system was collected in the river gravel bed and transmitted to two 250-gal. conical bottom tanks. The cells were open to the atmosphere and subject to atmospheric changes. During spraying, a 0.5-A buffer zone around the perimeter of the cells was maintained to minimize short circuiting by the PVC liner. Each week, 0.5 in. of wastewater was evenly applied over the sprayed area. The wastewater application was followed by a 6-day resting period to restore aerobic conditions. This cycle continued for 1 full year. Influent sampling occurred on the day of application. Effluent sampling occurred 1 day after a spraying and/or significant rain event, and continued on a daily or every 2-day cycle. in Fig. 6, influent and effluent O/G concentrations are presented for both cells. O/G removal was greater than 97% over the length of the study. There was little difference in performance between the vegetated and nonvegetated cells other than an increase in water loss due to evapotranspiration in the vegetated cells. In a full-scale system, vegetation would be included because of the increase in evapotranspiration and operational considerations (minimizing erosion, wind-blown soil, runoff, etc.).

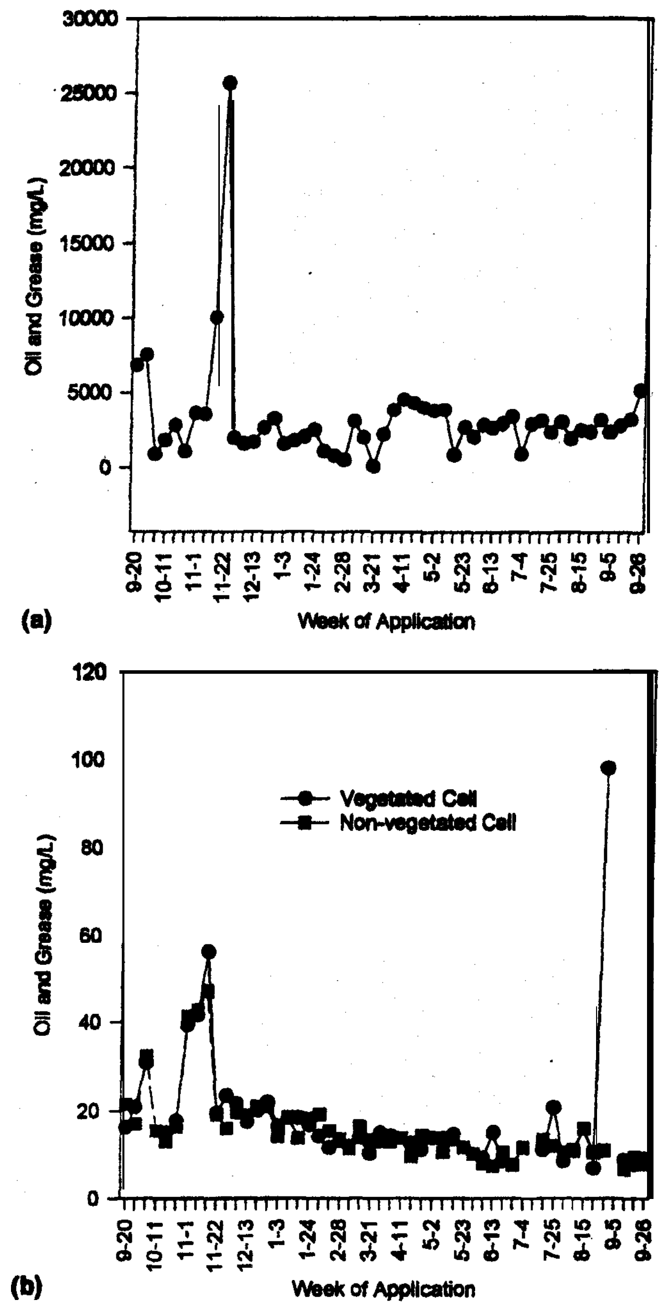


FIG. 6. Influent (a) and Effluent (b) O/G Concentration from Land Application Cells

CONSTRUCTED WETLANDS

A schematic of a constructed wetland cell is presented in Fig. 7. In this study, two wetlands series were constructed with two cells in series (cell 1 → cell 2; cell 3 → cell 4). Each cell was 14 ft wide and 26 ft long and had an average depth of 20 in. Cells 1 and 4 had cattails planted aud cells 2 and 3 had bulrush. The ability of the constructed wetlands to polish the effluent from either the CA-DAF or UF system (secondary treatment systems) was evaluated using pond effluent that was diluted with tap water. O/G, total organic carbon, chemical oxygen demand, biochemical oxygen demand, TSS, and dissolved oxygen were measured along the length of each cell; however, only O/G results will be presented here. In Table 3, O/G concentration for constructed wetland series 1 (ceils 1 and 2) are presented. Results for series 2 (cells 3 and 4) were similar. Initially, O/G removal was not effective, but as the vegetation matured O/G removal increased. There was also a noticeable increase in O/G during the colder months. Overall O/G removal efficiency was about 94% with the lowest efficiencies (~88%) occurring in the winter months.

RESIDUAL TREATMENT

Oil skimmings from primary treatment (settling ponds) were disposed of through an oil recycler. Residuals from tertiary treatment (constructed wetlands/land application) consist only of vegetative harvests. Thus, the primary residual requiring treatment was from the secondary treatment system (either CA-DAF or UF). Acid cracking of CA-DAF and UF residuals was not effective because the amount of acid required was similar to the amount of effluent produced. A high-shear rotary UF membrane system was tested to determine the efficacy of concentrating secondary treatment residuals. The high-shear rotary UF system is described in greater detail in Reed et al. (1997b). In Fig. 8, the permeate flux versus concentration factor is presented for a 5% oil waste. Five percent oil represents a typical oil content from secondary treatment. The high-shear rotary UF system was very effective in concentrating the oily waste high oil contents. Concentrate from the rotary UF system will be disposed of off-site while the permeate from the system will be sent back to the head of the treatment system.

PRELIMINARY DESIGN

The existing settling ponds will be used for storage and to remove free oil. Effluent from the ponds will then pass through an oil-water separator prior to entering the UF system. A UF

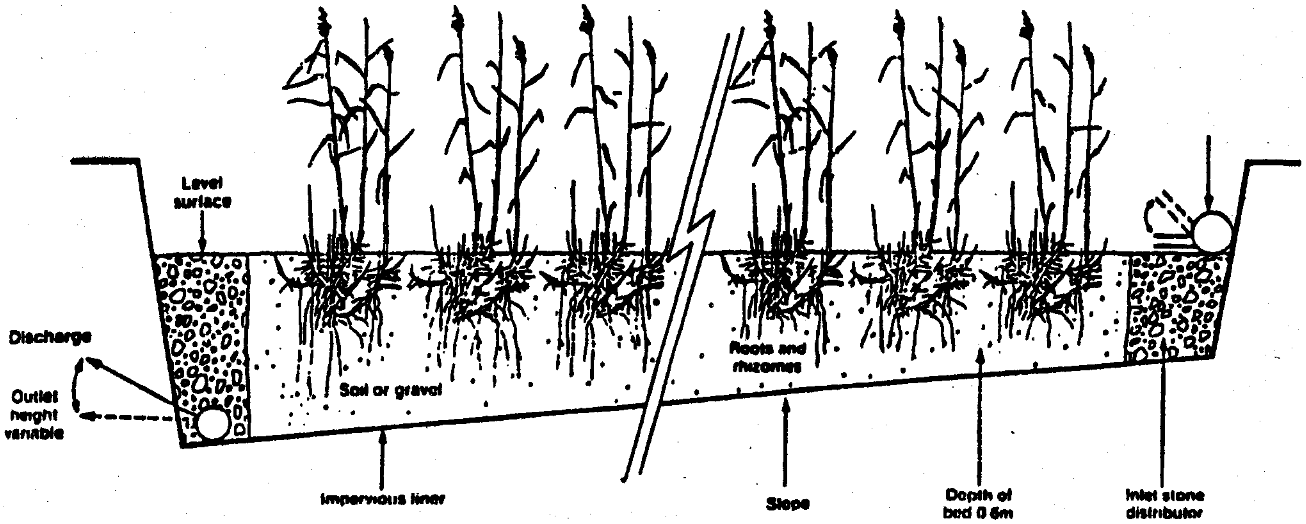


FIG. 7. **Schematic** of **Constructed** Wetland

TABLE 3. O/G Concentrations for **Constructed** Wetland **Series** 1: Cells 1 and 2

Month (1)	Cell 1 Influe			Cell 1 Effluent			Cell 2 Effluent		
	Average (2)	Minimum (3)	Maximum (4)	Average (5)	Minimum (6)	Maximum (7)	Average (8)	Minimum (9)	Maximum (10)
May	209	47	319	56	36	89	59	42	88
June	231	26	633	34	11	68	20	S	39
July	298	63	672	16	2	44	8	4	16
August	162	90	407	12	3	30	5	1	14
September	60	25	109	8	1	26	3	<1	8
October	98	17	223	9	3	15	6	1	17
November	233	69	342	23	5	53	19	9	45
December	158	44	264	20	11	36	20	10	35
January	131	43	216	10	<1	15	6	<1	13
February	185	11	618	9	1	23	8	4	10

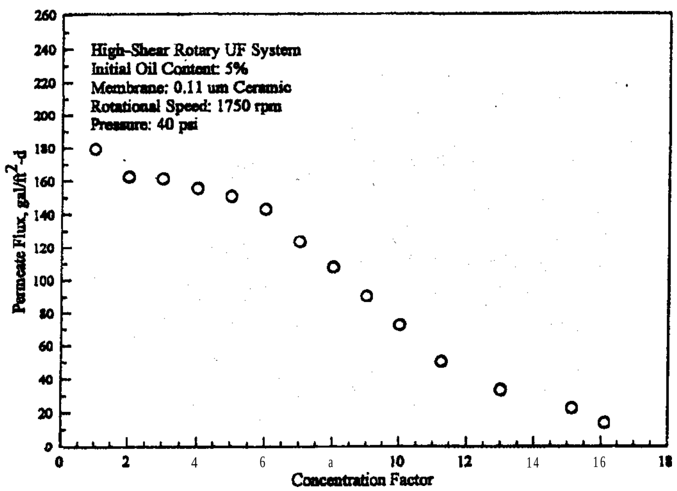


FIG. 8. Permeate Flux versus Concentration Factor for 5% Oil Using High-Shear Rotary UF System

tubular membrane **system** having membranes with a **MWCO** = 120,000 will be used; 3,100 ft^2 of membrane (1,410 tubes) is required. The UF will run until the concentration factor of 32 is reached. At this point, the **UF** system will be shutdown and cleaned and the UF residual (-2,600 gpd) sent to the high-shear rotary UF system (50 ft^2 of membrane) for further concentration. The rotary system will concentrate the residual by about a factor of 10, resulting in approximately 260 gpd of concentrated oily waste that will be disposed of via an off-site oil **recycler.** Effluent from the UF system will be applied to a constructed **wetlands/land** application hybrid system using land mass from the existing land treatment system. The **exist**ing land treatment application devices will be used to apply **the UF effiuent** in such a manner as to maintain near saturated conditions and an aerobic environment. Bulrush, tall **fescue,** and orchard grass will be planted throughout the system. Required land **areas** for winter and summer conditions are 15 and 5 acres, respectively.

APPENDIX. REFERENCES

Cooper, I? F., and Hobson, J. A. (1989). **"Sewage** treatment by reed **bed** systems: The present situation in the United Kingdom" *Constructed Wetlands for Wastewater Treatment: Municipal, Industrial, and Agricultural*, D. A. Hammer, ed. *Lewis* Publishers, Chelsea, Mich.

Reed, B. E., Lin, W., Dunn, C.. Carriere, P.. and Roark, G. (1997a). "Treatment of an oil/grease wastewater using ultrafiltration: Pilot-scale results." *Separation Sci. and Technol.*, 32(9), 1493 – 15 11.

Reed, B. E., Lin, W.. Viadero. R. C., and Young, J. (1997b) "Treatment of oily wastes using high-shear rotary ultrafiltration." *J. of Envir. Engrg*, ASCE, 123(12), 1234-1242.

Zhu, X.. Read, B. E., Carriere, P., and Roark, G. (1997). "Investigation of several polymers for the treatment of an emulsified oil wastewater." *Separation Sci. and Technoi..* 32(13), 2173-2187.