**Report on Bioventing of Petroleum Contaminated Soils at
108-3C: Active Extraction and Passive Injection (Barometric
Pumping) of a Gaseous Nutrient (U)**

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DOE Contract No. **DE-AC09-96SR18500**

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ABSTRACT

A bioventing system was constructed with horizontal extraction wells and vertical injection wells in an area which had previously been excavated and then backfilled. Initial in-situ respiration rates (air addition only) suggest that hydrocarbon degradation may be nutrient limited. The rate of TPH degradation was maximum (0.8-1.2 mg/kg/day) between 10-15 ft (bgs), but dropped to essentially zero 30 ft (bgs) within the contaminated zone (even though previous analysis at this depth indicated a TPH concentration of 3800 ppm). Analysis of the soil at 17 ft showed that NO_3 and PO_4 were below detection limits (0.5 ppm), indicating that nutrient limitation may be occurring. Nitrate levels were highest at 10 ft (bgs), correlating with the highest respiration rates. However, phosphate levels were at/or below detection levels throughout the site (indicating possible PO_4 limitation). Viable cells increased from 3×10^6 cfu/g at 3 ft (bgs) to 1×10^7 cfu/g at 10 ft (bgs) and remained relatively constant down to 17 ft. Cell numbers in the control area were significantly lower than in the contaminated zone (4.5×10^3). Gas phase nutrients (triethylphosphate and nitrous oxide) will be injected to see if the hydrocarbon degradation rate can be increased.

INTRODUCTION

Since the 1950's, emergency power required for operation of Savannah River Site (SRS) Reactors has been met by diesel generator operations. These diesel engines were fueled via a system of above ground storage tanks (AST) in each Reactor Area (the 108-3 facilities K, L, and C). Past operating practices at these AST facilities resulted in the accumulation of diesel spillage for over 30 years.

In 1994, efforts to mechanically remove petroleum contaminated soils (PCS) found at the 108-3C AST site began. Following excavation down to 20 feet, in which approximately 500 cubic yards of contaminated soils were removed, several hand augured samples were taken at the bottom of the excavation pit. Extensive hydrocarbon contamination down to > 40 ft. was found

(up to 3800 ppm) and mechanical removal was terminated since costs for removal would significantly exceed originally estimates. The operating history of the 108-3K and 108-3L AST sites, which were typical to 108-3C, suggested that contamination at these sites would be similar to that discovered at 108-3C. Further characterization was performed at these sites using laser induced fluorescence probing (LIF) and cone penetrometer technology (CPT). With these technologies one site was determined to be clean (108-3K) and at the other a hydrocarbon plume was characterized (108-3L, TPH up to 3600 ppm). Based on data from the literature, site characterization data, and SOILS facility data at SRS, bioventing was proposed as an economical clean-up method.

Several technologies developed at the SRS have been implemented in these bioventing projects; gas phase nutrient addition, discrete zone piezometers, and passive air injection via a Baro-Ball. Bioventing systems installed and operated at these sites each utilize a vacuum pump to pull air (oxygen) through the vadose zone. At Site 108-3C, horizontal extraction wells were installed during backfilling and passive injection wells were installed around the pit. The injection wells were connected to a diffusion chamber and Baro-Ball. The diffusion chamber will be half filled with triethylphosphate (TEP), a volatile organic phosphate, which will act as a gaseous source of phosphate. The Baro-Ball will act as a check valve to allow air flow through the tank and sweep TEP into the subsurface, but prevent backflow in the system if the vacuum pump fails or is shut down for in-situ respiration studies. The bioventing design at site 108-3L is similar except that vertical extraction wells will be installed.

BACKGROUND

Biodegradation of petroleum hydrocarbons by stimulation of indigenous soil microorganisms, sometimes called biostimulation, is a proven remediation technology. Petroleum land farming has been used in the oil industry for decades to degrade large quantities of oil sludges (Bartha and Bossert, 1984). If adequate amounts of moisture, oxygen, and nutrients are available, complete degradation of petroleum hydrocarbons can occur. The process exploits the ability of many indigenous soil microorganisms to completely metabolize petroleum hydrocarbons to generate new biomass. Biostimulation can be applied both above ground in prepared beds (biopiles) and below ground (in-situ) via bioventing. In each case it is imperative to identify the rate limiting steps, such as low microbial numbers, low moisture content, nutrient limitation, low oxygen concentration... ect, and design the system to eliminate these steps.

Oxygen mass transfer is usually the initial rate limiting step in the degradation of petroleum hydrocarbons below the ground. Bioventing is a method of increasing oxygen mass transfer by either air injection or vacuum extraction and is appropriate for sites where the contamination is deeper and/or more extensive, provided the soil is relatively porous. However, contrary to soil vapor vacuum extraction, flow rates are relatively low to prevent stripping, but high enough to enhance microbial metabolism (Hinchee et al., 1991). Bioventing also has the advantage of being able to be integrated into a strategy that includes intrinsic bioremediation, thereby further

reducing the over all costs of the remediation. Bioventing has been used to remediate gasoline, diesel, and PAH contaminated soils (Miller, 1991).

In general, air (oxygen) is the only nutrient injected during a bioventing campaign (EPA, 1995). It has been reported that nutrients are not required in most field bioventing sites (Miller, 1990 and 1991). However, in many cases biodegradation can be limited by nutrient availability (once oxygen limitations have been overcome) and hence nutrient feed systems may have to be combined with the bioventing process (Breedveld et al., 1995; Brockman et al., 1995). A wide range of nutrients have been successfully used to enhance bioremediation at other sites. Nitrogen has been successfully introduced into the terrestrial subsurface for biostimulation using ammonia, nitrate, urea, and nitrous oxide (USEPA, 1989, Bioremediation of hazardous waste sites workshop, CERI-89-11, Washington, DC). Phosphorus is naturally quite low in most environments and in terrestrial subsurface environments even if phosphorus concentrations are high it may be in a mineral form that is biologically unavailable, eg. apatite. Several inorganic and organic forms of phosphate have been successfully used to biostimulate contaminated environments (EPA, 1989). The Savannah River Technology Center has successfully shown that triethylphosphate or TEP can be added in gas form to stimulate bioremediation (Hazen et al, 1994, Lawrence et al., 1994). Thus, nutrients can be added as a gas in the air injection wells (ie., nitrous oxide, ammonia and TEP) or in liquid form via infiltration galleries or sprinkler systems. Liquid nutrients such as (NH_4NO_3) have been added to contaminated soil via surface irrigation in biosparging processes (Lord et al., 1995) and are transported to the contaminated zone by percolation. Gas phase nutrient addition provides a more uniform and direct transport of nutrients to the contaminated zone and overcomes precipitation problems.

EXPERIMENTAL DESIGN

Site Characterization

Approximately 500 yd³ of petroleum contaminated soil was removed from 108-3C (down to a depth of 20'), which ranged in TPH concentration from 56 to 3000 ppm (based on 19 samples with a mean value of 1122 ppm). Petroleum contamination was also found another 10 ft below the pit (3800 ppm), but was found to diminish to very low levels 20 ft below the pit (11 ppm). Additionally, lateral soil borings indicated contamination ranging from 90 to 900 ppm TPH (Fig. 1). Because of the extensive contamination below the bottom of the pit, further excavation was not performed. It was decided to back-fill the pit with the contaminated soil and use bioventing to degrade the TPH.

Analytical and Microbial Methods:

Soil samples were analyzed for total petroleum hydrocarbons (diesel fraction, TPH-D) via EPA 8015M (Weston, PA). Groundwater samples were analyzed for TPH-D and BTEX (EPA 8240). Viable cell counts (aerobic heterotrophs) were performed using spread plates on 1% PTYG medium (Balkwill, 1989) and were based on dry weights. Nitrogen (NO_2 , NO_3) and phosphate

(PO₄) were extracted from the soil using water as the solvent and quantified using ion chromatography (Greenberg, et al., 1992). Soil gas samples were evacuated via instrument pumps or a diaphragm vacuum pump (twin-headed KNF Neuberger, INC., 17 L/min at 29.5 in Hg) from discrete zone sampling ports (1'x 1' tri-lock threaded chamber with a slotted sample chamber and hollow chamber swaged to 1/8" tubing). Oxygen was measured using an O₂ analyzer (Teledyne Brown, Portable Oxygen Analyzer - 0-5%, 0-10% and 0-25%) and a LandTec (GEM 500); the LandTec was also used to measure CO₂, static and differential pressure.

FACILITY DESIGN

First, eight passive injection wells were installed using CPT (2" ID and 45' deep with 10' of well screen) and were positioned around the pit as portrayed in Figure . The injection wells were located in such a manner as to provide air and nutrient flow to contact the lateral contamination and contamination below the bottom of the pit, as well as the back-filled soil. Next, 1" ID piezometers were positioned within the pit before back filling. Piezometers 1 and 2 were 30' in length and a sampling device was positioned at 10', 20', and 30' down (Fig. 1). Piezometers 3 and 4 were 20' in length and a sampling device was positioned at 5', 10', 15', and 20' down (Fig. 1). The sampling device was designed such that soil gas and pressure readings could be obtained without effect from other sample ports (the PVC sampling device was 1" in length and 3" in diameter with female and male tri-lock threads on each end, a counter bore and thread and a 0.55" opening for tubing connection, and a slotting sampling area). CPT technology was used to install three vadose zone piezometers outside the zone of contamination (piezometers 5, 6, and 7 on Fig. 1). These piezometers were screened at 10' (#5), 45' (#6), and 20' (#7). Once the piezometers were in place, the pit was back-filled. As the pit was back-filled, 10/10/10 fertilizer was spread over the soil; approximately 8, 50 lb bags of fertilizer were added to the soil. At certain points during back filling, a layer of corrugated ABS pipe with slots was positioned in the pit. One hundred feet of the corrugated pipe was snaked in the hole and connected by a tee. Straight PVC riser was connected at the tee and taken to the top of the pit. The corrugated pipe was positioned at three layers 10', 15', and 20' down. The last layer at 10' consisted of two 100' sections of corrugated pipe and two risers (Fig.1). The corrugated pipe was wrapped in burlap to prevent the soil from blocking the slots.

Each PVC riser (4") was connected to a tee and reduced to one inch pipe. One inch pipe from each riser was connected to a globe valve, elbowed to a flow meter, and teed into a manifold. The manifolded system was connected to a vacuum pump (Roots/Dresser, Rotary Lobe Blower with maximum allowable operating conditions of 5275 rpm, 14" Hg vacuum and 225°F). The top of each tee was capped and NPT threads added for a sampling port and addition of a pressure reading device. Each injection well was connected to a 3 gallon hot-dog tank (1" connection), this was the air/gas outlet, and the inlet was connected to a barrel ball via 1/2" stainless steel tubing. The Baro-Ball was constructed of PVC and is essentially a check valve to allow air flow through the tank, but prevent any backflow should in-situ pressure become greater than atmospheric. The diffusion chamber will be half filled with triethylphosphate (TEP), which is

volatile (vapor pressure of 0.27 mmHg at 20°C) and will be transported by air flow through the chamber into the injection well.

RESULTS

In-situ Respiration and Baseline Data

Before the vacuum pump was turned on, soil gas analysis was performed and soil samples were analyzed for nutrients and microbial numbers as a function of depth. Soon after start-up, soil gas analysis was performed to test the effect of the vacuum pump on O₂ mass transfer. Baseline in-situ respiration tests were also performed. Soil gas analysis clearly showed that certain areas within the backfill were metabolically active, since oxygen concentrations had dropped to 6 to 10 % relative to the control piezometers (points outside the contaminated area), but increased to near atmospheric levels once the extraction was initiated. In-situ respiration studies showed that the rate of oxygen consumption ranged between 0.8 and 1.7 %/day across the site (Fig. 2). The oxygen consumption rates were converted to estimated TPH degradation rates using a batch reactor design equation, assuming zero order with respect to hydrocarbon concentration, and using the stoichiometry of hexane oxidation (Equation from EPA Bioventing Manual).

$$\text{Equation 1: } k_b = (k_o)(\phi)(dO_2)(C)(0.01)/\rho_{\text{soil}}$$

where ,

$$-r_{\text{TPH}} = k_b = \text{mg TPH/ kg soil/day,}$$

$$k_o = \% \text{ O}_2/\text{day,}$$

$$\phi a = (\text{Gas-Filled Pore Volume}) = 0.25 \text{ cm}^3 \text{ gas/cm}^3 \text{ soil (assumed value)}$$

$$dO_2 = 1300 \text{ mg O}_2/\text{L}$$

$$C = 0.29 \text{ mg Hexane consumed/ mg O}_2 \text{ consumed}$$

$$\rho_{\text{soil}} = 1.4 \text{ g/cm}^3 \text{ (assumed value)}$$

Equation 1 can be derived from the heterogeneous batch reactor design equation

$$dN_j/dt = r_j W,$$

where W = weight of the catalyst (in this case the soil) and the oxidation reaction for hexane, a representative hydrocarbon, is

$$-rA = a/b \text{ } -rB \text{ or } -rO_2 = 9.5 \text{ } -rB$$

$$-rB = b/a \text{ } -rA \text{ or } -r\text{Hexane} = 1/9.5 \text{ } -rO_2 = 0.29 \text{ (mgHexane/mgO}_2\text{) } -rO_2$$

Thus,

$$-r\text{Hexane} = (0.29) \text{ } -rO_2 = (0.29) dN_j/dt \text{ } 1/W$$

Assuming zero kinetics

$$-r_{O_2} = (U_{\max} X/Y_{O_2}) O_2 / (K_{O_2} + O_2)$$

if $O_2 \gg K_{O_2}$

then $-r_{O_2} = U_{\max} X/Y_{O_2}$

$$-r_{O_2} = dN_j/dt \cdot 1/W$$

$$C_{O_2} = N_{O_2}/V;$$

Assuming a constant volume reactor

$$-r_{O_2} = (dC_{O_2}/dt) V/W$$

where,

dC_{O_2}/dt is the slope of the in-situ respiration test,

W = Volume of solids X Density of solids

$W = \{(1-\phi) V\}$ (Bulk Density)

ϕ = void volume / total bed volume (total porosity)

V = the reactor volume or volume of the contaminated soil

$$-r_{O_2} = dC_{O_2}/dt \cdot 1/(1-\phi) (\rho_{\text{soil}})$$

The estimated degradation rates appear to peak between 10' and 15' bgs, with lower rates near the surface and deeper in the pile (Fig. 3). These rates tend to correlate with an increase in cell numbers and nutrient levels at these points (Fig. 4), as well as TPH concentrations within the soil. The TPH concentration ranged from 64.5 ± 16 (n=4) at 5', 114.3 ± 18 (n=4) at 10', and 117 ± 57 (n=4) at 15-17' (from a limited number of samples). Total viable counts were higher at 10' and 15', and NO_3 levels were higher at these depths (Fig. 4). However, phosphate levels were low and ranged from 0-0.3 ppm regardless of the depth (2-17 ft: data not shown). Thus, in general, respiration rates increased with higher TPH concentration and nutrients, which in turn probably increased microbial counts within the soil.

Performance Assessment

To assess bioventing at 108-3C, the site was divided into two zones: the volume of soil backfilled (500 yd³) or zone 1 and the volume of soil beneath the backfill which was contaminated (approximately a 10' [depth] x 20' [wide] x 40' [length] volume or 8000 ft³) or zone 2 (see drawing below). The initial TPH concentration in zone 1 was assumed to be a mean value of 1100 ppm. Zone 1 appears to have been remediated close to acceptable levels (100 ppm), based on the limited number of sample taken from the area. A method to estimate the final sample size to verify clean-up for zone 1 is shown below.

$$\text{UCL (Upper Confidence Limit)} = X + (t=0.95(n-1)) S_x$$

where:

$n = 8$, number of samples

S_x = standard error = $S/\sqrt{n}$

S = standard deviation

$X = 115.75$ (ignoring samples taken at 5')

$$\text{UCL} = 115.75 + (1.895 * 39.1 / 2.82) = 142$$

Since the UCL is above the maximum allowable concentration of 100 ppm the lambda calculation is performed.

where:

$$\lambda = (RT - X) / S$$

RT = regulatory limit

S = standard deviation

$$\lambda = \text{abs}(100 - 116) / 39.1 = 0.41$$

From statistics tables ($\alpha = 0.05$ and $\beta = 0.05$), 70 more samples are required to verify the TPH concentration in zone 1. Instead of continued sampling at the site, it is suggested that treatment continue and concentrate on zone 2. Estimates of degradation rates within zone 1 will be used to approximate the time required clean-up.

There are two sets of data that can be used to estimate the rate of TPH removal within zone 2: in-situ respiration rates and actual TPH analysis of the soil. The maximum rate of TPH removal based on in-situ respiration rates was measured at 1.2 mg/kg/day. However, if the change in TPH concentration is used, a higher rate is calculated. The batch reactor design equation was used to estimate the degradation rate in the following manner.

$$-r_A = -\frac{dC_A}{dt}(\text{constant} - \text{volume})$$

$$-r_A = kC_A = k(\text{zero} - \text{order})$$

$$k = \int_{C_{Ao}}^{C_{A(t)}} \frac{dC_A}{dt} = \frac{C_{Ao} - C_{A(t)}}{t}$$

$$t = N_{Ao} \int_0^{X(t)} \frac{dX}{V(-r_A)}$$

Using the third equation in the set above, the zero order rate constant, k (mg/kg soil/day) was estimated. The initial TPH concentration, C_{Ao} was assumed to 1100 ppm(mg/kg), the final concentration, C_A , was assumed to be 116 ppm, and the total operating period was estimated at 8 months (240 days, which includes the time from backfilling to sampling). The rate constant, was found to be 4 mg/kg/day, approximately 4 times higher than those recently measured by in-situ respiration.

Using a rate of 4 mg/kg/day, several clean-up scenarios are projected for zone 2. First, the third equation in the above set was rearranged and solved for $C_{A(t)}$ and the cumulative % conversion calculated for zone 2. Assuming an initial concentration of 3800 ppm, it would take approximately 2-3 years to reach 100 ppm (Table 1). However, if the average TPH concentration for zone 2 is used (3800, 1000, 290; Mean = 1700) the required clean-up time is much shorter - 1 year (Table 2).

CONCLUSIONS

The estimated maximum degradation rate is relatively lower than rates reported for other diesel bioventing sites, 1-2 mg/kg/day compared to 6-30 mg/kg/day (EPA, 1995). The low rates could be due to many factors, such as low TPH concentration and low nutrient concentrations. A significant amount of degradation could have occurred in the excavated soil during storage and during the backfilling process when fertilizer was added to the soil, since in-situ respiration studies were performed 8 months after backfilling and installation of the biosystem. Recently,

soil samples taken at 10' and 15' show that the TPH levels have declined from 1100 ppm to 100-180 ppm.

However, the lower respiration rates at the deeper depths (20' to 30') are probably due to nutrient limitation and not low TPH levels. This is indicated by the fact that the rate of TPH degradation was maximum between 10-15 ft (bgs), but dropped to essentially zero 30 ft (bgs) within the contaminated zone, even though previous analysis at this depth showed a TPH concentration of 3800 ppm. Moreover, analysis of the soil at 17 ft showed that NO_3 and PO_4 were below detection limits (0.5 ppm), indicating that nutrient limitation may be occurring. For this reason the gaseous nutrient, triethylphosphate, will be injected into the pile. In-situ respiration rates will be periodically monitored to see if TEP will increase the respiration rate and hence the hydrocarbon degradation rate. Since the backfilled soil appears to be relatively clean (100-150 ppm), attempts will be made to concentrate nutrient injection in the soil beneath the bottom of the pit. Helium tracer analysis will be performed to measure the influence of each injection well. Clean-up for zone 2 (beneath the bottom of the pit) is estimated to take 1-3 years from the start of aeration.

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Figure 1 - Top view of site showing approximate location of injection wells, piezometers, extraction wells, approximate contour lines (horizontal), and horizontal wells.

Figure 2: In-Situ Respiration rates at 10 ft bgs (Top) and 15 ft bgs (Bottom) After Shut Down of the Vacuum Extraction Pump: Oxygen Consumption (■, ●, ★, +) and CO₂ Production (□, ○, ☆, ⬢), Compared to a Control Piezometer Located Outside the Contaminated Zone (O₂, ▼; CO₂, ▲).

Figure 3: Change in the Estimated TPH Degradation Rate (Based on Slope of Oxygen Consumption Rates) as a Function of Depth Within the Contaminated Zone and Time: 6/96 and 8/96 (●) and 10/96 (○).

Figure 4: Viable Cell Count and Nutrient Concentration As A Function of Depth Within the Contaminated Zone.

Table 1: Estimated Time Requirement for Clean-up at 108-3C (Zone 2)

Estimated Performance for Zone 2: Lower Portion of Area	
k,mg/kg/day	4
CAo,mg/kg	3800
Ca,final mg/kg	100
Equation	$CA(t) = CA0 - tk$
X Needed	0.97368421

Time,days	Time,months	Time,years	CA(t) ppm	X, conversion
100	3.33333333	0.27777778	3400	0.10526316
200	6.66666667	0.55555556	3000	0.21052632
300	10	0.83333333	2600	0.31578947
350	11.6666667	0.97222222	2400	0.36842105
400	13.3333333	1.11111111	2200	0.42105263
450	15	1.25	2000	0.47368421
500	16.6666667	1.38888889	1800	0.52631579
550	18.3333333	1.52777778	1600	0.57894737
600	20	1.66666667	1400	0.63157895
650	21.6666667	1.80555556	1200	0.68421053
700	23.3333333	1.94444444	1000	0.73684211
750	25	2.08333333	800	0.78947368
800	26.6666667	2.22222222	600	0.84210526
850	28.3333333	2.36111111	400	0.89473684
900	30	2.5	200	0.94736842

Table 2: Estimated Clean-up Time for Zone 2 at Site 108-3C

Estimated Performance for Zone 2: Lower Portion of Area					
k,mg/kg/day			4		
CAo,mg/kg			1700		
Ca,final mg/kg			100		
Equation	$CA(t) = CA0 - tk$				
X Needed			0.97368421		

Time,days	Time,months	Time,years	CA(t)	X, conversion	
100	3.33333333	0.27777778	1300	0.65789474	
200	6.66666667	0.55555556	900	0.76315789	
300	10	0.83333333	500	0.86842105	
350	11.66666667	0.97222222	300	0.92105263	
400	13.33333333	1.11111111	100	0.97368421	