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TREATABILITY STUDIES FOR ON-SITE BIOLOGICAL REMEDIATION OF SOILS AND LEACHATES CONTAMINATED BY COAL CONVERSION RESIDUALS AND BY-PRODUCTS

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Summary

The purpose of this study was to investigate the feasibility of using the Sequencing Batch Reactor (SBR) as a key component in an on-site biological remediation program for the treatment of contaminated soil and leachates present at Niagara Mohawk Power Corporation's Harbor Point site in Utica, New York. Soil and leachate samples brought from this site were tested for biological activity. Plate counts and carbon evolution from soil respirometers verified significant activity of indigenous soil microorganisms. Laboratory-scale SBRs were tested by using several operating strategies. The SBRs removed most of the soil and leachate constituents while producing "specialized bacteria" resistant to cyanide. Results from Chemical Oxygen Demand (COD) measurements and gas chromatograph analyses of selected target organics showed high removal efficiencies.

Introduction

Various residuals and by-products of the coal carbonization process that were produced by two former gas manufacturing plants that were owned and operated from 1902 to the mid 1950's by one of Niagara Mohawk's predecessors,

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the Utica Gas and Electric Company, were disposed of at a 70 acre parcel (about 280 km^2) known as Harbor Point. As a result, soils and leachates at Harbor Point are contaminated with typical coal gasification materials such as polynuclear aromatic hydrocarbons (PNAs), phenols, coal tars and oils, purifier waste containing cyanides and sulfates, ash, and other debris.

The purpose of this study was to investigate the potential of the sequencing batch reactor (SBR) as a key component in an on-site remediation program for the Harbor Point site. The laboratory-scale SBR was to be integrated into an overall test system which included the biological degradation of hazardous organics found in leachates and soils and the production of "specialized bacteria" in the SBR for eventual application to the soils. This unique approach to soil decontamination extends the innovative features of the SBR that were described by the United States Environmental Protection Agency [1,2], the National Science Foundation [3], and the U.S. Congressional Office of Technology Assessment [4].

The SBR has been shown to be a cost effective and energy efficient means for degrading hazardous wastes [5–7]. It is uniquely suited for the selection and enrichment of the desired microbial population because of the ease with which a diverse array of operating strategies and selective pressures can be implemented [8–11]. The convenience in operation stems from the time oriented nature of the process. Specifically, each tank in the SBR system is filled during a discrete period of time. During this period, organism selection can be controlled by manipulating the maximum growth rates of the microbes and by regulating the oxygen levels in the reactor (e.g., from anaerobic to aerobic). After a tank is filled, treatment continues as the SBR behaves as a batch reactor. During this period further selective pressures are applied by controlling the length of time the organisms are subjected to starvation conditions. After treatment, the microbes are allowed to settle and the clarified supernatant is drawn from the reactor. During these quiescent periods, the opportunity for plasmid exchange and general enrichment of genetic information are enhanced. "Specialized bacteria" developed in the SBR can be added to contaminated soils by using well-developed municipal sludge application technologies [12,13]. The results reported upon herein focus on SBR operation and performance and not on on-site soil detoxification.

Experimental approach and analytical methods

Experimental work of this study included two phases. Phase I was carried out over a five-month period and was aimed at determining if the soils and leachates at the Harbor Point site can be treated biologically. This phase consisted of four independent tests which were: (1) determination of chemical characteristics of the leachates; (2) determination of leaching properties of the soils; (3) analysis of biological characteristics of the soils, including growth

experiments and respirometer tests; and (4) operation of two "initial screening" batch reactors being fed contaminated leachates and soils. The "initial screening" studies were the basis of a rather extensive phase II investigation which followed Phase I. During phase II, the biological degradation of the soil and leachate contaminants were tested in four laboratory scale SBR's.

There have been several reports that bacteria present in sewage sludge degrade either phenols [14], PNAs [15], or cyanides [16]. The impact of soils and leachates contaminated with these compounds had not been previously tested in the SBR. In order to explore a relatively broad range of SBR operating strategies, the four reactors were operated with (1) either continuous or instantaneous feed modes in order to test the impact of shock loads; (2) either completely aerobic or an anaerobic/aerobic sequence with a nitrate supplement in order to compare removal efficiencies with these operating strategies; and (3) the addition of a carbon source (glucose) to determine if cyanide removal was enhanced during denitrification.

Most of the chemical analyses of this study were conducted according to procedures described in the APHA et al. Standard Methods [17]. Chemical Oxygen Demand (COD) was determined by the Dichromate Reflux Method (508C). Ammonia nitrogen (NH_4–N) as analyzed by the Nesslerization Method (417B), and nitrate and nitrite nitrogen (i.e., oxidized nitrogen, NO_x–N) by the Cadmium Reduction Method (Hach modification [18]). Total cyanide was determined by Method 412A.B.C. Total bacterial counting analyses were performed by the Spread Plate Method (907B [17]) on Trypticase Soy Agar (TSA).

Gas chromatograph (GC) analyses were conducted according to procedures in the Federal Register [19]. The GC used was a Varian 3700 with Vista 401 data processor. Three methods were used for detection of three compound groups typical to coal gasification wastes. In each method, specific compounds were selected as target compounds to be compared to standard solution data. The three methods and the selected target compounds were: (1) purgeable aromatics (Method 602) for benzene, toluene and xylenes; (2) phenols (Method 604) for phenol; (3) Polynuclear Aromatic Hydrocarbons (Method 610) for acenaphthene, acenaphthylene and naphthalene.

Results and discussion

Leachate chemical characteristics

Testing was conducted on three soil samples and one leachate sample. The soil samples were designated 1, 2, and 3 with soil 1 being the least contaminated and soil 3 the most contaminated. The leachate sample was collected at the soil 3 site. Four replicates of this leachate sample were shipped in separate containers. The composition of the filtered leachate is given in Table 1. As can be seen from these data, the leachates have a low pH of about 2.7, sufficient

TABLE 1

Leachate characteristics

Parameter	Unit	Replicate samples			
		1	2	3	4
pH	–	2.68	2.64	2.67	2.65
COD	mg/l	80	80	70	80
Ammonia nitrogen (NH_4-N)	mg/l	7	7	7	5
Phosphate phosphorus (PO_4-P)	mg/l	0.5	0.2	0.2	0.2
Iron (Fe)	mg/l	80	70	30	70
Free cyanide (CN)	mg/l	0.7	–	–	–

ammonia nitrogen and phosphate phosphorus to support biological growth, and less than 100 mg/l of soluble COD. Because of the low concentration of soluble COD in the leachate, leachates fed to the SBRs were supplemented with contaminated soils so as to promote bacterial growth, to develop a more diverse biological population, and to enhance the biological removal of target organics present in both the leachates and soils. A glucose supplement was also tested in some reactors.

Soils leaching properties

The leaching potential of the soils was tested by placing 2 gram soil samples in a 100 ml basal salts medium and shaking at 28°C for 14 days. The results of this procedure for the three soil samples collected at Harbor Point site is shown in Table 2. It can be clearly seen that all three soils demonstrated a high release potential for organics. Unfortunately, similar studies using tap water and soils, produced leachates which contained low levels of soluble COD. However, the resulting total COD was high and correlated well with high levels of suspended colloidal and pin-point materials. These materials served as the main source of feed to the SBR units during phase II of the experimental study.

TABLE 2

Release of soluble COD from 2 g of soil in 100 ml basal salts medium with shaking at 28°C

Day	Soluble COD (mg/l)		
	Soil #1	Soil #2	Soil #3
1	280	560	280
7	480	960	560
14	520	1700	1100

Bacterial examination of the soils

Growth experiments were conducted to characterize the organisms present in the soil and to determine if they were able to grow on the organic constituents, hazardous or non-hazardous. The growth potential of the organisms present in the soil samples were tested on TSA. This was done by adding a 2 g soil sample to 100 ml of basal salts medium in a 250 ml flask and shaking at 28°C for two weeks. Small aliquots removed from each soil flask on a regular basis were plated on TSA medium and the number of Colony Forming Unites (CFU) produced counted. Results obtained for all three soils are plotted on Fig. 1. As can be seen, soil 1 had the highest initial counts at about 4000 CFU/ml. The initial counts for soil 2 were 20 CFU/ml and for soil 3, 200 CFU/ml. This corresponds to approximately 200,000, 1,000 and 10,000 CFU per g of soil for soils 1, 2 and 3, respectively. Considering the fact that not all of the aerobic organisms present in the soil would be detected on the TSA medium and that none of the anaerobes would be measured by this procedure, these results indicate that a significant indigenous population is available at the Harbor Point site to assist in a biological remediation program if optimal growth conditions are provided. Indeed, the aerobic, mixed environment established in the flasks did result in a 1000 fold increase in numbers of organisms in soils 1 and 3 and a 10,000 fold increase in soil 2. The die off of organisms at the end of the experiment in flasks containing soils 2 and 3 may have resulted from the exhaustion of readily usable carbon sources and/or from the accumulation of toxic organics leached from the soils during the course of the test.

A total of 29 bacteria were isolated from the soil growth flasks and cloned to purity. Of these, 8 or 9 may be different strains of bacteria. These isolates were tested for their ability to grown on acenaphthene (0.01%), naphthalene (0.04%, and 0.1%), and phenol (0.01%, 0.1% and 0.5%) as sole carbon sources. The controls were plates containing no carbon sources. Two of the isolates were

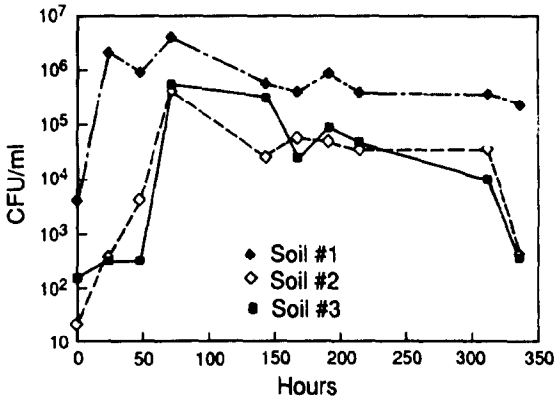


Fig. 1. Semi-log plot of growth of indigenous bacteria in soil samples.

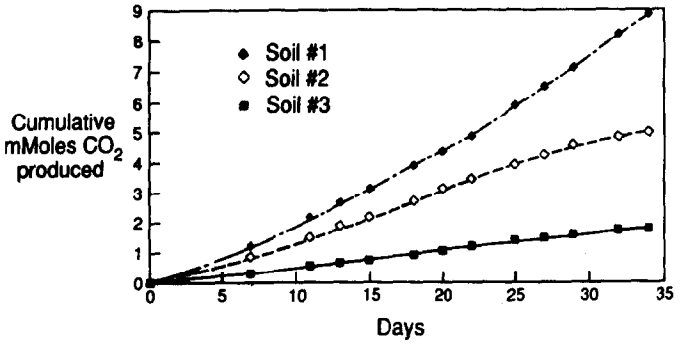


Fig. 2. Cumulative CO_2 evolution from soils.

capable of growing on 0.01% acenaphthene, five of the isolates on 0.04% naphthalene, and five on 0.01% phenol. Two of the colonies having distinct morphological characteristics on the plates were able to grow on all three carbon sources. No growth was observed in 0.1% naphthalene or on either 0.1% or 0.5% phenol.

Further evidence of the biological activity of the naturally occurring organisms at Harbor Point was obtained from the measurement of carbon dioxide evolution in soil respirometers. In these studies, 50 g of each of the soils 1, 2, and 3 used in the growth studies described above were placed in separate soil respirometers. Carbon dioxide evolution was measured by trapping in KOH and titration with acid. Results from these experiments are presented in Fig. 2. From this figure it can be seen that soil 3, the most heavily contaminated soil, showed the lowest rates of CO_2 evolution. Soil 2 had an intermediate rate of evolution and soil 1 had the highest rate. All data points have been corrected with data from sterilized control soils. On day 27, inorganic nutrients (0.05 g K_2HPO_4, 0.045 g KH_2PO_4, 0.02 g $(NH_4)_2SO_4$, and 0.0016 g $MgCl_2$) were added to the soils. No increase in the rate of CO_2 evolution was seen as a result of this addition. The results from the soil respirometer studies strengthened the bacterial count findings, indicating that an active indigenous population of microorganisms is present at the Harbor Point site.

Screening biological activity in batch reactors

After reviewing the results from the preliminary chemical and microbiological analyses, it was decided to operate two "initial screening" batch reactors, one fed with a mixture of leachate and soil (designated unit 1) and the other (unit 2) fed with the low COD leachate. Two 3.85 l reactors were operated with a 24 hour cycle on an instantaneous fill basis. Performance and biological activity were measured mainly by feed/effluent COD balances and by oxygen uptake rate (OUR) measurements. The OUR test served as an excellent in-

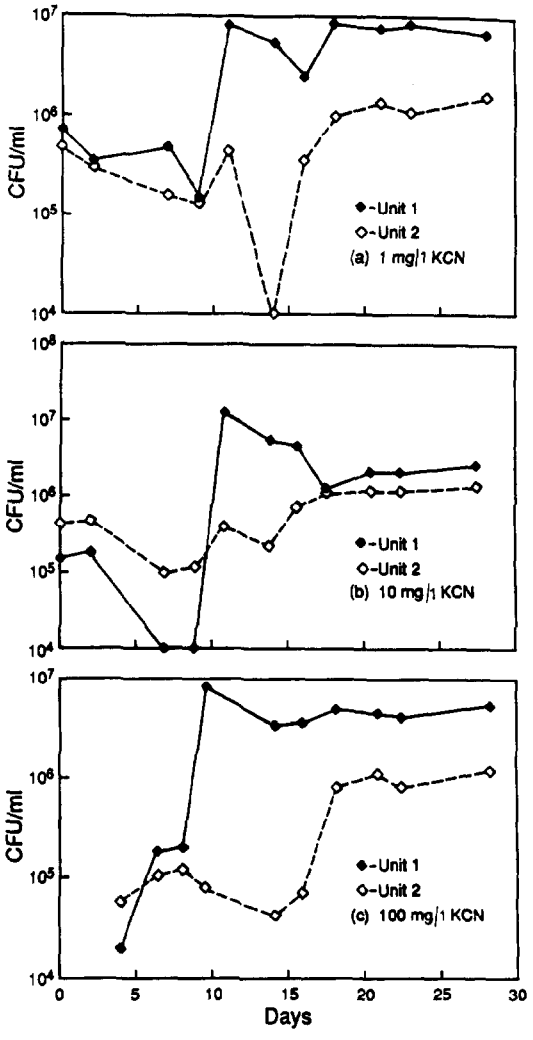


Fig. 3. Bacterial counts on MLSS from the "initial screening" reactors on TSA + KCN.

dicator for activity inhibition caused by sudden shock loading. Occasional gas chromatograph analyses of selected target organics generally showed agreement with the overall COD removal trends.

Additional information regarding biological activity in the laboratory reactors was obtained from bacterial plate counts on the mixed liquor suspended solids (MLSS). The plating medium was TSA with various additional amounts of cyanide, one of the components of the contaminated soils. The results of these counts are summarized in Fig. 3. As can be seen, a population of cyanide resistant bacteria that reached a stable population in number was selected in

the reactors. The number of bacteria determined at each cyanide concentration was essentially the same for increasing cyanide levels. Indeed, bacterial counts in the 10^6/ml range were determined on the highest cyanide concentration, 100 mg/l KCN. Medium containing 1000 mg/l KCN was tried but no growth was detected at this level at any dilution of biomass.

Phase II bench scale SBR operation

Based on the experience gained by the operation of the two batch reactors, four SBR units were established and operated simultaneously. Each reactor had a total liquid volume of 3.85 l and received a biomass inoculum which was composed of a mixture of domestic sewage sludge and the remaining sludge from the two batch reactors. All four SBRs were operated at room temperature (19 to 21 °C). The basic feed solution was prepared by mixing of 12 l of tap water with 240 g of contaminated soil for three days followed by 30 minutes of clarification. This feed solution contained low levels of soluble COD, ranging from 30 to 75 mg/l. However, the total COD was between 350 and 900 mg/l and the suspended solids (SS) concentrations ranged from 250 to 600 mg/l. Air to the reactors was introduced through porous diffuser stones. During an-

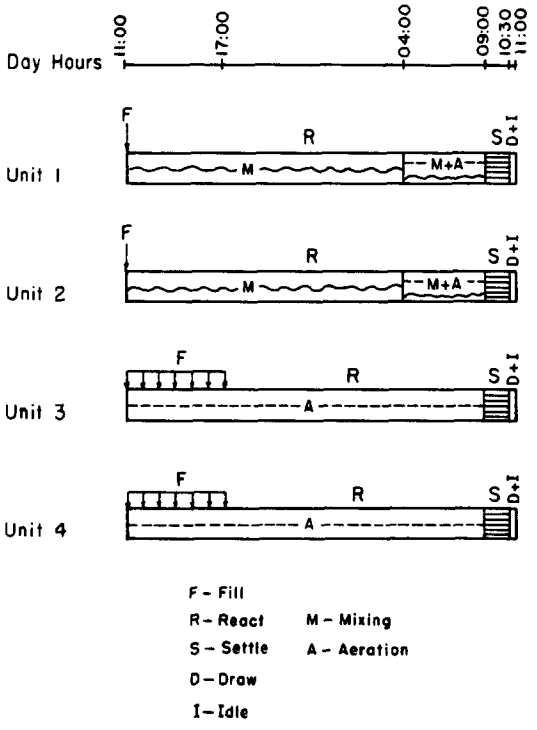


Fig. 4. SBR units operating strategies.

TABLE 3

Summary of operating conditions following initial three week acclimation period

Operating conditions	Unit 1	Unit 2	Unit 3	Unit 4
Max. reactor liquid vol., l	3.85	3.85	3.85	3.85
Daily feed vol., l	0.5	0.5	1.0	1.0
Detention time, days	7.7	7.7	3.85	3.85
Supplemental components Added to Basic Feed[a], mg, for:				
Glucose as COD	0	600	0	600
Ammonia Nitrogen as N	64	64	96	96
Phosphorus as P	10	10	15	15
Alkalinity as $CaCO_3$	0	0	600	600
Time in hours, for:				
Aerated fill	0	0	6	6
Mixed fill	0.01	0.01	0	0
Mixed react	17	17	0	0
Aerated react	5	5	16	16
Settle	1.5	1.5	1.5	1.5
Draw and idle	0.5	0.5	0.5	0.5
Cycles per day	1	1	1	1

[a]Basic feed was composed of leachate and contaminated soils obtained after mixing 12 l of tap water with 240 g of soil for 3 days followed by 30 minutes of clarification. Feed concentration of total COD ranged from 350 to 900 mg/l and that of suspended solids, from 250 to 600 mg/l.

aerobic periods magnetic stirrers were used for mixing. All aeration, mixing, and discharge functions were sequenced with laboratory timers. Reactor operating conditions established after an initial acclimation period of three weeks are summarized in Fig. 4 and Table 3. As can be seen, units 1 and 2 were filled on an instantaneous basis and units 3 and 4 were filled (using piston pumps) over a six hour period.

During the initial three week acclimation period, nitrate nitrogen was added to units 1 and 2. Mixing only was provided throughout the 22 hour react period in order to develop a denitrifying bacteria population. During the acclimation period, units 3 and 4 developed a nitrifying population. As a result, the original operating mode of units 1 and 2 was altered to include the five hour aeration period shown in Table 3, so as to promote nitrification and eliminate the need for an external supplement of nitrate. Nitrogen and phosphorus were added daily to the four reactors. Units 3 and 4 were supplied with additional alkalinity in order to promote nitrification and to minimize pH fluctuations. Table 3 summarizes the composition and amounts of the supplemental constituents added to the reactors. Glucose was chosen to be the additional carbon source for units 2 and 4 for its known tendency to form cyanohydrin with the cyanide

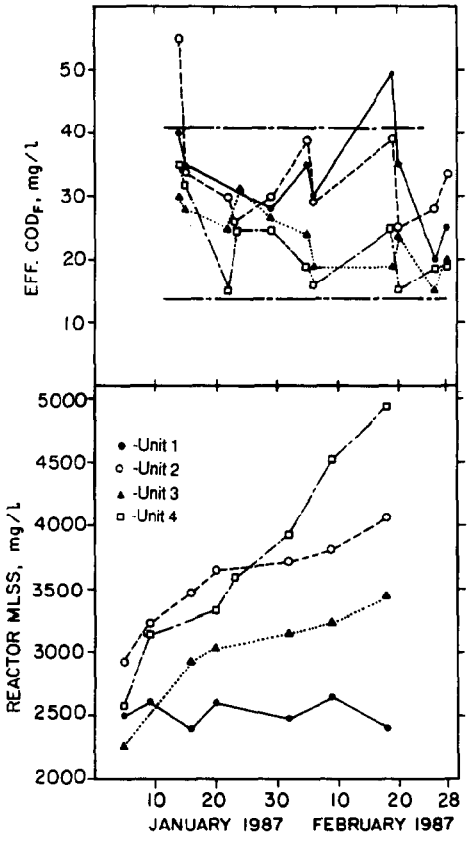


Fig. 5. Chronological plot of SBR treatment conditions: effluent COD and reactor suspended solids.

ion. The cyanohydrins have been reported to hydrolyze to aldonic acids. These acids can serve as carbon and energy source for either aerobic or anoxic bacteria [20–22].

Operation of the SBR units was monitored by routine sampling and bi- or tri-weekly analyses of COD, MLSS, mixed liquor volatile suspended solids (MLVSS), effluent suspended solids (total and volatile), ammonia nitrogen, and nitrate and nitrite nitrogen. A description of effluent soluble COD and each reactor's MLSS is given in Fig. 5. The soluble effluent COD was in the range of 15 to 40 mg/l reflecting high removal of the waste organic constituents. Moderate sludge wastage resulted in increased levels of MLSS. This increase in reactor suspended solids was due to bacterial growth and to the accumulation of the suspended solids present in the feed. As expected, units 2 and 4 had a high yield of solids (because glucose is added to the feed) and units 1 and 3 had poor yield in spite of having high removal efficiencies for most

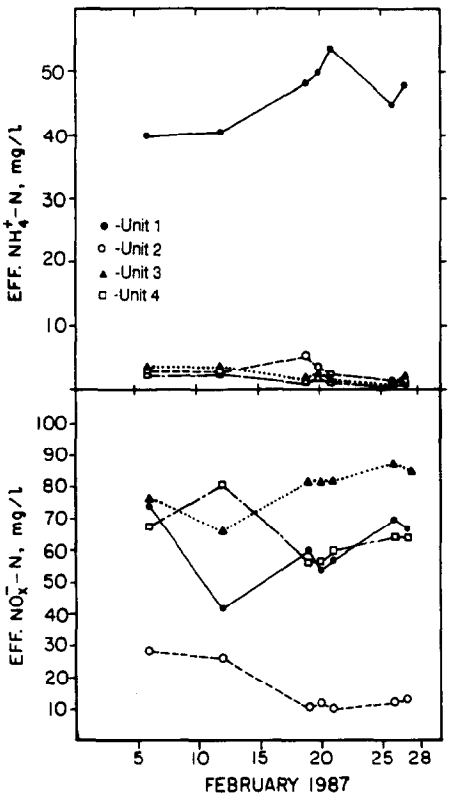
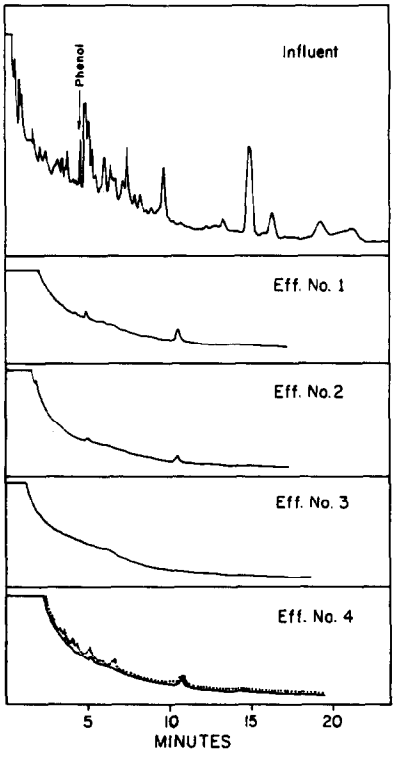


Fig. 6. Chronological plot of SBR treatment conditions: effluent ammonia and oxidized nitrogen.

constituents. This low yield for leachates containing toxic materials has been observed by others [23] and may be explained by high maintenance energy requirements that result in catabolism of large amounts of substrate carbon to CO_2 and less assimilation into new cell mass. In general, the reactors' effluents were turbid with high levels of suspended solids that probably resulted from the oily nature of the feed mixture. Units 2 and 4, the glucose augmented systems, however, developed better clarification with lower levels of effluent suspended solids (i.e., 50–70 mg/l versus 120–150 mg/l for units 1 and 3). In addition, the glucose served as a means for improving settling characteristics of the sludge, perhaps by stimulating growth of flocculating bacteria or by contributing to higher production of extracellular polymers assisting in bioflocculation.

The variation of nitrogen compound concentrations in the reactors' effluents is shown in Fig. 6. The influent ammonia nitrogen concentration for units 1 and 2 was 132 mg/l and was 100 mg/l for units 3 and 4. As can be seen from Fig. 6, nitrification easily developed in all four reactors with highest levels



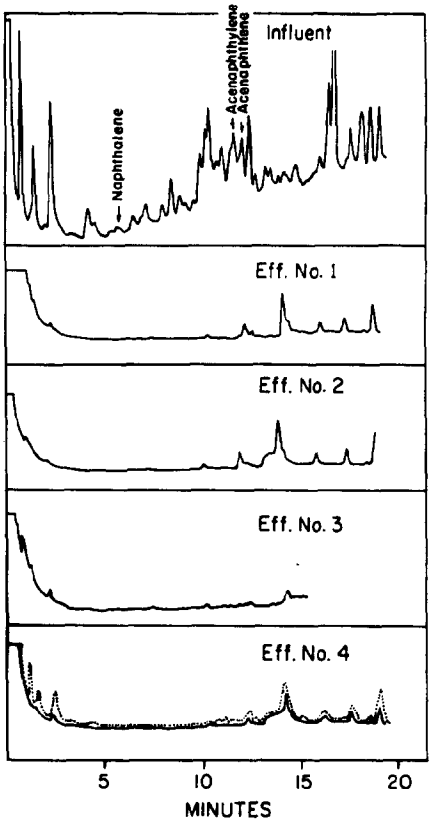


Fig. 7. GC-FID fingerprints of the phenols. (Influent is total; dotted line for effluent #4 is total; all other effluents (solid lines) are for soluble. Sampling date January 14, 1987.)

Fig. 8. GC-FID fingerprints for the polynuclear aromatic hydrocarbons. (See Fig. 7 for further details.)

found in units 3 and 4 which were supplied additional alkalinity. Nitrification was poorest in unit 1, the reactor with neither glucose nor alkalinity added to the feed. Nitrification, in the presence of cyanide, was also reported in another study [24]. As expected, denitrification was observed primarily in unit 2, the only unit that was provided sufficient conditions for denitrification [25,26]. While unit 1 had the potential for developing denitrification, it did not have sufficient carbon to drive the denitrification oriented reactions during the mixed react period because of the aerobic heterotrophic activity taking place during the five hour aerated react period. It should be noted, however, that this unit did develop denitrification capabilities during the three week initial acclimation period when nitrate was added to the feed and the reactors were maintained with a 22 hour mixed react.

TABLE 4

Removal of specific constituents in the SBR units (average values for January 14-16, 1987)

Contaminant		Influent[a]	Effluent[b]
COD, mg/l	Unit#1	500	35.0
	Unit#2	1700	34.0
	Unit#3	500	30.0
	Unit#4	1100	32.0
Ammonia, mg/l as N	Unit#1	132.2	11.9
	Unit#2	132.2	2.5
	Unit#3	100.2	1.7
	Unit#4	100.2	1.8
Cyanide, mg/l as CN	Unit#1	28.6	2.9
	Unit#2	28.6	3.8
	Unit#3	28.6	7.0
	Unit#4	28.6	2.6
Phenol, μg/l	Unit#1	13.1	0.5
	Unit#2	13.1	<0.14
	Unit#3	13.1	<0.14
	Unit#4	13.1	<0.14
Acenaphthene, μg/l	Unit#1	47.8	1.9
	Unit#2	47.8	2.3
	Unit#3	47.8	<1.8
	Unit#4	47.8	<1.8
Acenaphthylene, μg/l	Unit#1	99.5	<2.3
	Unit#2	99.5	<2.3
	Unit#3	99.5	<2.3
	Unit#4	99.5	<2.3
Naphthalene, μg/l	Unit#1	17.5	<1.8
	Unit#2	17.5	<1.8
	Unit#3	17.5	<1.8
	Unit#4	17.5	<1.8

[a]Total, except for ammonia.
[b]Soluble, except for cyanide.

GC analyses and cyanide determinations were conducted on the feed and effluent. Representative GC fingerprints for the phenols and the PNAs are given in Figs. 7 and 8 respectively. As can be seen from these figures, these compound groups were easily removed in all four reactors even though the reactors were operated with markedly different strategies. A comparison of soluble and total effluents can be seen in both Figures for SBR unit 4. GC analyses for the purgeable aromatics both for the leachate feed and the effluent resulted in undetectable levels of these compounds. As a result, their removal, if any, could not be determined. Detailed data for some of the target organics

and for cyanide, COD, and ammonia are presented in Table 4. The quantitative analyses for the phenols and the PNAs verify the qualitative trends shown in Figs. 7 and 8, and also agree with the aggressive COD removals reported. Cyanide removal was not complete. While the differences in cyanide removal between the four SBR units are difficult to attribute to the different operating conditions, it can be noted that unit 3, the system with the highest effluent cyanide, had no glucose added and was fully aerobic: conditions which are least favorable for denitrification.

Conclusions

Laboratory studies were conducted in order to determine the chemical and biological characteristics of leachates and soils from Harbor Point site. Leachate and soil mixtures served as the basic feed for bench scale SBRs operated by using various control strategies which were aimed at optimizing biological degradation of soil and leachate contaminants. The main conclusions drawn from this study are:

- Contaminated soils from Harbor Point site contain high variety of indigenous bacteria which can tolerate and degrade the toxic constituents present in the soils.
- Growth of these bacteria can be intensified in SBRs treating mixtures of contaminated leachates and soils.
- Removals of COD, phenol, and some PNA's were high under a broad range of SBR operation policies.
- Nitrification developed easily under all operational conditions and feed compositions tested.
- Cyanide did not interfere with biological activity and was satisfactory removed.
- The SBRs were shown to be flexible in operation and easily maintained on the constituents of the soils from Harbor Point, and are likely to be an excellent source of "specialized bacteria" for on-site soil remediation systems.

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