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MGP SOIL REMEDIATION IN A SLURRY-PHASE SYSTEM: A PILOT-SCALE TEST

by

Bill Y. Liu, Vipul J. Srivastava, J. Robert Paterek, and Salil P. Pradhan
Institute of Gas Technology
Chicago, Illinois 60616, U.S.A.

J. Ronald Pope
Elizabethtown Gas Company
Union, New Jersey 07083, U.S.A.

Thomas D. Hayes and David G. Linz
Gas Research Institute
Chicago, Illinois 60631, U.S.A.

Douglas E. Jerger
OHM Corporation
Findlay, Ohio 45839, U.S.A.

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ABSTRACT

An overall protocol for remediating manufactured gas plant (MGP) soils generally includes bench-scale evaluation of the technology, pilot-scale demonstration, and full-scale implementation. This paper summarizes the results of the bench-scale and pilot-scale study for treating an MGP soil with IGT's integrated Chemical/Biological Treatment (CBT) or Manufactured Gas Plant Remediation (MGP-REM) process in the slurry-phase mode of application. MGP soils are contaminated primarily with polynuclear aromatic hydrocarbons (PAHs).

An MGP site in New Jersey was the subject of this study. Soils from the site were used for the bench-scale evaluation of the integrated Chemical/Biological Treatment. The bench-scale study started with biological pre-treatment followed by chemical treatment and biological polishing. Results of the bench-scale study showed that this process was effective in degrading EPA Total as well as EPA Carcinogenic PAHs.

A test matrix was developed to assess this technology at a pilot-scale facility. The test matrix consisted of at least eight semi-continuous runs designed to evaluate the effects of PAH concentration, total solids concentration, residence time, and a number of

chemical reagent additions. An operating permit for 14 days was obtained to evaluate the process primarily for air emission data and secondarily for PAH degradation data. The PAH data showed that the MGP-REM process was very effective in degrading carcinogenic PAHs even under sub-optimal operating conditions. The field data also showed that the emissions of volatile organic compounds were well below the regulatory limits.

MGP SOIL REMEDIATION IN A SLURRY-PHASE SYSTEM: A PILOT-SCALE TEST

INTRODUCTION

Background

Plants for the manufacture of gas from coal and crude oil, referred to as manufactured gas plants (MGPs), have existed in many parts of the world for the past two centuries. These plants have resulted in the contamination of soils with polynuclear aromatic hydrocarbons (PAHs). Certain PAHs are known carcinogens and are an environmental hazard. As a result, those former MGP sites require cleanup to ensure environmental safety. Over 1500 MGP facilities have been identified to date in the United States [3].

Among the alternatives for MGP site remediation, bioremediation is considered to be one of the better options because its treatment end products, such as carbon dioxide and water, are environmentally safe. In addition, bioremediation is generally more economical than other processes, such as incineration or stabilization [2]. However, the bioremediation of many persistent contaminants such as the carcinogenic PAHs is often incomplete and/or insufficient.

Several major factors influence bioremediation of organic contaminants in soil: the low solubility and bioavailability of PAHs, soil characteristics, and the presence of metabolically effective microbial populations [4]. The low solubility of PAHs poses special problems for bioremediation. On one hand, low solubility is beneficial because it limits the migration of PAHs into groundwater. On the other hand, it limits microbial degradation by making these compounds unavailable to microorganisms that are capable of utilizing these PAHs in a bioremediation application. If the kinetics of PAH degradation are the same or better than the kinetics of solubilization, waste reduction can be achieved efficiently without increasing the possibility of further contaminating groundwater. Also, the management of water addition and the maintenance of zero water discharge during the treatment to avoid movement of contaminants into the groundwater are very important considerations.

Previous work suggests that the binding of PAHs to soil particles and/or the presence of PAHs within the micropores of soil aggregates may also limit their biodegradation by restricting availability of PAHs to microorganisms [5,6]. Thus, the soil mechanism that governs the rate and extent to which PAH biodegradation will be achieved is pollutant mass transfer from the soil and/or the bulk tar phase to the phase where soil microorganisms thrive. Generally, biodegradation of 2 and 3-ring PAHs occurs more readily than biodegradation of 4 to 6-ring PAHs.

Integrated Chemical/Biological Treatment, MGP-REM Process

To overcome these limitations of conventional bioremediation, the Institute of Gas Technology (IGT) has developed an integrated Chemical/Biological Treatment process (also known as the MGP-REM process).

The MGP-REM process helps to overcome these limitations by combining two complementary remedial techniques: 1) chemical oxidation as the pre-treatment for hard-to-degrade contaminants and 2) biological treatment using an aerobic biosystem. The MGP-REM process uses a mild chemical treatment with Fenton's reagent (hydrogen peroxide and ferrous salt) that produces hydroxyl radicals that start the chain reaction with the organic contaminants, resulting in modification and degradation of organics to biodegradable and environmentally benign products. These products and other organics are later degraded in the biological step. Results from bench-scale studies conducted with MGP soils show that the MGP-REM process is capable of enhancing the rate as well as the extent of PAH degradation. The results indicate that when a soil is dominated by 4 to 6-ring PAHs, chemical treatment is performed as a pre-treatment step. However, when a soil is dominated by 2 to 3-ring PAHs, chemical treatment is used as a co-treatment step. Added cycles of combined chemical and biological treatments could be implemented as deemed necessary to reduce the end point further.

Slurry-Phase Application of the MGP-REM Process

Slurry-phase biological treatment is a relatively new development for the remediation of hazardous wastes, and it offers significant advantages over other bioremediation approaches currently in use. It is highly effective for a variety of wastes, and the rate of degradation is up to 10 times faster than land treatment [5,6]. Addition of Fenton's reagent to the bioslurry reactor has been shown to be helpful in bench-scale reactor studies and solid-phase bioremediation studies. Fenton's reagent, as mentioned above, chemically oxidizes high-molecular-weight organic compounds into smaller and more soluble organic compounds, thereby enhancing bioavailability. Addition of Fenton's reagent generates free hydroxyl radicals. The free radicals then oxidize PAHs to simpler and biodegradable aromatic hydrocarbons [4]. In addition to hydrocarbon oxidation, Fenton's reagent has the ability to disaggregate silt and clay particles -- similar to, but stronger than, the effect of intense shear mixing provided in the bioslurry system.

There are two major benefits of Fenton's reagent. The first benefit is the enhancement of removal rates and the subsequent reduction of treatment time and cost. Biodegradation is enhanced because simpler, more soluble organic compounds are produced as a result of the attack of Fenton's reagent chemicals on target organic compounds. The second benefit is the reduction in the concentration of the insoluble compounds by virtue of oxidizing these compounds into more soluble, biologically susceptible products. The ultimate benefit of using Fenton's reagent in a bioslurry application would be to enhance biotreatability of some persistent compounds, thus allowing a final concentration lower than that achieved by using biological treatment alone.

The MGP-REM process can be applied in a variety of modes. The process has been demonstrated successfully in a field-scale land treatment application using MGP soils [6]. Also, researchers have concluded that chemical oxidation -- namely, Fenton's reagent oxidation -- can enhance *in-situ* biodegradation of hydrocarbons in groundwater aquifers [1,4]. In addition to its application to solid-phase and *in-situ* bioremediation, the MGP-REM process can also be used in slurry-phase bioremediation [7].

Slurry-Phase Bioremediation Protocol

In slurry-phase bioremediation, excavated contaminated soil is normally screened to remove debris and large objects before being placed in an onsite stirred-tank reactor where the soil is combined with water to form a slurry. The solids content of the slurry depends on the type of soil, the type of mixing and aeration equipment available, and the rates of contaminant removal that need to be achieved. Typical solids contents range from 10% to 40% by weight. If required, nutrients, microorganisms, or surfactants are added to the slurry to enhance the biodegradation process. The residence time in the reactor varies with the slurry matrix; the physical/chemical nature of the contaminants, including concentration; and the biodegradability of the contaminants. Once biodegradation of the contaminants is completed, the treated slurry is typically dewatered, and the cleaned soil can be reused depending on the regulatory constraints.

To use a slurry reactor effectively in a soil remediation project, some pre-treatment is required to remove all oversize material. The excavated contaminated soil is first sieved through a 2-inch screen to remove objects, such as rocks or bricks. The screened soil is then slurried in an attrition scrubber to wash contaminants from the larger soil particles. After this, it passes through a shaker screen to remove any gravel, debris, and other oversize material. The soil passing through the shaker screen is then fed into the slurry reactor. Most of the sand and gravel will be clean after the washing step and can be discharged. The finer materials and the excess wash water that cannot be recycled are then passed into a series of bioslurry reactors.

Total hydraulic residence time in these reactors will vary depending on the nature of the organic contaminants, their concentrations, and the cleanup level required. The soil slurry is finally dewatered in a thickener, the effluent of which is decanted and recycled for the next batch of treatment.

IGT/GRI Treatability Protocol for the MGP-REM Process

If site characteristics indicate that bioremediation might be appropriate for any part of the site, the soil treatability potential is evaluated by performing what is termed a treatability study. A treatability protocol, summarized in Figure 1, has been developed to determine the potential of conventional bioremediation as well as the MGP-REM process within a 2 to 3-month period [7]. Phase I of the three-phase protocol consists of the following four major steps:

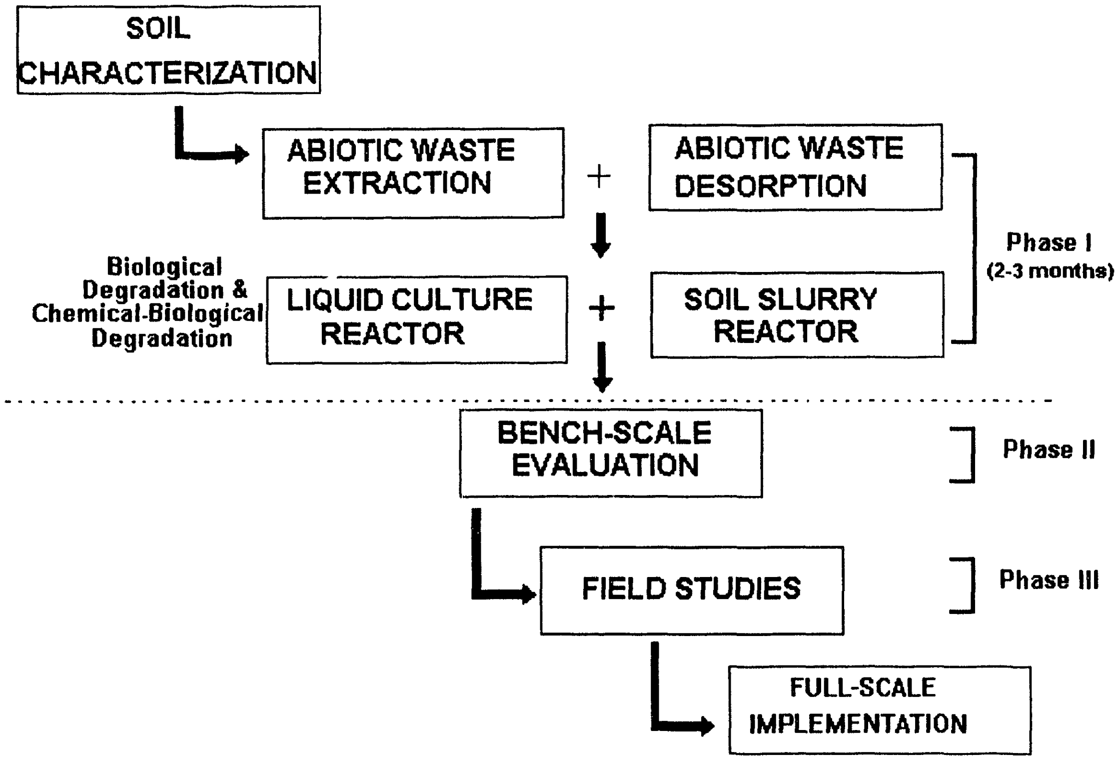


Figure 1. TREATABILITY PROTOCOL FOR EFFECTIVE REMEDIATION OF MGP SOILS

1. Soil Characterization -- The composite soil samples are characterized for physical, chemical, microbiological, and geotechnical properties.

2. Abiotic Waste Extraction -- The soil samples are extracted with Soxhlet extraction using EPA protocols and an organic solvent such as dichloromethane or a 1:1 mixture of acetone and hexane. This provides a measurement of the maximum amount of PAHs bound to the solid matrix. The soil sample is also extracted with methanol or ethanol to assess the PAH level easily available for biodegradation.

3. Abiotic Waste Desorption -- Soil samples are gently mixed in an aqueous solution and solutions of surfactants and co-solvents. Periodic water samples are measured for PAHs desorbed from the solid matrix into the aqueous solution. This provides an assessment of bioavailability of solids-bound PAHs for microbial degradation. In other words, the data can be used to assess the extent of mass-transfer problems to be encountered during the biodegradation.

4. Soil-Slurry Reactor Studies -- Contaminated soils at 10% to 30% solids concentrations are treated in a variety of conditions representing controls,

conventional bioremediation, and the MGP-REM process for 4 to 8 weeks. Samples are periodically analyzed for PAHs bound to the solid matrix as well as those present in the aqueous phase to determine the rate and extent of PAH degradation in each bioreactor. The off-gas is often measured to determine the extent of volatilization. Test results have shown that for most MGP soils, the extent of volatilization is insignificant. The results of these investigations provide an indication of how effective the process is likely to be for a given site and the extent of improvement anticipated as a result of chemical treatment, surfactant addition, etc.

The results of Phase I of the treatability protocol can be used to assess the potential of bioremediation. If the results are encouraging, Phase II is initiated where the process is optimized in soil-slurry systems. Soil pans and soil columns are used for evaluating solid-phase and *in-situ* bioremediation, respectively. Phase III is the field-scale demonstration prior to the full-scale implementation.

This paper describes a complete treatability protocol for remediating soils from an MGP site in New Jersey. The study started with soil characterization and bench-scale studies (Phase I) of soils from the site. After completion of the bench-scale study, the results were analyzed and showed great potential for slurry-phase bioremediation (Phase II). The step that followed was a pilot-scale slurry-phase bioremediation (Phase III).

The pilot-scale field study followed all guidelines set by the New Jersey Department of Environment and Energy (NJDEPE). The goals of the treatment are to meet the New Jersey soil standards for non-residential areas and to limit VOC (volatile organic compound) emissions to below 0.5 pound per hour.

METHODS

Bench-Scale Study

The bench-scale slurry reactor study was performed in 10-liter stainless steel reactors. Each reactor was equipped with an air sparger and mechanical mixer to ensure complete mixing of the slurry contents. The soil-slurry experiments employed a mixture of contaminated soil and water with solids content of about 10%. The treatment scheme evaluated the integrated Chemical/Biological Treatment of MGP soils for PAH removal. The test started with 3 weeks of biological treatment, followed by Fenton's reagent chemical treatment with 2.5% hydrogen peroxide, and then biological treatment for final polishing. The total operation time was 34 days. The bench-scale study was very successful in MGP soil treatment. As a result, a pilot-scale study was planned for field demonstration.

Pilot-Scale Study

Figure 2 is the flow diagram of this pilot-scale study, in which the slurry-tank system is divided to three independent, but interconnected, reactor units. Each unit can be operated either as the bioreactor or the chemical reactor. One of the three units is used as the slurry reactor. In the slurry reactor, the sieved slurry is mixed with water to reach a desired solids content. After reaching the designed slurry content, slurry is pumped to the respective reactors for either biological or chemical treatment. To prevent volatile organic compound emissions, off-gas from the reactor system is collected and treated by two granular activated carbon (GAC) canisters operated in series. The GAC can adsorb any VOCs released during the process and eliminates the possibility of air pollution. The off-gas monitoring devices are connected prior to the GAC canisters.

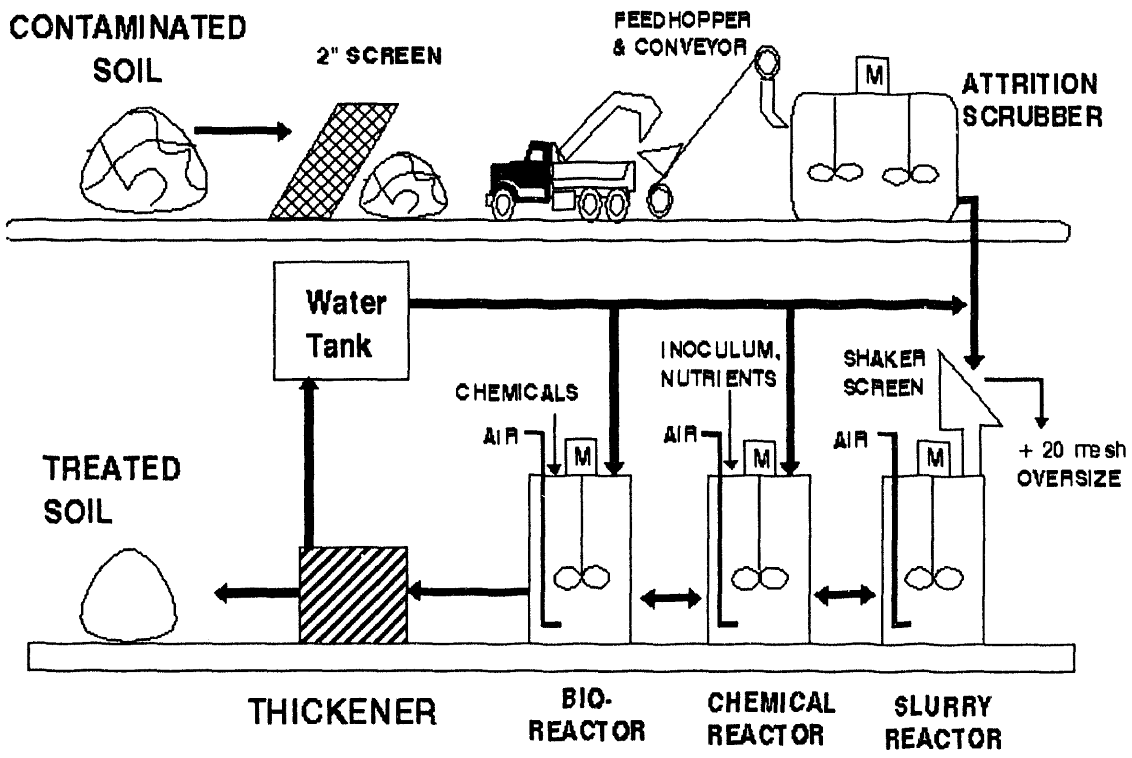


Figure 2. SLURRY-PHASE BIOREMEDIATION DIAGRAM

<u>Operating Protocol for Test No. 1</u>

As shown in Figure 2, excavated soil was screened to remove objects greater than 2 inches. The screened soil was then transported to an attrition scrubber where soil was mixed with water to make a 50% soil slurry. Slurry from the attrition scrubber was sent to the 20-mesh vibrating screen to remove particles greater than 1 mm, which were sent to the reject stockpile.

The slurry reactor, chemical reactor, and biological reactor each have a capacity of 2100 gallons. Slurry passing through the screen was diluted to 10% solids in the slurry reactor in preparation for the following chemical/biological treatment. In the chemical treatment, 105 gallons of 50% hydrogen peroxide (H_2O_2) were added to the chemical reactor to achieve 2.5% volume H_2O_2/volume slurry. A high concentration of H_2O_2 was selected primarily to obtain the worst-case scenario for air emission. Normally, 1%

H_2O_2 has been found to be appropriate. Commercial-grade $FeSO_4 \cdot 7H_2O$ was added to achieve a predetermined Fe^{+2} concentration during chemical treatment.

The slurry-reactor system was operated in a batch mode. As described in the work plan approved by NJDEPE, the test started with soil of low PAH concentration and approximately 10% solids concentration. Upon receipt of the full operating permit, subsequent tests will involve varying operating conditions. See Table 1 for the work plan.

Table 1. TREATMENT PLAN FOR PILOT-SCALE STUDY

Parameter	Treatment Range
PAHs, mg/kg	100 - 15,000
Solids % in Slurry Reactor	10 - 30
Slurry Residence Time in Slurry Reactor, days	5 - 15
Hydrogen Peroxide, % vol/vol	1.0 - 2.5

Only solids were treated with Fenton's reagent during the study because PAHs are insoluble and should remain in the solid matrix. In Test 1, slurry was allowed to settle for 4 hours prior to chemical treatment, and the supernatant (about half of the total volume) was aerated in the slurry tank for later addition in the biological reactor. This procedure served as a way to oxidize only the PAHs in soil and keep the microbial population in the supernatant unaffected. After the chemical oxidation was completed, which was indicated by the complete utilization of added hydrogen peroxide, the supernatant was added back to the solid slurry to provide the microbial population. During the chemical treatment of slurry, the supernatant remained aerated and was monitored for dissolved oxygen (DO) and pH. During the biological treatment, the slurry reactor was regularly monitored for pH, temperature, DO, and nutrient levels.

During the first test run, an automatic sampler took emission samples at 1-hour intervals for BTEX (benzene, toluene, ethylbenzene, and xylenes) and PAH analyses to ensure no emission to the atmosphere.

PAH Analysis

PAHs were determined in the samples using a modified EPA SW-846 Method 8100 analysis of Soxhlet extracts. Soxhlet extraction was performed by EPA SW-846 Method 3540 using a 1:1 mixture of acetone and hexane as the extraction solvent. The modifications of Method 8100 involved the use of mesitylene (1,3,5-trimethylbenzene) as an internal standard and detection using ion-trap detecting gas chromatography/mass spectrometry. A 30-meter-long PET-5 (Supelco) column (0.25-mm ID and 0.25-μm film) was installed in a Perkin Elmer 8320 gas chromatograph fitted with an ion-trap detector.

The injector temperature was 325°C, and the following temperature program was used: 40°C for 4 minutes, followed by an increase to 300°C at a rate of 10°C per minute, isothermal at 300°C for 1 minute.

RESULTS AND DISCUSSION

Bench-Scale Study

Initial characterization of the soil used for the bench-scale study is given in Table 2. Results of the bench-scale batch study are summarized in Table 3, in which the initial PAH concentration and the concentrations after 7 and 34 days of treatment were compared with the non-residential soil standards set by NJDEPE. Table 2 shows that the slurry reactor can reduce total PAHs from about 1000 to lower than 50 mg/kg in 34 days of treatment, which also included start-up. More importantly, the carcinogenic PAHs were reduced to less than 15 mg/kg from their original concentration of about 230 mg/kg. This represents approximately 93% reduction in the concentration of carcinogenic PAHs. These results imply that the combination of Chemical/Biological Treatment in the slurry-phase bioremediation can treat MGP soils to meet regulatory requirements and deserves serious consideration for further study and/or full-scale implementation.

Pilot-Scale Study

The pilot-scale test plan consisted of several experiments evaluating the process efficiency as a function of PAH concentration, total soil solids concentration, residence time, chemical pre-treatment reagent concentration, and the number of cycles of chemical treatment. However, due to certain regulatory confusion and constraints, the operating permit by NJDEPE was issued for only 14 days. (Initially the host company was led to believe that no permit was required to operate this experimental test facility.) The temporary permit required subjecting the soil to the entire treatment train scheme during this period so that appropriate measurement of air emissions could be made. It was believed that if the data showed emission of VOCs to be within the regulatory limits, a full permit would be issued by the NJDEPE. As a result, the objectives of this 14-day operating permit, which included the system start-up, were to --

- Assess the emission of volatile organics within the treatment scheme
- Evaluate the PAH-degradation efficiency.

Test 1 started with a 6-day biological pre-treatment with bioaugmentation, followed by chemical treatment, and another 6-day biological polishing. A final chemical treatment was conducted on the last day of the operation. Characterization of the soil after excavation, which began only a day prior to the start of the pilot plant, indicated that the PAH concentration was 100 mg/kg, much lower than the expected concentration of

Table 2. SOIL CHARACTERIZATION FOR BENCH-SCALE STUDY

Parameter	Value
Total PAHs, mg/kg	1014
Carcinogenic PAHs, mg/kg	231
Noncarcinogenic PAHs, mg/kg	783
pH	5.4
Organic Mater %	5
Phosphorus, ppm	25
Potassium, ppm	70
Nitrate-N, mg/kg	5.5
Sand %	81
Fines, %	19

Table 3. BENCH-SCALE BATCH TREATMENT OF MGP SOIL WITH NO PROCESS OPTIMIZATION (All Units in mg/kg)

	Day 0	Day 7	Day 34	NJDEPE Standards
Total PAHs	1013.7	204.4	44.8	54,257.32
Noncarcinogenic PAHs	782.5	93.3	30.1	54,200
Carcinogenic PAHs	**231.2**	**111.1**	**14.7**	**57.32**

500 mg/kg and the 1000 mg/kg used for the bench-scale study. A target PAH concentration of approximately 500 mg/kg was selected to allow microorganisms to get acclimated for biological treatment. Table 4 outlines the soil characterization.

Table 4. FIELD SOIL CHARACTERIZATION FOR PILOT-SCALE STUDY

Parameter	Value
Total PAHs, mg/kg	100
Carcinogenic PAHs, mg/kg	39
Noncarcinogenic PAHs, mg/kg	61
pH	6.0

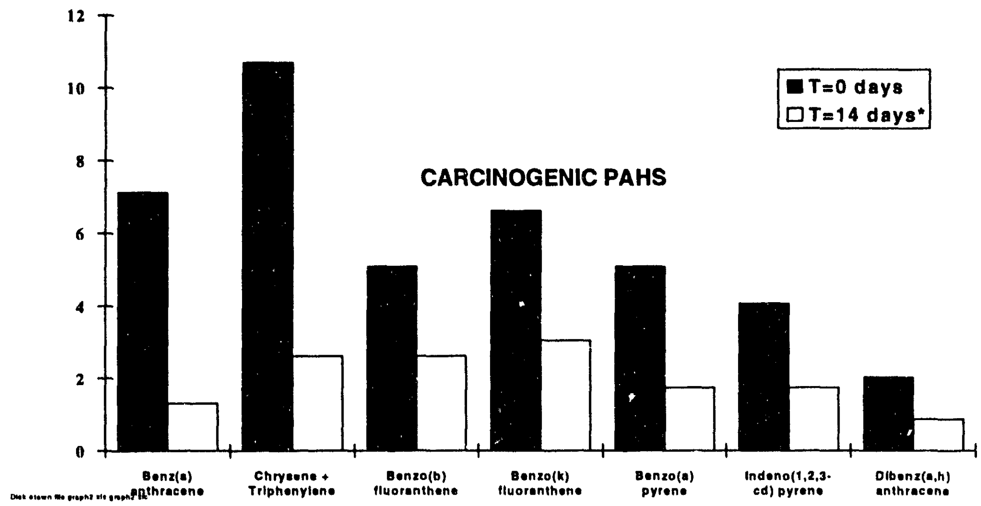
Table 5 summarizes the NJDEPE standards for non-residential soils and the performance of the MGP-REM process in the pilot study. In spite of the limited time for acclimating the biological system, the results indicate that the process can effectively remediate the soil to meet the standards. Most carcinogenic PAHs were removed to levels lower than the NJDEPE standards after 14 days of treatment. The efficiency of carcinogenic PAH reduction was approximately 65%. Figure 3 shows the initial and final concentration of all the carcinogenic PAHs monitored during this test period. It is believed that if the total PAH concentration were higher, it would have provided a sufficient carbon source for a healthy microbial population and would have further enhanced the PAH reduction efficiency. In the case of this test soil, the microbial population appears to have been starved. These results are especially encouraging in light of the fact that the process was not operated under the optimal conditions.

Table 5. PILOT-SCALE BIOSLURRY TEST NO. 1 RESULTS*
(All Units in mg/kg)

Parameter	Original Conc. Day 0	Final Conc. Day 14	NJDEPE Standards
Total PAHs	100	26.5	54257.32
Benz(a)anthracene	7.1	1.3	4
Chrysene + Triphenylene	10.7	2.6	40
Benzo(b)fluoranthene	5.1	2.6	4
Benzo(k)fluoranthene	6.6	3	4
Benzo(a)pyrene	5.1	1.7	0.66
Indeno(1,2,3-c,d)pyrene	4.1	1.7	4
Dibenzo(a,h)anthracene	2	0.9	0.66
Carcinogenic PAHs	**40.7**	**13.8**	**57.32**

* Includes system start-up -- normally 1-2 weeks for bioslurry when the initial microbial count is low, as in this test soil.

In addition to the PAH results, air monitoring results also show that this process is environmentally safe. After 14 days of operation, only a negligible amount of BTEX was detected in the monitoring system occasionally. About 90% of the readings were below detection limits. The total amount of VOCs released during the process was well below the 0.5-pound per hour limit set by NJDEPE.



* Includes system start-up -- normally 1 - 2 weeks for bioslurry

Figure 3. PILOT-SCALE BIOSLURRY TEST NO. 1 -- CARCINOGENIC PAHs (All Units in mg/kg)

In summary, results of the first test run demonstrate good treatment efficiency despite the fact that the process was not operated at optimal conditions. The process can reduce carcinogenic PAHs to meet NJDEPE standards with minimal VOC emissions. As a result, this pilot study should be able to continue next year after a permit for complete pilot-scale operation is received from NJDEPE by the host company.

Treatment Cost Estimate

A preliminary economic evaluation of the full-scale treatment of contaminated MGP soils was also performed. The proposed treatment system includes soil screening and slurry preparation, slurry treatment, and slurry dewatering. Preliminary costs for biological treatment of approximately 10,000 cubic yards of material range from $220 to $230 per cubic yard of material treated by slurry-phase treatment. The use of Fenton's reagent causes an acceleration of the biodegradation kinetics and improves the treatment efficiency. As a result, the treatment time is expected to be reduced to about one-third the time required by biological treatment alone. Although it is optimistic to assume that the biological treatment alone would reduce PAHs to the treatment end points, the treatment cost will be similar to that of the MGP-REM process.

The total soil treatment cost for the MGP-REM process depends upon the required dosage of Fenton's reagent. It ranges from approximately $220 per cubic yard for 0.5% reagent to approximately $250 per cubic yard for 2% reagent. The normal dosage of hydrogen peroxide application is between 0.5% and 1.5%. A more detailed economic evaluation of the integrated treatment process will be performed at the completion of the pilot-scale demonstration project.

CONCLUSIONS

Several significant conclusions can be drawn from this study:

1. The bench-scale study demonstrated that the integrated Chemical/Biological Treatment can effectively treat MGP soil contaminated with PAHs to meet regulatory requirements in slurry-phase bioremediation. Results of the bench-scale study and the pilot-scale test showed that total carcinogenic PAHs can be reduced to below 15 mg/kg.

2. Despite the fact that the system was not operated under the optimal conditions, the first run of the pilot study confirmed the finding of the bench-scale study -- that an integrated chemical/biological process can remediate MGP soils to meet standards for most carcinogenic PAHs in slurry-phase bioremediation. Because only one test was conducted, more tests are recommended to fully understand this process.

3. Air monitoring from the first run of the pilot study did not detect any significant VOC emissions, suggesting that the pilot-scale operation will not cause any problem for air pollution and is safe for the environment.

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