

Enhanced Intrinsic Bioremediation of Hydrocarbons Using an Oxygen-Releasing Compound

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An "oxygen barrier" was formed by depositing an oxygen-releasing compound in a series of wells that were placed perpendicular to the direction of groundwater flow at a site in Belen, New Mexico. The objective was to enhance the intrinsic bioremediation of dissolved phase BTEX contamination in the aquifer and to quantify the results. The oxygen was supplied by a controlled release formulation of magnesium peroxide called Oxygen Release Compound (ORC®), a virtually insoluble powder that is packaged in polyester filter socks. The areal distributions of the initial concentrations of dissolved oxygen and BTEX were measured and compared to the concentration changes at various times in the first 93 days of system operation. The concomitant reduction in BTEX can be seen in a series of contour plots. In 93 days, dissolved oxygen had dispersed at least 20-feet downgradient from the ORC source wells based on the pattern of decreasing BTEX concentrations.

Traditional methods for the remediation of source areas, and the control of downgradient movement of contamination, are being increasingly replaced or augmented by bioremediation and other emerging technologies; this shift has resulted from issues of efficiency and economics (Norris et al., 1995). Furthermore, there has been increasing interest in approaching the wide variety of affected sites with a risk reduction philosophy (ASTM RBCA, 1994). In concert with these changes, intrinsic bioremediation is becoming a well-established and scientifically defensible strategy for risk reduction or full cleanup. At sites where total reliance on intrinsic methods is questionable because of the existence of limiting factors, certain enhancement practices become attractive.

In groundwater remediation systems, dissolved oxygen can be the major factor limiting biodegradation (Salanitro, 1993). Where this occurs, the use of supplemental oxygen becomes an imperative. Traditionally, supplemental oxygen is provided by active systems that include air sparging and the continuous addition of hydrogen peroxide (Brown, 1995; Norris, 1994). Recently, solid peroxygen compounds, such as sodium

percarbonate, calcium peroxide, and magnesium peroxide, have become useful as a basis for more passive oxygen delivery systems. These materials can be deposited where needed for continuous release of oxygen in soil or groundwater systems (Davis-Hoover et al., 1991; Davis et al., 1995).

Materials like calcium and magnesium peroxide are virtually insoluble and can be deposited by various means, using wells, trenches, or by direct injection by push-point technology into an aquifer to make contact with contaminated groundwater. When contacted with water, ordinary calcium and magnesium peroxide will release oxygen for several weeks, and proprietary formulations of magnesium peroxide, such as ORC, can release oxygen for several months to a year. The rate control feature is a function of the synthesis of the molecular matrix and not achieved by a coating process. While the technology can be applied to several other solid peroxygens, only calcium and magnesium peroxide are feasible to use in bioremediation. The higher solubility and pH of calcium peroxide directed efforts on magnesium peroxide as the peroxygen of choice in the preparation of ORC.

Since the solid peroxygens simply release oxygen when hydrated, they are ultimately converted to their respective hydroxides. ORC therefore becomes ordinary magnesium hydroxide which, like magnesium peroxide, is also virtually insoluble. ORC is packaged in exchangeable filter socks. Since an ORC sock lasts approximately six months, as shown in **Exhibit 1**, this operation can be performed as little as twice a year. Therefore, the use of ORC is the basis for passive treatment of subsurface hydrocarbon contamination with biologically based source treatments and migration barriers.

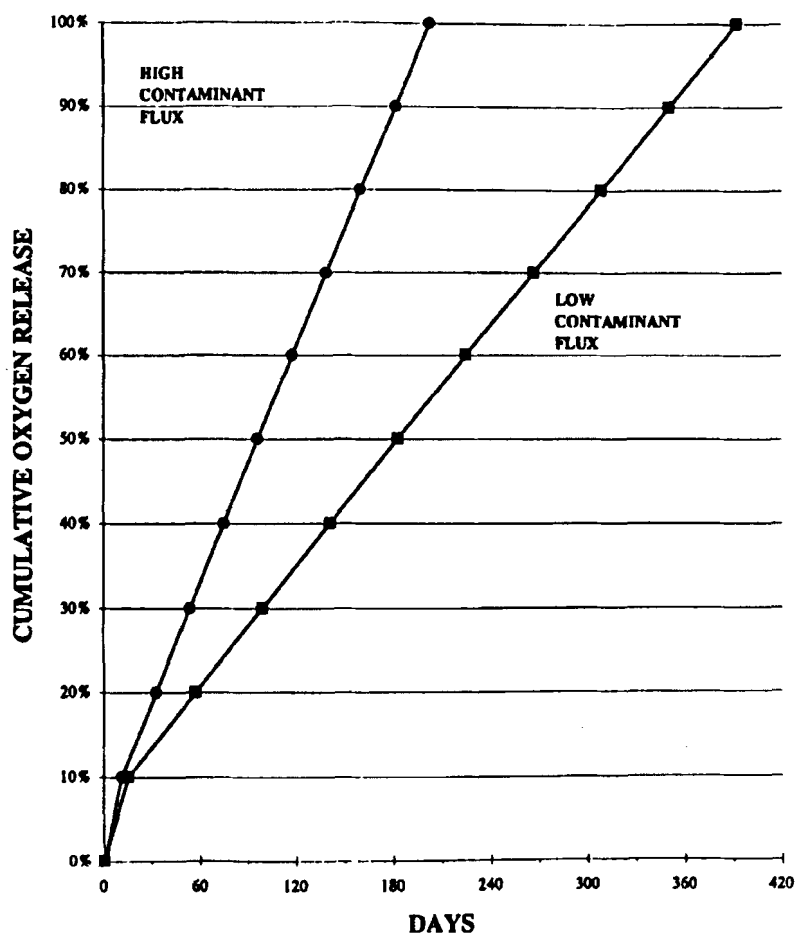
The first implementations of oxygen barrier technology with ORC were conducted at the University of Waterloo (Bianchi-Mosquera et al., 1994) and at North Carolina State University (Kao and Borden, 1994). These studies established proof-of-concept for the use of ORC in the formation of a passive migration barrier. A quantitative field demonstration (QFD) of ORC's ability to function as the basis for a passive groundwater bioremediation system was conducted at an abandoned service station site in Belen, New Mexico, from April 1, 1995 to July 5, 1995. **Exhibit 2** is the overall site plan which shows the QFD region and the location of the excavated source area. **Exhibit 3** details the placement of the oxygen barrier and the array of monitoring points. This article describes the changes in areal distribution of the concentrations of dissolved oxygen and BTEX (benzene, toluene, ethylbenzene, and xylene) as they occurred at various times in the first 93 days of system operation.

SITE HISTORY

Initial contamination at the site was discovered in the winter of 1989. An unknown amount of gasoline had leaked into the subsurface for an unknown length of time. A site assessment was performed in the spring of 1993 and indicated that petroleum hydrocarbon contamination had affected soil and groundwater (ATEC, 1993). The contaminated groundwater had migrated off-site to the southeast. In June 1994, a hydrogeological investigation was conducted and indicated that the groundwater contami-

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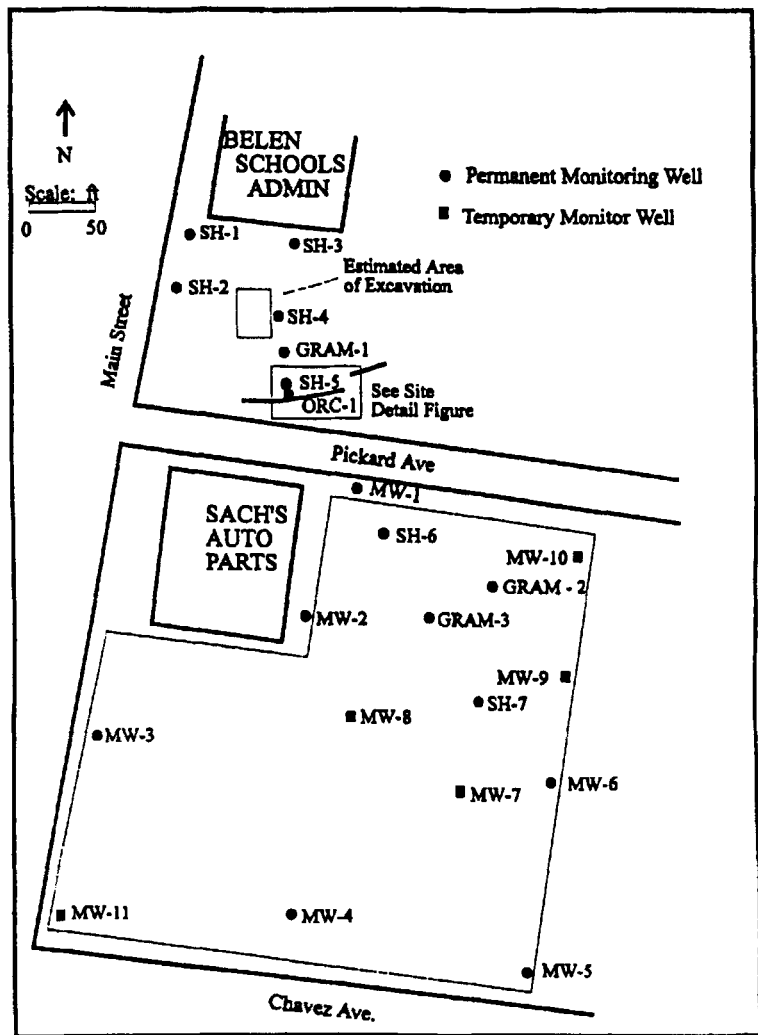
Exhibit 1. Oxygen Release Kinetics of ORC



nation extended downgradient to SH-6 and MW2. The vertical extent of the contamination was approximately 15 feet below ground surface (bgs).

In September 1994, a study with ORC was conducted (GRAM, 1994). A single six-inch diameter PVC source well was installed for the placement of ORC and 26 monitoring points were placed at various positions downgradient to monitor changes in dissolved oxygen (DO) and BTEX. After four months of operation it was apparent that oxygen was being released from ORC, that it was being distributed downgradient, and that BTEX was being remediated. Based on these results, the UST Bureau of the New Mexico Environment Department approved a full-scale ORC mediated oxygen barrier.

Exhibit 2. Overall Site Plan

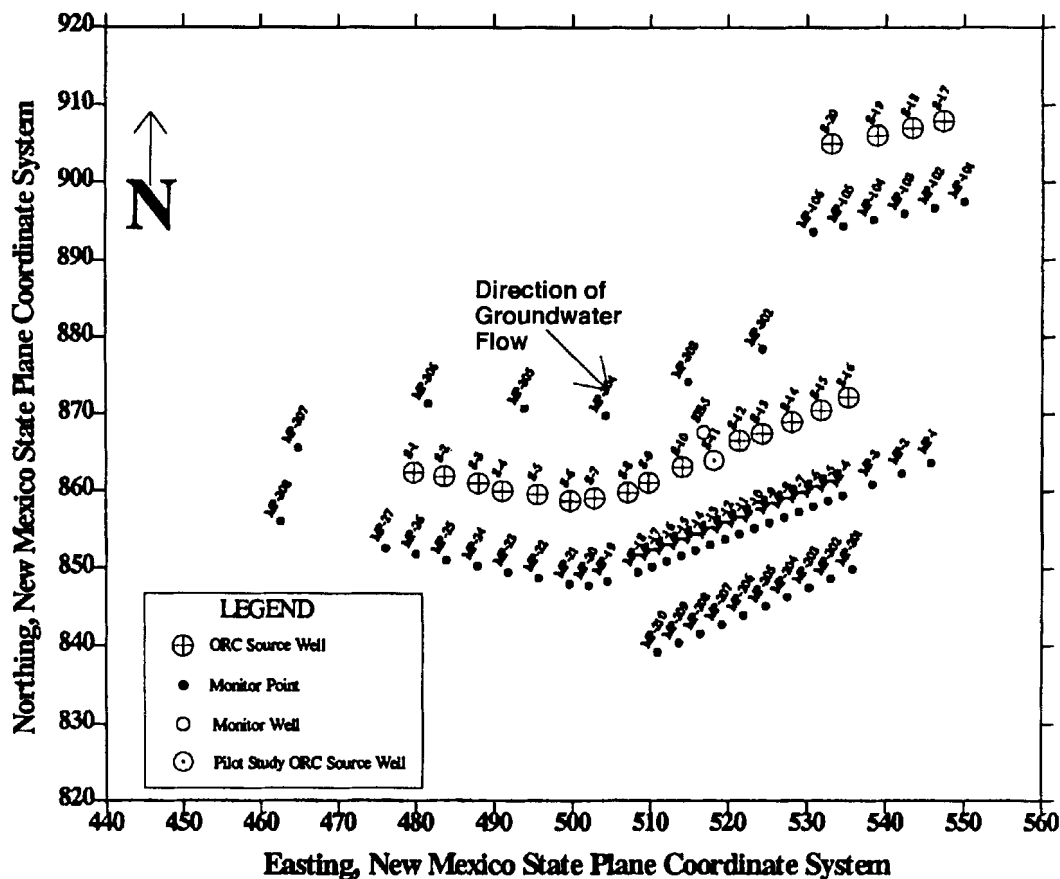


The full barrier was designed around the pilot source well. In March 1995, 19 additional PVC wells, plus an additional array of monitoring points, were installed (**Exhibit 3**). Contaminated soil in the vicinity of the former tank pit was excavated to minimize recharge of the aquifer with additional contamination.

SITE DESCRIPTION

The site is situated in the lower section of the Albuquerque-Belen Basin

Exhibit 3. Detailed Site Map



within the Rio Grande Rift. The rift is bordered by the Lucero uplift and the Rio Puerco fault zone on the west and by the Sandia/Manzano uplift to the east. The sediments that exist beneath the site are of alluvial origin and reflect the geomorphic setting. The soils at the site consist of interbedded clays, sand, and gravel.

The aquifer is shallow and unconfined and is comprised mainly of well-sorted sands. The depth to groundwater is approximately five feet bgs. The average groundwater gradient at the site was 0.0016 in February 1994 and 0.0015 in November 1994, both toward the south-southeast. The hydraulic conductivity from SH6 was measured to be 1.5.5 ft/day as determined by a slug test (Geohydrology Associates, 1994). Assuming the average hydraulic gradient and a range of possible effective porosities from 25

percent to 30 percent, the range of interstitial velocity at the site was 0.10 to 0.11 ft/day. This would account for 10 to 11 feet travel distance during the 93 days the system was being monitored.

DESIGN AND CONSTRUCTION OF THE SOURCE AND MONITORING NETWORK

Exhibit 3 illustrates the ORC oxygen barrier which is comprised of 20 ORC source wells and a variety of monitoring points. Due to the existence of an overhead power line on the east side of the site, the oxygen barrier was split into two unequal sections as shown. The main objectives of the two lines of ORC wells were to create an oxygen barrier to control the forward movement of plume and to draw the leading edge back toward the source.

The placement of the ORC source wells is perpendicular to the prevailing groundwater flow direction. One row of monitoring points (MP) is located ten-feet downgradient of the source wells. These include MP-4 to MP-18, which are spaced two feet on center, and the series consisting of MP-1 to MP-4, MP-18 to MP-27, and MP-101 to MP-106, which are spaced four feet on center. Another series of monitoring points, MP-201 to MP-210, is 20-feet downgradient with a spacing of three feet on center.

The source wells have three KVA miniature shield points fastened to the outside of the well casings. The shield point is made of aluminum and is six inches long, 1/4 inch in diameter, and has 0.010 inch slots. The source wells had these probes placed at 7 feet, 14 feet, and 21 feet bgs. The average vertical position of these probes are referred to as the C-zone, B-zone, and A-zone, respectively.

MATERIAL AND METHODS

ORC filter socks were purchased from the manufacturer (Regenesis Bioremediation Products, San Juan Capistrano, California). The socks are composed of a tight weave polyester, which is the same material used for well screen filter sock. Each sock, containing a one-to-one mixture of ORC and #90 silica sand, weighs 12.6 pounds and is 4 percent oxygen by weight.

Water levels were measured at the site prior to sampling. One-eighth-inch-diameter Teflon line tubes were connected to each of the probes. A peristaltic pump was used for water sampling and the first 100 mL of sample drawn up was discarded. The second 100 mL drawn was measured for temperature, pH, and electrical conductivity. A 300 mL BOD (biochemical oxygen demand) bottle was then filled as the sample for dissolved oxygen and a 40 mL VOA was filled and preserved with mercuric chloride for the Ohmicron BTEX immunoassay.

Six sampling points were selected as QA/QC points, and an additional 40 mL VOA was filled for outside laboratory analysis. BTEX was analyzed using EPA Method 8020. The laboratory BTEX readings were correlated with the significantly lower-cost field immunoassay tests, enabling the collection of a much larger number of samples than would have otherwise been possible. All samples indicated were collected during 11 sampling events; before the ORC was installed at Day 0 and at Days 3, 5, 9, 12, 20, 30, 47, 60, 75, and 93. Monitoring wells were sampled using dedicated HDPE tubing.

The main objectives of the two lines of ORC wells were to create an oxygen barrier to control the forward movement of plume and to draw the leading edge back toward the source.

FIELD SCREENING

Field screening of environmental samples was accomplished using three different methods. A Hydac digital tester was used to measure pH, conductivity, and temperature of the samples in the field. Dissolved oxygen was measured using Hach modified Winkler digital titration method in the field. BTEX was screened using Ohmicron RAPID Assay Total BTEX kits upon returning from the field. Each of the methods are discussed in detail below.

Quality Assurance/Quality Control (QA/QC). QA/QC of field screening procedures were accomplished by running duplicates of 10 percent of all samples collected for all field screening methods.

Hydac Meter. A Hydac digital tester was used to measure pH, conductivity, and temperature of all water samples collected in the field. The Hydac tester was calibrated in the field before the start of each sampling event.

Dissolved Oxygen. DO was measured using the Hach modified Winkler digital titration method. The azide modification of the Winkler Method is the standard test for dissolved oxygen. In the analysis, Mn^{2+} (manganous ion) reacts with the dissolved oxygen present in the alkaline solution to form a Mn^{4+} oxide/hydroxide flocculent. Azide is added at this time to suppress interference from any nitrate present which would react with the iodide. The solution is then acidified and the manganese floc is reduced by iodide to produce Mn^{2+} and free iodine as I_3 ($I_2 + I$ in solution) in proportion to the oxygen concentration. The iodine gives the clear supernatant a brown color. Thiosulfate is then used to titrate the iodine to a colorless end point. The samples were collected in 300 mL BOD bottles, and the DO was measured in the field. The thiosulfate titrate was checked before each sampling event by performing an accuracy check using an Iodate-Iodide Standard Solution equivalent to 10 mg/L dissolved oxygen.

BTEX. BTEX field screening was performed using the Ohmicron RAPID Assay Total BTEX kits, which are immunoassay-based technologies. The antibodies are attached to microscopic particles and are mixed with the water samples. A known antigen competes with any BTEX present for binding sites, and the BTEX concentration becomes inversely proportional to a color change induced by the known antigen complex. The results are quantitative within the limits of the calibration, 0.02–3.0 ppm, and qualitative outside of the range. Results greater than the highest calibration point are qualitative because it is not known what happens to the calibration curve in that range.

Quantification is made with a RPA-1 RAPID Analyzer, which was calibrated for each batch of samples. Calibration curves were run using duplicates of four standards, 0.0, 0.09, 0.35, and 3.0 mg/L. A control sample was also included as part of each batch. Replicate samples were sent to the laboratory for analysis by EPA Method 8020, as noted.

Field screening of environmental samples was accomplished using three different methods.

RESULTS AND DISCUSSION

The results will be presented as a time series of contour plots and mass curves for BTEX and DO. The data interpretation followed decisions with respect to the use of certain contouring algorithms, geostatistical methods, and selection of boundaries. The areal distribution of BTEX and DO was contoured using two kriging algorithms. GEOEAS, an environmental geostatistical package, was used to examine the spatial correlation features of the data sets (U.S. EPA, 1988). An exponential model fit the variograms for BTEX and DO quite adequately. The main purpose of implementing GEOEAS was to provide the most realistic representation across the computational domain where sampling information was unavailable. The kriging algorithm within SURFER was used to contour the data across the site for the purpose of generating mass estimates.

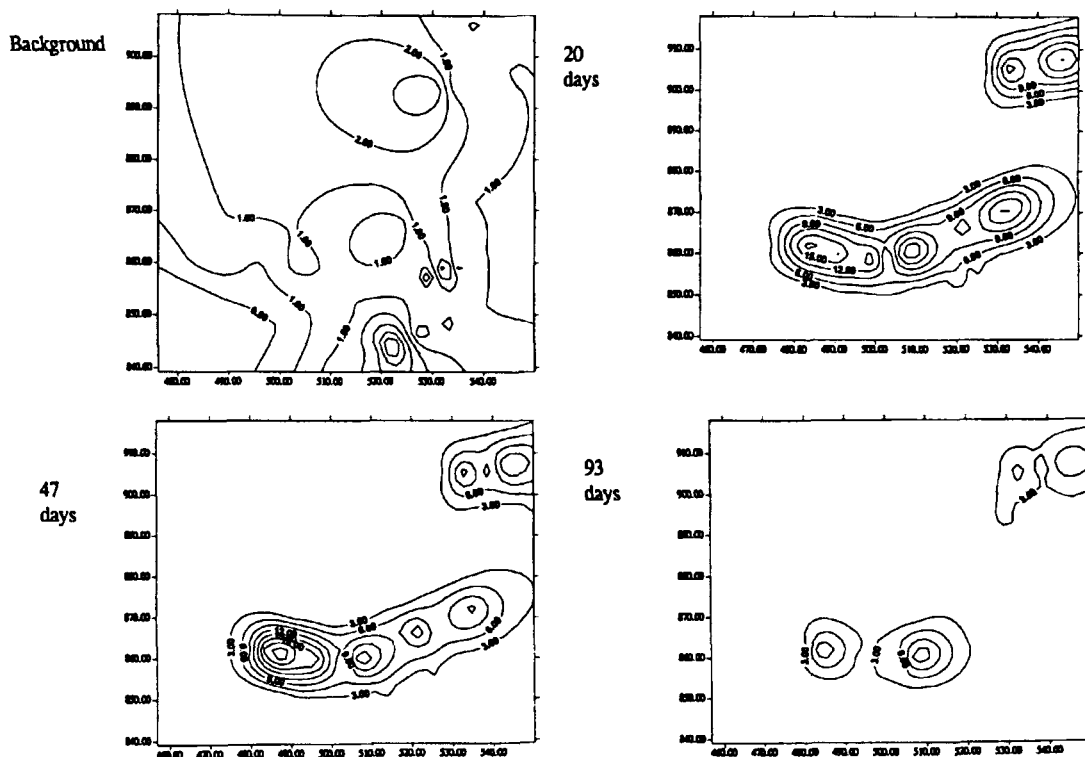
Contour plots of oxygen and BTEX were made for the 11 sampling events; however, it is not necessary or practical to show them all in this article. Four contour plots of BTEX and DO were selected from the total series based on the following rationale. The initial (background) and final contour plot were selected for obvious reasons. The first intermediate time, 20 days, was selected because the oxygen mass was established at its peak relative to two previous sampling events and there was significant BTEX degradation from background conditions. The second intermediate time, 47 days, was selected because of a slight apparent increase in BTEX concentration in the well samples, coupled with the first sign of appreciable oxygen mass depletion.

Exhibit 4 is a plot of the contoured DO concentrations at the sampling events of days 0, 20, 47, and 93. The average background concentration of DO in the domain was approximately 1 mg/L with a maximum of concentration of 4.54 mg/L at MP-106. The need for adding oxygen is clear. Salanitro (1993) states that there may be a minimum threshold concentration for which BTEX degradation is slowed, even at levels greater than 1 to 2 mg/L of DO. The contour plots at 20 and 47 days show a buildup of dissolved oxygen around the barriers with some skewness in the direction of flow. A small amount of fingering is apparent from the complete time history of data at 515 to 530 feet (MP-6 to MP-14). There is obvious oxygen migration to the monitoring wells ten-feet downgradient and less evidence of it reaching the well series located 20-feet downgradient. The 3 mg/L contour line was chosen as the threshold concentration for new oxygen. DO concentrations fluctuate above the background levels in well MP-201 to MP-210; however, they are generally below 3 mg/L. The significant decrease in the size of the oxygen plume at 93 days, as well as a lower maximum concentration, is a result of consumption due to the remediation of BTEX.

Exhibit 5 presents the same series of contour plots for BTEX. The background concentration is approximately 2 mg/L of BTEX, and most of the mass appears to be located upgradient of the barriers. There is a shift in the areal distribution of BTEX throughout the time periods presented. There is a region of higher concentration near the east side of the large barrier and a region of lower concentration near the west side of the barrier at Day 20. At Day 47, there appears to be a separate plume forming toward

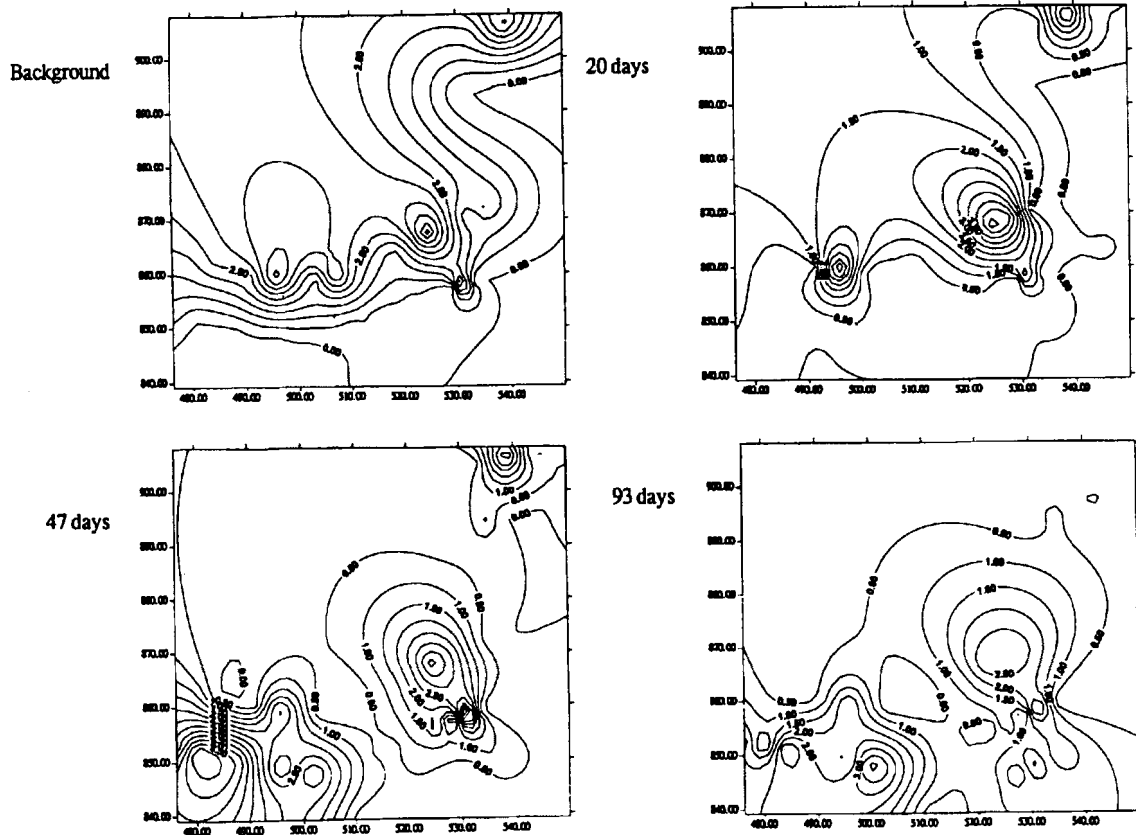
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Exhibit 4. Contour Plots of DO



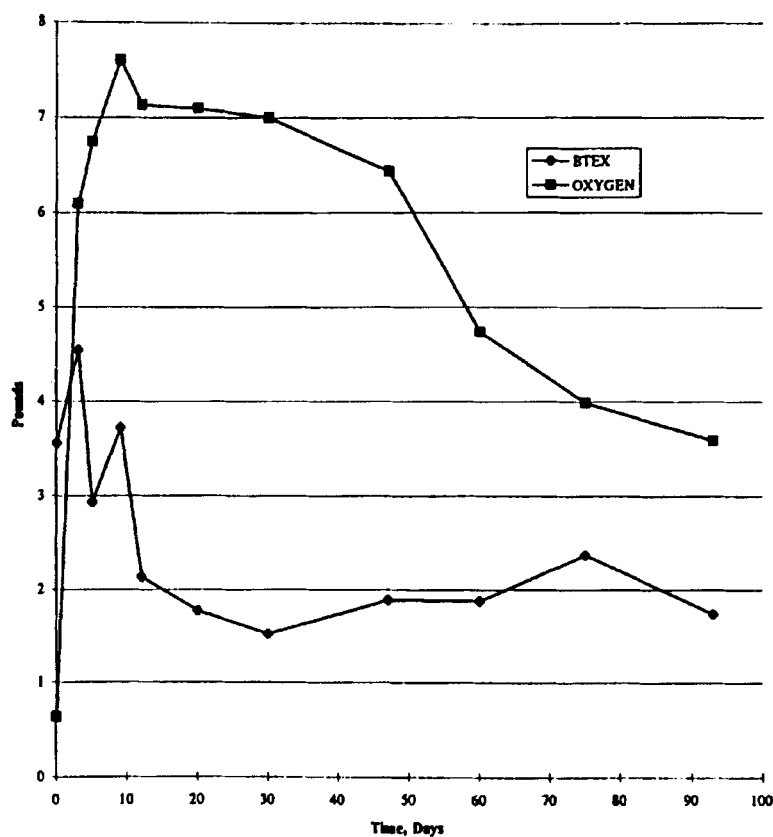
the east side of the large barrier as well as a pinched off plume near the small barrier. The increase in concentration near the west side of the large barrier most likely results from a recharge of BTEX to the aquifer. At Day 93, there appear to be two plumes remaining, one near the east side of the large barrier and the other toward the west side. The plume located near the small barrier contains less than 0.50 mg/L of BTEX.

The macroscopic behavior of BTEX and DO in the system is illustrated succinctly with the mass curves shown in **Exhibit 6**. The mass was calculated by assuming the C-zone concentrations are depth-averaged and that there is a total porosity of 37.5 percent, with a saturated thickness of 17 feet. The volume was calculated using the routines in SURFER, and the unit conversions were performed in a spreadsheet. The DO mass reached its peak in approximately nine days, remained relatively constant for the next month, and then started to decline. The total DO mass in the system

Exhibit 5. Contour Plots of Dissolved BTEX

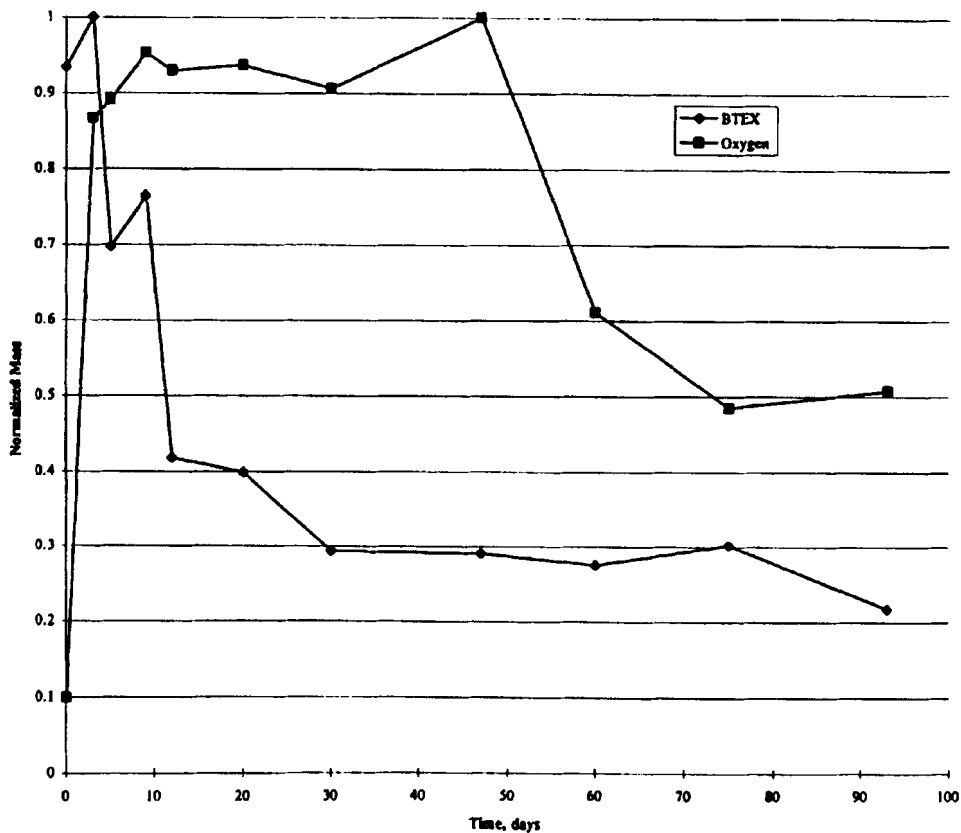
increased by about an order of magnitude and remained at approximately five times its initial mass at Day 93.

The effect of the increase in DO mass in the system is seen by examining the total BTEX mass curve. A reduction in BTEX mass of approximately 50 percent occurred in the first 30 days without a significant change in the maximum oxygen mass. As a probable result of recharge of BTEX into the aquifer, BTEX mass increased slightly from Day 47 to Day 75, as confirmed by the relatively rapid decrease in oxygen mass from Day 47 to Day 75. The new recharge of BTEX essentially increased the kinetics of oxygen release by exerting a large demand. The comparison of the two curves simultaneously presents a textbook description of aerobic bioremediation dynamics.

Exhibit 6. Mass Curve of DO and BTEX


As a sideline to the data analysis presented thus far, consideration should be given to the normalized mass curves for the large barrier in cross section. The A-zone, B-zone, and C-zone oxygen and BTEX concentrations were used to estimate the mass across a vertical slice of the aquifer. **Exhibit 7** is a plot of the normalized mass curves of DO and BTEX at the large barrier. The normalized curves are necessary due to the large oxygen mass compared to the small BTEX mass. Thus, the same general behavior can be observed at the wall, as already seen in the C-zone data. With respect to Exhibit 6, while the oxygen concentration at the barrier represents the vast majority of the oxygen in the system, it is also important to note that the BTEX concentrations away from the barrier represent the vast majority of the BTEX mass in the system. One hypothesis to explain the increase in BTEX mass from Day 30 to 47 is a lowering of the water table between

Exhibit 7. Normalized Mass Curves of DO and BTEX

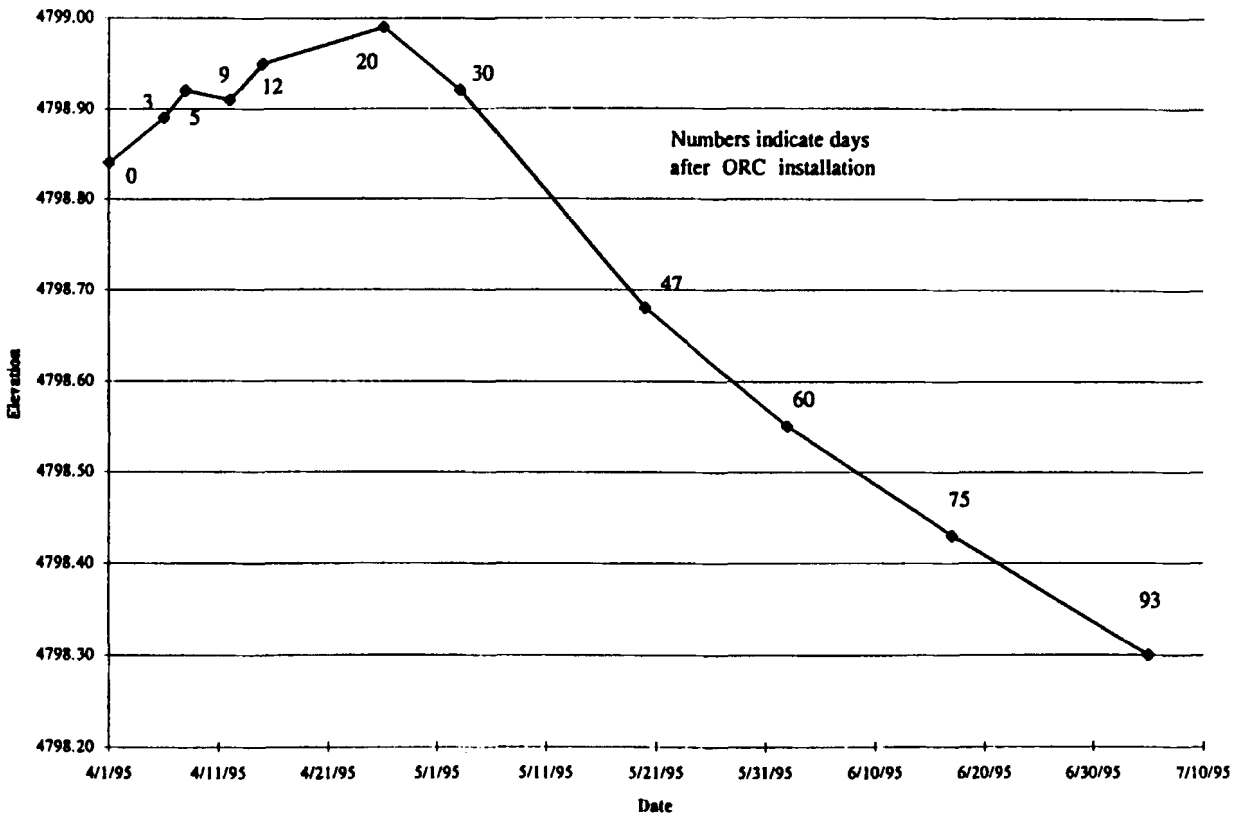


the two time periods. **Exhibit 8** is a time history of water levels at the large barrier over the duration of data collection. The water table dropped approximately 0.24 ft in 17 days or 0.014 ft/day. The decline continued for the remainder of the QFD (Day 47 to Day 93) at an average rate of 0.0081 ft/day (75 percent slower). The average increase in water table elevation for the first 20 days was 0.0075 ft./day. The fixed position of the C-zone probes and the decrease in sampling volume above the probe support the notion that an increase in BTEX concentrations for days 30 through 47 likely was caused by a lowering of the water table.

MICRO-SCALE VERSUS MACRO-SCALE OXYGEN DYNAMICS

Although there may be some issues related to measuring the dissolved oxygen concentrations at a micro scale near the barrier, there is no question

Exhibit 8. Time History of Water Levels at the Large Barrier



that the mass curve for oxygen presented in Exhibit 6 represents a macroscopic average condition in the system. The kinetics of oxygen release must be combined with the demand for oxygen exerted by the BTEX, the recharge of groundwater passing by the source wells, and the local mixing characteristics such as those caused by well screen effects.

Exhibit 1 represents the laboratory-generated kinetics of oxygen release in the form of cumulative oxygen release from ORC versus time. If the ORC were placed in the wells with no BTEX, a relatively constant source of oxygen would be supplied to the aquifer for a period of approximately one year. The rate of mass transfer of release would be largely a function of the groundwater velocity flowing past the source wells. The average groundwater velocity at the site is approximately 10 to 11 feet for a 93-day time period or 0.11 ft/day. This velocity is relatively

slow and therefore would lead us to conclude that molecular diffusion plays a fairly significant role for ORC release (Peclet number = 0.21; assuming a particle diameter of 0.5 mm and a molecular diffusion coefficient of 1 cm²/day).

There is a supply of BTEX upgradient of the source wells for the duration of the QFD and, considering this demand on the kinetics of oxygen removal from the ORC, there are two conclusions: (1) there was enough oxygen released to cause an increase in dissolved oxygen concentrations in monitoring wells located 10- and 20-feet downgradient and (2) as the mass curve of oxygen in Exhibit 6 suggests, there was significant BTEX demand, resulting in a shortened life for the ORC. These conclusions are evident without the use of a groundwater transport model, which accounts for advection, hydrodynamic dispersion, sorption, and biodegradation.

HYDROCARBON BACTERIAL CONCENTRATION ACROSS THE LARGE BARRIER

On August 15, 1995, samples were collected from selected source wells and monitoring points across the site. **Exhibit 9** is a plot of the suspended hydrocarbon degrading bacterial concentration starting at MP-303 and ending at MP-203. There is an increase in concentration of approximately three orders of magnitude across the barrier. Although there is no background data to support the contention of ORC-induced bacterial growth, such large concentrations of aerobic hydrocarbon degrading bacteria would not be thriving in the absence of oxygen.

CONCLUSIONS

The following conclusions can be made regarding the performance of an ORC-mediated oxygen barrier in Belen, New Mexico:

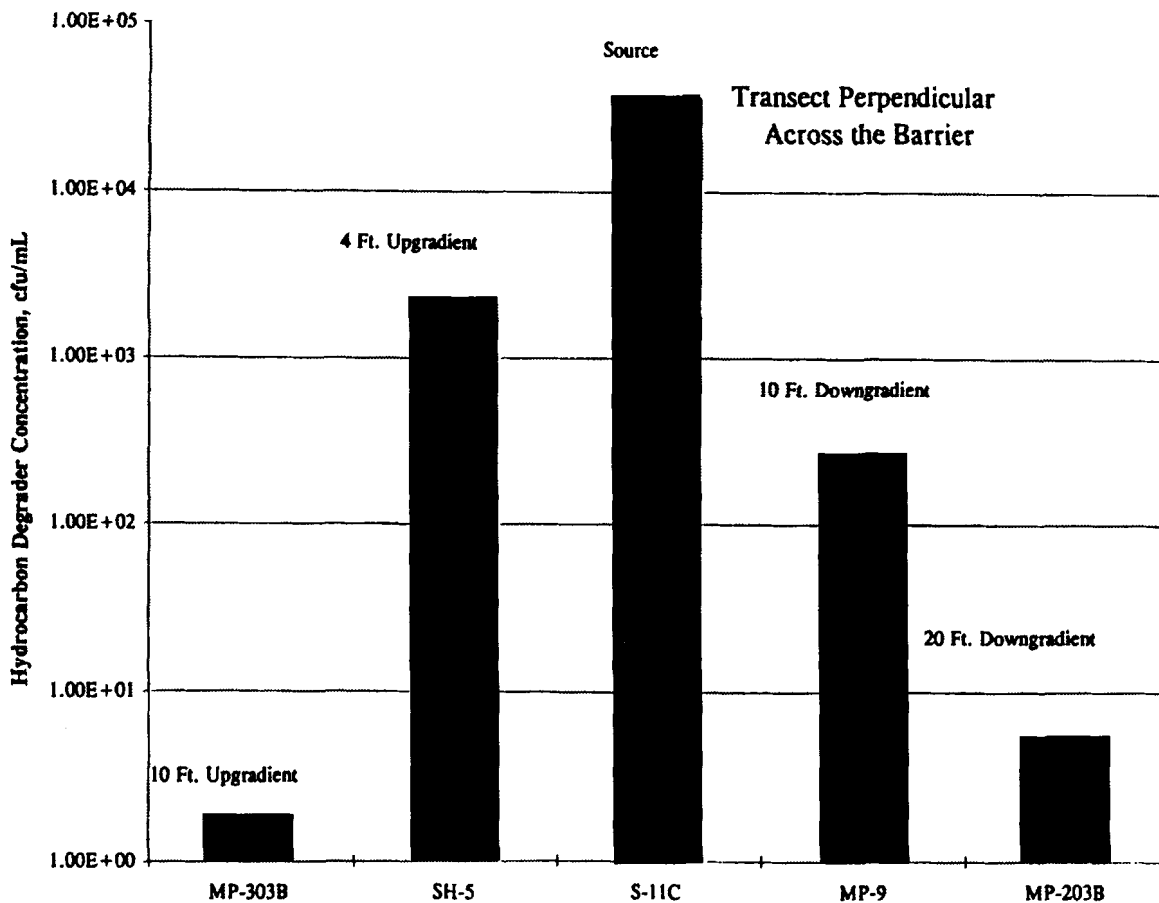
- The oxygen mass increased an order of magnitude near the leading edge of the dissolved-phase BTEX plume.
- The increase in dissolved oxygen concentration had a significant impact on the dissolved-phase BTEX plume.
- An ORC-mediated oxygen barrier appears to be one means of achieving enhanced intrinsic aerobic bioremediation and overall risk reduction of dissolved-phase hydrocarbon plumes in groundwater.

DISCUSSION

This work is the largest of several field studies that have demonstrated the use of ORC in passive bioremediation systems and have highlighted the potential of these methods for the prevention of the migration, and/or reducing the source, of aerobically biodegradable contaminants. These systems are alternatives to slurry wall or pump-and-treat installations where intrinsic remediation is not applicable or reliable. ORC-based systems can also be viewed as a lightly engineered and cost-effective method of enhancing intrinsic remediation. ORC systems can be used as a sole treatment method

This work is the largest of several field studies that have demonstrated the use of ORC in passive bioremediation systems.

Exhibit 9. Plot of the Hydrocarbon Degrading Bacterial Concentration



or be employed in conjunction with more conventional technologies that might be planned for or that already exist on a site. These efforts may serve to supplement limits that exist with air-sparging arrays, or pump-and-treat systems, or to function as a polishing step, as when, for example, pump-and-treat systems reach a treatment asymptote.

The use of ORC has been focused on typical petroleum hydrocarbon spills such as those found at gas stations; however, a wide range of other problems are candidates for solutions based on passive oxygen release systems. With respect to DNAPLs, it is hoped that efforts may soon begin in areas such as: (1) the control of TCE and PCE by co-metabolic strategies that require oxygen; (2) the prevention of the anaerobic formation of vinyl

chloride from TCE and PCE; and (3) the control of vinyl chloride by aerobic biodegradation once it is formed.

ACKNOWLEDGMENTS

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