

Advanced Oxidation Technologies for the Treatment of
Contaminated Groundwater

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ABSTRACT

This paper presents information on two pilot-field applications of advanced oxidation technologies for contaminated groundwater with organics. The two UV/oxidation technologies were developed by Ultrox International of Santa Ana, California and Peroxidation Systems, Inc. of Tucson, Arizona. The Ultrox technology was demonstrated in 1989 with the U.S. Environmental Protection Agency's Superfund Innovative Technology Evaluation (SITE) program at the Lorentz Barrel and Drum (LB&D) site in San Jose, California. Peroxidation Systems technology was applied at the Old O-Field site located within the Aberdeen Proving Ground, in Maryland.

The Ultrox system was evaluated for its effectiveness in treating the volatile organic compounds (VOCs) present in groundwater at the LB&D site. Achievement of VOC removals were greater than 90 percent under best operating conditions at that time. Most VOCs were removed through Chemical oxidation, however, for a few VOCs, stripping also contributed toward removal. The treated groundwater met the applicable discharge standards at 95 percent confidence level for discharge into a local waterway. There were no harmful air emissions to the atmosphere from the Ultrox system, which is equipped with an off-gas treatment unit.

The Peroxidation Systems technology, achieved contaminant removal efficiencies of about 96 percent, and the treated water met the federal maximum contaminant levels for drinking water.

The information presented includes a description of the technologies, factors affecting the technologies, and results from the two pilot-scale studies of the UV/oxidation treatment system applications.

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INTRODUCTION

The Superfund Innovative Technology Evaluation (SITE) program was created in 1986 to provide information on alternative and innovative technologies. The SITE program also generates reliable performance and cost data for these technologies from each demonstration, as well as a broader range of data on each process from non-SITE activities. Therefore, technologies to destroy, treat, detoxify, reduce mobility, volume or recycle hazardous waste materials are being developed and demonstrated within the Environmental Protection Agency (EPA).

Most conventional treatment processes, such as air stripping, steam stripping, carbon adsorption, and biological treatment, are quite effective in treating water contaminated with organics, but have certain limitations. These limitations include transfer of contaminants from one medium (water) to another (air or carbon) when using stripping and adsorption. In addition biological treatment processes generate sludge that may require further treatment and disposal.

Most of these limitations can be eliminated by chemical oxidation processes using ozone, hydrogen peroxide or some other conventional oxidant. However, because of kinetic limitations chemical oxidation by conventional oxidants has yet to become a competitive treatment option. Several studies have shown that the kinetic limitations could be overcome by using hydroxyl radicals to carry out the oxidation reactions (1-3). The hydroxyl radicals are known to be less selective in carrying out oxidation reactions and have much higher rate constants compared to ozone, hydrogen peroxide, or ultraviolet (UV) radiation.

Hydroxyl radicals are generated by the combined use of (1) UV radiation and hydrogen peroxide, (2) UV radiation and ozone, or (3) ozone and hydrogen peroxide. These processes are commonly referred to as "advanced oxidation processes" or, when UV radiation is used to generate hydroxyl radicals, "UV/oxidation technologies."

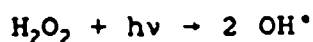
This paper briefly describes (1) the chemistry of UV/oxidation technologies and factors affecting these technologies and (2) the results from pilot-field scale operations of two UV/oxidation systems. These technologies differ in design and application, and therefore present unique features that demonstrate the efficiencies of both processes under different conditions. It is noted that Ultrox International has already been demonstrated under the EPA's SITE program in 1989 and has successfully been in the market for several years. Likewise, the Peroxidation Systems technology has been available in the market for a number of years, the technology, however, will be in the SITE demonstration program in 1992 at the Lawrence Livermore National Laboratory, in Livermore, California.

UV/OXIDATION PROCESS CHEMISTRY

The generation of hydroxyl radicals is the key principle of UV/oxidation technologies through UV photolysis of hydrogen peroxide and/or ozone. When UV radiation is used to photolyze hydrogen peroxide or ozone, the UV radiation may also photolyze some organic contaminants. A summary of the chemistry of UV/oxidation technologies is given below. More information of the chemical reactions that occur during these applications is available elsewhere (4).

UV Photolysis of Hydrogen Peroxide

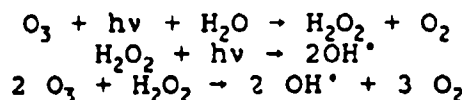
Generation of hydroxyl radicals by UV photolysis of hydrogen peroxide may be described by the following equation:



Most commercial applications are using low-pressure mercury vapor UV lamps to produce UV radiation. The maximum absorbance of UV radiation by hydrogen peroxide occurs at about 220 nanometers (nm). However, the dominant emission wavelength of low-pressure mercury vapor UV lamps is at about 254 nm. Also, the molar extinction coefficient of hydrogen peroxide at 254 nm is low, 19.6 liters per mole-centimeter ($\text{M}^{-1} \text{cm}^{-1}$). Because of the low molar extinction coefficient, a high concentration of hydrogen peroxide is needed in the medium to generate sufficient hydroxyl radicals.

UV Photolysis of Ozone

UV photolysis of ozone in water yields hydrogen peroxide, which in turn reacts with UV radiation or ozone to form hydroxyl radicals as shown below.



Because the molar extinction coefficient of ozone is $3,300 \text{ M}^{-1} \text{cm}^{-1}$ at 254 nm, the UV photolysis of ozone is not expected to have the same limitation as that of hydrogen peroxide when low-pressure mercury vapor UV lamps are used.

FACTORS INFLUENCING PERFORMANCE

Factors influencing the performance of a UV/oxidation technology can be grouped into three categories: (1) waste characteristics, (2) operating parameters, and (3) maintenance requirements. Following is a brief discussion of these factors.

Waste Characteristics

The type of contaminants to be treated influence the removal efficiencies of the UV/oxidation processes. For example, organics with double bonds, such as trichloroethene (TCE), tetrachloroethene (PCE), and vinyl chloride, and aromatic compounds, such as phenol, toluene, benzene, and xylene, are easily removed because they are readily oxidized. In systems that use ozone, organics without double bonds and with high Henry's law constants, such as 1,1-dichloroethane (1,1-DCA) and 1,1,1-trichloroethane (1,1,1-TCA), are also removed. However, because they are difficult to oxidize their removal is primarily due to stripping. Organics without double bonds and with low Henry's law constants, such as diethylamine and 1,4-dioxane, would be difficult to remove because they are not easily oxidized or stripped.

UV/oxidation technologies are intended for the destruction of organic contaminants, other species that consume oxidants are considered an additional load for the system. These species are called scavengers and include anions such as bicarbonate, carbonate, sulfide, nitrite, bromide, and cyanide. Metals present in their reduced states, such as trivalent chromium, ferrous iron, manganous ion, and several others, are likely to be oxidized. These reduced metals, in addition to acting as scavengers, cause additional concerns. For example, trivalent chromium is oxidized to more toxic hexavalent chromium, and ferrous iron and manganous ions are oxidized to less soluble forms, which precipitate in the reactor and can cause UV lamp scaling and suspended solids formation. Nontarget organics (for example, humic compounds) could also act as scavengers. Other parameters such as suspended solids and oil and grease would reduce UV transmission, thereby decreasing the treatment efficiency. For these reasons, pretreatment may be required for proper functioning of UV/oxidation units depending on the waste characteristics.

Operating Parameters

Operating parameters are those parameters that are varied during the treatment process to achieve the desired treatment efficiencies. Such parameters include hydraulic retention time, ozone dose, hydrogen peroxide dose, UV lamp intensity, influent pH level, and gas-to-liquid flow rate ratio.

In general, increasing the hydraulic retention time will increase treatment efficiency up to a certain point. At this point, the system tends to proceed toward equilibrium, and increasing the hydraulic retention time no longer increases treatment efficiency.

The higher the dose of oxidants, the better the treatment rate. However, the molar ratio of the oxidant doses must be considered. For example, when treating water containing TCE and PCE, maximum removals were observed when the molar ratio of ozone dose to hydrogen peroxide dose was equal to two, and the removals were significantly less when the ratio was not equal to two. In this

case, the expected stiochiometry for pure water agreed with the molar ratio at which optimum removal was observed; however, several factors may influence the molar ratio (1). These factors are summarized as follows:

Hydrogen peroxide can act as a free radical scavenger itself, thereby decreasing the hydroxyl radical concentration if it is present in excess.

Ozone can react directly with hydroxyl radicals, consuming both ozone and hydroxyl radicals.

Ozone and hydroxyl radicals may be consumed by scavengers present in the water being treated.

Therefore, the optimum proportion of the oxidants for maximum removals cannot be predetermined. Instead, the proportion needs to be determined for the waste under consideration using pilot- or bench-scale treatability tests.

In addition to photolyzing hydrogen peroxide and ozone to generate hydroxyl radicals, the UV radiation may also photolyze some organic contaminants, such as PCE, aromatic halides, and pesticides, increasing the contaminant removal.

If water has bicarbonate and carbonate alkalinity at a level greater than 400 milligrams per liter (mg/L) as calcium carbonate, lowering the pH to a range of 4 to 6 should improve the treatment efficiency. Low pH decreases the concentration of these scavengers by shifting the equilibrium toward carbonic acid. If the carbonate and bicarbonate alkalinity is low, then a high pH should improve the treatment efficiency. High pH favors hydroxyl radical formation because of the reaction between ozone and the hydroxyl ion.

The ozone gas flow rate can also influence treatment rate. In practice, once the ozone dose is selected, several combinations of ozone gas phase concentration and ozone gas flow rate can be applied. According to Venosa and Opatken (5), the ratio of gas flow rate to liquid flow rate will dictate the hydraulic characteristics of the reactor, as shown in Figure 1. This figure shows that, at low gas-to-liquid flow rate ratios, the mixing regime in a reactor is close to that of a plug flow reactor (shown as Curve A); whereas at high ratios, the reactor mixing regime is close to that of a mixed reactor (shown as Curve C). For reactions with a positive reaction order, plug flow mixing characteristics offer higher treatment rate than mixed reactor mixing characteristics (6). Since most reactions have a positive reaction order, low gas-to-liquid flow rate ratios should be considered. In addition to increasing the treatment rate, low gas-to-liquid flow rate ratios reduce stripping of volatile organics.

Maintenance Requirements

Regular maintenance by trained personnel is essential for a successful treatment operation. The following components require maintenance: (1) ozonation system, (2) UV lamp assembly, (3) ozone decomposer unit, and (4) miscellaneous components. A brief summary of the maintenance requirements for these components is presented in Table 1.

PILOT-FIELD STUDY ONE

The Ultrox technology demonstration occurred in February, 1989 at the Lorentz Barrel and Drum (LB&D) site in San Jose, California, through an agreement between EPA's Region IX, Ultrox International, and the EPA's SITE program. The LB&D site was used primarily for drum recycling operations from about 1947 to 1987. The drums contained residual aqueous wastes, organic solvents, acids, metal oxides, and oils. The preliminary site assessment report for the LB&D site showed that the groundwater and soil were contaminated with organics and metals.

The upper aquifer at the LB&D site was selected as the waste stream for evaluation of the UV/oxidation technology. Samples from the shallow aquifer were collected in December 1988 which indicated that volatile organic compounds (VOCs) were present in the groundwater. VOCs detected at high levels included TCE (280 to 920 μ /L), vinyl chloride (51 to 146 μ g/L), and 1,2-trans-dichloroethylene (42 to 68 μ g/L). The pH and alkalinity of the groundwater were about 7.2 and 600 mg/L as CaCO₃, respectively. These measurements indicated that the bicarbonate ion (HCO₃), which acts as an oxidant scavenger, was present at high levels. Other oxidant scavengers, such as bromide, cyanide, and sulfide were not detected. Iron and manganese were present at low levels (less than 1 mg/L). Detailed information is available (7) to describe the parameters and design of the SITE demonstration.

Ultrox System

The Ultrox UV/oxidation treatment system uses UV radiation, ozone, and hydrogen peroxide to oxidize organics in water. The major components of the Ultrox system are the UV/oxidation reactor module, air compressor/ozone generator module, hydrogen peroxide feed system and the catalytic ozone decomposition (Decompozon) unit (Figure 2).

The UV/oxidation reactor used has a volume of 150 gallons and is 3 feet long by 1.5 feet wide by 5 feet high. The reactor is divided by five vertical baffles into six chambers and contains 24 65-watt UV lamps in quartz sheaths. The UV lamps are installed vertically and are evenly distributed throughout the reactor (four lamps per chamber). Each chamber also has one stainless steel sparger that extends along the width of the reactor. These spargers uniformly diffuse ozone gas from the base of the reactor into the water. Hydrogen peroxide is added to the influent line to the reactor. An

in-line static mixer is used to disperse the hydrogen peroxide into the contaminated water in the influent feed line.

The Decompozon unit (Model 3014 FF) uses a nickel-based proprietary catalyst to convert reactor off-gas ozone to oxygen. The Decompozon unit can accommodate flows of up to 10 standard cubic feet per minute and can destroy ozone concentrations in ranges of 1 to 20,000 ppm (by weight) to less than 0.1 ppm.

Testing Approach

The study was designed to evaluate the Ultrox system by adjusting the levels of five operating parameters: (1) influent pH, (2) hydraulic retention time, (3) ozone dose, (4) hydrogen peroxide dose, and (5) UV radiation intensity. Eleven test runs were performed to evaluate the Ultrox system under various operating conditions. After these Runs, two additional runs were performed to verify that the system's performance was reproducible. The verification Runs (Runs 12 & 13) were at the best operating conditions which were determined to be those of Run 9, pH 7.2; hydraulic retention time 40 minutes; ozone dose 110 mg/L; hydrogen peroxide dose 13 mg/L; and all UV lamps operating.

During the study, a preliminary estimate of Ultrox system's performance in each run was obtained from the effluent concentrations of three indicator VOCs. The VOCs selected for this purpose were TCE, 1,1-DCA, and 1,1,1-TCA. TCE was selected because it is a major volatile contaminant at the site, and 1,1-DCA and 1,1,1-TCA were selected because they are relatively difficult to oxidize. At the end of the study, data from all samples was used to evaluate the system's effectiveness.

Results and Conclusions

Results of the Ultrox system are summarized to present the overall effectiveness of the UV/oxidation technology in removing VOCs from the groundwater at the LB&D site. The removal efficiencies and concentration profiles of all VOCs are not presented in this paper, but additional information can be obtained from the Technical Evaluation Report and the Application Analysis Report published by EPA (8).

Summary of Results for VOCs

Based on overnight analysis performed during the demonstration (when two of the six replicates determined the average effluent concentrations for each indicator VOC), Runs 8 and 9 showed that the effluent met the discharge standard at either set of conditions. Since a lower hydrogen peroxide dose was used in Run 9, compared to Run 8, Run 9 was chosen as the preferred operating run. However, based on a complete analysis of the four remaining replicates for Run 9 performed after the demonstration, the mean concentration of 1,1-DCA was found to be slightly higher $\mu\text{g/L}$, the discharge standard for the VOC. Since

preferred operating conditions during the demonstration, the verification runs (12 and 13) were also performed at those conditions.

Figure 3 shows that the total VOC removals were about 90 percent, while removal efficiencies for TCE were about 98 percent and those for 1,1-DCA and 1,1,1-TCA were about 60 and 85 percent, respectively. Higher removal efficiencies for TCE than for 1,1-DCA and 1,1,1-TCA support the rationale used in selecting the indicator VOCs.

Figure 4 compares the 95 percent upper confidence limits (UCLs) for the effluent VOCs with the National Pollutant Discharge Elimination System (NPDES) limits. The UCLs were calculated using the one-tailed Student's t-test. The effluent met the discharge limits for all regulated VOCs at the 95 percent confidence level in Runs 12 and 13; in Run 9, the mean concentrations for 1,1-DCA and 1,2-DCA exceeded the discharge limits.

The gas chromatography (GC) and GC/mass spectrometry analyses performed for VOCs, semivolatile organics, polychlorinated biphenyls, and pesticides did not indicate the formation of new compounds in the treated water. Because VOCs made up less than 2 percent of the total organic carbon, the general claim that UV/oxidation technologies convert VOCs to carbon dioxide and water could not be verified.

Because the Ultrox system treated the groundwater by bubbling ozone gas through it, some VOC removal could be attributed to stripping in addition to oxidation. To determine the extent of stripping within the treatment system, VOC samples were collected from the reactor off-gas and emission rates for four VOCs were compared to the VOC removal rates from groundwater. The results are summarized in Table 2. Because the extent of stripping for any particular VOC is expected to be proportional to the ratio of air flow rate to water flow rate, this ratio is also presented in the table. The ratio for Runs 1 to 5 is approximately 2; for Run 6 and Runs 8 to 13, it is about 4.5; and for Run 7, it is 1. If stripping contributed to the total removal of the four VOCs, the extent of stripping would be expected to be least in Run-7, and most in Runs 6 and 8 to 13. The data presented in the table follow this trend for three of the four VOCs (except for the vinyl chloride in Runs 6,7, and 9). A quantitative correlation of the extent of stripping cannot be made, because the operating conditions were different in each run. For example, at a given air to water flow ratio, when oxidant doses are varied, the extent of oxidation also varies. Therefore, the extent of stripping will be indirectly affected.

Table 2 presents Henry's law constants for the four VOCs (9). By comparing these constants for the VOCs, their volatility is expected to increase from left to right as shown below:

1,1-DCA → TCE → 1,1,1-TCA → vinyl chloride

However, significant removal fractions for 1,1,1-TCA and 1,1-DCA were observed to be due to stripping. Conversely, the extent of stripping was low for vinyl chloride and TCE. This is because it is easier to oxidize vinyl chloride and TCE than 1,1-DCA and 1,1,1-TCA because of the double bonds between the carbon atoms in TCE and vinyl chloride. Therefore, UV/oxidation processes using ozone, stripping is a significant removal pathway for compounds that are difficult to oxidize.

Performance of the Decompozon Unit

The ozone concentrations in the influent to and the effluent from the Decompozon unit were analyzed in each run. Ozone destruction efficiencies greater than 99.99 percent were achieved in Runs 1 to 10. The effluent ozone concentrations were low (less than 0.1 ppm) for Runs 1 to 8, approximately 1 ppm in Runs 9 and 10, and greater than 10 ppm in Runs 11, 12, and 13. The high ozone levels (greater than 1 ppm) in the effluent are attributed to the malfunctioning heater in the Decompozon unit. The temperature in the Decompozon unit should have been 140° F for the unit to properly function, whereas the temperature for Runs 11 to 13 was only about 80° F.

Although the primary function of the Decompozon unit is to remove ozone, significant VOC removal also occurred when the unit functioned as designed (Runs 1 to 8). For example, the Decompozon unit removed TCE, 1,1-DCA, 1,1,1-TCA and vinyl chloride (present in the gas phase in the reactor at levels of approximately 0.1 to 0.5 ppm) to below detection levels.

PILOT-FIELD SCALE STUDY TWO

This pilot-field scale study was performed at the Old O-Field site located in the Edgewood Area of the Aberdeen Proving Ground, Maryland by the Peroxidation Systems, Inc. (PSI). The site was used for disposal of the chemical-warfare agents, munitions, contaminated equipment, and various other hazardous materials during the 1940s and early 1950s. The disposal of these hazardous materials contaminated several media at the Old O-Field site, including groundwater, surface water, and sediments.

During the pilot-field study, groundwater samples were collected which indicated that several organics, including VOCs, organosulfur compounds, and explosives, were present at the site in concentrations of 10 µg/L to 500 µg/L. Iron and manganese were present at levels of 120 mg/L and 2.5 mg/L, respectively (10). Trace levels of arsenic were also observed in some locations which were sampled before the pilot-field scale study.

Treatability studies were performed in April and May 1991 as part of the remediation process. A total of 37,000 gallons of groundwater from three wells was used to perform the treatability studies. The contaminated groundwater was pumped to two holding tanks and then treated by a metals precipitation system. The

metals precipitation was performed at pH 11, primarily to remove arsenic observed in samples collected before the pilot study began; however, the pilot study samples showed no arsenic contamination. Iron and manganese were removed to levels of 0.2 mg/L and 0.02 mg/L, respectively. The pH of the metals precipitation system effluent was adjusted to 7 and then treated using two parallel systems: (1) an air stripping system followed by carbon adsorption for both liquid and vapor phase effluents from the air stripper and (2) a UV/oxidation system. The air stripping/carbon adsorption system was developed by Carbonair and the UV/oxidation system was developed by Peroxidation Systems, Inc.

Peroxidation Systems Process

Figure 5 shows a schematic of the PSI UV/oxidation system. The system used at the Old O-Field site consisted of two parallel cartridge filters rated at 10 micrometers (μm) followed by the UV/oxidation reactor. The UV/oxidation reactor had a volume of 80 gallons and was divided by three horizontal baffles into four chambers. Each chamber contained one high intensity, broad band, mercury-arc-type 15-kw UV lamp. A splitter was used so that hydrogen peroxide could be added at multiple points, such as the influent line and at several locations inside the reactor, making hydrogen peroxide available for hydroxyl radical formation throughout the reactor. The effluent from the reactor was passed through an optional manganese-greensand filter to remove any residual hydrogen peroxide, followed by a pH adjustment to raise the pH to an acceptable level.

Testing Approach

Four tests were conducted at a flow rate of 15 gpm (hydraulic retention time of about 5.3 minutes). In Tests 1, 2, and 3, hydrogen peroxide doses were 45 mg/L, 90 mg/L and 180 mg/L, respectively, with the splitter in operation; and in Test 4, hydrogen peroxide dose was 45 mg/L with the splitter not in operation. When the splitter was used, the total hydrogen peroxide dose was split into three equal parts, which were added at (1) the influent line to the reactor, (2) the effluent line from the first chamber, and (3) the effluent line from the second chamber. In Test 4, when the splitter was not used, all hydrogen peroxide was added at the influent line to the reactor. Treated and untreated water samples were collected for (1) chemical analyses to estimate removal efficiencies and compare them with federal maximum contaminant levels (MCLs) for drinking water, and (2) bioassay to evaluate whether the water was acutely toxic to fathead minnows, daphnia magna, sheepshead minnows, and mysid shrimp.

Results and Conclusions

A discussion of the optimum operating conditions of Test 3 is presented here, a more detailed description on the PSI system performance is available in an unpublished report by the U.S. Army Corps of Engineers Toxic and Hazardous Materials Agency (10). Also, Table 3 summarizes the influent and effluent contaminant levels and the removal efficiencies of several contaminants. These results show that for most compounds the effluent levels were below detection levels, and the removal efficiencies for these compounds were greater than 82 to 99 percent. The effluent levels of chloroform and 1,3,5-trinitrobenzene were 1.2 $\mu\text{g/L}$ and .53 $\mu\text{g/L}$, respectively. The removal efficiencies for these compounds were in the range of 96 to 97 percent.

The treated effluent met the federal MCLs for all compounds. The influent to and the effluent from the PSI system passed the bioassay tests. The pH decreased by about one unit, indicating that some of the oxidation byproducts were acidic. Although the manganese-greensand filter was effective in removing residual hydrogen peroxide, it increased the manganese levels in treated water from about 0.02 mg/L to 1.4 mg/L, which is above the National Secondary Drinking Water Standard (50 $\mu\text{g/L}$). Therefore, the use of manganese-greensand filter is not recommended. Instead, other methods should be considered to neutralize any residual hydrogen peroxide in the treated water samples (for example, addition of ascorbic acid, thiosulfate, or catalase-D). If the residual oxidant level is greater than 1 mg/L and is not neutralized, it would continue to react with the contaminants in the sample bottles until analysis could be performed. This continued reaction may introduce a bias in the treatment system evaluation.

CONCLUSIONS ON THE UV/OXIDATION TECHNOLOGIES

The UV/oxidation technologies present an efficient and competitive alternative treatment, especially for the removal of organics present in water at low concentrations (less than about 100 mg/L). For higher concentration levels of contaminants, these technologies may prove cost-effective when used in combination with biological or adsorption processes. The UV/oxidation technologies are often preferred over adsorption or biological processes, because in the UV/oxidation technologies (1) contaminants are destroyed rather than transferred to some other medium and (2) no residuals requiring further handling, such as sludge or spent carbon, are generated. Due to the contaminants present it may be necessary to implement pretreatment processes to minimize shut down or delays.

Operation and maintenance data are currently being documented, and this information is instrumental in moving the technology to more efficient design and application techniques.

A greater understanding of the actual chemistry needs to be researched further, especially to understand the byproducts of the UV/oxidation of organics. Several technology developers claim that byproducts are carbon dioxide, water, and halides, but little published data are available to support these claims when UV/oxidation is used to treat the contaminants present in groundwater. Research is also being established in the area of using the UV/oxidation technologies for the destruction of organics in the air phase. Promising results are being generated for this application.

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