**GAS PHASE PHOTOCATALYTIC DEGREGATION ON TiO₂
PELLETS OF VOLATILE CHLORINATED ORGANIC
COMPOUNDS FROM A SOIL VAPOR EXTRACTION WELL
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Gas Phase Photocatalytic Degradation on TiO₂ Pellets of Volatile Chlorinated Organic Compounds from a Soil Vapor Extraction Well

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Abstract

The mineralization of trichloroethylene (TCE) and tetrachloroethylene (PCE) in gas stream from a soil vapor extraction (SVE) well was demonstrated with an annular photocatalytic reactor packed with porous TiO₂ pellets in field trials at the Savannah River Site in Aiken, SC. The TiO₂ pellets were prepared using a sol-gel method. The experiments were performed at 55 to 60 °C using space times of 10⁸ to 10¹⁰ g s/mol for TCE and PCE. Chloroform (CHCl₃) and carbon tetrachloride (CCl₄) were detected as minor products from side reactions. On a molar basis, CCl₄ and CHCl₃ produced were about 2 % and 0.2 % of the reactants.

Introduction

Many soils and groundwater supplies have become contaminated with volatile organic compounds (VOCs) such as trichloroethylene (TCE) and tetrachloroethylene (PCE) resulting from leaking underground storage tanks and improper disposal practices (1). Recognition of this problem has resulted in urgent calls for research to develop effective, inexpensive techniques for groundwater remediation. The treatment methods now in use for VOC-contaminated groundwater include soil vapor extraction (SVE) and groundwater air stripping. SVE is used to remove VOCs from the vadose (unsaturated) zone. Depending on the types of structures and materials encountered within this zone, a considerable fraction of the VOCs can be retained that

will leach slowly to groundwater, frustrating efforts to clean up groundwater. Interbedded layers of clay and sand within the vadose zone at the Savannah River Site are known to contain high levels of contaminants. When treatment of the effluents from SVE systems is required under air quality regulations, granulated activated carbon (GAC) columns are most frequently used. However, remediation by these methods is incomplete because pollutants are not destroyed, but shifted to another phase. Contaminants adsorb onto activated carbon until the adsorptive capacity of the carbon is reached. At that point, the carbon can either be regenerated for further use or disposed of as waste. After several regeneration cycles, the carbon loses its activity and must be discarded; the costs of transport and processing for regeneration or disposal can be significant. VOCs vented to the atmosphere contribute to smog and if persistent, lead to widespread environmental contamination.

Heterogeneous photocatalysis using semiconductor particles is capable of mineralizing most organic contaminants and is recognized as having great potential in the treatment of polluted water (2,3). Most studies have been performed with powder suspensions of TiO_2 in aqueous solutions (4-14). Dibble et al. reported the photocatalytic oxidation of TCE using TiO_2 powder in a gas phase system, but did not demonstrate that complete mineralization was achieved (15,16). Recently, Milne et al. have warned that the incomplete photocatalytic oxidation by TiO_2 of TCE, PCE and perhaps other chlorinated hydrocarbons in the gas phase can yield considerable quantities of phosgene (17). We previously reported that TCE can be completely mineralized to CO_2 and HCl in the gas phase by using TiO_2 pellets prepared using sol-gel techniques (18, 19). In June, 1992, a photocatalytic reactor was field tested for 5 days at the Savannah River Site in Aiken, SC. The reactor was used to treat the PCE- and TCE-contaminated effluent from a SVE unit. To the best of our knowledge, this is the first paper to

describe the use of a TiO₂-based photocatalytic reactor to degrade gas-phase contaminants in the field.

Experimental Section

The TiO₂ pellets were prepared using a sol-gel technique and fired at 300 °C (19). The diameter of the pellets was about 1 mm. Porosities of 50-56 % and specific surface areas of 160-194 m² g⁻¹ were measured by BET analysis. Analysis by powder X-ray diffraction indicates that the TiO₂ pellets are pure anatase. Annular photoreactor cells were made by a glass blower using Corning 7740 borosilicate glass tubing as illustrated schematically in Figure 1. The diameters of the inner and outer glass tubes are 2.8 and 3.8 cm; the thicknesses are 1.5 and 2 mm. The reactor cross-section is 2.9 cm² and its height is 13.4 cm. Four nipples (outer diameter: 6 mm, length: 2 cm) attached at each end of the photoreactor serve as inlets and outlets for the gas stream. The TiO₂ pellets (30 gram) were packed in the annulus surrounding a 4-W fluorescent black light bulb (GE F4T5-BLB) positioned in the middle of each photoreactor. The flowrate of photons reaching the catalyst in each reactor (wavelength < 400 nm) was found to be 1.24×10^{-6} einstein s⁻¹ using uranyl oxalate as an actinometer (20).

The demonstration photoreactor used in the field test incorporated four annular photoreactor cells arranged in parallel: the gas entering the reactor passed into a manifold that distributed the stream to the four cells and a second manifold collected the effluent. Pressure differences measured across each annular photoreactor cell were found to be equal, verifying that the gas distribution was balanced. The inlet and outlet manifolds were made of 316 stainless steel tubes (1/2" and 3/4" o.d.), sealed at both ends and each arrayed with silver soldered nipples to accommodate short pieces of Norprene plastic tubing (3/16") that connected the manifolds to the photoreactor cells. A pressure gauge, thermometer, flowmeter, and 2 valves incorporated into the reactor system (Figure 2) were joined by 1/4" o.d. 316 stainless steel

tubing. These components, a switch and ballasts for the lamps, and a cooling fan were installed in a suitcase (13" x 21" x 7.5") for portability (Figure 2).

A relative humidity meter (Fisher Scientific) was used to measure the humidity of the gas stream. The humidity of the inlet stream was controlled by passing the gas through coiled copper tubing (1/4" o.d.) immersed in an ice bath; a glass container at the bottom of the coil collected the condensate. A second copper coil leading from the collector and out of the ice bath served to rewarm the gas. Thermostat-controlled (Glas Col PL 312) heating wire (Cal-Cord) wrapped around this coil, the stainless steel inlet tubing and the inlet manifold was used to raise the temperature of the gas before it entered the photoreactor. Significant amounts of heat were also generated by the lamps. Liquid crystal temperature indicators (RLC-10, OMEGA) were attached to the outsides of the cells and by monitoring these, the thermostats were adjusted so that the reactor temperature was at least 55-60 °C. Heat loss was minimized by covering the cells and inlet lines with insulation.

The inlet gas stream was drawn from a soil vapor extraction well, located in the M-area of the Savannah River Site, Aiken, SC (Figure 3). In normal operation, gas is extracted from the well with a trailer-mounted SVE unit (maximum capacity of 500 scfm⁻¹ at 280 torr vacuum) and passed through two granular activated carbon (GAC) columns before being discharged to the atmosphere. As a result of the high VOC concentrations and a limited supply of GAC, the SVE unit had to be taken off line prior to the start of experiments. 1/4" i.d. teflon tubing was used to connect the wellhead to a small, oil-free pump (Gast model 1089). Because the photoreactor was designed to handle a smaller flowrate than that of the small pump, a system of valves was used to divide the inlet gas stream. A portion of the inlet gas stream flowed through the photoreactor system in a single pass. The unused fraction and the photoreactor effluent were routed to the GAC columns (Figure 2).

A photoionization detector (H-Nu model PI-101) was used at the site to make rough estimates of the amount of organic contaminants at the inlet and outlet. Inlet and outlet gas samples were taken using either Tedlar (Analytical Testing Corp., Model ZH 126, cap. 1 liter) or Teflon (Alltech Associates, Inc., Cat. 41184, cap. 1.2 liter) sampling bags with stainless steel valves and a septum port. The samples were stored at ambient temperature in the dark and transported to the laboratory for analysis. Samples were injected directly into a gas chromatograph/mass spectrometer (Hewlett-Packard, model 5890/5971) equipped with a 30 m x 0.53 mm x 3 μ m VOCOL column or preconcentrated on a Tenax/Silica Gel/Charcoal Trap with an OI Analytical 4460A purge and trap concentrator. Quantitation of trapped samples was performed by internal standardization with chlorobenzene-d5 and 1,4-difluorobenzene. It has previously been demonstrated that the alcohol diluent (methanol was used in this investigation) in the internal standard reacts with phosgene in the samples to form methylchloroformate (21)

Results and Discussion

Experiments were performed at three different reactant flowrates: 40, 100, and 200 ml/min as shown in Figure 4-6.

Reactant Gas. The reactant gas stream was found to contain 1,1,1-TCA (:trichloroethane, 64 - 160 ppm), TCE (222 - 1100 ppm), and PCE (1900 - 7000 ppm). 1,1-DCE (: dichloroethylene 30 - 55 ppm) was also detected in the chromatographic peak but the amount was less than the minimum reporting value. As shown in Figure 4, the concentrations of 1,1,1-TCA, TCE, and PCE fluctuated over a wide range. However, the ratios of [1,1,1-TCA]/[TCE], and [PCE]/[TCE] were constant: 0.18 ± 0.02 , and 5.16 ± 0.76 , respectively. These fluctuations did not seem to have any relationship with the ambient temperature but may have been due to diurnal changes in barometric pressure.

As we reported previously, O_2 and H_2O are needed for the complete mineralization of TCE (19), but recent experiments have suggested that excess H_2O is undesirable, possibly masking reaction sites on the catalyst surface. The water trap was designed to lower the inlet mole fraction from 0.22 (measured at the well head) to 0.007 atm. (the saturation vapor pressure of H_2O in air at $0^\circ C$).

According to the BET analysis, the TiO_2 pellets are highly porous materials with large internal surface areas. Considerable time is required to establish adsorption equilibrium between the bulk gas stream and the catalyst surface. In a bench-scale experiment using a similar annular photoreactor, it took about 4 hours for TCE (450 ppm) to reach a steady state passing through the TiO_2 bed at 300 mL/min without irradiation. For this reason, during the field experiment, the reactant gas stream was passed through the system at 1100 mL/min before irradiation. After about 3 hours, the level of organic contaminants at the outlet were almost equal to that at the inlet, as indicated by the photoionization detector. At this point, the flowrate was adjusted to 100 mL/min and the lamps were ignited.

Photodegradation. Figure 5 shows the concentrations of 1,1,1-TCA, TCE and PCE at the outlet in the same range as Figure 4. The reactor space time (defined as the amount of catalyst (W) divided by the molar flowrate of each reactant (F) (22)) for PCE and TCE were calculated to be $1.4 \times 10^8 - 2.1 \times 10^9$ and $7.5 \times 10^8 - 9.1 \times 10^9$ g s/mol, respectively. Table 1 summarizes the space times, conversion efficiencies and apparent quantum yields for PCE and TCE degradation. The apparent quantum yield, defined as the degradation rate for each reactant (mol s^{-1}) divided by the flowrate of photons of wavelengths < 400 nm entering the reactor (einstein s^{-1}). The conversions of PCE and TCE were always ca. 100 %, showing that there was enough space time for the degradation of these compounds and suggesting that it should be possible to treat higher concentrations or to run the reaction at higher flowrates. The apparent

quantum yields for the simultaneous degradation of PCE and TCE in this field demonstration were 4 - 18 % and 0.3 - 3.2 % (Table 1). In earlier experiments using a photoreactor with a tubular rather than annular configuration, the apparent quantum yield for the complete mineralization of TCE alone was about 17 % (19). If all of the steps in the reaction are photocatalytically mediated, this value is approximately the same as the theoretical maximum because six moles of electrons are transferred in the complete oxidation of each mole of TCE. Four moles of electrons must be transferred to mineralize PCE, so the maximum apparent quantum yield should be 25 %. For the simultaneous degradation of TCE and PCE, the calculated yields for each component were not compared with the theoretical maxima. However, assuming no other reaction mechanisms, the data collected during the field demonstration indicate that about 91 % of the available electrons were utilized to degrade TCE and PCE during the period of heaviest contaminant load. Free radical chain reactions, direct reaction of

Table 1. Volumetric flow rate, space time, conversion efficiency (X) and quantum yield (QY) for TCE and PCE during irradiation in the field demonstration reactor at Savannah River Site.

Time(h) (h)	Flow (mL/min)	TCE			PCE		
		Space time (gs/mol)	X (%)	QY (%)	Space time (gs/mol)	X (%)	QY (%)
3.5	100	20.1 x 10 ⁸	50.3	0.6	4.2 x 10 ⁸	63.9	3.7
5.4	100	29.0	0.3	0.7	4.8	85.4	4.3
3.3	100	16.8	58.3	0.9	2.6	96.5	9.0
27.3	100	15.3	97.3	1.5	2.3	99.2	10.4
30.3	100	18.9	97.8	1.3	3.9	97.1	6.1
44.8	100	23.7	99.8	1.0	5.0	99.5	4.8
49.3	40	78.8	98.9	0.3	18.7	97.4	1.3
52.3	40	69.6	99.6	0.3	16.6	98.6	1.4
54.7	40	91.0	97.6	0.3	21.3	88.6	1.0
62.4	200	7.5	100.0	3.2	1.4	99.9	17.8
68.8	200	12.8	99.9	1.9	3.0	99.9	8.1
70.8	200	36.4	98.6	0.7	4.3	99.6	5.7

intermediates with oxygen and accumulation of intermediates on the catalyst surface are other mechanisms that may account for the observed high efficiency of the system. Elucidation of these processes will require further investigation and is of interest as it may enable prediction of conditions under which the degradation rates could be greatly accelerated. 1,1-DCE was not detected in the effluent gas and appears to have been completely degraded. 1,1,1-TCA was detected in the outlet gas stream at levels comparable to those found in the inlet stream, and so does not appear to be degraded in this process (Figure 5). Reaction products. During operation of the reactor with the lamps on, chloroform and carbon tetrachloride were detected in the effluent, but not in the influent gas streams (Figure 6). The amount of CCl_4 was one order of magnitude higher than that of CHCl_3 , and the concentrations of both contaminants were low relative to the levels of TCE and PCE in the influent. The highest level of CCl_4 (277 ppm) was observed shortly after the lamps were turned on. During this time, the effluent gas was more humid than the influent. This may have been due to water desorbing from the catalyst: the catalyst was not heated to the normal operating temperature (55-60 °C) until the lamps were turned on. The large area of the catalyst surface and its hydrophilic nature suggest that significant amounts of water could have been sorbed during the sizing and packing of the TiO_2 pellets and during the operation to reach a steady state before irradiation. After 20 hours of operation at 55-60 °C, influent and effluent humidity levels were equal (0.007 atm) and the CCl_4 levels were 40 - 100 ppm. These results suggest that the humidity may be an important factor in the mineralization of PCE and TCE, for which optimal conditions should be determined. As shown in Figure 6, the concentration of CCl_4 does not appear to be correlated with the reactor flow rate, suggesting that it is not an intermediate in the main degradation path of TCE or PCE to CO_2 and HCl , but is produced in a side reaction. Excess water on the catalyst may favor this reaction or alternatively, water may occupy sites on the TiO_2 surface preventing CCl_4 from

being degraded further. Production of carbon tetrachloride and chloroform is consistent with a free radical reaction mechanism.

The allowable site boundary concentrations, issued by the South Carolina Dept. of Health and Environmental Control and the Threshold Limit Values (TLV) set by the American Conference of Governmental Industrial Hygienists, are lower for CCl_4 and CHCl_3 than for TCE and PCE (23,24). It has been reported in the case of TiO_2 powders using a slurry photoreactor that the degradation rates for 1,1,1-TCA, and CCl_4 are slower than those of TCE and PCE (7,25). These saturated organic compounds seem to have less interaction with the catalyst. It may be possible to degrade these compounds by increasing the space time, or optimizing the reaction temperature and water vapor content. The fact that CCl_4 is a highly oxidized compound suggests that finding operating conditions unfavorable to its formation may be a better strategy.

In the gas phase, oxidation of PCE with $\cdot\text{OH}$ radicals or homogeneous photolysis of TCE with high intensity of UV-lamps have been reported to produce a toxic gas, phosgene (26, 27). In these experiments phosgene was not detected as a reaction product.

Corrosion was observed on the stainless steel fittings of the gas sampling bags and the reactor outlet manifold, indicating production of HCl. Mineralization of TCE and PCE are expressed as the following equation:

The large quantities of HCl produced in these reactions would require that a full scale reactor have a scrubbing system to remove the acid before venting the effluent to the environment.

Conclusions

These field experiments have demonstrated that TCE and PCE can be mineralized by photodegradation at 55 - 60 °C in annular reactors packed with TiO_2 pellets without any

production of phosgene. The apparent quantum yield was calculated to be 4 - 18 % for PCE and 0.3 - 3.2 % for TCE, suggesting that the full capacity of the reactor is not being utilized. Further work needs to be performed in order to optimize the reaction for the mineralization of 1,1,1-TCA and CCl₄.

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Figure Captions:

Figure 1. Schematic diagram of an annular photoreactor cell.

Figure 2. A schematic diagram of the photoreactor system.

V: 2-way valve; **MV**: monitoring valve; **WT**: water trap; **RC**: rewarming coil; **P**: pressure gauge; **FM**: flowmeter; **T**: thermometer; **GAC**: granular activated carbon; **SVE**: soil vapor extraction.

Figure 3. Location of the Savannah River Site.

Figure 4. Concentrations of 1,1,1-TCA (∇), TCE ($\diamond$), and PCE (Δ) in the influent gas stream. Three different flowrates were used during the field test.

Figure 5. Concentrations of 1,1,1-TCA (∇), TCE ($\diamond$), and PCE (Δ) in the effluent gas stream. The earliest sample (3.5 hrs) was taken before the reactor lamps were turned on.

Figure 6. Concentrations of CHCl_3 ($\diamond$) and CCl_4 ($\square$) in the effluent gas stream. The earliest sample (3.5 hrs) was taken before the reactor lamps were turned on.

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