

AL/EQ-TP-1994-0302

**DECONTAMINATION OF WATER USING  
ADSORPTION AND PHOTOCATALYSIS  
(PREPRINT)**

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Contract No. F08635-90-C-0063

November 1994

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*Form Approved  
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|   |   |   |
|---|---|---|
| <b>1. REPORT DATE (DD-MM-YYYY)</b><br>11-NOV-1994 | <b>2. REPORT TYPE</b><br>Journal Article - PREPRINT | <b>3. DATES COVERED (From - To)</b><br>15-JUN-1990 -- 15-OCT-1994 |
|---|---|---|

|   |  |
|---|--|
| <b>4. TITLE AND SUBTITLE</b><br>Decontamination of Water Using Adsorption and Photocatalysis (PREPRINT) | <b>5a. CONTRACT NUMBER</b><br>F08635-90-C-0063 |
|   | <b>5b. GRANT NUMBER</b>                        |
|   | <b>5c. PROGRAM ELEMENT NUMBER</b><br>0602202F  |

|   |   |
|---|---|
| <b>6. AUTHOR(S)</b><br>Suri, Rominder P.S.; Perram, David W.; Crittenden, John C.; Hand, David W. | <b>5d. PROJECT NUMBER</b><br>1900       |
|   | <b>5e. TASK NUMBER</b><br>71            |
|   | <b>5f. WORK UNIT NUMBER</b><br>19007168 |

|   |   |
|---|---|
| <b>7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)</b><br>Department of Chemical Engineering<br>Michigan Technological University<br>1499 Townsend Drive<br>Houghton, MI 49931 | <b>8. PERFORMING ORGANIZATION REPORT NUMBER</b> |
|---|---|

|  |   |
|--|---|
| <b>9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)</b><br>Environics Directorate<br>Armstrong Laboratory<br>139 Barnes Drive, Suite 2<br>Tyndall Air Force Base, FL 32403-5323 | <b>10. SPONSOR/MONITOR'S ACRONYM(S)</b><br>AL/EQ                    |
|  | <b>11. SPONSOR/MONITOR'S REPORT NUMBER(S)</b><br>AL/EQ-TP-1994-0302 |

**12. DISTRIBUTION/AVAILABILITY STATEMENT**  
Distribution Statement A: Approved for public release; distribution unlimited.

**13. SUPPLEMENTARY NOTES**

**14. ABSTRACT**

A novel treatment strategy employing adsorption and solar assisted photocatalytic processes was field tested at Tyndall Air Force Base, Florida for the removal and destruction of organic contaminants in groundwater. The adsorbents were impregnated with photoactive catalysts and used in packed beds to remove the pollutants from the water stream for 18 hours. The spent adsorbents were regenerated for 6 hours by passing hot water in the presence of sunlight. The desorbed organics were destroyed using fixed bed photocatalysis. The process was examined for 10 cycles of adsorption and regeneration for treatment of water contaminated with BTEX (benzene, toluene, ethylbenzene, o-xylene, m-xylene, and p-xylene) compounds. The efficiency (ratio of amount of organics removed to amount of organics adsorbed) of the process to regenerate the adsorbents was examined for each cycle. A laboratory study was also conducted under controlled conditions using tetrachloroethylene as a model compound to examine the regeneration efficiency for 7 cycles of adsorption and regeneration.

**15. SUBJECT TERMS**  
decontamination, regeneration, phototcatalysis, adsorption

|  |                         |                          |   |                                  |  |
|--|-------------------------|--------------------------|---|----------------------------------|--|
| <b>16. SECURITY CLASSIFICATION OF:</b> |                         |                          | <b>17. LIMITATION OF ABSTRACT</b><br>UU | <b>18. NUMBER OF PAGES</b><br>24 | <b>19a. NAME OF RESPONSIBLE PERSON</b><br>Mark Smith |
| <b>a. REPORT</b><br>U                  | <b>b. ABSTRACT</b><br>U | <b>c. THIS PAGE</b><br>U |   |                                  | <b>19b. TELEPHONE NUMBER (Include area code)</b>     |

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# Decontamination of Water using Adsorption and Photocatalysis

Rominder P.S. Suri, David L. Perram, John C. Crittenden, and David W. Hand

## ABSTRACT

A novel treatment strategy employing adsorption and solar assisted photocatalytic processes was field tested at Tyndall Air Force Base (Florida) for the removal and destruction of organic contaminants in groundwater. The adsorbents were impregnated with photoactive catalysts and used in packed beds to remove the pollutants from the water stream for 18 hours. The spent adsorbents were regenerated for 6 hours by passing hot water in the presence of sunlight. The desorbed organics were destroyed using fixed bed photocatalysis. The process was examined for 10 cycles of adsorption and regeneration for treatment of water contaminated with BTEX (benzene, toluene, ethylbenzene, o-xylene, m-xylene, and p-xylene) compounds. The efficiency (ratio of amount of organics removed to amount of organics adsorbed) of the process to regenerate the adsorbents was examined for each cycle. A laboratory study was also conducted under controlled conditions using tetrachloroethylene as a model compound to examine the regeneration efficiency for 8 cycles of adsorption and regeneration.

*Key words: decontamination, regeneration, photocatalysis, adsorption.*

## INTRODUCTION

Adsorption technology has been used for decades for removal of organic contaminants from water. However, the spent adsorbents may have to be handled as hazardous waste. This could create a problem for small water treatment utilities where GAC usage rates are not large

enough to justify on-site thermal reactivation, and where hazardous waste landfills are not available locally.

Some researchers have shown that heterogeneous photocatalytic oxidation process can be employed to successfully regenerate spent adsorbents (Notthakun et al., 1993; and Liu et al., 1993), wherein the adsorbed organics are oxidized to non-toxic forms. The adsorbents were impregnated with the photocatalyst and exposed to a UV light source. It was observed that the adsorbents loaded with trichloroethylene, or p-dichlorobenzene could be successfully regenerated using this process. However, the regeneration process required a very long time, which made this process not suitable for practical applications. It was reported that the regeneration of adsorbents (F-400 GAC and Ambersorb 563 adsorbent) was limited due to diffusion of adsorbates from the interior surface of adsorbent to the exterior where the reaction takes place. One method to increase the desorption rate, and thereby increase the regeneration rate, is to heat the regeneration reactor system. During photocatalytic regeneration, there may be products of partial oxidation of parent adsorbate which may be more polar, and it may be easier to desorb these secondary compounds at elevated temperatures.

The major objective of this study was to enhance the regeneration rate of the spent adsorbents using photocatalysis by conducting the regeneration process at elevated temperatures. The adsorbents were impregnated with a photocatalyst, and regenerated at elevated temperatures in an attempt to develop an effective and practical reactor system. For this purpose there were three elements that were considered for the optimization of the process - adsorbent, photocatalyst, and photocatalyst support.

The Ambersorb 563 adsorbent (Rohm and Haas Co., Philadelphia, PA) was selected for

this study as it is reported to be resistant to fouling by natural organic matter (Parker and Bortko, 1991; and Hand et al., 1993). Fouling of activated carbon may occur by natural organic matter during field operation, and it may be difficult to regenerate it using photocatalysis. Also, it has been reported that oxygenated compounds such phenols can react on the surface of some GACs in the presence of dissolved oxygen forming polymeric species (Vidic et. al., 1993), and it may be difficult to regenerate the carbon. In an earlier study (Suri et al., 1993), a number of catalysts were examined for their photoactivity, and 1%Pt-TiO<sub>2</sub> was identified as a very active photocatalyst for destruction of a number of organic compounds. The 1%Pt-TiO<sub>2</sub> photocatalyst was used in this study. For photocatalytic destruction of organic compounds, several researchers have evaluated the option of fixing the catalysts on some support material to avoid the problem of catalyst separation from the aqueous slurry. Matthews (1987a, and 1987b), and Al-Ekabi and Serpone (1988) showed that phenolic compounds could be photocatalytically degraded over titanium oxide supported on a glass fiber matrix. Zhang et al. (1994) showed that Pt-TiO<sub>2</sub> catalyst supported on silica gel was very effective for destruction of a number of compounds such as benzene, toluene, ethyl benzene, xylenes, trichloroethylene, tetrachloroethylene, p-dichlorobenzene, and o-chlorobiphenyl. In this study, 1%Pt-TiO<sub>2</sub> catalyst was fixed on silica gel support (35X60 mesh, Aldrich Chemical Co.) following the method outlined by Zhang (1994).

## **PROCESS STRATEGY**

In this study, a reactor configuration was examined in which the organic contaminants were first removed from the influent water stream by adsorption, and then the organics were

destroyed by photocatalysis. A schematic of the reactor set-up for the field study is shown in Figure 1. The process operates in three phases. The first phase consists of fixed bed adsorption. In the second phase, the adsorbent is regenerated off-line by passing a hot regeneration solution (oxygenated Milli-Q water) through the fixed adsorbent bed to desorb the adsorbate. During this phase of the process, the adsorbent bed is exposed to a UV-light source. The photocatalyst supported on the adsorbent, in the presence of UV-light, can oxidize the adsorbed organics partially or completely. The products of partial oxidation may be more polar in nature than the parent compounds, and may desorb faster from the adsorbent surface, thereby assisting the regeneration process. Accordingly, the operative mechanisms of the regeneration process may be thermal desorption of the adsorbed organics, as well as their partial or complete oxidation. In the third phase, the desorbed organics from the regeneration unit are oxidized photocatalytically by catalyst supported on silica gel.

This process strategy was field tested at Tyndall AFB, Florida for the removal and destruction of BTEX (benzene, toluene, ethylbenzene, o-xylene, m-xylene, and p-xylene) compounds. A laboratory study was conducted under controlled conditions using tetrachloroethylene as a model compound to examine the regeneration process mechanisms.

## **MATERIALS AND METHODS**

### **MATERIALS:**

All chemicals were reagent grade or better. Tetrachloroethylene (PCE), high purity (99.9+ %) Aldrich TiO<sub>2</sub>, and Davisil™ silica gel were obtained from Aldrich Chemical Co. (Milwaukee, WI). X-ray diffraction showed more than 99% of the crystalline Aldrich TiO<sub>2</sub> is

anatase. The Ambersorb-563 adsorbent (20X50 mesh) was obtained from Rohm and Haas Co. (Philadelphia, PA).

**Adsorbent Preparation:** The commercial grade Ambersorb-563 adsorbent was impregnated with 1%Pt-TiO<sub>2</sub> catalyst following the method described elsewhere (Suri, 1994). In short, the impregnation of the adsorbent was conducted in two steps. In the first step, the adsorbent was soaked in titanium isopropoxide solution (with 20% by volume of 2-propanol). The amount of solution used was equal to that required to fill the pores of the adsorbent. The mixture was then placed in a humid environment for about 24 hours for titanium isopropoxide to hydrolyze on the adsorbent. The adsorbent/catalyst was calcined at 500 °C for 3 hours under an argon atmosphere. With this process, it is expected that the catalyst will be impregnated onto the internal surface of the adsorbent. This may facilitate the regeneration process by reducing the solute diffusion time from the internal surface. The impregnation of the catalyst onto the external surface of the adsorbent was performed by immersing the adsorbent in an aqueous slurry of Pt-Aldrich TiO<sub>2</sub> catalyst for 10 minutes. The adsorbent/catalyst was dried at 70 °C for 2 hours, washed with Milli-Q water, and dried at 105 °C for 12 hours. The amount of catalyst loaded onto the adsorbent was determined by ash content analysis. It was observed that the total amount of catalyst impregnated onto the adsorbent was 12% by adsorbent weight.

**Catalyst Preparation:** Platinized TiO<sub>2</sub> was prepared by photo-reducing 1 weight percent of Pt on the surface of TiO<sub>2</sub> from hexachloroplatanic acid as described by Kraeutler and Bard (1978). The 1%Pt-TiO<sub>2</sub> catalyst was fixed on silica gel support (35X60 mesh) following the method outlined by Zhang (1994). The amount of catalyst supported on the silica gel was about 1% by weight.



## SITE CONDITIONS AND GROUNDWATER MATRIX

The Tyndall AFB site was a fuel handling and storage area, and was contaminated from occasional spills and leaks from the underground storage tanks. The soil and groundwater were contaminated with JP-4 jet fuel which is a mixture of over 300 different compounds including about 2 to 3 wt. % of BTEX compounds. About half a meter of floating fuel was found on the water surface of the monitoring wells in the area. However, the BTEX concentration was observed to fluctuate with the rate and duration of pumping during the test. To minimize the fluctuations in the influent BTEX concentrations, the groundwater was collected in an air free teflon bag and spiked with a certain amount of floating fuel. The floating fuel was bailed out from a monitoring well only a few meters away from the extraction well. In about 24 hours, a stable concentration of the BTEX compounds was achieved in the bags. Table 1 shows the time average concentration of the BTEX compounds in the influent groundwater. The dissolved oxygen was less than 2 mg/L, and the pH was about 6.5 in the water well. The water hardness was about 140 mg/L as calcium carbonate.

All other water, except the groundwater at Tyndall AFB, used in the study was distilled, and further purified by a Milli-Q purification system (Millipore Corp., Bedford, MA).

Table 1: Average influent BTEX compound concentrations in groundwater at the Tyndall AFB

| Organic Compounds | Concentration(ug/L) |
|-------------------|---------------------|
| Benzene           | 837                 |
| Toluene           | 1791                |
| Ethylbenzene      | 421                 |
| o-xylene          | 380                 |
| m-xylene          | 1670                |
| p-xylene          | 585                 |

## **ANALYTICAL METHODS**

PCE was analyzed by direct aqueous injection (Supelco GC Bulletin 816A, Supelco, Inc., Houston, TX) using a Hewlett-Packard (HP; Palo Alto, CA) 5880A gas chromatograph (GC) which was equipped with an electron capture detector. Chloride ion concentration was measured using Dionex DX500 (Sunnyvale, CA) Ion Chromatograph. Dissolved oxygen (DO) was analyzed using a YSI (Yellow Spring, OH) Model 54 DO meter. Light intensity was measured using EG&G Gamma Scientific (San Diego, CA) Model GS 3100 spectroradiometer equipped with a fiber optic probe. It was calibrated to measure light intensity for wavelengths between 275 to 387.5 nm because this is the range of light which can activate TiO<sub>2</sub> catalysts (Sakata and Kawai, 1983). For the field study, the BTEX compounds were analyzed using a Hewlett-Packard (Palo Alto, CA) 5890A gas chromatograph according to the EPA Method 8020.

## **REACTOR SETUP AND EXPERIMENTAL PROCEDURES**

The reactor units were constructed using plastic tubes (American Energy Technologies, Inc., Green Cove Springs, FL). The mechanical and UV transmissive properties of the plastic material have been reported to be stable by the manufacturer under a continuous solar illumination for 5 years. A 1/8" thick plate of this plastic material has an average sunlight transmittance of 80% in the wavelength range of 300 - 400 nm. For the laboratory study, the artificial light source was a 1.75 meter long, 88 watts fluorescent tanning lamp (United States Lighting Corp., Fenton, MI; model FS7212/UVB/HO). The light intensity of these lamps was 1.8 mW/cm<sup>2</sup> in the range of 275 to 387.5 nm and peaked at 310 nm. For all laboratory experiments involving artificial light, the reactors were placed at a distance of 1 cm from the lamp.

***Field Reactor Setup and Procedures:*** Two M-7 plastic tubes were packed with impregnated adsorbent. The length and inner diameter of these adsorbent tubes were 100 cm and 0.645 cm, respectively. One adsorbent column, the control column, was loaded in the dark and was not regenerated. Figure 1 shows the experimental setup for the adsorption/regeneration processes. Contaminated groundwater was passed through the control and test adsorbent columns for a period of 18 hours. Samples were taken periodically at the influent and effluent ends, and analyzed for BTEX concentrations. Following each adsorption period for the test column, adsorption feed pumps were shut off, and the adsorbent was acid washed with 45 ml of 10% HCl. This acid washing was conducted to prevent catalyst fouling in the regeneration unit. After the acid washing, the adsorbent bed was regenerated by exposing it to sunlight and passing hot water (90 °C) counter current to the direction of the loading cycle. The hot water at about 90 °C was pumped at a flow rate of about 14 ml/min to enhance the desorption of the organics. The regeneration cycle was conducted during the day light for a period of 6 hours. Since the adsorbent was impregnated with the photocatalyst, it was expected that partial or complete oxidation of the organics would occur in the adsorbent bed itself in the presence of light. It is possible that the products of partial oxidation were more polar than the parent compound, thereby assisting in the regeneration of the adsorbent. During regeneration, the effluent carrying the desorbed organics from the test column was passed through the photocatalyst/silica gel bed which was exposed to sunlight. Samples were taken periodically at the effluent end of the photocatalyst bed to determine the removal of organics. The control column was operated in the dark under the same influent conditions as the test column, and was not regenerated. A comparison of the effluent profiles of the test and control columns would show the effectiveness

of the regeneration process.

***Laboratory Reactor Setup and Procedures:*** In the laboratory study, three adsorbent columns were used. One M-7 tube was packed with virgin Amborsorb 563 adsorbent (non impregnated), and two tubes were packed with impregnated adsorbent. The length and inner diameter of these adsorbent tubes were 25 cm and 0.645 cm, respectively. The virgin adsorbent column, which was used as a control column, was operated in the dark under the same influent conditions as the test column, and was not regenerated. For the regeneration process, two M-7 tubes of 100 cm in length and 1.27 cm in inner diameter were packed with catalyst coated silica gel. Figures 2 and 3 show the schematic of the adsorption and regeneration reactor systems, respectively. Tetrachloroethylene was used as a model compound in this study. The adsorption cycle was conducted in the dark for 18 hours, followed by 6 hours of regeneration process under artificial light illumination. Six hours of regeneration time represents the daylight time period available for the regeneration process during the field application. After each loading, the impregnated adsorbent columns were regenerated by passing hot water (about 90 °C) at a flow rate of about 5 to 20 ml/min counter current to the direction of the loading cycle. One of the impregnated adsorbent column was regenerated in the dark, as shown in Figure 3, as a control column to investigate the effect of photocatalysis on regeneration. During the regeneration process, the effluent from the test column was passed through the two silica gel columns connected in series, as shown in Figure 3. Samples were taken periodically from the influent and effluent end of each column, and analyzed.

## RESULTS AND DISCUSSION

**FIELD STUDY:** The adsorbent regeneration reactor was tested at the Tyndall AFB for treatment of groundwater contaminated with BTEX compounds, and it was operated for a total of 10 adsorption/regeneration cycles. Table 2 lists the conditions under which it was operated. The influent and effluent BTEX profiles for the adsorbent regeneration reactor and the control column are compared in Figure 4. The adsorbent column was regenerated for 6 hours after every 18 hours of loading, although the first adsorption cycle was for more than 18 hours. The control column was loaded in the dark and was not regenerated. It may be observed from Figure 4 that the effluent profile from the adsorbent regeneration reactor is significantly lower than the control column. This shows that the cyclic regeneration process was effective in retarding the BTEX breakthrough. Table 2 summarizes the mass of BTEX compounds adsorbed during each adsorption cycle. It was observed that the total amount of BTEX compounds adsorbed onto the adsorbent in the test column for 10 adsorption cycles was about 500 mg. During regeneration, the total amount of BTEX compounds desorbed during the 10 regeneration cycles was about 100 mg. However, this does not account for the amount of BTEX compounds that may have been either partially or completely oxidized by photocatalysis during the regeneration process. Initially, it was observed that some fouling species (which were adsorbed onto the adsorbent during the loading cycle) were eluting out and deactivating the photocatalyst bed during regeneration. This problem of catalyst deactivation was eliminated by acid washing the adsorbent bed prior to regeneration.

From Table 2, it may be observed that the efficiency of the adsorption process to remove the BTEX compounds was more than 96%. This removal efficiency is based on the ratio of

amount of BTEX adsorbed to the amount of BTEX fed into the reactor. On the other hand, it may be observed from Table 2 that although the overall regeneration efficiency of the process was only about 21%, the regeneration efficiency of a cycle increased dramatically from about 1 to 60%. The regeneration efficiency of about 60% for the last cycle is based on the ratio of amount of BTEX compounds desorbed as compared to the amount of BTEX adsorbed in the last cycle. The overall regeneration efficiency of 21% is based on the ratio of total amount of BTEX compounds desorbed as compared to the total amount of BTEX adsorbed. It may be observed from Table 2 that as more of the organics were being adsorbed onto the column, higher was the amount desorbed.

It seems that a stage may be achieved when almost all of the organics adsorbed in the adsorption cycle can be removed in the subsequent regeneration cycle. Based on the results shown in Figure 4 and Table 2, it appears that steady state was not achieved. However, it may be possible to design an adsorbent bed which can effectively remove organics without replacement of the bed. The regeneration of adsorbent is probably due to partial and/or complete oxidation of organics by photocatalysis, as well as desorption and flushing of organics by hot regeneration water in the direction opposite to loading cycle. Conceptually, it can be imagined that the mass transfer zone of adsorption is being pushed back during the regeneration cycle. However, some significant length of mass transfer zone is required for significant removal of organics by the regeneration process, so that sufficient number of adsorbent sites are regenerated to adsorb all of the organics during the next adsorption cycle. Hence, after a certain period of operation, a steady state can be achieved where the amount of organics adsorbed is equal to the amount removed. It may be possible to design a reactor which is long enough to

contain this steady state mass transfer zone, and which can meet the effluent requirements at all time.

**LABORATORY STUDY:** To examine in detail the mechanisms of this process to regenerate spent adsorbents, a laboratory study was performed using tetrachloroethylene (PCE) as a model compound. A chlorinated hydrocarbon was selected as chloride produced from photocatalytic oxidation could be quantitatively related to amount of parent compound destroyed. In an earlier study (Suri et al., 1993) it was shown that chloride ions could be used to determine the destruction of parent chlorinated compound. The process was evaluated for 8 cycles of adsorption (18 hr) and regeneration (6 hr), and the results are shown in Table 3. During regeneration, the PCE profile in effluent stream from adsorbent bed was integrated to calculate the amount of PCE desorbed, and that value is reported in Table 3. The effluent from adsorbent bed was the influent for photocatalytic bed. The PCE concentration in effluent from photocatalytic reactors was below 5 ug/L.

From chloride ion analysis, no destruction of PCE was observed for the adsorbent column regenerated in the dark. For the adsorbent column regenerated in presence of light, some chloride was observed in effluent which indicated destruction of adsorbate in the bed. However, it is possible that the adsorbed PCE was partially oxidized and there were some chlorinated by-products. The effluent from this test column was passed through photocatalyst columns where these chlorinated by-products, if any, were expected to get oxidized. The chloride ions measured in effluent from photocatalytic beds corresponded to a PCE value which was significantly higher than the PCE desorbed from adsorbent bed. For example, the PCE desorbed from adsorbent bed for third cycle was about 21 mg, as shown in Table 3, and chloride

ions measurement showed that about 9.2 mg of PCE was destroyed in the adsorbent bed itself. However, chloride ions measured in effluent of catalyst beds showed that about 53 mg of PCE was destroyed on catalyst bed (this was in addition to about 9.2 mg of PCE destroyed on adsorbent bed, as shown in Table 3). Hence, about 32 mg of PCE was converted to by-products in the adsorbent bed due to photocatalysis, and these compounds were subsequently destroyed on silica gel bed by photocatalysis. Table 3 shows that a significant amount of adsorbed PCE is not only destroyed on the adsorbent bed, but is also partially oxidized to chlorinated compounds, and these by-products are subsequently oxidized in photocatalyst beds. Hence, for regeneration of adsorbents loaded with organics, both thermal desorption and photocatalytic oxidation processes were observed to be important.

For the eight adsorption/regeneration cycles conducted, the efficiency of the regeneration process for the individual adsorption/regeneration cycle increased from about 30% to 60% (this efficiency is based on the ratio of amount of PCE destroyed as compared to amount of PCE adsorbed), as shown in Table 3. However, it is possible that if this adsorption/regeneration process was conducted for some more cycles, a steady state may have been achieved where the amount of organics removed were same as amount adsorbed in a cycle.

## **CONCLUSIONS**

A treatment strategy employing adsorption and solar assisted photocatalytic oxidation processes was field tested at Tyndall Air Force Base (Florida) for removal and destruction of organic contaminants in groundwater. The adsorbents, impregnated with the photocatalyst, were packed in plastic tubes having high UV-light transmission. The adsorbent columns were



employed to remove BTEX pollutants in groundwater for 18 hours, and then regenerated for 6 hours by passing hot water at about 90 °C counter current to the direction of the loading cycle and in the presence of sunlight. The desorbed BTEX compounds from adsorbent bed were destroyed by catalysis in the photocatalytic bed. The field study demonstrated that the adsorption process was more than 96% efficient for treatment of water contaminated with BTEX (benzene, toluene, ethylbenzene, o-xylene, m-xylene, and p-xylene) compounds. The study showed that for 10 cycles of adsorption/regeneration conducted, the regeneration efficiency for a cycle increased from about 1 to 60%. However, it is possible that if this adsorption/regeneration process was conducted for some more cycles, a steady state may have been achieved where the amount of organics removed were same as amount adsorbed. It may be possible to design a reactor which is long enough to contain the steady state mass transfer zone, and which can meet effluent requirements at all time without replacement of the bed. A long term feasibility study is warranted to determine the feasibility of this process for practical applications. This regeneration process would be more attractive for adsorbents with faster desorption properties, and organics with weak adsorptive character.

To determine the mechanisms of this adsorption/regeneration process, experiments were conducted in laboratory under controlled conditions using tetrachloroethylene as a model compound. Laboratory study showed that a significant amount of adsorbed organic was either completely or partially oxidized in the adsorbent bed itself. The regeneration of adsorbents was due to partial and complete oxidation of organics by photocatalysis, and desorption and flushing of organics by hot water. The desorbed organics from adsorbent bed were destroyed in photocatalytic bed to below detection level. For the eight adsorption/regeneration cycles

conducted, the efficiency of the regeneration process for individual adsorption/regeneration cycle increased from about 30% to 60%.

## **ACKNOWLEDGMENTS**

**Credits:** This research was based on work supported by the U.S. Air Force, Environics Division, Tyndall AFB, Florida; and Michigan Technological University, Houghton, Michigan. The authors thank Air Force project officers Major Mark Smith and Capt. Edward Marchand for their constructive criticism. Opinions, findings, and conclusions expressed in this article are those of the authors and do not necessarily reflect the views of the US Air Force or Michigan Technological University. Initial analysis of data from the field study at Tyndall AFB was performed by Eric Klun who was a graduate student at Michigan Technological University.

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Table 2 Mass balance on BTEX compounds for 10 adsorption/regeneration cycles at Tyndall AFB, FL.

| Cycle no. | Loading flow rate, ml/min | Loading duration, min | BTEX fed, mg | BTEX adsorbed mg | Overall BTEX removal efficiency | BTEX desorbed mg | Regeneration Efficiency for the cycle | Cumulative Regeneration Efficiency      |
|-----------|---------------------------|-----------------------|--------------|------------------|---------------------------------|------------------|---------------------------------------|---|
|           |                           |                       | (a)          | (b)              | $\frac{(b)}{(a)} \cdot 100$     | (c)              | $\frac{(c)}{(b)} \cdot 100$           | $\frac{\sum(c)_i}{\sum(b)_i} \cdot 100$ |
| 1         | 9.8                       | 3631                  | 201          | 201.0            | 100%                            | 1.2              | 1%                                    | 1%                                      |
| 2         | 8.9                       | 1047                  | 53           | 52.5             | 99%                             | 6.8              | 13%                                   | 3%                                      |
| 3         | 6.5                       | 1756                  | 65           | 64.6             | 99%                             | 10.2             | 16%                                   | 6%                                      |
| 4         | 5.0                       | 1062                  | 30           | 30.0             | 100%                            | 11.5             | 38%                                   | 9%                                      |
| 5         | 5.3                       | 1041                  | 27           | 27.0             | 100%                            | 6.5              | 24%                                   | 10%                                     |
| 6         | 5.1                       | 1020                  | 27           | 25.8             | 96%                             | 5.6              | 22%                                   | 10%                                     |
| 7         | 5.2                       | 1238                  | 34           | 33.2             | 97%                             | 15.9             | 48%                                   | 13%                                     |
| 8         | 5.0                       | 1032                  | 27           | 26.9             | 100%                            | 20.2             | 75%                                   | 17%                                     |
| 9         | 5.0                       | 918                   | 26           | 25.5             | 98%                             | 16.2             | 62%                                   | 19%                                     |
| 10        | 5.0                       | 910                   | 24           | 23.6             | 98%                             | 13.8             | 58%                                   | 21%                                     |

Regeneration flow rate = 13.6 ml/min; regeneration duration = 6 hours

Sample calculation for Cumulative regeneration efficiency after cycle 3:

$$\frac{\sum(c)_i}{\sum(b)_i} \cdot 100 = \left( \frac{1.2 + 6.8 + 10.2}{201 + 53 + 65} \right) \times 100 = 5.7\%$$

**Table 3** Mass balance on PCE for 8 adsorption/regeneration cycles for the laboratory study using catalyst impregnated Ambersorb-563 adsorbent and catalyst coated silica gel beds. Adsorption time: 18 hours; Regeneration time: 6 hours.

| Cycle no. | PCE adsorbed on resin bed, mg<br>(a) | Regeneration flow rate, ml/min | PCE desorbed from resin bed, mg | PCE destroyed* on resin bed, mg<br>(b) | PCE destroyed* on silica gel bed, mg<br>(c) | Total PCE destroyed, mg<br>(= b+c)<br>(d) | Efficiency for the cycle<br>$\frac{(d)}{(a)} \cdot 100$ | Cumulative Process Efficiency<br>$\frac{\Sigma (d)_i}{\Sigma (a)_i} \cdot 100$ |
|-----------|--------------------------------------|--------------------------------|---------------------------------|--|---|---|---|--|
| 1         | 46                                   | 10                             | 2.4                             | 2.9                                    | 12.3  | 15.2                                      | 33%   | 33%  |
| 2         | 56                                   | 10                             | 4.9                             | 4.4                                    | 15.6  | 20.0                                      | 36%   | 35%  |
| 3         | 106                                  | 20                             | 21.0                            | 9.2                                    | 53.1  | 62.3                                      | 59%   | 47%  |
| 4         | 99                                   | 5                              | 8.6                             | 5.3                                    | 36.4  | 41.7                                      | 42%   | 48%  |
| 5         | 108                                  | 20                             | 28.0                            | 6.0                                    | 39.0  | 45.0                                      | 42%   | 46%  |
| 6         | 143                                  | 20                             | 39                              | 7.8                                    | 39  | 46.8                                      | 33%   | 42%  |
| 7         | 126                                  | 20                             | 47.5                            | 9.2                                    | 61.8  | 71.0                                      | 56%   | 44%  |
| 8         | 118                                  | 20                             | 43.4                            | 7.3                                    | 62.7  | 70.0                                      | 59%   | 46%  |

\*: determined from chloride ion analysis

Process efficiency is defined as the ratio of adsorbate mass destroyed to mass adsorbed.

Sample calculation for Cumulative process efficiency after cycle 3:

$$\frac{\Sigma (d)_i}{\Sigma (a)_i} \cdot 100 = \left( \frac{15.2 + 20 + 62.3}{46 + 56 + 106} \right) \times 100 = 47\%$$

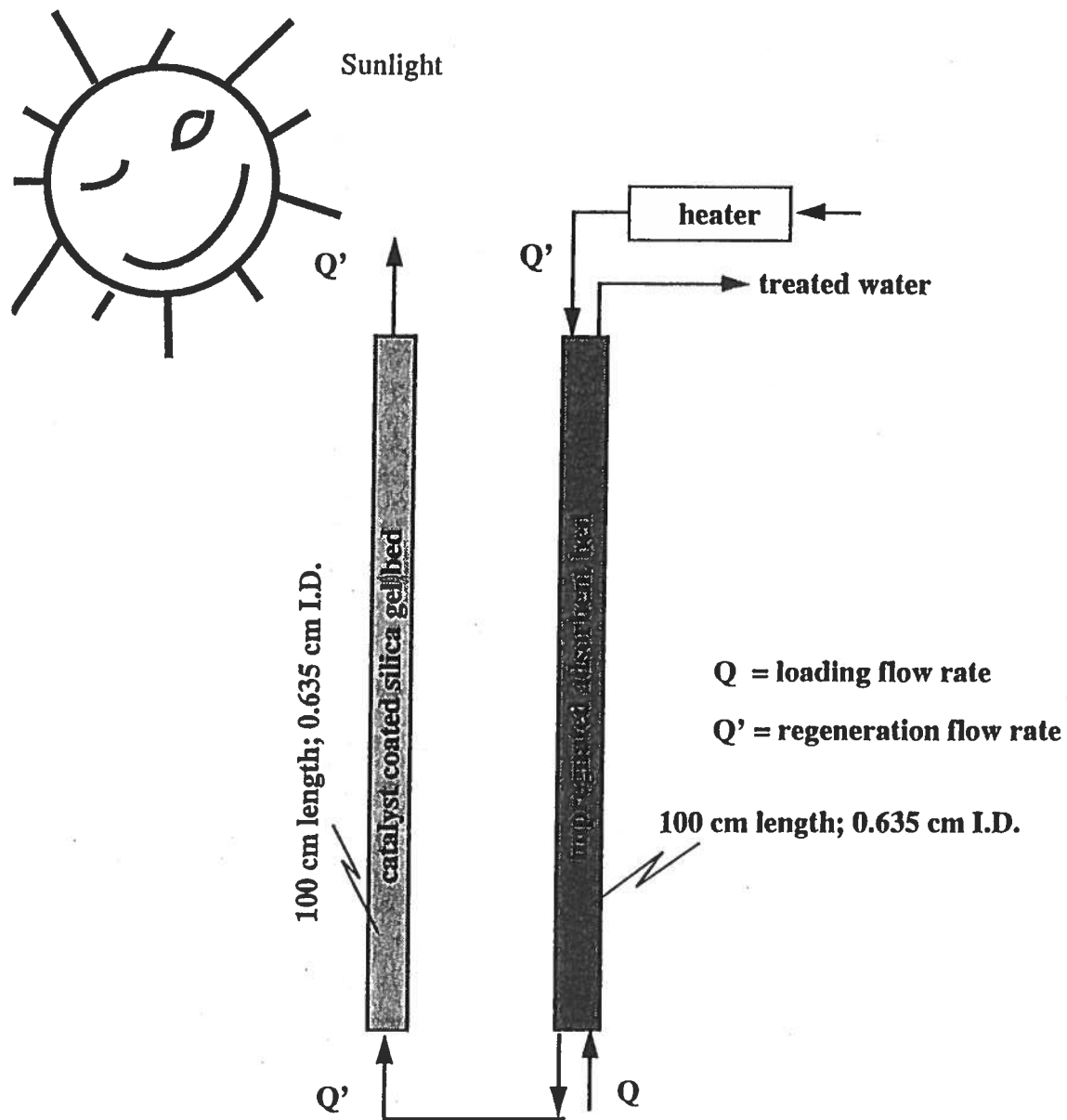
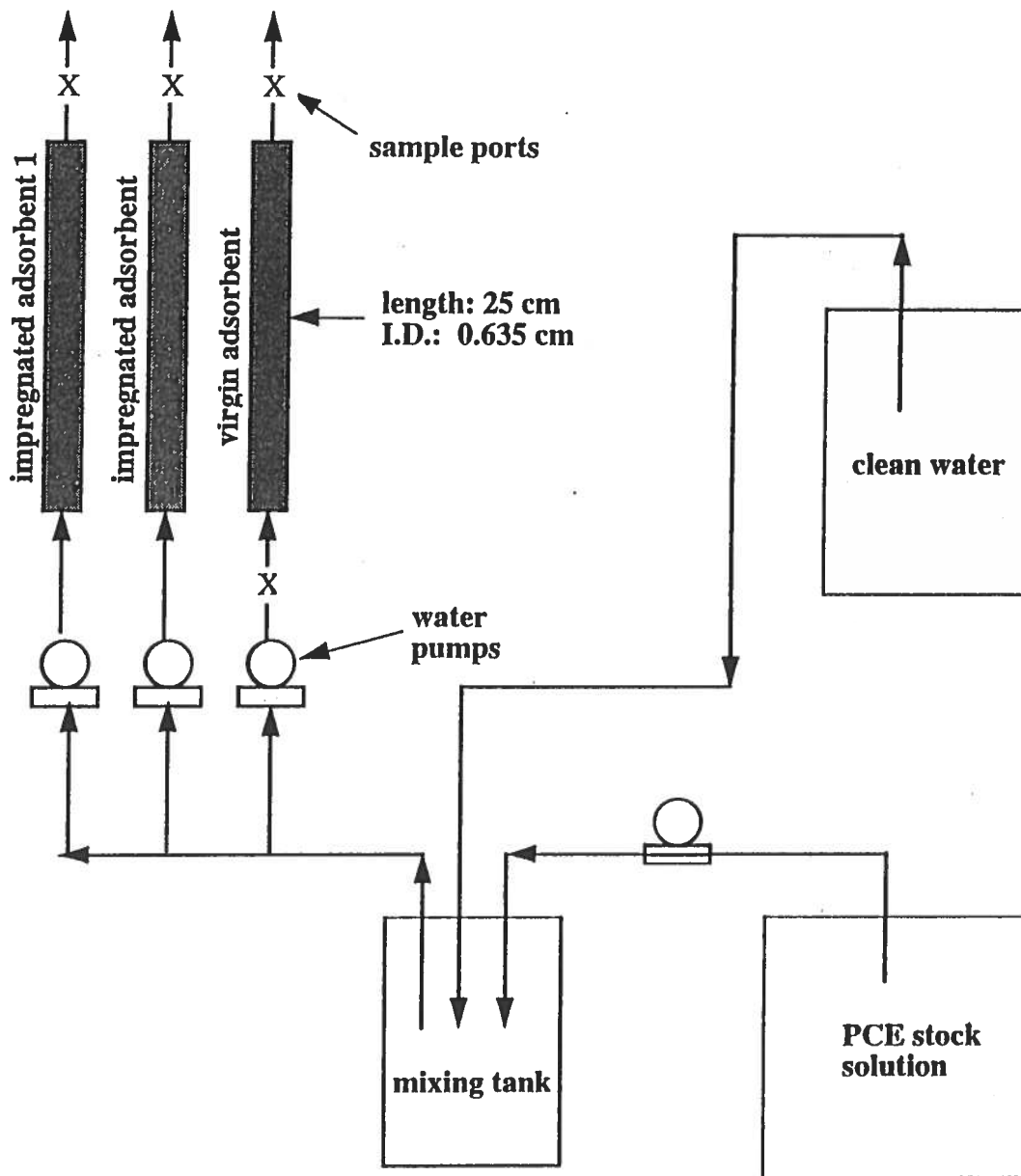


Figure 1. Schematic of the reactor setup for the field study.





**Figure 2. Schematic of the adsorption reactor set up for the laboratory study.**

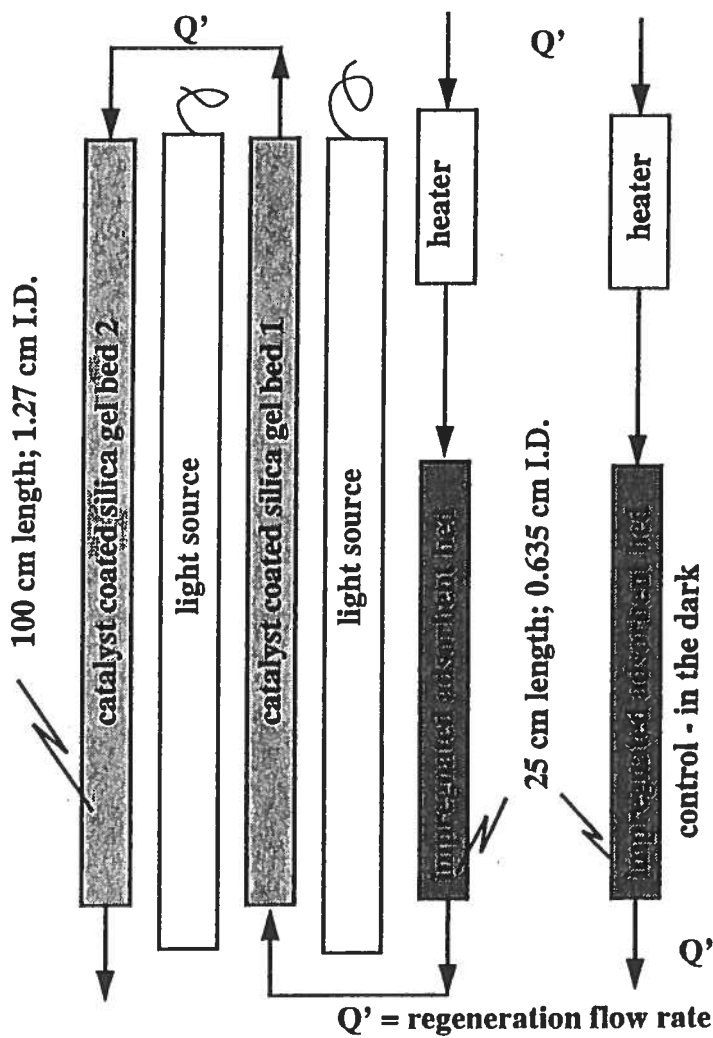


Figure 3. Schematic of the regeneration reactor set up for the laboratory study.

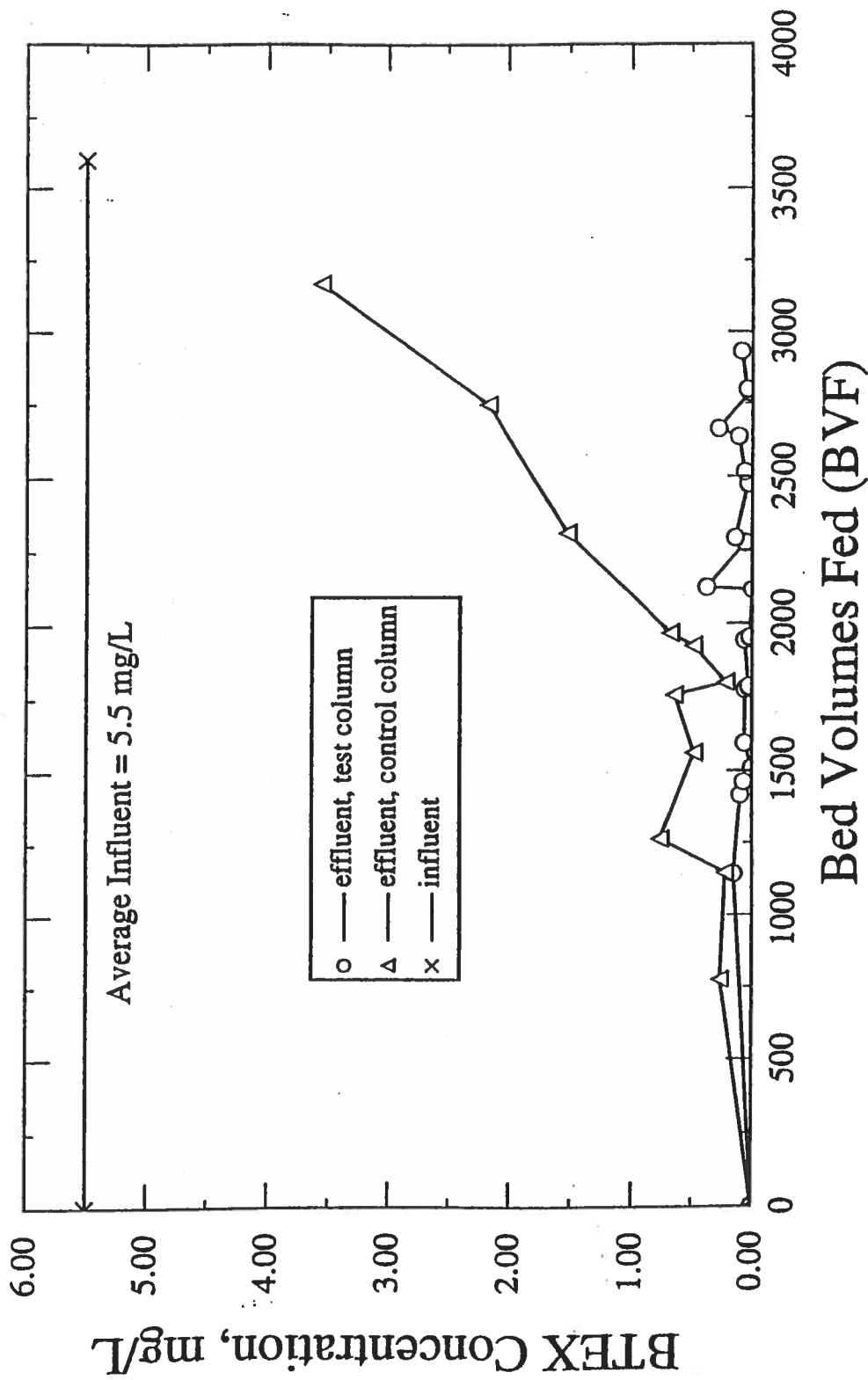


Figure 4. Comparison of BTEX effluent profiles from control and test (regenerated) column for the adsorption process. Empty bed contact time (EBCT) for test and control columns: 4.7 min and 3.4 min, respectively; loading velocity for test and control columns: 13 m/hour and 17 m/hour, respectively; column diameter: 0.635 cm; column length: 100 cm; adsorbent mass: 19.3 gm; bulk density: 0.61 gm/cc.