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Microwave Process for VOC Abatement

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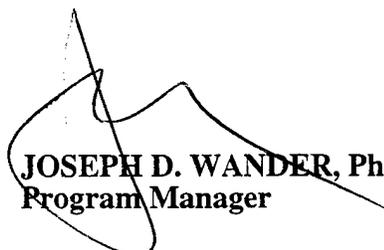
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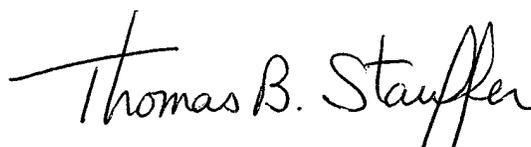
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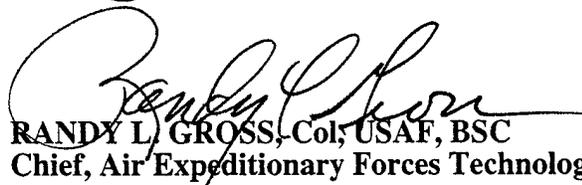
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Microwave Process for VOC Abatement

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ABSTRACT

The CHA Corporation has completed U.S. Air Force Phase II Small Business Innovation Research program to investigate the feasibility of using a novel microwave-based process for the removal and destruction of volatile organic compounds in effluents from non-combustion sources such as paint booth ventilation streams. Removal of solvents by adsorption followed by the regeneration of saturated granular activated carbon (GAC) by microwave energy was achieved in a single fixed-bed reactor. Microwave regeneration of the fixed-bed-saturated carbon restored the original GAC adsorption capacity. During microwave regeneration, the concentrated desorbed paint solvent was easily oxidized by passing the solvent mixture through a fixed bed of an oxidation catalyst mixed with silicon carbide. After completing the fixed-bed pilot testing, we assembled a pilot-scale continuous system and tested it during a three-month continuous demonstration at McClellan Air Force Base from 15 January to 16 April 1999. For this demonstration, a 100-CFM stream of extracted soil vapors was continuously passed through the pilot system and the airborne VOCs were removed. The success of this demonstration and pilot-scale testing at CHA Corporation show the technical feasibility of using this system at an Air Force paint booth installation to continuously clean ventilation air. Laboratory and pilot plant testing results are presented.

Keywords: microwaves, VOCs, abatement, solvent, catalytic oxidation, activated carbon, air purification.

INTRODUCTION

During paint stripping, painting, metal plating, surface finishing and cleaning operations, a large volume of air containing solvents and other volatile organic compounds (VOCs) is produced. These emissions may be continuous or episodic and quite variable in terms of solvent content ranging from a few parts per million (ppm) to parts per thousand (ppt). Thermal oxidation or incineration has been applied broadly to destroy VOCs¹. In the case of a dilute fume in air, the two methods for incineration are direct thermal oxidation

and catalytic oxidation. The main advantage of incineration is its potential for very high efficiency. If held for a sufficient length of time at a sufficiently high temperature, organic compounds can be oxidized to any desired degree of completeness. However, high temperature incineration requires a substantial amount of supplemental fuel and also produces secondary air pollutants such as NO_x. Microwave catalytic oxidation can reduce the required temperature by hundreds of degrees and saves a considerable amount of space for equipment as compared to thermal oxidation. Both high- and medium-temperature catalytic oxidation are costly. The filter techniques often used in these methods also have problems of saturation and ultimate disposal. Furthermore, the service conditions of low temperature and non-steady state operations that prevail in the U.S. military operations of cleaning, spray painting, and paint stripping, are ill-suited for catalytic oxidation. This is because catalytic oxidation performs best when the gas flow rates are nearly constant and the gas temperature is above 260°C. Therefore, there is a need to develop a technology that controls the fugitive emission of solvent vapors, such as those resulting from operations at Air Force bases.

As a part of the ongoing environmental cleanup of McClellan AFB, the U.S. Air Force has evaluated several innovative and conventional technologies for the treatment of VOCs². Conventional off-gas treatment technologies were found effective but costly. It was concluded that of the innovative technologies demonstrated only Flameless Thermal Oxidation, Microwave Regenerable Granular Activated Carbon Adsorption, and Photolytic Destruction Technology have the performance characteristic needed for full-scale implementation at McClellan AFB.

For the past 10 years, CHA Corporation has focused on the utilization of microwaves to induce or enhance chemical reactions. These efforts, made possible through industrial and governmental support, emphasize the development of new technologies for gas and water cleanup through selective destruction of pollutants in contaminated waste streams. The CHA microwave-based gas cleanup process is designed to capture and destroy a wide variety of both chlorinated and non-chlorinated VOCs, many of which are commonly found at industrial and military sites. In addition, the CHA process also removes the pollutants SO₂ and NO_x from flue gases. The technology has been successfully demonstrated at McClellan Air Force Base in March 1998 where NO_x and VOCs were removed from diesel engine exhaust gas^{3,4}. The microwave air purification process is also used in the site remediation field for the treatment of extracted soil vapors. We have successfully completed a pilot-scale demonstration at McClellan Air Force Base from 15 January to 16 April 1999. As a result from the pilot-scale demonstration, a significant advance in this technology has been made and commercialization of this microwave air purification process is immanent.

PROCESS DESCRIPTION

To overcome the disadvantages and limitations of current emission control options, CHA Corporation has developed a new approach for removing solvents and other air pollutants in air vented from U.S. military operations and university chemical laboratories. To prepare the microwave processing system, for large airflow rates, we have developed a microwave process utilizing granular activated carbon (GAC) moving-bed system for removal and destruction of hydrocarbon vapors in the air. Furthermore, a zeolite bed is

added to the moving-bed to remove ammonia and acid gases that are not easily adsorbed onto GAC. Figure 1 shows a process flow diagram for the microwave air purification process. The process consists of four main components, moving-bed GAC/zeolite adsorber, microwave regenerator, microwave oxidizer, and acid gas adsorber. If the air does not contain ammonia and/or acid gases, the zeolite bed and microwave regeneration reactor for saturated zeolite is not needed. The acid gas adsorber is not needed if the air does not contain chlorinated hydrocarbons. The following describes each of the four main components.

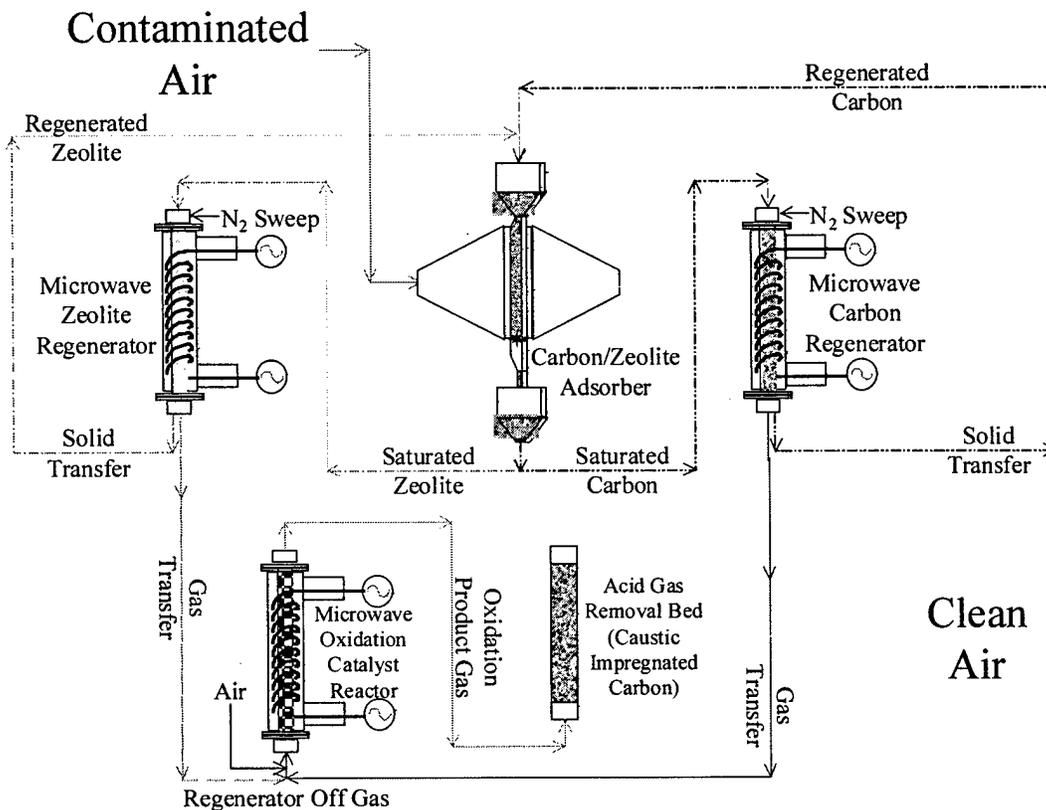


Figure 1. Process Flow Diagram for Microwave Air Purification Process

The moving-bed adsorber continuously treats the ventilated air stream containing VOCs, hydrocarbons, solvents, and acid gases. In the adsorber the VOCs, hydrocarbons, and solvents are removed from the ventilation air by adsorption onto the 4-inch thick moving bed of 3-mm, 80 carbon tetrachloride number (CTC) pelletized granular activated carbon. The 2-inch, 4 to 8-mesh natural zeolite bed removes any acid gases and ammonia contained in the air stream. These two adsorbents act as a concentrator in this type of process. The adsorbents remove the contaminants from the large air stream, and microwave regeneration concentrates them into a smaller stream that is more easily treated. The concentrated stream produced during regeneration of the saturated adsorbents usually has a gas flow rate that is approximately 1% of the original contaminated air stream. This concentrating effect is unique to the two adsorbents used in the process. Using these adsorbents keeps the cost of the treatment system low. The

adsorbents fed through the adsorption unit via a feed hopper at the top and travel downward via gravity.

The used adsorbents exit through star valves at the base of the adsorber, from which they are transported to the respective microwave regenerator feed hopper by pneumatic conveyor. The conveyor air is passed through a cyclone to remove any entrained solid particles during carbon transfer. After passing through the adsorber the solvent-free air is then filtered to remove the remaining fine carbon or zeolite particles.

Saturated GAC and zeolite are fed into their respective regenerator through the top feed hopper. Regenerated GAC and zeolite exit through a rotary valve at the base of their respective regenerator. The regenerator operates as a moving bed and regenerates the saturated GAC and zeolite via microwave energy. The microwaves cause the contaminants to rapidly desorb from the adsorbent and return to the vapor phase. The solvent vapors are removed from the bottom of the regenerator by a small, constant stream of nitrogen purge gas injected at the top of the regenerator. This sweep gas containing VOCs, hydrocarbons, solvent, or acid gas flows into a water-cooled condenser and liquid solvent is collected in a knockout pot. Remaining solvent vapor in the N_2 sweep gas and air that is needed for combustion of the solvents are then fed to the microwave-based catalytic oxidizer. It should be noted that the solvent vapor concentration in the painting ventilation air is low enough to disallow solvent condensation on the GAC. The regenerated GAC and zeolite are then returned to the top of the adsorber by a pneumatic conveyor.

The microwave catalyst reactor is packed with an oxidizing catalyst of platinum (Pt) and palladium (Pd) coated onto alumina beads impregnated with 25 percent 800-mesh SiC. The nitrogen stream containing solvents and acid gases from the regenerators is mixed with sufficient air for combustion and fed to the oxidizer. The microwaves and catalyst work together to efficiently oxidize the concentrated solvent/acid gas-laden stream at low temperatures. For non-chlorinated solvents the combustion products will be CO_2 and H_2O , which can be safely vented to the atmosphere. If chlorinated solvents are present, the combustion products will be CO_2 , H_2O , and HCl. The gas containing HCl leaves at the top of the reactor and flows through an acid gas scrubber. The acid gas scrubber is a caustic impregnated carbon bed.

The acid gas removal-bed removes any acid gases remaining in the oxidation product gas stream after the microwave catalytic oxidizer. Carbon, impregnated with sodium hydroxide (NaOH), is loaded into a column designed for proper treatment of the oxidation product stream. Any acid gases contained in the oxidation product gas reacts with the caustic to form water and the respective salt. For instance, hydrogen chloride (HCl) will leave water (H_2O) and sodium chloride (NaCl), both of which are harmless. The carbon bed is replaced when the acid gas removal efficiency diminishes. Clean gas exiting from the acid gas removal-bed is mixed with the clean air from the carbon/zeolite adsorber and vented to the atmosphere.

The microwave process offers a number of advantages over other available conventional treatment technologies. The microwave technology gives the option of either recovering or destroying the solvents, ammonia, acids, and other hazardous air pollutants. The use

of microwave energy allows a continuous regeneration of GAC and zeolite on site with a minimum loss of each adsorbent. Additionally, less energy is used in the regeneration process because the bulk temperature is much lower than conventional steam regeneration processes. No secondary pollutants such as NO_x are produced during regeneration. Microwave catalytic oxidation of solvents, ammonia and acids eliminates any need for handling and disposal of recovered wastes. Furthermore, the volume of gas produced from regeneration is about 1% of the volume of the contaminated ventilation air being treated. This volume reduction or contaminant concentration step allows for a 100 to 1 reduction in oxidation equipment scale. The use of NaOH coated activated carbon eliminates the need for an acid gas wet scrubber.

LABORATORY RESULTS

Adsorption Experiments

Solvent removal experimental work using granular activated carbon (GAC) has been conducted for several different solvent types. These solvents include methyl ethyl ketone (MEK), 2-butanol and a mixture of 83% methyl *n*-propyl ketone (MPK) and 17% butyl acetate. Adsorption breakthrough testing was performed on all three solvents while carbon regeneration cycling was implemented using only MEK.

Adsorption testing for 2-butanol and MPK was performed using a 2-inch-ID tube containing the adsorbent bed. Adsorbent height was varied from 1.5 to 6 inches to manipulate the gas space velocity as desired. Inlet gas flow was held constant at 130 SCFH. The solvent was added to the flow stream by sparging a small amount of the solvent into a nitrogen carrier gas flowing into an air stream. The average inlet concentration for all experiments was 200 ppm solvent.

Testing of the MEK was carried out using a quartz tube (1-inch-ID) containing a column of GAC. The bed height was fixed and the volumetric gas flow rate was manipulated to achieve the desired gas space velocities. MEK was applied using a sparger apparatus similar to the one mentioned above. All regeneration tests were performed in the quartz tube with a nitrogen purge stream to prevent oxidation of the carbon adsorbent. Saturated carbon samples were regenerated using 200 W of microwave power.

The surface area is proportional to the available active adsorption sites for a given amount of adsorbent. The surface area trend throughout the lifetime of the carbon remained virtually constant. The adsorption capacity of the GAC for MEK is a measure of the adsorbent weight gain due to adsorbed MEK in grams MEK per 100 grams of GAC. The experimental adsorption capacity data remain relatively constant with a slight variance comparable to reasonable experimental deviation.

In addition to the extended adsorption capacity and surface area data taken for MEK adsorption on GAC, average adsorption capacity data were experimentally determined for the various solvent types using 200 ppm solvent concentration in the sample gas and presented in Table 1. The gas hourly space velocity (GHSV) used in these adsorption experiments was 20,000 hr⁻¹. Gas hourly space velocity represents the ratio of the

volumetric gas flow, Q (ft^3/hr), to the volume of the adsorbent or catalyst bed, V (ft^3), providing a scalable parameter with the unit hr^{-1} .

Table 1 Solvent Adsorption Capacities for Granulated Activated Carbon
(GHSV=20,000 hr^{-1} , Inlet concentration=200ppm)

Solvent	Adsorption Capacity, g Solvent/100 g C	Solvent	Adsorption Capacity, g Solvent/100 g C
Methyl n-propyl Ketone	13.1	Hexane	17.5
2-Butanol	16.5	Methylene Chloride	4.7
Methyl Ethyl Ketone	13.2	Acetone	3.9
Trichloroethylene (TCE)	30.5	SC-100	36.3

Complete microwave regeneration of GAC saturated with MEK proved successful for 20 cycles without diminishing adsorbent performance using inlet 200 ppm MEK concentration and 20,000 hr^{-1} GHSV. Figure 2 illustrates the surface areas and adsorption capacity data for the GAC over the 20 adsorption/regeneration cycles.

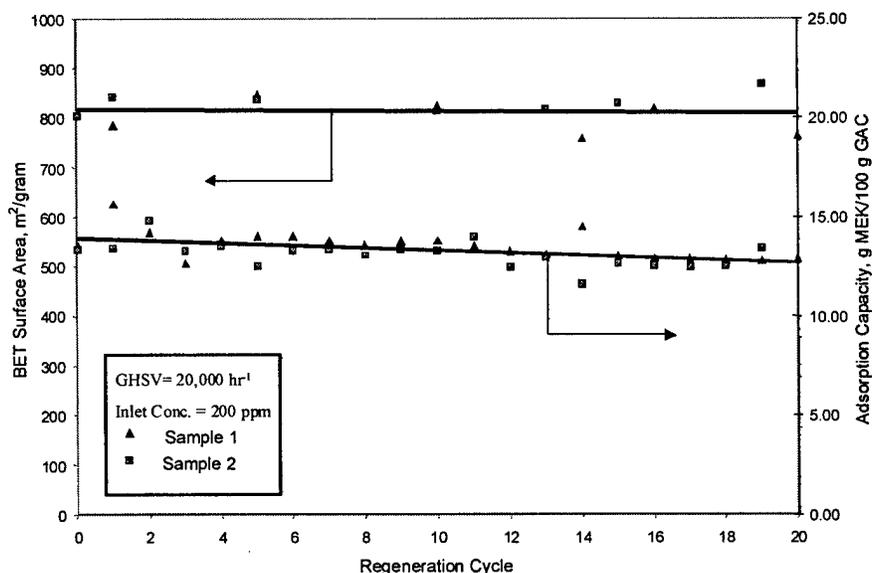


Figure 2. Effect of Microwave Regeneration of GAC on Surface Area and Adsorption

The consistency of the surface area and adsorption capacity results over extended microwave regeneration shows that the microwave regeneration of the carbon adsorbent does not decrease adsorption performance, even after many cycles. This consequently demonstrates the feasibility of economically utilizing GAC for the cleanup of low-concentration solvent streams.

Adsorption breakthrough testing was performed for each of the three solvent types at a variety of gas space velocities. Breakthrough curves represent the behavior of an adsorbent as it approaches saturation. Initially, assuming sufficient adsorbent, no solvent

will be present in the outlet gas stream. As the adsorbent begins to saturate, a point is reached at which the adsorbed species appears in the outlet gas stream. This is the point of breakthrough. After initial breakthrough, the outlet concentration slowly increases until it equals the inlet concentration upon reaching adsorbent saturation. Figure 3 represents dimensionless breakthrough profiles for the three tested solvents each at four different GHSVs. The dimensionless time shown is the ratio of the experimental time to the time required for a 50% breakthrough, t_{50} , (i.e., when the outlet concentration is equal to $\frac{1}{2}$ the inlet concentration).

Each of the runs shows similar breakthrough behavior for the respective solvents over the range of tested GHSV. As the GHSV is increased, the initial breakthrough time as a fraction of the 50% breakthrough time decreases. This decreased initial breakthrough time serves to decrease the average removal efficiency as calculated at the 50% breakthrough time.

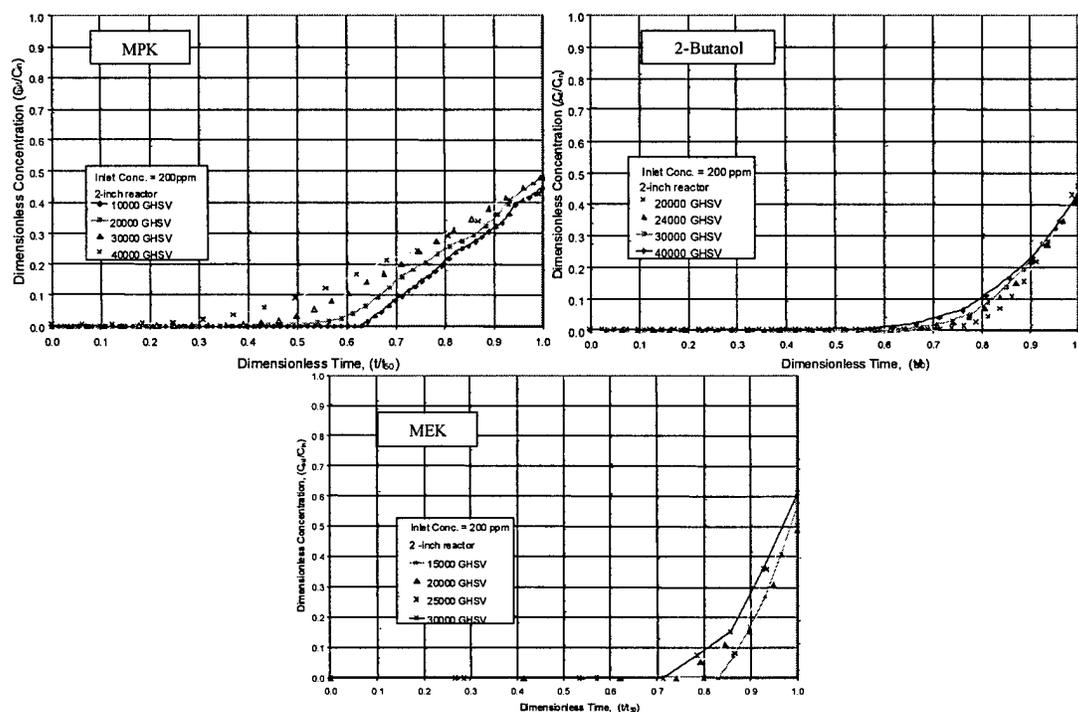


Figure 3. Adsorption Breakthrough Curves for MPK Mixture, 2-Butanol, and MEK on GAC

Actual breakthrough times for all tested samples and conditions are presented in Figure 4. These breakthrough curves, as expected, show a clear correlation between initial breakthrough time and GHSV. Lower GHSV correspond to later breakthrough times, while higher velocities yield earlier breakthrough times.

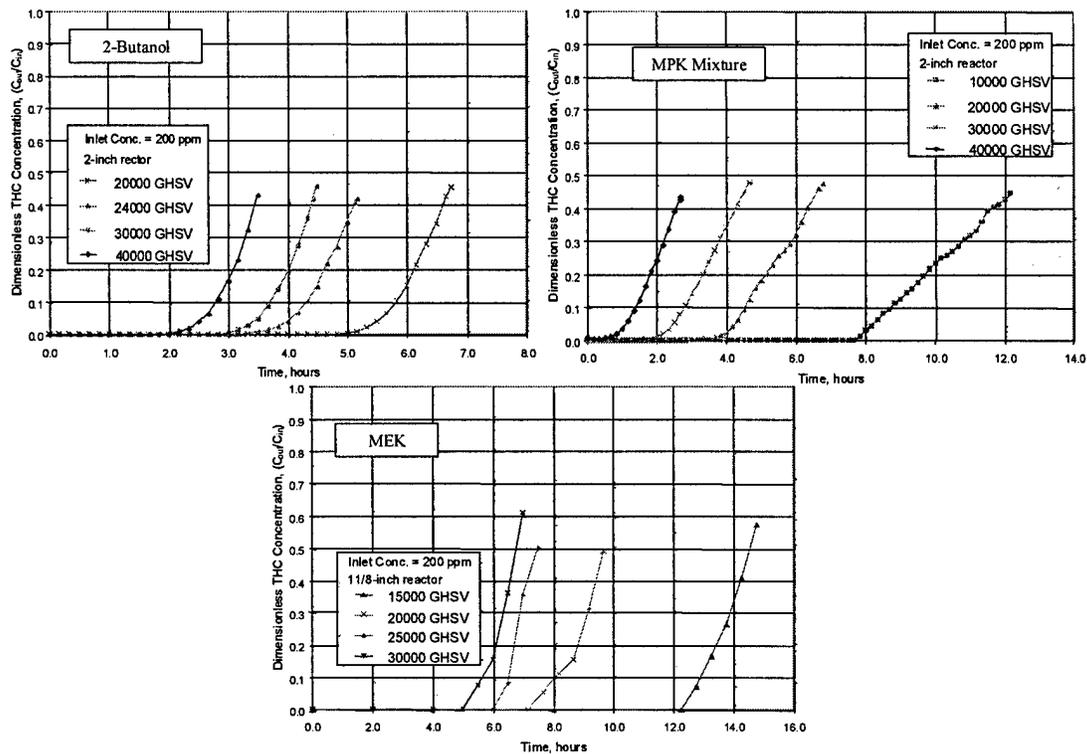


Figure 4. Breakthrough Curves for 2-Butanol, MPK Mixture, and MEK on GAC

Figure 5 depicts the average solvent removal efficiencies for GAC as a function of GHSV. The reported removal efficiencies are calculated by integrating the dimensionless 50% breakthrough curves and dividing the total solvent adsorbed by the total solvent entering the system. This provides a means of analyzing the adsorption performance over a range of GHSVs. As Figure 5 illustrates, the average removal efficiency decreases slightly with increased GHSV for all solvent types, but even at very high GHSV levels, average removal efficiencies above 90% can be expected.

The actual 50% breakthrough times for GAC adsorption of the tested solvents are given in Figure 6. Over the range of experimental testing for all solvents, 50% breakthrough times were decreased with increasing gas space velocities. Extended microwave regeneration cycling of the granulated activated carbon has shown that adsorption capacity and adsorbent surface area do not suffer over-long term use. This longevity makes the carbon adsorbent an economic choice for effective gas cleanup.

The adsorption and microwave regeneration testing of solvents on GAC has demonstrated the propensity for effective cleanup of spray paint-booth solvent. Analysis of the carbon adsorption performance as a function of GHSV provides scalable data that will allow for the design of the demonstration-scale spray paint booth clean-up device.

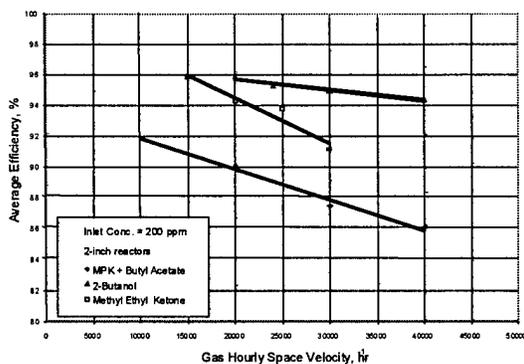


Figure 5 Average Solvent Adsorption Efficiency for GAC

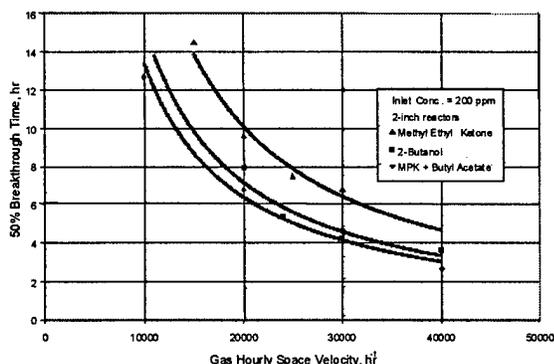


Figure 6 Solvent 50% Adsorption Breakthrough Time for GAC

Destruction of Solvents during Regeneration of Saturated Carbon

After activated carbon is saturated with spray paint solvents, microwave energy is used to regenerate the saturated carbon. The solvent adsorbed onto the activated carbon quickly desorbs when exposed to microwave energy. The solvent released from the adsorbent can be either collected by condensation or destroyed by microwaves. A series of experiments were performed to determine collection and destruction efficiencies as a function of microwave power absorbed and gas residence time through the oxidation catalyst bed.

A series of solvent destruction tests were performed to determine the minimum microwave power required achieving greater than 99% solvent destruction. Microwave solvent oxidation testing was carried out under ambient conditions. Low-level microwave power was used to facilitate oxidation. Microwave solvent destruction testing for each sample was performed using a 7/8-inch diameter quartz tube containing a bed of oxidation/reduction catalyst (PRO*VOC*7, manufactured by Prototech Company) on SiC. The bed height and volumetric gas flow rate were varied to manipulate GHSV and determine the effect of bed height on conversion performance. The solvent was sparged into the inlet gas stream with nitrogen. The solvent-rich nitrogen stream was then combined with a flowing air stream prior to entering the reaction chamber. The ratio of the flows of the two gas streams was held constant over all experiments. Inlet solvent concentrations were maintained well below the lower explosive limit for all of the microwave destruction tests.

Minimum microwave power data were compiled by manipulating the applied microwave power while the solvent-laden air was continuously flowing through the catalyst bed of the reactor. Microwave power was slowly increased and allowed to stabilize at each increment, until no remaining solvent could be found in the outlet stream. Figure 7 depicts the minimum microwave power required per cubic inch of oxidation catalyst as a function of GHSV to achieve complete solvent destruction for various solvents at 200-ppm inlet concentration. Slightly higher microwave power is required for oxidation of the solvent mixture when using the 3-inch catalyst bed rather than a 4-inch bed.

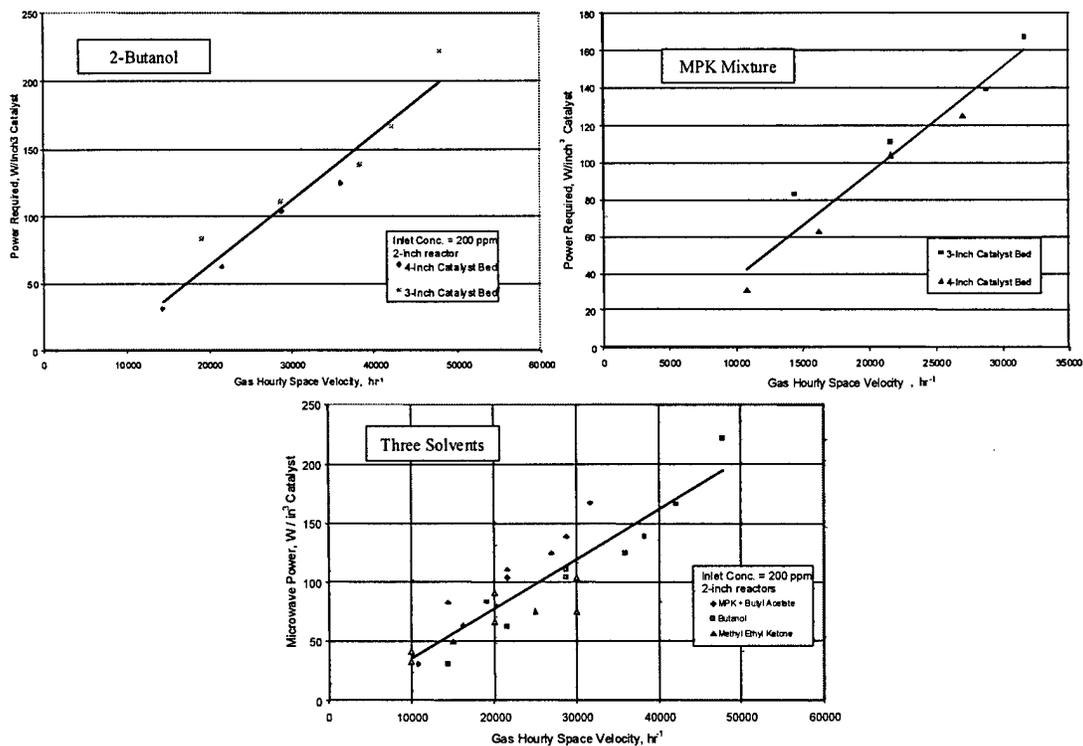


Figure 7. Minimum Microwave Power Level for Complete Solvent Destruction

Similar to the data from the solvent mixture of methyl *n*-propyl ketone and *n*-butyl acetate, using 4 inches of catalyst bed, data from both runs hold relatively well to the trend line. Destruction efficiency and minimum power required were shown to be dependent on bed height only to the extent that the GHSV was altered, whenever a minimum bed height was achieved. This minimum bed was experimentally determined to be approximately 1-1/2 inches.

PILOT-SCALE DEMONSTRATION

Design, Fabrication, and Installation of Pilot-Scale Control Device

A representative cycle of large aircraft painting operation is 11 days: 9 days for preparation and 2 days of painting. Consequently, a batch process is suitable for the removal and destruction of solvents in the air that is vented from painting and paint removal operations. A pilot-scale solvent removal system was designed to treat a 100-CFM airflow rate. The pilot-scale control system is depicted in Figure 8. A fixed-bed adsorber was fabricated. The finished adsorber picture is shown in Figure 9. The bed was of square configuration, and was able to serve as a single- or two-stage adsorber. The adsorber was fitted with copper meanderlines for distribution of the microwaves, and two magnetrons were mounted to each meanderline. A picture of this meanderline is also shown in Figure 9.

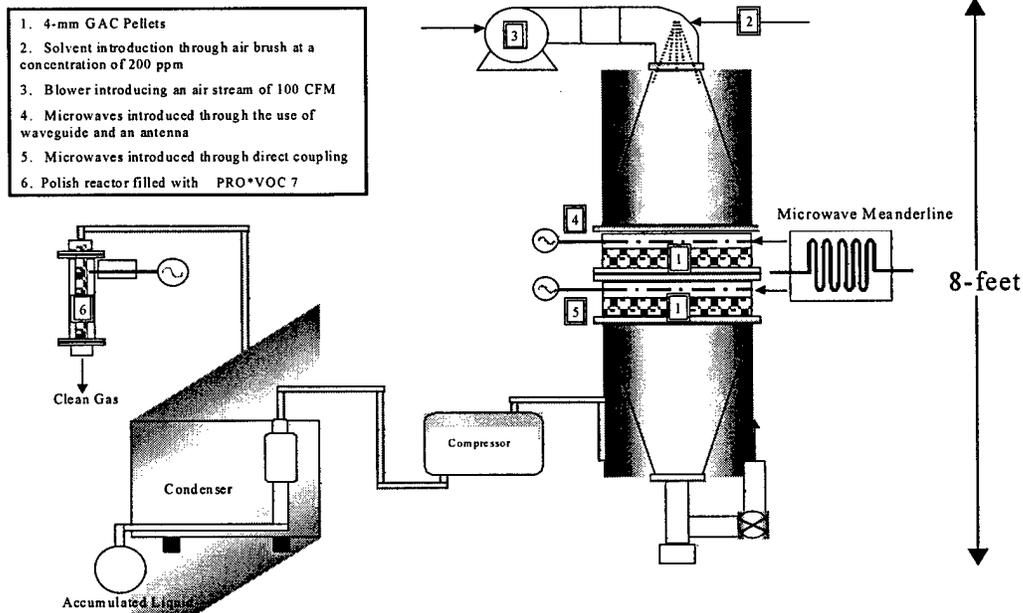


Figure 8. Pilot-Scale Control System for Removal and Destruction of Solvent in Air

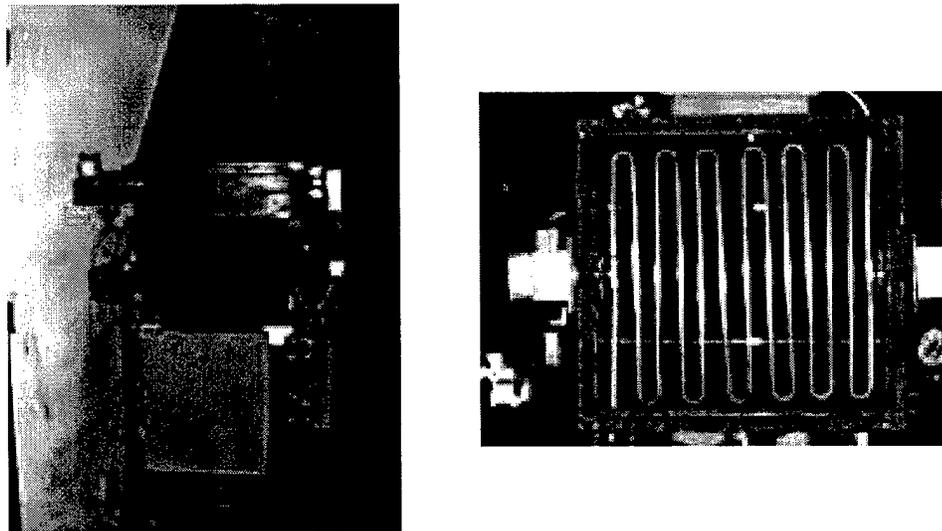


Figure 9. Fixed-Bed Adsorber and Meanderline Inside Adsorber

A 120-CFM blower supplied air to the fixed-bed adsorber. A precision liquid metering pump attached to an airbrush was used to inject solvent into the air stream. A total hydrocarbon (THC) analyzer was used to determine inlet and outlet solvent concentrations. The blower, pump, and analyzer setup is shown in Figure 10. A water-cooled condensing unit collected paint solvents during the regeneration process. A 2.31-inch microwave catalytic oxidation reactor was used for oxidation of any remaining solvent in the regeneration gas after the condenser. The microwave catalytic oxidation reactor is also pictured in Figure 10.

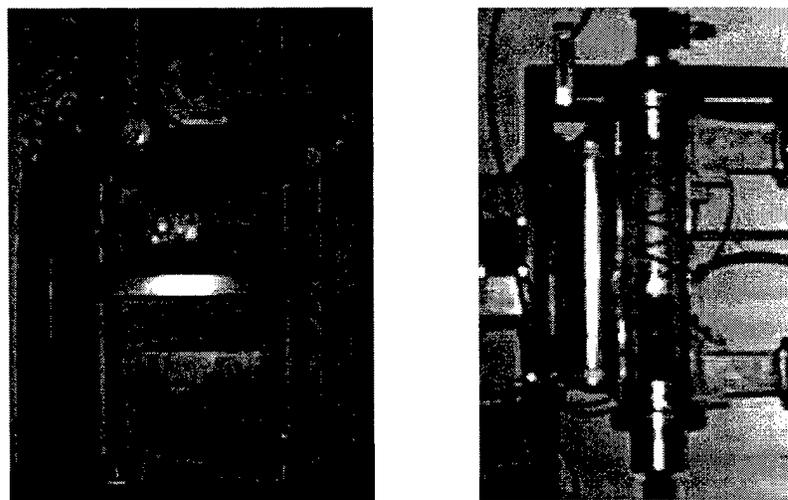


Figure 10. Blower and Airbrush Setup, with THC Analyzer and Microwave Catalytic Oxidation Reactor

Test Operation of the Pilot-Scale Treatment System

Several variables were investigated during the test operation of the solvent-removal system. First saturation time was established. To determine this time several experimental runs were performed using 2-butanol as the solvent. The experiments were run using a total air flow rate of 100 CFM, which contained 200 ppm of the solvent. Only one stage of the adsorber was filled with two inches of GAC. The experiments were continued until the outlet concentration of solvent in the air stream reached 50% of the inlet concentration. The air samples were analyzed continuously using a THC analyzer. The bed dimensions were 18x18x2 inches. The time that it took for the solvent concentration in the outlet gas to reach 50% of inlet concentration for these conditions averaged about 10 hours.

The second system characteristic tested was the rate at which the carbon could be regenerated. During the regeneration process, the adsorption bed was exposed to 2 kW of applied microwave power that was distributed throughout the bed by a meanderline. During regeneration, the unit was swept with carbon dioxide to remove the solvent vapors and carry them to the condenser. Any remaining vapors were directed to the microwave catalytic oxidizer. The total regeneration time was found to be 3 hours. Figure 11 presents adsorption breakthrough curves for three adsorption/regeneration cycles. The amount of solvent removed when the outlet concentration reached 50% of inlet concentration remained approximately the same after two regeneration cycles.

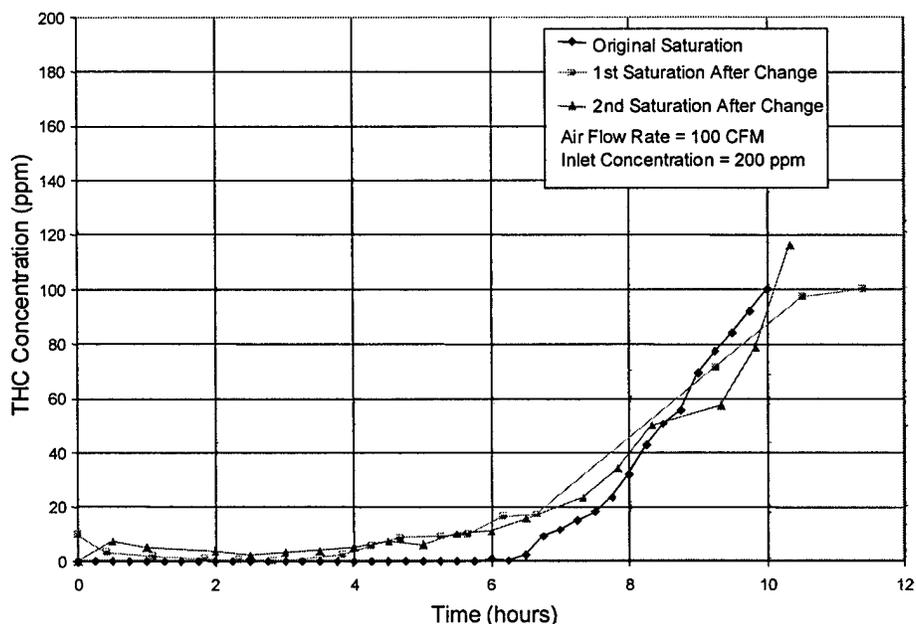


Figure 11. Adsorption Breakthrough Curves for 2-Butanol Comparing Original GAC and Microwave-Regenerated GAC.

The oxidation catalyst bed was exposed to microwave powers of 700, 800, and 900 W, and the bed gave destruction rates of over 98% at each of these power levels. A material balance was completed to determine the amount of 2-butanol that could be accounted for either in the vapor form, the liquid condensed form, or remaining on the GAC. The material balance was calculated by measuring the inlet 2-butanol adsorbed and comparing it to the amount of butanol recovered as a liquid and vapor in the outlet gas. Table 2 shows 2-butanol recovery during each cycle. For the first saturation/regeneration cycle, the carbon bed adsorbed 792 mL of 2-butanol and 500 mL of 2-butanol was subsequently recovered in liquid form during regeneration. 194 mL was recovered in vapor form. For the second cycle, 989 mL was adsorbed on the carbon, 400 mL was recovered in liquid form, and 568 mL was recovered in vapor form. The final material balance closures were 88% with the first regeneration and 98% for the second regeneration. Cracking during microwave regeneration and fluctuations in the solvent concentration in the outlet gas are possible source of errors in the material balance determinations.

Table 2. 2-Butanol Material Balance

Cycle Number	Amount of 2-butanol Input, mL	Amount of 2-butanol recovered in liquid, mL	Amount of 2-butanol recovered in vapor, mL	Material Balance
1	792	500	194	88%
2	989	400	568	98%

After testing was completed using conventional GAC, pelletized activated carbon was tested. Pelletized activated carbon is a much more durable adsorption agent than the conventional GAC, making it an attractive alternative. The pelletized carbon had a

comparable surface area to the conventional GAC (1000 m²/g), and in the single-stage setup showed an initial adsorption capacity approximately the same as the conventional GAC. The regeneration of the pelletized carbon took much longer than the conventional GAC. The regeneration time for the single-stage system containing the pelletized carbon was found to be 12 hours. The second stage of the reactor was then installed for testing. Use of the second stage brought the breakthrough time for the sec-butanol up to 23 hours. The initial adsorption capacity of the pellets for this run was 14g solvent per 100g carbon. The adsorption capacity of the carbon dropped to 11g solvent per 100g carbon for the second cycle. As a result, conventional GAC was used in subsequent solvent adsorption/recovery tests.

Testing of the two-stage adsorption arrangement followed. Furthermore, we prepared a mixture of solvents identical to that found in Deft Chemicals Paint solution used in the Air Force painting operations. The mixture consists of 38% methyl *n*-propyl ketone, 32% SC-100 and 30% 2-butanol by weight. SC-100 is an aromatic hydrocarbon replacement manufactured by Exxon Chemicals. Two bed depths, 1.5 and 1.75 inches, were tested for adsorption/regeneration characteristics to determine the optimum bed thickness. Both bed thicknesses provided equivalent adsorption characteristics, but during the regeneration process the thicker bed retained more solvent. Therefore, the arrangement having two 1.5-inch beds was selected for further testing. Figure 12 shows the THC concentration of the outlet nitrogen sweep gas as a function of time during regeneration of the carbon beds.

Extended testing of the two-stage adsorption system, using two 1.5-inch thick GAC-bed configurations was then performed. Six adsorption/regeneration cycles were completed to see how the adsorption capacity would be effected over time. For all six cycles, comparable adsorption capacities were seen. Table 3 presents the adsorption capacity of regenerated GAC, breakthrough time, and regeneration time for each of five cycles. For the first cycle, liquid solvent was accumulated inside a condenser and only 40 g of liquid solvent was recovered.

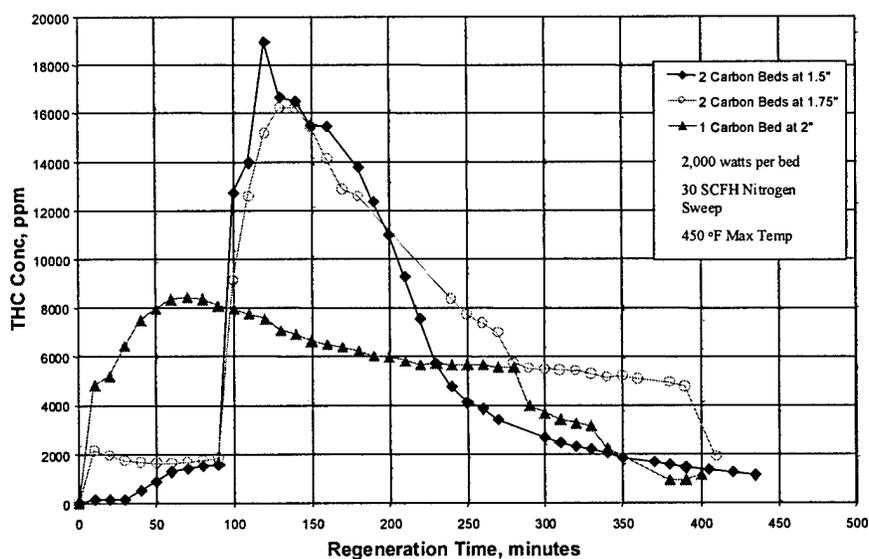


Figure 12. THC Conc. in the Outlet N₂ Gas as a Function of Regeneration Time

Table 3 Adsorption/Regeneration Results of Two-Bed System

Cycle Number	1	2	3	4	5
Adsorption Capacity g Solvent/100g Carbon	16.0	13.0	16.2	13.2	16.5
Breakthrough Time, hrs	12.0	9.8	12.1	10.0	12.4
Regeneration Time, hrs	13.0	21.5	7.0	6.0	6.5
Liquid Solvent Collected, g	40	350	345	478	490

Figure 13 illustrates the results of the regeneration for each cycle. In the first regeneration cycle, microwaves were applied to the top bed first and after one hour to the bottom bed. In the second cycle, microwaves were applied to both top and bottom beds simultaneously. In Cycles 3 through 6, microwaves were applied to the top bed first and then to the bottom bed after 1.5 hours. More liquid solvent was recovered in the final four cycles than in the first two. This suggested that a sequential regeneration is better than simultaneous regeneration. When higher liquid solvent volumes were recovered, corresponding subsequent higher adsorption capacity of regenerated carbon should be obtained. However, this is not clear in adsorption data shown in Table 3.

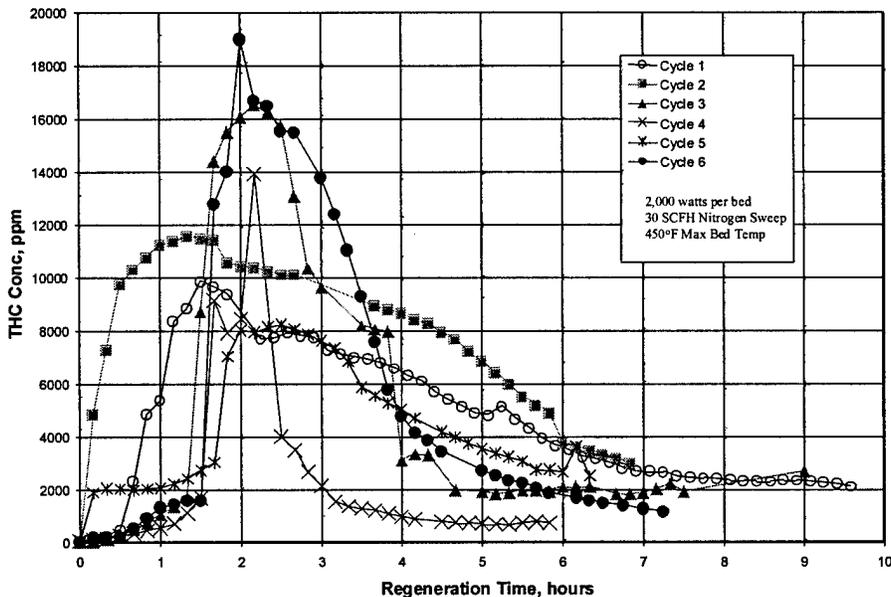


Figure 13. THC Concentration in Outlet Gas as a Function of Regeneration Time

Vertical Moving-Bed Adsorber

The microwave regeneration of a fixed bed of GAC is very inefficient, as shown above. The amount of GAC contained in two 1.5-inch beds can be regenerated in a 2.31-inch moving-bed within one hour compared to 6 hours for the fixed bed, as shown in Table 3 and Figure 13. Consequently, a moving-bed system, Figure 1, will be more cost effective for the removal and destruction of solvents in the air that is vented from painting and paint-removal operations. Microwave regeneration of saturated GAC in the 2.31-inch moving bed has been successfully demonstrated at McClellan AFB in Sacramento,

California. As a result, we have sufficient data to design a moving-bed microwave reactor for regeneration of saturated GAC.

A new vertical moving-bed adsorber was designed and has been fabricated. A schematic diagram of the new adsorber is shown in Figure 14. The newly constructed adsorber is shown installed for testing in Figure 15. The vertical adsorber is 22 inches square. Bed thicknesses of 2, 4, and 6 inches can be tested. The adsorber is attached to a blower with an airbrush for solvent injection.

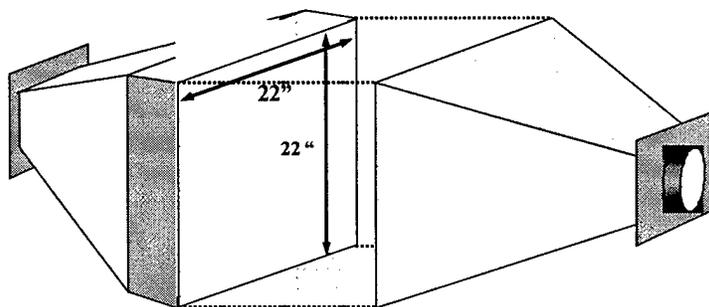


Figure 14. New Vertical Moving-Bed Adsorber Diagram

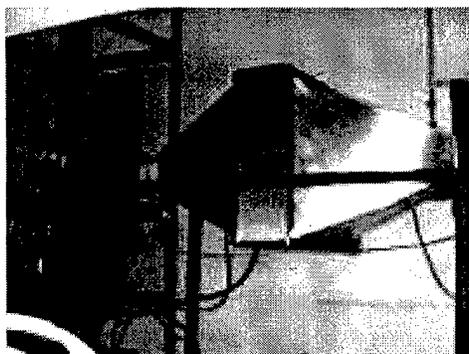


Figure 15. Vertical Moving-Bed Adsorber Installed for Testing

An air flow rate of 90 CFM was used and a mixed solvent, as used before, was injected into the air stream at a concentration of 90 ppm. The bed was loaded with 3-mm pelletized GAC. Bed thicknesses of 2, 4, and 6 inches were tested to determine the adsorption capacity of GAC and breakthrough time. Airflow continued until the removal efficiency of the GAC adsorber dropped below 95%. Figure 16 shows the concentration of solvent in terms of THC (propane standard) as a function of time for three different bed thicknesses. Breakthrough time, 95% removal efficiency time, and adsorption capacity results are presented in Table 4. The solvent adsorption capacity of pelletized GAC increased with increasing bed thickness as expected. The increase in breakthrough time due to the increase in the bed thickness is also non-linear.

**Table 4 Adsorption Test Data for 3-mm GAC beds
(Vertical Adsorber with 90 CFM Air Flowrate)**

Bed depth, Inches	Breakthrough Time, hrs	Time to Reach 95% Solvent Removal, hrs	Adsorption Capacity, G solvent/100g carbon
2	18.50	24.6	19.2
4	50.50	55	19.3
6	79.50	84	20.6

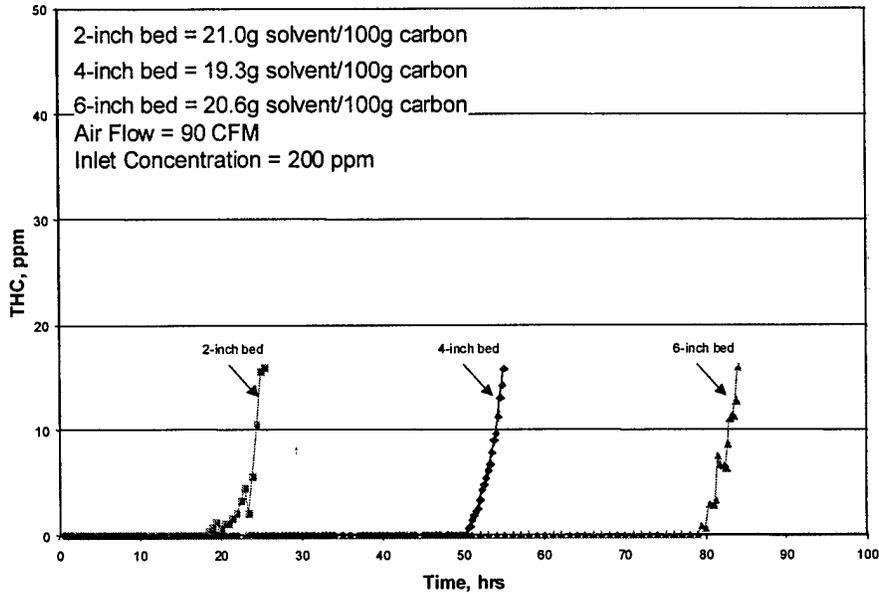


Figure 16 Breakthrough Curves for 2-, 4-, and 6-inch Carbon Beds.

CAPITAL AND OPERATING COST ESTIMATE

A typical cycle of C-130 aircraft painting operations at Hill Air Force Base is 11 days: 9 days for preparation and 2 days of painting. A batch process is compatible for the removal and destruction of solvents in the air that is vented from the painting and depainting operations. However, the microwave regeneration of a fixed GAC bed was found to be very ineffective. Operating the process shown in Figure 1 in a semi-continuous mode would be an economically superior treatment method. The granular pelletized activated carbon (GAC) adsorber shown in Figure 1, would be operated as a batch contactor during the two-day painting cycle. The microwave regenerating system, including the microwave catalytic oxidizer would be then operated continuously to regenerate saturated GAC in the adsorber. The description of three main sections is given below:

1. **GAC Adsorption Unit.** The air vented from the paint booth will flow through a GAC adsorption unit. The adsorber will be operated as a batch fixed bed during painting operations. Pelletized activated carbon will be used as the adsorption medium. The adsorption bed will be 8 feet x 8 feet x 6 inches and contain 1,000 pounds of GAC.

2. **Regeneration Unit.** The spent GAC removed from the adsorber will be pneumatically transferred to an on-site regeneration unit utilizing microwave energy. The carbon will be continually fed through a microwave reactor that is operated with 8 kW of microwave power. A 4-inch rotary valve will control flow of the carbon through the reactor. During the course of regeneration the carbon column in the reactor is swept with nitrogen.
3. **Microwave Catalytic Oxidation Unit.** The nitrogen stream containing solvent vapor produced during regeneration flows through the condenser and is then combined with air and passed through an oxidation unit. The oxidizer contains oxidation catalyst (PRO*PEL*VOC, manufactured by Protech Company) coated onto silica beads and imbedded with 800-mesh SiC. A continuous 4 kW of microwave energy will be applied to the catalyst bed. At these conditions, we have established that VOC destruction exceeds 99%.

This estimate provides the direct cost elements, on a current basis, for constructing a plant to treat 3,000 SCFM of a gas containing approximately 200 ppm mixed solvent vapors using a semi-continuous microwave air purification process. The planned treatment cycle would be to run the adsorber during the two days of painting, and regenerate the saturated pelletized activated carbon during the two following days. The estimating technique used in this report is based on the establishment of the process flow diagram shown in Figure 1, a major equipment list and equipment purchase costs. Where possible, equipment costs were obtained from the manufacturer. In some cases, equipment costs were obtained from established pricing charts based on a characteristic of the unit operation⁵. In some cases, a preliminary design and material take-off were made and the equipment priced on an appropriate "per pound" material basis

The total capital cost for the microwave air purification system treating 3,000 SCFM was estimated to be \$218,018. To obtain this estimate we multiplied the equipment purchased cost by a Lang factor⁵ of 3.45 to get the installed equipment cost of \$174,415. Then, a 25% contingency allowance was added to estimate the total capital cost. This estimate includes only the battery components shown in the process flow diagram in Figure 1, and does not include such things as site preparation expenses, including power supply and telephone lines, spare parts, or fees for permits, royalties, or licenses. Since all of the costs were based on factored estimates, the overall accuracy is judged to be $\pm 30\%$.

The plant is assumed to operate at the treatment rate of 3,000 CFM for two 8-hour workdays of adsorption, followed by two days of regeneration. This estimate provides the necessary operating elements, on a current basis, for continuous operation of the plant to treat air containing solvent vapors using a microwave air purification process. The estimating technique used was based on the previous capital cost estimate, and established industry costs. Included in the estimate are labor, maintenance, supplies, and utility costs.

For labor costs it was assumed that one technician could operate the treatment plant during the regeneration. No worker would be needed during the adsorption time, as the system will be automated. Assuming a salary of \$30 per hour during regeneration, the

annual labor cost is then calculated to be \$10,000. Utility costs are those provided by facilities outside the specific plant boundaries. Energy requirements can be calculated from energy balance equations or the sum of specific unit power requirements. For our plant the total electric power needed is 5,000 kW-hr. This power will supply 12 magnetrons, a 3,000-CFM blower, and two vacuum lifts. A cost of \$0.075 per kW-hr was used to estimate the annual utility costs. An energy efficiency multiplier of 0.9 was used to get the major equipment utility cost. An additional 10% was added to account for any supporting energy equipment. This includes additional pumps, chillers, fans, etc. that may be needed. The final value estimated for utility costs was found to be \$500 per year.

Maintenance and material supply costs are those associated with maintaining the plant equipment in operating order. These costs include equipment and labor used in repairs, magnetrons, transformers, and replacement of the activated carbon. The estimate for maintenance and supply costs is obtained by multiplying the total capital investment by 3%. For our estimate the annual maintenance and material supply costs are \$6,540.

Since the treatment plant is a part of the overall of painting and paint removal facility operations, taxes and insurance are not included in this estimate. The total annual operating cost for the microwave air purification system is estimated to be \$17,040.

CONCLUSIONS AND RECOMMENDATIONS

The CHA Corporation has completed an investigation of the feasibility of using a novel, microwave air purification process to remove and destroy VOCs in effluents from non-combustion sources such as paint booth ventilation streams. During SBIR Phase II work, laboratory and pilot-scale testing were conducted to obtain data necessary for process design. A pilot-scale demonstration of a microwave-based control device has proven technical and economic advantages of microwave air purification processes. During the Phase II work this microwave air purification system has also been successfully operated at McClellan Air Force Base for three months, starting 15 January 1999. This field demonstration of removal and destruction of chlorinated and non-chlorinated solvents from a soil vapor extraction site has clearly established advantages of this microwave process. The following summarizes conclusions drawn from the Phase II work and our recommendations.

- Pelletized activated carbon has the same adsorption capacity and surface area as granular activated carbon but has much higher strength. Microwaves can easily desorb solvents adsorbed on the activated carbon. Microwaves also enhance catalytic oxidation of solvents desorbed from the activated carbon. Microwave regeneration of saturated activated carbon restores the adsorption capacity and surface area.
- Microwave regeneration of spent carbon is much more effective in a moving bed than in a fixed bed. Microwaves couple to activated carbon more effectively when the carbon is moving. Because of greater penetration depth, a 915-MHz microwave generator is more suitable for a larger-scale microwave air purifier.
- For Air Force spray painting operations, a semi-continuous microwave process is more economically applicable than either a continuous or batch process. Two days of adsorption using a fixed bed of activated carbon followed by two days of microwave regeneration of spent carbon in a moving-bed are the best option. Solvent vapor can

be destroyed in a microwave catalyst bed without generating any secondary air pollutants. A 6-inch bed can effectively remove solvents in air for more than two 8-hour working days when superficial air velocity through the adsorber is ≤ 50 ft/min and solvent concentration in the vented air is approximately 200 ppm.

- The microwave air purification process has been successfully demonstrated on a pilot-scale (100 CFM airflow rate) removing solvents in air and also removing and destroying solvents from a soil vapor extraction project. This microwave air purification process is ready for commercial demonstration. Therefore, we recommend that a field demonstration of the microwave air purification process be carried out at an U.S. Air Force base to remove and destroy solvents in the air vented from aircraft spray painting operations. A 3,000-CFM microwave air purification facility will be a proper scale for this commercial demonstration.
- Microwave regeneration of used activated carbon is less expensive and more convenient than conventional steam regeneration. We recommend that used activated carbon produced at the various Air Force bases be regenerated on site using microwave devices rather than off-site regeneration. A technical and economic feasibility for microwave regeneration of wet activated carbon used in water cleanup operations should be investigated. Preliminary experimental data indicate that wet spent carbon can be regenerated without being dried using microwave energy.

ACKNOWLEDGMENTS

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