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Microwave Plasma Conversion of Volatile Organic Compounds

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ABSTRACT

A microwave-induced, steam/argon/oxygen, plasma "torch" was operated at atmospheric pressure to determine the feasibility of destroying volatile organic compounds (VOCs) of concern. The plasma process can be coupled with adsorbent technology by providing steam as the fluid carrier for desorbing the VOCs from an adsorbent. Hence, nitrogen can be excluded by using a relatively inexpensive carrier gas and thermal NOx formation is avoided in the plasma.

The objectives of the study were to evaluate the technical feasibility of destroying VOCs from gas streams by using a commercially available microwave plasma torch and to examine whether significant byproducts were produced. Trichloroethene and toluene were added as representative VOCs of interest to a flow that contained argon as a carrier gas in addition to oxygen and steam. The oxygen was necessary to ensure that undesirable byproducts were not formed in the process. Microwave power applied at 500 W to 600 W was found to be sufficient to achieve the destruction of the test compounds, down to the detection limits of the gas chromatograph that was used in the analysis. Samples of the post microwave gases were collected on sorbent tubes for the analysis of dioxins and other byproducts. No hazardous byproducts were detected when sufficient oxygen was added to the flow. The destruction efficiency at a fixed microwave power improved with the addition of steam to the flow that passed through the torch.

Keywords: microwave, plasma, regeneration, VOC, conversion, destruction, control

INTRODUCTION

As of 1997, total U. S. emissions of volatile organic compounds (VOC) had fallen to an estimated 19 x 10^6 tpy (tons per year) while total NO_x emissions continued to rise to about 24 x 10^6 tpy [1]. Among VOC categories, solvent usage increased to roughly 6.5 x 10^6 tpy [2]. Of especial concern are those compounds that are toxic air contaminants, or that may produce toxic byproducts while undergoing capture and treatment. Chlorinated solvents, chlorofluorocarbons and sulfur hexafluoride fall into such a category because of their higher activation energies and low biodegradability under oxidative conditions. In terms of millions of U.S. tons of carbon equivalents, *i.e.*, a measure of greenhouse gas potential, an increase of about 67% (16 x 10^6) tpy has occurred in the emissions of HCFCs, PFCs, and SF_6 during the decade of the 1990s [2]. Although increases in emissions have occurred, concentrations of emitted streams may actually be decreasing because of improved production methods or capture of these pollutants. Control costs generally increase as concentrations decrease, *e.g.*, the cost of catalytic incineration of VOCs increases from about \$5,000/ton to \$50,000/ton for a compound such as benzene as its concentration drops from 100 ppm_v to 10 $ppm_{v,}$ rendering even the least costly of combustion control measures impractical for low-concentration streams. Control costs for NO_x range from less than \$1000/ton for advanced burner technologies to over \$5,000/ton for exhaust gas treatments [3]. Development of efficient control strategies for low concentrations of these compounds while avoiding NO_x formation is highly desirable and is the focus of this feasibility study.

Conventional methods for removing volatile organic compounds (VOCs) from gas streams include absorption, adsorption, condensation, and incineration (including thermal and catalytic). Among these technologies, adsorption is an efficient and economical method for moderate-to-low-concentration streams. Nevertheless, adsorbates (such as VOCs) must be removed periodically and require further treatment after they saturate the adsorbents (*e.g.*, activated carbon or zeolites).

Microwave regeneration of an adsorbent utilizes "dielectric heating," which eliminates many of the above drawbacks and provides benefits unobtainable with conventional regeneration [4] In the microwave regeneration process, heat is generated internally, *i.e.*, within the adsorbent bed, either by heating the adsorbate directly and/or the adsorbent. It does not need to be conveyed through a fluid; therefore a minimal amount of purge gas is used and large concentration factors can be obtained. Our earlier experiments [4] have shown that the addition of water vapor can aid the regeneration process because water vapor can absorb microwave energy far more rapidly and effectively than molecules that have low polarizability, thereby heating those compounds indirectly. Unlike steam regeneration, however, condensation of the water vapor in the adsorbent bed is unnecessary as a means of heating the bed, thus avoiding aqueous-phase reactions and shortening heating and drying cycles. Minimal heating of ancillary mass occurs, reducing overall energy requirements and cooling time. As a result, a microwave regeneration process makes it possible to desorb VOCs from adsorbents rapidly and efficiently. In addition, the microwave system can be used for the destruction of the desorbed waste stream. Since steam can be used as the regenerating fluid carrier, nitrogen in the air can be excluded. Thus, by coupling the process with a plasma source, toxic compounds can be efficiently destroyed without production of thermal NO_x. The major objectives of this study were:

1) To study the technical feasibility of destroying VOCs from gas streams by using a commercially available microwave plasma torch
2) To examine byproducts of destruction of TCE and toluene by the microwave plasma process

Microwave technology for waste treatment

Plasma processing for environmental remediation applications is a developing technology. The primary interest in plasma processes has been in the area of combustion, due to the ability to generate extremely high temperatures, approaching 10,000 K, in the gas phase. By comparison, most chemical thermal processes, such as incineration, operate at temperatures ranging from 2000 to 2500 K. The higher temperatures attainable in a plasma process minimize the potential for the *in situ* formation of polynuclear aromatic hydrocarbons (PAH) and chlorinated dioxins and furans, which are major concerns of incineration processes. The cooling of a plasma is rapid and not conducive to molecular growth processes that yield complex molecules. However, thermal systems that are operated with air suffer from a common problem—the production of oxides of nitrogen (NO_x).

Recently there has been a growing interest in potential applications of high-frequency plasmas. For example, Wang *et al.* [5] reported the use of a RF plasma system to convert dichlorodifluoromethane into methane and acetylene in a hydrogen environment. Microwave plasmas may also be applied to the remediation of off-gas streams. Microwave plasmas possess a number of advantages over plasmas generated by other types of electrical excitation which make them attractive for this application: (1) production of high ionization levels and molecular dissociation without excess heating of the contained gas; (2) construction of reaction vessels which are simple, free from contamination and less subject to damage because of the absence of internal electrodes; (3) production of little electrical interference; (4) absence of high voltages, which can be easily contacted by operating personnel, *i.e.*, absence of shock hazards; (5) potentially lower power consumption; and (6) the ability to tolerate high concentrations of water. The latter feature is particularly important because it means that energy can be fed directly into a flow with high water concentration and a stable plasma can be established.

Although the microwave plasma process has been successfully applied in the microelectronics industries [6], the application of microwave plasma technology to hazardous waste treatment is limited. Bailin *et al.* [7] first investigated the decomposition of organic compounds by passage through a microwave-induced oxygen plasma. The basic idea in their study was to apply microwave discharge energy to break chemical bonds of organic compounds under reduced pressure conditions. Hertzler *et al.* [8] oxidized halocarbons with molecular oxygen directly in a low-pressure tubular flow microwave plasma discharge reactor. Although conversion of parent compounds exceeded 99.99%, a complete product analysis was not provided, and, therefore effluent toxicity could not be determined. Moreover, in the above studies, the organic compounds were introduced in liquid form into the plasma reactor. In other words, a microwave plasma was used to treat liquid organic wastes.

Researchers in Japan have been particularly active in promoting plasma technology for waste remediation. They have included microwave plasma systems in their studies. Suzuki *et al.* [9] investigated the use of a microwave-heated oxygen plasma for the conversion of metal chlorides into oxides. The method was shown to be successful in the conversion of a wide range of metals, from transition metals to lanthanides, with results that were consistent with an equilibrium analysis. Shimizu *et al.* [10,11] studied the decomposition of trichlorotrifluoroethane with a microwave-induced argon plasma. They used SiC fibers to assist in the generation of the plasma. The plasma was operated at one atmosphere pressure with small amounts of O_2 added to ensure complete conversion of C into CO_2. Somewhat surprisingly, they found that pulsed microwave operation achieved better destruction of the waste feed than DC operation. However, the reason for this behavior was not discovered.

More recently, McAdam [12] reported the use of a microwave plasma system for the destruction of Freon 134a and other fluorinated compounds. The concentration of Freon was about 1000 ppm in a flow of 20 Lpm. A microwave power of 800W achieved almost total removal of the Freon. Fitzsimmons *et al.* [13] explored the use of a microwave plasma to destroy dichloromethane in an atmospheric pressure flow of air.

Many hazardous waste streams include chlorine as part of the mix. Chlorine that is present in waste materials is most conveniently sequestered as hydrochloric acid (HCl). When Cl is bonded to H it is effectively removed from participation in all further reactions, except at very high temperatures. Ultimately, HCl can be handled readily with devices such as wet scrubbers where it can be neutralized. Dechlorination of wastes is an important first step in reducing the toxicity of remediation byproducts through the prevention of formation of compounds such as tetrachloro-dibenzodioxin (TCDD).

The dechlorination of waste compounds is thermodynamically favored by reaction in a reducing environment. Barat and Bozzelli [14] showed that an overall reaction of the form,

$$CCl_4 + 2H_2O \Leftrightarrow 4HCl + CO_2$$

exhibited large equilibrium constants. When oxygen is present in the system, O competes with Cl for bonding with H atoms. Hence, the ideal conditions for dechlorination are reducing, not oxidative. Barat and Bozzelli used a quartz flow reactor, driven by a microwave plasma, to examine the reaction of H_2 and H_2O with chlorocarbons. Reactions with H_2 yielded non-parent chlorocarbons, low-molecular- weight hydrocarbons, and soot. Reactions with water yielded only carbon monoxide and carbon dioxide. The production of carbon monoxide was a necessary side effect of the stoichiometry that was used; insufficient oxygen was available to ensure complete oxidation of the available carbon. Ravindran *et al.* [15] have shown from thermodynamic arguments that a similar dechlorination in hydrocarbon-rich environments is also possible. These observations suggest that dechlorination is possible under the correct conditions, conditions that may be possible in a microwave plasma.

MATERIALS AND METHODS

A continuous microwave generator (low ripple magnetron, 1.5 kW, 2450 MHz; Gerling Laboratory) and a resonant plasma tuner (ASTEX) were used to generate an argon/steam-based plasma at one atmosphere pressure. A schematic diagram illustrating the gas flow paths is shown in Figure 1. The main components of the system consisted of a plasma reactor (AX 7200), a plasma tuner, microwave generator and microwave waveguide. Continuous microwave power from the magnetron was conducted through a waveguide to the plasma torch. The forward and reflected powers from the plasma torch could be maximized and minimized by adjusting the tuning stubs on the plasma tuner. The forward power was operated at a level up to 600 W and the reflected power was maintained below about 100 W. The plasma reactor consisted of a 6 mm O.D. ceramic tube through which the mixture to be reacted was passed, and an outer quartz tube housing. It should be noted that at the input power levels applied, the mean outlet gas temperature was less than 1200 K and that the torch produces a non-equilibrium (non-thermal) plasma [16].

A plasma was generated from a flow of argon (Ar), oxygen and steam. All plasma gas flow rates were controlled by rotameters and were introduced to the plasma torch as shown in Figure 1. The total flow rate of gases was held constant at 10 slpm (standard liters per minute). The Ar was utilized as a basic carrier and reference gas; the O_2

provided stoichiometric oxidation requirements for the chlorinated hydrocarbon tested; and steam provided a reactive atmosphere containing additional hydrogen. In an H_2O/O_2 plasma process, H_2O can serve as both a source of oxygen and/or hydrogen. However, oxygen is added as dictated by stoichiometric requirements of a particular oxidation reaction. For example in the case of TCE (C_2HCl_3) one H_2O molecule and 3/2 of an O_2 molecule are required:

$$C_2HCl_3 + H_2O + 3/2O_2 \Leftrightarrow 3HCl + 2CO_2$$

Steam was generated by a coiled-tubing heater and was carried by Ar gas. The proportion of steam was established by trial-and-error to obtain an intense plasma. All the lines from the flow meter to the torch were heated to prevent steam condensation.

A common solvent, trichloroethene (TCE), was selected as one of the target compounds. Its vapor was introduced through the ceramic tube that is housed on the centerline of the plasma reactor. The other target compound was toluene (TOL). Destruction and removal efficiencies (DRE), measures of the effectiveness of the steam plasma, were calculated from concentrations of TCE and TOL measured by GC in the effluent (with and without the plasma turned on) and the known input flow rates. The effluent gas from the plasma reactor was passed through two traps. The first trap consisted of a coiled water concentrator and an Erlenmeyer flask, in which most of the steam was condensed. A second back-up trap was used to condense the remaining water vapor. Gas samples were collected with high efficiency on an adsorbent bed of Carbotrap C. A gas-sampling loop and switching valve were used to inject gas samples into a Varian 6000 GC from a by-pass line exiting the second trap. Liquid samples were also collected from the first and the second traps.

The reactor effluents were analyzed with an on-line Varian 6000 GC equipped with a TCD detector for TCE and TOL, and by GC/MS analysis of adsorbent tube (Carbotrap) extracts for TCE and other byproducts. A specific ion meter was used to measure Cl^- concentrations in the condensate. The Carbotrap samples were Soxhlet extracted for 20 hours with dichloromethane. The samples were evaporated down to 0.5 mL prior to injection into a Varian 3800 GC/MS, using a DB-5MS, 30m x 0.32 μm column. Dueterated phenanthrene and terphenyl were used as internal standards at a concentration of 100 μg/mL of MeCl.

RESULTS

The DRE for TCE was evaluated in the microwave system for a series of microwave powers at a condition of 30% steam with an O_2 flow rate of 0.5 slpm and an argon flow rate of 6.0 slpm. The concentration of TCE in the input flow to the plasma was 1700 ppm. The small amount of O_2 was added to ensure that sufficient O_2 was available in the system to complete the oxidation of TCE. The post-plasma gases were analyzed on a GC. The results are shown in Table 1. Experiments were also conducted with a power of 600 W and a TCE input concentration of 1700 ppm, but with varying concentrations of steam in the plasma. The results of those tests are shown in Table 2.

Finally, the Carbotrap samples were analyzed on the GC/MS system for products of incomplete combustion at conditions of microwave power of 600 W with 30% steam and 1700 ppm of TCE. This analysis did not find any evidence, down to the detectable limit of dioxins or furans (LOD approximately 0.2 ng/m^3), demonstrating that a high DRE can be achieved without the production of other products of incomplete reaction.

The effectiveness of the microwave system was also investigated for toluene. Two concentrations of toluene were added to the input flow. The destruction efficiency is shown in Table 3. Based upon the ability of the GC software to discriminate peak area, the detection limit for toluene was approximately 50 ppm and for trichloroethene, the detection limit was 300 ppm. With an estimated uncertainty in the measured concentrations of 2%, an uncertainty analysis showed that the estimated uncertainty in the reported destruction and removal efficiencies (DRE) was about 0.05%. The maximum DRE's are reported as greater than 99.95%.

DISCUSSION

It is apparent that the plasma system is capable of achieving significant destruction at operating powers as low as 400 W. The chloride ion concentration is a measure of the HCl that is formed, and is a second measure of the accuracy of the sampling system and analysis. The observation of a constant Cl^- concentration at powers above 400 W supports the conclusion that the majority of the input chlorine was sequestered as HCl under these conditions.

The presence of steam is seen to have an important impact on the effectiveness of the microwave destruction process. The effect of steam that is shown in Table 2 is consistent with the observation of Barat and Bozzelli that at the temperatures of the microwave plasma, thermodynamics favors the dechlorination of chlorinated hydrocarbons in the temperature window in which the plasma is operated. Sequestration of the Cl in the system as HCl prevents its further incorporation into hazardous byproducts such as TCDD. The removal of the HCl could be readily achieved in a practical system through a condensing wet scrubber and subsequent neutralization. It should be noted that the volume of gas that would require treatment is quite small since it consists only of the amount of argon used to blend with the steam, and the carbon dioxide from oxidation of the carbon atoms in the VOC. The hydrogen chloride and additional water are condensed in the scrubber. If there is sulfur in the waste stream, one would presumably remove sulfur dioxide and sulfuric acid with an alkaline scrubbing solution.

The microwave system was equally as effective with a non-chlorinated aromatic hydrocarbon (toluene) as it was with the chlorinated aliphatic compound. Once again, input power levels greater than 400 to 500 W were necessary to ensure complete destruction. An analysis of heavier polynuclear aromatic hydrocarbons (PAH) in the post-plasma effluent stream was not undertaken in this case due to limited personnel resources for the project. Two tests to screen for the presence of chlorinated dioxins as byproducts of TCE destruction were performed by GC/MS on a Carbopak C sorbent trap. Results from those analyses indicate that in the absence of stoichiometric oxygen addition, a trace amount of dioxin was detected. In the case when stoichiometric oxygen was added, no detectable chlorinated dioxins were observed (level of detection approximately 0.2 ng/m^3).

The energy requirement to destroy TCE completely, per unit volume of treated gas, was about 1000 W-hr m^{-3} or 3600 J L^{-1}. This figure is significantly higher than the energy cost for a pulsed corona plasma [12] that was used to process toluene. A high level of destruction was achieved with an energy input of about 30 W-hr m^{-3}. Pulsed corona discharges are typically useful for treating large flows, as a result of their open structure. Smaller flows are more conveniently treated by other non-thermal plasmas such as the microwave plasma. Fitzsimmons et al. [13] reported a 20% reduction in the concentration of dichloromethane in a packed dielectric bed plasma reactor with an energy input of about 66 J L^{-1}; some N_2O and NO_2 formed as a result of the large energy density and heating of the flow. The formation of NO_x is not an issue for the current system as a result of the exclusion of N from the system. Nonetheless, the microwave plasma is expensive to operate in comparison with other plasma technologies.

From the power consumption and the TCE feed rate into the plasma torch, an estimate of the energy used to destroy a gram of TCE was computed to be about 300 kcal per gram of TCE destroyed. Assuming a power cost for electricity of $0.10/kW-hr, then the power cost alone in the plasma torch system was about $35/kg-TCE or $3500/metric ton of TCE. The figure does not take into account the system capital costs which would need to include an adsorbent bed system and microwave generators, nor the pressure losses through the system. A recent report by Agnihotri *et al.* [17] on the destruction of trichloroethane (TCA) estimates that the comparable cost for a non-thermal dielectric-barrier discharge plasma to be about $20/kg-TCA for a humid airstream (88% relative humidity) . Thus, while the microwave plasma costs do not appear prohibitive, they are substantial. However, the size of the unit, and its high destruction efficiency, may make it very competitive for applications to small, compact sources such as dry cleaners and small scale paint shops, especially in light of the report that low-cost half-wave rectified microwave generators have been used successfully to produce plasmas [16].

SUMMARY AND CONCLUSIONS

Preliminary tests of a microwave plasma system have shown that this is a feasible technology for the treatment of waste streams that include chlorinated hydrocarbons and non-chlorinated hydrocarbons. The prototype system was shown to be very successful in destroying trichloroethene and toluene. Destruction was achieved with acceptably moderate power levels supplied to the microwave torch. It was found to be important to maintain the correct stoichiometry in the mixture. To this end, oxygen needed to be added to the flow to ensure that toxic byproducts were not formed. Sampling onto a sorbent tube, followed by subsequent solvent extraction and analysis on a mass spectrometer, revealed that with sufficient oxygen no chlorinated dioxins or furans, or other hazardous polynuclear aromatic hydrocarbons, were formed. Steam was also shown to be an important factor in the destruction process. An increasing fraction of steam in the flow led to improved destruction efficiencies. This is consistent with a thermodynamic analysis that showed that the dechlorination of chlorinated hydrocarbons is quite efficient in the temperature window from about 800 K to 1100 K in which the plasma torch operates. The successful demonstration of microwave destruction suggests that a combined system, with a sorbent bed followed by steam desorption and microwave destruction, could be put into practice for real-world systems designed to control dilute streams of volatile organic compounds. The economics of the system have not been fully analyzed, but the moderate power levels that are required suggest that the economics may very well be favorable.

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Table 1. DRE and Cl ion concentration in post-plasma gases at different operating powers with an input of 1700 ppm of TCE, 30% steam and flows of 6.0 Lpm of argon and 0.5 Lpm of O_2

Power (W)	DRE (%)	Cl^- (ppm)
200	58	10800
300	99.8	38700
400	>99.95	52000
500	>99.95	56200
600	>99.95	56900

Table 2. DRE at 600 W of input microwave power with varying steam concentrations and 1700 ppm of TCE

Steam fraction of flow (%)	DRE(%)
0	99.75
5	99.9
10	>99.95
20	>99.95
30	>99.95

Table 3a. DRE of toluene at input concentrations of 500 ppm

Power(W)	DRE(%)
200	90.5
300	99.5
400	>99.95
500	>99.95
600	>99.95

Table 3b. DRE of toluene at input concentrations of 800 ppm

Power(W)	DRE(%)
200	72
300	98
400	99.95
500	>99.95
600	>99.95

FIGURE CAPTIONS

Figure 1. Microwave apparatus illustrating components: gas supplies, microwave source, resonant tuning stubs, plasma torch, steam generation and condenser system.

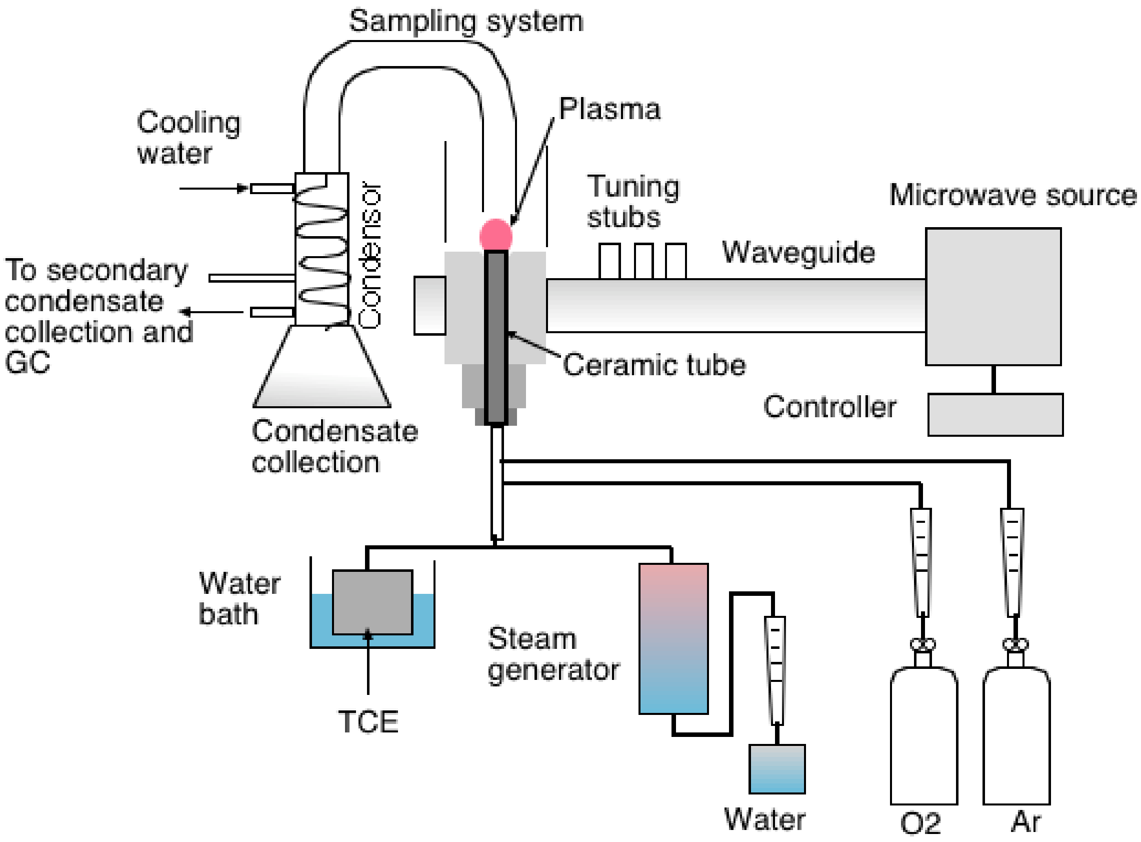

Figure 1