

## Regeneration of granular activated carbon saturated with gaseous toluene by microwave irradiation

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### Abstract

This paper describes laboratory-scale experiments examining the impact of microwave irradiation on the regeneration of granular activated carbon (GAC) exposed to toluene vapor. A stream containing 300 ppm of toluene was passed through the GAC. The saturated adsorbent was treated by microwave irradiation at a heating frequency of 2450 MHz at various power levels to evaporate the adsorbate. The adsorption capacity was evaluated through breakthrough curves and the residue was analyzed by gas chromatography. We found that exposing GAC saturated with toluene under microwave irradiation at 900 W for 15 min could remove the pollutant from the adsorbent effectively. The regeneration efficiency was more than 95% after 5 cycles at higher power levels. The GAC surface area was increased from 1150 m<sup>2</sup>/g to 1185 m<sup>2</sup>/g over regeneration cycles. We concluded that GAC could be reused after repeated regeneration cycles under microwave irradiation while maintaining its original adsorption capacity and physical properties.

**Key Words:** Toluene vapor, granular activated carbon, adsorption, microwave irradiation, regeneration efficiency

### 1. Introduction

Organic solvents are volatile organic compounds (VOCs) with varying chemical compositions. The EPA has defined VOCs as compounds with a room temperature vapor pressure, greater than 70 Pa (USEPA, 2005). Industrial use of solvents results in emission of VOCs in occupational settings as well as nonindustrial indoor environments. Industrial emissions account for 50% of VOCs in the environment (Pohanish, 2002; Jin et al., 2004). Occupational exposure to VOCs is harmful and toxic to workers. They are considered as carcinogenic and mutagenic agents (Rutnick et al., 2004; Semple et al., 2004; OSHA, 2005). VOCs also have negative impacts on the environment. Outdoor emissions are responsible for destroying the stratospheric ozone layer,

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causing acid rains, producing ground level ozone pollution due to photochemical reactions, and changing the global climate. Solutions for controlling volatile organic compounds are described in the literature (Khan et al., 2000; Moretti, 2001; DiNardi, 2003). Various control methods involve thermal incineration (Dranca et al., 2001), catalytic incineration (Sinquin et al., 2000; Everaert et al., 2004; Wang et al., 2007), and photocatalytic oxidation (Hitchman et al., 2002; Wang et al., 2003; Zou et al., 2006) to control and treat VOCs. Thermal combustion uses high temperatures (700-1000 °C) and is thus costly and suitable for large volumes of gas emissions with high concentrations. Adsorption by activated carbon is an effective method to remove low VOC concentrations from gas streams in indoor ventilation systems (Benkhedda et al., 2000; Tiemey et al., 2006; Villacanas et al., 2006), but the adsorbent becomes saturated after constant use and requires further treatment. The adsorption performance of activated carbon mostly depends on the porous structure of the activated carbon (Abril et al., 2009). Activated carbon regeneration by hot steam decreases the adsorbent capacity and damages the surface structure (Ania et al., 2005; Chen et al., 2005), resulting in short-term use and disposal. Therefore, efficient regeneration of activated carbon is required to ensure its long-term reuse and simultaneously maintain its adsorption capacity at an acceptable level. Jones et al. (2002) discussed microwave heating applications in environmental engineering in detail. Compared with conventional heating, microwave (MW) heating has many advantages: 1) it heats the material from the inside out, 2) selective heating depends on the nature of the object, 3) there is no need for heat convection through a fluid, 4) it provides rapid heating with no direct contact between the MW heating source and the heated material, 5) there is ease of heating process control, 6) it has high temperature capabilities, 7) it saves time and energy, and 8) it increases chemical reactivity. There is a growing body of research on MW applications in industrial and environmental engineering (Bathen, 2003). Studies have shown MW energy to be a possible method in waste treatment for extracting contaminants and organic solvents from soil (Liu and Yu, 2006) and metals from sludge (Menendez et al., 2002), and for processing minerals (Jones et al., 2002) and water purification (Bandosz, 2006). MW radiation has also been used for the production of activated carbon (Nabais et al., 2004). Studies have shown that granular activated carbon (GAC) can absorb MW radiation, leading to rapid heating (Guo et al., 2000). MW energy was applied to regenerate GAC-adsorbed pentachlorophenol in water and powdered activated carbon spent with salicylic acid in wastewater (Liu et al., 2004; Ania et al., 2007) where adsorption and regeneration reactors were separate and thus required movement of the adsorbent into the MW chamber, resulting in attrition.

The purpose of the present study was to examine the regeneration of GAC exposed to toluene vapor by applying MW radiation. The proposed laboratory-scale system was designed with a single adsorption and regeneration reactor. Adsorption capacity and the efficiency of adsorbent regeneration at various cycles and changes in the adsorbent texture were investigated.

## 2. Experimental materials and methods

### 2.1. Materials

Activated carbon, in the form of pellets manufactured by Merck, was used in this study. The size of the pellets was around 2.5 mm with a molecular weight of 12.01 g/mol. The pellets were crushed into GAC. The characteristics of the adsorbent used in the study are summarized in Table 1. Before using the adsorbent in the experiment, the adsorbent was treated with 10% hydrochloric acid and immersed in deionized water to remove impurities and fine particles, then dried in an infrared heating oven at 120 °C for 1 h and stored in a desiccator

for 48 h before use in the relevant experiments. The moisture content of GAC was measured with a Sartorius moisture analyzer. Toluene with a purity of 99.97% was introduced into the gas stream to pass through and saturate the GAC, which was then treated with MW irradiation.

## 2.2. Experimental methods

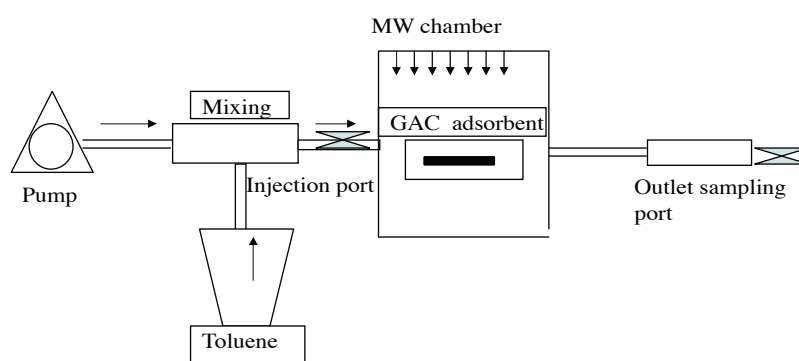
In this study, the integrated system was composed of a vapor generator, a column containing a GAC adsorbent bed, a MW chamber, and analytical measuring instruments.

### 2.2.1. Vapor generator

The laboratory-scale reactor system consisted of a dynamic vapor generation system. An ejector was used to supply a given vapor stream. Purified air was supplied by a pump at a controlled flow rate, and saturated toluene vapor was introduced by suction into the ejector at desired room conditions. Inside the ejector, the toluene vapor merged with the air stream and passed through a venturi tube, and was then channeled into a chamber for complete mixing, resulting in a desired concentration and controlled flow of vapor. Before entering the adsorbent, the gas stream was metered and set by a mass flow meter to supply a desired and constant flow rate (Figure 1). A gas analyzer (PhoCheck 5000EX) was used to determine the vapor concentration flowing into the reactor.

**Table 1.** Characteristics of fresh GAC used in the study.

Mesh size	BET surface area (m <sup>2</sup> /g)	Mesopore volume (cm <sup>3</sup> /g)	Micropore volume (cm <sup>3</sup> /g)	Moisture content (%)
20/40	1150	0.412	0.2712	< 1.20



**Figure 1.** Laboratory-scale experimental set-up for toluene removal and GAC recovery by MW irradiation.

### 2.2.2. GAC adsorption and regeneration experiments

The adsorption capacity of GAC was examined and linked with the regeneration efficiency of GAC under MW irradiation in successive adsorption-regeneration cycles. Adsorption laboratory tests of toluene vapor were carried out in a quartz glass column with a 2-cm inner diameter and a length of 30 cm. The column was loaded with a fixed adsorbent bed containing 10 g of GAC. The adsorption experiments were performed at the desired temperature of  $25 \pm 4$  °C. Previous studies found that the presence of water vapor along with volatile organic compounds had no significant impact on the adsorption capacity of the activated carbon until

the relative humidity exceeded 75% (Tiemey et al., 2006). Thus, the relative humidity of the gas stream was controlled at  $55 \pm 4\%$ . A hygrometer (TH2-SIBATA) was used to monitor the relative humidity of the stream. The inlet concentration of the toluene vapor stream was 300 ppm at a volumetric flow rate of 50 mL/min, passed through the GAC bed. The concentration of the outlet stream was monitored continuously to obtain breakthrough curves. The adsorption breakthrough profile was measured by monitoring the outlet concentration as a function of time. When the adsorbent bed reached the saturation point, the adsorbate was extracted and the adsorption capacity was measured in grams of toluene loaded per gram of GAC. A microwave oven with power levels of 175-900 W provided MW irradiation at a heating frequency of 2450 MHz. A quartz glass column reactor was installed into the MW cavity. A stream containing toluene vapor flowed from the bottom of the column and passed through the GAC adsorbent bed and the outlet at the top of the column, connected to a Tedlar air sampling bag. After the passing of the gas stream contaminated with toluene vapor and the saturating of the GAC bed, the adsorbent bed was exposed to MW heating irradiation under various power levels and time durations. The regenerated GAC samples were used for the adsorption of toluene vapor at the same previous laboratory and ambient conditions through a series of successive tests conducted to determine the adsorption capacities' and recovery cycles' efficiency as a function of MW energy in view of modification of the GAC properties. Regeneration efficiency was defined as equal to: (adsorptive capacity of the regenerated GAC/adsorptive capacity of the fresh GAC)  $\times$  100.

### 2.2.3. Analytical measurement

The samples adsorbed on the GAC bed were desorbed by carbon disulfide, and gas collected in the bag was analyzed with a gas chromatograph (GC-Philips PU4410) using a flame ionization detector, as proposed by OSHA Method 111 (OSHA, 2002). A 30-m capillary column with an inner diameter of 0.32 mm was used for chromatographic separation. The analysis was performed with nitrogen as a carrier gas at a flow rate of 30 mL/min; the zone temperatures were set as 60 °C for the column, 250 °C for the injector, and 275 °C for the detector. The injection volume was 1  $\mu$ L and the external standards were prepared to obtain a calibration curve to bracket the sample concentrations.

The surface area of the GAC was determined by the Brunauer-Emmett-Teller (BET) analytical method. Nitrogen adsorption isotherms were used to determine the micropore and mesopore volumes by the Dubinin equation (Banosz et al., 2003). The surface temperature of the GAC after exposure to MW heating irradiation was measured with an infrared pyrometer.

## 3. Results and discussion

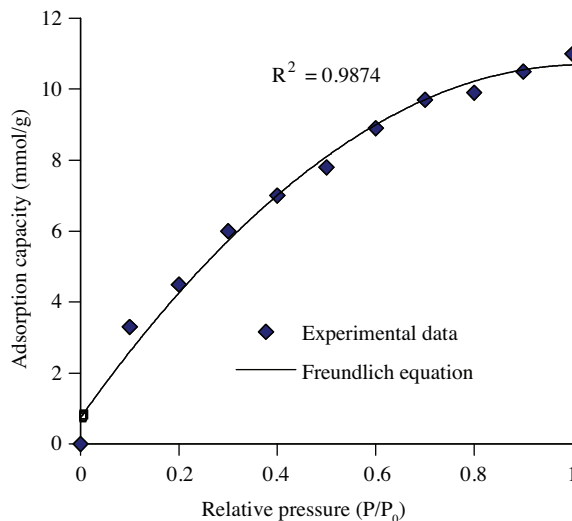
### 3.1. Equilibrium adsorption capacity

The adsorption experiments were conducted by passing a vapor stream carrying toluene, at an inlet concentration of 300 ppm and a flow rate of 10 mL/min at 25 °C, through the adsorbent bed of 10 g of GAC. The toluene vapor reached its equilibrium at the adsorption period of around 9 h. The maximum adsorption capacity was determined to be 0.35 grams of toluene per gram of GAC. The adsorption values achieved for toluene at 300 ppm in this study were slightly better than those previously reported (Benkhedda et al., 2000). This may be explained by the origin, preparation method, and mesh size of the GAC, as well as the concentrations and flow rates used in the experiments. Figure 2 shows the adsorption isotherm data, plotting the vapor relative pressure of toluene versus the toluene adsorbed on the GAC adsorbent at 25 °C. We found that the adsorption isotherm

data could be explained and fitted by the Freundlich isotherm equation model (Villacanas et al., 2006), which is given by:

$$V = K.(P/P^\circ)^{1/n},$$

where  $V$  is the adsorbate concentration adsorbed on the adsorbent,  $K$  and  $n$  are regression parameters, and  $P/P^\circ$  is the ratio of adsorbate vapor pressure to saturated vapor pressure (relative pressure). The regression of the isotherm experimental data was performed in a log scale (the plot of  $\log V$  versus  $\log P/P^\circ$ ), yielding  $r^2 = 0.98$ ,  $1/n = 0.410$ , and  $K = 1.60$  mmol/g.



**Figure 2.** Toluene adsorption isotherm at 25 °C.

### 3.2. Toluene desorption under microwave irradiation

Microwave power level is considered a crucial variable in examining the impact of irradiation on GAC and toluene. To understand this, a series of 10 g of GAC saturated with toluene was exposed to various MW power level settings at 180, 360, 540, 720, and 900 W for different microwave irradiation times of 5, 10, 15, 20, and 25 min. The impact of MW power levels and irradiation periods on the residual amount of toluene in GAC is presented in Table 2. We found that at high power levels of 540, 720, and 900 W, the amounts of residue in the GAC were not detectable after 25, 20, and 15 min, while at low power levels of 180 and 360 W, irradiation was not adequate to remove the contaminant.

Previous studies have indicated that MW energy could be used for carbon materials; basically, the GAC absorbs MW heating energy quickly (Jones et al., 2002). We observed sudden and intense bright sparks at high powers, resulting in a hot and red carbon. The whole GAC turned red within a few minutes and remained in that condition for the entire treatment process under MW irradiation. These results are in agreement with studies previously reported, that the temperature increase in GAC exposed to MW radiation depends on microwave power levels (Bathen, 2003; Liu et al., 2004). The surface temperature measurements were around 730 °C and 800 °C, respectively, at output power levels of 720 W after 20 min and 900 W after 25 min of MW irradiation. It is evident that increased power levels enhanced the removal rate of adsorbed toluene due to the rapid increase in adsorbent bed temperature. Theoretically, MW irradiation energy is directly supplied to the GAC bed and the adsorbate. Energy transfer is not by conduction or convection, but is readily transformed into heat inside

the particles by dipole rotation and ionic conduction. Meanwhile, MW heating supplies energy independent of the mass of gas passed into the bed (Nabais et al., 2004). More than 90% of the toluene loaded on the GAC was removed over periods of 15 min of irradiation at 720 W and 10 min of irradiation at 900 W. We were not able to obtain detectable residue in the GAC exposed to irradiation at higher power levels, and as the microwave power increased, less time was required to remove the contaminant. Therefore, the treatment of pollutants by heating is more rapid and less time-consuming in a MW field than an electric furnace.

**Table 2.** Amount of toluene remaining on GAC adsorbent following treatment with various MW powers and irradiation periods.

MW power levels (W)	MW irradiation duration (min)					
	0	5	10	15	20	25
	Toluene residue on GAC adsorbent (mg toluene/g GAC)					
180	330	275	272	268	260	258
360	330	270	255	70	43	25
540	330	220	87	32	5	...
720	330	195	65	4	...	...
900	330	76	4	...	...	...

...: not detectable

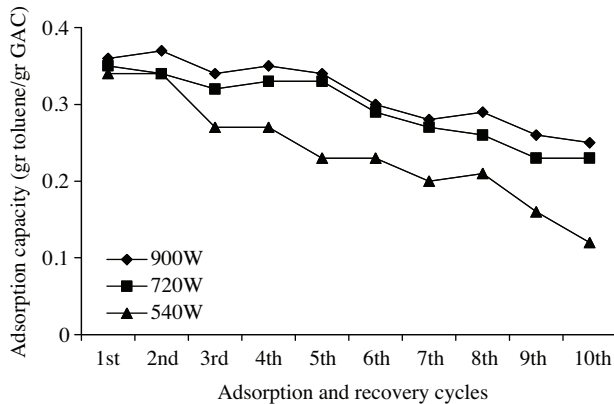
### 3.3. Multiple GAC regeneration in a microwave field

After saturated GAC was exposed to MW irradiation, we measured the effectiveness of the radiation on the GAC's adsorption capacity. It was crucial to examine the impact of various cycles of adsorption and recovery processes on the GAC adsorption capacity. Therefore, the recovered GAC was reused to adsorb toluene vapors, and the regeneration and adsorption cycles were repeated 10 times. The GAC adsorption capacity when exposed to toluene is shown in Figure 3 as a function of multiple regeneration cycles. We obtained interesting results by comparing adsorptive capacities of fresh GAC and samples treated and recovered by MW irradiation. We found that after 5 repetitive treatment cycles by MW energy, the adsorption capacity remained approximately constant at higher power levels. Further treatment and recovery cycles resulted in slight decreases of GAC adsorption capacity. The results imply that repeated MW irradiation at power levels higher than 720 W for adsorbent recovery does not have a remarkable effect on the GAC adsorption capacity compared to its fresh value. However, the trend of adsorption capacity showed more reductions at the lower MW power of 540 W. Figure 3 shows the trends obtained in recovery efficiency values during several GAC treatments under MW irradiation. Data provided in Figure 4 indicate that after 10 cycles of GAC recovery, the decrease in adsorption capacity compared to its untreated initial sample was about 30% at 720 and 900 W, while it was about 64% at 540 W. Furthermore, at higher output powers, the adsorption capacity was preserved and remained almost constant after 5 regeneration cycles; subsequent cycles resulted in slight reductions in adsorption capacity. Figure 5 shows the breakthrough adsorption curves for the fresh and multiple-treated GAC at 900 W. This suggests that, contrary to activated carbon heating in an electric furnace that will damage its adsorptive feature, the activated carbon's adsorptive properties do not change adversely under MW irradiation. Thus, it is important to note that, as observed from data points in Figure 5, the shapes of breakthrough adsorption curves for GAC did not change over repeated MW treatment cycles when compared with the curve of fresh GAC. It can be

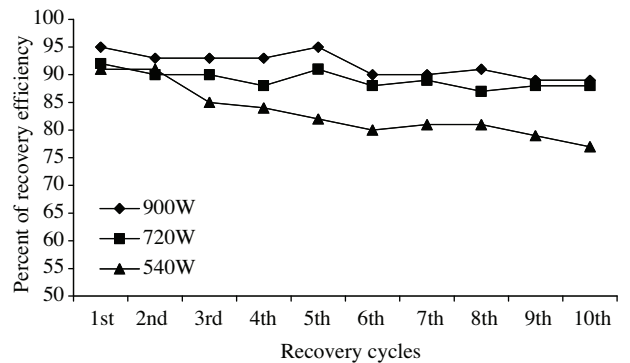
inferred from the experiments that MW energy can maintain the adsorption capacity at an acceptable level compared to fresh values and increase the life of GAC for VOC treatment. The experiments showed changes in GAC porosity results in the weight loss of the GAC. After 10 successive adsorption and regeneration cycles under MW irradiation at 900 W for periods of 10 min, the GAC weight loss was 4.2%.

### 3.4. GAC physical characteristics under microwave irradiation

Conventional thermal heating of activated carbon after successive treatment and recovery cycles in an electrical furnace decreases the adsorption capacity significantly, which is attributed to the adverse changes in the adsorbent physical structure (Bathen, 2003). Table 3 illustrates details concerning the surface and porous structure of GAC saturated with toluene after exposing to MW radiation at 900 W for 25 min. The BET, mesopore, and micropore volumes of MW-treated GAC loaded with toluene slightly changed over successive MW recovery and treatment cycles. Meanwhile, the relevant features increased compared with the fresh untreated GAC, as shown in Table 1. This implies that improvement in surface and porosity characteristics may probably be connected to the nearly constant and undeteriorated adsorption capacity of the GAC in successive cycles. The findings confirm previous literature on the effect of MW irradiation on the textural properties of carbon (Ania et al., 2005). According to Abril et al. (2009), the performance of activated carbon depends on its micropore volume, as a smaller micropore volume influences the breakthrough and saturation times. The heating of the adsorbate within the pores generates an internal pressure as the vapor is vaporized at its interior and gas expansion occurs; thus, the diffusion of the vapor out of the adsorbent may be dominated by a pressure-



**Figure 3.** Adsorption capacity in terms of MW treatment cycles at various power levels for 25 min of MW irradiation.



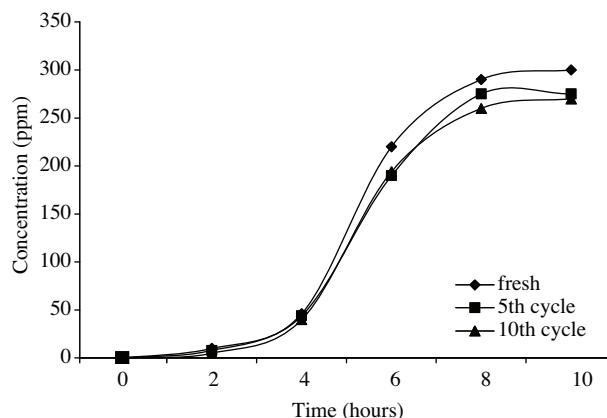
**Figure 4.** Recovery efficiency obtained under MW radiation at various levels.

**Table 3.** Physical characteristics of GAC exposed to MW irradiation.

GAC characteristics			
	Surface area BET (m <sup>2</sup> /g)	Mesopore volume (cm <sup>3</sup> /g)	Micropore volume (cm <sup>3</sup> /g)
MW treatment, 900 W*			
1st	1176	0.510	0.326
5th	1180	0.620	0.379
10th	1185	0.593	0.435

\*Results for 15 min of irradiation.

driven flow. The increase in mesopore and micropore volumes that enhance the adsorbate transportation and adsorption abilities of the GAC respectively suggests that longer application of the GAC can be obtained under MW irradiation treatment.



**Figure 5.** Breakthrough adsorption curves for toluene at 300 ppm over 10 gr of GAC in the fresh and MW recovered cycles.

#### 4. Conclusion

The results of our laboratory-scale tests demonstrated that MW heating energy may be used effectively for the treatment of VOCs adsorbed onto GAC. Microwave heating delivers energy throughout the volume of the adsorbent where the MW-absorbing adsorbate is located. Toluene removal increased steeply within a few minutes and reached over 90% under MW radiation at higher power levels. High recovery efficiencies were obtained under MW treatment. However, we noticed GAC weight loss after 10 successive regeneration cycles. We concluded that microwave recovery of saturated activated carbon loaded with toluene maintained the adsorption capacity, surface area, and porosity structure of the GAC in favorable circumstances. This may allow the reuse of GAC for longer periods. Conventional heating technologies, including incineration for treating VOCs, are suitable and economically effective when large amounts of these compounds can be provided, while MW treatment and recovery could be used for low concentration emission streams as encountered in industrial buildings and ventilation systems. Briefly, the system is interesting for its simplicity, effectiveness, speed, and economic utility for treating and recovering volatile vapor pollutants. We expect that this approach could be used in ventilation systems for the treatment of VOCs in work environments. However, as the system reaches high temperatures rapidly due to the generation of arcs in the GAC bed, further studies are required to examine the decomposition of the volatile vapors collected on the adsorbent.

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**Nomenclature**

ppm	parts per million	cm	centimeter
min	minute	mm	millimeter
Pa	pascal	m <sup>2</sup> /g	squared meters/gram
W	watt	cm <sup>3</sup> /g	cubic centimeters/gram
g	gram	°C	degrees centigrade
g/mol	grams/mole	mL	milliliter
		μL	microliter

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