

APPLICATION OF MICROWAVE IRRADIATION FOR THE TREATMENT OF ADSORBED VOLATILE ORGANIC COMPOUNDS ON GRANULAR ACTIVATED CARBON

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ABSTRACT

The purpose of this laboratory scale experimental research was to investigate the application of integrated microwave irradiation and granular activated carbon adsorption for removing volatile organic compounds (VOCs) from emissions released from industrial processes and considered major pollutants of the environment. A stream containing 300 ppm toluene was supplied and passed through the granular activated carbon (GAC). The saturated adsorbent was placed in a quartz glass reactor and treated by microwave (MW) irradiation at heating frequency of 2450 MHz at different power levels. Adsorption capacity was evaluated through breakthrough curves and the residue analyzed by gas chromatography. Breakthrough results showed that the high relative humidity of the inlet gaseous stream could lead to decreased GAC adsorption capacity. We found that GAC could absorb microwave irradiation and dissipate heating energy increasing its temperature up to 600°C in a short time mainly depending on the microwave power level. Our experimental results indicated that most of the toluene vapor passed over GAC was decomposed under 900 watt microwave irradiation after around 10 minutes. The addition of water vapor in the inlet stream slightly decreased pollutant removal rate over the GAC while the overall oxidation removal remain the same compared to stream with low content of water vapor. Adsorption isotherms showed that GAC surface area and porosity values increased slightly under microwave irradiation. We concluded that simultaneous application of GAC and microwave irradiation may be an effective approach for removing VOCs from gaseous stream.

Key words: Microwave irradiation; Granular activated carbon; Adsorption; Volatile organic compounds

INTRODUCTION

Reduction in volatile organic compounds (VOCs) emitted into the atmosphere has become a concern because of the emissions and their potential health effects to humans and deleterious effects to the environment (USEPA, 2005). New control technologies are being developed to separate and remove toxic compounds from gas streams. The various VOC control methods can be classified under three general categories. The first category involves the recovery of VOCs,

and specific examples include condensation, adsorption, and absorption. The second category is made up of methods that utilize the conversion of organic vapor pollutants into less harmful products, and examples include biological and ultraviolet treatment. The third category involves combustion methods, and examples are thermal incineration, flaring, and catalytic incineration. Details of each of these methods can be found in several books that address the general subject of VOCs (Hunter and Oyama, 2000; DiNardi, 2003; Moretti, 2001; Khan and Ghoshal, 2000).

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Thermal combustion uses high temperature (700-1000 °C) and is costly and suitable only for large volume of gas emissions with high concentrations (Sinquin *et al.*, 2000). This method also requires large space for equipment. Catalytic and photocatalytic techniques are kinetically slow processes. Adsorption by activated carbon is an effective method to remove low VOCs concentrations from gas stream in indoor ventilation systems (Villacanas *et al.*, 2006; Abril *et al.*, 2009; Mohan *et al.*, 2009) but the adsorbent becomes saturated after constant use and requires further treatment.

In recent years microwave (MW) heating energy has attracted attention as a tool for thermal process due to its capacity in heating at molecular level leading to homogenous and quick thermal reactions (Ania *et al.*, 2005). Microwaves are electromagnetic waves related to wavelengths of 1mm to 1m. The heating of materials in a microwave field occurs when there are free ions or dipolar molecule rotation. Jones *et al.*, (2002) discussed microwave heating applications in environmental engineering in details. Compared with conventional heating, microwave heating has many advantages: 1-microwave energy heats the material from inside out; 2-selective heating depends on the nature of the object; 3-there is no need for heat convection through a fluid; 4-microwave energy provides rapid heating; no direct contact between the microwave heating source and the heated material; 5-ease of heating process control i.e. as soon as the microwave radiation is switched off the source of the heat is quickly removed from the adsorbate and adsorbent; 6- high temperature capabilities; 7- time and energy savings; and 8- increase of chemical reactivity. Moreover, microwave processing systems are also relatively compact, portable, maintainable and cost effective.

There are growing researches of microwave processing applications in industrial and environmental engineering due to its advantages in the area of combustion (Bathen, 2003). Studies showed microwave energy as a possible method in waste treatment to extract contaminants and organic solvents from solution (Movahedyan *et al.*, 2009), soil (Liu *et al.*, 2006), metals from sludge

(Menendez *et al.*, 2002), and processing minerals (Jones *et al.*, 2002) as well as water purification (Bandosz *et al.*, 2006). It has also been suggested that microwave treatment is less time consuming. Microwave radiation has also been used for the production of activated carbon (Nabais *et al.*, 2004). Studies showed that Granular activated carbon (GAC) can absorb microwave radiation leading to rapid heating (Guo and Lua, 2000; Liu *et al.*, 2004). Therefore using microwave radiation may be a promising alternative in treating volatile organic compounds.

The principle objective of the present study is to integrate microwave radiation energy of an electric furnace with granular activated carbon adsorption technique to treat volatile organic vapors. Toluene was used as the organic vapor pollutant to conduct relevant experiments for the reason that it is one of the selected primary work environment air pollutants due to its high occurrence and also its suitability for the laboratory work.

MATERIALS AND METHODS

In this study the integrated system composed of vapor generator, column containing GAC adsorbent bed, microwave chamber and analytical measuring instruments (Fig. 1).

Materials

Activated carbon in the form of pellet manufactured by Merck Company was used. The characteristics of the adsorbent are summarized in Table 1. Toluene with a purity of %99.97 was introduced into the gas stream to pass through and saturate GAC and treated with microwave irradiation. The main chemical and physical properties of toluene are presented in Table 2.

Experimental setup and methods

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, toluene vapor merged with the air stream and passed through a Venturi tube

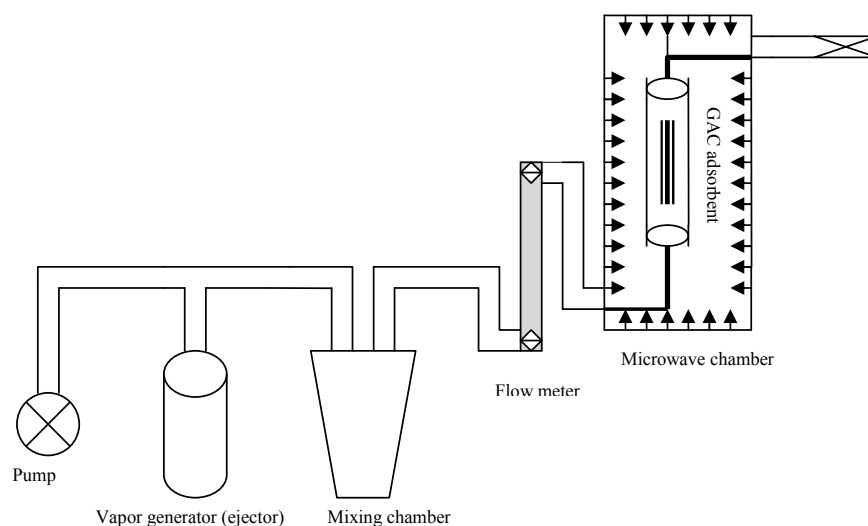


Fig. 1: Schematic illustration of experimental setup

which 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.

Toluene breakthrough experiments

Adsorption laboratory tests of toluene vapor were carried out in a quartz glass column with 2-cm inner diameter and the length of 30 cm. The column was loaded with a fixed adsorbent bed containing 10g of granular activated carbon with a bed height of 36mm. The inlet concentration of toluene vapor stream was 300 ± 25 ppm at a volumetric flow rate of 50 mL per minute passed through the GAC bed.

The adsorption experiments were performed at desired temperature of 25 ± 2 °C and pressure of 102.23 kpa. Humidity was added to the air stream in the system by bubbling air through water in a fritted bottle. Then the humid air merged with the main stream to obtain the desired humidity. A

Hygrometer (TH2-SIBATA) was used to monitor the relative humidity of the stream.

The concentration of outlet stream was monitored continuously to obtain breakthrough curves. The adsorption breakthrough profile was measured by monitoring outlet concentration as a function of time. Breakthrough time and saturation adsorption time were calculated from adsorption curves. When the outlet toluene concentration reached 5% of the initial concentration in the inlet, breakthrough time was determined. Saturation adsorption time was measured once the outlet toluene concentration increased to 95% of the inlet concentration (Mohan *et al.*, 2009).

Table 2: Toluene chemical and physical properties (Poling *et al.*, 2001)

Molecular formula	C ₇ H ₈
Molecular weight(g/mol)	92.14
Boiling point (°C)	110.6
Auto ignition temperature (°C)	480
Vapor pressure at 25°C (KPa)	3.79
Saturated vapor pressure(mm Hg)	28.3
Vapor molar volume (cm ³ /mol)	316
Kinetic diameter(Å)	5.8
Density at 20°C (g/cm ³)	0.86
Surface tension at 20°C (dyne/cm)	28.5
Depole moment(debyes)	0.45
Solubility in water at 25°C(g/L)	0.5
Liquid density at 20°C(g/ cm ³)	0.87

Table 1: Characteristics of fresh GAC sample used in the study

Mesh size	20/40
BET surfacearea (m ² /g)	850
Mesopore volume (cm ³ /g)	0.412
Micropore volume (cm ³ /g)	0.271
Moisture content (%)	<1.20

Toluene gas phase adsorption isotherms study

In adsorption phase equilibrium is established for the distribution of toluene vapor and the adsorbent surface. The equilibrium is usually expressed in terms of relative pressure (sample vapor pressure P to saturated vapor pressure P_0) of the adsorbate in the gas versus the pollutant adsorbing on the adsorbent which is expressed as mass of adsorbate per unit mass of the adsorbent. Adsorption isotherm of toluene was determined by a simple constant volumetric method (Kim *et al.*, 2006). Tedlar gas sampling bag with a capacity of 10 liter was used as a fixed volume reactor. A mass of GAC adsorbent weighted 10 mg was placed inside the bag via the relevant port. Then by using gas tight syringe a given volume of toluene was injected into the bag to produce the desired vapor concentration.

GAC porous structure

The surface area of the GAC was determined by Brunauer-Emmett-Teller (BET) analytical method. DFT (Density Functional Theory) model was used to the nitrogen adsorption isotherms to assess total micropore (pore size diameter < 2 nm) and mesopore volumes (Badosz *et al.*, 2003).

Microwave irradiation experiments

A microwave oven with power levels ranged 180-900 watt provided MW irradiation at heating frequency 2450 MHz. Quartz glass column reactor installed into the MW cavity. Stream containing toluene vapor was 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 saturating GAC bed treatment was followed by exposing the adsorbent bed to heating MW irradiation under various power levels and time durations. Surface temperature was measured by an infra red pyrometer.

Analytical measurement

The samples adsorbed on the GAC bed were desorbed by carbon disulfide and gas collected on the bag were analyzed by a gas chromatograph (GC-Philips PU4410) using a Flame Ionization Detector as proposed by OSHA method no.111(OSHA, 2002). A 30-m capillary

column with 0.32 inner diameter was used chromatographic separation. The analysis was performed by Nitrogen as carrier gas at 30 mL/min flow rate and the zone temperatures set as 60 °C for the column, 250 °C for the injector and 275°C for the detector.

RESULTS*Toluene adsorption capacity*

The breakthrough curve and data obtained for the adsorption capacity of fresh GAC sample subject to toluene vapor at various adsorption intervals is given in Fig.2 and table 3. The test with different relative humidity resulted in breakthrough curves of the same characteristics 'S' shape. However we can observe that breakthrough time varies with increasing the relative humidity. From the breakthrough curves it was observed that the saturation time of the bed was more in lower humidity. It was an indication that at lower relative humidity the GAC bed saturates around 3.3 hours later than that of experiments carried out in a higher relative humidity. The adsorption capacity of the GAC was obtained by integration of the complete breakthrough curves was 0.13g more in lower relative humidity experiments.

Table 3: Adsorption breakthrough for toluene on different relative humidity, 10g GAC, 50mL/min flow rate, 4.5 cm bed length

Relative humidity (RH) %	Saturation time (t_s) S	Adsorption capacity (g toluene/g GAC)
29±3	8.5	0.33
75±4	5.2	0.20

Fig.3 shows the adsorption isotherm data plotted the vapor relative pressure of toluene versus the toluene adsorbed on the GAC adsorbent at 25°C. We found that the adsorption isotherms data could be explained and fitted by Freundlich isotherm equation model (Villacanas *et al.*, 2006) which is given by:

$$V=K. (P/P^0)^{1/n}$$

Where V is the adsorbate concentration adsorbed on the adsorbent and K and n are regression parameters and P/P° 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 square value ($R^2=0.98$), $1/n = 0.410$ and $K= 1.60$ (mmol/g).

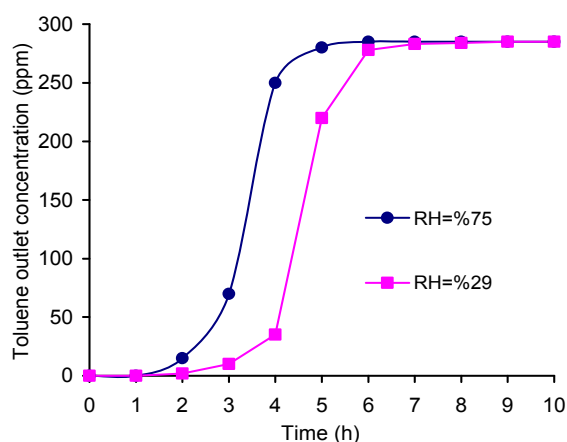


Fig. 2: Breakthrough curve for toluene adsorption in terms of relative humidity

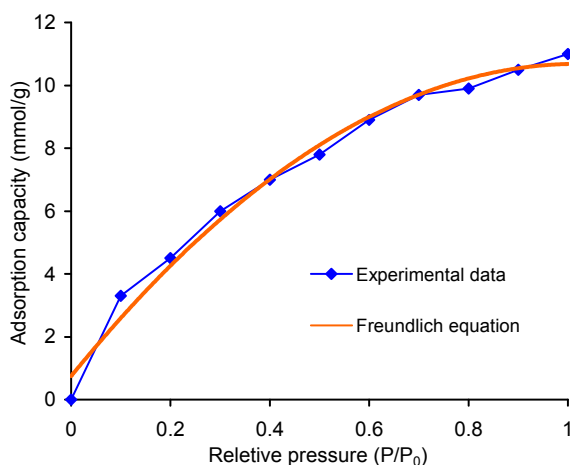


Fig. 3: Toluene adsorption isotherm at 25 °C

GAC temperature rising under microwave irradiation

Fig. 4 shows the trend of temperature rising course at various power levels. As indicated the higher the microwave power output level was, the higher the temperature of GAC bed could reach. It can be seen that at lower power levels less than 360 watt the temperature rising course is not considerable. Meanwhile a rapid heating rate was occurred when microwave power level was set at 540 or higher. In addition, it could be observed that at power levels more than 540 initial rapid increases in temperature were eventually followed by a slow down temperature increase finally reach a constant. Surface temperature of granular activated carbon measured around 600 °C within 90 seconds under microwave radiation at power levels of 900 watt, which is higher than the ignition temperature of toluene i.e. 480 °C. Thus thermal treatment experiments were followed by high power level of microwave at 900 watt.

Toluene removal under microwave irradiation process

First the gaseous stream containing toluene was treated without passing through granular activated carbon to examine if decomposition of toluene vapor was only due to microwave

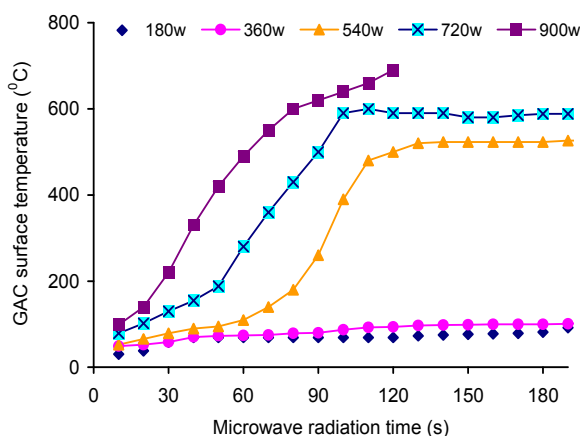


Fig. 4: GAC temperature rising trend at various MW power level

irradiation. The analysis of the outlet streams in the experiment carried out with only microwave radiation for 30 minutes showed that less than 1% of organic compound in the inlet stream was reduced.

To investigate the continuous thermal treatment of toluene under microwave radiation, inlet gaseous stream passed through fresh and preheated granular activated carbon respectively. Fig. 5 illustrates the decomposition levels of the organic compound over fresh and preheated granular activated carbon bed as a function of microwave irradiation time. As can be understood from Fig. 5 toluene was effectively removed after 10 minutes of microwave radiation for

both fresh and preheated granular activated carbon. However the decomposition rate of toluene over preheated granular activated carbon was remarkably higher than that of experiment conducted over fresh granular activated carbon as observed at laboratory scale.

The treatment of inlet streams containing toluene vapor over the preheated granular activated carbon bed under microwave heating radiation were carried out as a function of time duration at various relative humidity. As results shown in Fig. 6 the thermal oxidation rate of toluene in terms of time duration in the presence of higher level of humidity was observed nearly close to those experiments performed in ordinary low level of laboratory relative humidity.

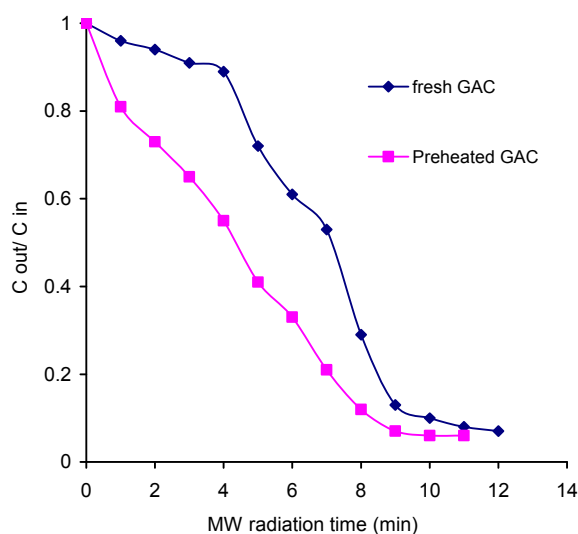


Fig. 5: Toluene removal over GAC under MW radiation

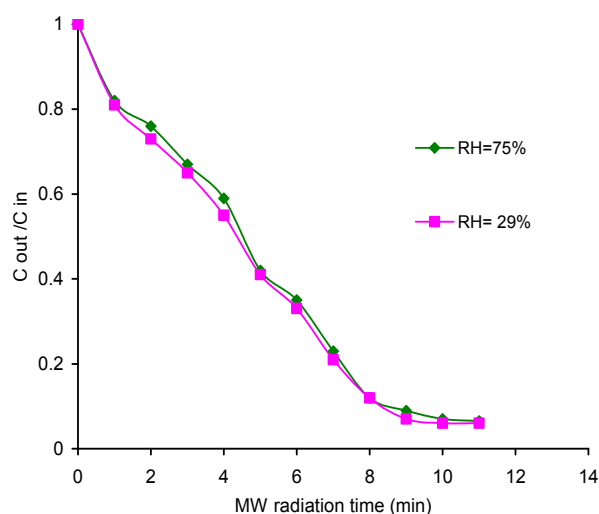


Fig. 6: Toluene removal rate in various relative humidity of the inlet stream under MW radiation

Table 4: Physical characteristics of GAC exposed to MW irradiation

	GAC characteristics		
	Surface area BET m ² /g	Mesopore volume cm ³ /g	Micropore volume cm ³ /g
*MW treatment 900W	876	0.510	0.326

*results related to 15 min. irradiation

GAC physical characteristics under MW Irradiation

Table 4 illustrates the details concerning the surface and porous structure of the GAC saturated with toluene after exposing to MW radiation at 900 W for 15 minutes. The BET, mesopore and micropore volumes of MW treated GAC loaded with toluene slightly changed over microwave treatment process. Meanwhile the relevant features increased comparing with the fresh untreated GAC as stated in Table 1. Results indicate rises in GAC surface area and micropore volumes under microwave irradiation.

DISCUSSION

There are five main findings in this study. First we found that the increase in relative humidity of the inlet gaseous streams decreased breakthrough and saturation time and resulted in decreased adsorption of toluene over granular activated carbon. Second we showed that microwave heating energy can be adsorbed and changed into heat effectively and relatively in a short time by GAC bed. Third the experimental results concluded that the raised GAC temperature under microwave radiation was able to remove and destroy completely toluene passing continuously through GAC bed in a few minutes. Fourth the texture of GAC was not deteriorated under microwave heating. Finally we demonstrated that the increase in inlet water vapor could not impact considerably on decomposition rate over GAC bed under microwave radiation.

The shape of adsorption breakthrough curve obtained for toluene is similar to previous reported experiment (Abril *et al.*, 2009, Mohan *et al.*, 2009). It indicates that toluene vapor has reached equilibrium at the adsorption period of around 9 hours. The maximum adsorption capacity was determined 0.35g toluene per g GAC. The adsorption values achieved for toluene at 300 ppm in this study is slightly better than those previously reported (Villacanas *et al.*, 2006). This may be explained by the origin, preparation method and mesh size of the GAC as well as the

concentrations and the flow rates used in the experiments. Previous studies found that the presence of water vapor along with volatile organic compounds has no significant impact on the adsorption capacity of the activated carbon until the relative humidity exceeds 75% (Tiemey *et al.*, 2006). The result that lower relative humidity of the gas stream caused a delayed breakthrough suggests that increasing in water vapor can reduce adsorption capacity which is in agreement with previous research reported by Mohan *et al.* (2009).

Previous studies have indicated that microwave could be used for carbon materials and basically the GAC could absorb microwave heating energy quickly (Jones *et al.* 2002). We observed sudden and intense bright sparks at high powers result in a hot and red carbon. The whole GAC was turned to red in few minutes and remained in the same condition for the entire treatment process under microwave irradiation. The results are in agreement with studies previously reported that the temperature rise in GAC exposed to MW radiation depends on the nature of adsorbent as well as on the microwave power level applied to the sample (Bathen, 2003; Liu *et al.*, 2004). Previous experiment reported that under microwave field an electric power dissipated within the material is proportional to the square of the electric field strength (Guo and Lua, 2000). This suggests that an increase of microwave electric field will give rise to more rapid heating on the GAC bed. At output power levels of 720W and 900W after 2 minutes of MW irradiation, the surface temperature measurements were around 600 °C and 700 °C respectively. It seems that GAC absorb microwave heating radiation more effectively and its temperature increases more quickly at the beginning. But the temperature rising rate decreased and remained constant later. The reason was considered to be that more energy is needed to maintain high temperature. It is evident that increasing power levels enhanced the removal rate of adsorbed toluene due to rapid increase

in adsorbent bed temperature. Theoretically, under microwave irradiation energy is directly supplied to the granular activated carbon 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, microwave heating supplies energy independent of the mass of gas passed into the bed (Nabais *et al.*, 2004). More than 90% of toluene passed over GAC was removed within 10 minutes 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 was setting to higher levels, less time duration was required to remove the contaminant. Therefore the treatment of pollutant by heating is more rapid and less time consuming in a MW field comparing to heating in an electric furnace. In this study a reduction in the required toluene oxidation time was observed when the GAC bed was preheated by microwave for 2 minutes before allowing the inlet stream passing through the reactor under microwave heat treatment process.

Several experiments indicated that the presence of water vapor in inlet stream may lead to a decrease in oxidation process of volatile organic compounds (Lou and Lee, 1997). On the contrary Siquin *et al.* (2000) showed that thermal decomposition of CCl_4 can be enhanced in presence of water vapor. Our experiments indicated a slightly decrease in toluene removal rates in higher relative humidity compared to that of experiment carried out in lower relative humidity. This may be explained by phenomenon that a little part of microwave radiation is absorbed by water vapor in the inlet stream.

Conventional thermal heating of activated carbon after 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). Considering toluene molecular diameter 5.8 Å, it appears that increasing in the GAC surface area and the volume of micropores due to microwave irradiation implies the nearly constant and not deteriorated adsorption capacity of the GAC. Our findings confirm earlier research on the effect of MW irradiation on the textural properties of the carbon (Ania *et al.*, 2005). According to Abril *et al.* (2009), the performance of activated carbon depends on micropore volume in a manner that narrower micropore volume influences the breakthrough and saturation times. The increase in mesopore and micropore volumes that enhances the adsorbate transportation and adsorption abilities of the GAC respectively suggests that longer application of the GAC can be obtained under microwave irradiation treatment.

We concluded that in laboratory scale test, microwave heating energy may be used effectively for the treatment of volatile organic compounds adsorbed onto granular activated carbon. Microwave heating delivers energy throughout the volume of adsorbent where the microwave-absorbing GAC is located and used as a thermal medium in combustion of toluene. We found that microwave power level was a key factor in rapid temperature rise of the GAC sample. Toluene removal increased steeply within a few minutes and reached over 90% under microwave radiation at higher power levels. This implies a lower consumption of energy. The experiments concluded that a preheated GAC under microwave heating energy could speed up thermal oxidation. The addition of water vapor in the inlet stream could lead to a slight decrease in organic compound removal rate over the GAC while the overall oxidation removal remain the same compared to stream with low content of water vapor. Conventional heating technologies including incineration for treating volatile organic compounds are suitable and economically effective when large amount of these compounds could be

provided while microwave treatment could be used for low concentrations emission streams as encountered in industrial indoors and ventilation systems. Briefly, the system is interesting from its simplicity, effectiveness, fastness and economic point of views for treating volatile vapor pollutants. Therefore microwave heating energy on GAC bed may used to decompose a variety of volatile organic compounds in gaseous stream. It is expected that this approach could be used in ventilation systems for the treatment of volatile organic compounds in work environment. However as the system reaches high temperature rapidly and due to generation of arcs in the GAC bed, further studies are required to study the decomposition reaction mechanisms of the various volatile organic vapors collected and adsorbed on the adsorbent.

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