

Using Microwave Radiation to Recover Granular Activated Carbon Exposed to Toluene Vapor

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ABSTRACT: *This paper describes laboratory scale experiments examining the impact of microwave irradiation on the Granular Activated Carbon (GAC) exposed to toluene, a common organic vapor frequently released into work environment as well as outdoor. A stream containing 300 ppm toluene was supplied and passed through the granular activated carbon. The saturated adsorbent was placed in a quartz glass reactor and treated by microwave irradiation at heating frequency of 2450 MHz at different power levels. After successive recovery cycles the adsorption capacity was evaluated through breakthrough curves and the residue analyzed by gas chromatography. We found that exposing GAC saturated with toluene under microwave irradiation at 900W for 15 minutes can remove the pollutant from the adsorbent effectively. The recovery efficiency was more than 95% after 10 cycles at high power levels. GAC surface area and porosity values were increased slightly over recovery cycles. Our experiments indicate that GAC could be reused after several cycles of treatment and recovery processes while maintaining its original adsorption capacity and physical properties.*

KEY WORDS: *Toluene vapor, Granular activated carbon recovery, Adsorption, Microwave irradiation.*

INTRODUCTION

Organic solvents are Volatile Organic Compounds (VOCs) with varying chemical composition. Environmental Protection Agency (EPA) has defined VOCs as compounds with a room temperature vapor pressure greater than 70Pa. [1]. Industrial solvents have a wide variety of uses and are usually used in degreasing of metals, painting and coating, dry cleaning, extraction, printing, fuels and polymer manufacturing. They commonly used to dissolve other organic materials such as oil, fats, resins, rubber, lacquers, waxes and plastics. Industrial use of solvents resulted in emitting VOCs in occupational settings as well as

environment and non industrial indoor environment. Industrial emissions account for 50% of VOCs in environment [2,3,4]. Occupational exposures to VOCs are harmful and toxic to workers with numerous damaging effects including mild irritation, headache, nausea, and damage to kidneys, liver and central nervous system. They are considered as carcinogenic and mutagenic agents [5,6,7]. VOCs also have negative impacts on environment. outdoor emissions are responsible for destroying stratospheric ozone layer, causing acid rains, producing ground level ozone pollution due to photochemical reaction

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(chemical reaction of VOCs with oxides of nitrogen in the presence of sunlight) and changing global climate. Regulations are set in many countries to control workers' exposure and environmental emissions. OSHA proposed the time weighted average exposure limit for toluene 200ppm [8]. Solutions for controlling volatile organic compounds are described in literatures[9-11]. Various control methods involves thermal incinerations [12,13], catalytic incinerations [14-16] and photocatalytic oxidation [17-19] are used to control and treating VOCs.

Thermal combustion uses high temperature (700-1000 °C) and thus costly and is suitable for large volume of gas emissions with high concentrations. 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 [20-22] but the adsorbent become saturated after constant use and require further treatment. The adsorption performance of activated carbon is mostly depends on the porous structure of activated carbon [23]. Recovery of activated carbon by hot steam decreases the adsorbent capacity and damages activated carbon surface structure [23,24] resulting in short term use and disposal of activated carbon. Therefore, Efficient recovery of activated carbon is required to ensure the reuse of it for a long term period and simultaneously maintain its adsorption capacity at an acceptable level.

Jones et al.[25] discussed Micro Wave (MW) heating applications in environmental engineering in details. Compared with conventional heating, MW heating has many advantages: 1-MW energy heats the material from inside out, 2-selective heating depend on the nature of the object, 3-there is no need for heat convection through a fluid. 4-MW energy provides rapid heating, no direct contact between the MW heating source and the heated material, 5-Ease of heating process control i.e. as soon as the MW radiation is switched off the source of the heat is quickly remove from the adsorbate and adsorbent, 6- high temperature capabilities, 7- time and energy savings, and 8- increase chemical reactivity. Moreover, MW processing systems are also relatively compact, portable, maintainable and cost effective.

There are growing researches of MW processing applications in industrial and environmental engineering

due to its advantages in the area of combustion [26]. studies showed microwave energy as a possible method in waste treatment to extract contaminants and organic solvents from soil [27], and metals from sludge[28], processing minerals [25] as well as water purification [29]. It has also been suggested that microwave treatment is less time consuming. Microwave radiation has also been used for the production of activated carbon [30]. Studies showed that Granular Activated Carbon (GAC) can absorb MW radiation leading to rapid heating [31-33]. Therefore using MW radiation may be a promising alternative in treating volatile organic compounds.

In the present study we applied microwave radiation energy of an electric furnace together with granular activated carbon adsorption technique aiming to treat volatile organic vapors and regenerate the adsorbent. We used toluene in this study as the organic vapor pollutant to conduct our experiments. Toluene is one of the selected primary work environment air pollutants because it has high occurrence and also well suited for the laboratory work because it is easy to measure.

EXPERIMENTAL SECTION

Materials

Activated carbon in the form of pellet manufactured by Merck Company was prepared. The activated carbon used in the study was coconut based and the size of pellets was around 2.5mm with molecular weight of 12.01 g/mol. The pellets were crashed into granular activated carbon. A domestic mixer was used to crush the pellets of activated carbon into granular form. Activated carbon in the form of Granular Activated Carbon (GAC) with a mesh size of 20x40 was used in the experimental tests as proposed by OSHA for gas phase applications [8]. Granules were passed through a standard mesh size No. 20 sieve (0.84 mm pore size) and retained on a standard mesh size No. 40 sieve (0.42 mm pore size) to prepare the desired mesh size. 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 and then dried by an infrared heating oven (Infrared Sarterious) at 120 °C for 1 h. and stored in a desiccator for 48h before using in the relevant experiments. The moisture content of GAC was measured

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

Mesh size	BET surface area m ² /g	Mesopore volume cm ³ /g	Micropore volume cm ³ /g	Moisture content %
20/40	1850	0.412	0.2712	<1.20

Table 2: Toluene chemical and physical properties.

Properties	Toluene
Molecular formula	C ₇ H ₈
Molecular weight(g/mol)	92.14
Boiling point °C	110.6
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/m ³)	0.87

by Sartorius Moisture Analyzer. In the study toluene vapor was used as a pollutant and representative of VOCs. Toluene prepared from Merck Company with a purity of %99.97 was introduced into the gas stream to pass through and saturate the GAC and then treated with microwave irradiation. The main chemical and physical properties of toluene as pollutant used in the study are presented in Table 2.

Experimental process

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

Vapor generator

The laboratory scale reactor system consisted of a dynamic vapor generation system. Fig. 1 shows the microwave setup which was applied to carry out experiments. An ejector was used to supply a given vapor stream. Purified air is 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 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 (Omega) to supply a desired and constant flow rate. The experimental segments were connected by Tygon tubes. A gas analyzer (Phocheck :5000EX) was used to determine and monitor the vapor concentrations flowing into the reactor and ensure the constant concentration of toluene in the stream gas. In order to provide a constant vapor concentration the experiments were carried out under a laboratory hood by adjusting temperature, humidity and flow rate of carrier gas introduced to the reactor. When the concentration reached to a stable and equilibrium status the experiments were conducted by introducing the gas stream through the column containing the adsorbent.

Adsorption and MW recovery experiments

Adsorption laboratory tests of toluene vapor carried out in a Quartz glass column with 2-cm inner diameter and the length of 30 cm. The column loaded with a fixed adsorbent bed containing 10 g. of granular activated carbon. A perforated quartz plate was inserted in the column to hold the granular activated carbon bed diffusing the inlet gas through the bed. The adsorption experiments were performed at desired temperature 25 ± 4 °C. 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% [21]. 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 toluene vapor stream was 300 ppm at a volumetric flow rate of 50 ml per minute passed through the GAC bed. 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. The inlet and outlet concentrations were measured by Gas Chromatography (GC) to obtain

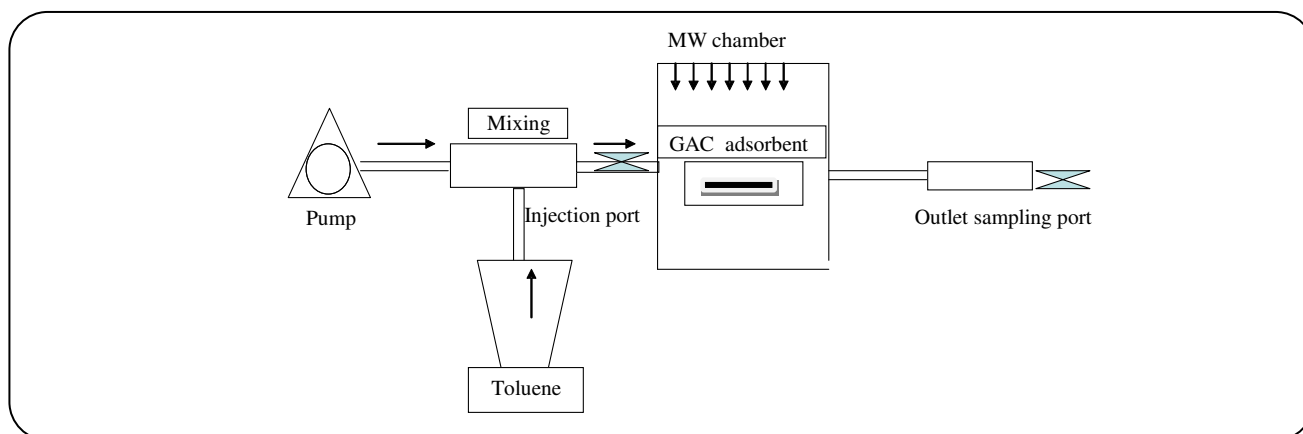


Fig. 1: Laboratory scale Experimental setup for toluene removal and GAC recovery by MW irradiation.

breakthrough data. Breakthrough time and saturation adsorption time were calculated from adsorption curves. When the outlet toluene concentration reaches 5% of the initial concentration in the inlet, which is 15 ppmv, breakthrough time is determined. Saturation adsorption time is measured when the outlet toluene concentration increased to 95% of the inlet concentration which is 285 ppmv in our experiment. When the adsorbent bed reached the saturation point the adsorbate extracted and the adsorption capacity was measured by gas chromatography in grams of toluene loaded per 1 gram GAC. The adsorption capacity of the GAC was obtained on the basis of inlet and outlet vapor streams by integrating the area above the curve that describes the toluene vapor's concentration as a function of time by the following relation:

$$q = \int_{t_0}^t \frac{Q_g(C_{in} - C_{out})}{M_{GAC}} dt \quad (1)$$

where q is the adsorption capacity of GAC, Q_g is vapor flow rate, M_{GAC} is the mass of GAC used in the column, C_o and C_i are outlet and inlet toluene vapor concentrations respectively.

A microwave oven with power levels ranged 175-900 W provided MW irradiation at heating frequency 2450 MHz. Quartz glass column reactor installed into the MW cavity in a vertical position. 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 SKC adsorbent air sampling tube. After saturating GAC bed by passing the gas stream contaminated with Toluene vapor the treatment followed by exposing the adsorbent bed to heating MW irradiation

under various power levels and time durations. The recovered GAC samples were used for the adsorption of toluene vapor at the same previous laboratory and ambient conditions through a series of successive tests carried out to determine the adsorption capacities and recovery cycles efficiency as a function of microwave energy in view of modification of the GAC properties. Recovery efficiency is defined as:

$$\text{Recovery Efficiency} = \left(\frac{\text{adsorptive capacity of the recovered GAC}}{\text{Adsorptive capacity of the fresh GAC}} \right) \times 100 \quad (2)$$

Analytical measurement

The samples adsorbed on the GAC bed were desorbed by carbon disulfide and gas collected on the sampling adsorbent tubes were analyzed by a gas chromatograph (GC-Philips Model: PU4410) using a Flame Ionization Detector as proposed by OSHA method no.111[33]. A 30-m capillary column with 0.32 mm inner diameter was used as 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. Injection volume was 1 μ l and external standards were prepared in Carbon disulfide (CS_2) solution to obtain a calibration curve to bracket the sample concentrations. The inlet and outlet gas samples were passed through and collected with SKC glass sampling adsorbent tubes with a length of 7 cm and internal diameter of 4 mm. The collected samples were extracted and recovered by Carbon disulfide (CS_2) of chromatographic grade. The extracted solution was injected into GC to determine toluene concentration.

Table 3: Amount of toluene remained on GAC adsorbent following various MW powers and irradiation periods.

	MW irradiation duration (min.)					
	0	5	10	15	20	25
	Toluene residue on GAC adsorbent (mg toluene/g GAC)					
MW power levels (W)						
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

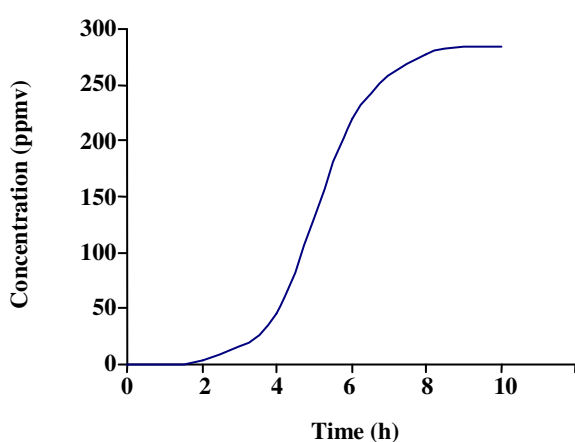


Fig. 2: Breakthrough adsorption curve for toluene at 300 ppm over 10 gram GAC.

The surface area of the GAC was determined by Brunauer-Emmett-Teller (BET) analytical method. Nitrogen adsorption isotherms were used to determine the micropore and mesopore volumes measured from nitrogen adsorption isotherms and determined by Dubinin equation [34]. The surface temperature of the granular activated carbon after exposure to microwave heating irradiation was measured by an infrared pyrometer.

RESULTS AND DISCUSSION

Equilibrium adsorption capacity

The adsorption experiments were determined by using 10 g GAC as the adsorbent bed in a vapor stream carrying toluene at an inlet concentration of 300ppm and a flow rate of 50 ml/min at 25 °C. The breakthrough curve obtained for the adsorption capacity of fresh GAC sample subject to toluene vapor at various adsorption intervals is

shown in Fig. 2. It indicates that toluene vapor has reached equilibrium at the adsorption period of around 9 hours. The maximum adsorption capacity was determined 0.35 g toluene per g GAC. The adsorption values achieved for toluene at 300 ppm in this study is slightly better than those previously reported [19]. 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.

Toluene removal under microwave irradiation process

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 GAC saturated with toluene was exposed to various microwave power level settings at 180, 360, 540, 720, 900 watts for different MW 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 3. We found that at high power levels at 540, 720 and 900 watt, the amounts of residue in the GAC were not detectable after 25, 20 and 15 minutes while at low power levels at 180 and 360 W irradiation was not adequate to remove the contaminant.

Previous studies have indicated that microwave could be used for carbon materials and basically the GAC absorb microwave heating energy quickly [26]. 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 increase

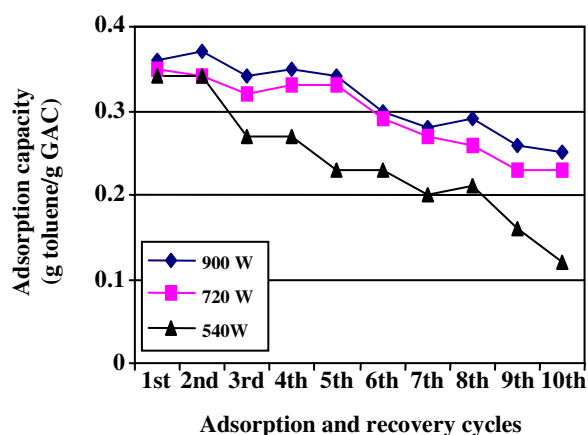


Fig. 3: Adsorption capacity in terms of MW treatment cycles at various power levels for 25 min. MW irradiation.

in GAC exposed to MW radiation depends on microwave power levels [27,33]. The surface temperature measurements were around 730 °C and 800 °C respectively at output power levels of 720W after 20 minutes and 900W after 25 min of MW irradiation. 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 [31]. More than 90% of toluene loaded on GAC was removed over periods of 15 minutes irradiation at 720 W and 10 minutes irradiation at 900 W. GAC is an excellent microwave adsorbing material to absorb and convert microwave energy into thermal energy. Thus the rapid microwave induced heating on GAC could remove adsorbed toluene efficiently at high power levels. 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 with heating in an electric furnace.

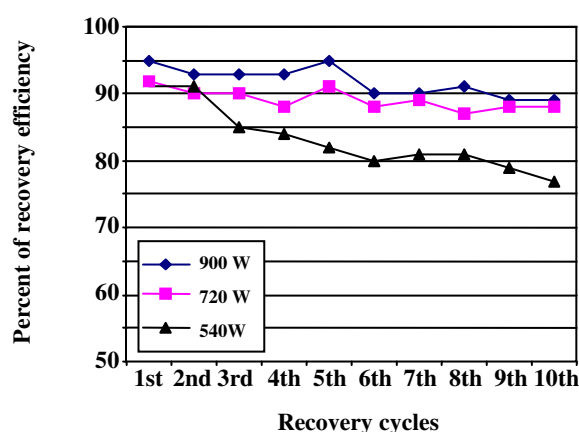


Fig. 4: Recovery efficiency obtained under MW radiation at various levels.

Multiple GAC Recovery in microwave field

After saturated GAC was exposed to microwave irradiation, we measured the effectiveness of MW on the GAC 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 reused to adsorb toluene vapors and the recovery and adsorption cycles were repeated for 10 times. The GAC adsorption capacity exposed to toluene is shown in Fig. 3 as a function of multiple recovery cycles. We obtained interesting results comparing adsorptive capacities of fresh GAC and samples treated and recovered by MW irradiation. We found that after five repetitive treatment cycles by MW, 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 mean that repeated microwave irradiation at power levels more than 720 W for adsorbent recovery does not have a remarkable weaken effect on the GAC adsorption capacity comparing to its fresh value. However the trend of adsorption capacity showed more reductions at lower MW power of 540 W. Fig. 4 shows the trends obtained in recovery efficiency values during several GAC treatments under MW irradiation. Data provided in Fig. 3 indicates that after 10 times of GAC recovery the decrease in adsorption capacity compared to its untreated initial sample was about 10% at MW power of 720 W and 900 W while it was 23% at MW power of 540 W. Furthermore at higher output powers the adsorption

Table 4: Physical characteristics of GAC exposed to MW irradiation.

GAC characteristics			
*MW treatment 900W	Surface area BET m ² /g	Mesopore volume cm ³ /g	Micropore volume cm ³ /g
1st	1876	0.510	0.326
5th	1880	0.620	0.379
10th	1923	0.693	0.235

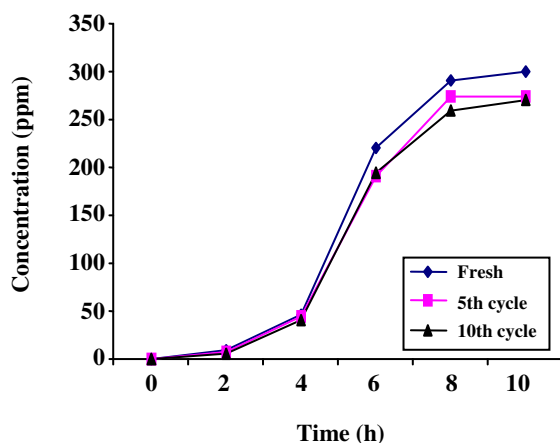


Fig. 5: Breakthrough adsorption curves for toluene at 300 ppm over 10g GAC in the fresh and MW recovered cycles.

capacity preserved and remained almost constant after 5 recovery cycles and subsequent cycles resulted in slight reductions in adsorption capacity. Fig. 5 indicates the breakthrough adsorption curves for the fresh and multiple treated GAC under MW irradiation at 900W. It is important to note that as observed from data points in Fig. 5 the shapes of breakthrough adsorption curves for GAC did not change over repeated microwave treatment cycles when comparing with the curve of fresh one. It can be inferred from the experiments that MW can maintain the adsorption capacity at an acceptable level comparing to its fresh value and increase the life of GAC for VOCs treatment. However after repeated recovery adsorption cycles will eventually lead to decreased adsorption. Previous studies showed that the adsorption capacity of low concentration of toluene and benzene mainly depend on adsorbent micropore volume since most molecules of gaseous pollutants range in diameter from about 0.4 to 0.9 nm [31,35]. These changes in adsorption capacity during adsorption-recovery cycles can be attributed to GAC structural modifications under heating microwave irradiation [23].

GAC physical characteristics under MW 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[27]. 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 25 minutes. The BET, mesopore and micropore volumes of MW treated GAC loaded with toluene slightly changed over successive microwave recovery and treatment cycles. Meanwhile the relevant features increased comparing with the fresh untreated GAC as stated in table 1. This implies that improvement in surface and porosity characteristics may be probably connected to the nearly constant and not deteriorated adsorption capacity of the GAC in successive cycles. The findings confirm previous literature on the effect of MW irradiation on the textural properties of the carbon [24]. According to *Abril et. al.* [22] the performance of activated carbon depends on micropore volume in a manner that narrower 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 the interior and gas expansion occurs, thus the diffusion of the vapor out of the adsorbent may be dominated by a pressure-driven flow. 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. However after 10 cycles of adsorption and MW recovery the structural textures shifted to dominant mesopore size distribution and micropore volume decreased. In our experiments decreased micropore volume after ten cycle adsorption-recovery with MW led to a lower concentration capacity. This is in agreement with *Ania et. al.* [23] experiments

pointing to microporous carbons shifted to considerable development of mesoporosity and decreased volume of microporosity after successive MW treatment. However, Liu et. al. [36] used GAC adsorption-MW recovery process for treatment of PCBs in a liquid phase adsorption indicated that although the decreased micropore area resulted in a lower adsorption capacity, the increased external surface was in favor of the dynamic adsorption of PCBs molecules with relatively large size thereby the adsorption amount was increased.

CONCLUSIONS

The results of our laboratory scale tests demonstrate that 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 adsorbate is located. 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. Moreover, High recovery efficiencies obtained after 10 treatment and recovery cycles. We concluded that microwave recovery of saturated activated carbon loaded with toluene maintain 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 volatile organic compounds are suitable and economically effective when large amount of these compounds could be provided while microwave treatment and recovery 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 and recovery of volatile vapor pollutants. We expect 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 examine the decomposition of the volatile vapors collected on the adsorbent.

Abbreviation and nomenclature

GAC	Granular Activated Carbon
VOCs	Volatile Organic Compounds
EPA	Environmental Protection Agency
MW	Microwave
OSHA	Occupational Safety and Health Administration
ppm	part per million
q	Adsorption capacity
Q_g	Vapor flow rate
M_{GAC}	Mass of adsorbent
C_i	Inlet (upstream) concentration
C_o	Outlet (down stream) concentration
Å	Angstrom

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