

Prediction of a Mathematical Model to Validate Maximum Regeneration Efficiency of Phenol Exhausted Activated Carbons used in Water Treatment Operations by Domestic Microwave Oven



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Abstract

This paper describes laboratory-scale experiments examining the impact of microwave irradiation (MW) on the regeneration of spent granular activated carbons (GAC) saturated with phenol. The process was conducted in an aqueous solution using domestic microwave oven. The investigation was achieved to evaluate its adsorption efficiency after successive regeneration cycles and to ensure its practical feasibility. Different factors have been studied such as microwave power level, pH of the solution, weight of the active carbons; volume of the regenerated solution, number of adsorption and regeneration cycles. A mathematical model has been developed to measure the regeneration efficiency of the GAC adsorption capacity. It was found that, for the same power level, as the time of exposure increases, the regeneration efficiency increases. As, the microwave oven power increase from 160 to 800 W, the regeneration efficiency increases from 60 to 90% after 1.5min of radiation time, during the regeneration process the pH of the solution increase from about 6.03 to 7.15. We concluded that (GAC) could be reused after repeated regeneration cycles under microwave irradiation while maintaining its original adsorption capacity and physical properties, it is expected that; this approach could be used in ventilation systems for the treatment of Volatiles organic compounds (VOCs) in work environments.

Keywords: Volatiles organic compounds; Regeneration efficiency; Adsorption; Desorption, Microwave irradiation; Active carbon; Phenol removal

Statement of Novelty

A mathematical model has been developed to measure the regeneration efficiency of the GAC adsorption capacity through using of domestic microwave oven to study the regeneration of GAC after saturation with phenol in aqueous solution. The pre-step of adsorption revealed that pseudo first-order is a good model to represent kinetic adsorption of phenol on GAC. Some of the most important uses of activated carbon are air pollution control, solvent recovery, food processing, chemical and pharmaceutical industries, wastewater and water treatment processes (i.e., purification of drinking water). The main advantages overcoming thermal regeneration techniques (steam or inert gas) which involve high energy consumption and reduction of adsorption capacity (5-15%), chemical regenerations which require supercritical conditions (large investment cost). Thus, a widespread and great progress in this area can be expected in the future.

Introduction

Granular activated carbons is one of the most effective and important adsorbents for the removal of a wide variety of pollutants from both aqueous and gaseous environment [1]. This is due to their high adsorption capacity, fast adsorption process and the ability for regeneration. Some of the most important uses of activated carbon are air pollution control, solvent recovery, food processing, chemical and pharmaceutical industries, wastewater and water treatment processes (i.e., purification of drinking water). 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, while MW treatment and recovery could be used for low concentration

emission streams as encountered in industrial buildings and ventilation systems [2-7]. The microwave irradiation (MW) is the one of important methods for regeneration due to its ability for molecular level heating that leads to homogeneous and quick thermal reactions [8-10]. Many of technical works are used this method; in addition, there is no evidence that his technique can damage the carbon, although it may change its pore distribution to some extent [11]. The main advantages overcoming thermal regeneration techniques (steam or inert gas) which involve high energy consumption and decreasing of adsorption capacity to (5–15%), the biodegradable substances are only effective in the chemical regenerations which require subcritical or supercritical conditions (large investment cost) and biological regenerations. Thus, a widespread and great progress in this area can be expected in the future [5].

Phenols are volatile organic solvents and belong to volatiles organic compounds (VOCs) which varies in chemical compositions of a group of common environmental contaminants. The industrial sources for these contaminants are: oil refineries, coal gasification sites, petrochemical units, etc. Which generate large quantities of phenols. In addition, phenolic derivatives are widely used as intermediates in the synthesis of plastics, dyes, pesticides, insecticides, paper, pulp and dye manufacturing [12,13]. These contaminants, even at low concentrations, can cause unpleasant taste and odor of drinking water and most of these compounds are recognized as toxic carcinogens [6]. There are many different methods were designed to remove phenol from aqueous media: such as aerobic and anaerobic biodegradation, oxidation by ozone [6]. Besides this, uptake by ion exchange resins, etc. [14,15]. However, adsorption by activated carbon is best and most frequently used method [16,17]. Moreover, the process is economical because it requires low capital cost and there are abundant low-cost materials available to produce active carbon from it [7]. The regeneration processes are very important to extra cost effect the whole task, and the advantages of adsorption technique over other conventional advanced treatment methods are; it can remove both organic as well as inorganic constituents even at very low concentrations, it is relatively easy and safe to operate, both batch and continuous equipment can be used, no sludge formation and frequently the adsorbent can be regenerated and used again [18-21]. In this work, the regeneration of spent granular activated carbon (GAC) in aqueous media using domestic microwave set under different operating conditions is investigated to evaluate its adsorption efficiency after successive regeneration cycles and to ensure its practical feasibility.

Methodology

Experimental work

Materials

Materials used in this study are crystalline grade phenol (98% purity), bi-distilled water and commercial granular active carbon,

having (specific surface area 830 m²/g, particle size ranged from 1500 to 250 μm, bulk density=447 Kg/m³ and ash content 4.22%). A concentrated stock solution of phenol (1000 ppm) was prepared and diluted solutions for samples were prepared from this stock using bi-distilled water. Prior to use, GAC was washed by bi-distilled water to remove any foreign materials and impurities, it is then dried at 110 oC for 24 hour and stored in desiccators for planned experiments. Phenol-adsorption on active carbon was firstly performed to assist the adsorption behavior. Next, the phenol-removal processes by MW were performed to evaluate the regeneration process.

Phenol adsorption process and kinetics

Kinetic study and isotherm experiments were carried out to assess the behavior of this specific GAC for the phenol adsorption process. This achieved by placing a known quantity of the adsorbent, 0.2 g, in a 250 ml, conical flask, containing 100 ml of an aqueous solution of phenol with concentration 140 ppm. Adsorption was performed by batch method using shaker (STUART SCIENTIFIC ORBITAL SHAKER SO1) at 120 rpm. Residual-phenol concentration was determined with time, 60 minutes interval until equilibrium, by measuring the absorbance at $\lambda_{max} = 270$ nm using (SHIMADZU UV-160 recording spectrophotometer). The concentration was calculated from a pre-determined calibration curve. Adsorption capacity was calculated according to $Q = (C_i - C_t) V / W$; where C_i and C_t are the initial and timed concentrations (mg/l), respectively, of phenol in solution; W is the weight (g) and V is the volume (l), of the adsorbent. To determine the rate constant of phenol-adsorption on GAC, data were collected based on triplicate measurements and pseudo first order model was selected and employed; and its linear formula is as follows [8]:

$$\text{Log}(q_e - q_t) = \text{log } q_e - kt/2.303$$

Where q_e , q_t , k and t are adsorbed amount (mg/g) at equilibrium, adsorbed amount (mg/g) at time t , rate constant (min^{-1}) and time in (minutes), respectively.

Adsorption isotherm experiments

The maximum adsorption capacity of GAC used in this work was determined using the following Langmuir model [9]:

$$1/q_e = 1/Q_0 + 1/(bQ_0C_e)$$

Where q_e (mg/g) is the adsorbed amount at equilibrium, C_e (mg/l) is the concentration at equilibrium. Q_0 (mg/g) and b (l/mg) are the maximum adsorption capacity and adsorption affinity, respectively. The adsorption isotherm experiments were performed at ambient temperature of about 20°C, using sample dosages of 0.2 g/100 ml aqueous solution of initial phenol concentrations 40, 60, 80, 120, 140, 160 and 200 ppm. The conical flasks were shaken at 120 rpm. To ensure maximum adsorption, samples were left for 24 hours which was pre-determined to be sufficient to attain equilibrium.

Microwave regeneration process

The adsorption and desorption process was repeated 5 times, considering the same conditions, to determine the maximum efficient number of regeneration cycles. The regeneration process of the spent GAC, was carried out in a domestic microwave set (MW hot point model 6640) of maximum output power of 800 W at frequency 2400 MHz with adjustable power settings and time programming. Each GAC sample, 0.2 g, was placed in a 250 ml, flat-bottomed flask, containing 100 ml distilled water, and then exposed to microwave irradiation under controlled operating conditions. The power level (PL) of this set is as follows: PL1= 160 W, PL2= 320 W, PL3= 480 W, PL4= 640 W and PL5= 800 W. For each PL, the effect of time was considered by measuring the absorbance of phenol-residue in the solution at different time intervals: 0.5, 1, and 1.5, 2, 2.5 and 3 minutes. It was always considered to avoid evaporation state.

pH change Monitoring

The change in pH of the medium after regeneration, for the different PL and time conditions, with respect to the regeneration of the media was determined by using pH meter (pH-211 microprocessor pH meter Hanna instruments).

Regeneration water Volume

The amount of phenol retained on GAC was determined using the adsorption technique. To assess the economic value of the regeneration process, the volume of bi-distilled water used for regeneration process was varied to this study. To investigate this effect on the regeneration process: A series of volumes (30, 50, 70, 100 ml) were used for regeneration of 0.2 g GAC saturated with phenol at PL5 and irradiation time 1.5 minute.

Weight loss of the regenerated GAC

The weight loss in the original weight of GAC sample due to successive regeneration cycles was investigated as an important factor affecting the regeneration process. A laboratory scale experiment was designed to study this effect by subjecting one gram of GAC sample to a repetitive regeneration process for 4-5 cycles under operating conditions of PL5 and irradiation time 1.5 minute, the change in weight has been determined using a 4 digits analytical balance (Scaltec SBA 31).

Characterization of the fresh and regenerated GAC

The specific surface area of the fresh, exhausted and regenerated GAC was determined using the Micrometrics ASAP 2405N adsorption analyzer by measuring nitrogen adsorption/desorption isotherms at 77.4 K. IR spectra of the selected samples were recorded using Fourier-transform infrared spectroscopy technique (SHIMADZU FTIR-8400).

Theoretical Aspects

A mathematical model has been developed to measure the

regeneration efficiency of the GAC adsorption capacity; based on both of the following mathematical models:

f2

Pseudo first-order mathematical model is matched and applied as follow: from Figure 2: Pseudo first-order plot of phenol adsorption on GAC at 20°C and the equation of the graph is:

$$y = m x + c$$

$$M = \frac{y_2 - y_1}{x_2 - x_1} = \frac{1.29 - 0.99}{4 - 5.52} = -0.197$$

$$C = 2.07$$

$$Y = -0.197X + 2.07$$

Comparing this with pseudo first order:

$$\log(q_e - q_t) = \log(q_e) - \frac{kt}{2.303}, \text{ We find:}$$

$$\frac{k}{2.303} = -0.197, K = 0.454 \text{ min}^{-1}$$

$$\text{Log } q_e = 2.07, q_e = 120 \text{ mg/g}$$

To get activation energy required by the reaction:

From Arrhenius equation and taking the pre-exponential factor as 0.004 (common).

$$\ln k = \ln A - \frac{E_a}{RT}$$

$$\ln(0.454) = \ln(0.004) - \frac{E_a}{8.314 * (20 + 273.15)}$$

$$E_a = -11532 \frac{\text{J}}{\text{Mole}} = -11.53 \frac{\text{KJ}}{\text{Mole}^*}$$

Mathematical model selected; Langmuir model is chosen: The equation of the graph is: $y = m x + c$

$$M = \frac{y_2 - y_1}{x_2 - x_1} = \frac{0.025 - 0.015}{0.12 - 0.04} = 0.125$$

C = 0.00823

Y = 0.125X + 0.00823

Comparing this with $\frac{1}{q_e} = \frac{1}{Q_0} + \frac{1}{bQ_0c_e}$, it is found that:

$$\frac{1}{Q_0} = 0.00823, Q_0 = 121.5 \text{ mg/g}$$

$$\frac{1}{bQ_0} = 0.125 = \frac{1}{b * 121.5}, b = 0.0658$$

Mathematical model: Relationship between Langmuir & pseudo 1st order:

From Langmuir:

$$\frac{1}{q_e} = \frac{1}{Q_0} + \frac{1}{bQ_0c_e}$$

$$q_e = \frac{b * C_e * Q_0}{b * C_e + 1}$$

From pseudo 1st order:

$$\log(q_e - q_t) = \log(q_e) - \frac{kt}{2.303}$$

We get:

$$\log\left(\left[\frac{b * C_e * Q_0}{b * C_e + 1}\right] - q_t\right) = \log\left(\frac{b * C_e * Q_0}{b * C_e + 1}\right) - \frac{kt}{2.303}$$

Knowing b, q_e, Q₀, C_e, t will result in determination of the reaction constant k.

Validation:

Find the reaction constant k @ t=6h knowing that:

C_e=1.418 mg/l, b=0.0658, Q₀=121.5m/mg.

First,

@ t=6h, log(q_e-q_t)=0.0996, (form Figure 2).

$$\log\left(\left[\frac{b * C_e * Q_0}{b * C_e + 1}\right] - q_t\right) = 0.996$$

$$\left[\frac{b * C_e * Q_0}{b * C_e + 1}\right] - q_t = 9.91,$$

$$\left[\frac{0.0658 * 1.418 * 121.5}{0.0658 * 1.418 + 1}\right] - q_t = 9.91$$

q_t=0.46m/mg

Applying into the equation,

$$\log\left(\left[\frac{b * C_e * Q_0}{b * C_e + 1}\right] - q_t\right) = \log\left(\frac{b * C_e * Q_0}{b * C_e + 1}\right) - \frac{kt}{2.303}$$

$$\log\left(\left[\frac{0.0658 * 1.418 * 121.5}{0.0658 * 1.418 + 1}\right] - 0.46\right) = \log\left(\left[\frac{0.0658 * 1.418 * 121.5}{0.0658 * 1.418 + 1}\right]\right) - \frac{k\left(\frac{6}{60}\right)}{2.303}$$

By solving the above equation; it is found that: K= 0.454 min⁻¹

Results and Discussion

Adsorption kinetics of Phenol

The adsorption experiments were carried out under shaking conditions which limits the effect of film diffusion .The time profile (8 hours) for phenol removal from water by GAC, at 20°C, is shown in Figure 1, it can be noticed that phenol removal is smooth and continuous with time which suggest open pores texture of GAC and its rate of adsorption can be suggested to be constant over this time range [20,22]. As shown in Figure 2, pseudo first-order kinetic model can be a reasonable candidate under this case. This relation is linear with correlation factor R= 0.974 which indicate good confidence in data. The adsorption rate constant from this relation is k = 0.454 min⁻¹ which is considered high enough in this type of application [18,21].

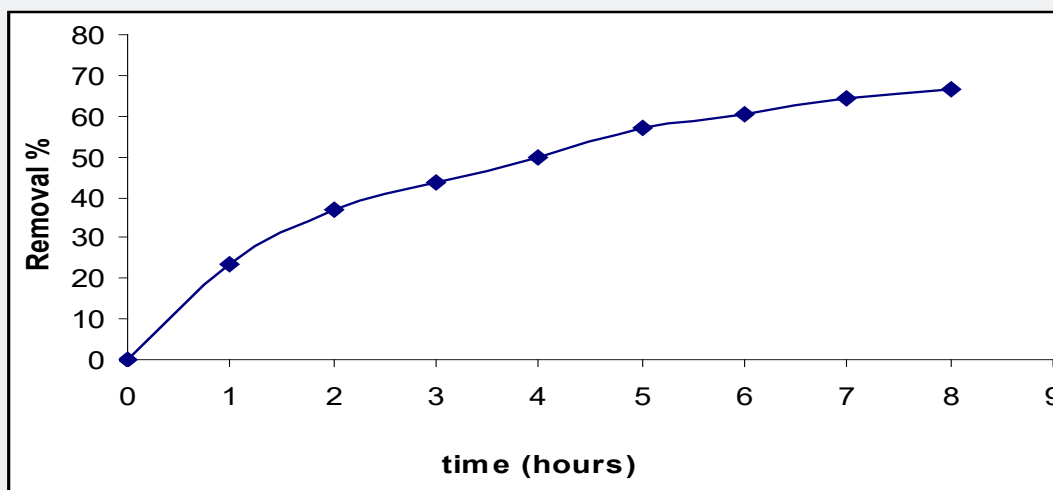


Figure 1: Removal percentage of phenol with time at 20°C.

Adsorption isotherm of Phenol

It is indicated from Figure 3 the Langmuir adsorption isotherm of phenol at 20 °C. The relation is linear with correlation factor R= 0.970 which indicate good confidence in data. The maximum capacity derived from equation is Q₀ = 123.5 mg/g

which is comparable with some related published values [10]. This indicates that phenol monolayer adsorption on GAC and adsorption energy is the same for all sites [1,23]. It was found that phenol adsorption on activated carbon can be achieved following a complex interplay through three possible mechanisms: [11].

- a. π - π dispersion interaction between the phenol aromatic ring and the delocalized π electrons present in the aromatic structure of the graphite layers [12].
- b. Formation of hydrogen bond [12].
- c. Formation of electron donor- acceptor complex at the carbon surface where the oxygen of the surface carbonyl group

acts as electron donor while the phenol aromatic ring being the acceptor [13].

d. However, to have a good knowledge about the mechanism of adsorption a several isotherm models it must be applied on the obtained data; these models can give a clear picture about the main mechanism of phenol adsorption.

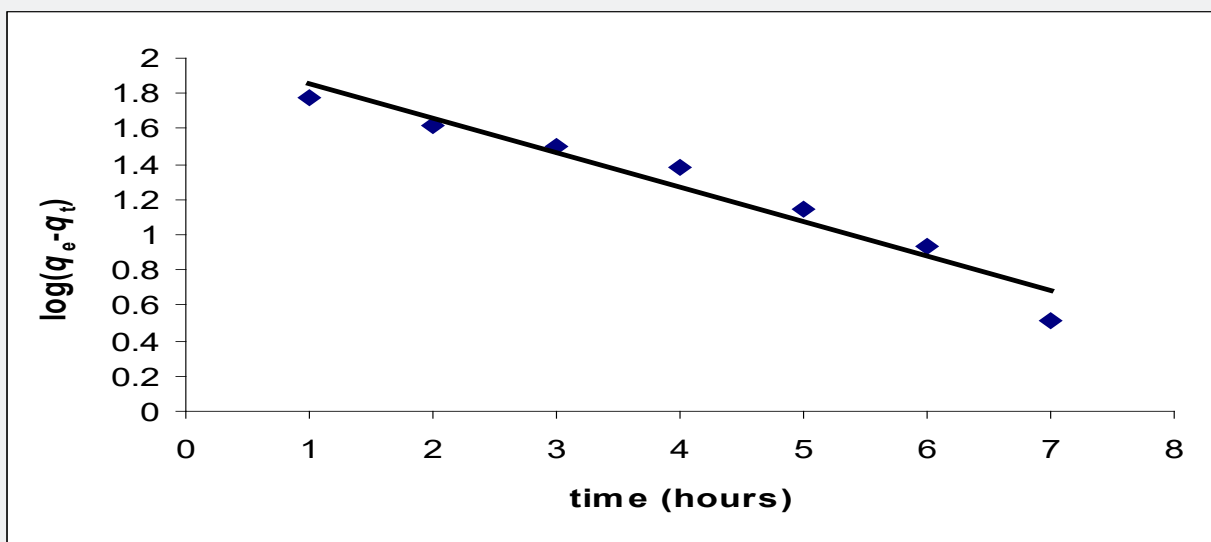


Figure 2: Pseudo first-order plot of phenol adsorption on GAC at 20°C.

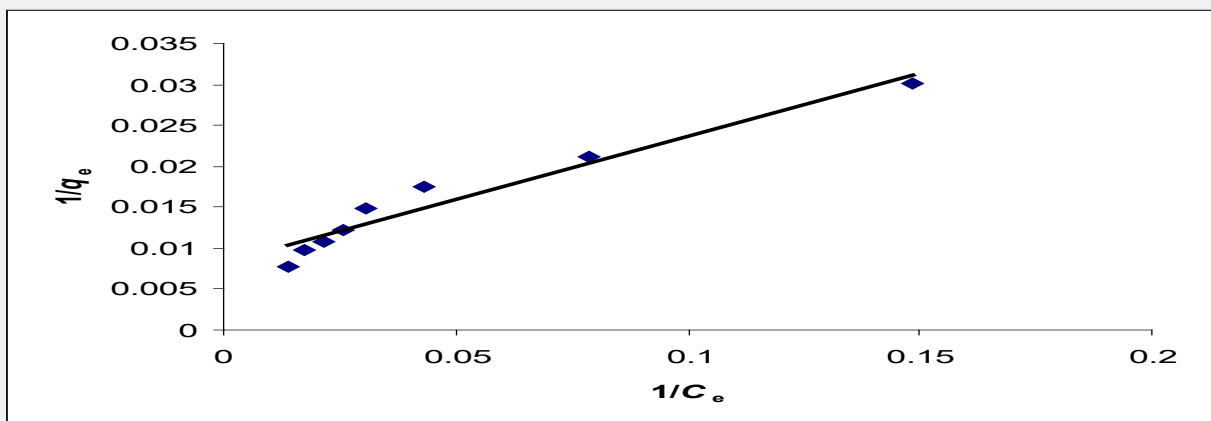


Figure 3: Langmuir adsorption isotherm of phenol on GAC at 20°C.

Micro wave regeneration of spent GAC

In this study, the regeneration efficiency is defined as the percentage amount of phenol adsorbed in the second adsorption process (after regeneration) divided by the amount of phenol adsorbed by fresh active carbon. Figure 4 indicates that: the regeneration efficiency reached about 90% at PL5 for 1.5 minute irradiation. It is worth to mention that; the regeneration efficiency

(was reached to 97-99%) as reported by [14,15]. however, they apply a different technique by using microwave for regeneration of dry GAC in inert atmosphere [24]. It is observed also as PL increases, the regeneration efficiency increases. It is suggested that phenol molecules suffer from excessive local heating which leads to quick thermal decomposition. By MW, heat energy readily transformed into dipole rotation some present groups or space charge polarization (as some free electrons are facially distributed

over carbon surface) [3,5]. This in turn heats adsorbate, phenol, and causes its decomposition. The decomposition is also practically highly suggested from experimental observations for each PL, the regeneration efficiency also increases with time. This gives more duration for microwave to decompose the phenol. For PL4 and PL5, regeneration efficiency was not determined

after 1.5 minute because of solution evaporation. Regeneration occurs within some minutes range. This is due to rapid heating of activated carbon by this technique. The measurement of supposed GAC-released phenol, by spectrometry, shows lower values of phenol compared to be expected than which is calculated from the next adsorption process [25,26].

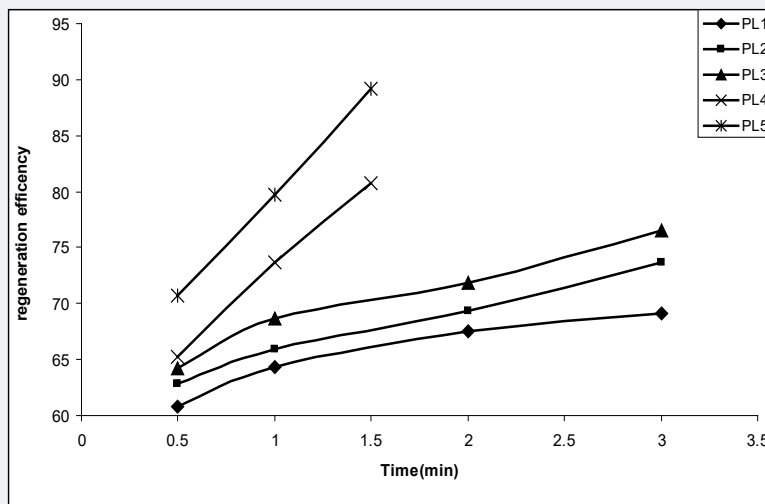


Figure 4: Regeneration efficiency of GAC saturated with phenol at different power levels.

Change of pH with MW treatment

Figure 5 shows the change in pH value of the solution, Δ pH, due to MW irradiation. This change in pH increases with PL, i.e. it shows more alkalinity. Also, increase of time, for each PL, has the same effect. The maximum Δ pH is 1.02 at PL5 and 1.5 minutes duration. The general trend indicated in this figure is matched, nearly, to the trend of regeneration efficiency. This change also suggests the decomposition mechanism mentioned earlier as this

change emanates from a decomposition product not from phenol. This is approved by auxiliary experiment shows that GAC/water system does not give significant change in pH under considered PL studied. On the other hand, phenol/water system does give almost the same Δ pH as for phenol/GAC/water system. This is present in Figure 6 that which shows the change in absorbance for phenol/water system after MW-irradiation at PL5 and time 1.5 minutes (Table 1).

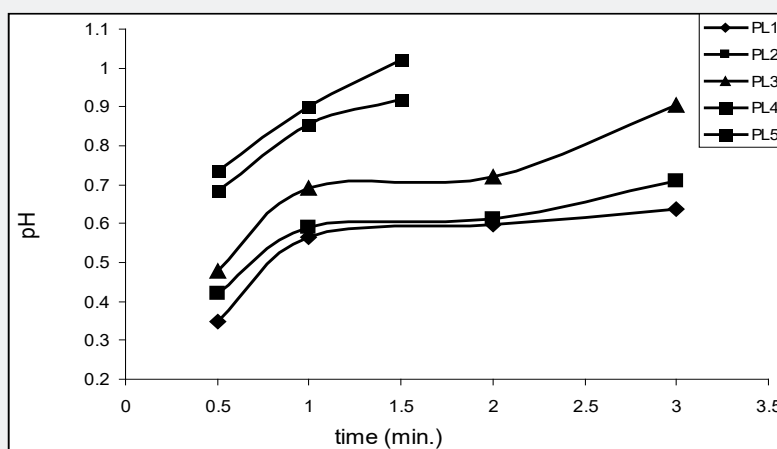


Figure 5: change in pH with microwave irradiation time and different power levels.

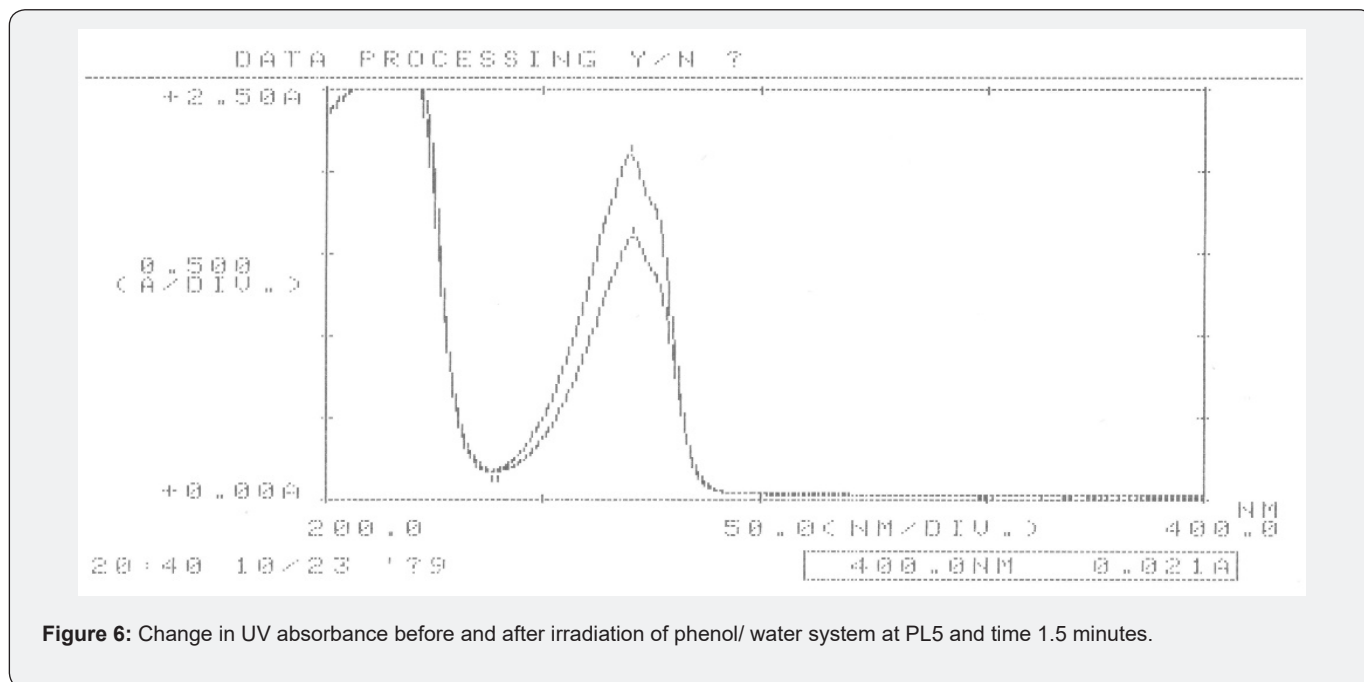


Figure 6: Change in UV absorbance before and after irradiation of phenol/ water system at PL5 and time 1.5 minutes.

Table 1: pH Change for phenol/ water system due to MW irradiation.

System	Power level	Time (min)	pH before	pH after	ΔpH
phenol/water	1	3	6.59	7.21	0.62
	2	3	6.54	7.26	0.72
	3	3	6.59	7.48	0.89
	4	1.5	6.56	7.49	0.93
	5	1.5	6.49	7.59	1.1

Regeneration water volume

From the data recorded in Table 2, it is clearly noticed that

the increase in volume from 30-100 ml of the water used in regeneration of GAC give only a slight increase of the Regeneration efficiency at PL5 and irradiation time 1.5 minutes.

Table 2: Volume change of the regeneration water with respect to regeneration efficiency.

Sample No.	Volume (ml)	PL	Time(min.)	Regeneration efficiency
1	30	5	1.5	88.02
2	50	5	1.5	88.99
3	70	5	1.5	89.48
4	100	5	1.5	89.80

Weight reduction of regenerated GAC

Figure 7 indicates that the weight reduction of GAC sample ranges from 2% to about 5% of the original weight after 5 regeneration cycles for PL5 and irradiation time 1.5 minutes. The weight reduction during regeneration can be noticed as a black dusty layer on the surface of regeneration solution, this may be due to wear effect between GAC particles during MW irradiation [27,28].

Characteristics of fresh and regenerated GAC

Brunauer-Emmett-Teller (BET) surface area analysis; indicates the specific surface area for Fresh GAC is 830 m²/g, exhausted 130 m²/g, regenerated 1 cycle 680 m²/g regenerated 5 cycles 300 m²/g. This successive decrease in the specific area may be due to the permanent adsorption of a certain amount of phenol inside the pores and this phenomenon indicates that a strong chemisorption mechanism and has a moderate sharing in the overall adsorption

process [29,30]. Infrared spectroscopy (IR spectra) of the 4 samples of GAC were analyzed as follow: the appearance of bands at the region about $3448.5\text{-}3421.5\text{ cm}^{-1}$ refers to (O-H) stretching vibrations in the hydroxyl, carboxylic or phenolic groups, since the location of the hydrogen-bonded OH groups is usually in the range of $3200\text{-}3650\text{ cm}^{-1}$ [16,17]. The band appears at 2854.5 cm^{-1} can be assigned to C-H group stretching, which usually appear at $2800\text{-}3000\text{ cm}^{-1}$ for aromatic and aliphatic C-H groups [17,18]. The bands present in the region between $1635.5\text{-}1627.8\text{ cm}^{-1}$ has

been observed by many earlier workers. It is known that olefinic C=C stretching band is located at 1650 cm^{-1} and the absorption maximum may shift toward lower wave numbers when the C=C bond is conjugated with another C=C bond, an aromatic nucleus, or a C=O bond. In fact, the C=C stretching absorption frequently occurs at approximately 1600 cm^{-1} for carbonaceous materials [2]. IR spectra of samples; fresh GAC, saturated GAC, and regenerated GAC at PL1, PL5) are shown in Figure 8.

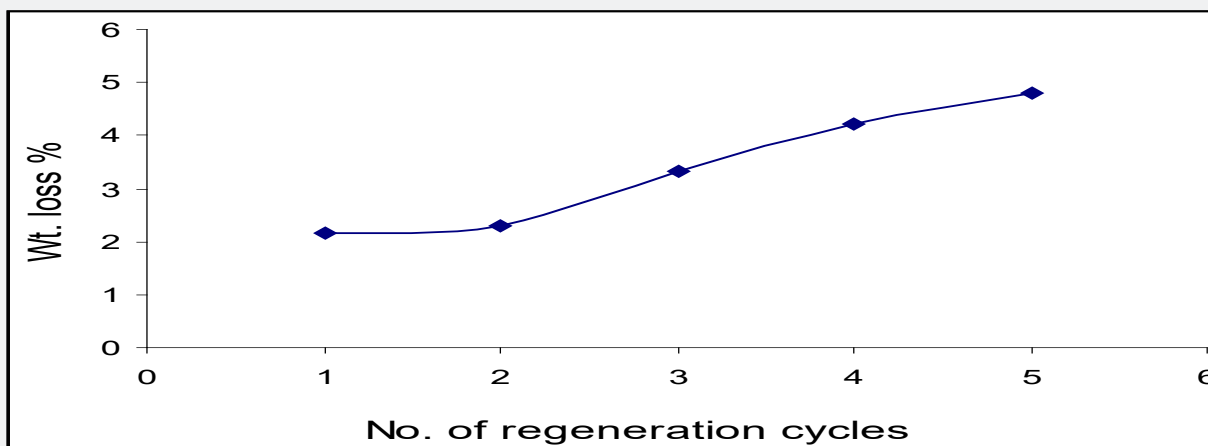


Figure 7: GAC Wt. loss% with respect to No. of regeneration cycles at PL5 and irradiation time 1.5 minutes.

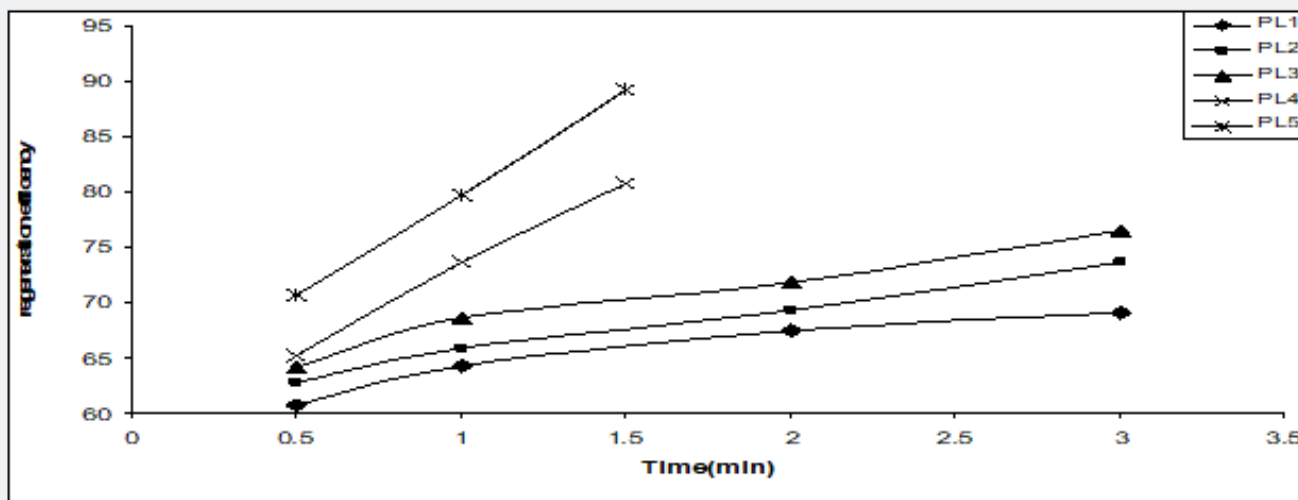


Figure A: Regeneration efficiency of GAC saturated with phenol at different power levels.

Conclusion

In this work, a mathematical model has been developed to measure and validate the maximum regeneration efficiency of the GAC adsorption capacity. the regeneration of GAC in aqueous media using domestic microwave set under different operating conditions is investigated to evaluate its adsorption efficiency after successive regeneration cycles and to ensure its practical feasibility.

Domestic microwave oven was used to study the regeneration of GAC after saturation with phenol in aqueous solution. The pre-step of adsorption revealed that pseudo first-order is a good model to represent kinetic adsorption of phenol on GAC. The adsorption rate constant was calculated and equal to be $k = 0.454\text{ min}^{-1}$. Langmuir adsorption isotherm at 20°C gives maximum adsorption capacity as $Q_0 = 121.5\text{ mg/g}$. For regeneration of GAC by micro

wave, it is indicated that the regeneration efficiency increases with power level and time up to certain limit. It is clear that, the increase in volume of the water used in regeneration of GAC give a slight increase of the regeneration efficiency. The GAC weight loss due to regeneration cycles was found to be in the range of 3-5%. BET analysis elucidates that the specific surface area decrease with the regeneration cycles. We concluded that microwave recovery of saturated activated carbon loaded with phenol 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. Briefly, the system is interesting for its simplicity, effectiveness, speed, and economic utility for treating and recovering volatile vapor pollutants. We expected that; this approach could be used in ventilation systems for the treatment of volatile organic compounds in work environments

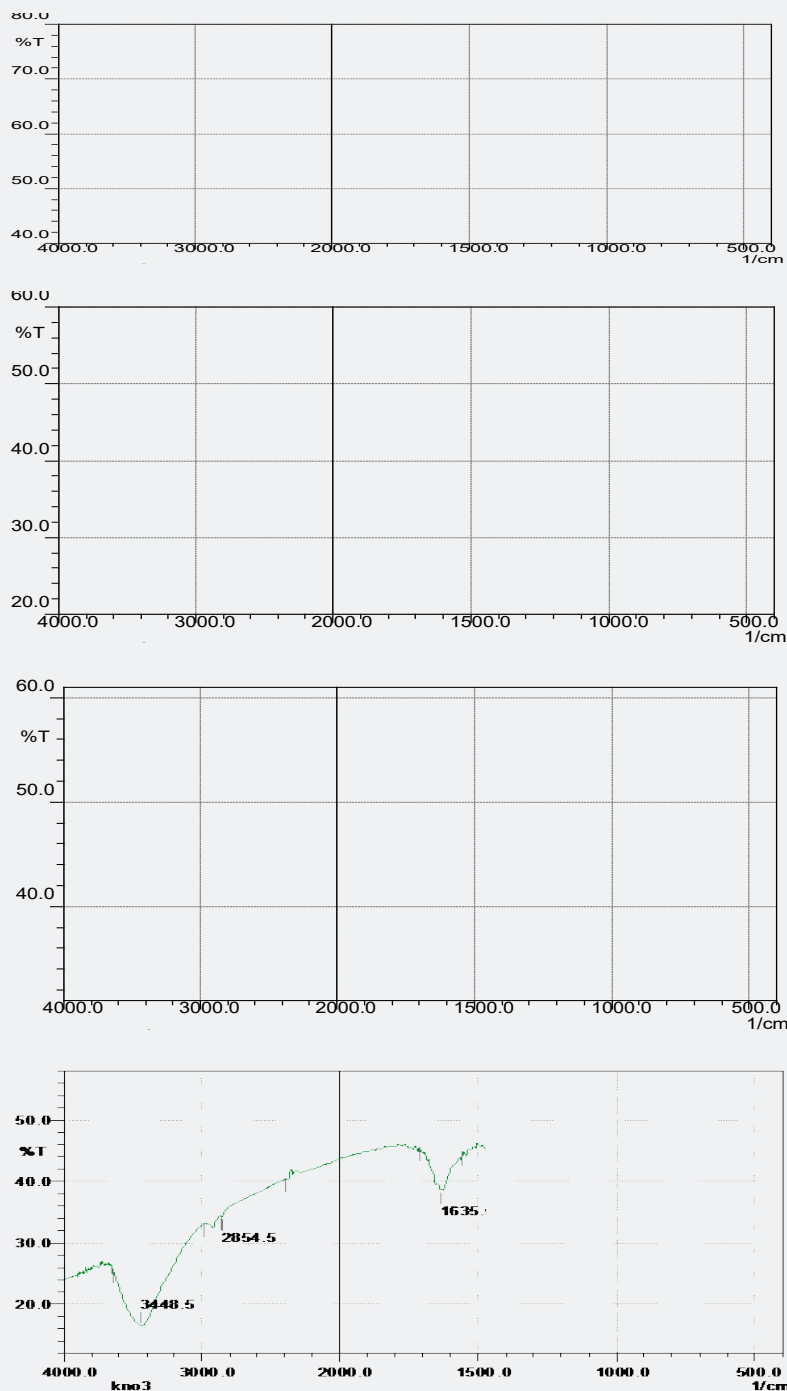


Figure 8: IR spectra for (A) Fresh GAC, (B) GAC saturated with Phenol, (C) GAC Regenerated at power level [1] and (D) GAC Regenerated at power level [5].

Recommendation

It is recommended to: Apply of the regeneration technique of spent active carbon used in water treatment operations by Domestic microwave oven because: the adsorption by activated carbon is best and most frequently used method ;and this due to the following reasons: -

- a. It can remove both organic as well as inorganic constituents even at very low concentrations.
- b. It is relatively easy and safe to operate.
- c. Both batch and continuous equipment can be used.
- d. No sludge formation and frequently the adsorbent can be regenerated and used again.
- e. Moreover, the process is economical because it requires low capital cost and there are abundant low-cost materials available to produce active carbon from it.
- f. MW regeneration in aqueous medium appears to be a real promising technique and thus a wide spread and great progress in this area can be expected in future.

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