

Preparation Of Activated Carbon From Fuel Oil Wastes For Removal Of Ortho-Xylene From Aqueous Solution By New Circulating System

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ABSTRACT

This investigation was concern to study the removal of o-xylene pollutants from aqueous solution using activated carbon synthesized from fuel oil wastes. Characterizations of activated carbon were also discussed. The thermodynamic parameters were calculated using adsorption process on an active carbon in the o-xylene solutions concentration of (50,200,300) ppm at four different temperatures and periods of time. Equilibrium adsorption study was done by using a new circulating system, which indicated that synthetic activated carbon fit very well with the Freundlich and Temkin isotherm models. The specific adsorption percentage of o-xylene was highly affected by addition of activator and decreasing with temperature compared to that of control sample. It has been found that the adsorption rate was increased by increasing the o-xylene concentrations, and these values indicated that the o-xylene adsorption onto active carbon was spontaneous and exothermic in nature. The o-xylene adsorption onto active carbon was fitted through the pseudo-first order model with high correlation coefficient values.

Key words: Activated carbon; Xylene; Adsorption; Pollution; Kinetics

Introduction

In recent years, water pollution is becoming a most serious problem due to technological activities, especially in developing countries [1]. Xylene is the important organic compound which is widely used as a solvent during a variety of many industries and commercial processes, such as chemical production, printing, paint, synthetic resin, rubber, mobile emission and petroleum derivatives [2,3]. So, environmental pollution which can be found in soil, water and air is highly affected by this pollutant. It penetrates human organs through ingestion, inhalation or absorption, causing serious diseases [4]. Therefore, the removal of these pollutants is an important target for environmental treatment systems. Since xylene is a volatile organic compound, the control technologies, such as condensation, adsorption, extraction, membrane separation, have been commonly utilized to control its volatilization. However, these technologies require high operating costs or a double disposal treatment.

Activated carbon is one of the mostly used in adsorption for removal of pollutants from aqueous solutions and gases [5]. Therefore, the demand of this method was growing widely over the years and

many investigations have focused on removal of low concentration of volatile organic materials [6, 7, 8]. Many factors showed a high potential effect on adsorptive capacity of activated carbon [9, 10]. These parameters are including a surface functionalization such as surface functional groups and ash content, while a texture of activated carbon such as surface area and pore size distribution showed a real effect. Meanwhile, the characteristics of adsorbate such as pK_a , polarity and molecular mass and size pointed out that pH, temperature, adsorbate concentration, presence of competitive compounds and polarity of solvent are the most important parameters in adsorption processes. However, activated carbon synthesis was followed by physical and chemical aspects which include many techniques for activator processes [11-17].

The main goal of this investigation is concern on the removal of o-xylene pollution from aqueous solutions using activated carbon synthesis from fuel oil wastes as adsorbent.

Materials And Methods

2.1. Vacuum distillation of fuel oil residue:

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The samples of fuel oil residue were collected from the power plant. The sample were placed under low pressure of 20 mmHg, heated at a range of 280–325 °C for the purpose of removing volatile substances and get rid of additional quantity of liquid ingredients in waste oil and to ensure an increase of the density of carbon from fuel oil wastes. The refining operation was done using a laboratory apparatus as shown in Fig. (1). Distillation process continues until the mass becomes highly dense, and just like solids.

2.2. Preparation of activated carbon:

A certain weight of the waste of the fuel oil was taken and soaks with 20% anhydrous zinc chloride ratio of 1:2. The mixture was heated, gradually with continuous stirring for two hours by homogenizer and then left stand for (48) hours.

The resulting material burned at a temperature of 400-500 °C in the muffle furnace under atmospheric pressure for two hours and then the sample was cooled to room temperature. The sample washed with 0.1 M of hydrochloric acid, filtered and then washed with distilled water several times in order to eliminate the content of inorganic materials in the resulting carbon samples. The active carbon was dried at 110-120 °C for 24 hours and then cooled, grinded and passed through sieves of 16-20 mesh and stored at desiccators.

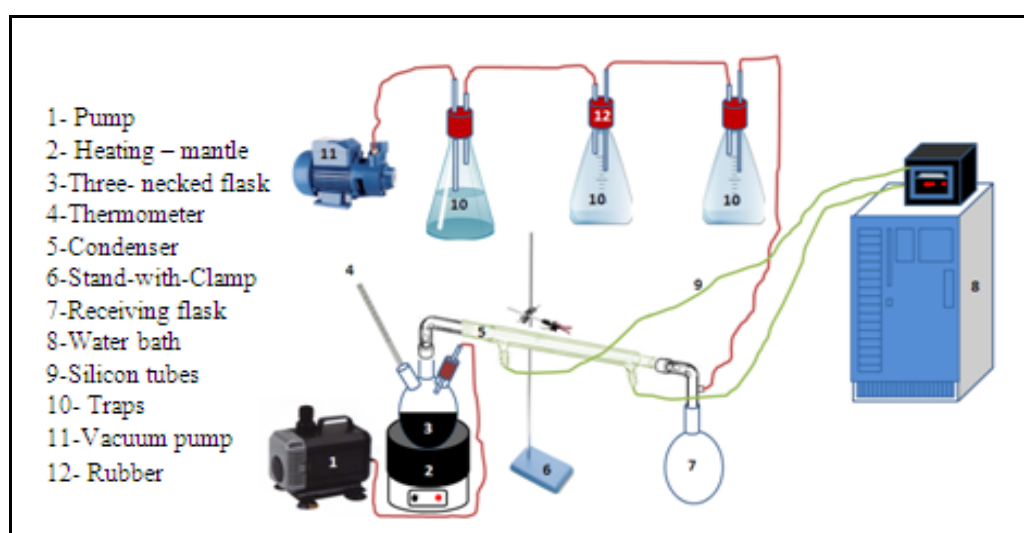


Fig. 1: Schematic diagram of vacuum distillation for the preparation of carbon from heavy fuel oil residues

2.3. Measurements:

The specific surface area for the samples of carbon was recorded by using Adsorption isotherm method (B.E.T isotherm). The determination of surface area were performed by adsorption isotherm of nitrogen gas-196 °C in liquid nitrogen followed by desorption isotherm for adsorbed gas on surface of carbon. This process called (Adsorption-Desorption Isotherm).

The pH value of prepared activated carbon was determined by mixing 1.0 g of sample with 10 ml distilled water were carried out by shaking for 30 min , the solution was filtrated at room temperature and then measured by pH-meter. The ash content was transferred estimated by 1.0 g of activated carbon to crucible heated in muffle furnaces at 1000-1100°C for three hours, cooled in desiccators theremaining weight was supposed to be ash contain then the percentage was calculated [18]. Moisture content was measured by heat 1.0 g of prepared activated carbon in an oven at 150 °C for four hours then

cooled in a desiccators, the difference in weight before and after heating was calculated [19]. The density of the prepared activated carbon was determined by weighing 10 cm³ of the carbon [20].

2.4. Adsorption studies:

Adsorption process was done by using a new circulating system as described in Fig. (2). Five grams of active carbon sample have been placed in container (no.6) fitted to a three-necks flask (no.5) containing 200 ml of o-xylene solution with three different concentration 50,200,300 ppm at temperatures of 283,293.313 and 333 K for 120 minutes using a water bath (no.3). The equilibrium time was achieved by a recycling solution using a peristaltic pump (no.4).To minimize the evaporation and contamination, a cooling system (no.7) and a trap (no.2) were in contact along with a cycling process. Samples of 4 ml were taken for each 10 min from the valve (no.8).

UV-Vis spectrophotometer has been used to measure the absorbance for o-xylene at equilibrated solutions at certain wavelength (215 nm). The quantity of adsorbate was calculated by using the following formula:

$$Q_e = V_{\text{Sol}}(C_o - C_e)/M \quad (1)$$

Where:

Q_e = Quantity of adsorbate (mg/g).

V_{Sol} = Total volume of adsorbate solution (L)

C_o = Initial concentration of adsorbate solution (mg/L)

C_e = Concentration of adsorbate solution at equilibrium (mg/L)

M = Weight of adsorbate (g)

While the percentage removal of o-xylene and equilibrium adsorption q_e (mg/g) was uptake, using the formula.

$$\% \text{ adsorption Efficiency} = (C_o - C_e) / C_o \times 100 \quad (2)$$

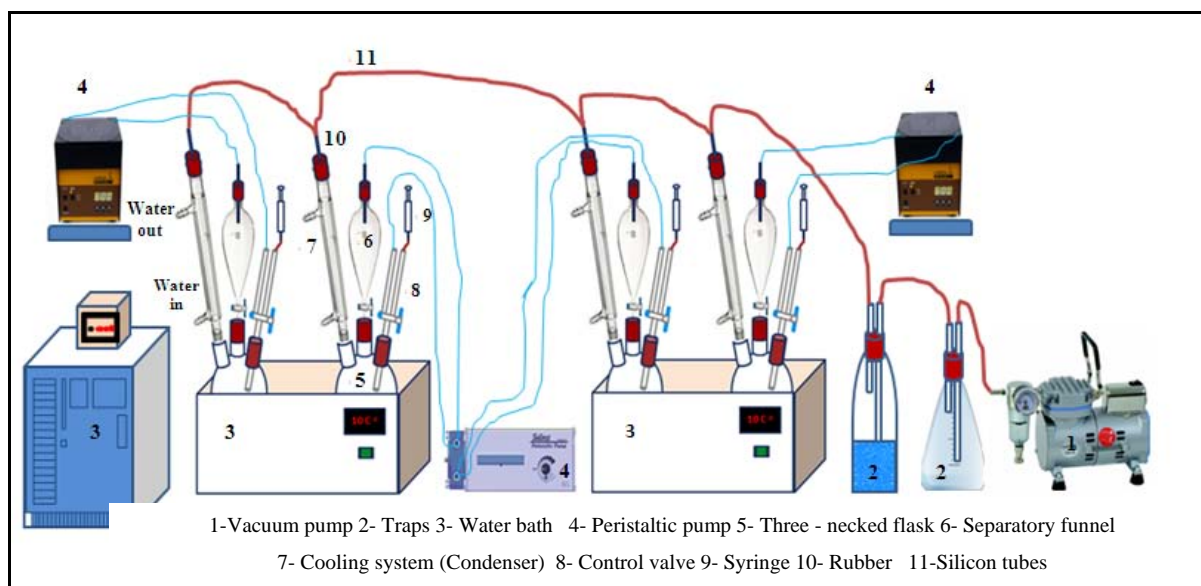


Fig. 2: Schematic diagram of new circulation system for equilibrium adsorption

Results and Discussions

3.1. Physical Properties:

The B.E.T isotherm method was used including adsorption of nitrogen gas to determine specific surface area due to large number of used freeze materials in isotherms processes which have uniform shapes and porous nature such as activated carbon, thus the surface area cannot be determined straight forward but can be determined using isotherm B.E.T with multiple layers [21]. The surface areas values of the resulting carbon prepared from fuel oil waste impregnated with concentrations of 20% $ZnCl_2$ solution. The results showed that the values of surface area of prepared activated carbon equal to 399.75 m^2/gm compared to that of non-active carbon 72.32 m^2/gm . These results indicated that activated carbon plays an important role in increasing the surface area.

The average value of pH of the prepared activated carbon was 5.53 which is more acidic compared to that of non-active 6.15. The surface acidity was due to the presence of carbon-oxygen surface chemical structures that have been postulated as carboxyl's and lactones. The activated process has

increased the surface area and porosity as well as the surface basicity of activated carbon.

The prepared activated carbon has low ash content 1.23% compared to that of raw sample which indicates that the activated carbon has high purity. High ash content is undesirable for activated carbon since it reduces the mechanical strength of carbon and affects adsorptive capacity [22].

It is known that permissible range of moisture content of activated carbon should be less than 10% [23]. However, the moisture content of produced activated carbon was 1.43% which indicates a very good result.

The density of activated carbon derived from fueloil waste source is 0.538 g/cm^3 . Therefore the smaller the density, the greater the pores and accordingly the higher surface area available with active carbon.

3.2. Equilibrium Adsorption:

The equilibrium adsorption isotherms are of fundamental importance in the design of adsorption systems [24]. The isotherm of the o-xylene adsorption by synthetic activated carbon was represented by applying the Langmuir, Freundlich

and Temkin adsorption models. It was found that the adsorption process on the synthetic activated carbon fit very well with the Freundlich and Temkin isotherm models and poor with Langmuir isotherm model table 1.

The Freundlich isotherm is expressed as

$$Q_e = K_f C_e^{1/n} \quad (3)$$

This isotherm is usually used in special cases for heterogeneous surface energy and it is characterized by the heterogeneity factor $1/n$. Q_e is the equilibrium value of o-xylene adsorbed per unit weight of synthetic adsorbate, i.e. a liquid-phase sorbate concentration occurred at equilibrium, K_f as the Freundlich constant. The Freundlich equation was linearized by taking logarithms on each side of the equation and gives a straight line as shown in equation 4.

$$\log Q_e = \log K_f + 1/n \log C_e \quad (4)$$

Freundlich factors and relations are shown in table 1 with high correlation coefficients (R^2). The Freundlich model assumes that the uptake of any adsorbate occurs on a heterogeneous surface by multilayer adsorption and that the amount of

adsorbate increases infinitely with increasing the concentration. These assumptions led to conclude that synthetic activated carbon takes up o-xylene on a heterogeneous surface by multilayer adsorption. However, the Langmuir isotherm is expressed as

$$Q_e = \frac{Q_o C_e}{K_L + C_e} \quad (5)$$

Equation 5 is rearranged and gives a straight line as shown in equation 6.

$$C_e/Q_e = (1/K_L Q_o) + (a/Q_o) C_e \quad (6)$$

Where Q_o is the maximum amount of adsorption corresponding to complete monolayer coverage and K_L is the Langmuir constant (Table 1).

The linear form of Temkin isotherm is expressed as.

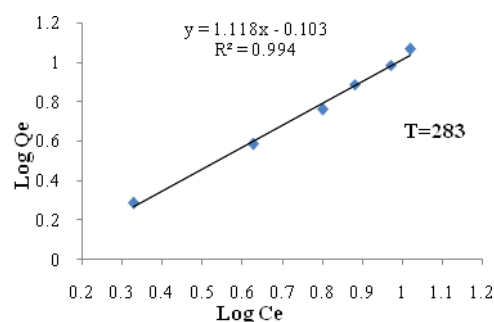
$$q_e = B \ln K + B \ln C_e \quad (7)$$

The adsorption data were analyzed according to eq.7. A plot of q_e versus $\ln C_e$ enables the determinations of the isotherm constants K and B (Table 1).

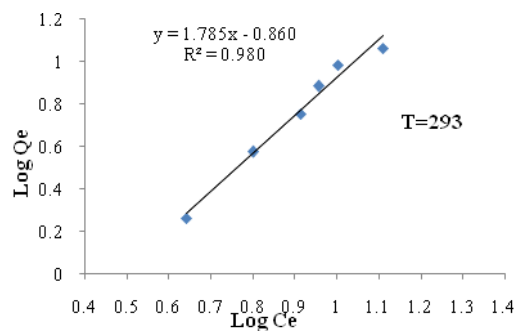
Table 1: The values of Langmuir, Freundlich and Temkin constants at different temperatures in Figs. 3,4,5,6.

	T K	Langmuir constants			Freundlich constants			Temkin constants		
		R^2	a	K_L	R^2	n	K_f	R^2	B	A
Active carbon	283	0.8329	-0.0211	0.8332	0.9943	0.8943	0.7885	0.9005	5.7364	0.5455
	293	0.7781	-0.0549	0.3618	0.9807	0.5602	0.1379	0.9486	9.3466	0.2546
	313	0.6881	-0.0511	0.2992	0.9596	0.5492	0.1009	0.9491	9.5280	0.2204
	333	0.4970	-0.0436	0.2931	0.9485	0.5495	0.0840	0.9154	9.7481	0.1951
Non-active carbon	283	0.9789	-0.0179	0.0115	0.9519	0.2208	1.21×10^{-7}	0.7329	14.435	0.0315
	293	0.7311	-0.0174	0.0060	0.9607	0.1561	5.21×10^{-11}	0.9089	19.671	0.0263
	313	0.2666	-0.0101	0.0073	0.8298	0.3415	1.40×10^{-5}	0.9213	7.763	0.0268
	333	0.3627	-0.0104	0.0057	0.8064	0.2771	5.75×10^{-7}	0.9684	8.4642	0.0240

The relations of equilibrium values of $\log Q_e$ vs. $\log C_e$ and Q_e vs. $\ln C_e$ for o-xylene adsorption isotherms using synthetic carbon powder at different temperature are shown in Figures (3,4,5 and 6).



(A)



(B)

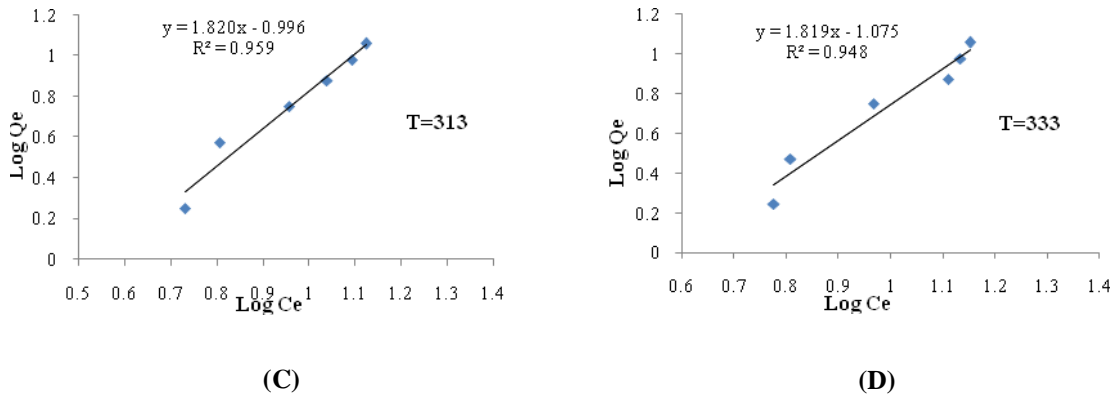


Fig. 3: Freundlich linear relationship for the adsorption of o-xylene solutions on an active carbon at different temperatures

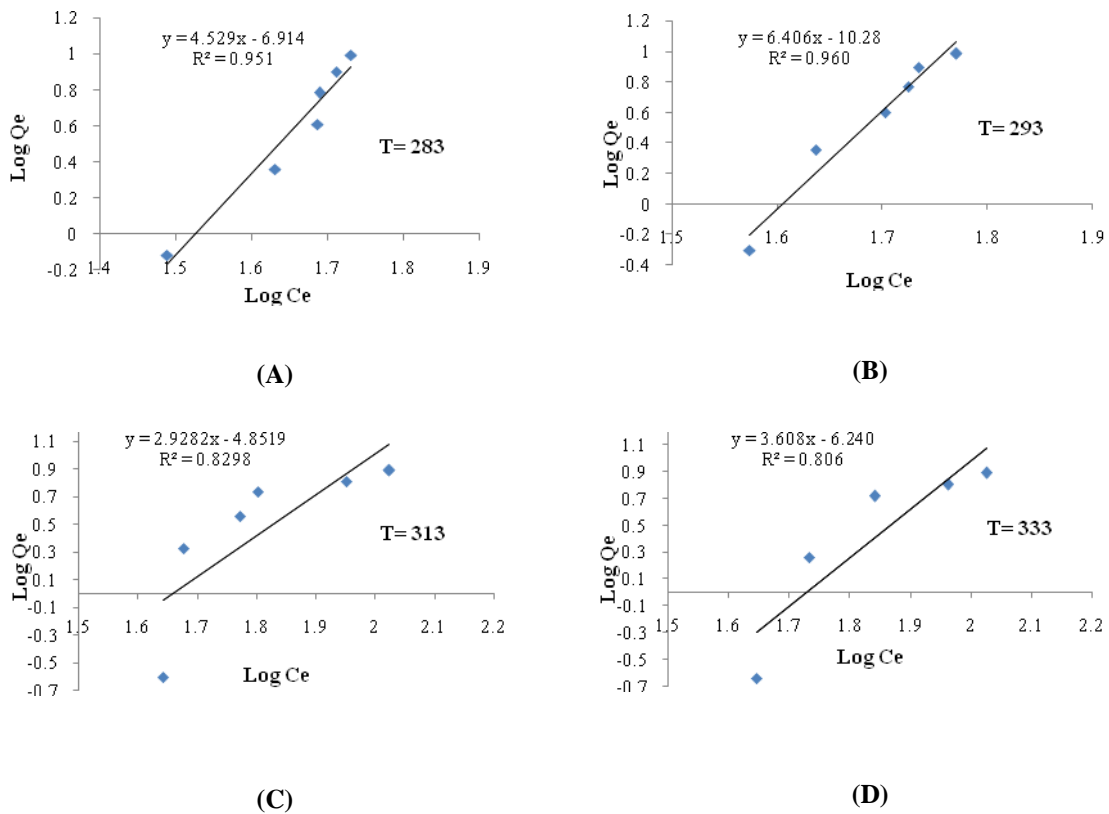
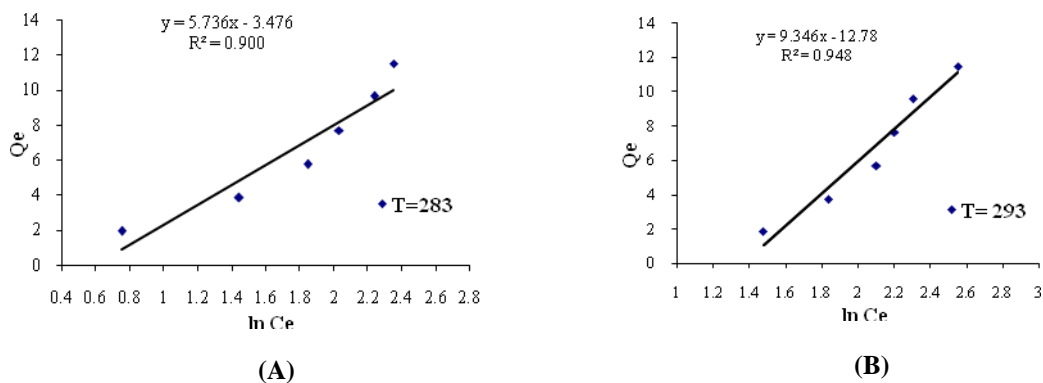


Fig. 4: Freundlich linear relationship for the adsorption of o-xylene solutions on non-active carbon at different temperatures



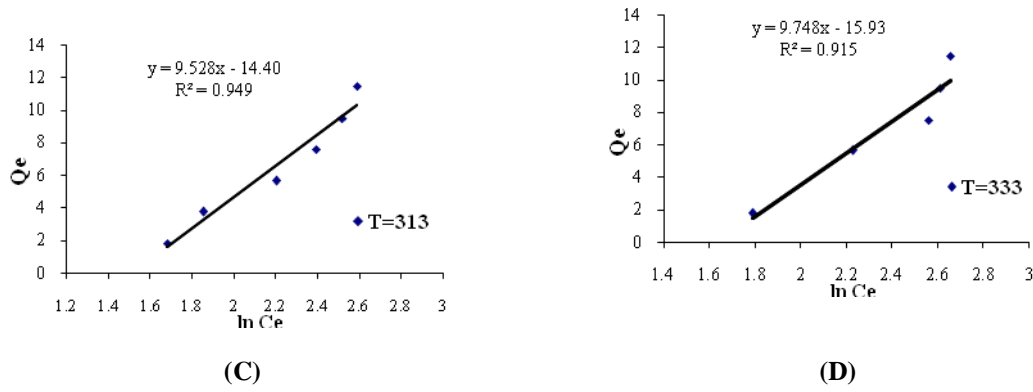


Fig. 5: The linear relationship of Temkin isotherm for the adsorption of o-xylene solutions on an active carbon at different temperatures

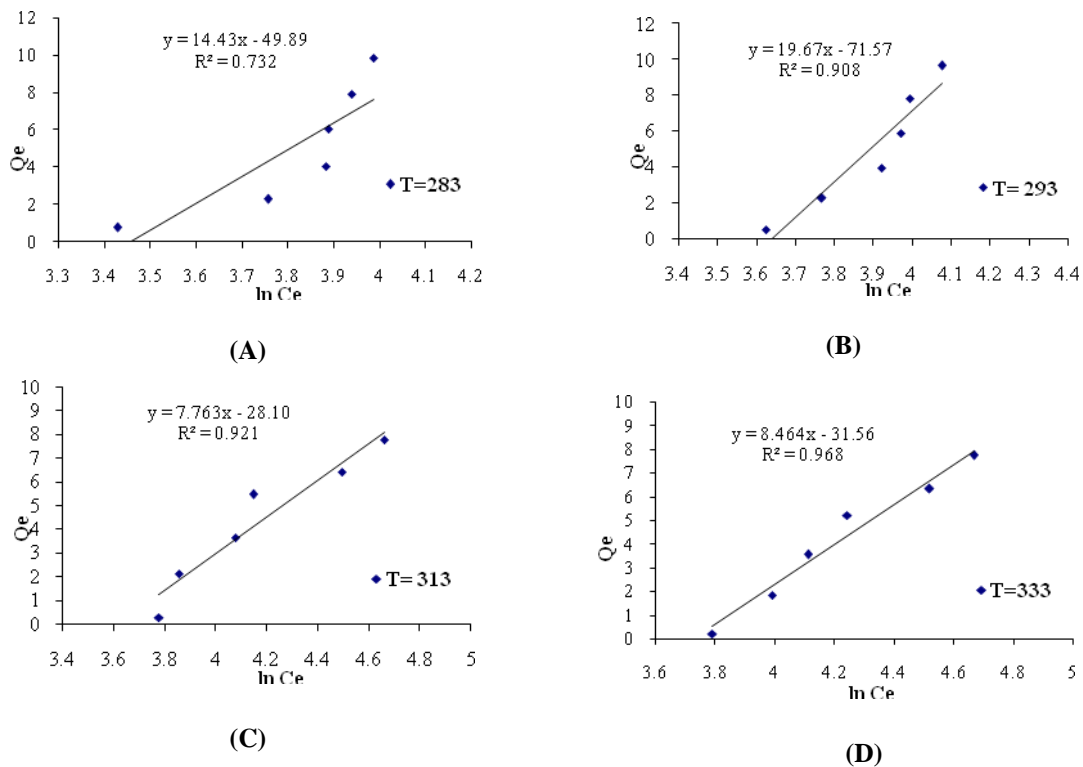


Fig. 6: The linear relationship of Temkin isotherm for the adsorption of o-xylene solutions on non-active carbon at different temperatures

The rate adsorption of the o-xylene increased with increasing concentration while the adsorption percentage of o-xylene decreases as temperature increases, whereas the adsorption process is an exothermic (Tables 2,3 and 4).It can be seen that adsorption capacity increases until an equilibrium concentration is obtained. About 96.51% reduction in concentration of o-xylene was obtained at 300 ppm and temperature of 283 K. Hence, it is clearly proved that o-xylene adsorption by synthetic activated carbon agrees fair enough with the Freundlich and Temkin adsorption models. Less fitting was occurred with Langmuir isotherm model. The correlation coefficients are very high throughout the

experimental range of o-xylene concentrations studied.

3.3. Thermodynamic parameters:

The thermodynamic parameter of ΔG° for adsorption processes is determined by using following equation [25].

$$\Delta G^\circ = -RT \ln K \tag{8}$$

Where K is the thermodynamic equilibrium constant. The parameters of ΔH° and ΔS° , and the temperature effect on thermodynamic functions are determined by:

$$d \ln K/dt = \Delta H^0/RT^2 \quad (9) \quad \text{Log } K = \Delta S^0/2.303 R - \Delta H^0/2.303 RT \quad (10)$$

Table 2: The adsorption percentage of *o*-xylene with concentration 50 mg/L at different temperature

Temp. Time	adsorption efficiency %							
	283K		293K		313K		333K	
	Active carbon	Non-active carbon	Active carbon	Non-active carbon	Active carbon	Non-active carbon	Active carbon	Non-active carbon
0	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
10	84.29	17.24	74.57	10.67	71.14	7.52	68.76	6.10
20	85.24	21.81	79.81	11.71	78.57	8.57	76.10	6.67
30	87.06	25.52	82.38	18.57	80.57	9.33	77.43	7.33
40	87.13	26.10	84.19	21.81	83.62	10.19	81.62	8.48
50	87.87	29.71	86.25	24.19	86.00	10.48	84.57	9.24
60	90.11	33.52	87.01	25.14	87.01	10.76	86.50	9.62
70	90.39	36.48	87.18	25.43	87.10	12.10	87.00	10.29
80	92.93	36.67	87.24	25.52	87.31	12.38	87.14	10.76
90	93.21	37.62	87.80	25.81	87.38	12.57	87.53	10.95
100	95.46	38.00	90.39	25.05	87.42	12.48	87.80	11.43
110	95.46	38.48	90.96	25.05	89.27	12.38	88.03	11.81
120	95.75	38.38	91.24	24.95	89.27	12.38	88.03	11.52

Table 3: The adsorption percentage of *o*-xylene with concentration 200 mg/L at different temperature

Temp. Time	adsorption efficiency %							
	283K		293K		313K		333K	
	Active carbon	Non-active carbon	Active carbon	Non-active carbon	Active carbon	Non-active carbon	Active carbon	Non-active carbon
0	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
10	73.43	41.60	72.67	45.48	72.07	39.21	69.17	38.17
20	74.14	51.74	73.83	49.83	72.62	42.02	72.19	41.05
30	79.43	55.81	78.95	51.12	77.26	43.33	76.36	42.29
40	83.24	60.74	81.86	56.93	79.74	45.21	78.26	44.21
50	86.71	62.00	86.57	59.57	85.60	45.74	83.52	45.21
60	89.36	66.91	88.86	62.45	85.74	46.74	84.83	46.26
70	91.45	69.45	90.45	63.98	89.93	51.41	89.26	50.19
80	92.74	72.14	92.43	66.88	91.76	52.21	89.83	51.48
90	94.79	72.69	93.60	68.91	93.00	57.52	92.45	55.14
100	95.67	73.19	94.62	71.21	94.50	62.24	93.50	57.26
110	96.19	75.52	95.45	73.46	94.52	68.29	93.55	65.17
120	96.19	75.57	95.48	73.46	94.52	68.29	93.55	65.24

Table 4: The adsorption percentage of *o*-xylene with concentration 300 mg/L at different temperatures

Temp. Time	adsorption efficiency %							
	283K		293K		313K		333K	
	Active carbon	Non-active carbon	Active carbon	Non-active carbon	Active carbon	Non-active carbon	Active carbon	Non-active carbon
0	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
10	77.95	58.87	77.57	58.50	77.35	58.05	73.76	57.73
20	80.95	60.27	78.71	59.25	78.38	58.51	75.87	58.27
30	84.13	63.95	82.51	59.65	79.29	59.32	78.24	59.24
40	86.87	67.33	85.83	64.05	81.54	59.83	80.02	59.95
50	89.84	71.03	87.54	66.76	84.62	60.81	82.22	61.21
60	91.97	75.89	89.60	69.86	86.19	61.54	84.57	61.79
70	93.06	77.83	90.78	73.14	87.40	62.56	85.81	62.90
80	94.64	79.51	91.84	76.67	88.44	63.08	88.33	63.29
90	95.43	80.22	93.22	77.73	89.94	63.48	89.51	63.75
100	96.10	80.87	94.57	79.78	93.24	64.02	93.00	63.95
110	96.51	82.00	95.98	80.30	95.54	64.69	95.29	64.46
120	96.51	82.05	95.98	80.32	95.56	64.76	95.27	64.52

The ΔH^0 and ΔS^0 values were calculated from slope and intercept of the linear plot, of $\log K$ vs. $1/T$ as shown in Fig. (7). The corresponding values of thermodynamic parameters are presented in table 5. The negative values of ΔG^0 indicated that the *o*-xylene adsorption process is spontaneous and feasible. The negative value of ΔH^0 showed the adsorption process is an exothermic in nature while the negative ΔS^0 indicated the decrease in

randomness at the solid/liquid interface during adsorption of *o*-xylene on carbon.

3.4. Adsorption kinetics:

Adsorption kinetic models can be useful to determine the mechanism of adsorption and the efficiency of the adsorbents for the removal of pollutants. In this study, the adsorption data of *o*-xylene by active carbon was fitted through kinetic

model including pseudo-first order as shown in equation 11 [26].

$$\ln(q_e - q_t) = \ln q_e - k_1 t$$

Where q_e (mg/g) is the amount of o-xylene at equilibrium time while q_t (mg/g) is the amount of o-

xylene at any time (t), and k (min^{-1}) is the pseudo-first order rate constant. k and q_e were determined from the slope and intercept of the linear plot of $\ln(q_e - q_t)$ against t, respectively (Fig.8). (11)

Table 5: Thermodynamic functions of o-xylene adsorption process in Fig. 7.

Samples	Initial Conc.(mg/l)	T(K)	K	ΔH°		ΔS°		ΔG°
				kJ.mol^{-1}	$\text{J.mol}^{-1}.\text{K}^{-1}$	kJ.mol^{-1}	kJ.mol^{-1}	
Active carbon	200	283	25.250	-8.505	-3.208	-3.676	-7.597	
	200	293	21.104					
	200	313	17.261					
	200	333	14.450					
non-Active carbon	200	283	3.094	-8.046	-19.042	-19.000	-2.657	
	200	293	2.767					
	200	313	2.153					
	200	333	1.877					

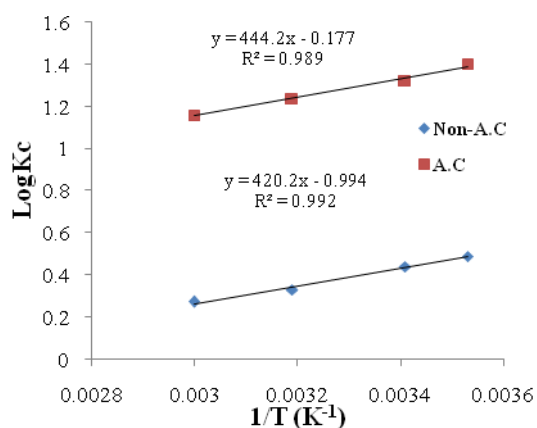


Fig. 7: The plot of Log Kc vs. 1/T with active carbon and Non-active carbon

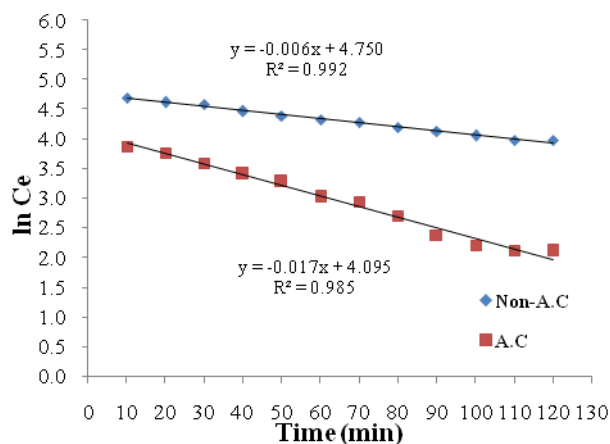


Fig. 8: Relationship between Ln (Ce) vs. Time (min) for o-xylene solutions at temperature 283K^0 and Con. (200 mg/L) on active carbon and Non- active carbon.

Conclusions:

The addition of activated carbon powder as an absorbent to aqueous sample of o-xylene reduces the concentration dramatically. About a 96.51 % reduction in concentration of o-xylene was obtained using 5.0 g of synthetic carbon powder for 200 ml of o-xylene, at concentrations of 50, 200 and 300 ppm. The process of adsorption on carbon powder fitted the Freundlich and Temkin isotherm equilibrium adsorption models with very high correlation coefficients.

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