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Furfural Removal from Refinery Wastewater by Adsorption on Commercial Activated Carbon

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Abstract

Furfural is a toxic aromatic aldehyde that can cause severe environmental problems, especially the wastewater discharge from petroleum refinery units. The effect of adsorption variables, which include time (30-240) min, initial concentration of furfural (40-5080) mg/l, and amount of adsorbent material (10, 15, and 20 g\250 ml). The commercial activated carbon was investigated in a batch process in order to obtain the maximum furfural removal from wastewater. The results obtained from the experimental investigations showed that furfural removal increases with the increasing adsorbent material and decreases with increasing furfural concentration. Best Furfural removal efficiency was obtained at pH value equal 7.0, agitation speed 150 rpm, contact time 240 minutes. Chemical adsorption takes place when increasing temperature adsorption capacity increases. The best solvent used to regenerate activated carbon was ethanol 50 wt%. Ethanol has been used in industrial applications due to its low cost and relatively eco-friendly solvent. The laboratory experiments were done, and the sump drum O3D4, D-303 site was chosen to execute the project. The maximum contamination in the furfural was 100 ppm. The dimension of the polluted area in the sump drum (O3D4, D-303) was 20.3, 45 m2. The amount of activated carbon used 327, 726.3 kg.

Keywords: Furfural; activated carbon; waste water; adsorption; batch method.

إزالة الفورفورال من المياه الصناعية للمصفى عن طريق الامتزاز على الكربون المنشط التجاري

الخلاصة

فورفورال هو ألدهيد عطري سام يمكن أن يسبب مشكلة بيئية خطيرة وخاصة مياه الصرف الصحي من وحدات تكرير البترول. تم دراسة تأثير متغير الامتزاز الذي يشمل الوقت (30-240) دقيقة ، والتركيز الأولي للفورفورال (40-5080) جزء في المليون، وكمية المادة الممتزة (10، 15، 20 جم \ 250 مل) للكربون المنشط التجاري في دراسة واحدة. عملية

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دفعية من أجل الحصول على أقصى قدر من إزالة فورفورال من مياه الصرف الصحي. النتائج التي تم الحصول عليها من البحث التجريبي أظهرت أن النسبة المئوية لإزالة فورفورال تزداد مع زيادة كمية المادة الممتزة حتى تصل إلى أقصى قيمة ثم تصل إلى قيمة ثابتة تقريبًا ، وتتناقص مع زيادة تركيز فورفورال. والتي حصل عليها عند درجة حموضة تساوي 7.0 ، سرعة التحريك 150 في الدقيقة ، زمن التحريك 240 دقيقة. يحدث الامتزاز الكيميائي عند زيادة قدرة امتصاص درجة الحرارة. أفضل مذيب يستخدم لتجديد الكربون المنشط هو الإيثانول 50٪ بالوزن. لأن الإيثانول له مزايا في تطبيقات الصناعة نظرًا لتكلفته المنخفضة ومدى ملاءمته للبيئة النسبية. يتم إجراء مقياس التجارب المعملية من أجل تنفيذ المشروع في موقع أسطوانة الحوض (O3D4، D-303). الحد الأقصى للتلوث في فورفورال يصل إلى (100 جزء في المليون). تصل مساحة التلوث في حوض الحوض (O3D4، D-303) إلى (20.3، 45 م 3) على التوالي. ستستخدم كمية الكربون المنشط (327 ، 726.3 كجم).

1. Introduction

Petroleum refineries are confrontation pollution problems linked with the accidental losses of furfural. This forms an economical loss as well as environmental hazards. Furfural is a toxic aromatic aldehyde with the chemical formula C_4H_3OCHO [1]. It is pale yellow or colorless oily liquid and turns into brown or red in the presence of air or light. Human exposure has been classified as a hazardous material that can damage the lungs, liver, kidneys and spleen. The permissible exposure limit (PEL) for the furfural is 5 ppm [2].

Furfural is an ideal solvent used to separate desired and unwanted components in petroleum derivatives. It has the following basic properties: Good solvent for extracting undesirable components as well as highly selective for desirable components, easy to be recovered does not react chemically with the components of oil, and it is not toxic industrial, when used has a high degree of stability [3].

Furfural has got main applications like in lube oil refining, in pharmaceuticals, and in the manufacture of phenolic resins. It is widely used in the solvent extraction processes of the petroleum refineries as a solvent extracting. It is also used as a chemical intermediate, weed killer, fungicide, and also as a flavoring agent. Sulfite pulping processes used in the pulp and paper industry are a major source of furfural contamination. Synthetic rubber plant wastewater has been found to have 1.7 g/l furfural. Leakage of furfural not only causes a pollution problem but also constitutes a sizable economic loss [4].

Activated carbons are among the best adsorbents utilized for adsorption processes. Activated carbons possess high surface area per unit mass and exhibit high adsorption capacities for many adsorbates. Essentially, the structure of Activated carbons contains pores which are arranged by

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the International Union of Pure and Applied Chemistry (IUPAC, 1972) order into three groups: micropores (pore measure < 2nm), mesopores (pore estimate 2– 50 nm) and macropores (pore estimate > 50 nm) [5]. .

Concept of Adsorption, in adsorption theory, The solid material that provides the surface for adsorption is referred to as adsorbent; the species that will be adsorbed are named adsorbate [6]. Adsorption occurs due to unsaturated and unbalanced molecular forces on the surface of the solid [7]. When molecules move from the liquid phase to the solid surface. Adsorbed species can be released from the surface and transferred back into the liquid phase. This reverse process is referred to as desorption [8].

The aim of present work removal of furfural from petroleum refinery wastewater by commercial activated carbon and Make regeneration for activated carbon and recovery of furfural.

2. Experimental Work:

2.1. Material and methods

- **Materials:** Adsorbent commercial activated carbon supplied by (Unicarbo, Italian, Lmt. Co.) Adsorbate Furfural was used as an adsorbate; it has synonyms of (2furaldehycle, Furyl and 2 Furyl methanol), Tables (1, 2) show the chemical and physical properties of furfural and ethanol respectively [9].

Table (1): Chemical and Physical properties of Furfural

Chemical formula	**($C_5H_4O_2$)**
Molecular weight	96.06 g/mol
Color	Colorless to yellow and red-brown when exposure to light and air
Odor	Aromatic odor as benzaldehyde
Specific gravity 20^{o}C	1.1598
Flash point, open cup ^{o}C	68.3
Heat of vaporization (kcal/mol) at 160 ^{o}C	9.22

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Table (2): Chemical and physical properties Ethanol

Chemical formula	CH_3CH_2OH
Molecular weight:	46.069 gm/mol
Appearance	Colorless
Odor	Mild, rather pleasant; like wine
Purity	99 %

- **Batch method:** To study the removal efficiency of furfural batch experiments were used. These experiments were summarized in Table (3) including the following variables (initial furfural concentrations, amount of adsorbent (dose) and temperature) with constant PH, agitation speed and particle size.

Table (3) Summary of Batch Experiments Variables

Variables	Initial Conc. Ppm	PH	Agitation speed rpm	Amount of adsorbent	Particle Size mm	Temp °C
Initial Concentration	40	7	150	40 g\ 1000 ml	0.91	25
	640	7	150	40 g\ 1000 ml	0.91	25
	780	7	150	40 g\ 1000 ml	0.91	25
amount of adsorbent	780	7	150	10 g\ 250 ml	0.91	25
	780	7	150	15 g\ 250 ml	0.91	25
	780	7	150	20 g\ 250 ml	0.91	25
Temperature	780	7	150	10 g\ 125 ml	0.91	35
	780	7	150	10 g \125 ml	0.91	50

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2.2. Theory/Calculation

The variation of furfural concentration in the aqueous solution due to adsorb by commercial activated carbon was determined calorimetric at wavelength (278nm) by UV-Visible DR5000 Spectrophotometer. The percent removal of furfural (Y_R) due to adsorption process was calculated according the equation:

$$Y_R = \frac{C_o - C_e}{C_o} \times 100 \quad \text{.........................} (1)$$

$$q_e = \frac{(C_0 - C_e)V}{W} \quad \text{.........................} (2)$$

Where $\mathbf{C_o}$ and **Ce** the initial and equipoise concentration of furfural in the aqueous solution.

$\mathbf{q_e}$ adsorption capacity and **V** the volume of beaker **W** the weight of amount activated carbon [10]

3. Results and Discussion:

The important properties of activated carbon are surface area and pore volume. Table (4) shows the characterization of activated carbon from this table the surface area was 950 m2⁄g. Table (4) shows the precise analysis of the ash content and moisture content and the apparent density of commercial activated carbon.

Table (4) Characterizations of commercial activated carbon

Characteristics	commercial activated carbon
Surface area, m^2/g	950
Iodine No.	850
Mesh size	12×40
Mean particle dam, mm	0,91
Effective size ,mm	0,6
Ash content, %	1.51
Moisture content, %	2
Bulk density, g/ml	0.538

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3.1 Effect of contact time, the initial concentration of furfural, the amount adsorbent.

The removal percent of furfural at different contact time from 30 to 240 minute. Figure (1) shows Furfural percent removal was 99.44, 99.54 and 99.73 at 240 min for 10, 15 and 20 g\ 125 ml respectively. With increase contact time until reach a maximum value after that the removal of furfural was constant with increasing the adsorbed time because reach to the equilibrium state[11]. The removal of furfural at different concentration (40, 640, and 780) ppm. Figure (2) shows furfural removal percent was 99.43, 99.45 and 99.25 at 240 min for 640, 780 and 40 ppm respectively. The removal depended on the initial concentration when increase the initial concentration the final concentration increase because the active site for activated carbon become lowest from furfural molecules, the removal percent of furfural increases with increasing time until reach to equilibrium[12] .The removal of furfural at different amount of adsorbent shown in Figure (3). Due to increase the amount of adsorbent material the surface area will increase therefore the removal percent increasing [13].

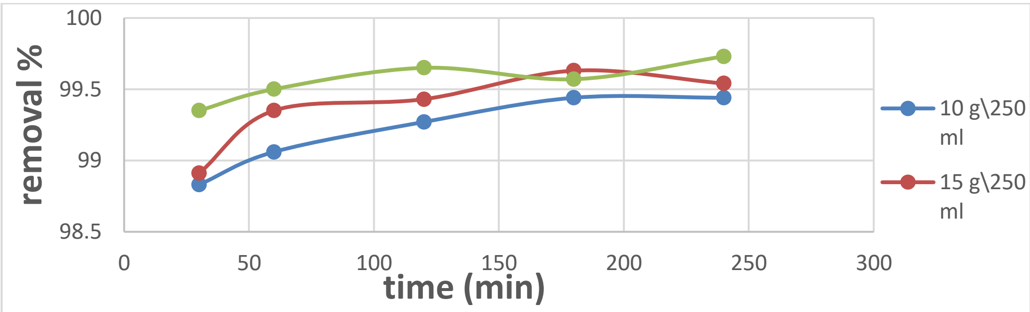

Fig. (1): The influence of contact time on the removal percent at different dose of activated carbon [initial concentration = 780 ppm, agitation speed= 150 rpm, volume of beaker=250 ml, PH=7 and temperature= 25 ºC]

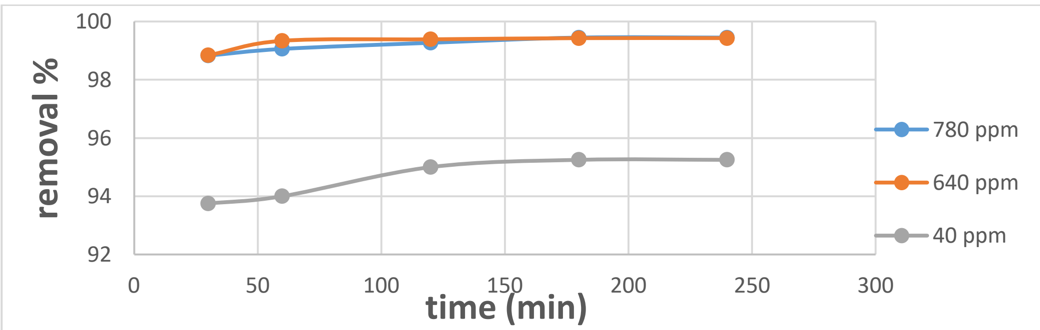

Fig. (2): The influence of contact time on the removal percent at different initial concentration of furfural [agitation speed= 150 rpm, dose = 40g, volume of beaker=1000 ml, PH=7 and temperature= 25 ºC]

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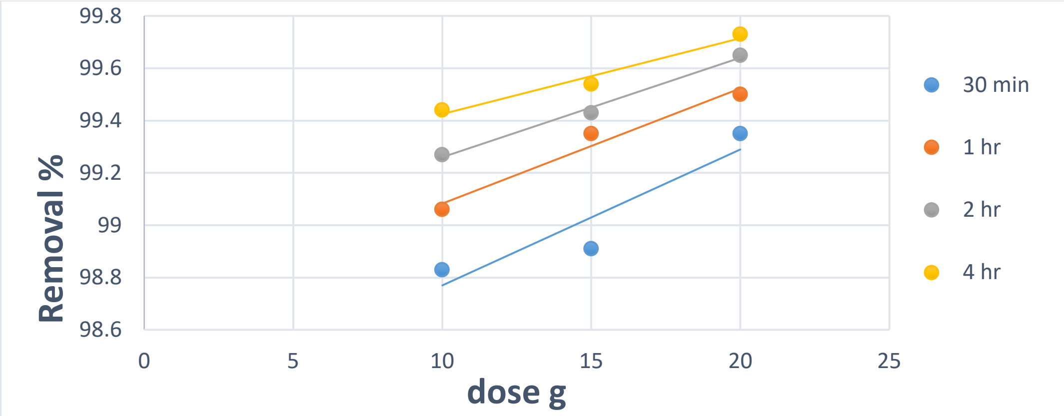

Fig. (3): The influence of dose of activated carbon on the percent removal [initial concentration = 780 ppm, agitation speed= 150 rpm, volume of beaker=250 ml, PH=7 and temperature= 25 °C]

3.2 Effect of temperature.

The temperature is the factor that greatly affects the adsorption capacity and the rate of adsorption. During the study for the adsorption of furfural on activated carbon, when increasing the temperature from 35°C to 50°C this will led to a increase in adsorption capacity as shown in Figure (5) . That means adsorption is chemical so it is not easy to adsorb furfural ions with pure water [14]. Removal percent increases with increase temperature as shown in Figure (4), but In general the temperature does not affect significant. Table (5) shows the final concentration and removal percent using different temperature at (concentration =780 ppm, volume of backer=125 ml, PH= 7, agitation speed =150 rpm and dose= 10 g) adsorption capacity was calculated by the equation (2) [14].

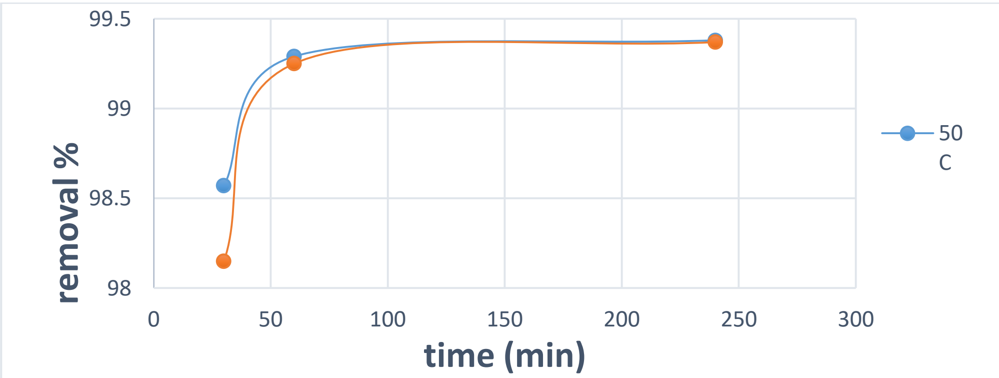

Fig. (4): The influence of contact time on the removal percent at different temperature [initial concentration = 780 ppm, agitation speed= 150 rpm, volume of beaker=125 ml, PH=7 and dose= 10 g

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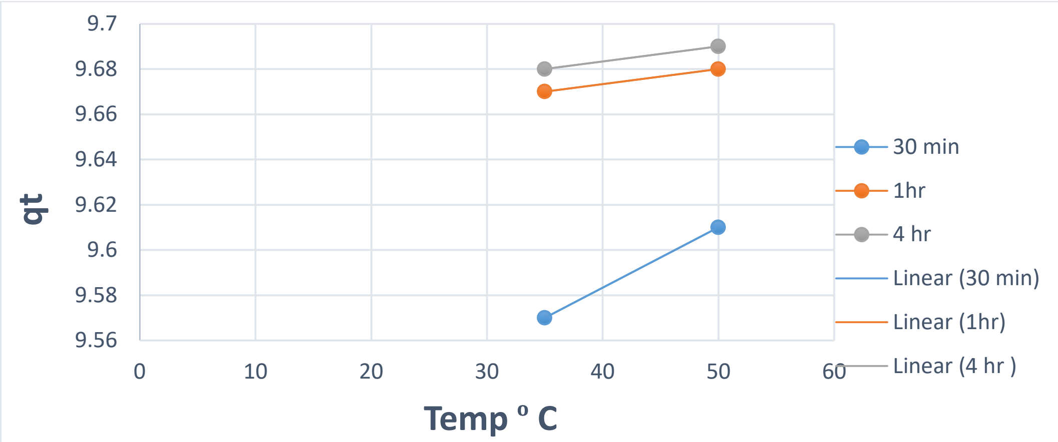

Fig. (5): The influence of temperature on the adsorption capacity [initial concentration = 780 ppm, agitation speed= 150 rpm, volume of beaker=125 ml, PH=7 and dose= 10 g]

Table (5) Shows the final concentration and removal percent using different temperature (concentration = 780 ppm, volume of backer=125ml, dose = 10g, PH= 7, and agitation speed =150 rpm)

Temperature	Cf (final concentration) ppm	Removal percent %	qt. mg\g
After 30 min			
35 °C	14.4	98.15	9.57
50 °C	11.1	98.57	9.61
After 1 hr			
35 °C	5.7	99.26	9.67
50 °C	5.5	99.29	9.68
After 4 hr			
35 °C	4.9	99.37	9.68
50 °C	4.8	99.38	9.69

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3.3 Applied conditions in the summer

Figure (6) Explains theoretical relationship between initial concentration of pollution and concentration after treatment [Temperature = 50 ºC, time= 1 hr, maximum ratio volume of activated carbon \ volume of pit = 4%, PH = 7, and agitation speed =150 rpm]. Note when the concentration of pollution is (550 ppm) and more the final concentration will exceed the contamination limit of the furfural (20 ppm). So the time should be increased from one hour to 3 hours when the pollution concentration is 650 ppm and more. When treated (1000 ppm) concentration under the same conditions for 3 hours the final concentration will reach (12.7 ppm) [15].

Figure (7) Explains the relationship between initial concentration of pollution and amount of activated carbon [Temperature = 50 ºC, time= 1 hr, rang removal (95.5- 98), %PH = 7, and agitation speed =150 rpm]. Note when the concentration of pollution (100 ppm) we can make ratio the amount of activated carbon to the volume of pit (2%) with increasing time more than 1 hr. [16].

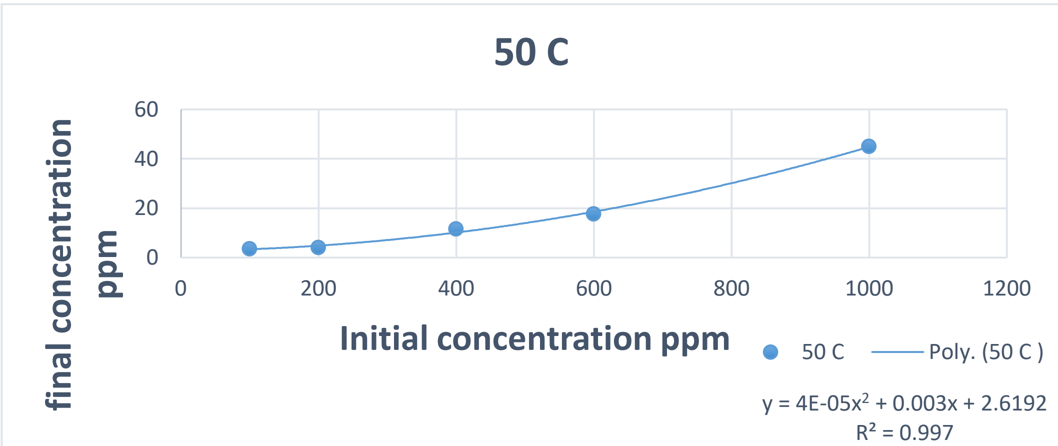

Fig. (6): The relationship between initial concentration of pollution and concentration after treatment [Temperature = 50 ºC, time= 1 hr, maximum ratio volume of activated carbon \ volume of pit = 4%, PH = 7, and agitation speed =150 rpm]

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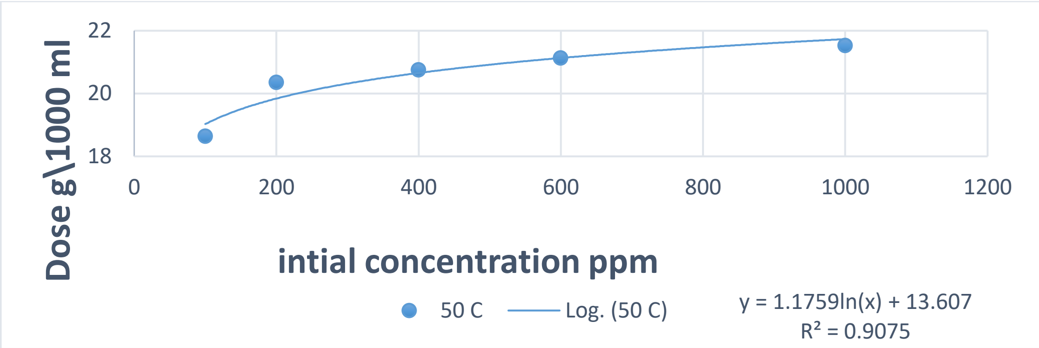

Fig. (7): The relationship between initial concentration of pollution and amount of activated carbon [Temperature = 50 °C, time= 1 hr, rang removal (95.5- 98),%PH = 7, and agitation speed =150 rpm]

3.4 Applied conditions in the winter.

Figure (8) Explains theoretical relationship between initial concentration of pollution and concentration after treatment [Temperature = 25 °C, time= 2 hr, maximum ratio volume of activated carbon\ volume of pit = 4%, PH = 7, and agitation speed =150 rpm]. Note when the concentration of pollution is (850 ppm) and more the final concentration will exceed the contamination near limit of the furfural (20 ppm). So the time should be increased from 2 hour to 3 hours when the pollution concentration is 1000 ppm and more [17].

Figure (9) Explains the relationship between initial concentration of pollution and amount of activated carbon [Temperature = 25 °C, time= 2 hr, rang removal (97.4-98.36), %PH = 7, and agitation speed =150 rpm]. Note when the concentration of pollution (100 ppm) we can make ratio the amount of activated carbon to the volume of pit (2%) with increasing time more than 1 hr. [18].

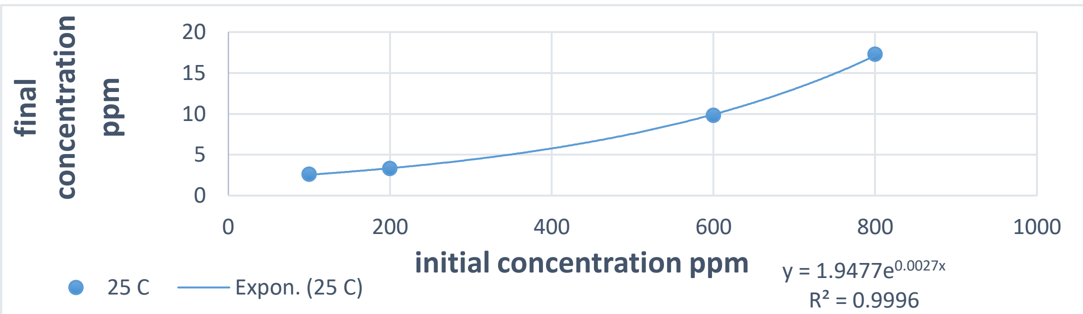

Fig. (8): The relationship between initial concentration of pollution and concentration after treatment [Temperature = 25 °C, time= 2 hr, maximum ratio volume of activated carbon \ volume of pit = 4%, PH = 7, and agitation speed =150 rpm]

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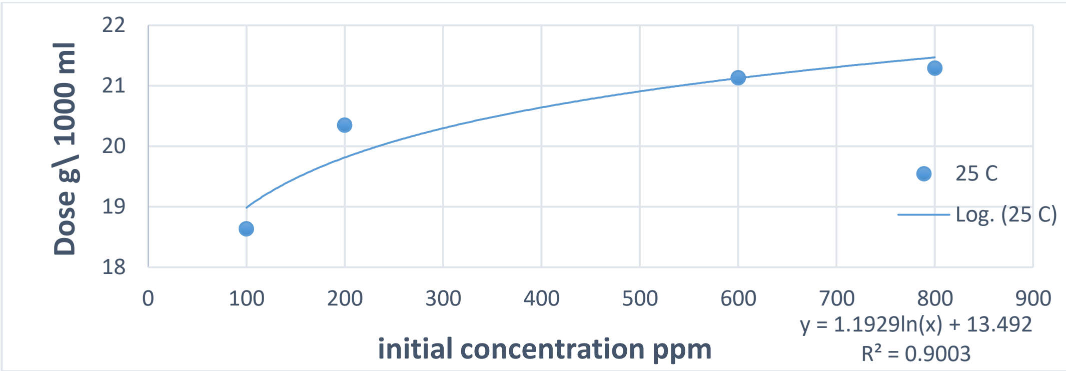

Fig. (9): The relationship between initial concentration of pollution and amount of activated carbon [Temperature = 25 °C, time= 2 hr, rang removal (97.4-98.36), PH = 7, and agitation speed =150 rpm]

3.5 Regeneration of activated carbon and recovery furfural

To evaluate the regeneration efficiency, adsorption batch tests were performed with regenerated adsorbents after regeneration, and the adsorption capacity of regenerated adsorbents was compared with the fresh adsorbents [19].

3.6 Condition of Desorption Tests

Tables (6) and (7) shows the condition of the desorption tests, in which activated carbon were contacted with pure water at different temperature. The content of furfural desorbed into the regenerates was analyzed by UV-Vis spectrophotometer after desorption tests.

Table (6) The condition for desorption furfural from activated carbon by pure water at 95°C

Initial conc. of furfural in activated carbon (ppm)	Ratio amount of activated carbon\Vol. of pure water in backer (g\ml)	Washing time (min) At 95°C and 150 rpm	Concentration of pure water after washing
600	3.228 g \ 500 ml	5	1.5
		15	4.1
		30	5.7
200	1.076 g \ 500 ml	5	0.2
		15	1.2
		30	1.8
100	2.959 g \ 500 ml	5	0
		15	0
		30	0.5

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Table (7) show the condition for desorption furfural from activate carbon by pure water at 100°C

Initial concentration of furfural in activated carbon (ppm)	Ratio amount of activated carbon\volume of pure water in backer (g\ml)	Washing time (hr) At 100°C and 800 rpm	Concentration of pure water after washing
1000	6.456 g \ 240 ml	1	15
		2	40
100	6.454 g \ 240 ml	1	0
		2	0

3.7 Desorption by Pure Water

DI water was first utilized in the desorption tests to desorb furfural from the activated carbon at different temperature. The results of UV spectra of furfural desorbed from activated carbon into water show the pure water not can adsorb furfural because the type of adsorption is chemical so we cannot use pure water as regenerates [20] as shown in Figure (10).

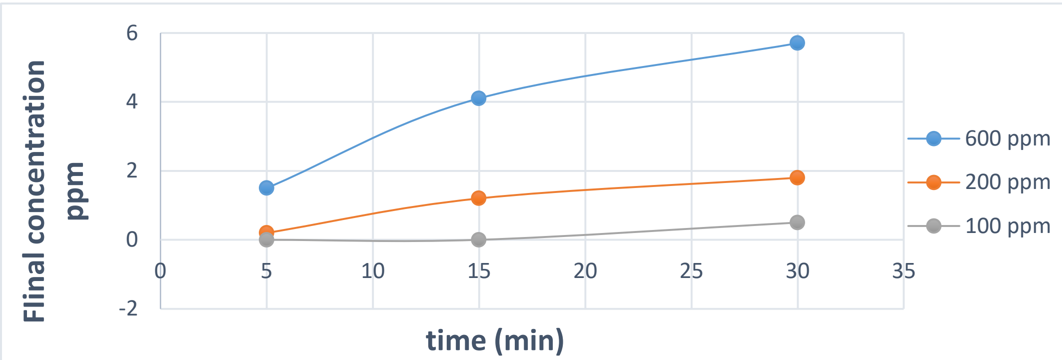

Fig. (10): desorption furfural from activated carbon by pure water at 95°C

3.8 Desorption by Ethanol

Ethanol water solution was further investigated to desorb furfural form activated carbon at room temperature. Ethanol water solutions with different ethanol concentration were utilized in the desorption tests, and the results are shown in Figure (11) and Table (8). The desorption of furfural from activated carbon increased with an increase of ethanol concentration up to 50 wt%. Ethanol has advantages in industry application due to its lower cost and relative eco-friendliness [21].

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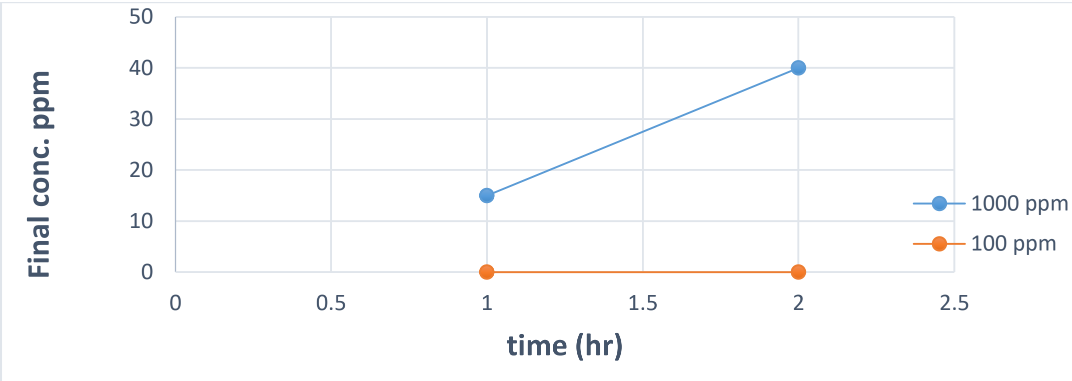

Fig. (11): desorption furfural from activated carbon by pure water at 100°C

Table (8) show the condition for desorption furfural from activated carbon by ethanol 50wt% at 25 °C.

Initial concentration of furfural in activated carbon (ppm)	Ratio amount of activated carbon\volume of ethanol in backer (g\ml)	Washing time (hr) At 25°C and 450 rpm	Concentration of solution after washing
200	2.69 g \ 25ml Ethanol 25wt%	1	18.7
		2	17.9
		3	16
200	2.69g \ 25 ml Ethanol 50wt%	1	100
		2	45
		3	30

3.9 Design

Oil 2 (Sump drum (O3D4), Length = 704 cm, Width = 288 cm, Depth =327 cm).

Oil 3 (Sump drum (D 303), Length = 10 m, Width = 4.5 m, Depth =3.5 m).

The volume of furfural contamination in the pit = length (`10 m)* width (4.5 m) *depth (1 m) = 45 m^3, 45 m^3 = 45000 L

The maximum pollution reach to (100 ppm), so the percent of activated carbon to the volume of furfural contamination in the pit (3%)

Volume of activated carbon = 0.03* 45000 = 1350

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Density of activated carbon = 0.538 g \ ml

Weight of activated carbon = Volume of activated carbon * Density of activated carbon

= 1350000 ml * 0.538 g \ ml = 726300 g = 726.3 kg

Dimensions of the basket (Length = 2.5 m, Width = 2.5 m, Depth = 1 m)

Volume of basket = (2.5 m* 2.5m* 1m) = 6.25 m^3, 6250 L, Table (9) explains design details.

Table (9) Design the basket

Position	The total volume of pit (Liter)	The volume of furfural contamination in the pit (Liter)	The volume of the basket (Liter)	The volume of activated carbon (Liter)	The amount of activated carbon (Kg)
Oil-2 Sump drum (O3D4)	66299.9	20300	810	609	327
Oil-3 Sump drum (D 303)	157500	45000	6250	1350	726.3

3.10 Economic costs

Oil-2

In a single operation in case maximum pollution (100 ppm) the cost of furfural lost (4519.2 $). We used activated carbon cost (528 $) for three treatment pollution also the cost of ethanol used for regeneration of activated carbon and recovery of furfural is (3060.8 $) after that we can used activated carbon, the equation will become [22]:

Profit ($) = [Cost furfural loss] – [cost activated carbon + ethanol +laborer

= [4 * 4519.2 $] – [528 $ + 1226.76 $ + 50 $] = 16272.04 $

The same method of calculation for **Oil -3, Tables (10, 11) explain economic costs details.**

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Table (10) Economic costs for Oil-2

Substance	The price per (Ton)	Quantity loss when contamination 100 ppm from furfural	Quantity used for treatment	the cost $
Furfural	2400 $	1.883 ton	-	4519.2 $
Activated carbon	1600 $	-	330 kg	528 $
Ethanol	800 $	-	1533.4 liter	1226.76$

Table (11) Economic costs for Oil -3

Substance	The price per (Ton)	Quantity loss when contamination 100 ppm from furfural	Quantity used for treatment	the cost ($)
Furfural	2400 $	4.176 ton	-	10022.4 $
Activated carbon	1600 $	-	726.3 kg	1162.08$
Ethanol	800 $	-	3375 liter	2700 $

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4.Conclusions

According to the results obtained from this study, the following conclusions are obtained:

1- Activated carbon with BET surface area of 950 m^2/g and iodine number of 850 $m^2\backslash g$, so it is having high ability to adsorb furfural.

2- The removal percent of furfural increases with increasing time until reach to equilibrium.

3- The removal percent of furfural decreases with increasing initial furfural concentration, the amount of activated carbon constant.

4- The removal percent of the furfural in constant of all the variables increases with increasing the amount of activated carbon.

5- Chemicals adsorption takes place as result of increasing adsorption capacity with increasing temperature.

6- The removal percent of furfural increases with increasing temperature, in constant of all the variables.

7- Ethanol 50wt% is ideal solvent used for regeneration activated carbon and recovery of furfural.

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