REMOVAL OF KEROSENE FROM WASTE WATER USING IRAQI BENTONITE

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ABSTRACT

The aim of the present research is to study the potentiality of Iraqi bentonite as adsorbent for removing of kerosene from wastewater. Also the capacity of bentonite for kerosene removal was compared to the activated carbon capacity. The sorption of kerosene onto bentonite and AC. were described by two well – known adsorption isotherm models namely Langmuir and Freundlich models. It was found that the Freundlich model can fit very well the equilibrium isotherm adsorption of kerosene onto bentonite and AC. Batch experiments were carried out to study the effect of adsorption of kerosene onto bentonite using various conditions such as initial concentrations of kerosene (100-500) mg/L, agitation speeds (125, 250, 500, 800) RPM, and weights of bentonite (0.05, 0.5,1.1) gm, particle sizes (0.5- 0.6) mm, and temperature 303 k . It was found that the best results for removing kerosene onto bentonite were obtained at C_o=500 mg/L, RPM =800. Activated carbon was used as powder and granular of particle sizes ranged (1-1.18) mm and (0.5-0.6) mm. The results indicated that the activated carbon was more active than bentonite for removing of kerosene from wastewater

KEYWORDS:
Bentonite; activated carbon; kerosene; adsorption

الخلاصة

الهدف من هذا البحث هو دراسة قدرة البنتونايت العراقي كمادة ممتزة (adsorbent) على ازالة الكيروسين من الماء. وكدلك مقارنة كفاءة ازالة الكيروسين بواسطة البنتونايت مع كفاءة ازالة الكيروسين بواسطة الكاربون المنشط.

ان عملية امتزاز الكيروسين على البنتونايت و الكاربون المنشط تم وصفها بمودلين معروفين(لا نكمير وفرندلش) والتي تعتبر من الموديلات الشائعة في مجال معالجة المياه الملوثة,وقد وجد بأن معادلة (فرندلش) هي اكثر تطابقا من معادلة (لانكمير) في عملية امتزاز الكيروسين على البنتونايت والكاربون المنشط عند حالة التوازن(Equilibrium Isotherm). تم اجراء تجارب النمط الدفعي (Batch Process) لدراسة تأثير : تغير التركيز الابتدائي للكيروسين ، سرعة الرج، تغير وزن البنتونايت مع الزمن. وقد تمت كل التجارب تحت نفس الظروف. وجد بان افضل النتائج لازالة الكيروسين على البنتونايت هي بتركيز ابتدائي = 500 ملغرام/ لتر ، سرعة الرج = 800 ، حجم حبيبي= (0.6- 0.5) ملمتر. وتم استخدام الكاربون المنشط باحجام مختلفة (1.18-1) ملمتر ، (0.6- 0.5) ملمتر ،(بودر) . لقد اشارت النتائج بأن الكاربون المنشط هو اكثر فعالية من البنتونايت في ازالة الكيروسين من الماء .

INTRODUCTION:

Numerous standards and regulations were adopted for discharge of oily waste water into surface water or sewage systems. These regulations may vary from country to another, and even within a country itself. Environment Canada (1976b) established a discharge limit for oil and grease of 15 mg/L at federal establishment. In Iraq (Iraqi Specification 1967) the allowable oil and grease concentration for discharge into water is 10 mg/L

Oils that are found in contaminated water can be grouped into four categories (Patterson,1975): (1)light hydrocarbons including light fuels such as gasoline, kerosene and jet fuel; (2)heavy hydrocarbons; (3)lubricants and cutting fluid; and (4)fats that are found in both plants and animals. The aqueous effluents from industrial, municipal and petroleum refineries contain different pollutants including oil, furfural, phenolic compounds, sulfides, suspended solids, toxic metals, ammonia and biochemical oxygen demand. Methods of removal of those pollutants generally involve biological degradation, chemical oxidation, physical stripping and adsorption process (Prather, 1970). Adsorption techniques are widely used in the field of removing small quantities of pollutant present in the large volume of fluid, which can be carried out in batch wise or continuous manner of operation (Rao, 1994). Many factors may affect the decision of choosing an adsorbent for removal of oil from water, these are: (1) economical factor (cost of the adsorbent); (2) abundance and availability of the adsorbent, and (3) effectiveness of the adsorbent in removing a particular pollutant. The removal of organic material by adsorption using activated carbon or polymers is an effective treatment technique. Although, activated carbon is a preferred adsorbent, its wide spread uses are restricted due to its high cost and expends much of its capacity in removal of incidental constituents (Dentel et al.,1995). Therefore many studies are directed in finding an efficient and economical sorbent. Clay (bentonite) can be considered as an alternative adsorbent due to its effectiveness in removing certain organic compounds from water and its abundance, availability, and low cost (Baker & Luh, 1971).

Numerous quantitative empirical mathematical expressions, called isotherms, have been developed to describe sorption. The two most common sorption models are the Langmuir and Freundlich isotherms (Sulaymon, A. H. and Ahmed K. W., 2008).

The Langmuir model is based on the assumption that a single monolayer of sorbate accumulate at the solid surface. As the concentration of the sorbate is increased in the liquid phase, proportionately more of the sorbent surface is covered with the sorbate. The equation that describes Langmuir system is the

$$q_e = \frac{x}{m} = \frac{ab \ \mathrm{Ce}}{1 + \mathrm{bCe}} \qquad (1)$$

The Langmuir equation may be transformed to a linear expression by inverting Equation (1) and separating variables:

$$\frac{\mathrm{Ce}}{q_e} = \frac{1}{ab} + \frac{\mathrm{Ce}}{a} \qquad (2)$$

The empirical coefficients (a) and (b) may be obtained by plotting Ce/qe as a function of Ce. Using linear regression, the slope = 1/a and the y- intercept = 1/ (ab) can be obtained. (Richard, 1997).

The Freundlich model is characterized by sorption that continues as the concentration of sorbate increases in the aqueous phase. The mass of material sorbate is proportional to the aqueous phase concentration at low sorbed concentrations and decreases as the sorbate accumulates on the sorbent surface. Sorption then continues with increasing aqueous phase sorbate concentrations, but to a diminishing degree. The Freundlich isotherm is quantified by:

$$q_e = \frac{x}{m} = K_F C_e^{1/n} \qquad (3)$$

The coefficients "K_F" and "n" may be determined by plotting experimental data on log –log paper. Alternatively, Equation (3) can be transformed logarithmically:

$$\text{In}\ q_e = \text{In}\ K_F + \frac{1}{n}\ \text{In}\ C_e \qquad (4)$$

Logarithmic transformations of experimental data may then be plotted on arithmetic paper as In q_e as a function of In C_e ; through linear regression, $K_F = 10^{\text{y-intercept}}$ and 1/n = slope can be found. (Richard, 1997)

EXPRIMENTAL PROCEDURES:

Materials:

- **Adsorbate:**

  Kerosene is obtained by atmospheric distillation of crude oil and consists mainly of normal and branched chain alkanes, cycloalkanes, alkyl benzenes and alkyl naphthalene's (Ellison et al., 1999). Aliphatic hydrocarbons are the primary components (80%) ranging from C_9 to C_{16}. Aromatic hydrocarbon makes up about 20% of the components and consists mainly of single- ring compounds such as alkyl benzene. The kerosene used in this study was supplied by "AL-Dora Refinery "; it was distilled at range (180-270) ^{o}C.

- **Adsorbent:**

  Bentonite:

  bentonite used in this study was an Iraqi bentonite (Calcium base). It was supplied by "State Company of Geological Survey and Mining" as pecieses of rocks. These rocks were destructed by a crusher (type: Jaw Crusher – BBI Restch) to granules different sizes, and then sieved using sieves (type: Restch, Germany) to produce granules of sizes (0.5-0.6) mm. The granules were grained to powder (0.074-0.088) mm by grainder (type: National MK – 51N). The granules of bentonite were dried at 100 ^{o}C for 30 min before used.

  Activated carbon (AC.):

  Granulated activated carbon (GAC) was supplied by "Unicarbo, Italians". The physical properties were measured by Oil Research and Development Center-Ministry of Oil and were coincided with that supplied by the manufacture. The granulated activated carbon (GAC) was crushed, sieved into (0.5- 0.6) mm, (1-1.18) mm, and then grained to powder (0.074-0.088) mm by a grainder (type: National MK – 51N). The require sieve fraction was removed and washed by distilled water to remove fines from the crushed GAC. The GAC was placed in a clean beaker filled with distilled water. Then it was stirred with a glass rod and allowed to settle. After allowing the GAC particles to settle for (5) minutes, the supernatant was poured off and new distilled water was added. This process was repeated until the supernatant was clear. The wet GAC was dried in an oven that was maintained at (100) ^{o}C for (24) hours, after which the GAC was kept in a desiccators for experimental use.

- **Solvent:**

  Iso-propyl alcohol [$(CH_3)_2CHOH$] was used as a solvent for kerosene in water. It's molecular weight is (60.1) g/g mol.

Equilibrium isotherm experiment:

Batch studies were adopted to obtain the equilibrium data. Solutions were prepared containing the desired solute (kerosene) concentration of 100, 200, 300, 400, and 500 mg/L. 100 ml of each prepared solution were placed in nine bottles each of 250 ml volume. Adsorbent of 0.05, 0.1, 0.2, 0.3, 0.5, 0.75, 0.9, 1, and 1.1 gm were placed in nine individual bottles, each sample of them should be gathered with another sample containing the same quantity of adsorbent solution (adsorbent and distilled water) as a blank. The bottles were then placed on a shaker (Type: B.BRAUN) and agitated continuously for 30 hours at 30 °C. Ten ml from each sample was taken and mixed with 10 ml of iso-propyl alcohol (extra pure) as co- solvent (Bastow et al., 1997) and the mixture was shaked by shaker (type: Stuart scientific, Auto vortex SA6, UK) for (1.5) min,and then filtered by filter paper (type: Whatman 542, England).

The equilibrium concentrations were measured by means of UV- spectrophotometer (Type: GENESYS^{TM} 10 series spectrophotometers, thermo). At that point the concentration was in equilibrium. The adsorbed amount is calculated using the following equation:

$$qe = \frac{V_L (C_0 - Ce)}{m} \qquad (5)$$

The adsorption isotherms curves were obtained by plotting the weight of solute adsorbed per unit weight of adsorbent (qe) against the equilibrium concentration of kerosene in the solution (C_e) (Crittenden& Weber, 1978). All batch experiments were conducted at constant temperature 30 °C.

Swelling test experiment:

The swelling of bentonite clay in water and kerosene was measured. One gm of powderd bentonite was gradually added in small portions to 50 ml of water and the same quantity of bentonite was added to 50 ml of kerosene contained in a 100 ml graduated cylinder, without stirring .

After (24) hours at room temperature, the volume of the column of clay was measured, after which the content was stirred with a glass rod and allowed to stand for another (24) hours. The swelling was then recorded in both water and kerosene.

Analytical technique:

The UV – technique was used to measure the concentration of kerosene in a single system.

The optimal wavelength of kerosene was found to be 298 nm, using a Shemadzu model UV-160A ultraviolet / visible spectrophotometer, The concentration of the individual single component solute were determined using UV spectrophotometer directly from the calibration curve, figuer (1).

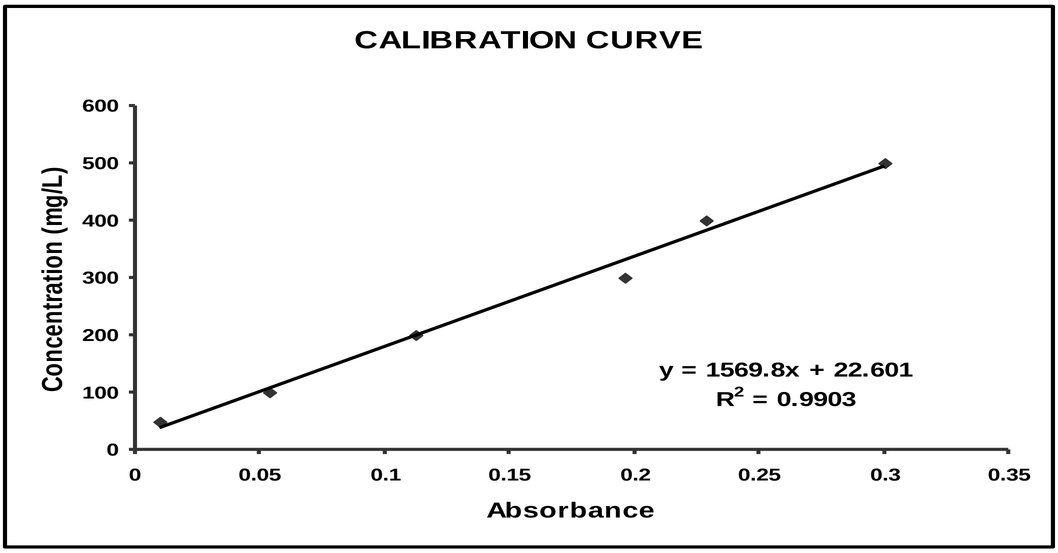

Fig. (1) Calibration curve for kerosene system

RESULTS & DISCUSSION:

Batch isotherm studies of bentonite:

The data collected from adsorption isotherm experiments were subjected to non – linear estimation using the two well – known adsorption isotherms commonly adopted in the field of environmental engineering (Langmuir and Freundlich isotherm). The non – linear estimation was performed on a statistical package "AXCEL for Windows". The adsorption isotherms of kerosene onto bentonite of concentrations 100, 200, 300, 400, and 500 mg/L, agitation speed (RPM=250), and particle size 0.5- 0.6 mm at 30 ^oC are shown in figure (2).

Plotting Ce/qe versus Ce to obtain the value of constants in Langmuir model (a and b). These values were obtained from the slop and intercept of the lines, and tabulated in table (1).

Plotting ln qe versus ln Ce to obtain the value of constants of the Freundlich model (K_F and 1/n). These values were obtained from the slop and intercept of the lines, and tabulated in table (1).

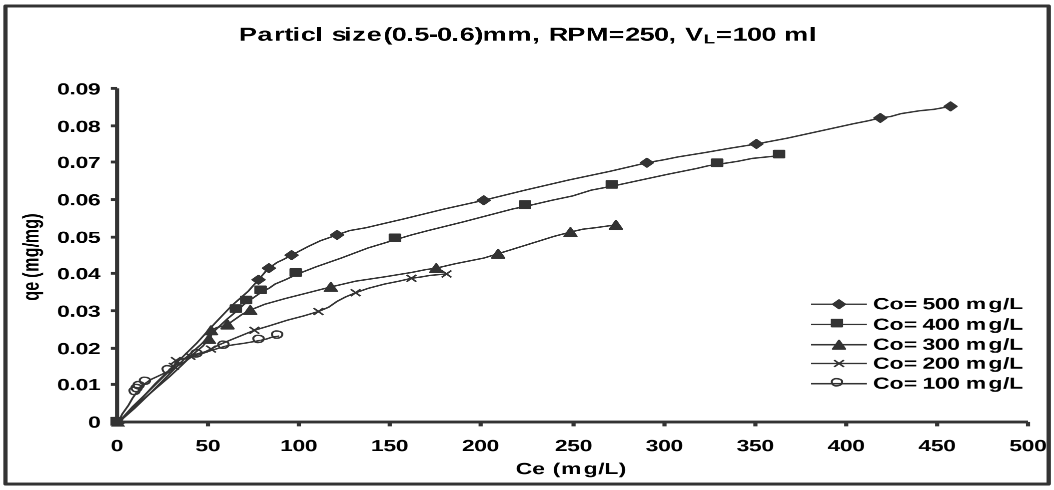

Fig (2) Adsorption of kerosene onto bentonite at different initial Concentrations

Table (1) Regression equations of (Langmuir and Freundlich) isotherm models for various initial concentrations of kerosene and bentonite.

Initial Concentration	Isotherm model	Equation of regression	R^2
C_o = 500 mg/L	Langmuir	$\frac{x}{m}=\frac{(0.1084)(0.0070)Ce}{1+0.0070Ce}$	0.9921
	Freundlich	$\frac{x}{m}=0.0065\ Ce^{1/2.3872}$	0.9936
C_o = 400 mg/L	Langmuir	$\frac{x}{m}=\frac{(0.1008)(0.0065)Ce}{1+0.0065Ce}$	0.9947
	Freundlich	$\frac{x}{m}=0.0029\ Ce^{1/1.7439}$	0.9952
C_o = 300 mg/L	Langmuir	$\frac{x}{m}=\frac{(0.0714)(0.0094)Ce}{1+0.0094Ce}$	0.9858
	Freundlich	$\frac{x}{m}=0.0040\ Ce^{1/2.0325}$	0.9895
C_o = 200 mg/L	Langmuir	$\frac{x}{m}=\frac{(0.0606)(0.0103)Ce}{1+0.0103Ce}$	0.9775
	Freundlich	$\frac{x}{m}=0.0024\ Ce^{1/1.8480}$	0.9955
C_o = 100 mg/L	Langmuir	$\frac{x}{m}=\frac{(0.0305)(0.0357)Ce}{1+0.0357Ce}$	0.9933
	Freundlich	$\frac{x}{m}=0.0028\ Ce^{1/2.1000}$	0.9935

It is clear from figure (2) and table (1) that:

- The equilibrium isotherm for each initial concentrations of kerosene is of favorable type (figure 1), for being convex upward and the amount adsorbed is proportional to the concentration in the fluid.
- The results of regression equations obtained for various initial concentrations of kerosene are presented in table (1). Higher values of correlation coefficient it were observed in Freundlich model for initial concentrations of 100, 200, 300, 400, and 500 mg/L.
- The sorption capacity of bentonite for various kerosene initial concentrations varied from (0.0082) to (0.0852) mg of kerosene per mg bentonite table (2). The average kerosene sorption capacity of bentonite was the highest for initial concentration of 500 mg/L and the lowest for initial concentration of 100 mg/L.

Table (2) Experimental kerosene sorption capacity values (x/m) of bentonite for various kerosene initial concentrations, Co

Kerosene initial concentration	mg kerosene sorbed per mg bentonite, $\frac{x}{m}$	
	Range	Mean
C_o = 500 mg/L	0.0384 - 0.0852	0.0608
C_o = 400 mg/L	0.0304 - 0.0722	0.0503
C_o = 300 mg/L	0.0227 - 0.0534	0.0369
C_o = 200 mg/L	0.0153 - 0.0402	0.0265
C_o = 100 mg/L	0.0082 - 0.0232	0.0152

Batch kinetic studies of bentonite:

The plots of kerosene concentrations versus time for kerosene emulsion (C_o = 500 mg/L) used in this study and bentonite of 0.05, 0.5, and 1.1 gm are presented in figure (3). Based on this plot, an equilibrium time of 4 hours were chosen for kerosene, (based on 1.1 gm). It is notable that the kerosene concentrations decreased sharply at the beginning of adsorption indicating a rapid sorption rate(this is due to high surface area available for sorption at the beginning of the process), and then the decreased gradually, indicating a slow sorption rate.

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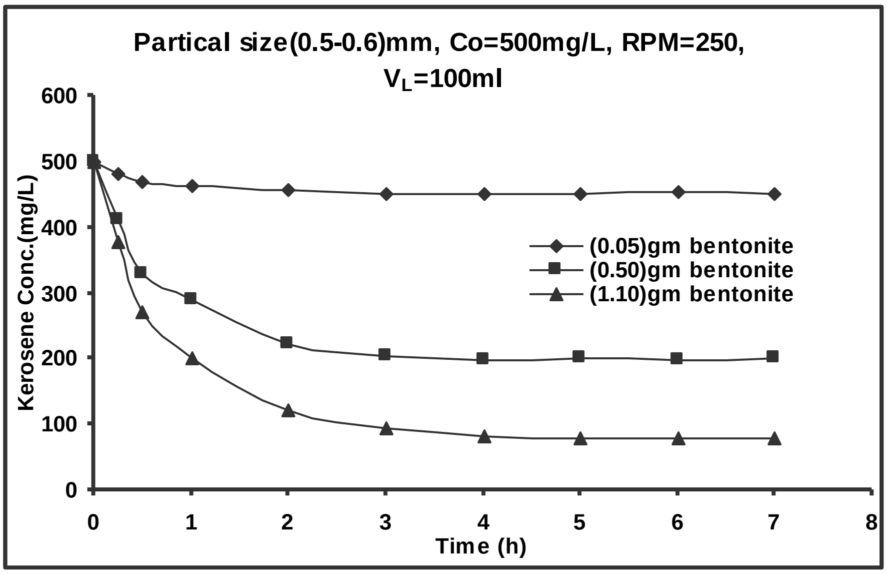

Fig.(3)Equilibrium times for the sorption of kerosene by various quantities of bentonite

The plots of kerosene concentrations at equilibrium Ce versus quantities of bentonite for various kerosene initial concentrations 0f 100, 200, 300, 400, 500 mg/L, RPM= 250 are presented in figure (4). As shown in this figure (4), when the quantity of bentonite is higher than 1.1 gm, the kerosene removal will not be increased due to reaching the equilibrium state.

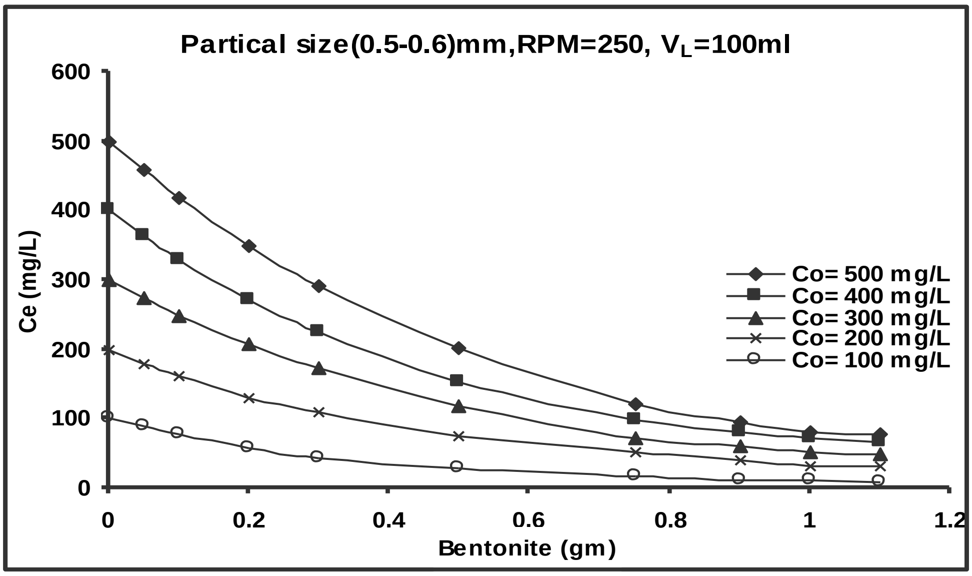

Fig. (4) Kerosene removal by bentonite for various initial concentrations

Effect of agitation speeds of bentonite:

The typical concentration decay curves of kerosene in batch experiments at different agitation speeds are shown in figure (5). It is observed that with increasing agitation speed, the kerosene concentration decreased.

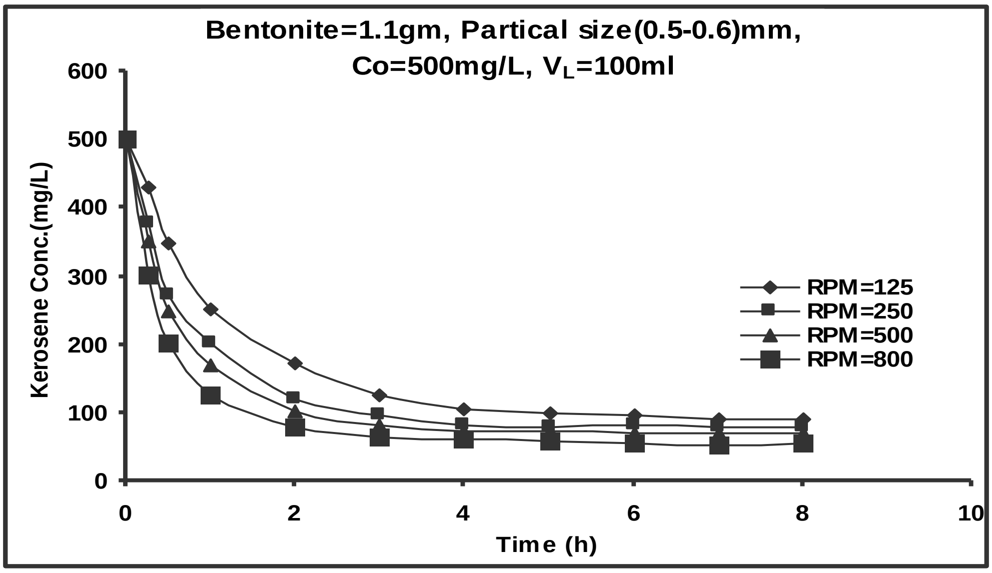

Fig.(5) Concentration – time decay curves for kerosene onto bentonite in batch process at different agitation speeds

Swelling tests of bentonite:

Figure (6) shows the swelling of the bentonite. The bentonite showed high swelling values in water with and without mixing (i.e. 4 and 6.5 ml/gm, respectively), and a low 1.5 ml/gm swelling value in kerosene (with and without mixing). No significant increase was found when the swelling was recorded after 48 hours and after mixing. This test emphasized that bentonite was posed hydrophilic nature more than organophilic nature.

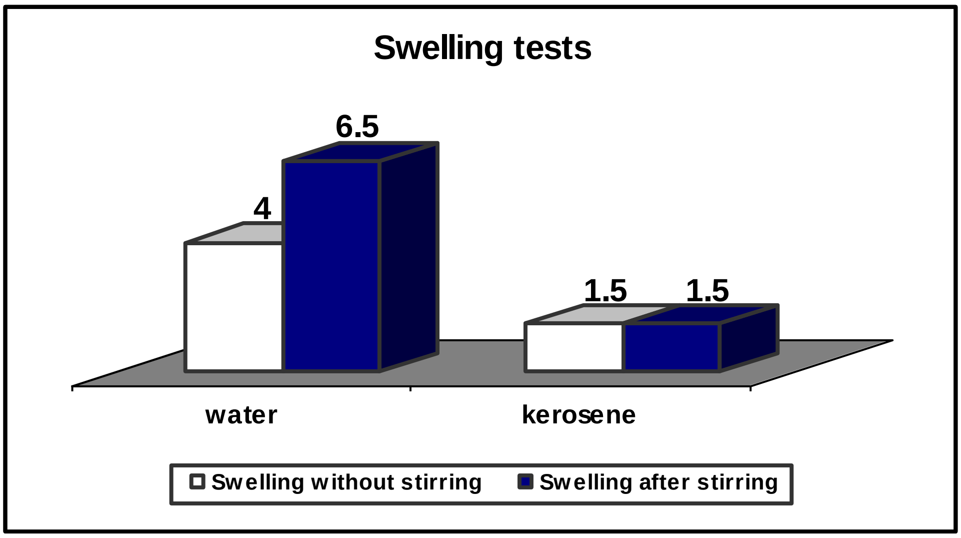

Fig. (6) Swelling values with and without stirring

Batch isotherm studies of activated carbon:

The adsorption isotherm of kerosene onto activated carbon of C_o= (500) mg/L, agitation speed RPM= 800, powdered and granular of size activated carbon (0.5-0.6) and (1-1.18) mm, at 30 ^oC is shown in figure (7).

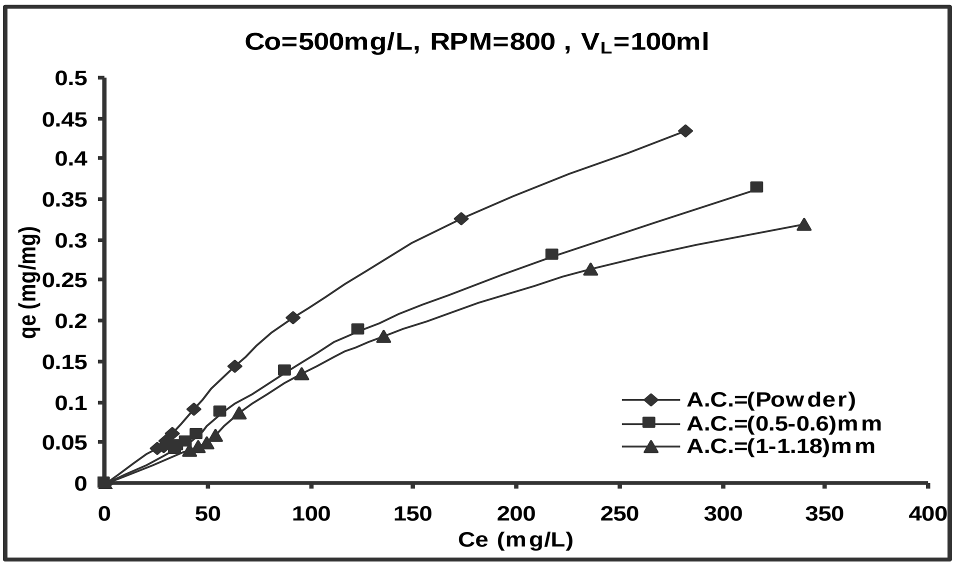

Fig. (7) Adsorption of kerosene onto activated carbon at different particle sizes.

The values of empirical constants for two models (Langmuir and Freundlich) were similarly as for bentonite, and are presented in table (3).

Table (3) Regression equations of (Langmuir and Freundlich) isotherm model for various particle sizes of activated carbon.

Particle size (mm)	Isotherm model	Equation of regression	R^2
(powder)	Langmuir	$\frac{x}{m}=\frac{(0.0940)(0.0006)Ce}{1+0.0006Ce}$	0. 1536
	Freundlich	$\frac{x}{m}=0.0020\ Ce^{1/1.0146}$	0.9748
(+0.5, -0.6)	Langmuir	$\frac{x}{m}=\frac{(3.0618)(0.0004)Ce}{1+0.0004Ce}$	0.1581
	Freundlich	$\frac{x}{m}=0.0014\ Ce^{1/1.0183}$	0.9817
(+1, -1.18)	Langmuir	$\frac{x}{m}=\frac{(3.3134)(0.0003)Ce}{1+0.0003Ce}$	0.0604
	Freundlich	$\frac{x}{m}=0.0011\ Ce^{1/0.9983}$	0.9634

It is clear from above figures (7) and table (3) that:

- The equilibrium isotherm for various particle sizes of activated carbon is of favorable type, figure (7).
- The results of regression equations obtained for various particle sizes of activated carbon are presented in table (3). Higher value of correlation coefficient indicated that the Freundlich model was valid isotherm for all particle sizes.
- The sorption capacity of activated carbon for various particle sizes varied from (0.0417) to (0.4358) mg of kerosene per mg activated carbon, (table 4). The average kerosene sorption capacity of activated carbon was the highest for the powder and the lowest for particle size(1-1.18) mm. As shown in table (4), it is notable that as the particle size of activated carbon decreases, the sorption capacity increases due to the increase of surface area.

Table (4) Experimental kerosene sorption capacity values (x/m) of activated carbon for various particle sizes.

Particle size(mm)	mg kerosene sorbed per mg activated carbon, x/m	
	Range	Mean
(1-1.18)	0.0417 to 0.321	0.1318
(0.5- 0.6)	0.0423 to 0.3646	0.1401
(powder)	0.0431 to 0.4358	0.1566

- The results indicated that as the particle size of activated carbon decreased the kerosene removal efficiencies increased, at certain constant agitation speed and weight of adsorbent .The plot of kerosene concentration at equilibrium C_e versus quantities of activated carbon for various particle sizes is presented in figure (8). It is of interest to note that the kerosene concentration at equilibrium Ce decreased very sharply at lower quantities of A.C. (up to 0.3 gm), and then the decrease became gradual.

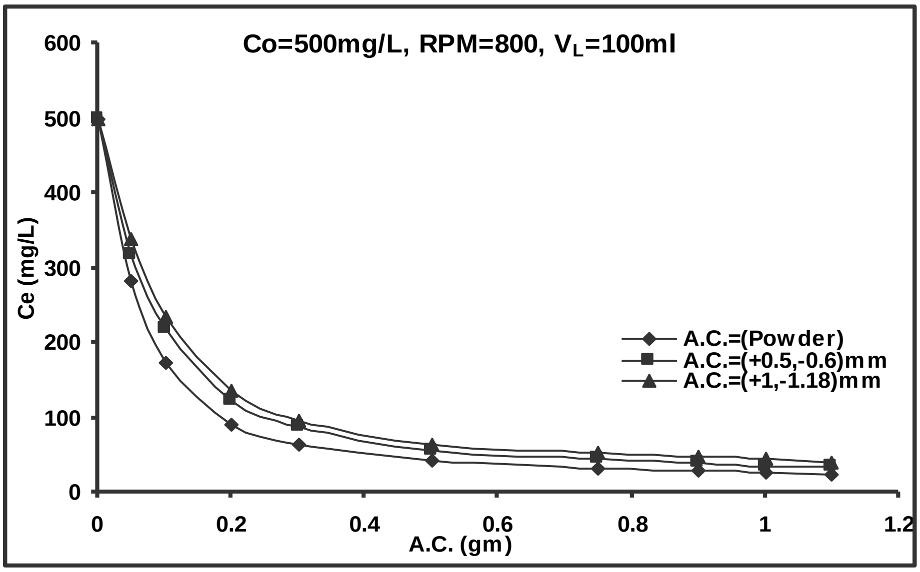

Fig. (8) Kerosene removal by activated carbon for various Particle sizes

Batch isotherm studies of bentonite and activated carbon:

The adsorption isotherm of kerosene onto bentonite and activated carbon of Co = (500) mg/L, agitation speed RPM = 800, particle size (0.5-0.6) mm at 30 ^{o}C is shown in figure (9).

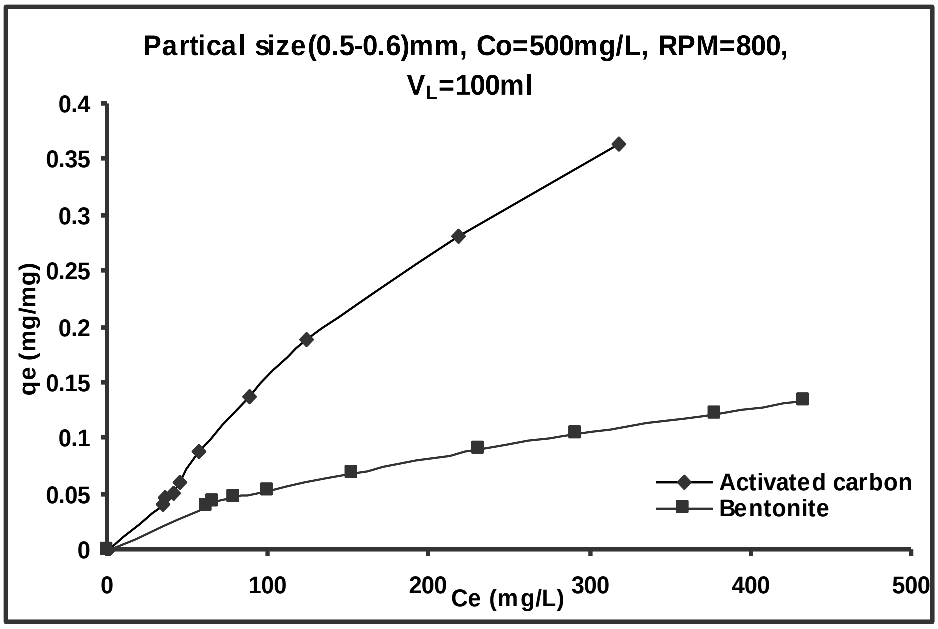

Fig. (9) Adsorption of kerosene onto bentonite and activated carbon

The values of empirical constants for two models (Langmuir and Freundlich) were calculated by the same previous manner, their values were tabulated in table (5).

Table (5) Regression equation of (Langmuir and Freundlich) isotherm model for bentonite and activated carbon

Adsorbent	**Isotherm model**	**Equation regression**	**R^2**
Bentonite	Langmuir	$\frac{x}{m}=\frac{(0.2128)(0.0034)C_e}{1+0.0034C_e}$	0.9737
	Freundlich	$\frac{x}{m}=0.0033\ C_e^{1/1.6455}$	0.9987
A.C.	Langmuir	$\frac{x}{m}=\frac{(3.0618)(0.0004)C_e}{1+0.0004C_e}$	0.1581
	Freundlich	$\frac{x}{m}=0.0014\,C_e^{1/1.0183}$	0.9817

It is clear from above figure (9) and table (5) that:

- The equilibrium isotherm for bentonite and activated carbon are of favorable type and nearly follow the same behavior, figure (9).

- Freundlich model was the most valid isotherm for bentonite and activated carbon.
- The sorption capacity of bentonite varied from 0.0398 to 0.1334 mg of kerosene per mg bentonite, and sorption capacity of activated carbon varied from 0.0423 to 0.3646 mg of kerosene per mg activated carbon, table (6). The average kerosene sorption capacity of activated carbon was the highest and the lowest for bentonite.

Table (6) Experimental kerosene sorption capacity values (x/m) of bentonite and activated carbon

Adsorbent	**mg kerosene sorbed per mg (bentonite or A.C.), (x/m)**	
	Rang	**Mean**
Bentonite	0.0398 to 0.1334	0.0780
A.C.	0.0423 to 0.3646	0.1401

The kerosene removal efficiencies obtained at the equilibrium time (4 h) for bentonite and activated carbon are presented in table (7)

Table (7) Kerosene removal efficiencies for bentonite and activated carbon

Adsorbent	**Co (mg/L)**	**Ce (mg/L)**	**Kerosene removal %**
Bentonite	500	61.9	87.6
A.C.	500	34.1	93.1

The plot of kerosene concentration at equilibrium Ce versus quantities of adsorbent (bentonite or A.C.) is presented in figure (10). At the lower adsorbent mass (lower than 0.3 gm) the kerosene concentration decreased more sharply for activated carbon than bentonite, and then the decrease became gradual for both.

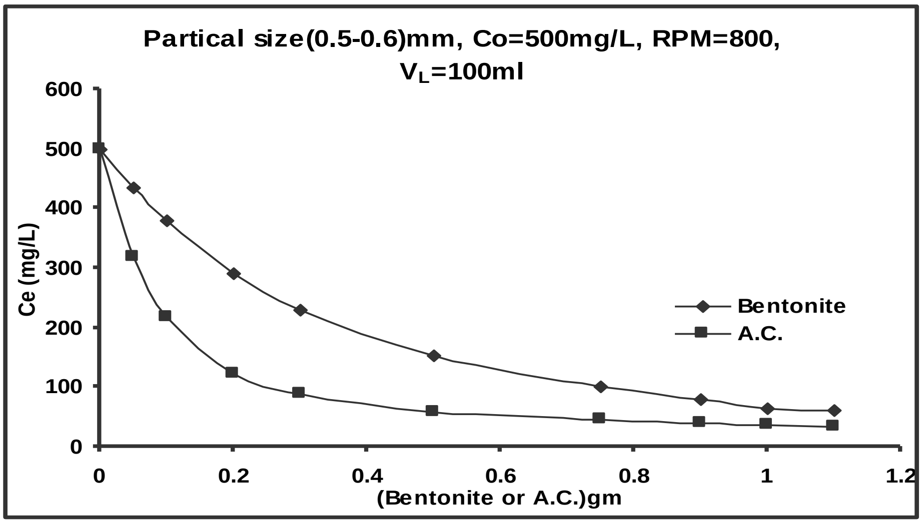

Fig. (10) Kerosene removal by bentonite and activated carbon

CONCLUTIONS:

- The equilibrium isotherm for removal of kerosene onto bentonite at various initial concentrations was of favorable type. The correlation coefficient shows that Freundlich equation fits the experimental data more than Langmuir equation.
- The sorption rate of bentonite increased with increasing initial concentration of kerosene.
- The batch results of kinetic studies of bentonite showed that the equilibrium time was four hours.
- It was found that increasing the mass of bentonite will increase the percentage of kerosene removal until (1.1) gm of bentonite for the given conditions [particle size= (0.5-0.6) mm, Co=500mg/L, RPM=250, V_L=100ml] due to the system reaches equilibrium state.
- The percentage of kerosene removal increased with increasing the agitation speed. The best removal is 87.4% for RPM = 800.
- The swelling of bentonite in water was more than its swelling in kerosene, which insure that bentonite is a hydrophilic substance.
- The equilibrium isotherm for adsorption kerosene onto activated carbon for various particle sizes was of favorable type. The correlation coefficient shows that Freundlich equation fits the experimental data more than Langmuir equation.
- The sorption capacity of activated carbon increased with decreasing particle size, because of increasing the surface area with decreasing the particle sizes.
- For a given conditions the sorption capacity and efficiency of activated carbon were higher than sorption capacity and efficiency of bentonite, Which means that the activated carbon was more active for removal of kerosene from waste water than bentonite. The activated carbon is non- selective for this purpose because the oil can blind the pore space of activated carbon during operation as well as it is a costly material. Therefore bentonite can be considered as alternative adsorbent.
- At the beginning of adsorption, the kerosene concentration decreased more sharply for activated carbon than bentonite, and then the decrease became gradual, indicating a higher sorption rate for activated carbon than bentonite.

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NOMENCLATURE:

a	Empirical constant (mg/mg)
b	Saturation coefficient (L/mg)
C_e	Concentration of contaminant remaining in solution at equilibrium (mg/L)
K_F	Freundlich equilibrium constant indicative of adsorptive capacity, (mg/mg)
m	Mass of sorbent (mg)
n	Freundlich constant indicative of adsorption intensity,(mg/L)
q_e	Contaminant concentration sorbed on the solid(mg/mg)
x	Mass of material sorbed on the solid phase (mg)