Performance of Activated *Ricinus Communis* Leaves for Degradation Oily Wastewater from Aqueous Solution

Wisam Shamkhi Jaber
Civil Engineering department,
Colege of Engineering,University of Babylon
Babylon/ Iraq
Email: w2011222@gmail.com

Abeer Ibrahim Alwared
Environmental Engineering department,
Colege of Engineering, University of Baghdad
Baghdad/ Iraq
Email: abeerwared@yahooo.com

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Abstract:

In this work, *Ricinus Communis* leaves used as adsorbent to eliminate dissolved oil in oil-water emulsion, the adsorbent was used in activated state, the influence of different parameters like initial oil concentration, adsorbent dosage, and contact time were examined in this study. The particle size used was (300 - 150 micron), at (pH < 2), 200 rpm. and adsorbent concentration of 400 mg/L, The oil uptake increased from 78 % to 88% for oil concentration of 2000 mg/L by using natural and activated respectively, the study was characterized by FTIR, SEM, and BET tests. FTIR test. Activated adsorbent proved good capability of adsorption aliphatic and aromatic hydrocarbons. Isotherm study had used to understand the mechanism of adsorption. Langmuir and Freundlich isotherm were fitted very well to describe the adsorption resulting by activated adsorbent, this study also characterized by kinetic models, Pseudo second order was the best fitted model for adsorption by adsorbent.

Key words: Oil in water; Activated *Ricinus Communis* leaves; Adsorption; pH: Contact time.

1.Introduction

The form of dissolved oil that take places in oily wastewater has considered as big problem attacked the environment and had negative effect on treatment plants. The removing of dissolving oil from solution is not easy process; this formation could pass by solution to different treatment units and decease the overall efficiency of that unit. The most known chemical compounds that lead to dissolve oil in water are detergents [1], in other words, detergents includes compounds named as "surfactants", these chemicals have both hydrophilic and hydrophobic ends [2]. The surface tension of oil molecules start to be decreased by the acting of hydrophobic end, these molecules attached to the surfactant and distributed in all water body because of the surfactant attach the water molecules by the other end (hydrophilic end) [3] the form resulting by dissolving oil in water named as oil-water emulsion [4].

Several environmental problems caused by oil when spilled or be in mixing with surface or ground water, this chance of this terrible case has increased day by day because of increasing the number of vehicles, machines, or any other devices using fuels and oils, for instance, many car wash-stations were found in Iraq, number of these stations released their wastewater toward sewers without any pretreatment [5].

Ricinus Communis is found widely in different zones, it can grow up in different weather conditions and types of soil, the plant had known popularly in healing some types of wounds [6]

Formation of activated carbon was important process for enhancing the ability of adsorbent to attach more amount of adsorbate, when a material be transformed to activated carbon, the porosity increased according to the activated process type, activated agent used, and type of adsorbent, two general process types of activation used: chemical activation were chemical agent (like KOH, H3PO4, NaOH, or H2SO4) be mixed with adsorbent in specific temperature, the other type is physical activation when activated takes place in high temperature by using stem or CO2 [7].

We had used Ricinus Communis leaves as adsorbent for removing oil in aqueous solution, maximum adsorbent capacity reached was 4.05 g/g [8]

In previous researches, activated form of Ricinus leaves had used in removal of Methyline Blue dye [9], also it used for removing Pb(II) from a solution by using H2SO4 as activated agent [10]

The aim of present study was investigation of using of activated Ricinus Communis leaves as inexpensive and environment friendly sorbent for degradation oil in aqueous solutions. Isotherm models of Langmuir and Fruendlich were used for describing the batch sorption recorded, also kinetic models were used to analysis the adsorption progress with time.

2. Materials:

2.1 Sorbent:

Sufficient quantity of Ricinus leaves had been collected from Al-Dolab village/ Hilla city/ Babylon province/ Iraq , the improper parts like stalks were removed from the plant, leaves were washed number of times with distilled water until no strange color be released, then withered in oven 100° C for 24 hr. the plant then sieved to particle sizes of (300-150) micron then placed in closed containers, the next stage was transforming of natural Ricinus to activated form, this process was done by two steps: first one was burning of the plant leaves after cleaning -as mentioned- at 300 C° for one hour then cooled to room temperature, the second step was mixing potassium hydroxide KOH with the result form of step one at ratios (g KOH/g adsorbent) of (0.5:1)

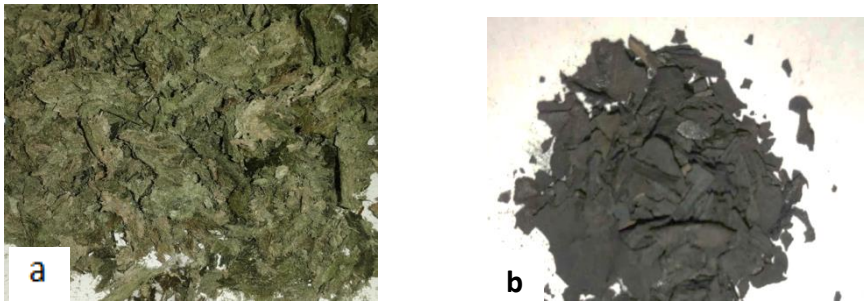

Fig.1 *Ricinus* leaves in a: natural form and b: activated form.

BET test according to ISO- 9277- 2010 was done in Iraqi Petroleum Research and Development Center, the specific area was for natural and activated forms was 1.1175 m2/ g, and 2.7246 m2/ g respectively.

In this study, analytical grade of NaOH and HCL solutions were used to adjust pH Sodium Dodecyl Sulfate (SDS) was used as surfactant, the chemical formula of this compound is NaC12H25SO4 in chemical formula-, it has hydrophilic end acting by hydrocarbon tail and Hydrophobic part denoted by sulfate and Sodium end, when SDS mixed with oil and water its hydrophobic end be in contact with oil drops, the surface tension of these drops start to decreasing so the drops be inside the water body as the surfactant has other end acts as water lover .

The simulation solution used in this study was prepared by mixing specific amount engine oil to solution contained water and SDS, the new solution was stirred at 200 rpm. several times until the final solution became milk-like and no oil drops be noticed at the surface: different oil concentration were used at this study to examined the ability of adsorbent. The properties of oil used was included in Table 1.

Table 1. Oil properties

Item Name	Oil
density	0.887 g/cm^3
flash point	218 °C
viscosity	1230 centipoise

2.2 Experimental work:

Batch study was carried out by placing samples of 200 ml of solution in number of flasks and be shacked at 200 rpm the influence of different parameters were examined in study like oil concentration, adsorbent/adsorbate dosage (g/g), contact time, and pH, the results were characterized by FTIR and SEM tests, and fitted with isotherm and kinetic models.

2.3 Residual Oil concentration Test:

The removal efficiency of oil was determined by using the liquid-liquid extraction method: EPA 1664A, the procedure was done using n-hexane as solvent [11], the adsorbent first removed from solution by using sieve with smaller openings than adsorbent size, after that n-hexane was mixed with the solution and stirred to ensure dissolving of oil in it, because the lighter density of n-hexane than water, it separated by using separator funnel and be collected in beaker then placed on hot plate to be heated at 70°C, the remaining after heating is the residual oil in solution so it weighted, the difference between this record and the initial oil weight was the amount of oil adsorbed [12].

3. Results

Oil uptake for natural form at pH=7 was recorded [8], the removal efficiency of both natural and activated form of adsorbent shown in Fig. 2 different experiments for activated form in batch study were done with different parameters to evaluate best conditions leading to high removal efficiency.

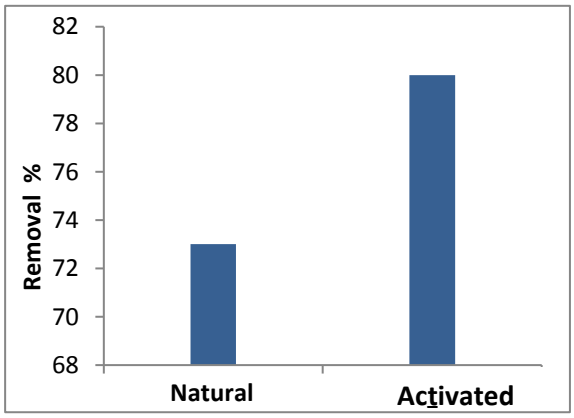

Figure 2.Comparison of removal efficiency between natural and activated *Ricinus* leaves

3.1 Stability

The stability of oil emulsion in different periods of time by the attendance of surfactant was studied. The solution was stable for 48 minutes, the oil drops then start to be in closeness case gradually to form more bigger drops relatively, the separated phase of oil drops moved toward the solution surface was noticed after 80 minutes, the stability life of the solution was illustrated in Fig.3.

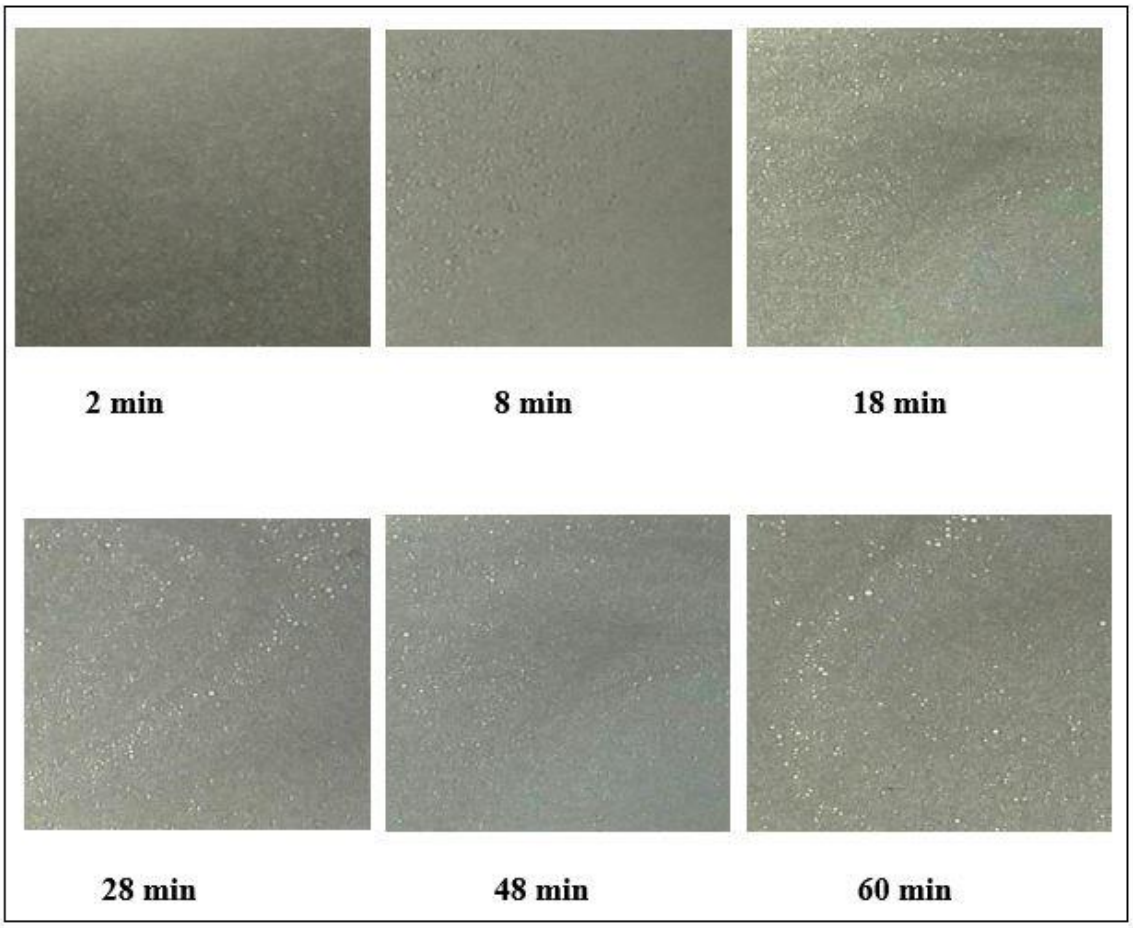

Figure 3. Oil emulsion stability at different periods of time.

3.2 Characterization tests:

3.2.1 FTIR test:

The study was characterized by FTIR spectroscopic test using (SHIMADZU FTIR, 8000 series spectrophotometer) at (Iraqi Minister of Science and Technology) for activated adsorbents before and after adsorption, the results were shown in Fig.4 , different peaks had appeared because of oil formed by many compounds , the main compounds were aliphatic and aromatic hydrocarbon that can be noticed in the diagram, non-hydrocarbons also found in oil structure such as nitrogen compounds and sulfur (like thiophene, pyridine, pyrrol, and sulfides) [13] [14].

According to test result, O-H group found as noticed by the peak in range (3200-3500 cm-1) when hydroxyl group bonded with hydrocarbon chain, the other group was C-H as shown by the peaks (2850-2990 cm-1), these group found in aliphatic hydrocarbon the main compounds of oil, range (1440-1625cm-1) also appears which referred to C=C bond in aromatic hydrocarbons, aliphatic hydrocarbon had also (C=C) bond noticed by (1620-1680 cm-1) peak range. Range of (1000-1300 cm-1) appeared in test was due to the function group of (C-O) because Oxygen be attached by hydrocarbons [15].

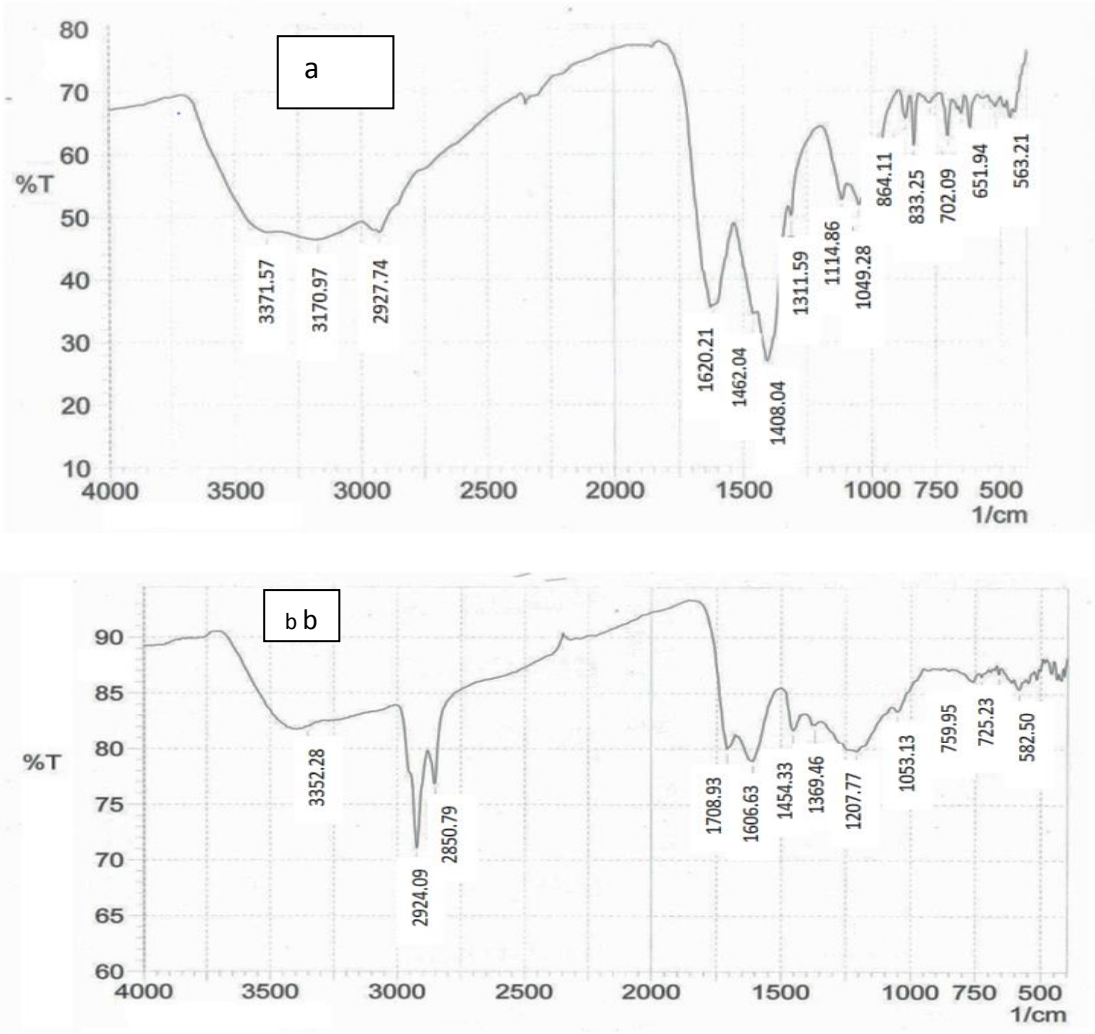

Figure 4.FTIR test result: (a) before adsorption, (b) after adsorption

3.2.2 SEM test:

The study was characterized by the test of Scan Electron Microscopic (Faculty of Pharmacy, Babylon University), it showed in Fig.5, the pores had appeared clearly, these pores increased the ability of the plant to adsorb more oil molecules by increasing the active surface of the plant to attach the adsorbate.

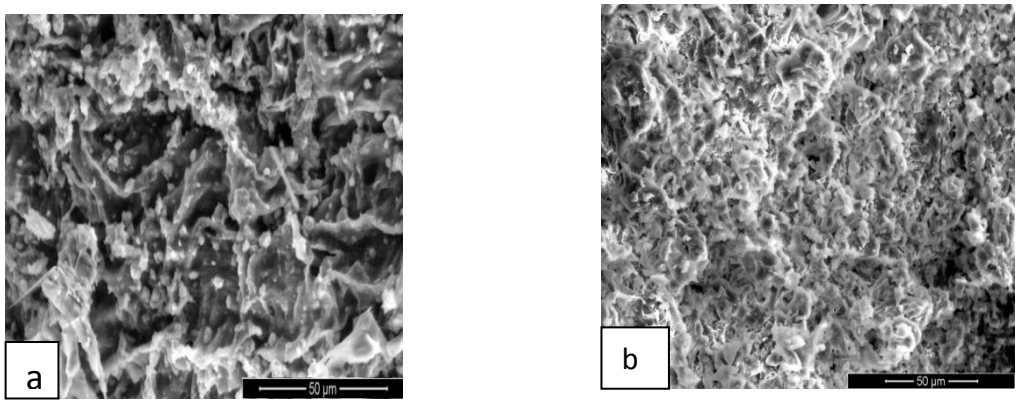

Figure 5.SEM test result: (a) before adsorption, (b) after adsorption

3.3 pH effectiveness on adsorption:

pH was noticed as effective parameter of the sorption progress, the difference in pH number led to changing in the surface characteristics of the bio-sorbent and ionization degree [16]. The effectiveness of pH was tested in the range of (2-10) while fixing other solution properties at same values. Fig.6 showed that when (pH<2), high removal of oil was recorded as to natural or alkalinity solution, the reason was that acidity was lead surfactant to be in weak condition reflecting on the bond between oil drops and the hydrophobic ends start to leave the surfactant structure, but this freedom was as shorter as they attached to the moving solid surface.[17]

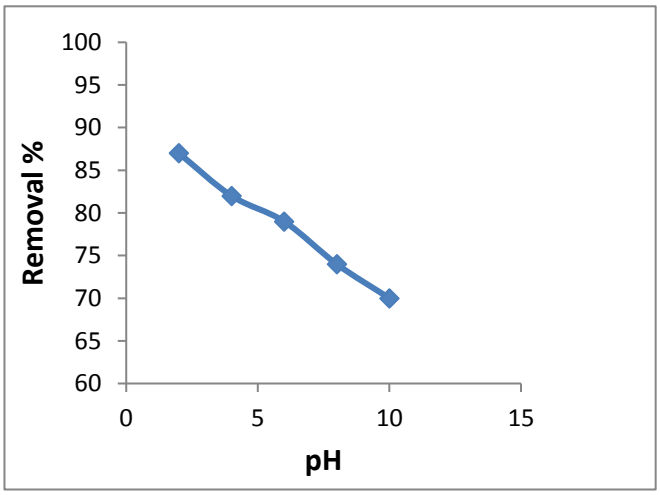

Figure 6. Relationship between removal of oil and pH

3.4 initial oil concentration affection :

The effectiveness of oil concentration was studied by using different amounts of oil while other parameters fixed at constant values, as shown in Fig.7, concentration of (250, 500, 1000, and 2000) mg/L were used to be adsorbed by (400 mg/L) of adsorbent, greater than before of oil concentration made the removal efficiency decreased because the solid that available in solution cannot uptake more than its adoption capacity when the amount of adsorbate increased at fixed dosage of adsorbent [18, 19].

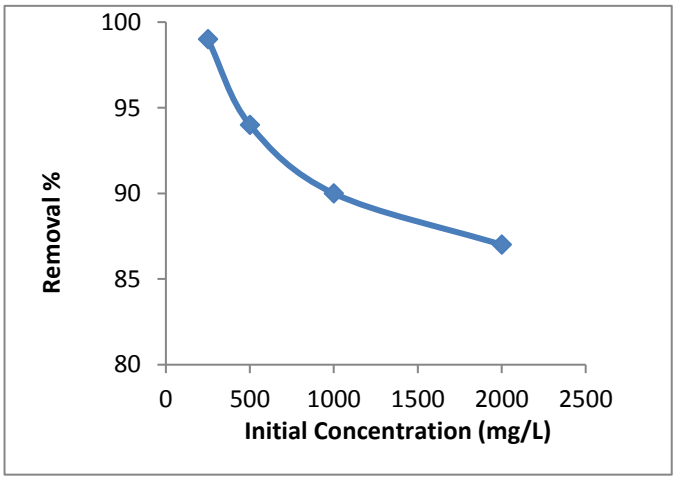

Figure 7.Relationship between removal efficiency and initial oil concentration

3.5 Effect of adsorption time:

The removal efficiency of oil in water by using activated Ricinus was shown in Fig.8, oil uptake was rapidly for 15 minutes from beginning of process then the slope of removal curve start to decrease meaning

that the mass transfer of oil became in lower rate, after 90 minutes of operation, the solution be in equilibrium state, the fact could be noticeable for the three oil concentrations used [19].

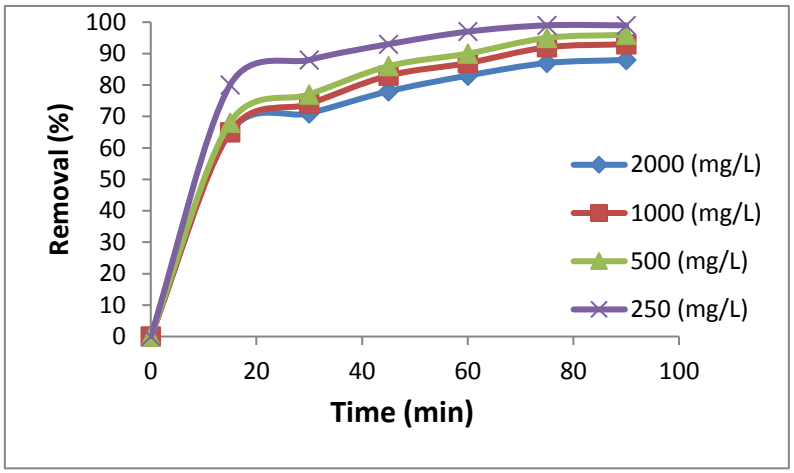

Figure 8. Relationship between removal of different oil concentrations and time

3.6 Effect of adsorbent dose:

The adsorbent dosage was good index about the capability of adsorbent for attaching great amount of oil during the process, adsorbent concentration of (400 mg/L) was used to treat the three concentrations, activated adsorbent showed the ability of this plant to uptake up to 88% of adsorbate in 90 minutes for oil concentration of (2000 mg/L). when more adsorbent weight was used, the removal increased relating to the more active solid available in solution be able to uptake more portion of adsorbate [19]. The effect of adsorbaent dosage were showed in Fig. 9.

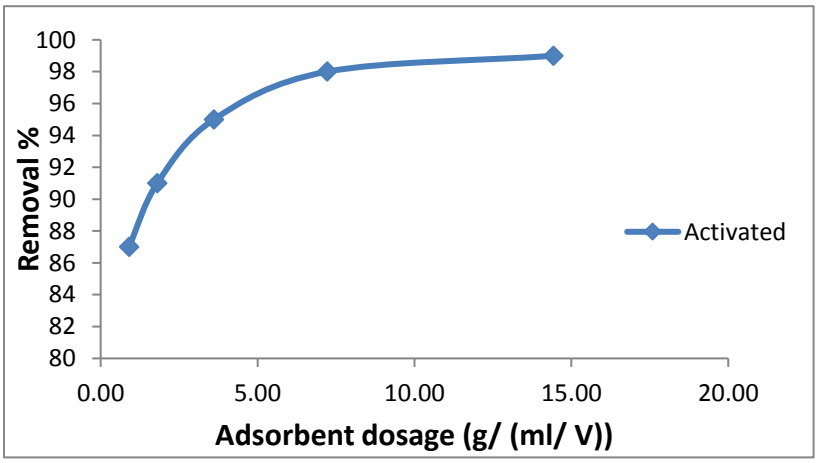

Figure 9. Relationship between removal efficiency and adsorbent dosage

3.7 Effect of temperature:

The influence of temperature on adsorption was studied at range of (20-60)°C, as shown in Fig.10, gradual eliminating was noticed in oil uptake when the temperature be changed from 20°C to 40 °C, then the decreasing was more noticeable as reached to lower removal efficiency at 60°C. the recorded values described the fact that the adsorbent was acted as exothermic [20], thermodynamic parameters were evaluated from the following formula [21]:

$$\log\left(\frac{qe}{Ce}\right) = \frac{\Delta S}{2.303\,R} - \frac{\Delta H}{2.303\,R\,T} \ldots(1)$$

$$\Delta G = \Delta H - T\,\Delta S \ldots..(2)$$

By using equations (1) and (2), the values of (ΔS) , (ΔH), and (ΔG) where (-58), (-24.28), and (17.26) respectively, the consideration of Ricinus as exothermic adsorbent was appeared as the negative sign of (ΔH) [22].

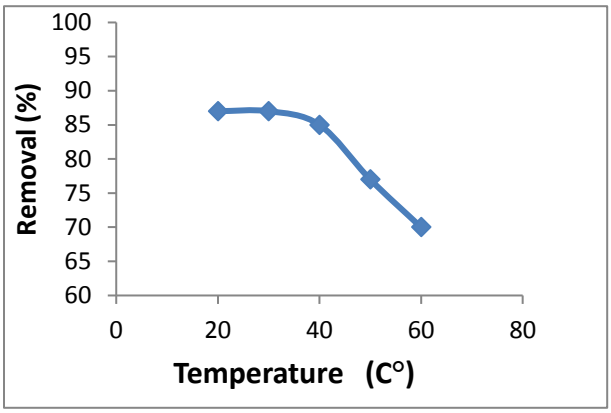

Figure 10. Relationship between removal efficiency and and temperature (C°)

3.8 Isotherm study:

The adsorption capacity representing by the weight of adsorbate that be adsorbed to that weight of adsorbent in equilibrium condition was examined by using well – known isotherm models: Langmuir model and Freundlich model, each model based on special assumption to describe the mechanism of adsorption.

3.8.1 Langmuir model:

The assumption of this model was single layer of adsorbate covers the adsorbent, [23], also the energy of adsorption assumed to be in same magnitude on the active solid surfaces [24] the important parameter that be evaluated in this model:"Q" the maximum uptake at equilibrium (mg adsorbate/ g adsorbent) "k" the adsorption rate (L of solution / mg of adsorbate), the equation of Langmuir model was [23] :

$$\frac{1}{qe} = \frac{1}{KL\,Q}\,\frac{1}{Ce} + \frac{1}{Q} \quad \text{.....(3)}$$

3.8.2 Freundlich model:

This model assumed that the solid surface available for adsorption is in heterogeneous form, the model used empirical equation to evaluate the rate of adsorption [25], the very noticeable thing in this model for distinguish it from Langmuir model was the assumption of multilayer of adsorbate that take place on the surface of adsorbent in other word the mass transfer of adsorbate toward the adsorbent structure continue to happen after a monolayer be adsorbed [26], Fruendlich isotherm equation was: [25, 27]

$$\text{Log qe} = \text{Log KF} + \frac{1}{n}\,Ce \quad \text{.....(4)}$$

The resulting data of experimental, Langmuir model, and Freundlich model were illustrated in Fig.11 and the parameters for the two models was included in Table 2, it can be seen depending i=on R2 , the data can be fitted very well with two models.

Table 2.Parameters values of Langmuir and Freundlich isotherm models

Isotherm model	Parameter	Value
Langmuir	Q	4382
	KL	0.0383
	R^2	0.9752
Freundlich	KF	58.955
	n	1.325
	R^2	0.9854

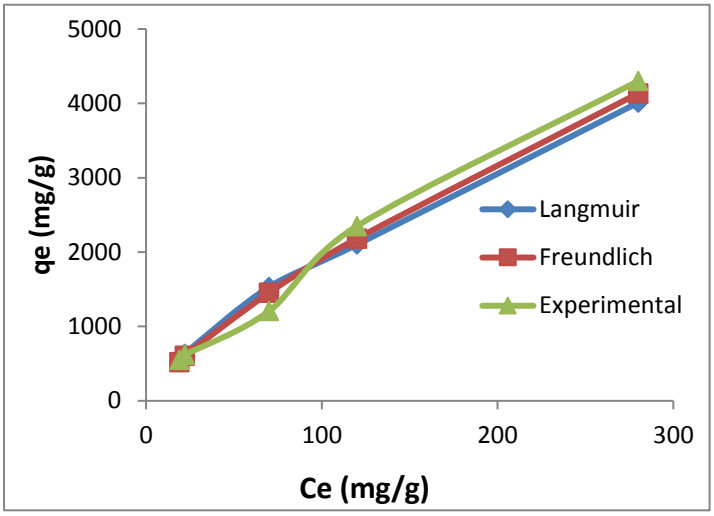

Figure 11. Data Comparison of experimental and isotherm models

3.9 Kinetics models:

Rate of adsorption represented by the amount of adsorbate that be attached by adsorbent per spesific period of time was studied, many models were used in this type of analysis according to the formula order used. Different values of removal efficiency per time were recorded at batch expermints.

3.9.1 Pseudo first order:

This model based on Linear formula to fit the increment of the amount of adsorbate that be taken by the adsorbent per spsesific time (qt) as shown in Fig.12, the model depends on the amount of adsorbate that be adosrbed at equilibrium state (qe), the equation equation of this model was [28]:

$$Ln(q_e-q_t)= \ln qe-K_1*t \ldots\ldots\ldots (5)$$

3.9.2 Pseudo second order:

The model describs the case when amount of adsorbate in that adsorbed was not in linear relashionship, the model covers the influencing of pore size on adsorpstion, in other word, fiiting of adsorption results with this model means the asorption process was controlled by diffusion of adsorbate molecules inside the pores of the solid particles [29].

To analysis data with this model, plot of linear form was adopted as following:

Linear form equation [30]:

$$\frac{t}{qt} = \frac{1}{k2\ qe^2} + \frac{1}{qe}t \ldots\ldots (6)$$

Plot of linear form of this model showed best fitting than previous model, this result mean the adsortion took place in non linearty increasing and it controlled by the diffusion inside pores and grooves in the structure of adsorbent surface.

The results in Fig.13 showed good fitting to the experimental adsorption data for concentration ranges between (250-2000) mg/L with time, these obeying illustrated linear increasing of adsorbed quantity of oil on the solid surfaces of adsorbent.

The kinteic parameters for both models were evaluated as showed in Table 3.

Table 3.kinteic parameters:

Oil concentration (mg/L)	qe (mg/g) experimental	Pseudo fisrt order			Pseudo second order		
		K1 (l/min)	qe (mg/g)	R^2	K2 (g/mg min)	qe (mg/g)	R^2
250	625	0.044	265	0.964	0.287	657	0.998
500	1210	0.049	999	0.971	0.074	1333	0.999
1000	2350	0.046	1860	0.976	0.036	2596	0.999
2000	4350	0.046	2821	0.947	0.021	4840	0.999

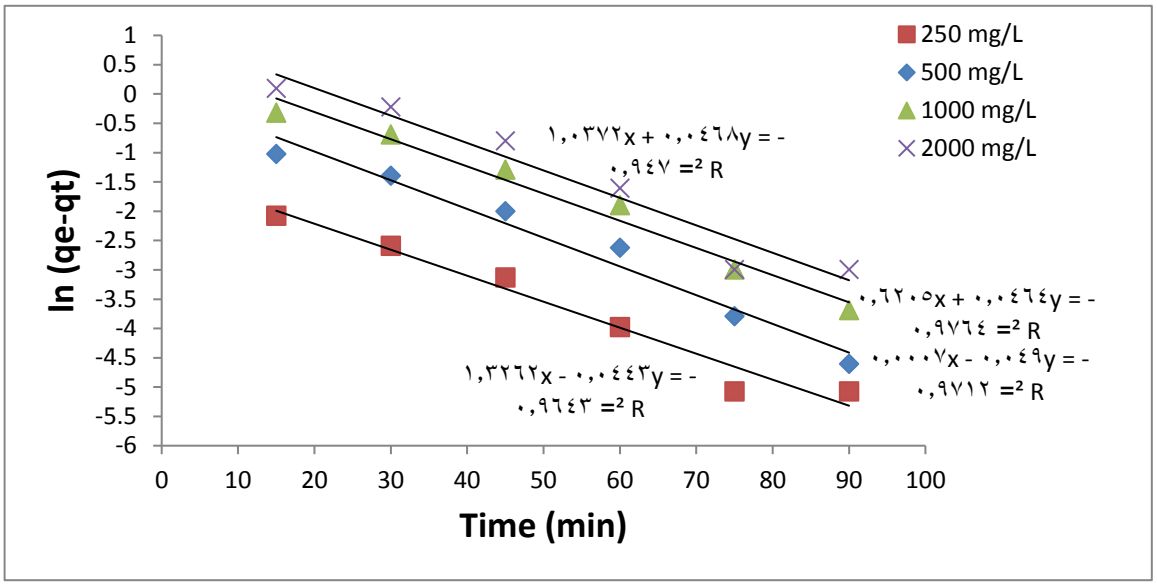

Figure 12. Pseudo 1st order kinetic model for oil adsorption

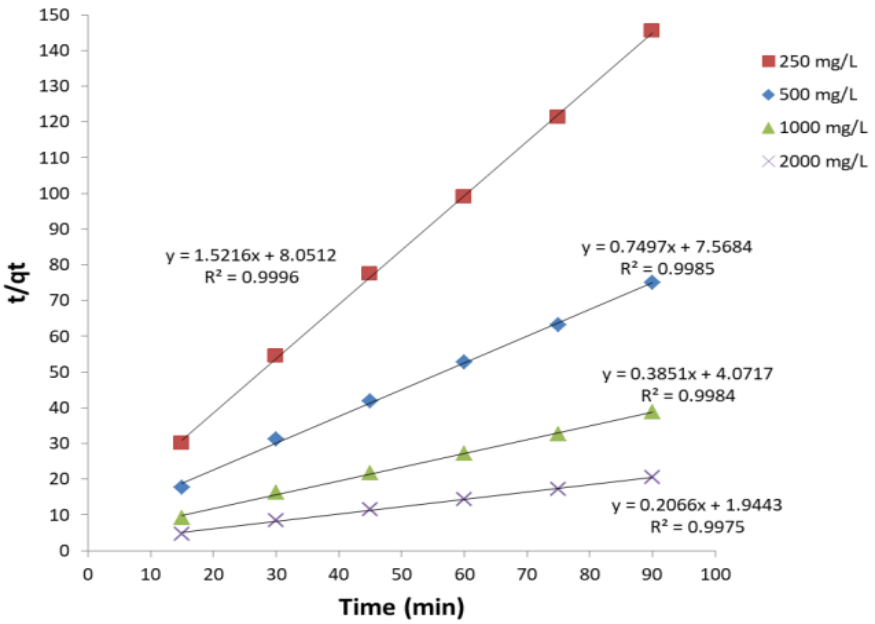

Figure 13. Pseudo 2nd order kinetic model for oil adsorption

Conclusion:

Oil in water emulsion can be formed by mixing of oil with water body by attendence of surfactants, the result solution contained dissolved oil which hard to be treated in conventional units, Ricinus leaves (150-300) micron showed good ability to adsorbed this kind of pollutants in acidified solution, Both of Langmuir isothrm and Freundlich isotherm were best fitted the adsorption process, rate of quantity adsorped increased in nonlinear form as the results of kinetic models adopted in this research, Psedo second order model well fitted the adsorption rate than first order model.

Abbreviation:

Ce: Remaining concentration of adsorbate in solution (mg/L)
k1: Pseudo first order constant
k2: Pseudo second order constant
KB: BET adsorption rate (L/mg)
KF: Freundlich isotherm adsorption constant
KL: Langmuir isotherm constant (L/mg)
n: Adsorption intensity according to Freundlich isotherm model
Q: Maximum adsorption capacity according to Langmuir isotherm model (mg/g)
qe: Adsorbate that be adsorbed per weight of adsorbent (mg/mg)
qs: Maximum adsorption capacity according to BET isotherm model (mg/g)
qt: Adsorption capacity after specific time (mg/g)
t: Time (min)
V : volume of water
ΔG Gibbs free energy $KJ.mol^{-1}$
ΔH Enthalpy $J.mol^{-1}$
ΔS Entropy $J.mol^{-1}$

Conflicts of Interest

The author declares that they have no conflicts of interest.

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أداء اوراق نبات الخروع المفعلة في تحلل دهون مياه الصرف الصحي من المحاليل المائية

وسام شمخي جابر
قسم الهندسة المدنية،
كلية الهندسة، جامعة بابل،
بابل/ العراق

عبير ابراهيم الورد
قسم الهندسة البيئية،
كلية الهندسة، جامعة بغداد،
بغداد/ العراق

الخلاصة

في هذا العمل، تم استخدام اوراق نبات الخروع كمادة مازة لازالة الدهن الذائب في محلول مستحلب الدهن– الماء، المادة المازة تم استخدامها بحالة التفعيل، في هذه الدراسة تم اختبار تأثير مختلف العوامل مثل تركيز الدهن الابتدائي، جرعة المادة المازة، ووقت الامتزاز، حجم الحبيبات المستخدمة للمادة المازة كان (150–300) مايكرون وقيمة الأس الهايدروجيني اقل من 2 ضمن 200 دورة بالدقيقة وتركيز للمادة المازة 400 ملغرام/ لتر. نسبة كمية الدهن الممتزة ازدادت من 78% الى 88% عند تركيز اولي للدهن 200 ملغرام/ لتر عند استخدام مادة مازة بالصورة الطبيعية والصورة المفعلة على التوالي. تم اجراء الفحوصات الخاصة بالمساحة السطحية BET وامتصاص الطيف FTIR والمجهر الالكتروني الماسح SEM، ان المادة المازة المفعلة اثبتت قدرة جيدة في امتزاز المركبات الاليفاتية والاروماتية، تم دراسة مدى تطابق نتائج الدراسة مع الموديلات الخاصة بسعة الامتزاز ووجد ان كلا من Langmuir model وكذلك Freundlich model يمكن استخدامهما لوصف الامتزاز بالمادة المفعلة، تم دراسة كمية المادة الممتزة مع الزمن وتم التوصل الى ان Pseudo Second order هو الموديل الافضل لوصف تغير مقدار الدهن المتز مع الزمن.

الكلمات الدالة: الدهن في الماء، اوراق نبات الخروع المفعلة، الامتزار، الدالة الحامضية، زمن التماس.