Research Article

Removal of oil emulsion from aqueous solution by using *Ricinus communis* leaves as adsorbent

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Abstract
Waste engine oil is a highly pollutant material that requires responsible management. Waste engine oil may cause damage to the environment when dumped into the ground or into water streams including sewers. The *Ricinus communis leaves* are one of the common wastes abundantly available in Iraq. The present study aims to investigate the performance and effects of *Ricinus communis* leaves as adsorbent for removing dissolved oil in oil–water emulsion, and the study investigated the effect of different parameters like particle size, pH, initial oil concentration, contact time, adsorbent/adsorbate (g/g) ratio. The maximum removal efficiency of *Ricinus communis* leaves was 98% at adsorbent particle size (150–300) micron, acidified solution (pH < 2), 90 min for adsorbent concentration of 400 mg/L. Several tests like BET, FTIR and SEM had been conducted for best interpretation of adsorption mechanism in this study. FTIR test displayed good ability of the adsorbent in adsorption of aliphatic and aromatic hydrocarbons. When isotherm study was applied to the results, best fitted data were close to Freundlich isotherm, and the results also had been fitted with kinetic adsorption equations; in the present work, pseudo-second-order equation was the best equation of oil engine uptake.

Keywords Oilly wastewater · Batch adsorption · *Ricinus communis* leaves · FTIR · SEM

Abbreviations

- ΔG Gibbs free energy (KJ mol^{-1})
- ΔH Enthalpy (J mol^{-1})
- ΔS Entropy (J mol^{-1})
- Ce Remaining concentration of adsorbate in solution (mg/L)
- q_e Adsorbate that is adsorbed per weight of adsorbent(mg/mg)
- K_L Langmuir isotherm constant (L/mg)
- Q Maximum adsorption capacity according to Langmuir isotherm model (mg/g)
- n Adsorption intensity according to Freundlich isotherm model
- K_F Freundlich isotherm adsorption constant
- K_B BET adsorption rate (L/mg)
- q_s Maximum adsorption capacity according to BET isotherm model (mg/g)
- q_t Adsorption capacity after specific time (mg/g)
- t Time (min)
- k_1 Pseudo-first-order constant
- k_2 Pseudo-second-order constant

1 Introduction

The problem associated with dissolved oil in water has played a negative role in the surrounding environment. This form of oil cannot be removed easily as floating form so it could pass treatment units and decrease their efficiency. Dissolved oil can form in oil by the presence of detergents [8]; in other words, because detergents contain compounds called "surfactants," these compounds contain two ends: hydrophobic and hydrophilic [45]. Hydrophilic end attached to water molecules and the other end attached to oil molecules lead to decrease the surface tension of oil drops and disperse them all over

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solution, especially when there is mixing force like high-speed flow or high pressure [23]. The result is usually known as oil-in-water emulsion [22]. Oil-contaminated wastewater has led to several environmental problems because of its hazardous nature. The volume of oil-contaminated wastewater has increased in streak with the number of petroleum stations required to supply for the increasing number of vehicles. In Iraq, there are more than 500 car-wash facilities, excluding the region of Kurdistan, and some of them release their wastewater as an emulsion to the sewer system before any treatment [32]. Different treatment methods of oil in water were adopted in past time, and one of these methods was liquid–liquid extraction using toluene, ethanol and butanol by adding specific amount of these solvents to oil–water emulsion to extract oil molecules; the validity of these methods depends on type and amount of extracted solvent [36], and the other method was by using centrifugal force to enhance discounting of oil drops from solution toward the upsurface to be removed and then by skimming [19]. Adsorption of oil in water was done in several researches using different adsorbents such as potato peels [43], eggshell [1], bentonite [34] and organoclay [42].

Ricinus communis grows up rapidly up to 6 meters in height which contains leaves with average size of 30 cm [21], and it has resistance to draught and could grow up in different types of soil [39] and is the source of castor oil which contains ricinoleic acid in amount of 85–90%. Ricinoleic acid ((12-hydroxy-cis-9-octadecenoic acid) contains hydroxyl groups and can be hydrogenated to form ricinoleic acid methyl ester which could be reacted with chlorosulfonic acid to produce sodium methyl ester sulfonate that can be used as low-cost sulfonated surfactant [4, 6].

Ricinus had been used for healing accumulation of bloods under skin and wounds [46]; in previous researches, *Ricinus communis* waste was used for adsorption of Ni^{+2} [26] and treating Methylene Blue dye [27]. The aim of the present work was the investigation of the adsorption behavior of *Ricinus communis* leaves as environmentally friendly and inexpensive cost sorbent for the degradation of oil engine emulsion from aqueous solutions. Langmuir, Freundlich and BET models were used for explaining the experimental sorption records. The data were fitted with different types of kinetic models to comprehend the mechanism of biosorption.

2 Materials and methods

2.1 Sorbent and chemicals

Ricinus communis leaves had been collected in sufficient quantity from Al-Dolab village, Hilla city, Babylon province, Iraq, which are available at much quantities and then sorted from any other unwanted pieces of stalks attached to them. Leaves were cleaned several times with distilled water until no foreign color was noticed, dried in an oven at 100° C for 24 h, allowed to be cooled at room temperature after sieving to particle sizes of 4760–2360, 2360–1200, 1200–600, 600–300 and 300–150 microns (Fig. 1) and then packed into stoppered bottle until use.

For evaluating whether there remains amount of castor oil in the particle solid textures, 80 mg of Ricinus particles at size of 300–150 micron was placed in a flask which contained 200 mL of distilled water and was shaken at 200 rpm for 90 min; then, the sample

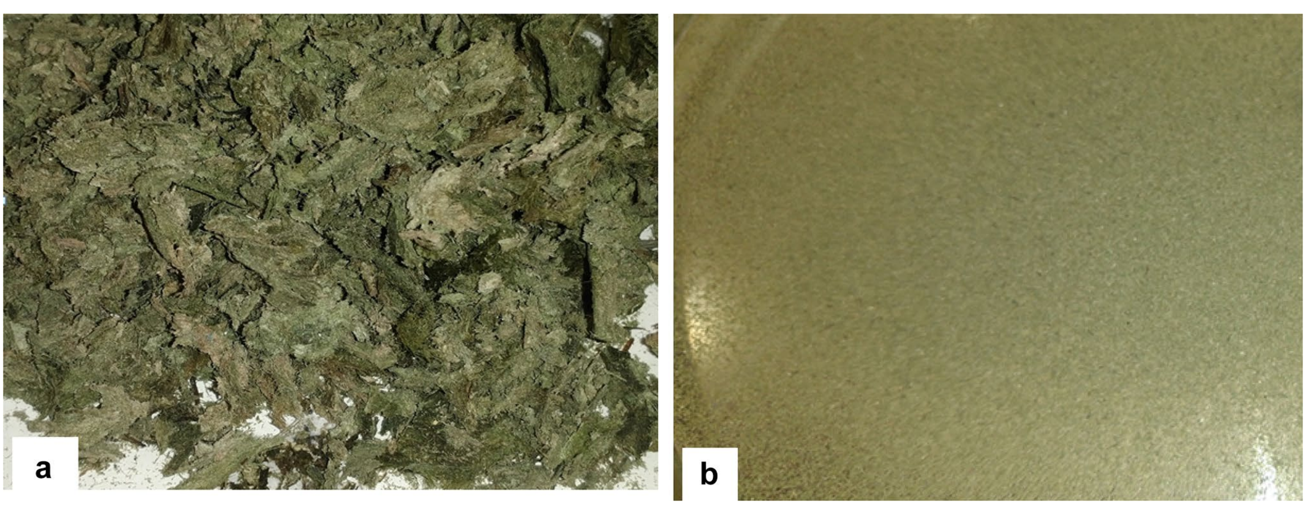


Fig. 1 *Ricinus* leaves **a** before sieving, **b** after sieving

of solution was tested for COD by Lovibond COD test and the result was zero, meaning the particles could be used in adsorption without any effect on the solution characteristics.

BET test analysis was performed according to ISO-9277-2010 in Iraqi Petroleum Research and Development Center, and the surface area was 1.1175 m^2/g for particle size of 300–150 micron. All chemicals used in this study were of analytical grade. HCL and NaOH solutions were used to adjust pH. Oil engine was used as pollutant source with characteristics shown in Table 1. Surfactant compound used in this study was sodium dodecyl sulfate (SDS)—$NaC_{12}H_{25}SO_4$ in chemical formula—it has two ends: hydrophobic part, represented by sulfate and sodium, and hydrophilic part, represented by hydrocarbon tail; when surfactant mixed with water and oil, hydrophobic part of surfactant will be attached with oil drops leading to decrease their surface tension and spreading them in water body as the other surfactant part is water lover.

The oil emulsion was prepared with the desired concentrations of 250,500, 1000 and 2000 mg/L by injecting the required volume of oil and SDS into specific volume of distilled water and then stirring at 200 rpm for about 15 min; after that mixing, oil drops disappeared from water surface and water solution and then milk-like solution was obtained, which exhibited the characteristics of chemically stabilized solution. Diameter distribution of the emulsion was measured at different time intervals to ensure the stability using microscope, and the scanning cycles were set to three times to ensure the accuracy.

2.2 Experimental work

Two hundred milliliters of oil-in-water emulsion in suitable flask was placed in shaker (Jlab model) at 200 rpm, and several parameters were changed in batch study such as contact time, pH, particle size, oil concentration, adsorbent/adsorbate dosage (g/g). Scanning electron microscope (SEM) test for determining the pore sizes and adsorption morphology appearance was conducted, and also FTIR test was conducted for *Ricinus* leaves before and after adsorption process. Adsorption results data were used for fitting adsorption mechanism with best isotherm and kinetics equations.

Table 1 Characteristics of oil engine

Item name	Oil engine
Density	0.887 g/cm^3
Flash point	218 °C
Viscosity	1230 centipoise

2.3 Oil content test

EPA 1664A method was adopted in this research to evaluate oil content in oil–water emulsion after adsorption, and this method is liquid extracted method using n-hexane to dissolve and extract oil in suitable funnel [14]; then, n-hexane layer can be easily separated in beaker from solution because of its lighter density. n-hexane solution in beaker was heated on hot plate at 70 °C and distilled to be used again while oil remained in bottom of the beaker to be weighted, and difference between that weight and weight of blank one was the weight of dissolved oil in solution [28].

3 Results and discussion

Different experiments in batch study were conducted with different parameters to evaluate best conditions leading to high removal efficiency.

3.1 Stability

As can be seen from Fig. 2, the stability of oil engine emulsion was studied under different time intervals with 200 rpm and the size of oil engine droplet is quite stable up to 48 min and then exhibits slight instability later and finally oil engine droplet is separated into the oil and internal phases after 80 min.

3.2 Characterization studies

3.2.1 FTIR test

The FTIR spectroscopic analysis was performed using Shimadzu FTIR 8000 series spectrophotometer at Iraqi Ministry of Science and Technology for *Ricinus communis* leaves before and after adsorption, and the results are shown in Fig. 3; it can be seen from this figure that as oil is a mixture of different compounds, the FTIR spectrum reveals numerous principal functional groups assigned according to the wave numbers of their peaks. This figure shows peak in the range of 3200–3500 cm^{-1} and this was due to O–H groups found in oil mixture when hydrocarbon chain attached hydroxyl group toward its molecular structure, and other peak was in the range of 2850–2990 cm^{-1} which represented C–H groups found in aliphatic hydrocarbon the major oil compounds, and also the other range (1620–1680 cm^{-1}) supported this fact because it refers to (C=C) group in aliphatic hydrocarbon. Range of 1440–1625 cm^{-1} also appears in this test which refers to C=C bond in aromatic hydrocarbons the other content of oil, (C–O) bond can be noticed also by the range

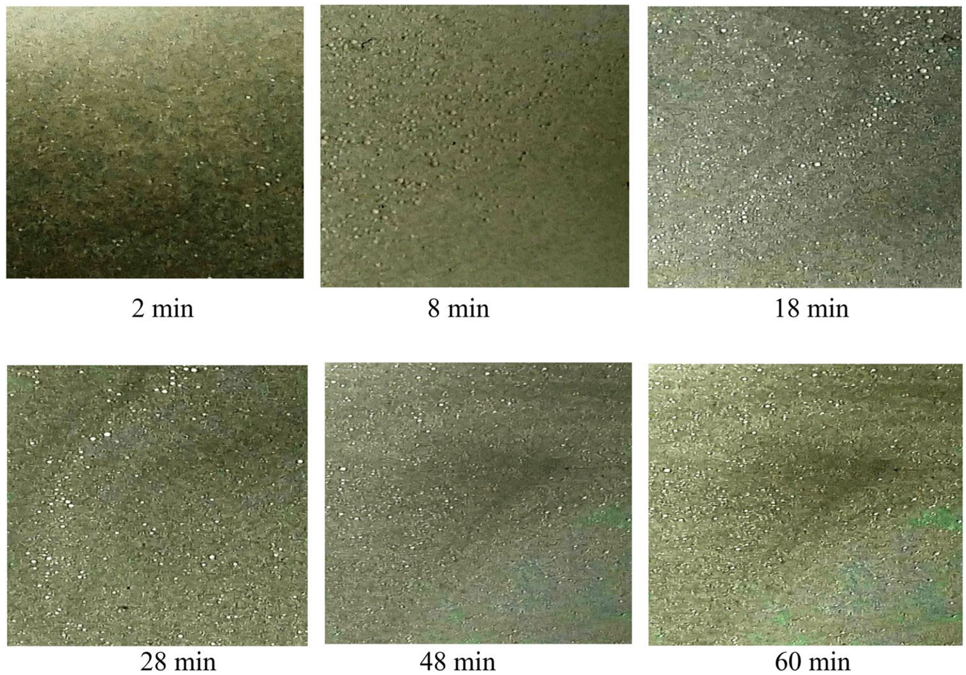


Fig. 2 Stability of oil engine emulsion at different time intervals

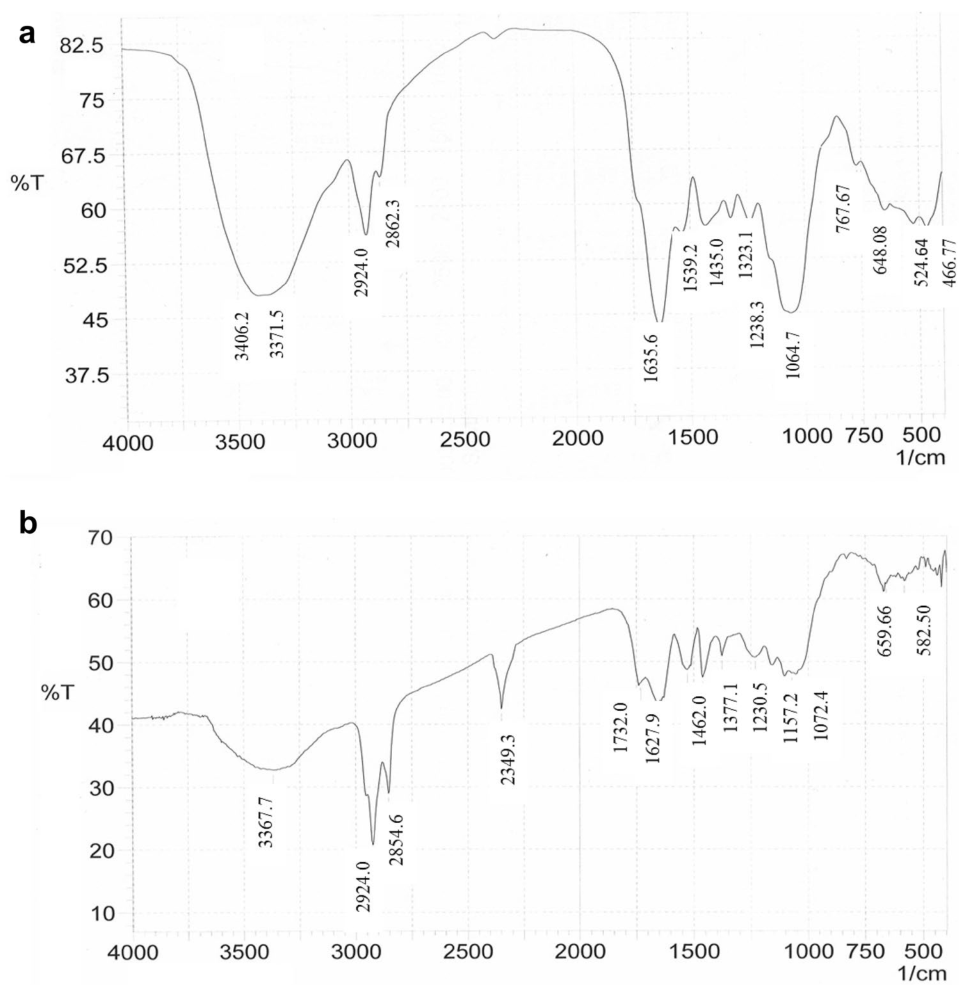


Fig. 3 FTIR test result: **a** before adsorption, **b** after adsorption (adsorbent (g)/adsorbate (g)) = (0.2), time = 90 min and pH = 2)

(1000–1300 cm^{-1}) indicated that some hydrocarbons attached Oxygen atom to their structure [24].

3.2.2 SEM test

Scanning electron microscopic test was conducted at Faculty of Pharmacy, Babylon university, and the attachment of oil molecules on the surface of adsorbent is clearly shown in Fig. 4. Grooves appeared in the texture of *Ricinus* leaves, and they provided good zones and attachment toward adsorbate to form dense layer and cover the texture.

3.3 Effect of particle size

Different particle sizes ranging from 150 to 4760 micron were tested in this study while other parameters were kept constant (pH = 2, 90-min contact time and with 1:5 adsorbent g/adsorbate g), and their results are presented in Fig. 5, which reveals that the removal efficiency decreased with increasing particle size, which is because more smaller particle and more specific area provided for solution and also less mass resistance provided to surrounding stream [3]. This finding was with agreement

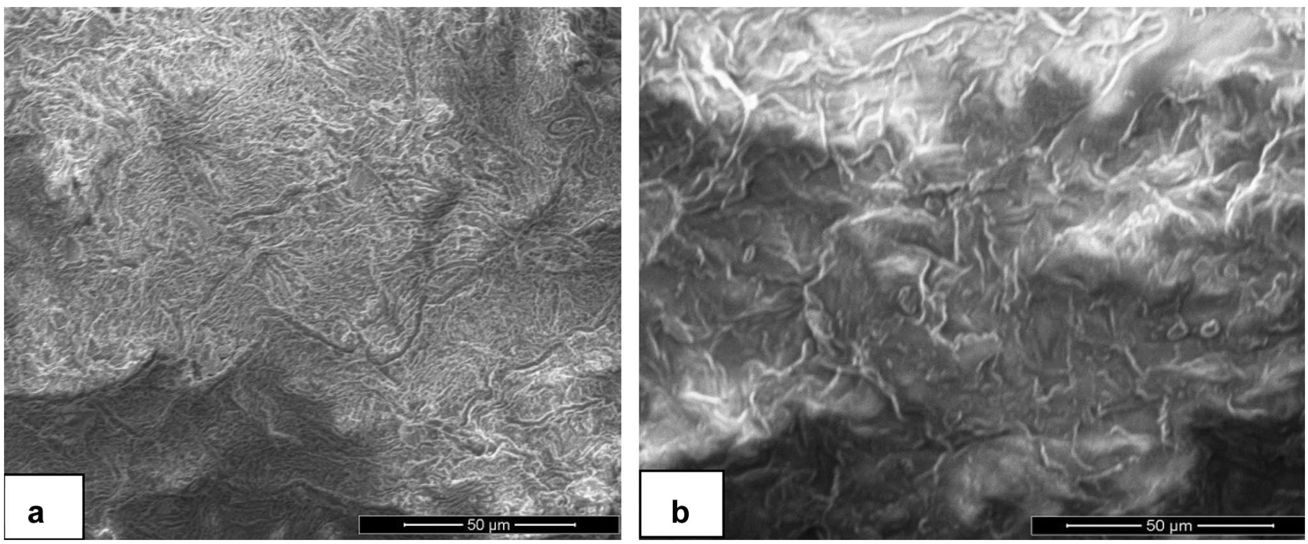
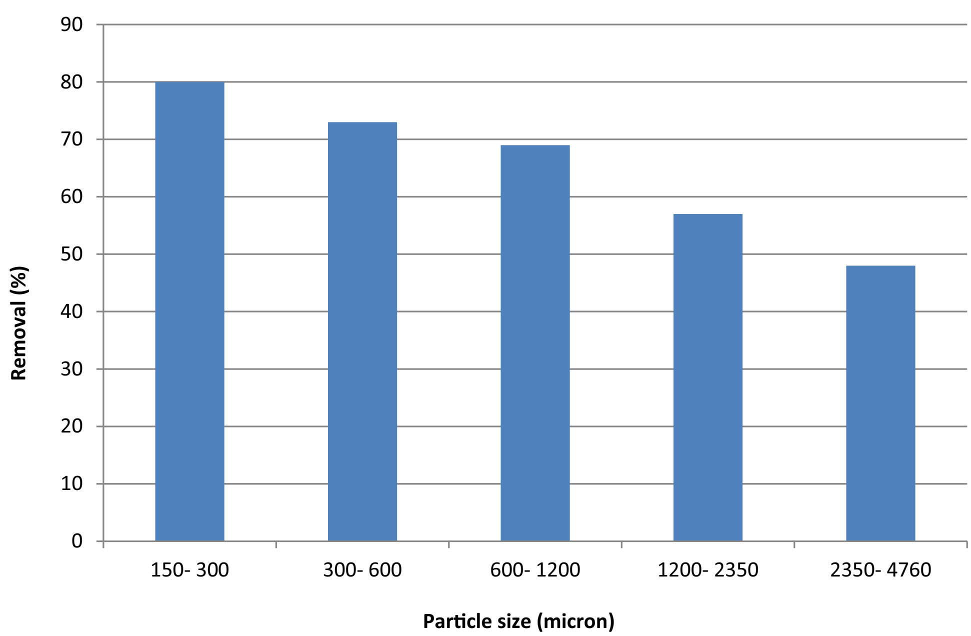


Fig. 4 SEM test result: **a** before adsorption, **b** after adsorption (adsorbent (g)/adsorbate (g)) = (0.2), time = 90 min and pH = 2)

Fig. 5 Relationship between degradation of oil by fresh *Ricinus* and particle size



with finding of [7] and [35]. So particle size in the range of 150–300 micron was adopted in this study.

3.4 Effect of pH

The efficiency of the sorption process is pH dependent owing to the fact that the variation in the pH value leads to difference in the surface properties of the biosorbent and degree of ionization [2]. pH in the range of 2–10 was tested while keeping other parameters constant, and their results are presented in Fig. 6, which shows that acidified solution leads to high removal of dissolved oil compared to moderate or alkalinity state because acidity made attachment of oil drops by surfactant in weakness case so this drops start to separate and catch by solid surfaces of motioned adsorbent particles [17]. These results were in agreement with the finding of [13] who found that increase in pH value leads to decrease in the removal efficiency.

3.5 Effect of initial oil concentration

Different concentrations of oil engine (250, 500, 1000, 2000 and 4000) mg/L with adsorbent mass/adsorbate mass equal to 0.2 were used while other parameters were kept constant, and their results are presented in Fig. 7, which shows that increase in oil concentration leads to decreased removal efficiency because fixed adsorbent dosage means solid surfaces of particles reached their capacity before attaching another quantity of oil content [15, 34]. These results were in agreement with the finding of [3, 37].

3.6 Effect of contact time

As shown in Fig. 8, *Ricinus* leaves showed removal efficiency increased rapidly for the first 15 min and then slightly increased until reaching its maximum for about 60 min; rapid mass transfer of oil molecules starts by separating and attaching toward the solid surface, building textures of adsorbate molecules on adsorbent surfaces; after that, solid surfaces of particles starts to reach their capacity so removal curve starts to be less effective by time; in this state, the amount of uptake is near zero and adsorption process reaches equilibrium [15].

3.7 Effect of adsorbent dose

From the economic point of view, the evaluation of optimum biosorbent quantity happens to be one of the vital characteristics. Therefore, the dependency of oil engine biosorption on the biosorbent content was investigated by varying the dose of *Ricinus* leaves particles as can be seen in Fig. 9 which shows good removal efficiency in small adsorbent/adsorbate (g/g) ratio; more than 80% of oil content was removed in 90 min by adsorbent/adsorbate ratio equal to 0.2, and increase in adsorbent weight leads to more available solid surface to more attached oil drops [15]. when the concentration of adsorbent was 400 mg/L and concentration of both surfactant and adsorbate was 2000 mg/L, removal efficiency reached 94%. The adsorbent dosage used was less than that used in different previous researches: 1800 mg/L [30] and 4000 mg/L [5].

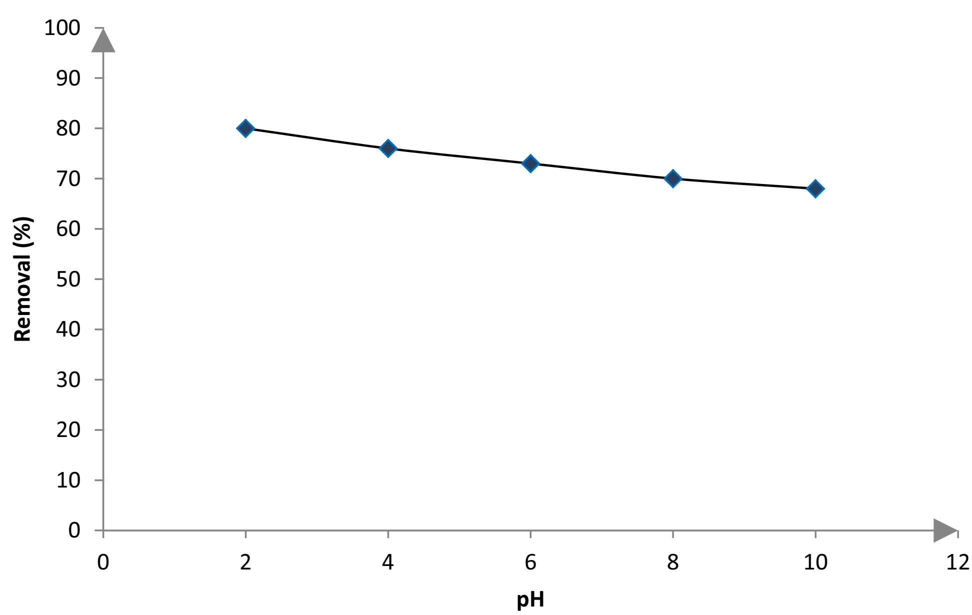


Fig. 6 Relationship between removal of oil by fresh *Ricinus* and pH

Fig. 7 Relationship between removal of oil by fresh *Ricinus* leaves and initial oil concentration

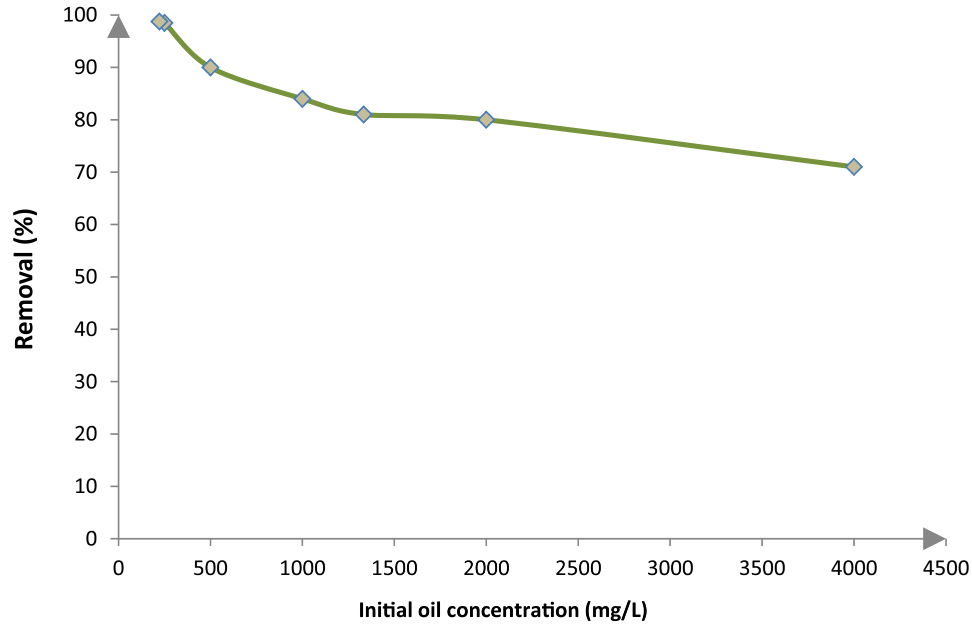
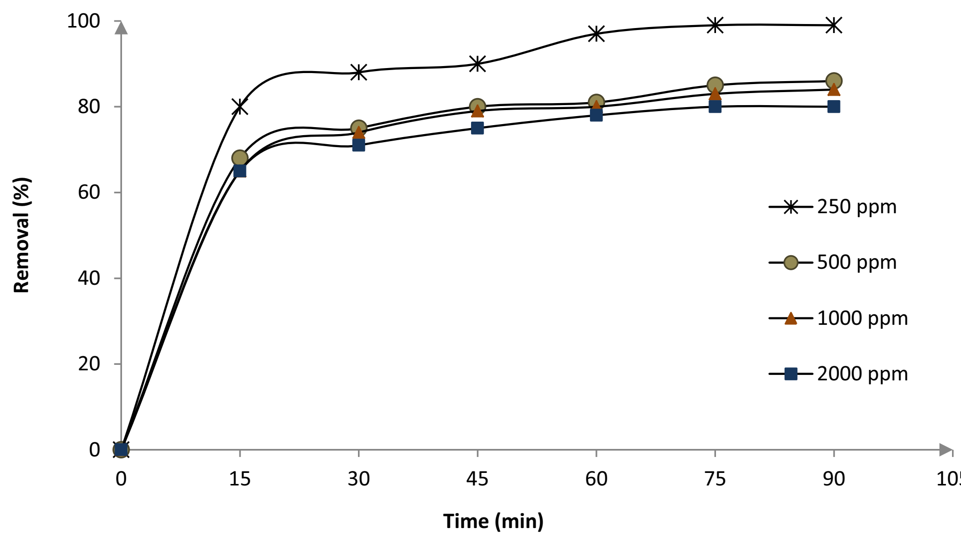


Fig. 8 Relationship between removal of different oil concentrations by fresh *Ricinus* and time



3.8 Effect of temperature and thermodynamics

The effect of temperature on adsorption process was investigated in the range of 20–60 °C, and their results are shown in Fig. 10. It can be seen from this figure that slight decrease in oil uptake was noticed by increasing temperature from 20 to 40 °C, and then the oil uptake continues to decrease noticeably by increasing the temperature to 60 °C. The decrease in removal efficiency by increasing temperature means *Ricinus* particles acted as exothermic adsorbent, and the mass transfer at heating up to high temperature starts to be reversed so portion of oil molecules had been separated from solid surface of adsorbent.

The thermodynamic parameters including change in Gibbs free energy ΔG, enthalpy ΔH and entropy ΔS were determined by using the following equations [41]:

$$\log\left(\frac{q_e}{\mathrm{Ce}}\right) = \frac{\Delta S}{2.303R} - \frac{\Delta H}{2.303RT} \tag{1}$$

$$\Delta G = \Delta H - T\Delta S \tag{2}$$

where R is the gas constant = (8.314 J/mol K) and T temperature in (°K).

By using Eqs. (1) and (2), the values of (ΔH), (ΔS), and (ΔG) where (− 10.45), (− 10.14), and (3.06) respectively, the negative sign of (ΔH) confirm the exothermic nature of *Ricinus* [44].

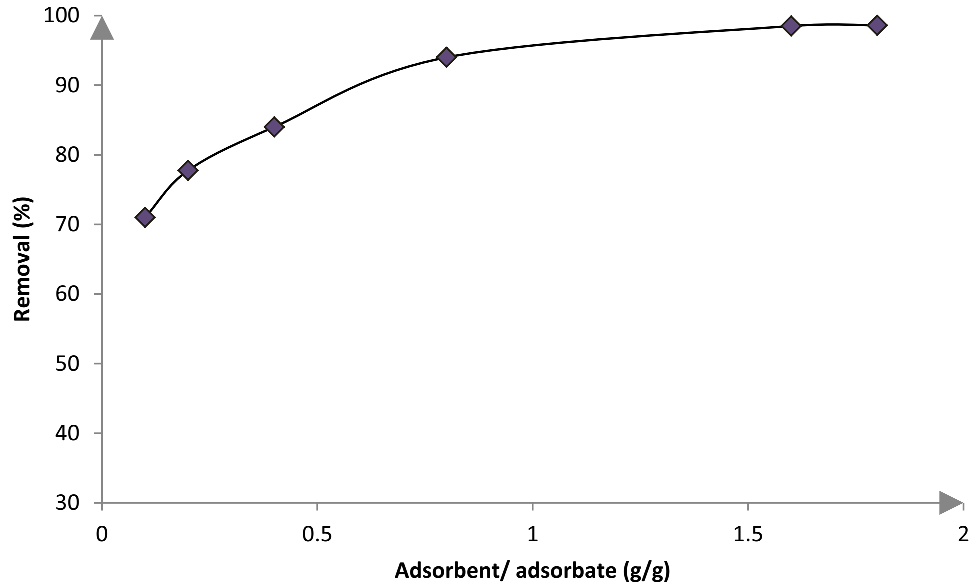


Fig. 9 Relationship between removal of oil by fresh *Ricinus* and adsorbent dosage

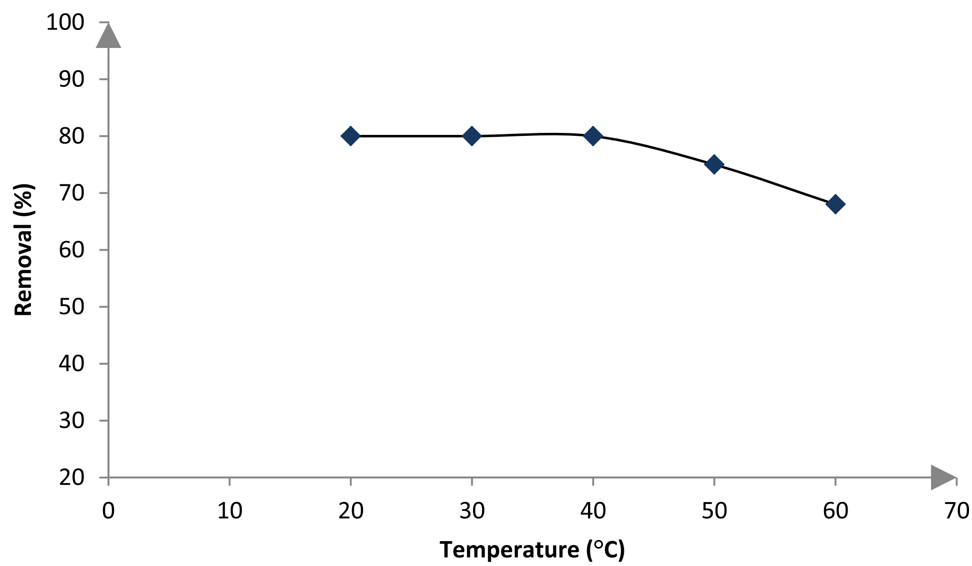


Fig. 10 Relationship between removal of oil by fresh *Ricinus* and temperature (°C)

3.9 Isotherm analysis

This analysis explains the behavior of adsorption related to adsorbent capacity by determining the amount of pollutant adsorbed to the available amount of adsorbent in equilibrium state [12]. Multiple models were built to evaluate this capacity, and each model used has special assumptions, and in this study, the analysis investigated applying the well-known models: Langmuir, Freundlich and BET.

3.9.1 Langmuir model

This model assumes monolayer is adsorbed to the solid surface of adsorbent [10, 25] and also adsorption energy is in equal distribution on solid surfaces [11]; two important parameters must be determined in this model: maximum adsorption in equilibrium and rate of adsorption. Langmuir model equation is as follows: [10]

$$\frac{1}{q_e} = \frac{1}{K_L Q} \frac{1}{\mathrm{Ce}} + \frac{1}{Q}. \tag{3}$$

3.9.2 Freundlich model

This isotherm was governed by empirical equation to analyze adsorption capacity and rate of adsorption in heterogeneous surfaces of adsorbent [29], and also this model covered the mechanism of multilayer adsorbed rather than monolayer as in Langmuir model [38]; the equation of Freundlich isotherm is as follows: [20, 29]:

$$\mathrm{Log}\, q_e = \mathrm{Log}\, K_F + \frac{1}{n} Ce. \tag{4}$$

3.9.3 BET isotherm model

This isotherm model is used widely because it can be used for monolayer as well as for multilayer in order to describe the mechanism of adsorption, and also this model can be considered to be applicable for the determination of active surface and pore sizes of adsorbents; it was assumed in this model that the interaction between solid surface of adsorbent and adsorbate molecules is larger than that between molecules of adsorbate neighbor [9].

The linear form of BET model is as follows [16]:

$$\frac{\mathrm{Ce}}{q_e(\mathrm{Cs}-\mathrm{Ce})} = \frac{1}{q_s K_B} + \frac{(K_B-1)}{K_B q_s}\left(\frac{\mathrm{Ce}}{\mathrm{Cs}}\right). \tag{5}$$

The results of the experimental and theoretical oil engine emulsion biosorption isotherms for *Ricinus communis* are portrayed in Fig. 11, and their correlation coefficients are presented in Table 2. Based on the values of the regression coefficient (R^2), one can infer that the Freundlich equation was the best fitted model for oil engine emulsion biosorption isotherms on the *Ricinus communis*, thereby illuminating that multilayer adsorption occurred in this process. The reason can be illustrated by the complex composition and molecular size of oil engine. Besides, the Freundlich model constant ($1/n$) is 0.722 for emulsified diesel oil, revealing the adsorption process was favorable.

Table 2 Isotherm parameters

Isotherm	Parameters	Oil engine emulsion
Langmuir	K_L (L/mg)	0.027299769
	Q (mg/g)	3832.19842
	R^2	0.9618
Freundlich	K (mg/g)(L/mg)1/n	45.325
	n	1.3846
	R^2	0.994
BET	K_B (L/mg)	8.466
	q_s (mg/g)	4102.47
	R^2	0.9516

3.10 Kinetic analysis

Kinetic models can be helpful in understanding the mechanism of sorption and evaluating the performance of an adsorbent [31]. In this study, the sorption kinetics was modeled using the pseudo-first-order and pseudo-second-order kinetic models.

3.10.1 Pseudo-first-order kinetic model

Linear form of this model was used to fit the increase of (q_t) which refers to amount of adsorbate adsorbed at specific interval of time as shown in Fig. 12a. Analysis took place toward the amount adsorbed at equilibrium (q_e), and the linear equation is as follows [33]:

$$\ln(q_e - q_t) = \ln q_e - k_1 t \tag{6}$$

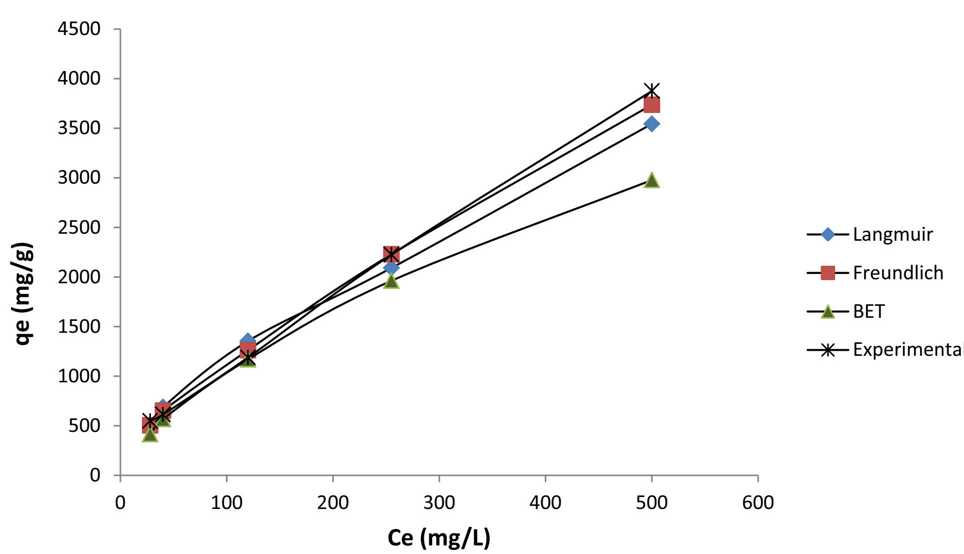
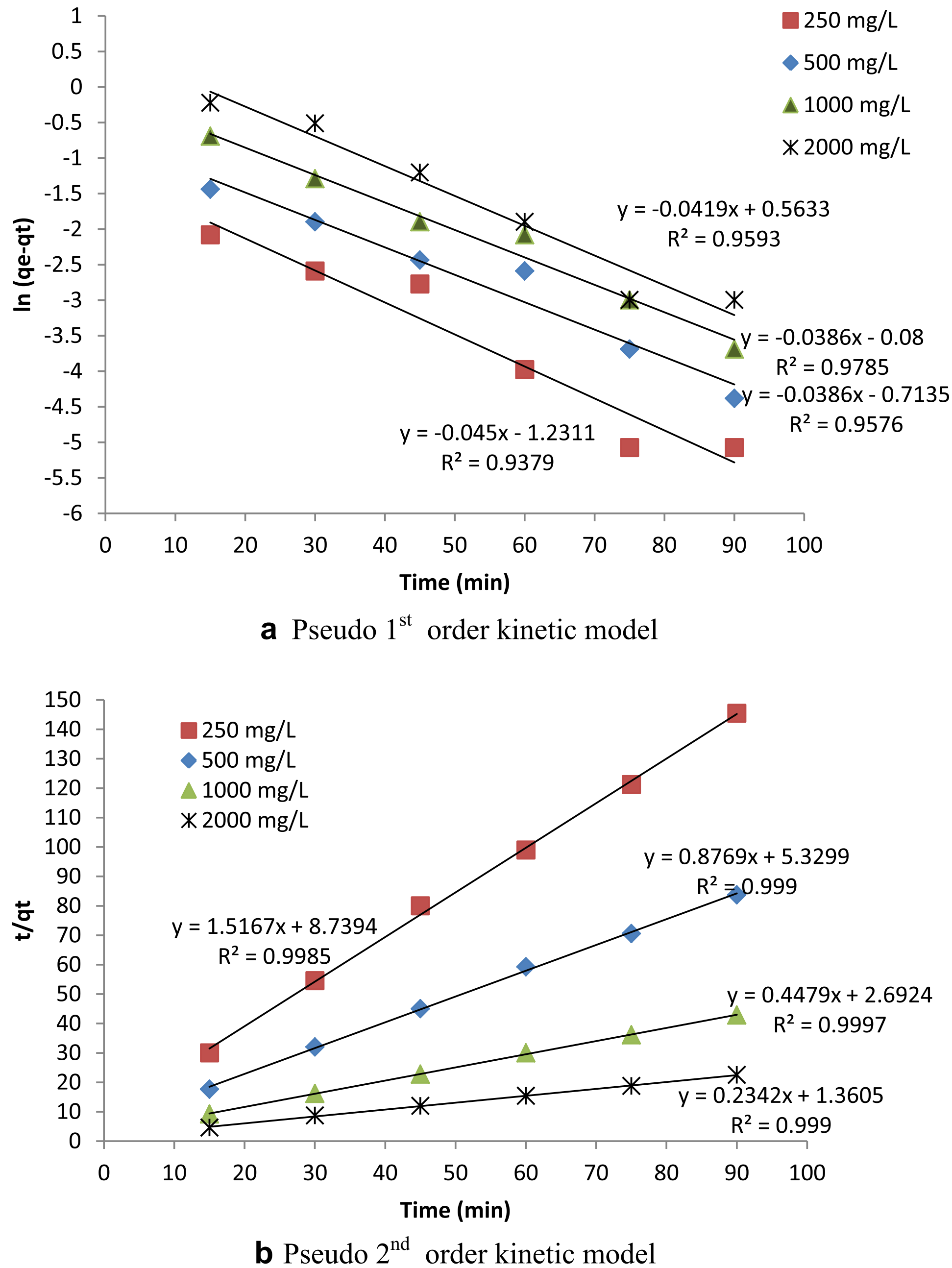


Fig. 11 Data comparison of experimental and isotherm models of Langmuir, Freundlich and BET

Fig. 12 Kinetic models for oil adsorption by *Ricinus*



3.10.2 Pseudo-second-order kinetic model

In this model, uptake of adsorbate on the structure of adsorbent surface was not in linear form, which pretended the effectiveness of pore size on the adsorption mechanism because if the process obeys this model, it means adsorption was controlled by diffusion of adsorbate molecules into pores [40].

To analyze data with this model, plot of linear form was adopted as follows:

Linear form equation [18]:

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e} t \tag{7}$$

The results in Fig. 12b showed good fitting to the experimental adsorption data for concentration ranges between 250 and 2000 mg/L with time, which obey illustrated linear increase in adsorbed quantity of oil on the solid surfaces of adsorbent.

The kinetic parameters for both models were evaluated as shown in Table 3.

Table 3 Kinetic parameters

Kinetic analysis	Oil concentration			
	250 mg/L	500 mg/L	1000 mg/L	2000 mg/L
q_e experiment (g/g)	0.625	1.0875	2.125	4.05
Pseudo-first-order				
q_e (g/g)	0.292	0.490	0.923	1.756
k_1 (min^{-1})	0.045	0.0386	0.0386	0.0419
R^2	0.9379	0.9576	0.9785	0.9593
Pseudo-second-order				
q_e (g/g)	0.695	1.140	2.232	4.269
k_2 (min^{-1})	0.263	0.144	0.074	0.040
R^2	0.9985	0.999	0.9997	0.999

4 Conclusion

Oil-in-water emulsion can be formed by mixing oil with water body in the presence of surfactant, and the resultant solution contained dissolved oil which is hard to be treated in conventional units. *Ricinus* leaves of 150–300 micron showed good ability to adsorb this kind of pollutants in acidified solution, and multilayer of oil molecules was attached to solid surfaces of adsorbent because Freundlich isotherm best fitted the adsorption process, rate of quantity adsorbed increased in nonlinear form as the results of kinetic models adopted in this research. Pseudo-second-order model well fitted the adsorption rate than first-order model.

Compliance with ethical standards

Conflict of interest The authors declare that they have no conflict of interest.

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