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A feasibility study for recycling biodegradable adsorbent in the oil spill clean-up from seawater

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ABSTRACT

In the present work, the efficiency of *Conocarpus* leaves to remove oil from seawater was estimated. *Conocarpus* leaves were characterized by Brunauer–Emmett–Teller, scanning electron microscopy, Fourier-transform infrared spectroscopy and contact angle. The sorption parameters (sorption time, pH, oil ratio, biomass dose, initial oil concentration and temperature were investigated. The change in the pH of the oil/seawater system does not influence the oil sorption. Maximum oil sorption percent reached 88% at 2 min, 0.35 g at room temperature. The kinetic studies show rapid sorption and the pseudo-second-order kinetic governed the oil sorption. The isotherm modeling displayed that the oil uptake was fitted by the Langmuir isotherm. The outcomes of this work indicated a good sorption capacity (1.76 g/g) of *Conocarpus* leaves. In addition, *Conocarpus* leaves can be recycled for 4 cycles until the performance decays under 40%.

Keywords: Oil spill; *Conocarpus* leaves; Sorption; Kinetic; Recycle

1. Introduction

Petroleum pollutants leaking into water are a disaster for the environment and the aquatic life system. Petroleum oil contains many organic substances, many of which are considered poisonous to living organisms [1]. One of the most dangerous of these compounds is benzopyrene, a carcinogenic hydrocarbon that leads to the death of aquatic organisms [2,3].

The methods used to remove oil pollutants are very difficult and expensive, some of them require a long time and a lot of labor, and others harm organisms and the marine environment [4,5]. These methods include the use of chemical dispersants that penetrate the oil slick and turn it into small spots, burning at the site of the spill, or mechanical collection of oil from the surface of the water [6–8]. Water pollution with oil can be combated by a biological solution using bacteria, as some scientists have found that several microscopic microorganisms that can decompose oil materials can transform oil slicks into very fine droplets in water [9]. The method that proved more cost-effective and easy to extract oil slicks is through adsorption using sorbents that convert the oil layer into solid or semi-solid particles that are easy to recover [10,11]. However, these materials have disadvantages represented in their low ability to adsorption and instability during the adsorption process, In addition to the narrow scope of their applications [12]. Recently, polymeric nano-adsorbents have achieved good results in large-scale oil spill removal; also, polymer blending has become an important approach for preparing polymeric materials with novel properties in this field [13]. Several agricultural adsorbents such as graphene oxide/β-cyclodextrin [14,15], amine-functionalised graphene/polyethylene nanocomposite [16], wheat straw [17], sugarcane [18], cotton fibers [19], and walnut shell [20] have a good competence to remove oil from seawater. In this study, *Conocarpus* leaves as biodegradable agricultural biomass was used as an adsorbent to remove oil from seawater in a batch system. *Conocarpus* is a plant that is widespread and available because they are widely used in afforestation. Experimental parameters of the adsorption system were determined. In addition, kinetics, and isotherms models studied the results. Besides, the reusability of *Conocarpus* leaves was investigated.

2. Materials and methods

2.1. Preparation of Conocarpus leaves

Used motor oil obtained from motor maintenance is used in the study. The properties of used motor oil are presented in Table 1. *Conocarpus* leaves are obtained from the waste gardens. 100 g of *Conocarpus* leaves were censored into 0.5 cm pieces and washed with distilled water, then dried at 80°C for 24 h. Also, 3.5% salinity seawater was used in the adsorption procedures.

2.2. Method

Oil ratio range from 0.3 to 0.8 g (5–10 mL) of used oil was mixed with 1 L of seawater in a batch glass contactor for a sorption time range (0.5–5 min) at room temperature. The biomass dose range of 0.1–0.5 g was added to the oil/seawater system. The loaded samples were separated from the oil/seawater system then drained for 5 min, and re-weighted (Electronic Analytical Balance, Model: ALE-223). Then, the drained samples were dried for 12 h at 85°C to remove adsorbed water. In addition, the effect of sorption temperature (30°C–50°C) and pH from 3 to 8 (BR Biochem Black Digital PH Meter) were studied at the optimum operating condition. The experiment was done three times and the average was taken for the accuracy of obtaining the results.

The oil removal efficiency (E %) was described by Eq. (1):

$$E\% = \frac{\left(W_D - W_I\right)}{W_I} \times 100 \quad (1)$$

Oil sorption capacity q_e (g of oil/g of biomass) is determined by Eq. (2):

$$q_e\left(\text{g/g}\right) = \frac{O_m}{W_I} \quad (2)$$

where W_I, is the initial weight of biomass, W_D is the weight of biomass with oil after drying. O_m is the weight of the sorbed oil (g).

Table 1
Characteristics of used motor oil

Characteristics	
Water content (%)	0.02
Density (kg/m^3)	872.5
Viscosity, mm^2/s at 100°C	58.94
Mechanical impurities content, %, no more than	0.087
Flash temperature, °C, no less than	188

3. Results and discussion

3.1. Characterization

3.1.1. Brunauer–Emmett–Teller and scanning electron microscopy analysis

The result of Brunauer–Emmett–Teller (BET; ASAP 2020, Norcross, GA, USA) analysis is 3.44 m^2/g, which indicates that the *Conocarpus* leaves (Fig. 1a) have a good surface area, which increases the oil adsorption capacity. In addition, the scanning electron microscopy (SEM) analysis (Hitachi, Tabletop TM3000) shows that *Conocarpus* leaves have prominent circular points (Fig. 1b) and that the surface is covered with a dense layer of wax, which increases the hydrophobic nature and thus the ability to absorb oil.

3.1.2. Contact angle

The contact angle (θ) was determined using a Krüess Processor Tensiometer K100. Contact angle has been an imperative constraint to determine the wetting aptitude of the biomass surface [21]. Results displayed that the wetting force relates well to the proceeding contact angle, the higher the θ the poorer the surface wettability [22]. Fig. 2 shows the biomass has θ = 112° with seawater (Fig. 2a) indicating that the *Conocarpus* leaves have a hydrophobic nature. In addition, Fig. 2b displays θ = 16° with the oil interface representing a good oil spread over the leaves' surface. It is obvious that a low contact angle at the biomass–oil interface is indispensable to apply high capillary forces on the oil and hence attain high oil adsorption capacity [23].

3.1.3. Fourier-transform infrared spectroscopy analysis

Fig. 3 displays the functional groups of *Conocarpus* leaves by Fourier-transform infrared (FTIR) spectroscopy (Thermo Fisher Scientific, USA). The band at 3,431 cm^{-1} is attributed to the hydrogen bonded (O–H) in cellulose [23]. The bands at 2,933 cm^{-1} correspond to the attendance of C–H in CH_3 and CH_2 groups [17]. The bands detected at 1,611 and 1,510 cm^{-1} are related to the vibrations of C=C bonds. The band at 1,455 cm^{-1} was attributed to the C–O or –OH deformation in COOH group [24]. The band at 1,045 cm^{-1} is ascribed to –C–O–C– stretches [19]. FTIR analysis presented that *Conocarpus* leaves have hydrophilic ((OH), (C=O) and (COOH)) and hydrophobic ((C–H), (R–COO–R) and (C–O–C)) functional groups, which increase the oil sorption capacity of *Conocarpus* leaves.

3.2. Sorption studies

3.2.1. Effect of sorption time

The effect of batch sorption time (0.5–5 min) on the oil removal percent of *Conocarpus* leaves sorbent is displayed in Fig. 4a under operating conditions (1.0 g oil/1L seawater, 0.25 g biomass, 28°C and pH 6). It can be observed that the oil removal percent was rapid until reached 88% in the first 0.5–2 min and afterward no obvious change in the oil removal percent was noticed indicating that the optimum contact time was 2 min. This firstly high rate of

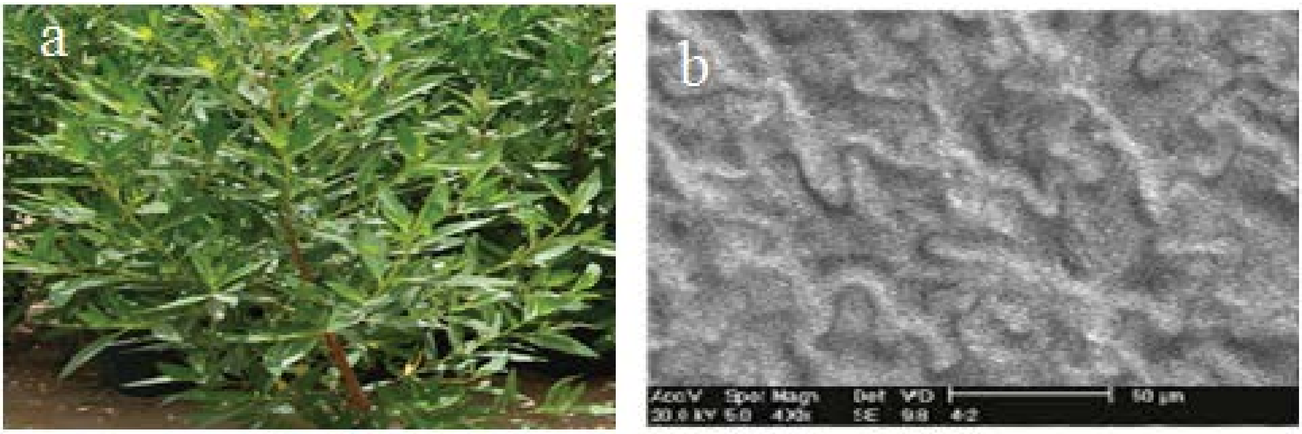

Fig. 1. *Conocarpus* leaves (a) and SEM analysis of leaves (b).

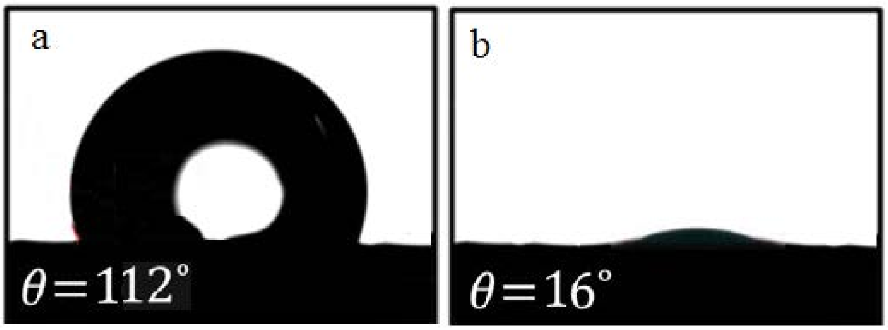

Fig. 2. Contact angle analysis with water (a) and oil (b) onto *Conocarpus* leaves.

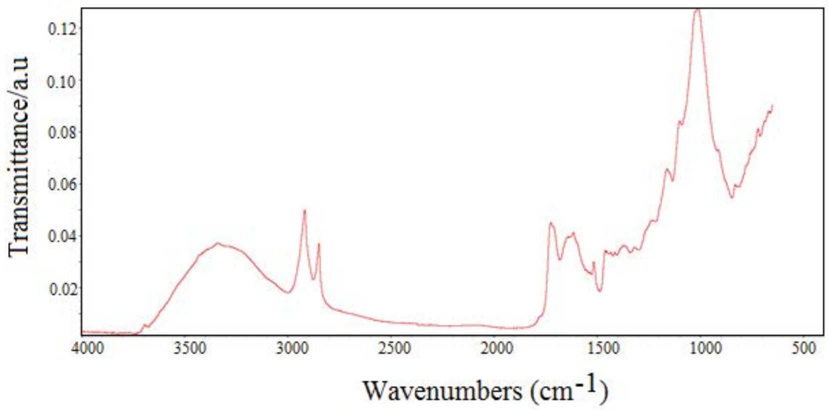

Fig. 3. FTIR analysis of *Conocarpus* leaves.

oil removal percent may be attributed to the existence of a great number of empty cavities available for the oil sorption uptake on the *Conocarpus* leaves [25]. The kinetic of oil sorption onto *Conocarpus* leaves is described using kinetic models of pseudo-first and second-order models.

3.2.1.1. Pseudo-first-order model

The pseudo-first-order model is proposed by the following linear Eq. (3) [15,26]:

$$\log(q_e - q_t) = \log q_e - K_1 t \tag{3}$$

3.2.1.2. Pseudo-second-order model

The pseudo-second-order model is described by the succeeding linear Eq. (4) [25,27]:

$$\frac{t}{q_t} = \frac{1}{q_e^2 K_2} + \frac{1}{q_e} t \tag{4}$$

where K_1 (L/min) and K_2 (g/g min) are the constants of pseudo-first and second-order. While, q_e and q_t are the oil sorption capacities at equilibrium and time t (g/g), respectively.

The values of the kinetic parameters are determined from the intercept and the slope of t vs. $\ln(q_e - q_t)$ and t/q_t plots of pseudo-first and second-order rate constant respectively (Fig. 5).

The rate coefficients of kinetic models are presented in Table 2. According to the R^2 coefficients, the pseudo-second-order model has a good fitting with the experimental outcomes demonstrating that the rate-limiting step of oil sorption attributed to monolayer formation of chemical interaction between the surface of *Conocarpus* leaves and oil, which involved co-valent interaction forces by the sharing of electrons amid the adsorbent and oil particles [27]. The sorption of spilled used oil onto *Conocarpus* leaves exhibited the pseudo-second-order kinetics model and was controlled by the film-diffusion system and ruled by the interior transport mechanism [28].

3.2.2. Effect of pH

The effect of pH on oil removal percent (1.0 g oil/1 L seawater, 0.25 g biomass, 28°C and 2 min). While the pH of the liquid sorption system is an imperative constraint in the sorption of ions, the current work proves that the pH had no important effect on the oil sorption by *Conocarpus* leaves in the pH range from 2 to 8, the oil removal percent remained constant around 88%. Consequently, all further

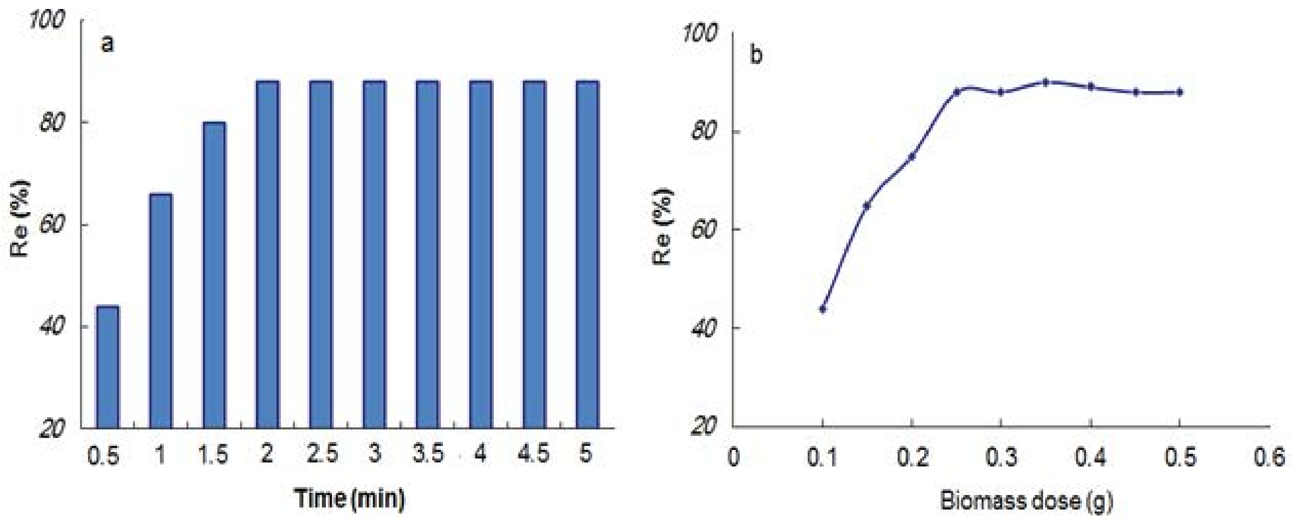

Fig. 4. Time effect (a) conditions (1.0 g oil/1 L seawater, 0.25 g biomass, 28°C and pH 6) dose effect and (b) (1.0 g oil/1 L seawater, 2 min, pH 6, and 28°C) on the oil sorption onto *Conocarpus* leaves.

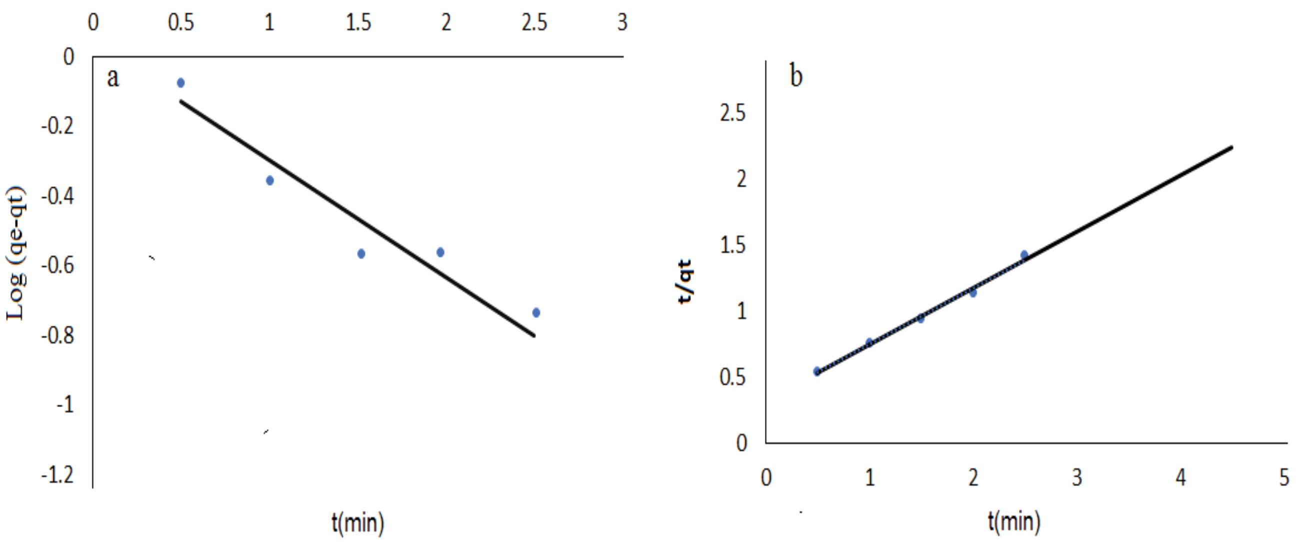

Fig. 5. Pseudo-first-order (a) and pseudo-second-order (b) kinetic models of used oil sorption onto *Conocarpus* leaves under operating conditions (1.0 g oil/1 L seawater, 0.25 g biomass, 28°C and pH 6).

experiments were completed at pH 6. The pH ineffectiveness on the oil sorption may be owing to the existence of the high stability of numerous compounds of heavy metals, metalloids, and hydrocarbons in the oil content, which led to the resistance of these compounds against ionization in the changed pH of aqueous solutions [22].

3.2.3. Effect of biomass dose

The dose range 0.1–0.5 g of *Conocarpus* leaves was utilized under operation conditions (1.0 g oil/1L seawater, 2 min, pH 6, and 28°C) to investigate the optimum economic dose in the maximum oil removal percent. Fig. 4b shows an increase in the oil removal percent from 44% to 88% and sorption capacity from 0.88 to 1.76 g/g with increasing biomass mass from 0.1 to 0.35 g/L, respectively. These results owing to the increase of oil sorption sites in the *Conocarpus* leaves, then the oil removal percent remained constant at around 88% (1.76 g/g) indicating that the optimum dose of biomass was 0.35 g [22,28].

Table 2
Sorption kinetic models and results for oil sorption onto *Conocarpus* leaves under operating conditions (1.0 g oil/1 L seawater, 0.25 g biomass, 28°C and pH 6)

Kinetic model	Parameter value
Pseudo-first-order	
K_1 (min^{-1})	0.4231
q_e (mg/g)	26.765
R^2	0.8731
Pseudo-second-order	
K_2 (g/mg·min)	0.1330
q_e (mg/g)	1.965
R^2	0.9825

3.2.4. Effect of temperature

Fig. 6a shows the effect of temperature range 28°C–50°C in the oil sorption onto *Conocarpus* leaves under operation conditions (1.0 g oil/1L seawater, 2 min, pH 6, and 0.35 biomass dose). The experimental results showed a decline in the oil removal percent from 88% to 24% and oil sorption capacity from 1.76 to 1.21 g/g, with increasing the temperature from 28°C to 50°C, respectively. These results owing to the decrease in the oil viscosity with increasing temperature, which leads to a decrease in the oil retention in the biomass pores during the draining period [29].

3.2.5. Effect of initial oil concentration

Fig. 6b shows the oil and seawater sorption capacities of *Conocarpus* leaves in the different oil ratios (0.3–0.8 g/1 L seawater) under operating conditions (0.35 g dose biomass, 28°C, pH 6 and 2 min). The results showed that the oil sorption capacity increased with increasing the initial oil concentration until reached 1.76 g/g at an oil ratio of 0.5 g/1 L seawater then no noticeable change in the oil sorption capacity with increasing the initial oil concentration indicating that the sorption sites in the *Conocarpus* leaves are saturated [30]. While the seawater sorption capacity was decreased from 0.5 to 0.022 g/g with increasing the initial oil concentration from 0.3 to 0.8 g respectively. The outcomes show that the *Conocarpus* leaves can be used as an exceptional adsorbent in the oil sorption from seawater.

The isotherms of Langmuir and Freundlich equations have been used to evaluate the oil sorption equilibrium onto *Conocarpus* leaves.

3.2.5.1. Langmuir isotherm

Langmuir isotherm supposes that monolayer oil sorption onto the *Conocarpus* surface and the model described by Eq. (5) [17,21].

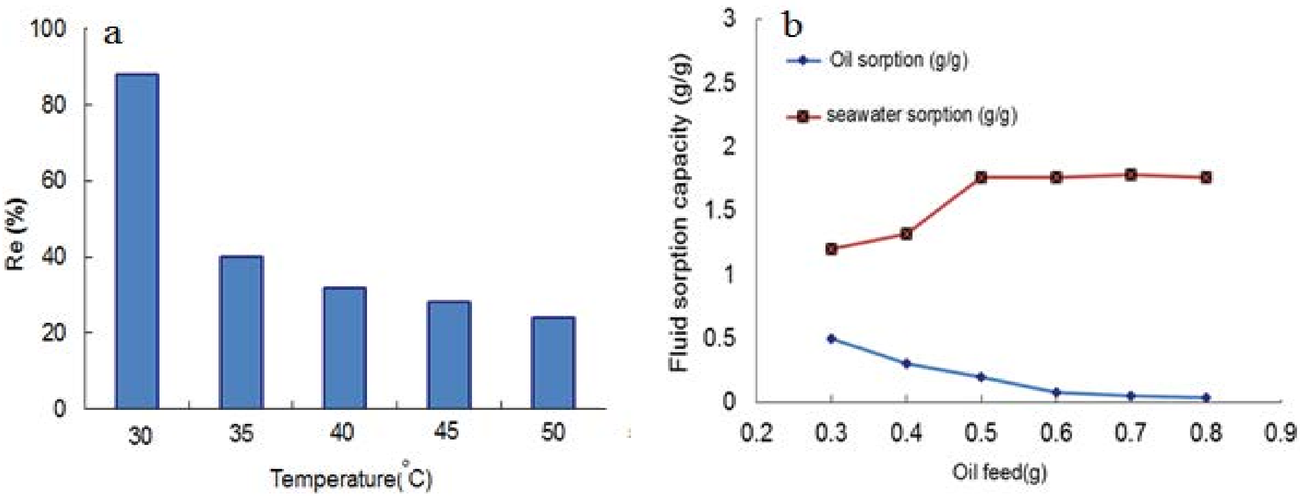

Fig. 6. Temperature effect (a) under operation conditions (1.0 g oil/1 L seawater, 2 min, pH 6, and 0.35 biomass dose) and oil ratio (b) under operating conditions (0.35 g dose biomass, 28°C, pH 6 and 2 min) on the oil sorption onto *Conocarpus* leaves.

$$\frac{C_e}{q_e} = \frac{C_e}{Q_l} + \frac{1}{K_L Q_L} \quad (5)$$

3.2.5.2. Freundlich isotherm

Freundlich isotherm assumes that a heterogeneous oil multilayer interacted with the *Conocarpus* surface and described by Eq. (6) [29,30].

$$\log(q_e) = \log K_F - \frac{1}{n}\log C_e \quad (6)$$

where q_e is the oil sorption capacity (g/g) and C_e is the oil concentration at equilibrium (g/L).

K_F is the Freundlich constant and K_L and Q_L are Langmuir constants. n is a parameter representing to the strength of oil sorbed onto the *Conocarpus* surface.

The constants of Langmuir and Freundlich isotherm were determined from the C_e vs. C_e/q_e plot (Fig. 7a) and lnC_e vs. lnq_e plot (Fig. 7b), respectively and listed in Table 3. The fitting between kinetic and experimental results is assessed by the correlation coefficient (R^2) [21]:

$$R^2 = \frac{\sum_{i=1}^{n}\left(q_{e\text{ experimental}} - q_{e\text{ theoretical}}\right)^2}{\sum_{i=1}^{n}\left(q_{e\text{ experimental}} - q_{e\text{ theoretical}}\right)^2 + \sum_{i=1}^{n}\left(q_{e\text{ experimental}} - q_{e\text{ theoretical}}\right)^2} \quad (7)$$

Table 3
Isotherm results of used oil sorption onto *Conocarpus* leaves under operating conditions (0.35 g dose biomass, 28°C, pH 6 and 2 min)

Isotherm model	Parameter	Value
Langmuir isotherm	Q_L (g oil/g biomass)	1.982
	K_L (L/g)	0.532
	R^2	0.995
Freundlich isotherm	K_F (g $(^{1-1/n})$/$L^{1/n}$·g)	1.367
	n	2.345
	R^2	0.886

The results showed that the correlation coefficients (R^2 = 0.995) of the Langmuir isotherm are higher than the value achieved for the Freundlich isotherm (R^2 = 0.886). For that reason, the Langmuir isotherm is the best-fit model for oil sorption onto *Conocarpus* leaves, which is attributed to a monolayer sorption process [31].

3.2.6. Oil sorption thermodynamics

The values of thermodynamic parameters (entropy (ΔS), free energy (ΔG), and enthalpy (ΔH)) in the oil sorption onto *Conocarpus* leaves were studied to determine the oil sorption nature of *Conocarpus* leaves. The thermodynamic parameters are described by using the following equations [22,32].

$$\Delta G = \Delta H - T\Delta S \quad (8)$$

$$\Delta G = -RT \log K_c \quad (9)$$

$$K_c = \frac{q_e}{C_e} \quad (10)$$

$$\ln K_c = \frac{\Delta S}{R} - \frac{\Delta H}{RT} \quad (11)$$

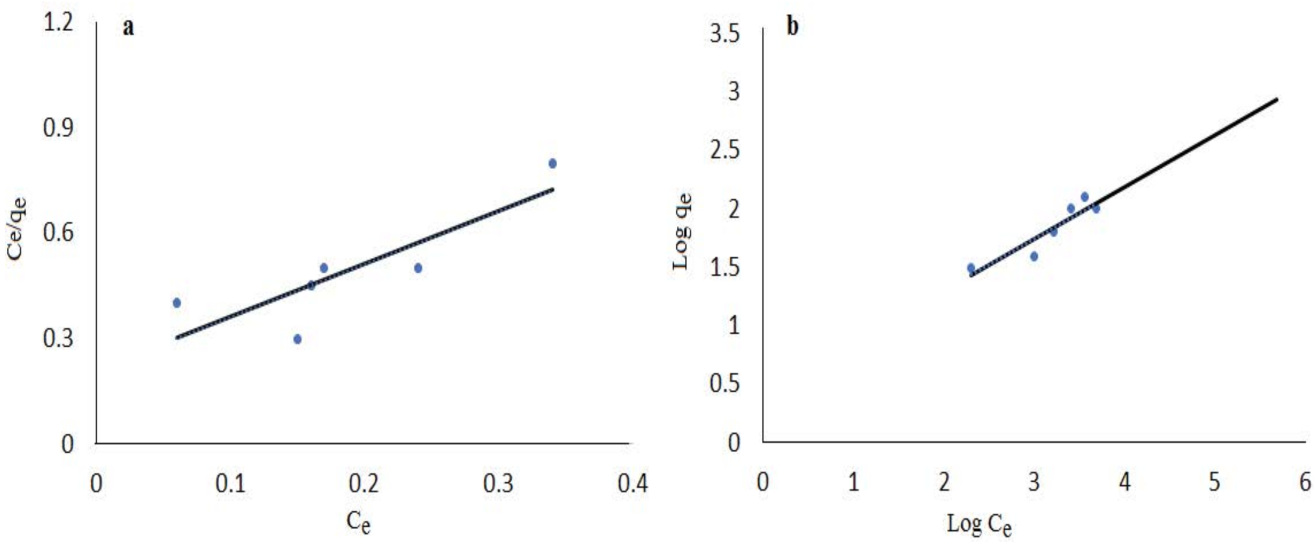

Fig. 7. Langmuir (a) and Freundlich (b) isotherm of used oil sorption onto *Conocarpus* leaves under operating conditions (0.35 g dose biomass, 28°C, pH 6 and 2 min).

where q_e: oil sorption uptake (g/g), C_e: oil concentration at equilibrium (g/L), T: temperature (K) and R: gas constant (8.314 J/mol·K).

Fig. 8a shows a reduction in ΔG and oil removal percent with the increase in the temperature from 28°C to 50°C. The positive ΔS (0.4689 J/mol·K) refers to spontaneous oil sorption [22,25]. Furthermore, the negative value of ΔH (–157.209 J/mol) represents the exothermic nature of oil sorption onto *Conocarpus* leaves (Table 4).

3.3. Reusability studies

The economic feasibility of *Conocarpus* leaves in the removal of the oil spill is important. The oil desorption system was carried out by Squeezing loaded biomass under centrifugation force at 500 rpm. Fig. 8b shows the effect of the sorption/desorption system on the oil removal percent of *Conocarpus* leaves. The results showed a decrease in the oil removal percent with the increase of sorption/desorption which indicates that the *Conocarpus* leaves can be recycled for 4 cycles.

Table 5 shows a comparison between the oil sorption capacity of *Conocarpus* leaves and other adsorbents indicating an acceptable outcome of *Conocarpus* leaves relative to the other adsorbents.

4. Conclusion

In this work, the performance of *Conocarpus* leaves for the oil sorption from seawater was investigated. The outcomes showed that pH had no important effect on the oil sorption. Maximum oil sorption reached 1.76 g/g at 2 min, 0.35 g at room temperature The kinetic studies demonstrate that oil adsorption on *Conocarpus* leaves is rapid until sorption time 2 min and the pseudo-second-order kinetic ruled oil adsorption. The isotherm modeling showed that the oil sorption was fitted by the Langmuir isotherm. The outcomes of this work indicated a good sorption capacity of *Conocarpus* leaves without any pretreatment. *Conocarpus* leaves can be recycled for 4 cycles until the performance decays under 40%. Therefore, the use of *Conocarpus* leaves as low cost, biodegradable and available sorbents could be acceptable.

Table 4
Thermodynamic results for used oil sorption onto *Conocarpus* leaves under operation conditions (1.0 g oil/1 L seawater, 2 min, pH 6, and 0.35 biomass dose)

T (K)	$\ln q_e/C_e$	ΔG (J/mol)	ΔH (J/mol)	ΔS (J/mol·K)
301	2.126514	–16.0701		
305	1.548813	–14.1945		
309	1.311673	–12.3189	–157.209	0.4689
316	0.804543	–09.0366		
323	0.182321	–05.7543		

Table 5
Oil sorption capacity and reusability of *Conocarpus* leaves and different sorbents

Biomass material	Sorption capacity (g/g)	Reusability cycles	References
Banana peel	6.35	10	[2]
Raw cotton fiber	30.5	5	[24]
Bagasse	11.3	5	[11]
Raw flax fiber	13.75	6	[22]
Raw kapok fiber	38.1	5	[19]
Wheat straw	4.0	5	[21]
Saw dust	6.4	4	[23]
Solanum leaves	11.56	5	[28]
Conocarpus leaves	1.76	4	Present study

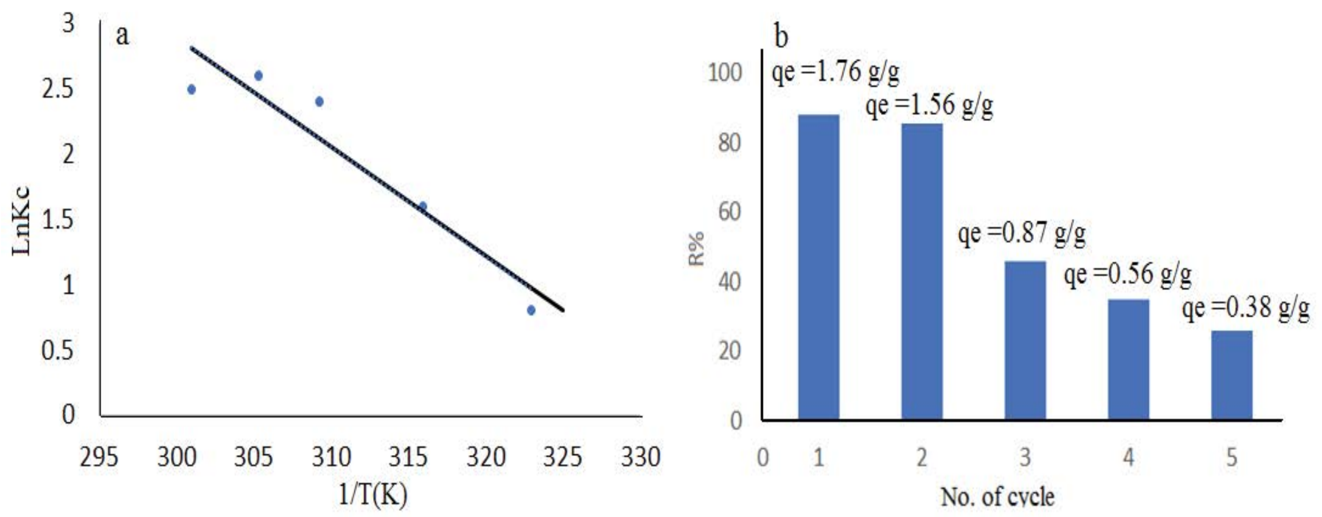

Fig. 8. Van't Hoff plot (a) under operation conditions (1.0 g oil/1 L seawater, 2 min, pH 6, and 0.35 biomass dose) and sorption–desorption cycles and (b) of used oil sorption onto *Conocarpus* leaves.

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