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Utilization of Date Palm Trunk Fibers (DPTFs) Waste as a Sorbent for the Removal of Crude Oil from Aqueous Media: Equilibrium Isotherms and Kinetic Studies

***Hamid Boleydei*[1]*, Zahra Razavi**[1]

1) *Department of Natural Resources, Isfahan University of Technology, Isfahan, Iran.*

**Corresponding author:* z.razavidinani@gmail.com

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ABSTRACT

In the present study, the efficiency of date palm trunk fibers (DPTFs) to remove crude oil from aqueous solution was evaluated. The sorbent were characterized by Fourier transform infrared (FT-IR) spectroscopy. The effect of various parameters including contact time, pH of oil solution, particle size, initial oil concentration and adsorbent dose on oil adsorption were investigated. The results showed that the adsorption kinetic was described by pseudo-second-order model. The change in the pH of oil solution from 2–10 had no effect on the oil adsorption. The decrease of particle size of DPTFs reduced oil sorption percent. The crude oil adsorption on DPTFs was well fitted by the Langmuir model. Maximum capacity of adsorption of crude oil by DPTFs was 2500 mg g-1. The results obtained revealed that crude oil adsorption onto the date palm wastes as the abundantly available low cost and readily biodegradable material is feasible and can successfully be used as an effective adsorbent to oil spill cleanup in water basins.

Key words: Adsorption Kinetic, Isotherm; Agricultural byproduct; Crude Oil; Date Palm Trunk Fibers (DPTFs)

INTRODUCTION

Unsystematic discharge of oil containing wastewater into an aqueous environment is considered one of the most serious threats to protection of water bodies because of toxicological and esthetical reasons such as unpleasant odor and taste as well as undesirable appearance. In addition, oil and its derivatives are stable to light and to heat, they are also very difficult to undergo biodegradation so oil pollution in water media causes severe environmental damage on water fowl, material life and affects tourism and economy [1-3].

Of the available physical, chemical and biological methods for oil spill cleanup, adsorption process is one of the interesting methods used in wastewater systems. Oil sorbents are able to change liquid oil to the semi-solid or solid phase, which can then be removed from the water without significant oil draining out [4].

Recently, adsorption processes using solid waste products particularly by-products from agriculture as natural adsorbents have drawn the increasing attentions. It offers various advantages such as simple technique, little processing cost, readily availability, high buoyancy, reusability (by simple squeezing or as a fuel in heating processes) and ability to biodegradation [5-8].

Date palm biomass or date palm trunk fiber (DPTF) is also an agricultural product has recently gained wide attention as effective adsorbent for the removal of various contaminants such as dyes, heavy metals, pesticides, phenol and oil hydrocarbons from aqueous environments [9]. The date palm (*Phoenix dactylifera L.*) tree is a dioecious monocotyledon belonging to the *Arecaceae* family (Figure1). The date palm is a relatively tall tree, with cylindrical trunk, no branching stem and bearing edible fruit**.** It is a feather palm, composed of compound leaves with the leaflets on either side of the base of the petiole**,** radiating from one growing point at the apex of the trunk. [9, 10].

Date palm is adapted to the arid and semi-arid climates. On an average, it contains cellulose (40–50%), hemicellulose (20–35%), and lignin (15–35%) [9, 10]. As the cellulosic material acted like a solidifier with no dripping [9, 10]. As the cellulosic material acted like a solidifier with no dripping [11], the palm wastes can be a suitable sorbent for oil removal from aqueous media and the oil adsorbed on the material could be recovered by simple squeezing [7]. Of the date palm byproducts, date palm surface fibers were chosen in this study as it seemed most suitable for exploitation. Usually, after annually trimming operations, enormous quantities of date palm fibers are thrown away as waste except in smaller scales for artisan products [12]. Although most researchers used modified organic sorbents and in some cases reported their high efficiency for removing a variety of compounds such as oil and its derivatives, it should be noted that the pre-treatment steps are expensive and time consuming and even not environmentally friendly. Therefore, in some emergency oil spill conditions, the use of raw sorbents with high adsorption capacity for rapid and economic cleanup of contaminated water seems preferable [13].

Fig.1: (a) Schematic picture of the date palm (*Phoenix dactylifera L.*) tree and (b) date palm trunk fibers (DPTFs)

Therefore, the purpose of this work was to evaluate the adsorption potential of “in-nature” palm fiber (i.e. bark wastes) for the removal of crude oil from aqueous solution. The selected palm tree cultivar was Shahani which is the most popular cultivar in Jahrom [10]. In the present study the effective factors in the sorption process such as contact time, pH, sorbent particles size, the initial oil concentration, and sorbent dose were also optimized. The isotherm and kinetic data of the adsorption process were then studied to understand the adsorption mechanism of crude oil molecules onto the sorbent.

MATERIALS AND METHODS

Date palm trunk fibers (DPTFs) were collected from a farm in Jahrom region, Fars province, Iran. DPTFs were received in carton boxes. For cleaning away remaining dust of DPTFs, they were washed many times with distilled water subsequently DPTFs were dried at 75°C for 48 h in oven. After drying they were milled using an electric grinder mill and the samples were then sieved using a sieving machine into different average particle sizes of 0.053-0.106, 0.106-0.25, 0.25-0.5, 0.5-1 and 1-2 mm for experiments. Finally each sample was placed in a zipped bag and labeled.

The oil of Persian Gulf crude oil used whole experiments was obtained from Isfahan oil refining company that has 0.06 Poise viscosity (at 40$^{\circ C}$) and 0.85 gcm^3 density (at 25 $^{\circ C}$).

The surface functional groups present in the sorbent were characterized by Fourier transform infrared (FT-IR) spectroscopy (Bruker Equinox 55, Wissembourg, France). The samples were ground, mixed with KBr in ratio 0.1:3 and compressed into a thin tablet. The FT-TR spectra were recorded at ambient temperature in the wave number of 400–4000cm^{-1}.

For primary experiments, 10gl^{-1} of crude oil was poured into a 1000ml Erlenmeyer flask containing freshwater. The initial pH of solutions was adjusted with 1M NaOH or HCl solutions (Merck, Germany) and measured with a digital pH meter (3310 model, Jenway, UK). Then 10gl^{-1} of the sorbent was added to the mixture and shaken at 150 rpm for 10 min. After 10 min, the sorbent with adsorbed crude oil were separated with net filter. For separating crude oil and water from each other, Dichloromethane was added to Erlenmeyer flask. Then Erlenmeyer contents were transferred to a separation funnel. Based on the density difference the water phase and oil-dichloromethane phase were separated [14]. Finally for separating dichloromethane from crude oil, solution was placed in the heater at 40$^{\circ C}$ to evaporate dichloromethane [15]. Now by knowing the remaining and initial concentration of crude oil, adsorbed oil percentage was calculated as:

$$\mathbf{\%\ Sorption= (Ce\text{-}C0)\ V/M \times 100} \qquad \text{Eq. 1}$$

Where C_e is the concentration adsorbed crude oil (gl^{-1}), C_0 is the initial concentration of crude oil (gl^{-1}), V is volume of solution and M is DPTFs weight.

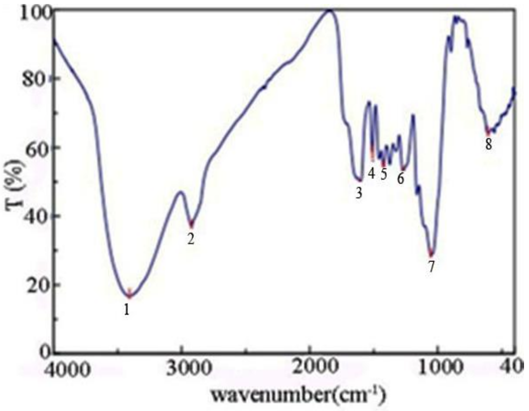
Similarly, in the following the influence of some factors that are contact time, initial pH of oil solution, DPTFs particle size, initial crude oil concentration and DPTFs dose on the crude oil sorption was studied. It is noteworthy that all tests were repeated three times at temperature of 21±3$^{\circ C}$.

Analysis of variance and Tuckey’s HSD in Minitab v.17 was used to determine which parameters had a significant effect on sorption crude oil by DPTFs and Origin Pro *v*.9.5 was applied to draw charts.

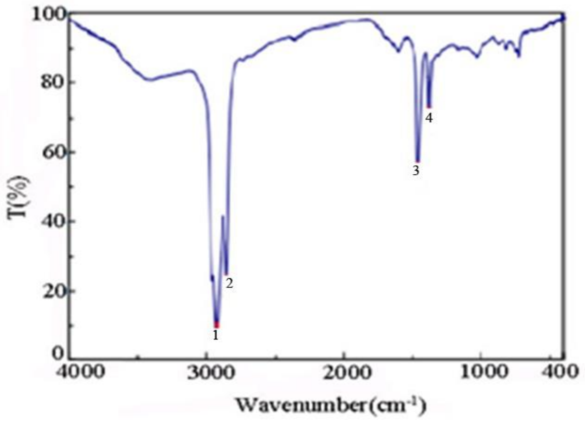
RESULTS AND DISCUSSION

Fig. 2 shows the FTIR spectrum of palm fibers before and after crude oil adsorption. The result is in agreement with that reported in the literature for Date palm biomass. The strong signal at 3408cm^{-1} is attributed to the intra- and inter-molecular hydrogen-bonded (O-H) stretching that occurs in cellulose [16].

The bands at 2921 cm-1 (before oil adsorption) and 2923cm^{-1} (after oil adsorption) correspond to the presence of C-H asymmetric stretching of CH_3 and CH_2 groups [17]. The adsorption bands observed at 1608cm^{-1} and 1509cm^{-1} are related to stretching vibrations of aromatic C=C bonds. The band at 1460cm^{-1} may represent the C-H bending vibration mode and the one at 1424cm^{-1} was due to C-O stretch or -OH deformation in carboxylic acids [18]. The peak at 1053cm^{-1} are attributed to ethers –C-O-C- stretches [17].



a) Before oil adsorption



b) After oil adsorption

Fig. 2: FTIR spectrum of DPTFs: (a) before and (b) after crude oil adsorption

The effect of batch contact time on the percentage removal of crude oil on palm fiber sorbent is shown in Fig.3 under various other fixed operating conditions (10gl^{-1} sorbent and sorbate concentrations, sorbent particle size 1-2mm, pH 8–10, 21±3$^{\circ C}$). It can be seen here that the removal of oil samples by the sorbent was rapid, occurring in the first 10–15min and thereafter beyond a contact time of about 15min no noticeable change in the percentage removal was observed. The percentage removals in these times were 80-85%. The relative increase in the extent of removal of crude oil after 15 min of contact time is not significant and hence it is fixed as the optimum contact time. This initially high rate of oil uptake maybe attributed to the presence of a large number of vacant voids accessible for the oil sorption on the sorbent. As the contact time was increased, less adsorption sites were available, hence oil uptake remained constant [6].

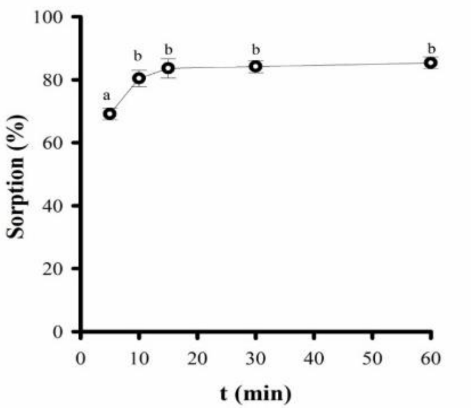


Fig. 3: Effect of contact time on adsorption of crude oil by DPTFs (10g L^{-1} sorbent and sorbate concentrations, sorbent particle size 1-2mm, pH 8–10, 21±3$^{\circ C}$). Different letters show significant differences according to Tukey's test (at p<0.05).

The kinetic of adsorption of crude oil onto DPTFs was characterized using pseudo first order and pseudo second order models. The pseudo first order linear kinetic model as proposed by Lagergren [19] is:

$$\boldsymbol{ln(q_e - q_t) = ln(q_e) - k_1 t} \qquad \text{Eq. (2)}$$

The values of the kinetic parameters (qe and k_1) are generally determined from the intercept and the slope of t versus ln ($q_e - q_t$) plot (Fig. 4a).

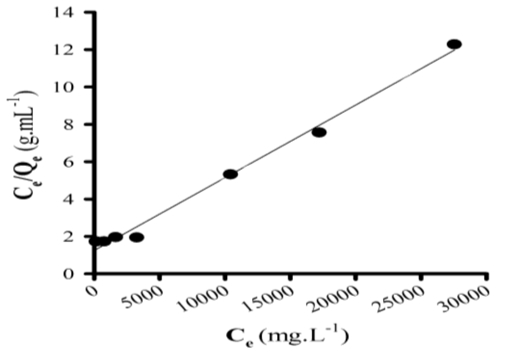
Also, the linearized-integral form of the pseudo second order kinetic model, as proposed by Ho and McKay [20] is:

$$\boldsymbol{\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e} t} \qquad \text{Eq. (3)}$$

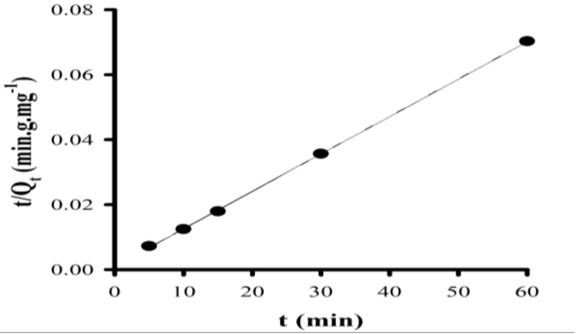
The values of the kinetic parameters (q_e and k_2) are generally determined from the slope and intercept of t versus t/q_t plot (Fig. 4b).

Where q_e and q_t are the amount of sorbet solute (mg g^{-1}) at equilibrium and time t, respectively, and k_1 and k_2 (g mg^{-1} min-1) is the pseudo first and second order rate constant respectively.

The pseudo first order and pseudo second order rate coefficients are presented in Table 1. According to the coefficients of determination R^2 which were close to 1, the pseudo-second-order model well represents adsorption kinetics in this study indicating that the rate limiting step may be a chemical interaction between the oil compound and the sorbent surface that it is due to the monolayer formation [21]. Formation of a monolayer could lead to early saturation of the sorbent [20].



a) pseudo first-order



b) Pseudo second-order

Fig. 4: Kinetic models for adsorption of crude oil by DPTFs: (a) pseudo first-order and (b) pseudo second-order.

Table 1: Kinetic parameters obtained from pseudo first-order model and pseudo second-order model for DPTFs (10gl^{-1} sorbent and sorbate concentrations, sorbent particle size 1-2 mm, pH 8–10, 21±3$^{\circ C}$).

Model	Parameter	value
Pseudo first-order (Lagergren)	qe (mg g^{-1})	693.8
	K_1 (min^{-1})	2×10-5
	R^2	0.428
Pseudo second-order	qe (mg g^{-1})	833.33
	K_2 (g mg-1min^{-1})	1×10-3
	R^2	0.999

The effect of pH on oil adsorption (15min, 10g L^{-1} sorbent and sorbate concentrations, sorbent particle size 1-2mm, 21±3$^{\circ C}$) is presented in Fig. 5. Although the pH of the solution is an important parameter in adsorption of ions, the present study clearly demonstrates that the pH had no significant effect on the oil adsorption by DPTFs and with increasing the pH of the oil solution from 2 to 10 (10gl^{-1} oil and sorbent), the adsorption percentage remained constant at about 80-85%. Therefore, all further experiments were performed in the initial pH of oil solutions in the range of 8–10. Brandão *et al.* also reported that the pH of the solution was not important in the adsorption of contaminants such as petroleum hydrocarbons on raw sugarcane bagasse [3]. The ineffectiveness pH on the oil compounds adsorption may be attributed to the presence of the several materials (heavy metals/metalloids, mixture of hydrocarbons and so on) in oil content which their high stability and the absence of hydrophilic compounds led to the resistance of these materials against ionization in different pH aqueous solutions [22].

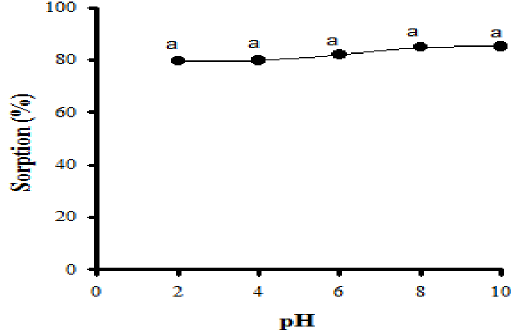


Fig. 5: Effect of oil solution pH on adsorption of crude oil by DPTFs (15min, 10gl^{-1} sorbent and sorbate concentrations, sorbent particle size 1-2 mm, 21±3$^{\circ C}$). The same letters show insignificant differences (at $p<0.05$).

The effect of the average particle size on the oil sorption percent of DPTFs (15min, 10gl^{-1} sorbent and sorbate concentrations, pH 8–10, 21±3$^{\circ C}$) shown in Fig. 6. It was found that crude oil adsorption on DPTFs is adsorbent particle size dependence. The highest percentage of removal for crude oil was achieved on the largest particle size. In fact, the decease of particle size of DPTFs from 1-2 to 0.053-0.106mm reduced the adsorption of crude oil from 83.62 to 70.80%, respectively. This is due to the fact grinding the sorbents caused degradation of the sorbent pores thus the sorbent was unable to hold more oil on the surface and the adsorption was reduced [13, 23]. In addition to, accumulation of small particles on each others can result in clogging pores and then reducing oil sorption on sorbent surface [1]. Husseien *et al.* [1], Kim *et al.* [24] and Lee *et al.* [23] in their studies on the oil adsorption by natural sorbents have confirmed that the oleophilic compounds tend to be adsorbed into the available large pores

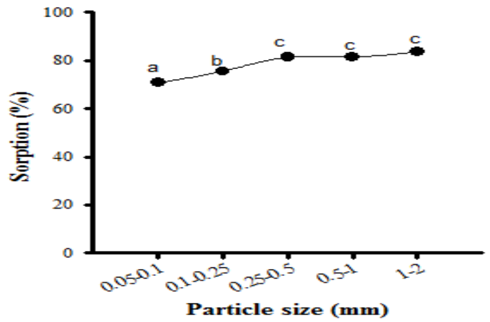


Fig. 6: Effect of adsorbent particle size on adsorption of crude oil by DPTFs (15min, 10gl^{-1} sorbent and sorbate concentrations, pH 8–10, 21±3$^{\circ C}$). Different letters show significant differences according to Tukey's test (at $p<0.05$).

Fig.7 shows the percentage of oil adsorption and sorption capacity of DPTFs (15min, $10 gl^{-1}$ sorbent concentration, sorbent particle size 1-2mm, pH 8–10, $21\pm3^{\circ C}$), versus the initial concentration of crude oil in aqueous solution respectively. It can be observed that that the oil adsorption showed a decreasing trend with the initial oil concentration so that with an increase of initial oil concentration ($1–50 gl^{-1}$), the percentage oil removal by DPTFs was reduced from 85.26 to 44.9%. Also, data shows increasing oil sorption capacity with increasing the initial oil concentration. Generally, at higher contaminant concentrations, the oil molecules themselves may be clustered into micelles making the adsorption process more difficult in some of the adsorbent pores [3]. Also, another hypothesis to explain this behavior is limitation of the adsorbent capacity in which the number of sorption sites is a determinant parameter so that at lower initial oil concentrations, the more amounts of oil compounds could be interacted with the active sites [33]. Whereas at higher concentrations, the adsorption percentage was reduced due to the saturation of adsorbent [15].

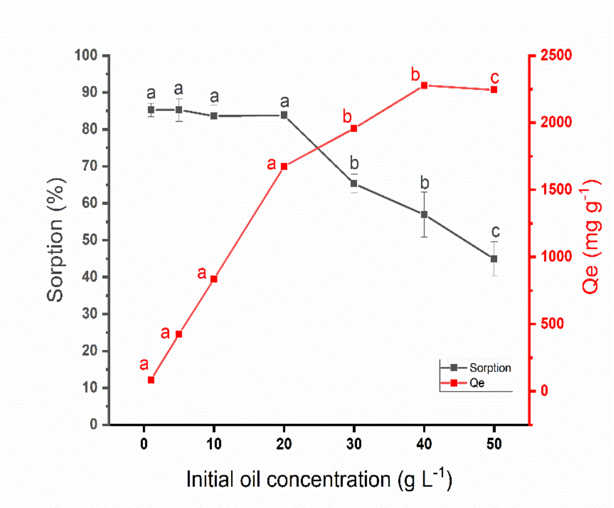


Fig. 7: Effect of Initial oil concentration on adsorption of crude oil by DPTFs: oil adsorption percent and sorbent capacity (15min, $10 gl^{-1}$ sorbent concentration, sorbent particle size 1-2 mm, pH 8–10, $21\pm3^{\circ C}$). Different letters show significant differences according to Tukey's test (at $p<0.05$).

The Langmuir and Freundlich isotherm equations have been used to describe the equilibrium characteristics of oil adsorption onto DPTFs. In Langmuir theory the basic assumption is that the sorption occurs at specific homogeneous sites with the formation of a monolayer sorbate on the outer surface The amount of DPTFs in crude oil solution was observed to significantly affect the oil uptake (15min, 10gL-1 sorbate concentration, sorbent particle size 1-2 mm, pH 8–10, 21±3°C) that shown in Fig. 9. It can be seen here that an increase in adsorbent dosage from 0.5-3gL-1 resulted in an initial increase in the oil removal percentages from 70.42 to 83.62% and beyond a certain dose it became constant (83-89%). This increase in oil adsorption efficiency was due to the availability of more active sites on the sorbent surfaces and after that with saturation of adsorbent, oil adsorption did not changed significantly [33, 34]. of the adsorbent. The linear Langmuir equation is [25]:

$$\frac{C_e}{q_e} = \frac{C_e}{q_{max}} + \frac{1}{q_{max} K_L} \qquad \text{Eq. (4)}$$

Where qe ($mg\ g^{-1}$) is the amount of adsorbed oil at equilibrium, Ce ($mg\ L^{-1}$) is the sorbate concentration at equilibrium, q_{max} is the maximum adsorption capacity and K_L (mgl^{-1}) refers to the Langmuir constant, which is related to the adsorption energy. The Langmuir constants q_{max} and KL were determined from the slope and intercept of the plot C_e/q_e versus C_e [26].

In Langmuir isotherm, a dimensionless constant, commonly known as separation factor [3] that is defined by the following equation:

$$R_L = \frac{1}{1+K_1C_0} \qquad \text{Eq.(5)}$$

The value of RL indicates that the adsorption nature could be either unfavorable if RL>1, linear if RL=1, favorable if 0<RL<1 and irreversible if RL=0[25]. Whereas the isotherm Freundlich assumes that the adsorption occurs at heterogeneous surface. The linear form of Freundlich can be represented as:

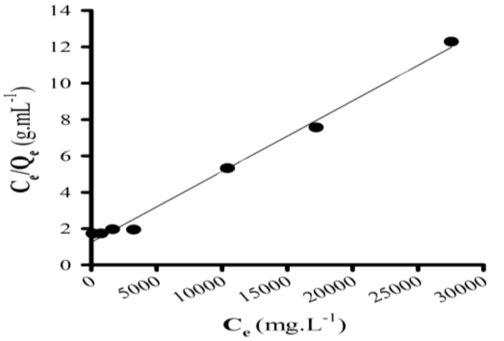
$$\ln q_e = \ln K_f + \left(\frac{1}{n}\right) \ln C_e \qquad \text{Eq.(6)}$$

Where q_e (mgg^{-1}) is the adsorbed amount at equilibrium, Ce (mgl^{-1}) is the equilibrium concentration of the sorbate. K_f (mgg^{-1}) is Freundlich constant for sorption capacity and 1/n is adsorption intensity of the sorbent. Generally, the values of n in the range of 2–10, 1–2, and <1 represent good, moderately difficult and poor sorption characteristics, respectively [13, 27].

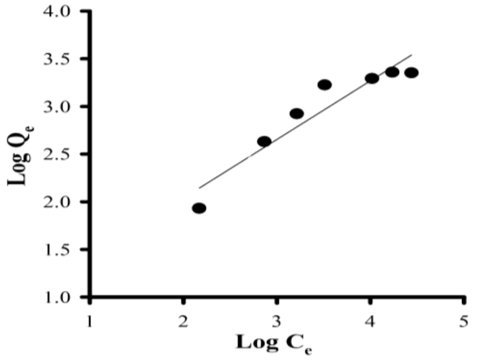
The Langmuir and Freundlich isotherm constants were determined from the plots of C_e/q_e versus C_e (Fig. 8a), $\ln q_e$ versus $\ln C_e$ (Fig. 8b). The constants for two isotherms, and the correlation coefficients, R^2 with the experimental data are listed in Table 2. The correlation coefficients for the Langmuir isotherm are highest in comparison to the value obtained for the Freundlich isotherm. Therefore, the Langmuir isotherm is the best-fit isotherm for the adsorption of crude oil onto DPTFs.

The oil adsorption on DPTFs was better fitted by the Langmuir isotherm (R^2=0.99) other than Freundlich model (R^2= 0.897) and the maximum adsorption capacity is 2500 mg g-1. Based on the value of RL factor ($0<R_L<1$), the crude oil adsorption on DPTFs was classified as favorable. It was clarified that the adsorption decrease of crude oil at the higher concentration could be attributed to a monolayer sorption process [3].

The adsorption capacity of DPTFs for crude oil in this study (2500mgg^{-1}) shows greater sorption advantages compared to some other natural organic sorbents used by other investigators (Table 3).



a) Langmuir



b) Freundlich

Fig. 8: Isotherm models for adsorption of crude oil by DPTFs: a) Langmuir and b) Freundlich

Table 2: Isotherm parameters obtained from Langmuir and Freundlich models for oil adsorption by DPTFs (15min, 10gl^{-1} sorbent concentration, sorbent particle size 1-2mm, and pH 8–10, 21±3 $^{\circ C}$).

Isotherm	Parameter	Value
Langmuir	q_{max} (mg g-1)	2500
	K_L (Lmg^{-1})	1.27×10-7
	R_L	0.99
	R^2	0.99
Freundlich	KF (Lmg^{-1})	0.8119
	n	0.3556
	R^2	0.897

Table3: Crude Oil adsorption capacity of some natural organic fibrous sorbents.

Adsorbent	Sorption capacity (mg g^{-1})	Reference
Meshed groundnut shell	1100	[28]
Rice husk	2860	[29]
Ground kenaf bast	450	[23]
Ground cotton	760	
Garlic peels	385	[30]
Onion peels	455	
Eggshell	108.69	[31]
Raw corncobs	0.0043	[32]
Date palm trunk fiber	2500	This study

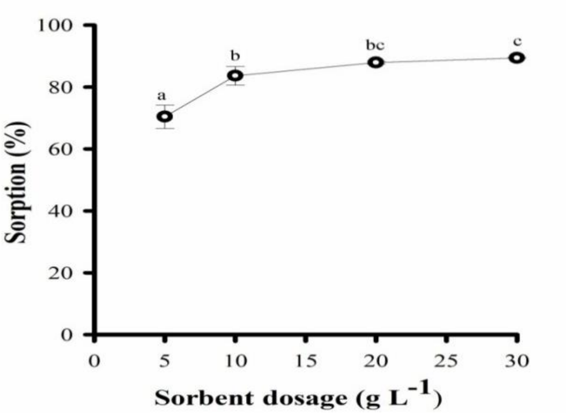


Fig. 9: Effect of contact time on adsorption of crude oil by DPTFs (15min, 10gl^{-1} sorbate concentration, sorbent particle size 1-2 mm, pH 8–10, 21±3$^{\circ C}$). Different letters show significant differences according to Tukey's test (at p<0.05).

CONCLUSIONS

In this study, the efficiency of DPTFs for the crude oil adsorption from aqueous solution was investigated. The results indicated that pH did not play a key role in the oil adsorption. It was also observed that the adsorption of oil on DPTFs is dependent on adsorbent particle size so that decreasing particle size of the adsorbent reduced the oil adsorption percentage due to damage of its active site. The kinetic studies reveal that oil adsorption on DPTFs is rapid and the pseudo second order kinetic governed oil adsorption. The isotherm study indicated that the oil adsorption was fitted well by the Langmuir model. In general, the results of this study indicated the high sorption capacity of DPTFs sorbent without any pretreatment (2500mg g-1). Therefore, in some emergency situations caused by oil and petroleum products spills, the use of such raw sorbents as a low cost, environmental friendly sorbents and with high adsorption for rapid and economic treatment of contaminated water could be justifiable.

ETHICAL ISSUES

Ethical issues such as plagiarism have been considered by the authors.

CONFLICT OF INTERESTS

The authors have declared no conflict of interest.

AUTHORS' CONTRIBUTIONS

Boleydei H. designed the study and carried out technical analysis of data. Razavi Z. coordinated research activities, contributed in conceptualization, preparing and results interpretation of the paper. All authors read and approved the final manuscript.

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This work was done with the author's personal budget.

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