

Mann's Cucumeropsis Seed Shell as an Oil Sorbent: Kinetic, Isotherm, and Thermodynamic Studies

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Abstract: Oil spill cleanup using agricultural wastes, which otherwise cause environmental contamination, has gained popularity. In this study, the feasibility of using Mann's cucumeropsis seed shell, a commonly found agricultural waste, as an oil spill mop, was investigated. To identify the surface characteristics of the seed shell, SEM and BET analyses were employed. Batch crude oil sorption tests were performed under various adsorption conditions to determine the effects of initial oil concentration, contact time, adsorption temperature, and adsorbent dosage. Kinetic study of the sorption process involved the pseudo-first order, pseudo-second order, Elovich, and intraparticle diffusion models. Isotherm analysis involved the Langmuir, Temkin, Freundlich, and Dubinin-Radushkevich isotherms. Additionally, the thermodynamic parameters for the sorption process were established. SEM analysis revealed the porous nature of the seed shell's surface. The BET surface area was 313.2 m²/g. The oil sorption process conformed best to the pseudo-first-order kinetics and Langmuir isotherm. The monolayer sorption capacity of the seed shell was 13.072 g/g. The sorption process was exothermic, with some order at the adsorbent–mixture interface. The study's findings demonstrate that Mann's cucumeropsis seed shell is a potent oil sorbent that is safe for the environment and can be enhanced for use in cleaning up oil spills.

Keywords: Mann's Cucumeropsis Seed Shell, Adsorption, Kinetics, Isotherm, Thermodynamics

1. Introduction

Over time, rising crude oil consumption has increased the amount of crude oil produced/explored, transported, and stored, which has raised the chances of an environmental spill. The environment gets contaminated whether the spill was accidental or deliberate. Harmful compounds are discharged into the environment after an oil spill which negatively impacts the ecosystem for a long time [1]. Therefore, it is essential to create a quick and efficient method for recovering any spilled oil and potentially recycling it.

Oil spills on land are significantly simpler to clean up than those on the surface of the sea [2]. The cleanup of spills on land may be done ex-situ or in-situ. Ex situ remediation requires physically removing the contaminated soil so that it

may be treated elsewhere, whereas in situ remediation addresses the polluted land where it is. Many physical, biological, chemical, and sorption technologies are being utilized to treat oil spills on water. However, the natural adsorption method, which uses readily available agricultural wastes and materials, is preferred because it is straightforward, inexpensive, and effective [3, 4], as well as because the sorbents are readily available, renewable, and biodegradable [5, 6]. The crude oil sorption potential of various agricultural materials and wastes has been investigated. These include mangrove bark peel [7], coconut coir [8], cotton fiber [9], banana peels [10], luffa fibers [11], orange peel [12], rice husk [13], Neptune grass fiber [14], and date palm trunk fiber [15].

Mann's cucumeropsis (*Cucumeropsis mannii*), also commonly called white seed melon, is a member of the family of fruits and vegetables known as cucurbits, or

Cucurbitaceae [16]. The plant, which is normally grown from March to September when it rains, yields a climbing vine that may reach a height of 4 meters and little yellow female and male flowers whose petals are under a centimeter long [17]. The fruits, which are egg-shaped or elongated ovate, are cream-colored with green stripes and can reach a length of 19 cm and width of 8 cm [18]. The shelled seeds are ground into a flour or paste and used as an emulsifier, thickener, fat binder, and flavoring ingredient in traditional soups such as egusi soup in Nigeria, Cameroon, and Benin and pistache soup in Côte d'Ivoire [19]. The shelled seeds can also be roasted and consumed as a snack, while the fermented shelled seeds are used to make ogiri, which is a sauce or soup condiment. Being inedible and typically discarded, the seed shell contributes to environmental pollution. As far as we are aware, no research has been done on the use of Mann's Cucumeropsis seed shell as a crude oil adsorbent. Therefore, this study aims to investigate the crude oil sorption behavior of Mann's Cucumeropsis seed shell. Kinetic, isotherm, and thermodynamic studies of the adsorption process were also conducted.

2. Materials and Methods

2.1. Materials

Mann's cucumeropsis seeds were obtained from Eke Awka market in Anambra state of Nigeria. The seeds were shelled, and the shells were washed, dried, ground, and sieved through a British standard sieve size 25. Crude oil was obtained from the Port Harcourt refinery, Rivers State, Nigeria.

2.2. Characterization of Mann's Cucumeropsis Seed Shell

The bulk and tapped densities, ash content, and moisture content of Mann's Cucumeropsis seed shell (MCSS) were determined using ASTM D 6683–08, ASTM D 1102–84, and ASTM D 4442–07, respectively, as described by Azubuike and Okhamafe [20]. A BET analyzer (Quantachrome NOVA 4200e) was used to determine the pore volume, size, and surface area of the MCSS. A scanning electron microscope (PhenomWorld) was used to observe the surface morphology of the MCSS.

2.3. Crude Oil Adsorption

The sorption of crude oil from an aqueous medium was conducted in a batch system according to ASTM F726-99 as described by Behnood *et al.* [21]. A weighed portion (0.2 g) of the MCSS was contacted with 100 g/L of crude oil at room temperature. After 5 minutes of sorption time, with a little agitation, a sieve net was used to remove the MCSS sorbent from the beaker, and the excess oil was allowed to drain. The oil-loaded sorbent was then weighed and the oil sorption capacity, q (g/g), was calculated using Equation (1).

$$q = \frac{W_1 - W_0}{W_0} \quad (1)$$

where W_1 and W_0 (g) are the weights of MCSS after and before oil sorption, respectively.

The effects of adsorption conditions were studied by varying the adsorbent dosage, initial crude oil concentration, and temperature from 0.2 g to 1.0 g, 50 g/L to 150 g/L, and 303 K to 323 K, respectively. The data obtained from the variations of initial crude oil concentration and temperature was used for isotherm and thermodynamic studies, respectively.

For kinetic analysis, the oil sorption tests were allowed to proceed for 1, 3, 5, 10, and 15 minutes of contact. The amount of crude oil adsorbed per unit weight of adsorbent, q_e (mg/g), was calculated using equation 2.

$$q = \frac{C_0 - C_e}{m} \quad (2)$$

where C_e and C_0 (mg/L) are the equilibrium and initial crude oil concentration, respectively. V (L) is the volume of the solution and m (g) is the mass of the MCSS adsorbent.

3. Results and Discussions

3.1. Characterization of Mann's Cucumeropsis Seed Shell

The characterizing properties and surface morphology of MCSS are shown in Table 1 and Figure 1, respectively.

Table 1. Properties of Mann's Cucumeropsis seed shell.

Parameter	Value
Surface area (m ² /g)	313.2
Pore size (nm)	2.118
Pore volume (cc/g)	0.1547
Bulk density (g/cm ³)	0.693
Tapped density (g/cm ³)	1.448
Moisture content (%)	5.438
Ash content (%)	0.726

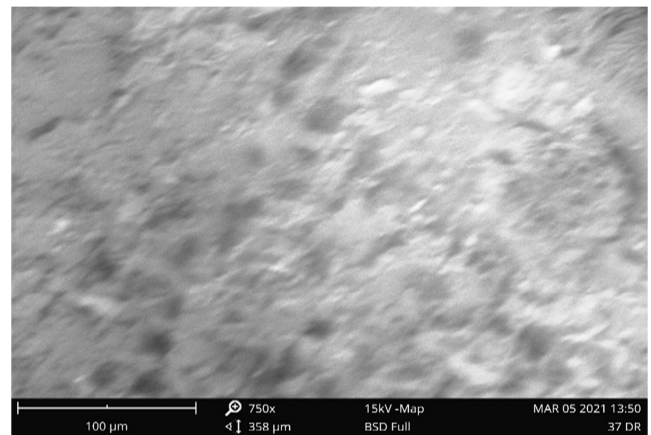


Figure 1. SEM image of Mann's Cucumeropsis seed shell.

The results show that MCSS is porous with a high surface area. This is desirable because large surface area enhances the contact between adsorbent and adsorbate [22]. The densities and moisture content of MCSS are moderate and are consistent with previous reports [23]. Ash content is known to promote hydrophilicity and can have catalytic

effects that cause restructuring processes during the regeneration of spent sorbent [24]. The low ash content of MCSS (0.726%) suggests that it has low hydrophilicity, and so will be suitable for oil sorption in an aqueous medium. It also suggests that the regeneration process will not lead to significant restructuring of the MCSS sorbent. Hence, it is likely that it can be reused multiple times, thereby making its use cost-effective.

3.2. Effect of Adsorption Conditions on the Oil Sorption Capacity of Mann's Cucumeropsis Seed Shell

The effects of contact time, initial crude oil concentration, adsorbent dosage, and temperature on the oil sorption capacity of MCSS are shown in Figure 2a, b, c, and d, respectively. The oil sorption capacity increased as contact time increased. The sorption process started quickly during the early phases of contact before slowing down as it approached equilibrium. This is likely because there were a lot of empty surface sites and microscopic spaces available for sorption in the early stages. But after some time had passed, the filling of the empty sites left became challenging due to the repelling interactions between the oil molecules on the adsorbent and those in the bulk phase [25].

An increase in initial crude oil concentration from 50 g/L to 125 g/L also resulted in increased oil sorption capacity (Figure 2b). However, a further increase in the initial oil concentration led to decreased oil sorption capacity. This can be explained by the fact that increasing initial sorbate concentration increases the concentration gradient between the bulk sorbate and the sorbent surface, thereby increasing diffusion of the sorbate to the sorbent, which subsequently increases the sorption capacity. However, at very high sorbate concentrations, the surface of the sorbent becomes

saturated, which could lead to a reduction of its sorption capacity [26].

Figure 2c shows that an increase in adsorbent dosage led to decreasing oil sorption capacity. This decrease may be the result of sorption sites clustering together or overlapping as the sorbent dose rises. This reduces the overall adsorption surface and lengthens the diffusion path, which in turn results in a decrease in the amount of adsorbate attached to a unit mass of the adsorbent, and subsequently lowers the sorption capacity [27, 28]. Similar behavior was observed by Abdelwahab [11], who found that the sorption capacity of luffa fibers for crude oil and diesel oil decreased from 15 g/g to 5 g/g and from 27 g/g to 10 g/g, respectively on increasing the sorbent dose from 0.1 g to 0.5 g.

The oil sorption capacity of MCSS also decreased with an increase in temperature (Figure 2d). This might be because a temperature rise accelerates the oil molecules' Brownian motion, necessitating a larger force to hold the molecules to the sorbent surface [29]. Additionally, as the temperature rises, crude oil's viscosity decreases and its solubility in water increases. As a result, the interaction forces between the solute and the solvent become stronger than those between the solute and the sorbent, making it harder to adsorb the oil and making the adsorbed oil easily drain off the surface of the sorbent [30]. The fact that the oil sorption capacity decreased with an increase in temperature indicates the exothermic nature of the adsorption processes. Decrease in oil sorption capacity with increasing temperature was also reported by El-Din et al. [10]. The crude oil sorption capacity of banana peel decreased from 7.14 g/g – 7.94 g/g to 3.25 g/g – 4.52 g/g on increasing the adsorption temperature from 20°C to 45°C.

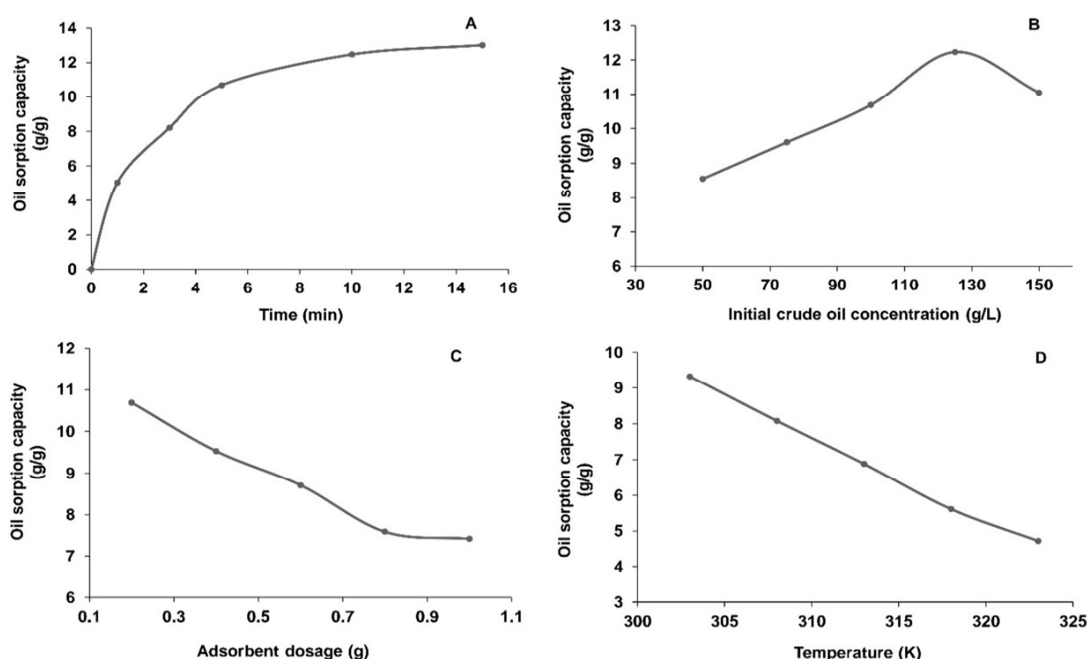


Figure 2. Effect of (a) contact time (b) initial crude oil concentration (c) adsorbent dosage, and (d) temperature on the oil adsorption capacity of Mann's Cucumeropsis seed shell.

3.3. Kinetic Studies

A kinetic study is used to investigate the kind of mechanism governing an adsorption process. The kinetics of the oil sorption on MCSS were investigated using a variety of kinetic models. The models used are the pseudo-first order, pseudo-second order, Elovich, and intraparticle diffusion models, expressed in equations 3, 4, 5, and 6, respectively [31, 32].

$$\ln(q_e - q_t) = \ln q_e - k_1 t \quad (3)$$

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^k} + \frac{t}{q_t} \quad (4)$$

$$q_t = \frac{1}{\beta} \ln(\alpha\beta) + \frac{1}{\beta} \ln t \quad (5)$$

$$q_t = K_{id} t^{0.5} + C \quad (6)$$

where q_e and q_t (mg/g) are the amounts of adsorbate adsorbed at equilibrium and at time t (min), respectively. k_1 (min^{-1}), k_2 ($\text{g}/(\text{mg} \cdot \text{min})$), and k_{id} ($\text{mg}/(\text{g} \cdot \text{min}^{0.5})$) are the pseudo-first order, pseudo-second order, and intraparticle diffusion rate constants, respectively. α ($\text{mg}/(\text{g} \cdot \text{min})$) and β are the initial adsorption rate and the desorption constant, respectively.

The pseudo-first order, pseudo-second order, Elovich, and intraparticle diffusion plots for the adsorption of crude oil on MCSS are shown in Figures 3 a, b, c, and d, respectively. The pseudo-second order plot gave the highest R^2 value, which should suggest that the sorption process was by chemisorption [33]. But, the Elovich model, which is also based on the chemisorption of adsorbate, gave an R^2 value

that is lower than that of the pseudo-first order plot. The R^2 value of the pseudo-first order plot is close to that of the pseudo-second order plot, and the obtained q_e value is the closest to the experimentally obtained value as shown in Table 2. Thus, it can be said that the oil sorption process is best described by the pseudo-first order kinetics. This implies that the sorption process was by physisorption [34].

Despite being linear, the intraparticle diffusion plot did not go through the origin. This demonstrates that intraparticle diffusion contributed to the oil sorption process, but it was not the only mechanism [35]. This indicates that there was surface sorption of the oil on the MCSS in addition to sorption within the fiber network.

Table 2. Kinetic parameters for crude oil adsorption on Mann's Cucumeropsis seed shell.

Parameter	Value
q_e , exp (mg/g)	13015
Pseudo-first order model	
k_1 (min^{-1})	0.3122
q_e , calc. (mg/g)	11891
Pseudo-second order model	
k_2 ($\text{g}/(\text{mg} \cdot \text{min})$)	4.90×10^{-5}
q_e , calc. (mg/g)	14286
Elovich model	
β (g/mg)	3.25×10^{-4}
α ($\text{mg}/(\text{g} \cdot \text{min})$)	16346
Intraparticle diffusion model	
K_{id} ($\text{mg}/(\text{g} \cdot \text{min}^{1/2})$)	2769.12
C	3236.24

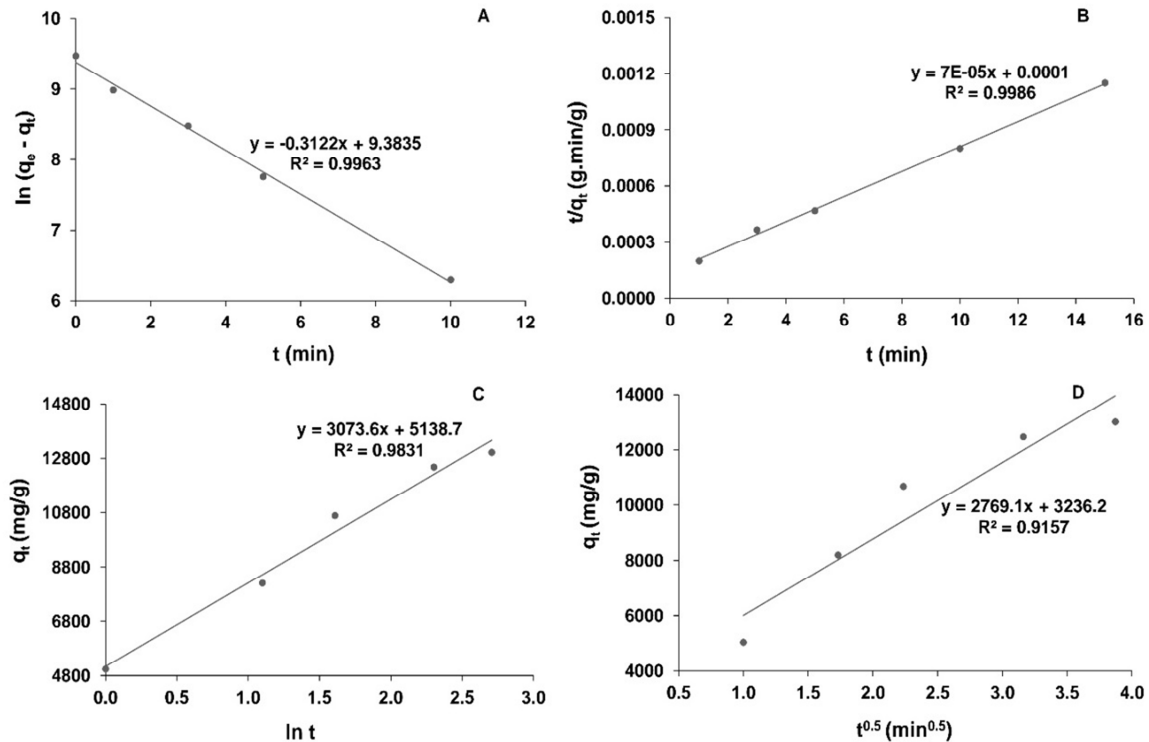


Figure 3. (a) Pseudo-first order (b) pseudo-second order (c) Elovich, and (d) intraparticle diffusion plots for crude oil adsorption on Mann's Cucumeropsis seed shell.

3.4. Isotherm Studies

Adsorption isotherms are essential for the efficient design of adsorption systems, optimization of the adsorption mechanism pathways, and for expressing the surface qualities and capabilities of adsorbents [36]. In this study, the oil sorption on MCSS was further studied using the Langmuir, Freundlich, Temkin, and Dubinin-Radushkevich isotherms, expressed in equations 7, 8, 9, and 10, respectively [37, 38].

$$\frac{C_e}{q_e} = \frac{1}{K_L q_m} + \frac{C_e}{q_m} \quad (7)$$

$$\ln q_e = \ln K_F + \frac{1}{n} \ln C_e \quad (8)$$

$$q_e = \frac{RT}{b} \ln K_T + \frac{RT}{b} \ln C_e \quad (9)$$

$$\ln q_e = \ln q_m - \beta \varepsilon^2 \quad (10)$$

$$\varepsilon = RT \ln (1 + 1/C_e) \quad (11)$$

where C_e (g/L) is the equilibrium concentration of the adsorbate, q_e and q_m (g/g) are the equilibrium and monolayer sorption capacities, respectively. K_L (L/g) is the Langmuir constant, K_T (L/g) and B are Temkin constants, K_F [(g/g)(L/g)^{1/n}] and n are Freundlich constants related to adsorption capacity and intensity, respectively. β is the Dubinin-Radushkevich coefficient, ε is Polanyi potential, R is the universal gas constant (8.314 J/mol.K), and T (K) is the absolute temperature.

The Langmuir, Freundlich, Temkin, and Dubinin-Radushkevich isotherms for crude oil adsorption on MCSS are shown in Figures 4a, b, c, and d, respectively. The isotherm parameters are listed in Table 3. The Langmuir isotherm gave the highest R^2 values, which suggests that the crude oil sorption process involved the crude oil covering the

MCSS surface in a monolayer [8]. The monolayer sorption capacity was found to be 13.015 g/g. The affinity between the crude oil and the MCSS was predicted using a dimensionless constant known as the separation factor (R_L), expressed in equation 12 [39].

$$R_L = \frac{1}{1 + K_L C_0} \quad (12)$$

C_0 is the initial crude oil concentration. The R_L value indicates that the adsorption irreversible if $R_L=0$; unfavorable if $R_L>1$, linear if $R_L=1$, and favorable if $0<R_L<1$ [33]. The R_L values for the oil sorption on MCSS obtained using various values of C_0 were in the range of $0<R_L<1$ as shown in Table 3. Hence, the oil sorption process was favorable. This is supported by the n value of 4.261 from the Freundlich isotherm which lies between 1 and 10, thereby indicating favorable adsorption [40].

Table 3. Isotherm parameters for crude oil adsorption on Mann's Cucumeropsis seed shell.

Parameter	Value
Langmuir isotherm	
q_m (g/g)	13.072
K_L (L/g)	5.87×10^{-2}
R_L	0.254 - 0.102
Freundlich isotherm	
K_F [(g/g)(L/g) ^{1/n}]	3.800
n	4.261
Temkin isotherm	
K_T (L/g)	1.155
b (J/mol)	1.052
D-R isotherm	
β	6×10^{-5}
q_m (g/g)	11.539
E (kJ/mol)	0.091

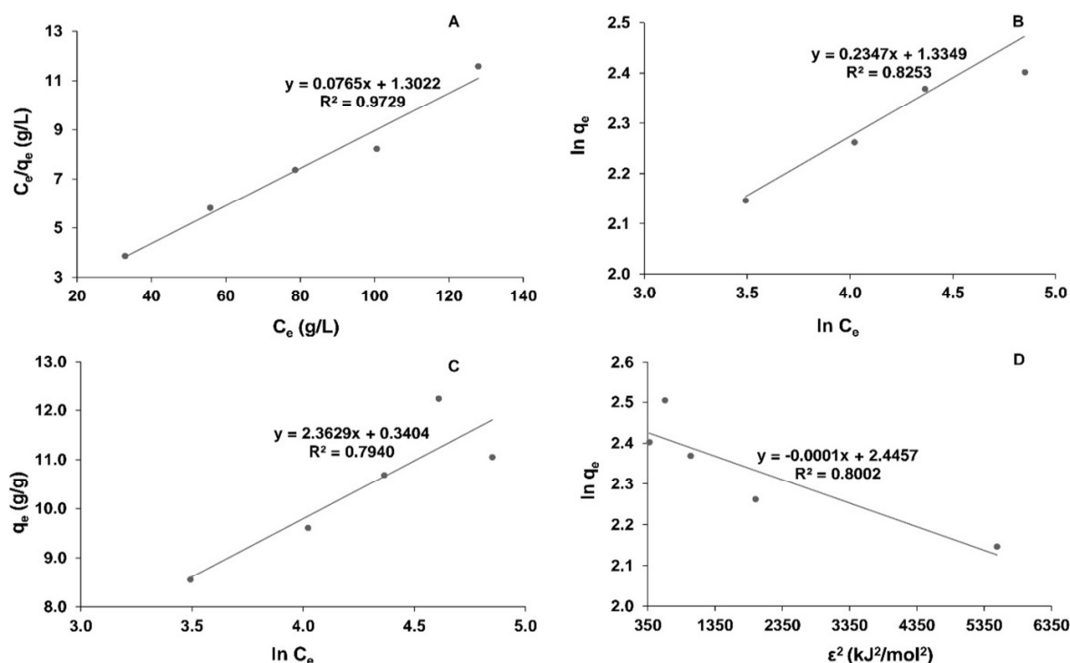


Figure 4. (a) Langmuir (b) Freundlich (c) Temkin, and (d) Dubinin-Radushkevich isotherms for crude oil adsorption on Mann's Cucumeropsis seed shell.

Dubinin-Radushkevich coefficient (β) can be used to obtain the mean free energy of adsorption, E (kJ/mol) as follows.

$$E = \frac{1}{\sqrt{2\beta}} \quad (13)$$

$E < 8$ kJ/mol indicates a physical sorption process, while E between 8 and 16 kJ/mol indicates a chemical sorption process [41]. The value of E for the oil sorption on MCSS was 0.091 kJ/mol. This indicates that the oil sorption was physical in nature, and supports the observation made during the kinetic study of the sorption process.

3.5. Thermodynamic Studies

The standard change in Gibbs free energy for the crude oil sorption process was calculated based on equation 14 [42].

$$\Delta G^\circ = -RT \ln K_d \quad (14)$$

$$K_d = \frac{C_o - C_e}{C_e} \quad (15)$$

where ΔG° (kJ/mol) is the free energy change, T is the absolute temperature (K), R is the universal constant (8.314 J/mol.K), and K_d is the linear sorption distribution coefficient. The values of ΔG° were found to be positive at all the considered adsorption temperatures (Table 4). This shows that the oil sorption on MCSS was non-spontaneous at the considered temperatures [43]. Lower negative ΔG° was obtained with increasing temperatures, indicating more effective adsorption at low temperatures. This is consistent with the earlier observation that MCSS's oil sorption capacity decreased with temperature rise.

Table 4. Thermodynamic parameters for crude oil adsorption on MCSS.

Temperature (K)	ΔG° (kJ/mol)
303	3.713
308	4.217
313	4.775
318	5.467
323	6.074

The changes in enthalpy and entropy (ΔH° and ΔS° , respectively) were calculated using the Van't Hoff equation expressed as follows [40].

$$\ln K = \frac{\Delta S^\circ}{R} - \frac{\Delta H^\circ}{RT} \quad (16)$$

A linear plot of $\ln K$ versus $1/T$ (Figure 5) yielded ΔH° and ΔS° from its slope and intercept, respectively. The ΔH° and ΔS° values are -32.383 kJ/mol and -0.119 kJ/mol · K, respectively. The ΔH° value is lower than 80 kJ/mol and is a negative value, showing that the oil sorption on MCSS was by physical adsorption and the process was exothermic in nature [42]. The negative ΔS° value indicates that there was decreased randomness at the adsorbent-adsorbate interface during the sorption process as a result of the orderly arrangement of the crude oil molecules on the MCSS's

surface [44].

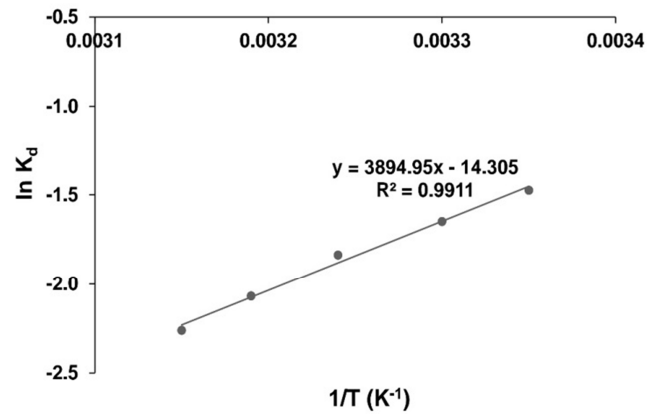


Figure 5. Van't Hoff plot for crude oil adsorption on Mann's Cucumeropsis seed shell.

3.6. Comparison of Oil Sorption Capacity of MCSS with Those of Other Natural Sorbents

Table 5 compares the crude oil sorption capacity of MCSS with those of some other agricultural materials and wastes. The sorption capacity of MCSS is lower than those of some other materials such as Neptune grass and cotton fibers. However, it is higher than those of mangrove bark, banana and orange peels, coconut coir, rice husk, and date palm trunk fiber. Thus, MCSS is an efficient crude oil sorbent that can be used for the removal of crude oil from an aqueous medium.

Table 5. Oil sorption capacity of some agricultural materials.

Adsorbent	Oil sorption capacity (g/g)	Reference
Banana peel	6.35	[10]
Mangrove bark peel	1.12	[7]
Rice husk	2.55	[13]
Coconut coir	9.2	[8]
Cotton fiber	30.5	[9]
Orange peel	5	[12]
Neptune grass fiber	14	[14]
Date palm trunk fiber	2.5	[15]
MCSS	13.02	This work

4. Conclusion

Crude oil sorption on MCSS was affected by variations in the contact time, initial oil concentration, sorbent dosage, and adsorption temperature. The oil sorption capacity of MCSS increased with increasing contact time and initial oil concentration until sorption equilibrium was attained. An increase in sorbent dosage and adsorption temperature, on the other hand, resulted in decreased oil sorption capacity. The sorption process was physical in nature and involved monolayer coverage of the surface of MCSS by the crude oil. The sorption process was exothermic and involved an orderly arrangement of the crude oil molecules on the surface of the MCSS. Thus MCSS can be used for effective sorption of crude oil from aqueous medium at ambient conditions with

minimal sorbent dosage requirement. The relatively high oil sorption capacity of MCSS shows that it is an efficient, alternative oil sorbent. Considering that oil sorption capacity of adsorbents increases with increasing surface area and porosity, modification of the MCSS using methods such as carbonization, acid impregnation or grafting is suggested in order to further increase its surface properties and consequently, its oil sorption capacity.

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