Isotherm Studies on Oil Removal from Produced Water Using Mango Seed Kernel Powder as Sorbent Material

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Abstract

Oil pollution has been a major pollution source in most countries where crude oil exploration takes place. Produced water is the largest waste containing oil usually obtainable from oil and gas exploration activities. This water contains ≥30% oil and is harmful to aquatic lives and the environment where it is disposed. In this work, mango seed kernel (MSK) was processed and used as oil sorbent from produced water with 93.33% oil removal from initial oil content of 30 ppm down to 2 ppm. The results showed that this bio-waste can satisfactorily substitute the costly means of conventional methods used in produced water treatment. The generated data from the sorption experiments were used in isotherm data fitting. Freundlich isotherm fitted the data better than Langmuir, Temkin, and Dubinin isotherms with coefficient of regression (R^2) 0.9638. This showed that the sorption behavior is physical in nature and can be desorbed easily. Elemental analysis on the sorbent revealed that it contains mainly carbon (60.95%) and oxygen (39.05%). It was found to have a surface area of 0.0036 m^2/g by BET method. The surface contained tiny pores suitable for sorption process. An FTIR run on the sample revealed the presence of several peaks indicating the presence of different functional groups.

Keywords: mango seed kernel, isotherm, characterization, produced water

1. Introduction

Equilibrium relationships between sorbents and sorbates are described by sorption isotherms which give the capacity of a sorbent for a sorbate. Isotherms can be obtained by examining batch reactions at fixed temperatures. Linear regression is frequently used to determine the best-fitting isotherm (Ho, 2006; and Itodo,*et al.*, December, 2010).

Wastewaters generated by many industries such as oil refineries and oil producing companies contain dissolved and dispersed oils which are harmful to the aquatic lives and the environment in general. Most oils are carcinogenic and mutagenic due to the presence and involvement of hazardous chemicals in their processes. Hence, oil removal from such wastewaters is known as one of the major environmental challenges.

There are currently numerous treatment methods for such wastewaters due to the presence of oil; amongst which were biodegradation, chemical oxidation, foam flotation, electrolysis, adsorption, chemical coagulation and photo-catalysis. Nevertheless, adsorption processes provide an effective alternative treatment approach for oil removal due to the lower initial cost, sludge free clean, flexibility and simplicity of design, and operation, easy recovery and resistance to toxic pollutants. Since most agricultural solid wastes are inexpensive, abundant and easily available, many researchers have focused on the feasibility of the low-cost materials that were derived from agricultural wastes for the removal of various oil types, heavy metals and other pollutants (Mohamad *et al*., 2012). Wastes or by-products of industries can be used directly or after some treatments as adsorbents in adsorption processes. Mango seed is one of the fruits that are abundant in northern Nigeria and is a by-product of fruit industries that has no application apart from its use as seed for growing the plant. Produced water usually represents a waste product in the petroleum industry; and its beneficial prior to disposal treatment is required in order to meet predisposal regulatory limits or meet beneficial use specification. While the petroleum industries quite often prefer to utilize low cost disposal options such as discharge to surface waters, government and environmental regulators require that the produced water must meet limits for key parameters. The parameters might be specific constituents of concerns such as ammonia or barium that can be toxic to sensitive animal and plant life. Or the parameters may be more broadly-based such as total dissolved solids (TDS) or sodium adsorption ratio (SAR) that can affect several aspects of environment.

The sorption of a chemical on a solid from a water solution may be seen as the result of a reversible process (sorption–desorption) which reaches a final equilibrium condition between the concentration of the chemical in the two hazes. Considerable research have been done on the possibility of utilizing agricultural waste materials in the removal of metals and salts from produced water but only few writers have targeted oil from produced

water. Table 1 provides a summary of various researches conducted and there target pollutants. In this paper, we report on our work aimed at testing isotherms with generated data from oil removal from produced water using mango seed kernel.

Table 1: Various researches conducted using Bio-waste materials

S/N	Biosorbents	Target Pollutant	Processing mode	Ref.
1.	Pyracanthacoccinea Berry biomass	Methylene blue	Washed with distilled water, dried in an Oven at 70°C, ground and sieved through 100 mesh	(Akar *et al*., 2009)
2.	Biomass immobilized	Pb II	Micro-organism isolated from polluted soil	(Cabuk *et al*., 2006)
3.	Hypneavalentiae Biomass	Cd ion	Dried seaweed washed with distilled water and shade dried	(Rathinam *et al*., 2010)
4.	Pyracnthacoccinea berry biomass	acid red 44 dye	Cathionic surfactant (HDEDMABr) mixed with as prepared Bio-sorbent and sieved through 212 µm with 1.5 g powder Suspended in 150 ml of 1% (w/v) surfactant solution and stirred for 24 h at room Temperature filtered and washed with deionised water and dried at 70°C in oven	(Akar *et al*., 2010)
5.	Red seaweed (marine algae)	Uranium	Seaweed algae washed with tap water & then washed (marine algae) with distilled water, dried in an oven at 90°C for 24h, ground and sieved. This is mixed with *S. cerevesiae* & silica gel and wetted with distilled water to form paste, heated in oven at 105°C for 24h and sieved through <125µm.	(Aytos*et al.*, 2011)
6.	Cashew nuts	Nickel II	Rinse with water, dried at room temperature, ground To fine powder and sieved to 200-30 mesh.	(Kumar *et al.*, 2011)
7.	Industrial fungus (rhizopuscohnii)	Cadmium II	Cultivated and washed with water	(Luo *et al.*, 2010)
8.	Cyanobacteria biomass	Cr (VI)	immobilization in calcium alginate matrix	(Mona *et al.*, 2011)
9.	Blue-green algae	Cr^{3+}, Cd^{2+}, Cu^{2+}	grown at 35°C in zarrouk liquid	(Chijnacka *et al.*, 2005)
10.	Capsicum annuum seed	reactive blue 49	seed washed with deionised water, dried, ground, sieved, mixed with acetone, filtered and washed with deionised water severally and dried at 80°C	(Akar *et al*., 2011)
11.	CephalosporiumAphidicola cells (fungus)	acid red 57	growth for 7days, dried, filtered, spread on petri dishes and dried in oven at 60°C.	(Kiran *et al*., 2006)
12.	EnterobacterSpj.	Pb, Cu, & Cd	Screened from industrial wastewater	(Lu *et al*., 2006)
13.	SaccharamycesCerevisiaestarin	Pb (II)	Grown on agar slants using malt	(Cabuk *et al.*,

			extract	2007)
14.	Granular sludge	cadmium II	Aerobic	(Liu et al., 2003)
15.	Penicillium Sp.	Acid violet dye	cultured and isolated from coal sample	(Anjaneya *et al.*, 2009)
16.	Trametesversicolor	textile dyes direct blue 1 & 128	Cultivated in liquid medium	(Bayramoglu and Arica, 2007)
17.	Rice husk ash, Activated alumina, Fuller's earth, Coal fly ash, Saw dust of teak wood and neem bark	Cr(VI)	Rice husk burnt to ash. Saw dust thoroughly washed with water then with 0.1N NaOH to remove lignin based colour material, followed by 0.1N H2SO4. Finally, it is washed with distilled water several times and dried in oven at 105±5oC for 6 hrs.	(Bhattacharya *et al.*, 2008)
18.	Citrus and durian rind peptin	Cd(II), Cu(II), Zn(II), Pb(II) and Ni(II)	Durian rind mix with mild acid solution (1:9 solid-liquid) and gently stirred. pH adjusted to 2 with 1M HCl. The Solution extracted at 90oC for 4hrs. Resulting slurry filtered through cheese cloth and allowed to cool to room temperature. Acidified ethanol (4% HCl in 95% EtOH) added in the ration 1:4 v/v and kept for 1 hr. The mixture centrifuged for 15 minutes. Gel-like Precipitate collected and re-suspended in distilled water 1:4 w/v and the solution washed twice with 95% EtOH 1:2 v/v and centrifuged for 15 minutes. Precipitate dried in vacuum oven at 25oC for 8 hrs.	(Wai *et al.*, 2010)
19.	Areca catechu betel-nut tree	Lead (II), cadmium (II)	cut into small pieces and washed with tap water and then with distilled water, sun dried for 7days, dried in oven at (heartwood) 70oC for 48 hrs, ground in blender and store in a desiccators Again washed with distilled water until colour and turbidity Free then dried at 70oC for 48hrs and sieve to 200 µm.	(Chakaravarty *et al.*, 2010c) (Chakaravarty, *et al.*, 2010)
20.	CinnamomumComphora leaves	lead (II)	fallen leaves collected and washed with distilled water several times and dried in oven at 80oC for 24 hrs. Dried Leaves crushed to fine powder and sieved through >200 mesh. Wash with distilled water till free from colour and turbidity, and finally dried in oven at 80oC for 2days.	(Chen *et al.*, 2010)

| 21. | Cone biomass thujaorientalis | acid blue 40 textile dye | cones washed with deionised water, dried at 80oC for 48 hrs and crushed to size | (Akar *et al*., 2008) |
| 22. | Barley straw | emulsified oil | Washed to remove dirt, ground and washed with distilled Water till colour free and dried over night at 65oC. ii. powder modified by soaking in 1L of 2.5 mmolL- CPC solution, shaken in orbited shaker at 170rpm at room temperature, separated from liquid, washed with distilled water to remove surface surfactant, dried at 60oC over night. | (Ibrahim *et al.* 2010) |

2.0 Materials and Methods

2.1 Preparation of Adsorbent

The waste samples (mango seeds) were collected and their kernels removed manually. The kernels were oven dried at 70°C and later crushed to finer particles. The oil contained in the powdered kernel was removed using n-hexane in a reflux condenser. The cake was later washed with distilled water severally until free of any trace of n-hexane which was later dried in an oven at 70°C until constant weight. The dried samples were then chopped and ground using a laboratory blender and the resulting powders then sieved with a nominal size of 212 µm and labelled as the mango seed kernel (MSK) sample.

2.2 Characterization of Adsorbent

The functional groups present at the surface of the MSK sample were investigated by Fourier Transform Infrared analysis (FT-IR) (Perkin Elmer, 100 FTIRNorwalk), ranging from 4000 to $400cm^{-1}$. Surface morphology of the sorbent was investigated using a Hitachi X-650scanning electron microscope (SEM)(Tungsten filament, EHT 20.00kV) and LEO 1450 Scanning Electron Microscope (Tungsten filament, EHT 20.00kV).Brenner Emmett and Teller (BET) equipment (TriStar 3000 V6.05A) was used to determine the surface area of the MKS. In order to determine the elements present on MSK sample, electron dispersion spectroscopy (EDS) was employed.

2.3 Sorption Study

The experiments were carried out by taking 300 ml of 30 mg/l produced water and different quantities of mango seed kernel (MSK) and both were charged in a 600 ml beaker. The contents were then agitated at 450 rpm for 30 minutes using mechanical shaker at room temperature. The biosorbent and sorbate were separated using a 63 micron sieve. Studies on the effects of agitation time, and biosorbent dose were carried out using known amounts of biosorbents212 microns size. Oil solutions (300 ml) with different amounts of biosorbents were taken to study the effect of adsorbent dosage on the removal of oil. The biosorption experiments were carried out at room temperatures.

The produced water was obtained from one of the off-shores in Nigerian oil rich areas. pH and temperature were kept constant during the experiment. The produced water samples were treated differently with various quantities of MSK for a period of 30 minutes and a stirring speed of 450 rpm. At the end of the treatment, MSK was removed from the oil/water mixture by passing through 63microns sieve; the residual oil in the water was determined using 1-1-1-tri-chloroethane as solvent. The extract was analyzed for oil content using HACH DR/2000 spectrophotometer at a wavelength of 450 nm. The test was repeated until optimum loading point was identified. With the optimum loading kept constant, the time was varied to determine the effect of contact time on the batch sorption study.

3.0 Results and Discussion

3.1 MSK Characterization Result

Fourier Infra-Red spectroscopy conducted on the MSK revealed the presence of many peaks indicating different functional groups as presented in Figure 1. All assignments were made in accordance with Coates (2000). To view the surface morphology of the MSK, a scanning electron microscope was utilized. The image shown in Figure 2, indicates that the MSK consist of several pores arranged on the surface that can be suitable for pollutant uptake. Electron dispersion spectroscopy (Figure 3) on the MSK revealed that it consists of mainly 60.95% carbon and 39.05% oxygen only. Figure 3 presents the chart as obtained from the equipment. As measured by the BET machine, the surface area obtained by single point was 0.0055 m^2/g, and was found to have a single point adsorption capacity of 0.003304 cm^3/g, with average pore width of 3717.63 nm. The MSK was found to have a surface area by Langmuir method as 0.0039 m^2/g while 0.0036 m^2/g was obtained by BET method.
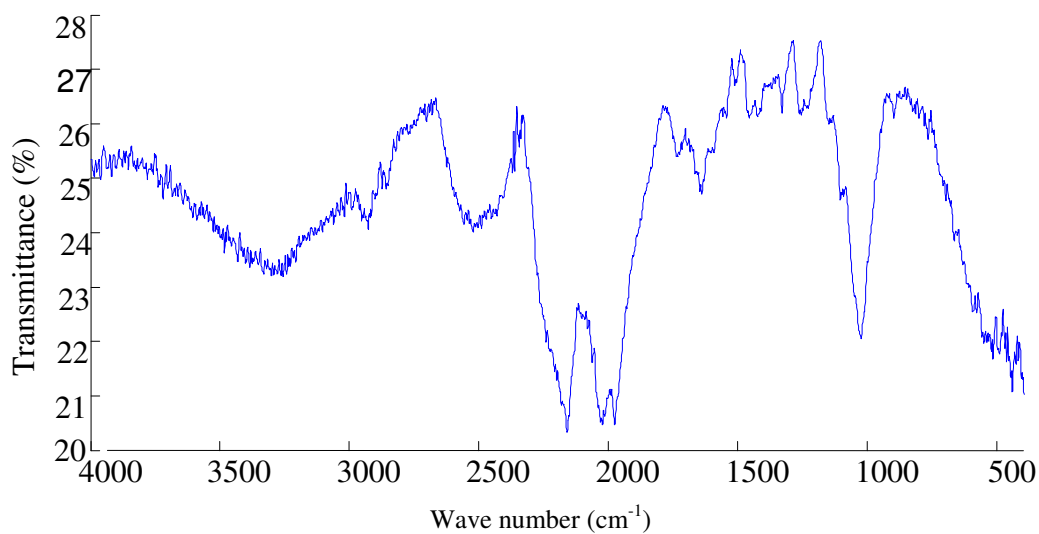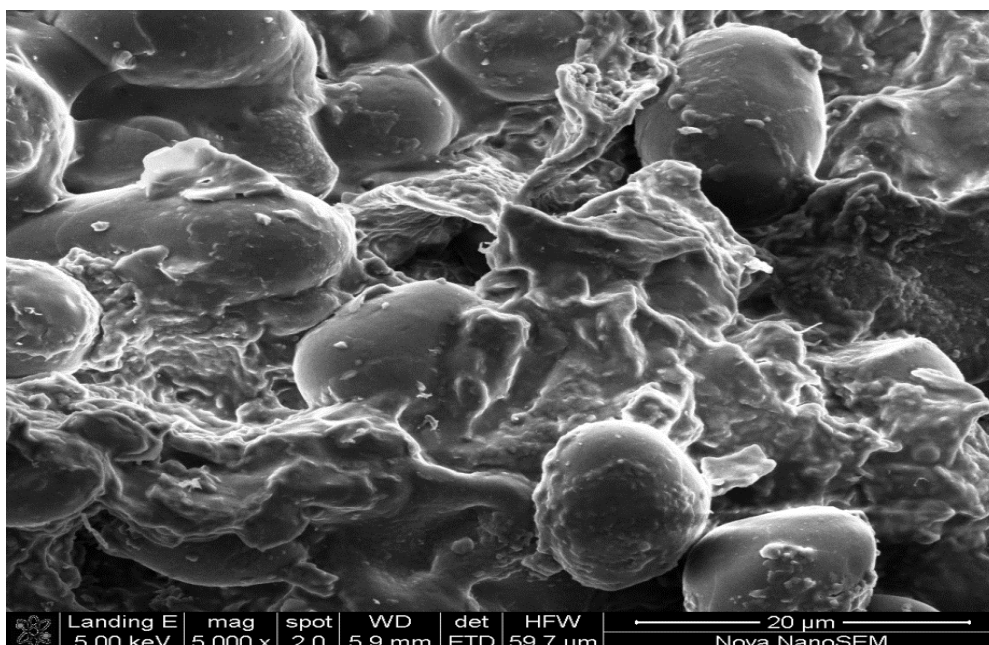

Figure 1: FTIR spectrum of MSK

Figure 2: Scanning Electron Microgram of MSK (magnification: 5000 times)

3.2 Sorption study

Table 2 presents the result of batch sorption studies using mango seed kernel as biosorbent with varying quantity of MKS. All other factors such as temperature (room temperature), pH (6.8), stirring speed (450 rpm), and sorption time (30 minutes) were kept constant.
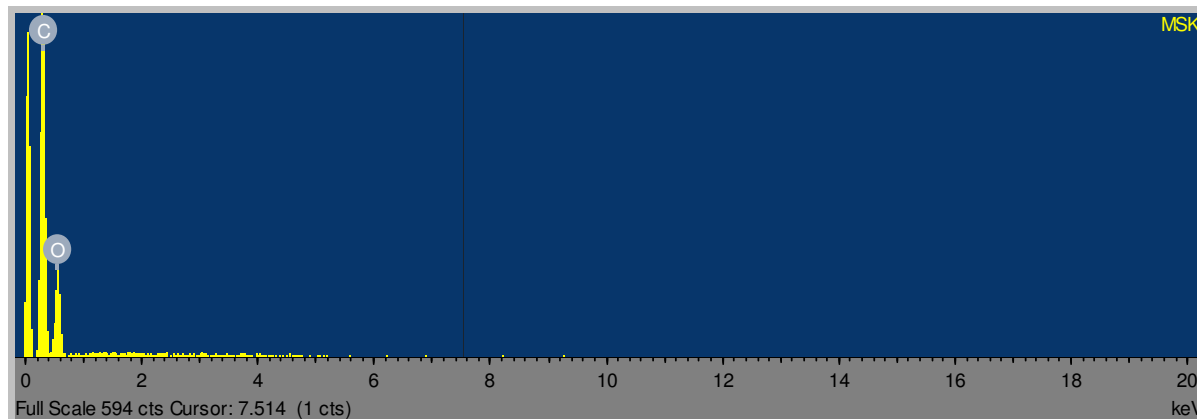

Figure 3: Elemental analysis made on mango seed kernel

Table 2: Data generated from sorption study with mango seed kernel biosorbent

Dosage (mg/L)	C_e	Oil Removal	% Oil Removal
0	30	0.0	0.00
333.33	8	22.0	73.33
500.00	6	24.0	80.00
666.67	4	26.0	86.67
1000.00	2	28.0	93.33
1333.33	2	28.00	93.33

3.2 Isotherms study

Sorption isotherm models are very important tools for the analysis of sorption process. Freundlich, Langmuir, Temkin-Pycher, and Dubinin-Radushkevich are the most widely used isotherms to investigate sorption process. The model parameters can be construed further, providing understanding on the sorption mechanism, surface properties, and an affinity of the sorbent.

3.2.1 Freundlich isotherm model

This sorption isotherm model is derived from the assumption of multi-layer sorption and for the sorption on heterogeneous surfaces. The theory says that the ratio of the amount of solute adsorbed onto a given mass of sorbent to the concentration of the solute in solution is not constant at different concentrations. The theory is presented in equation 1.

$$q_e = KC_e^{\frac{1}{n}} \tag{1}$$

The linear form of Eq. 1 is presented as Eq. 2.

$$\ln q_e = \ln K + \frac{1}{n} \ln C_e \tag{2}$$

where K is a constant indicative of the relative adsorption capacity of the sorbent (mg/g), 1/n is a constant indicative of the intensity of the adsorption. Both K and n are constants, being indicative of the extent of adsorption and the degree of non-linearity between solution and concentration respectively. The equilibrium sorption capacity q_e, defined by Eq. 3, is the mass solute adsorbed (mg) per gram of sorbent. C_e is the equilibrium concentration of solute in solution (mg/l).

$$q_e = \frac{V(C_o - C_e)}{m} \tag{3}$$

Here, V is the volume of solution (L), C_o is the initial concentration of solute in solution (mg/l), and m is the mass of the sorbent (g).The data in Table 2 was plotted using the linear forms of Freundlich (Eq. 2). A slope of 1/n and ln(K) as intercept as presented in Fig. 4.
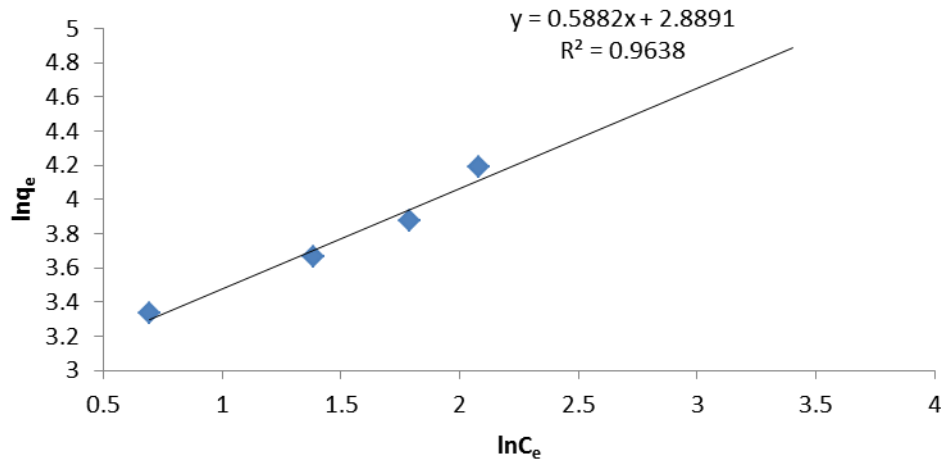

Figure 4: Mango seed kernel Freundlich isotherm model

From the figure, it can be seen that the R^2 correlation coefficient obtained showed an almost linear trend with a value of 0.9638. It can be deduced from this model that, the data generated from this sorbent material fitted the Freundlich isotherm model very well. The evaluated parameters are presented in Table 3.

Table 3: Evaluated isotherm Freundlich parameters

Isotherm model	Correlation coefficient (R^2)	Slope (1/n)	Intercept (lnK)	Degree of non-linearity (n)	Freundlich constant (K)
Freundlich	0.9638	0.5882	2.8891	1.7001	17.977

From the Table 3, it can be concluded from the evaluated value of degree of non-linearity 'n' (n=1.7001) that the oil sorption from produced water using mango seed kernel followed a physical sorption (Freundlich, 1906)process as indicated by the value of 'n' (n falls between 1-10) hence it can easily be recovered for re-use. This indicates that sorption pattern by this sorbent on oil produced water is heterogeneous in nature as it conforms to Freundlich isotherm model.

3.2.2   Langmuir isotherm

Langmuir sorption isotherm models the monolayer coverage of sorption surfaces and assumes that sorption occurs on a structurally homogeneous adsorbent and all the sorption sites are energetically identical. The saturated monolayer curve can be represented by Eq. 4.

$$q_e = \frac{k_L b C_e}{1 + k_L C_e} \tag{4}$$

The linear form of Eq. 4 presented as Eq. 5 can be used to test the data fitness.

$$\frac{C_e}{q_e} = \frac{1}{b k_L} + \frac{1}{b} C_e \tag{5}$$

A plot of C_e/q_e gave a slope 1/b and an intercept $1/bk_L$. The plot will help in determining the constant parameters and is presented in Fig. 5.
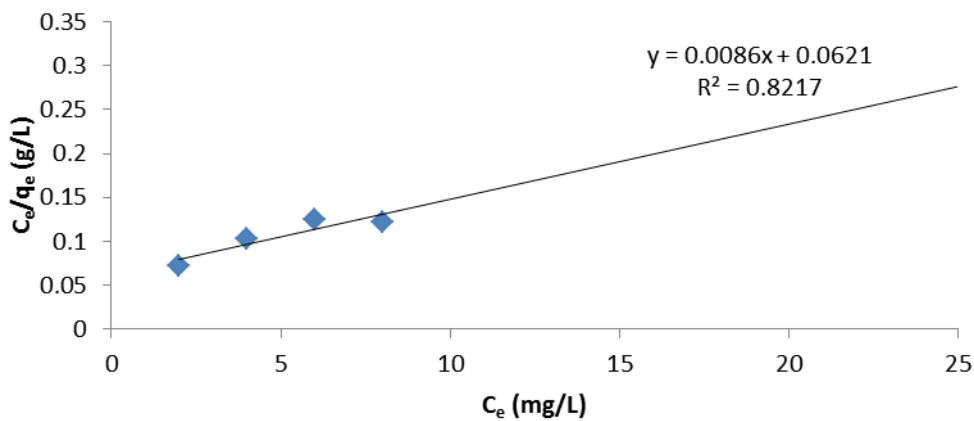
Figure 5: Mango seed kernel Langmuir isotherm model

The essential characteristics of the Langmuir isotherm can be expressed in terms of a dimensionless equilibrium parameter, R_L which is defined by Eq. 6 can be used to determine the sorption type.

$$R_L = \frac{1}{1 + bC_0}$$ (6)

Where, C_o is the highest initial solute concentration, b the Langmuir's adsorption constant (L/mg). The value of R_L indicates the type of the isotherm to be either unfavorable ($R_L > 1$), linear ($R_L = 1$), favorable ($0 < R_L < 1$) or irreversible ($R_L = 0$). The parameters evaluated for the Langmuir isotherm are presented in Table 4.

Table 4: Langmuir isotherm parameters using mango seed kernel

Isotherm model	Correlation coefficient (R^2)	Slope	Intercept	Isotherm constant (k_L)	b	Separation factor (R_L)
Langmuir	0.8212	0.0086	0.0621	116.28	0.138	0.195

The value of separation factor (R_L) showed that it falls between 0 and 1 ($0 < R_L < 1$) an indication that the sorption falls within the favorable region (Langmuir, 1916). The significance of evaluating R_L is to know the category of the sorption by the sorbent. It can be seen that the equilibrium constant is high ($k_L = 116.28$ mg sorbate/g sorbent). This is an indication that the sorbent has high oil sorption capacity.

The derivation of the Temkin-Pycher isotherm assumes that the fall in the heat of sorption is linear rather than logarithmic, as implied in the Freundlich equation. The Temkin isotherm (Eq. 7) has generally been applied in the following form:

$$q_e = \frac{RT}{b_T} \ln(A_T C_e)$$ (7)

Where, R is the universal gas constant (J/mol.K), T is the absolute temperature (K), A_T is Temkin isotherm constant (dm^3/mmol), and b_T is Temkin-Pycher isotherm constant. The linearized form of Temkin-Pycher isotherm is presented in Eq. 8.

$$q_e = \frac{RT}{b_T} \ln A_T + \frac{RT}{b_T} \ln C_e$$ (8)

When the data was plotted in the Temkin-Pycher isotherm model (Eq. 8), the result is presented in Figure 6.
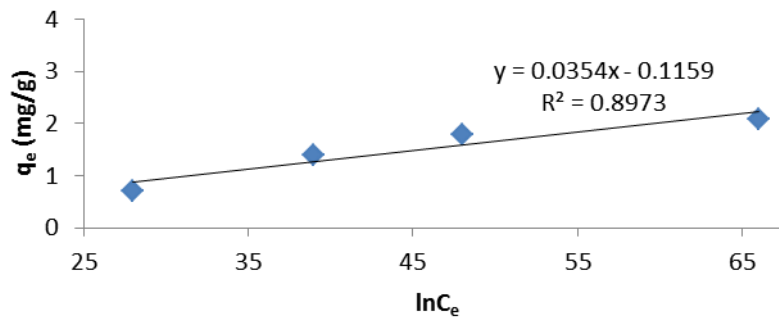
Figure 6: Mango seed kernel Temkin-Pycher isotherm model

From the generated figure, it can be seen that the correlation coefficient is better than that obtained from the Langmuir isotherm model. It can be said to be better fitted using the Temkin than the Langmuir isotherm. The parameters evaluated for the Temkin-Pycher isotherm data fit is presented in Table 5.

Table 5: Evaluated Temkin-Pycher isotherm for mango seed kernel

Isotherm	Correlation coefficient (R^2)	Slope	Intercept	Isotherm constants	
				b_T	k_T
Temkin-Pycher	0.8973	0.0354	-0.1159	0.9999	0.0114

Radushkevich (1949) and Dubinin (1965) have reported that the characteristic sorption curve is related to the porous structure of the sorbent. This isotherm is generally expressed as given in Eq. 9 (Dubinin, 1960).

$$q_e = q_D \exp\left(-B_D \left[RT \ln\left(1 + \frac{1}{C_e}\right) \right]^2 \right) \tag{9}$$

The constant, B_D, is related to the mean free energy of sorption per mole of the sorbate (E) as it is transferred to the surface of the solid from infinite distance in the solution and this energy can be computed using Eq. 10 (Hasany and Chaudhary, 1996):

$$E = \frac{1}{\sqrt{2B_D}} \tag{10}$$

The generated data was fitted into the linear form of Dubinin-Radushkevich isotherm model (Eq. 11).

$$\ln q_e = -B_D R^2 T^2 \left[\ln\left(1 + \frac{1}{C_e}\right) \right]^2 + \ln q_D \tag{11}$$

The result is presented in Figure 7. From the plot, it can be seen to be the least fitted isotherm with the generated data. This is because it has the least correlation coefficient (R^2=0.8142).
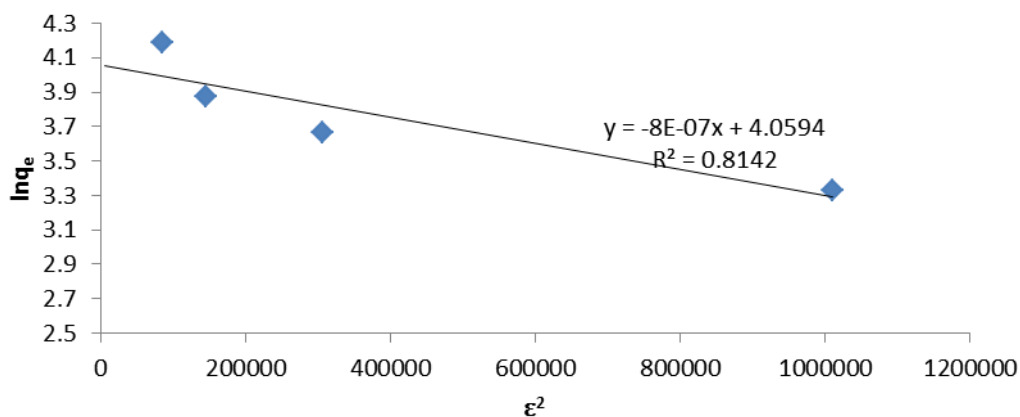

Figure 7: Mango seed kernel Dubinin-Radushkevich isotherm model

The constant parameters from the model were evaluated and the results are presented in Table 6.

Table 6: Evaluated constant parameters obtained from Dubinin-Radushkevich model

Isotherm	Correlation coefficient (R^2)	Slope	Intercept	β	Free energy (E) J/mol
Dubinin-Radushkevich	0.8142	--8E-07	4.0594	8E-07	790.57

From the free energy value (E), it can be concluded that oil sorption from produced water using mango seed kernel according to Dubinin-Radushkevich model followed a physical process since its value is less than 8 kJ/mol(Dubinin, 1960 and Dubinin, 1965).

4.0 Conclusion

Mango seed kernel was used as oil sorbent from produced water and it has successfully removed 93.33% oil initially present in produced water from 30 ppm to 2 ppm. Thus, mango seed kernel has proven to be very oil sorbent in the reduction of oil content from produced water. The data generated from the use of sorbent material was tried in fitting four different isotherms developed by Freundlich, Langmuir, Temkin-Pycher, and Dubinin-Radushkevich isotherms. It was deduced that, the data fitted better in the Freundlich isotherm model with heterogeneous sorption (Freundlich, 1906)which was concluded to be physical sorption process from the degree of non-linearity 'n' greater than 1. The dimensionless separation factor (R_L) evaluated from the Langmuir isotherm model showed that the sorption process is favorable (Langmuir, 1916). The free energy of sorption evaluated from the Dubinin-Radushkevich isotherm also gave a similar inference with that of the Freundlich isotherm, i.e. physical sorption process.

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