

Kinetics and Isotherms of Oil Spills Removal by Novel Bio-Adsorbent (Panseke) developed by a Natural Technique

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ABSTRACT

Kinetic and isothermal studies for the oil spills removal process have been conducted by Panseke powder prepared locally from a forest tree and evaluated successfully in the removal of heavy crude oil spills. The sorption process of crude oil onto panseke was studied using Langmuir adsorption models while the sorption kinetics was studied using Langmuir pseudo-first order, pseudo-second order. Suitable environmental applicability of both sorbents was also studied. The equilibrium study as it relates to the Langmuir isotherm model which is a mathematical model used to determine the rate of an adsorption reaction as well as the adsorption capacity of the adsorbent used in remediation was also studied. The adsorbent Panseke was used for investigation of soluble crude oil adsorbency and other properties such as: moisture content, buoyancy, water adsorbency and oil/water adsorbency ratio. In addition, relation between soluble crude oil adsorbency and the three factors of time of adsorbency, concentration of spill and dosage of the adsorbent were determined. The result values of moisture content of 4.84%, buoyancy of range: 8 – 17 %, water adsorbency of range 1.49 – 4.26 % and Langmuir values of $R_L = 0.0213$, $q_m = 1.527$ mg/g and $K_L = 0.0632$ L/mg; pseudo-first order rate constant of 0.111 min⁻¹ and pseudo-second order rate constant of 0.4329 mg⁻¹ min⁻¹ indicates that panseke is a better sorbent for oil removal. Panseke has the ability to retain sorbed oil which makes it a good sorbent when oil recovery is not required, but disposal. The sorption process for both sorbents follows the Langmuir isotherm.

Keywords: Panseke, Kinetics, Isotherms, Bio-adsorbent, Natural Technique, crude oil Spillage.

I. INTRODUCTION

Water is a very important constituent of the ecosystem on the Earth and essential component of life. The demand of water is increasing day by day due to an increase in population and in living standard. The quality of our water resources is deteriorating day by day due to the continuous addition of undesirable chemicals in them. Various water pollutants, such as metal ions, the residues of the drugs and pharmaceuticals (new emerging pollutants), dyes, Chiral pollutants and pesticides in water, are hazardous. Since these pollutants are very dangerous and have a direct and indirect impact on the human health life and the surrounding environment, many reviews and studies about have been published (Basheer, 2018). The different methods have been developed and used for water treatment. These methods include filtration, screening, oxidation, precipitation, coagulation, centrifugation, flotation, crystallization, sedimentation, distillation, evaporation, reverse osmosis, electrochemical, ion exchange, adsorption, etc. Among them, adsorption phenomenon is considered as one of the appropriate water treatment methodologies because of its ease of operation and the accessibility with a wide range of adsorbents for the removal of different pollutants (Basheer, 2018). When oil is explored, transported and stored and its derivatives are used, there is risk for spillage with the potential to cause significant environmental impact (Hussein et al., 2008). Due to its destructive properties, the entire character of the area is damaged once an area has been contaminated with oil. In addition, when oil encounters something to cling to (e.g., beach, rocks, feathers of a duck or a bather's hair), it is difficult to remove (Nwankwere, 2010). Therefore, pollution by petroleum oils affects sea life,

economy, tourism and leisure activities due to the coating properties of these materials (Hussein et al., 2008). When oil is spilled in water or on land, the physical and chemical properties of oil change progressively. The spilled oil contributes an undesirable taste and odor to drinking water and causes severe environmental damage (Nwadiogbu, et al., 2016). Oil pollution from food production, catering industry, petroleum chemical and petroleum mining has drawn extensive attention of many researchers in recent years. Many methods, such as mechanical extraction, in situ combustion and chemical degradation, have been used to the cleanup of oil from polluted areas. Owing to better economic and environmental benefits, the use of sorbent is considered as an effective method to concentrate, transfer and absorb spilled oil (Adebajo, et al., 2003). High-efficient oil sorbent is required to possess desirable characteristics, such as excellent hydrophobicity and oleophilicity, high uptake capacity and fast oil sorption rate, low water uptake and insolubility, low cost and high buoyancy. This study tends to use natural materials; panseke as adsorbent for soluble crude oil with our personal concentration on panseke.

II. EXPERIMENTAL

Materials

Collection of the adsorbents, Panseke (the flamboyant tree: *delonix regia*) was obtained from a forest in Lapai, Niger state, Nigeria. Crude oil was obtained from Shell Petroleum Development Company, Warri, Delta state, Nigeria. Equipment's used for the analysis are digital weighting scale, measuring cylinder, beaker, conical flask, magnetic stirrer stirring rod, dropper, stop watch, sample bottles, sieve mesh, shaker table, mortar and pestle, grinding stone, oven, filter paper and crucible were all obtained in chemistry and physics laboratory of the university of Abuja, Nigeria.

2.1 Methods

Preparation of Sample

In this study, the natural material is prepared to eliminate or minimize the effect of external factors that can cause a deviation from accurate readings. These factors are impurity, size effect and change of contact angle. The stems are manually harvested (the flamboyant tree: *delonix regia*). And then its impurities are removed by washing thoroughly with clean water. It is then chopped into approx 1 cm of length then sundried for days until weight remains constant. It is then stored in plastic bag to prevent moisture from air. The dried piece is then grinded into powder from by a mortar or grinding stone and then it is sieved

to collect only the finest of its powder. This powder is then stored in air tight containers to prevent moisture from air and will be used as adsorbent for soluble crude oil.

Determination of Moisture Content

In this study the tested material is exposed to room's temperature for 24 hours before investigating their moisture content and other characteristics. The procedures of determining moisture content are described below.

Place 4 g of the powdered panseke 24 hours exposed adsorbents in the crucible (the resolution must be + 0.01 g) and define it as W . Oven dry it at 103°C until its weight remains constant and totally transfer the dried adsorbents from crucible. Record the sorbent weight and denote it as w_d . Calculate the moisture content in adsorbents by using equation (1);

$$\text{Moisture Content} = \frac{W - w_d}{w_d} \times 100 \quad (1)$$

Buoyancy test

In the situation where adsorbents are used for combating oil spill incident recovering (or harvesting) of soaked materials is an important step to reduce their impacts on aquatic resources. Though, it is possible that some adsorbents may not be collected during the recovering process due to sinking. This effect can be expressed in terms of "Buoyancy" (Wang, 2014). However, in the case of soluble oil remediation the material has to pass through the entire depth of the water body for remediation to be effective. And since this adsorbent is biodegradable, there will be no deleterious impact to water body, or its aquatic life (Okoro and Ejike, 2007).

Determination of Water Adsorbency

Water adsorbency of materials is determined after the materials have been exposed in a room's temperature for 24 hours (Paulauskiene et al., 2014). The procedures for determination of water adsorbency are shown below.

Weigh 1 g of 24 hours exposed adsorbent (powdered panseke) and define it as W_o and transfer it to 10ml water filled test cell. Place the test cell cover on its opening and place it on a shaker table, then set its frequency to 200 rev/min. Shake for 5 min adsorption period (during the process investigate its appearance and changes on both adsorbent and water) and let it be settled for 2 minutes. Drain the content for 30 seconds and Weigh the drained sample. Record the drained

sample as $w_{drained}$ and calculate the water absorbency by using equation (2).

$$\text{Water absorbency} = \frac{w_{drained} - w_o}{w_o} \quad (2)$$

Soluble oil absorbency

Soluble oil adsorbency is the amount of soluble oil adsorbed on 1 g of adsorbent. Materials used to be oil spill adsorbent should have oil adsorbency more than 5 g oil / g adsorbent. In this study, adsorbate or oil to be investigated is crude oil. The procedures for the determination of soluble oil adsorbency are listed below:

- i. Standard solution of crude oil in water
- ii. Adsorption capacity in variation with time
- iii. Adsorption capacity in variation with concentration
- iv. Adsorption capacity in variation with dosage

2.2 Regeneration of Adsorbents

Regeneration being the process of renewal or restoration is an important process in the use of an adsorbent. It entails that an adsorbent can be reused after remediation. However, in the case of remediating soluble crude oil, where the adsorbent has to go through the entire depth of the water body, recovery of adsorbents is more or less an impossible feat (Nduka, 2012).

Nevertheless, in a case where adsorbents can be recovered, a regeneration process is

required. Hence, here is a set of procedures for the regeneration of adsorbents.

200ml of water is measured into a beaker and 1g of crude oil is added to the beaker. The solution is stirred for 30mins at a temperature of 40°C and the solution is filtered into a conical flask. 10ml of filtrate is measured into a shaking bottle, 0.1g of adsorbent was added to the filtrate and was then placed on a shaking table for 50mins at a shaking frequency of 200rpm. The sample was filtered into a bottle with the residue of adsorbent left on the filter paper. The paper containing residue was washed into beaker with 10ml of water and allowed to settle. It was then decanted into a sample bottle and labeled "1" cycle. 10ml of water was added again to the residue and allowed to settle then decanted again and was labeled "2" cycle. The process was repeated up to the 5th cycle and the residue was then placed in an oven to dry until the weight became constant.

III. RESULT AND DISCUSSION

Moisture Content

After careful experimental test using the detailed steps mentioned in materials and methods above over a range of 15 samples and using the statistical analysis presented below, it is concluded panseke has a moisture content of 4.84% moisture with a standard deviation of 0.11.

Table 1: moisture content of adsorbent

Adsorbent	Mean	N	Std	Minimum	Maximum
Panseke	4.83732	15	11084	4.623	4.968

Buoyancy

Buoyancy of adsorbent is investigated by studying its variation in time of adsorption. Just as stated earlier, the buoyancy of an adsorbent to be used for remediation of crude oil spill should be about 90% to be considered effective (not more

than 10% of adsorption should sink) however, in the case of remediating soluble crude oil, the adsorbent is required to pass through the depth of the entire water body, therefore a buoyancy of 10% or less would be considered effective (about 90% of adsorbent will go through the water body).

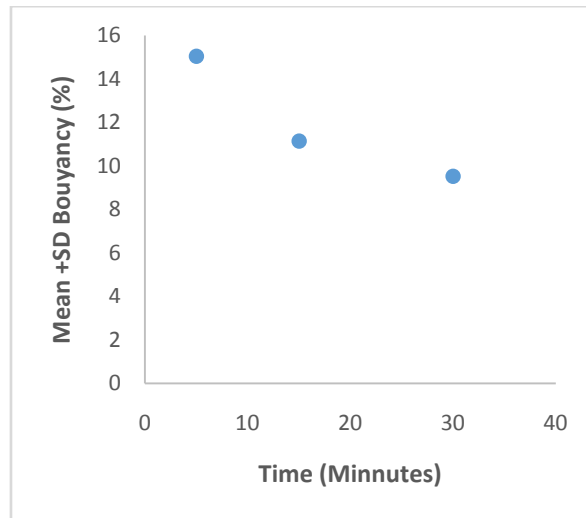


Figure 1: Buoyancy of Panseke with Time of adsorption

Buoyant of panseke varies in range of 8% to 17% at 0mins (upon introduction of adsorbents) it is already at 80%. At 5mins it is at 15.04% with a standard deviation of 2.001. At 15mins buoyancy is 11.14% with a standard deviation of 0.68 and lastly at 30mins buoyancy is 9.52% with a standard deviation of 1.63.

Water Absorbency

Amount of water (g) absorbed into 1g of absorbent “water absorbency” of the materials was investigated. The schematic plot between water absorbency and time of absorption of the absorbent is shown in the figure 2.

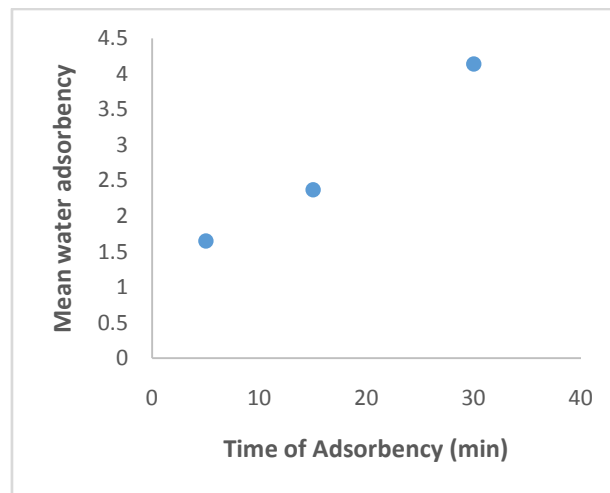


Figure 2: Water adsorbency of Panseke with time of adsorption

Water absorbency of panseke at time interval of (5 min., 15min, and 30min.) varies in range of 1.49 to 4.26. At 5 minutes, an average of water absorbency is 1.65 with standard deviation of 0.11. At 15 minutes, the average of water absorbency is 2.37 with standard deviation of 0.08. Lastly, at the time of absorption of 30 minutes, the average of water absorbency is 4.114 with standard deviation of 0.08.

Soluble oil absorbency

From the experimental procedure stated in materials and methods, soluble oil absorbency was carried out as absorption capacity was varied over three factors namely time, dosage and concentration. After spectroscopic analysis of samples and reviewing, results were obtained, the following were observed:

- **Absorption capacity in variation with concentration**

In the case of varying absorption capacity with concentration of crude oil, samples were created using a spill of 0.15g, up to 0.75g of crude oil and 0.1g of absorbent was used in remediation of 10ml of each sample.

The effect of initial spill concentration in the range of 0.05 to 0.75g on absorption (investigated under the specified conditions; contact time of 30 min; absorbent dosage of 0.1g; and temperature of 40°C) is shown below. The amount of adsorbate (soluble oil concentration) relating to the lower initial spill concentration of crude oil was just about the same with the amount when higher concentration was used. It is seen that the removal of soluble oil was not dependent on the concentration of the spill but on the percentage solubility in water of the type of crude oil as the decrease in the initial concentration had no effect in the amount of soluble oil removed, this is in line with the results of (Igwe and Abia, 2006). While the amount of spill concentration was varied, the soluble oil concentration remained the same at $30 \pm 0.02 \text{ mg/L}$.

• **Absorption capacity in variation with time**

After absorbents were introduced, the samples were placed on a shaker 5mins, 10mins and 20mins up to 100mins for different samples. The spectroscopic consult shows meaningful absorption and gradual change in sample properties up until 10mins.

Absorption of soluble crude oil was measured at given contact time for a constant spill concentration of 1g of crude oil to 200ml of water (10000mg/L), and 5g of absorbent to 10ml of its filtrate (500mg). From figure 3, the plot reveals that the rate of percent soluble oil removal is higher at the beginning. This is probably due to larger surface area of the absorbent powder being available at the beginning for the absorption. Most of the maximum percent soluble oil removal was attained after about 40 min of shaking time. The increasing contact time increased the soluble oil absorption and it remains constant after equilibrium is reached in 30 min. This depicts that it takes about 30mins for absorption of soluble crude oil by panseke to take place after which the absorbent becomes saturated if all of the soluble crude oil hasn't been absorbed.

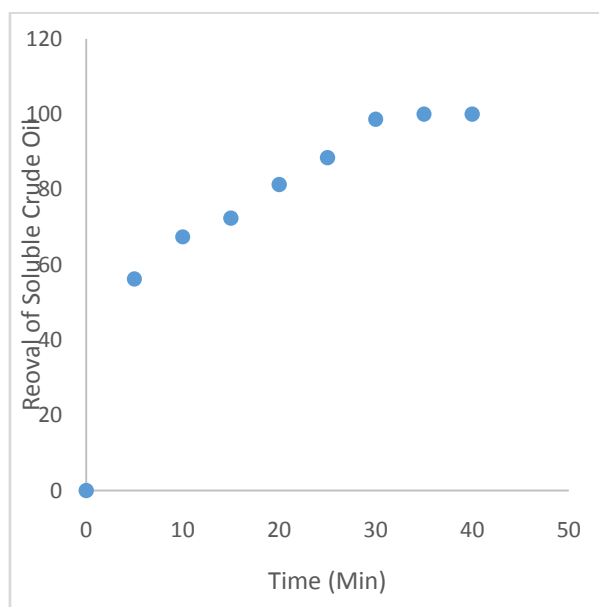


Figure 3: effect of contact Time on Adsorption

• **Absorption capacity in variation with dosage**

Spectroscopic result analysis shows that the absorption of soluble crude oil increases rapidly with increase in the amount of panseke powder due to greater availability of the surface area at higher concentration of the absorbent ascertained elsewhere (Dawodu and Akpomie, 2014). The significant increase in uptake was observed when

the dose was increased from 0.3 to 0.6g. any further addition of the absorbent beyond this did not cause any significant change in the absorption. This may be due to overlapping of absorption sites as a result of overcrowding of absorbent particles. From the results, it is revealed that within a certain range of initial soluble oil concentration, the percentage of soluble oil absorption on 0.3g of

panseke powder is determined by the sorption capacity of the absorbent. The maximum removal of soluble oil was obtained in the absorbent dose of

0.6g. It is also observed that with increased dose of absorbent, water was found to be more reddish.

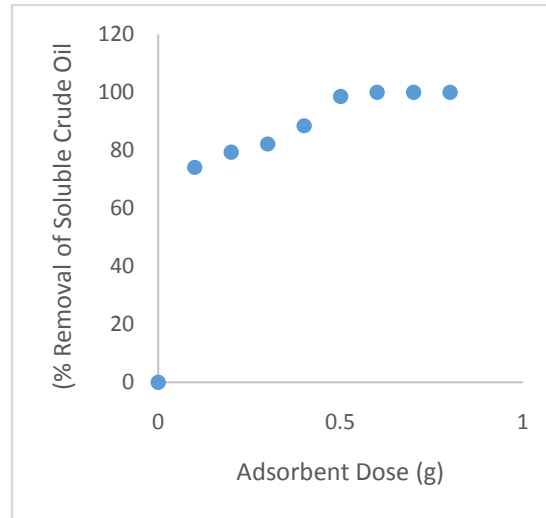


Figure 4: Effect of Adsorbent Dose

3.1 Regeneration of absorbents

From the experimental procedures in 3.3.6, concerning regeneration of absorbents, after the regeneration process, the second hand absorbents were found to be relatively lighter than the first hand ones, furthermore; the particles appear to be looser than the first hand absorbents. It also appears to have lost a lot of its coloration and has increased water absorbency soluble oil absorbency.

3.2 Equilibrium study

Having looked at the absorption process as it relates to different time frame, different

concentration of soluble oil, and different doses of absorbent; lets now review the equilibrium study of adsorption itself. Adsorption isotherms are mathematical models that describe the distribution of the adsorbate species among liquid and absorbent, based on a set of assumptions that are mainly related to the heterogeneity/homogeneity of absorbents, the type of coverage and possibility of interaction by the adsorbate species (Dong, Xu and Wang, 2015). Adsorption data are by describe by adsorption isotherms, such as Langmuir, Freundlich and Temkin isotherms. These isotherms relate soluble oil uptake per unit mass of absorbent, q_e to the equilibrium adsorbate concentration C_e .

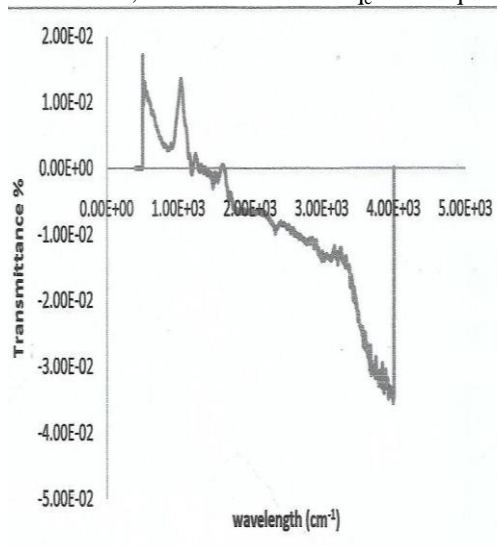


Figure 5: Spectrogram of adsorbent

Absorption isotherm is the relation in a constant temperature between partial pressure of adsorbate and amount of absorbed substance at its equilibrium condition. In the experiment, adsorbate is the soluble oil in liquid state. Thus, concentration of soluble oil, and amount of absorbed substance at equilibrium condition.

3.3 The Langmuir Isotherm

The Langmuir model is based on the assumption that the maximum absorption occurs when a saturated monolayer of solute molecules is present on the absorbent surface, the energy of absorption is constant and there is no migration of adsorbate molecules in the surface plane. The Langmuir isotherm is given by:

$$q_e = \frac{q_m k_l c_e}{1 + k_l c_e} \quad (3)$$

The constants in the Langmuir isotherm can be determined by plotting $(\frac{1}{q_e})$ versus $(\frac{1}{c_e})$ and making use of above equation rewritten as:

$$\frac{1}{q_e} = \frac{1}{q_m} + \frac{1}{q_m k_l} \cdot \frac{1}{c_e} \quad (4)$$

Where q_m and K_l are the Langmuir constants, representing the maximum absorption capacity for the solid phase loading and the energy constant related to the heat of absorption respectively. However, for easier representation, the linearized Langmuir can be used

$$\frac{c_e}{q_e} = \frac{1}{q_m k_l} + \frac{1}{q_m} c_e \quad (5)$$

Where C_e is the equilibrium concentration of the sorbate (mg/L), q_e the amount of sorbate per unit mass of bio-sorbent (mg/g), K_l is a constant representing the strength with which the solute is bound to the substrate (L/mg) and q_m is the absorption capacity of the substrate (gram solute / gram absorbent).

When $\frac{c_e}{q_e}$ was plotted against c_e a straight line slope

$\frac{1}{q_m}$ will be obtained and an intercept $\frac{1}{q_m k_l}$ while q_e is expressed as given below

$$q_e = \frac{v(c_i - c_e)}{m} \quad (6)$$

where v is the bio-sorption volume in liters, c_i is the initial concentration of the sorbate (mg/L) and m is the mass of the bio-absorbent (g)

The essential characteristics of the Langmuir isotherm can be expressed in terms of a dimensionless equilibrium parameter R_L which is defined by

$$R_L = \frac{1}{1 + q_m c_o} \quad (7)$$

Where c_o is the highest initial solute concentration, q_m the Langmuir absorption constant (L/mg). The value of R_L indicates the type of isotherm to be either unfavorable ($R_L > 1$), linear ($R_L = 1$), favorable ($0 < R_L < 1$) or irreversible ($R_L = 0$).

It can be seen from the figure 6, that the isotherm data fits the Langmuir equation well ($R^2 = 0.969$). The values of R_L , q_m and K_l were determined from the figure and were found to be 0.0213, 1.527mg/g and 0.0632L/mg, respectively.

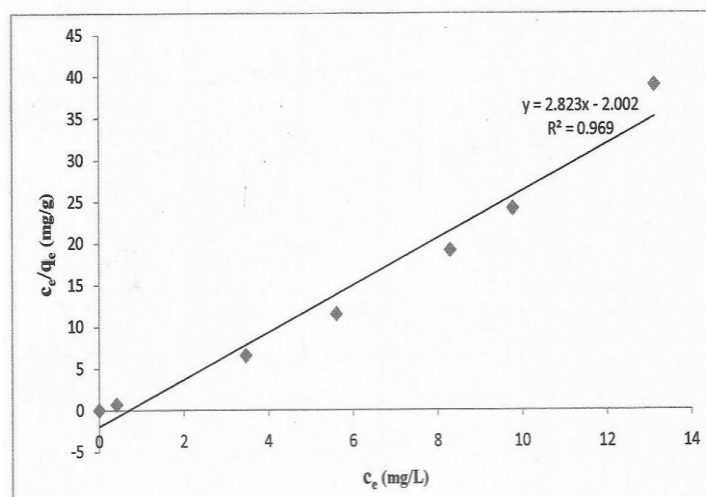


Figure 6: Langmuir adsorption isotherm of panseke

3.4 Kinetic Study

In order to investigate the controlling mechanisms of adsorption processes such as mass transfer and chemical reaction, the pseudo-first order and pseudo-second order equations are applied to model the kinetics of soluble oil adsorption onto panseke powder. The pseudo-first order rate equation is given as

$$\text{Log} (q_e - q_t) = \text{log} q_e - \frac{K_{ad}}{2.303} t \quad (8)$$

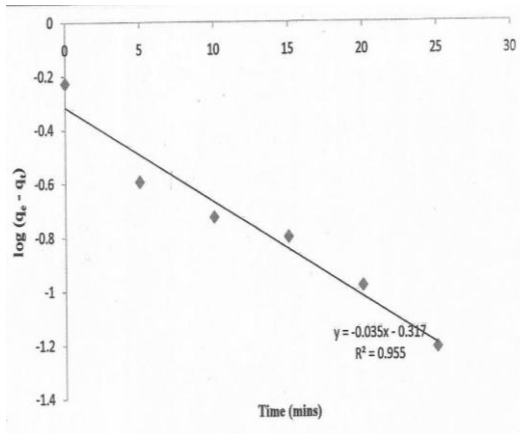


Figure 7: Pseudo-first order reaction for soluble crude oil adsorbed onto panseke powder at concentration of 30(g/L)

where $h = k q_e^2$ ($\text{mg g}^{-1} \text{min}^{-1}$) can be regarded as the initial absorption rate as $t \rightarrow 0$ and k is the rate constant of pseudo-second order absorption ($\text{g mg}^{-1} \text{min}^{-1}$).

The plot $\frac{t}{q_t}$ versus t should give a straight

line if pseudo-second order kinetics is applicable

Where q_t and q_e are the amount adsorbed (mg/g) at time, t , and at equilibrium respectively and K_{ad} is the rate constant of the pseudo-first order adsorption process (min^{-1}). Straight line plots of $\text{Log} (q_e - q_t)$ against time were used to determine the rate constant, K_{ad} and correlation coefficients, R^2 for the soluble oil concentration, as shown in figure 7.

and q_e , k and h can be determined from the slope and intercept of the plot, respectively. The plot of the linearized from the pseudo-second order reaction at concentration of 30(mg/L) by panseke powder is shown in figure 8.

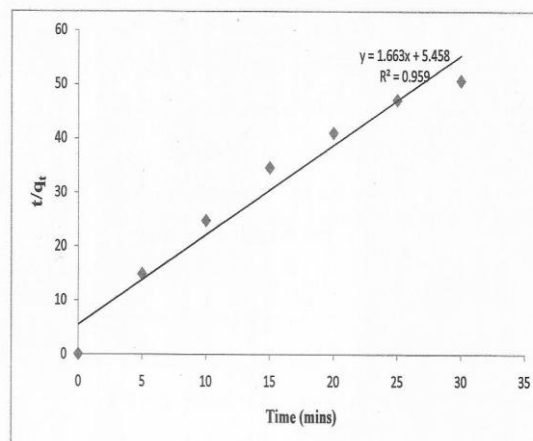


Figure 8: Pseudo-second order reaction for soluble crude oil adsorbed onto panseke powder at concentration of 30(g/L)

The plot of $\frac{t}{q_t}$ versus t for pseudo-second

order model yields a very good straight line with correlation coefficient, ($R^2 = 0.959$) as compared to the plot of pseudo-first order. The pseudo-second order rate constant was noted to be range of $0.4329 \text{ g mg}^{-1} \text{ min}^{-1}$. The theoretical values of q_e also agree very well with the experimental ones. Both facts suggest that the adsorption of soluble crude oil by panseke powder follows the pseudo-second order kinetic model, which relies on the assumption that physio-sorption may be the rate-limiting step. In physio-sorption. (physical absorption), the particles attach to the adsorbent surface by forming a chemical (usually covalent) bond and tend to find sites that maximize their coordination number with the surface.

The pseudo-second order kinetic analysis reveals that the value of the initial absorption rate, h , should increase with increase in the initial soluble oil concentration. The lower the concentration of soluble oil in the solution, the lower the probability of collision between the particle of soluble oil, and particle of adsorbent species is and hence the faster soluble oil could be bounded to the active site on the surface of the adsorbent. The equilibrium adsorption capacity q_e however will increase in initial soluble oil concentration due to large number of soluble oil particles adsorbed at the available absorption site. Based on the values of R^2 obtained from the plots of pseudo-first order and pseudo-second-order rate equations, it is obvious that increasing initial concentration of the adsorbate, the correlation of experimental data to the pseudo-second order kinetics model increase while that to the of pseudo-first order model decreases. The findings from this study are in complete agreement kinetics model developed by (Azizian 2004).

IV. CONCLUSION

Panseke has lower oil sorption capacity and sorbed oil recoverability than the standard (synthetic sorbent mat). However, Panseke has the ability to retain sorbed oil which makes it a good sorbent when oil recovery is not required. The sorption of crude oil onto Panseke and the standard follows Langmuir adsorption model, and pseudo first and second order kinetics. The equilibrium study shows that panseke powder is an effective adsorbent for the removal of soluble oil from water upon oil spillage. From the kinetic studies it is observed that adsorption of soluble oil is very rapid in the initial stage and decreases while approaching

equilibrium. The study on the kinetic also shows that the equilibrium time will increase with increase in initial soluble oil concentration. The percentage removal of soluble oil increase with the increase in adsorbent dosage and decreases with increase in initial adsorbate concentration. Experimental results are in good agreement with Langmuir absorption isotherm model, and have shown a good fitting on the experimental data. Adsorption of soluble oil obeys pseudo-second order equation with good correlation.

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