Surface Modified Malaysian Ceiba Pentandra (L.) Gaertn. As Natural Oil Adsorbent.

Ahmad Zakwan Bin Mat Noh, Dr. Putri Nadzrul Faizura Binti Megat Khamaruddin

Faculty of Chemical Engineering, Universiti Teknologi Mara

***Abstract* — The risk of oil spills escalate as the petroleum industry growing and the toxic hydrocarbons released to the environment are harmful to both animals and living things. Producing a cost-efficient and environmental-friendly sorbent are fundamentals. Sorbent must not leave traces of harmful chemicals which may later on affect the nature. Adsorption technique by using kapok fiber has been proven to be the most promising method compared to the other oil-containment approaches. Kapok have an excellent hydrophobic-oleophilic structure. In this study, four-hour esterification of fatty acids (oleic acid and decanoic acid) at 50˚C±5˚C will modify the surface characteristics of the kapok fiber in order to identify the effects on the oil sorption and dynamic oil retention capacity. The oil treatment protocol follows the standard F726-99 (ASTM, 1998c). This research focuses on analyzing the sorption capacity of raw kapok and its modified versions in accordance to the varied oil viscosity (diesel, engine oil, and light crude oil). In addition, the experimental results are compared with adsorption isotherms models which are Langmuir and Freundlich to identify the sorption behavior of the adsorbent. The functional groups of kapok can be retrieved from performing Fourier-Transform Infrared (FTIR). Raw, oleic grafted, and decanoic grafted kapok show high sorption intake for crude oil; 66.50g/g, 73.22g/g, and 79.27g/g respectively. Raw kapok shows highest sorption for diesel (50.41g/g) and engine oil (60.32g/g). However, the modified versions of kapok show better dynamic retention capacity compared to the raw kapok. Malaysian kapok behaves similarly as the Langmuir isotherm model.**

I. INTRODUCTION

Growing oil industry increases risk of oil spills. The possibilities of oil spill can occur during producing, transporting, as well as refining processes [1,2]. Weak wells or pipeline integrity can elevate the likelihood of leakage of oil producing oils. Thus, precautions need to be taken before the accident occurs. There are numerous existing oil containment method which has been proven to be efficient. However, several environmental concerns arise when selecting the most desired oil recovering method. In the event of oil spills, immediate action must be taken in order to prevent the hydrocarbons from devastating the environment and habitat. Oil spills recovering method by using natural sorbents caught many attentions because it is reliable in terms of cost, ample in nature, bio-degradable, and high absorptivity whereas synthetic sorbents such as polypropylene can be harmful to the environment [3]. In addition, sorption technique is a proven efficient method to contain the oil spreads on the seawater [4,5]. In the era of growing and advanced technologies, engineers and scientists ought to find the best improvised natural sorbents.

Protecting the environment is the main duty and must be put to attention when it comes to the selection of oil sorbents because we need to reduce any additional risks from worsening the incident. Natural sorbents are a good option and it comprises in various dimensions, origin, and sorption capability. Studies have shown that kapok fiber is the most suitable organic sorbent compared to the others due its unique surface characteristics' [5]. Kapok fiber is a yellowish cellulosic-lignin fiber and can be found as the fruit pod of *Ceiba pentandra* which known as kapok trees. Kapok fiber is light-weighted with good water repellent and was previously used as life-jackets filling. Kapok fiber has properties such as the huge hollow lumen, high buoyancy, and hydrophobic-oleophilic structure [3,6]. These special characteristics made kapok fiber as an excellent candidate towards removing spilled oil.

To cleanup a large body of oil spilled on seawater, cost-saving and efficient sorbents must be carefully selected to avoid unwanted wastes in terms of money as well as toxicity. In order of that, kapok fibers can be used because it is highly capable of absorbing oil instead of water due to its high hydrophobicity and waxy walls. Although, the main contribution to the sorption process is the consequence of big lumen of the kapok [7]. Nonetheless, the sorption and retention capacity of this organic sorbent can be improved to maximize the efficiency. The challenges arise when producing kapok fibers as a sorbent at industrial level because of the fluffy surface and poor interfacial adhesion [8].

In this study, oil absorbency effect when using raw and fatty acids (oleic acid and decanoic acid) grafted kapok fibers is analysed based on the oil sorption treatment with variable viscosity. The chemical compositions of the fibers are studied from the spectra of Fourier Transform Infrared (FTIR). The dynamic oil retention capacity of each kapok was evaluated. In addition, Analysis of Variance (ANOVA) and reproducibility elements are calculated to verify the results. The postulation of the adsorption isotherms between the raw kapok and the modified versions are identified based on the sorption behaviour.

II. METHODOLOGY

A. Materials

Malaysian kapok fibers used in this study were retrieved at the Ulu Dong Village, Raub, Pahang. Any debris found within the fiber was removed and stored away from moisture.

Decanoic acid, oleic acid, n-hexane, and concentrated sulphuric acid are analytically pure graded. The Bertam crude (light crude oil) is provided by the faculty, diesel was purchased at the Petronas pump station and engine oil (partly synthetic Castrol 10W-40) are used during the oil treatment processes. For the adsorption equilibrium test, Jabal seawater originated from Oman is used.

B. Kapok fiber and experimental oils characterization

The spectrum of kapok fiber is identified by using Spectrum One FT-IR Spectrometer (PerkinElmer, USA). The sample reader of the FTIR is cleansed by using acetone to remove any debris that may cause incorrect reading of functional groups. The sample is placed on the built-in diamond surface carefully and pressed by pressure gauge. The mechanical pressure gauge should be in the range of 60psi to 70psi. The wavelength is set to be from 500cm^{-1} until 4000cm^{-1}.

The viscosity of the experimental oils was measured by using Fann Viscometer 35SA which has three different speeds; 100rpm, 300rpm, and 600rpm. The measurement is triplicated to obtain an average result.

C. *Esterification of kapok fiber*

Kapok fiber is weighed at 1.0g and placed in the round bottom flask containing 0.2g of fatty acid (decanoic acid or oleic acid) mixed with 100ml of n-hexane and one drop of concentrated sulphuric acid, H_2SO_4 as a catalyst. The mixture is refluxed, stirred using condenser and a magnetic stirrer respectively at monitored temperature of 50˚C±5˚C for 4 hours at 200rpm. The obtained sample is washed using n-hexane repeatedly and dried in an oven at 45˚C for 24 hours. A final sample will be stored for later treatment with oils.

D. *Oil sorption capacity (OSC) and dynamic oil retention*

The method used are derived from the Sorbent Test Program 1999-2000 performed by Science Applications International Corporation (SAIC) Canada. This method computes after the existence of the revised method of F726-99 (ASTM, 1998c). The similarity between these two methods are known.

From the earlier kapok sample preparations, 0.1g of dried sample is placed in a round stainless-steel mesh loosely. The test cell is fully soaked inside an oil bath at a specific time according to the oil types until the adsorption reached equilibrium. A 500ml beaker is filled with 400ml oil (diesel, crude oil, or engine oil). The samples are left in the experimental oil bath (30 minutes in diesel, 60 minutes in crude oil, and 90 minutes in engine oil).

The oil-adsorbed test cells were lifted above the oil baths and left dripping for 30 minutes. For each minute of drip, the weight of oil bath is recorded and tabulated. Each oil sorption treatment was repeated for two times. The oil sorption capacity can be calculated by using Equation 1. The equation is used to calculate oil sorption capacity at every minute for 30 minutes. Then, the percentage of oil retention capacity is calculated using Equation 2.

Equation 1

$$OSC = \frac{W_i - W_t}{W_o}$$

Where W_i is the weight of the oil bath before the sorption treatment (g), W_t is the weight of oil bath at 1 min dripping (g), and W_o is the weight of dried kapok (g).

Equation 2

$$Oil\ retention\ capacity\ (\%) = \frac{W_{t=i}}{W_{t=1}} \times 100\%$$

Where $W_{t=i}$ is the weight of the oil sorption capacity (g/g) at t min dripping, i =2, 3, 4, …, and $W_{t=1}$ is the weight of the oil sorption capacity (g/g) at 1 min dripping.

E. *Oil adsorption equilibrium test*

Dried raw sample of kapok fiber weighed 0.1g is placed inside a test cell loosely. The test cell is fully immersed into a beaker which initially filled with Jabal seawater (100ml, 120ml, 140ml, 160ml, and 180ml) mixed with crude oil (100g, 80g, 60g, 40g, and 20g). The beaker with a mixture of oil and seawater is weighed and recorded (without test cell). The beaker is placed inside a horizontal shaker for 24 hours at a constant 100rpm and 30°C.

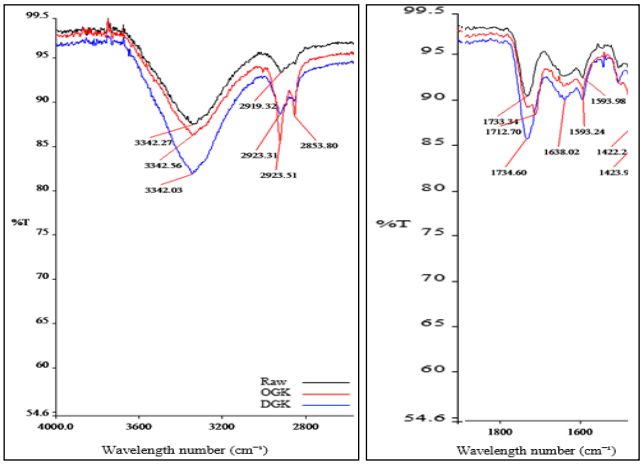
The retrieved test cell is left dripping for another 30 minutes to remove any adhered oil and seawater on the mesh surfaces. The beaker is weighed again to record and calculate the amount of adsorbed oil by the adsorbent.

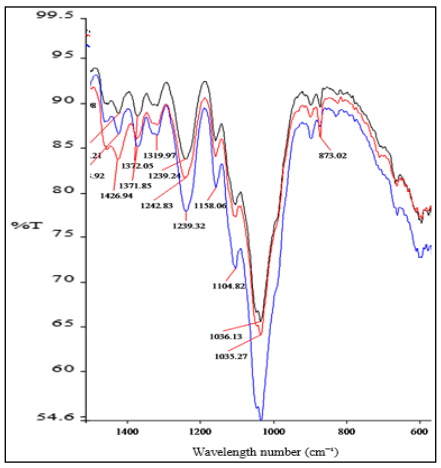
III. RESULTS AND DISCUSSION

A. *Characterization of kapok fiber by Fourier Transform Infrared (FTIR)*

Fourier Transform Infrared spectrometer used to identify the functional group presence within the sample of kapok fiber. This characterization of specific functional groups existence aid to determine the reactive elements of the adsorbent and relate with the sorption capacity outcomes. The FTIR spectra of all three kapoks was set to be overlapped with each other. Hence, the transmittance percentage of functional groups absorbed can be distinguished.

(a) **(b)**





(c)

Fig. 1: Spectra of raw and modified versions of kapok fiber (a) from 2800cm⁻¹ to 4000cm⁻¹ (b) from 1500cm⁻¹ to 1800cm⁻¹ (c) fingerprint region.

According to the Fig. 1, strong band from 3100cm⁻¹ until 3600cm⁻¹ with a broad peak at 3342cm⁻¹ shows that the functional groups of alcohol (ROH) and amine (RNH) are presented within the kapok [9]. The trough at 2919cm⁻¹ is assigned to be the vibration of asymmetric and symmetric aliphatic CH_2 and CH_3 stretching [1]. Among three versions of the kapok fiber, oleic acid grafted kapok (OGK) produced the most pronounced trough of the aliphatic vibrations. The band at 1733cm⁻¹ corresponds to the C=O stretching vibration of carboxylic groups, ester in lignin, ketone, and acetyl ester groups in xylan [10]. The absorption band at 1593cm⁻¹ is contributed by the stretching of C=C of aromatic rings. As a comparison, the raw kapok, oleic acid grafted kapok, and decanoic acid grafted kapok presents different levels of transmittance at each functional groups absorption. By referring to the trough at 3342cm⁻¹, chemical treatments caused the removal of plant wax from the surface of the fiber while the breaking of hydrogen bond increasing the production of cellulose hydroxyl groups in fiber walls. Hence, the removed wax causing the surface

of the fiber to become rougher and wrinkled. Moreover, the hydrophilic surface is exposed which can improve the sorption capacity of the oil [10]. This occurrence also helps to create an interlocking mechanism between the oil and the kapok which allows the oil to be trapped within the system.

B. Experimental oil viscosity

Three experimental oils having a different viscosity are used in this study. The varied oil viscosity can help to justify the effectiveness of each kapok reacting towards different oils. Rate of penetration of lower viscosity oil is faster compared to more viscous oil [8]. The respective viscosity for each oils can be found in Table 1.

Table 1: Dynamic oil viscosity

Type	100 rpm	300 rpm	600 rpm
Diesel	2.0 cP	4.5 cP	8.0 cP
Bertam crude oil	16.0 cP	41.3 cP	65.6 cP
10W-40 engine oil	33.0 cP	96.5 cP	188.0 cP

C. Oil sorption test

1) Oil sorption capacity

Different kapok fiber possesses different sorption capacity towards different oil viscosity. The surrounding temperature during the execution of the test is based on the ambient temperature. The tests are repeated two times and the oil sorption capacity is averaged. As shown in Table 1, the decanoic acid grafted kapok (DGK) fiber sorption capacity towards crude oil corresponds to be the highest (79.27g/g). Other than that, the oleic acid grafted kapok (OGK) fiber sorption capacity towards diesel oil shows as the lowest capacity (43.72g/g).

The waxy surface of kapok and van der Waals forces helps the hydrocarbon to be adsorbed. Then, the internal capillary movement of the oils entering the lumen of the kapok will continue until it reached its maximum sorption capacity. The oleophilicity of the kapok together with oil physical characteristics will determine the amount of oil adsorbed and retained [11]. Since the kapok is known for its high hydrophobicity, the treatment is performed in an oil bath instead of a mixture between oil and seawater.

High sorption of crude oil is because of the high amount of hydrocarbon content which react efficiently with the waxy surface of the kapok. Meanwhile, the engine oil and diesel oil are the derivation of the crude oil which may contain less organic hydrocarbons.

Table 2: Oil sorption capacity of kapok fiber at different oil viscosity.

Type of kapok fibers	Oil sorption capacity (g oil/g sorbent)		
	Diesel oil	Engine oil	Crude oil
Raw	50.41	60.32	66.50
OGK	43.72	50.99	73.22
DGK	50.04	56.88	79.27

OGK: oleic acid grafted kapok; DGK: decanoic acid grafted kapok.

The sorption capacity for raw, OGK, and DGK are varied when treated with different oils. The lightest oil is diesel followed by crude oil and engine oil. The crude oil sorption are higher than engine oil despite of the viscous engine oil. For diesel oil, the sorption capacity of raw kapok and DGK is nearly same whereas OGK sorb the least. For engine oil, raw kapok adsorbed the highest, followed by DGK and OGK. Finally, for crude oil, DGK sorption capacity is the most prominent among all followed by OGK and raw kapok. The effectiveness of the interaction between oil and the kapok surface wax depends on the chemical compatibility which aids in minimizing surface tension and contact angle. This allows minimum energy barrier for oil penetration into the fiber tubular structure. Eventually, the minimum energy barrier has been overcome, granting availability of void fraction and effective space inside the kapok assembly [1].

The modification of kapok fibers through esterification works better when treated upon crude oil. Despite the crude oil has a lower viscosity compared to the engine oil, the crude oil is physically thicker. Oil with higher viscosity are more likely to be adsorbed and retained within the fiber [12].

2) Dynamic oil retention

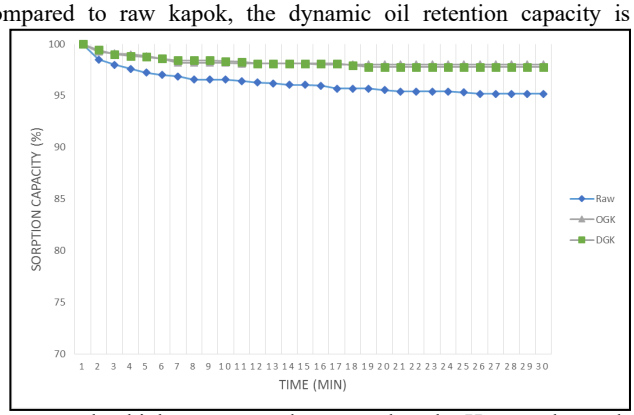
The kapok fiber has its maximum ability to retain the oil within its interstice. Therefore, the dynamic oil retention test is performed to determine the amount of oil that retainable by each of the kapok fibers. At specific times, the sorbent was allowed to immerse in each oils according to its varied viscosity. Thirty minutes dripping period is conducted permitting drip of excessive oils inside the test cells. The draining of oil is caused by the insufficient capillary pressure to hold the weight of the oils [1]. At some point, the draining process will come to an equilibrium state which will be considered as the maximum sorption capacity of the kapok fiber.

The diesel oil trapped inside the lumen of the kapok fiber seemed to be drained at a faster rate. This is mainly due to the low resistance of the oil to flow within the fiber which causes difficulty to retain the oil. Nonetheless, the fast pace of the flow allows the system to reach equilibrium quicker. Raw kapok fiber shows highest performance in retaining the diesel oil compared to both oleic acid grafted kapok fiber and decanoic acid grafted kapok fiber.

The rate of the system to reach equilibrium are a bit delayed due to the high viscosity of the engine oil. The performance of raw kapok and oleic acid grafted kapok show some similarity. However, the decanoic acid grafted kapok shows a very significant ability to retain the engine oil. This outcome can be seemed that the compatibility of the oil and the modified fiber are counterparts. The modified kapok fibers are also able to preserve the oil within its interstice higher in comparison with the raw kapok fiber.

As discussed above, the type of crude oil used is light crude and has a viscosity lower than the engine oil. However, the sorption capacity of the crude oil recorded to be the highest among other oils. As shown in Fig. 4, the decanoic acid grafted kapok retains the oil higher as to the other kapok fiber. This event occurred at both high oil viscosity which concludes that the esterified kapok fibers are compliant with a viscous oil. Most of the heavy oil trapped within the inter-fiber pores are secured by liquid bridges which are developed in the structure of the fibers [1].

In a nutshell, although the oil sorption capacity of the chemical modified kapoks are not significant and some are even lower compared to raw kapok, the dynamic oil retention capacity is



proven to be higher compared to raw kapok. Hence, the end-products of the modified sorbent capable to retain the oil at higher rates and increase the efficacy.

3) Analysis of variance (ANOVA) and reproducibility.

Based on the ANOVA, the P-values for each analyses show that are less than 0.05. Therefore, the null hypothesis can be

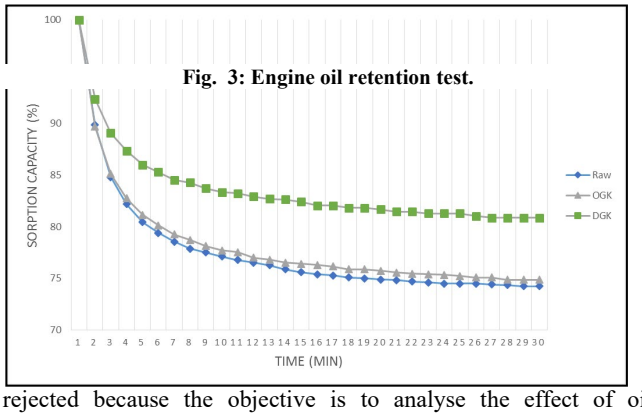


Fig. 3: Engine oil retention test.

rejected because the objective is to analyse the effect of oil absorbency when using different versions of kapok fiber. This

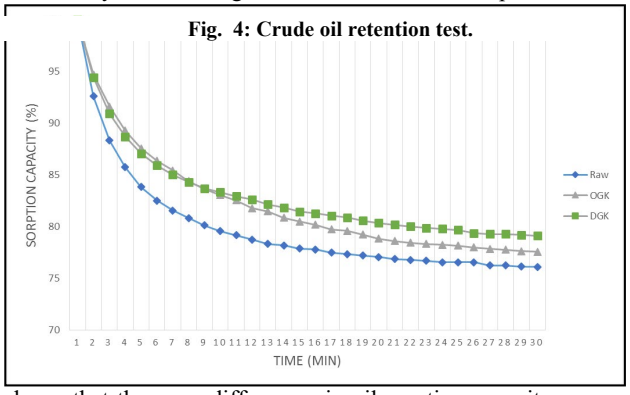


Fig. 4: Crude oil retention test.

shows that there are differences in oil sorption capacity possessed by different type of kapok fiber.

Table 3: ANOVA of raw, OGK, and DGK using different oil viscosity.

Type of oil	P-value	F	F crit
Diesel	1.12285E-78	2650.74	3.1
Engine	8.64418E-16	53.05	3.1
Crude	3.42635E-16	55.13	3.1

In addition, the F-value can also be used to determine the validity of the analysis. If the F-value is significantly higher in comparison with the F-critical value, the null hypothesis is rejected. Based on the ANOVA results, all the F-values are far surpassed the F-critical values which proves the above assumptions.

The reproducibility of the products can be identified by calculating the standard error of the test results. The calculated standard errors are based from the variance of two repeated tests. If the standard errors are minute, the product can be considered to be reproducible. In other words, significant errors conclude that the products simply cannot be replicated.

In Table 3, the standard errors for every data are very small and not significant. These small values represent that the raw kapok and its modified versions can be reproduced and still gives compelling outcomes. The reproducibility can be illustrated based on the following bar chart showing the standard errors for each sorption treatment.

Table 4: Standard error values for each sorption treatment using different kapok fibers.

Type of oil	Groups	Variance	Std Error
Diesel oil	Raw	51.31	0.92
	OGK	290.71	2.2
	DGK	313.58	2.29
Engine oil	Raw	75.96	1.13
	OGK	200.36	1.83
	DGK	72.11	1.1
Crude oil	Raw	8.27	0.37
	OGK	19.22	0.57
	DGK	7.44	0.35

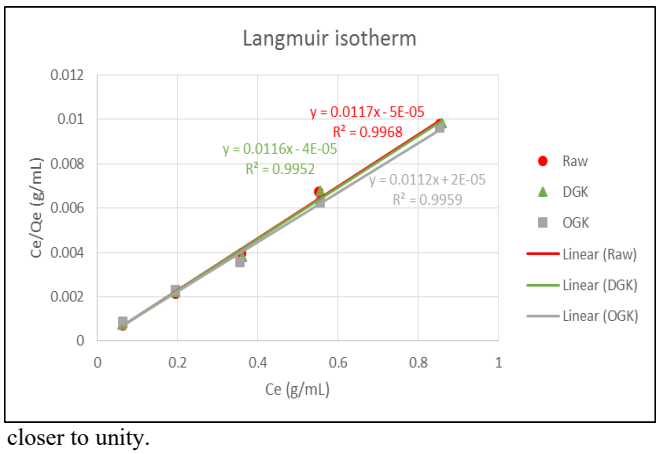
The existence of minus and plus errors are caused by the preparation of samples and executing sorption test at a different time. Since the samples are prepared separately, there are possibilities of dissimilarity in terms of surrounding temperature, chemical measurement, and period of samples stored until it is used in the sorption tests. The sorption treatments are carried out at different days which may cause differences in humidity. Hence, these varied situations lead to small changes of oil physical properties such as viscosity that affect the sorption test.

D. Application of adsorption isotherm models

To study the sorption behavior of the kapok, the adsorption isotherm modelling is applied to the experimental outcomes. The Langmuir isotherm is one of the adsorption models used to describe the sorption characteristic of the sorbent. Based on the assumption which states that the adsorption occurs uniformly at specific active sites and only monolayer adsorption exists. On the other hand, Freundlich isotherm model states that the adsorption rate occurs in terms of multilayer formation. There are possibilities that the experimental values to fit both of these models because of the small range of solute concentrations. Hence, the large sorption capacity of the adsorbent will able to approach both isotherm equations at linear form.

Fig. 5: Langmuir isotherm of crude oil on raw kapok, DGK (decanoic acid grafted kapok), and OGK (oleic acid grafted kapok).

Based on the plotted data in Fig. 5 (Langmuir) and Fig. 6 (Freundlich), the isotherm adsorption model can be selected by comparing the coefficient of regression (R^2) parameters from each model. The values of Langmuir regression coefficient are significant (raw – 0.9968, DGK – 0.9952, and OGK – 0.9959) and



closer to unity.

The coefficient of regressions (R^2) for Freundlich isotherm model is stated to be lower than the Langmuir model (raw – 0.0714, DGK – 0.0299, and OGK – 0.5576). This outcome indicates that the Langmuir model can be used to describe the equilibrium adsorption capacity of the system.

Nonetheless, R_L is defined to be the parameter which estimates either the Langmuir isotherm model are favourable, unfavourable, linear, or irreversible. As tabulated, the R_L (raw – 0.0043, DGK –

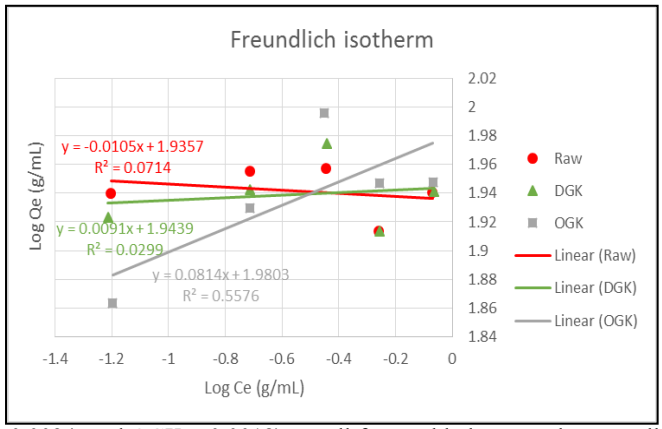


Fig. 6: Freundlich isotherm of crude oil on raw kapok, DGK (decanoic acid grafted kapok), and OGK (oleic acid grafted kapok).

0.0034, and OGK – 0.0018) are all favourable because they are all within the range of $0 < R_L < 1$.

Table 5: Parameters involved based on Langmuir and Freundlich models of crude oil.

		Crude oil		
		Raw	DGK	OGK
Langmuir	Qm	85.47	86.21	89.29
	b	234	290	560
	R^2	0.9968	0.9952	0.9959
	R_L	0.0043	0.0034	0.0018
Freundlich	k_F	6.9289	6.9859	7.2449
	1/n	-0.0105	0.0091	0.0814
	R^2	0.0714	0.0299	0.5576

IV. Conclusion

The ability of kapok to be used as a natural organic oil sorbent is validated by the outcomes of the study. Kapok fibers are applicable to be used during an event of oil spill based on the excellence sorption capacity of crude oil. The decanoic acid grafted kapok (DGK) is reliable to be used as a sorbent because it can adsorb high amount of oil and retain the oil within the fibers. The dynamic retention test proves that modified kapoks are competent in retaining the adsorbed crude oil. Thus, the process of retrieving oil-adsorbed sorbent from the spilled area will be easily done.

The Langmuir and Freundlich models have been used to mathematically illustrate the sorption manner of crude oil when using raw kapok, decanoic acid grafted kapok, and oleic acid grafted kapok. In accordance with the experimental results, the Langmuir model fitted better than the Freundlich model.

The impeccable characteristics exhibited by Malaysian kapok as a sorbent allows consideration to be selected and massively produced at an industrial scale. It is biodegradable and harmless to the environment conversely when using polymers. Hence, reduces the risk of post-auxiliary pollution after the sorption treatment process.

Acknowledgment

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