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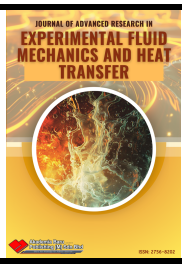

Effects of Temperature and Agitation on the Kinetics of Vegetable Oil Adsorption onto Kapok Fiber

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ABSTRACT

The unique properties of this natural fiber have sparked interest among researchers, opening possibilities for its potential use in oil spill cleanup. The oil adsorption may be influenced by temperature and agitation as revealed by past studies. This research focuses on both agitation speed at maximum of 200 rpm and temperature ranges from 30°C to 90°C in adsorption process to find an optimal condition to enhance the oil adsorption process. The analysis and characterization of kapok fiber by using scanning electron microscope (SEM), Fourier transform infrared spectroscopy (FTIR), and contact angle analysis that provided understandings into the structural and surface morphology of kapok fiber before and after oil adsorption. From the SEM morphology the kapok fiber observes the waxy surface and hollow lumen and porous structures believed can provide substantial capacity for oil storage. The FTIR analysis reveals three significant ester bands at 1732, 1370, and 1239 cm−1, which are related to the C=O stretching vibration. The contact angle measurements for raw kapok fiber before adsorption recorded at 141.5°C show high hydrophobicity and after adsorption with palm oil and waste palm oil, the sample show the contact angle at 0° indicated perfectly wetting. Temperature rise significantly affects adsorption, leading to a decrease in oil sorption capacity due to viscosity changes. Meanwhile, agitation speed notably influences oil sorption capacity, with 200 rpm showing slightly highest adsorption capacity, attributed to increased surface contact. 30 °C appears to be the optimum temperature for absorption capacity across various agitations. Pseudo-second order kinetics best fit to this study, supported by the closest R2 value to 1 and the relevance of Qe theoretical for this kinetic model. Once again, this study reaffirms kapok fiber potential as an effective oil adsorbent in water environments.

Keywords:

Kapok fiber; oil adsorption; kinetic study; agitation; oil adsorption

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1. Introduction

Considering the escalating threats posed by oil spills, which cause extensive harm to marine life, ecosystems, and coastal communities while polluting seawater, the need for effective oil adsorption methods becomes imperative [1,2]. Beyond immediate environmental consequences, spills adversely affect crops, fisheries, food security, and the overall marine community [3,4]. The pervasive impact on the marine environment, coupled with potential harm to tourism and economic losses, underscores the urgency for innovative solutions. Researchers worldwide are exploring eco-friendly and low-cost adsorbents based on natural fibers for this purpose [5].

Various natural adsorbents for pollutant removal have garnered significant attention due to their eco-friendliness, efficacy in diverse applications, and low cost [6-8]. These adsorbents include alginate, chitosan, and natural fibers such as kapok fiber, cotton fiber, and milkweed. One prominent application example of alginate is in the removal of heavy metals from wastewater [9]. Other natural adsorbents, such as chitosan, have also been extensively studied for their pollutant-removal capabilities [10]. Kapok fiber, on the other hand, is utilized as an adsorbent material in this study, given its distinctive physical and chemical characteristics, which have been researched and adapted to enhance its effectiveness in a variety of applications, most notably the treatment of oil and water [11-13].

Kapok fiber, obtained from the seeds of the Ceiba Pentandra tree, is notable for its significant low density and hollowness, thus enhancing its ability to absorb oil and has a remarkable buoyancy [14]. Traditionally used for bed fill and mattresses due to its hypoallergenic and moisture-resistant characteristics, recent research has unveiled its inherent ability to adsorb oil in water environments without modification [15]. The use of kapok fiber as an absorbent is justified due to its biodegradability and natural availability and its potential for chemical modification to enhance its adsorption characteristics [16].

Kapok fiber exhibits exceptional oil sorption capacity compared to other absorbents [17]. When compared to polypropylene fiber or polyurethane foam, this fiber demonstrates significantly higher oil sorption capability. Furthermore, milkweed and cotton fibers also display superior sorption properties, surpassing those of polypropylene fiber in various environments. The oil sorption capacity of cellulosic kapok fiber is approximately 1.5-2.0 times greater than that of polypropylene mat, as observed in comparative studies [18]. Additionally, chemically modified cotton has shown the potential to absorb up to 80 times its weight in oil, highlighting its remarkable efficiency as a sorbent. These findings underscore the distinct advantage of Kapok fiber, milkweed, and cotton fibers over synthetic materials, offering higher sorption capacities and presenting a more environmentally friendly alternative. The use of Kapok fiber stands out due to its hydrophobic properties, hollow structure, and the presence of a wax cutin layer on its surface, enabling it to repel water and absorb oil even in the presence of water. Overall, kapok fiber emerges as a highly effective and biodegradable absorbent with superior oil sorption capacity when compared to other absorbent materials.

The effect of modification of kapok fiber was studied by Wang *et al.,* [19], where the kapok fiber's natural hydrophobicity and ample lumen were enhanced for oil sorption by treating it with various solvents. Structural analyses compared untreated and treated fibers, revealing improved oil absorbency in all solvents except chloroform. $NaClO_2$ treated kapok showed the highest enhancement, with potential increases of 19.8% to 30.0% in oil absorbency. The treated fiber also demonstrated improved reusability, suggesting its promising role in oil recovery [19]. Given the concerns surrounding oil spills, extensive testing with various types of oil, including diesel, palm oil, and crude oil, has been conducted to assess the potential harm to marine ecosystems. Various

methods have been employed to determine the optimal conditions for utilizing kapok fiber in oil adsorption [20].

Accidental or intentional oil spills in water environments can result in severe damage, spreading rapidly through water movement, especially in drainage systems and posing greater threats when reaching vital water sources like rivers and seas. This study begins with the characterization of kapok fiber to identify its unique properties for subsequent oil adsorption testing.

The equilibrium of oil adsorption is the point at which the rate of oil molecules being adsorbed onto a solid surface equals the rate of oil molecules desorbing from the surface. This steady state is known as the equilibrium of oil adsorption. The equilibrium between the adsorption and desorption processes presents in this stated [21]. Oil molecules can adhere to a solid surface when they come into touch with it due to chemical interactions or intermolecular forces like van der Waals forces. The adsorption process begins quickly, with a rise in the amount of oil adsorbed on the surface. The rate of adsorption, however, progressively declines with time and finally reaches a point where it is equal to the rate of desorption [22] The surface is saturated with oil when the amount of oil molecules in the medium around it is constant at equilibrium. This indicates that no more adsorption takes place and that the ratio of oil molecules leaving the surface to oil molecules returning to it is equal. The equilibrium of oil adsorption can be influenced by various factors, including temperature, pressure, surface properties of the solid, and the nature of the oil. Understanding the equilibrium behavior is important in applications such as oil spill remediation, wastewater treatment, and the design of oil adsorbents for industrial purposes [23].

This study explored the oil sorption capacity of raw kapok fiber when exposed to vegetable oil, without any structural modification. The nature of kapok fiber was revealed through characterization techniques such as Scanning Electron Microscope (SEM), Fourier Transform Infrared Spectroscopy (FTIR), and contact angle analysis. Subsequently, the acquired data were analyzed using adsorption kinetic models to identify the most suitable fitting model. The utilization of adsorption kinetic models has been instrumental in assessing the effectiveness of the adsorbent and probing into the mechanisms of adsorption mass transfer. Nevertheless, there is a lack of well-established understanding regarding the physical meanings and solution methods associated with these kinetic models [24]. The importance rises to determine the most suitable kinetic model for studying the experimental process to gain a deeper understanding of whether the adsorption predominantly involves chemisorption or physisorption. Consequently, this research employed two widely recognized adsorption kinetic models, namely the pseudo-first order and pseudo-second order models.

2. Methodology

2.1 Materials

The kapok fiber utilized in this oil adsorption study was procured from a local vendor in Malaysia, acquired in 1 kg packs. The obtained kapok fiber exhibited impurities, including seeds and other non-fibrous components, which were subsequently removed as the initial step in the preparation process to mitigate potential inaccuracies in the results. Following this, the samples were stored in an airtight container to maintain a consistent level of moisture.

Palm oil, renowned for its widespread availability in Malaysia, was selected for this study and sourced from a local store. To enhance result accuracy, the Saji brand in 5 kg packaging was specifically chosen. Utilizing the same brand with consistent packaging ensures uniform viscosity and oil composition, facilitating the identification of results. Additionally, waste palm oil was acquired

from a local restaurant, obtaining 5kg from the same source to preserve uniformity in compound composition.

2.2 Oil Adsorption Testing

The oil adsorption testing was conducted following batch adsorption method to easier studied on adsorption characteristics. Adsorption is a process where molecules or particles adhere to the surface of a solid or liquid material which in this study is natural fiber as adsorbent. 1 g of kapok fiber is weighted and placed in petri dish to allow uniform shape of kapok fiber for all the sample for 2 days. This method distributes uniform thickness and surface exposed during testing. Kapok fiber later have been placed on top of mixture of 100 ml oil and 500 ml water in a glass beaker. The adjustment of temperature and agitation was achieved using a magnetic stirrer, as illustrated in Figure 1. Table 1 shows the experimental variables for this experiment. The mean values each sample were recorded.

The study was investigating adsorption, considering the influence of temperature and agitation. Temperature will be monitored using a mercury thermometer, while a magnetic stirrer was employed to regulate the temperature and agitation during the adsorption process. The adsorption process will extend for a duration of 25 minutes, with weight measurements taken at 5-minute intervals. This The oil measurement will follow Eq. (1).

$$\text{Oil sorption capacity, } q = \frac{m_f - m_i}{m_i} \quad (1)$$

where m_f is final weight of the sample and m_i is the initial weight of the sample. The oil sorption capacity is calculated as gram of sorption per gram of sample (gg^{-1}).

Fig. 1. Apparatus setting for oil adsorption testing

Table 1
Experimental variables

Number of samples	Oil type	Temperature, ℃	Agitation speed, RPM	Weight, g per time, min					Total oil absorbs, g
				5	10	15	20	25	
1	Palm Oil	30	0						
			125						
			200						
2	Palm Oil	60	0						
			125						
			200						
3	Palm Oil	90	0						
			125						
			200						
4	Waste Palm Oil	30	0						
			125						
			200						
5	Waste Palm Oil	60	0						
			125						
			200						
6	Waste Palm Oil	90	0						
			125						
			200						

2.3 Kinetic Study

In this study, batch adsorption was chosen to investigate the kinetics of oil adsorption onto kapok fiber. A 1 g sample of kapok fiber was placed on a 1000ml beaker containing an immiscible mixture of oil and water. The temperature difference within the mixture was set at 30, 60, and 90 ℃. To alter agitation, the speed was modified to 0, 125, and 200 rpm. The adjustment of temperature and agitation was achieved using a magnetic stirrer. The mean values each sample were recorded. The analysis and modelling of adsorption kinetics often involve the utilization of both Pseudo-First Order and Pseudo-Second Order kinetics. These approaches offer valuable insights into the underlying mechanisms and controlling factors of the adsorption process. To understand the mechanism of palm oil adsorption onto kapok fiber, two nonlinear kinetics models were applied using Eq. (2) and Eq. (3).

$$q_t = q_e\left(1 - e^{(-k_1 t)}\right) \tag{2}$$

$$q_t = \frac{q_e^2 k_2 t}{1 + q_e k_2 t} \tag{3}$$

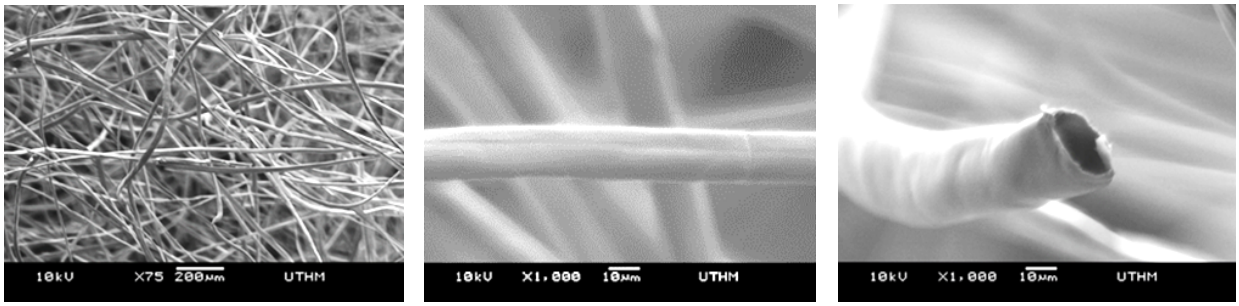
where k_1 (1/min) and k_2 (g/mg*min) represent the PFO and PSO constants, respectively. Researchers apply these models to fit experimental data, enabling the determination of essential parameters, including rate constants and equilibrium adsorption capacities. This study utilized the software OriginPro to calculate R_2 to find the closest value to 1, and comparing theoretical q_e with experimental q_e ensured the accuracy and reliability of the study.

3. Results

3.1 Surface Morphology of Kapok Fiber

SEM analysis revealed porous structures in kapok fibers, shown in Figure 2(a) at x75 magnification. These structures offer substantial oil storage capacity, accommodating oils through

intra-molecular interactions and Vander Waals forces [25]. This porosity enhances oil adhesion to the oleophilic foam surface, promoting accumulation within the porous structure. At x1000 magnification Figure 2(b), kapok fiber smooth texture and wax layer create hydrophobicity, making it ideal for water-based oil absorption. Removing the wax enhances hydrophilicity and surface roughness, improving oil adhesion and penetration. In Figure 2(c), a magnified x1000 view of kapok fiber reveals large lumens (approx. 15 µm diameter) with thin, oval to round walls measuring about 1.0 µm. Such characteristics enable the fiber to float in water and selectively absorb oil, making it well-suited for oil cleanup processes. This finding aligned with another study [5].



(a) (b) (c)

Fig. 2. Surface morphology of kapok fiber shown (a) Porous structure (b) Fiber surface (c) Hollow lumen

3.2 Functional Group Analysis

This section contrasts the FTIR spectra between raw kapok fiber and samples post-adsorption. As seen in Figure 3, the FTIR spectrum of raw kapok fiber exhibits distinctive characteristics. The band at 3339 cm^{-1} corresponds to O-H stretching vibration, while prominent peaks at 2918 cm^{-1} indicate asymmetric and symmetric stretching vibrations of aliphatic CH_2 and CH_3 groups. Spectral peaks associated with lignin between 1505 cm^{-1} and 1595 cm^{-1}, also hemicellulose peaks in between 1737 cm^{-1} and 1248 cm^{-1} are observable [26]. These peaks strongly suggest the presence of plant wax in the kapok fiber, with typical components such as long-chain alkanes, aldehydes, fatty acids, esters, ketones, and alcohols [6]. The peak obtained is also summarized in Table 2.

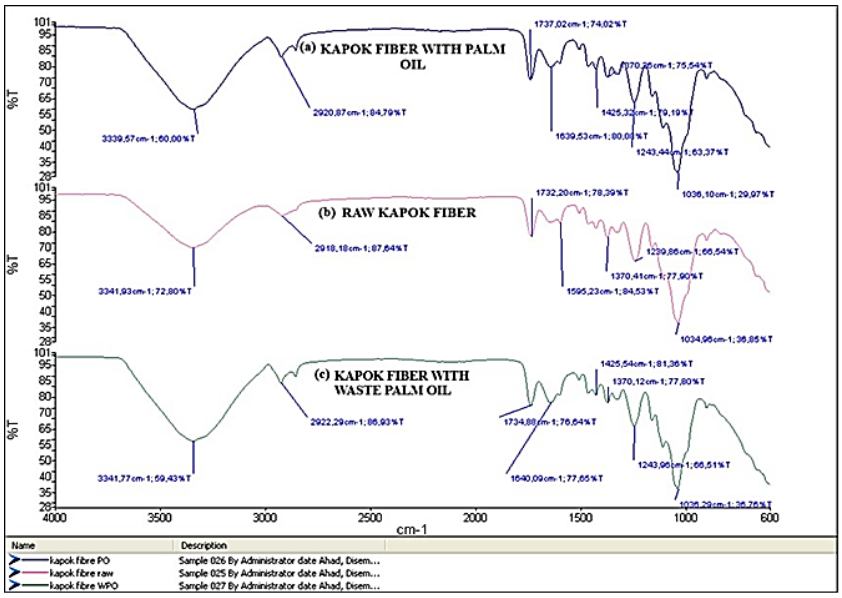


Fig. 3. FTIR spectra of (a) Kapok fiber after with palm pil (b)Raw kapok fiber (c) Kapok fiber with waste palm oil

Table 2
Major peaks observed from the FTIR spectra of raw kapok fiber samples

Peak no	Wavelength (cm^{-1})	Functional group
1	3,339	Broad intermolecular H Bonds in O-H stretching vibration group
2	2,198	Asymmetric and symmetric aliphatic CH2 and CH3 stretching vibrations
3	1732,1370,1239	Associated with the C=O stretching vibration group
4	1,595	Aromatic skeletal vibration
5	1,034	C–O–C stretching vibrations in Ester group

Figure 4 displays spectra examining functional groups in both palm oil and waste palm oil, revealing similar compositions supported by Hashim's findings [7]. Notably, both fresh and used palm oils exhibit consistent patterns, emphasizing the stability of functional groups. Analysis of palm oil spectrum identifies peaks at 2922 and 2853 cm−1 for –C–H stretching and 1743, 1465, and 1160 cm−1 for C=O, C=C, and C–O stretching, respectively, aligning with Jahangirian's study on palm kernel oil [8]. For further clarity, Table 3 provides a comprehensive summary of the identified functional groups in the analyzed sample. This detailed spectral analysis enhances our understanding of the chemical composition of both palm oil and waste palm oil, contributing valuable insights to the broader context of the study.

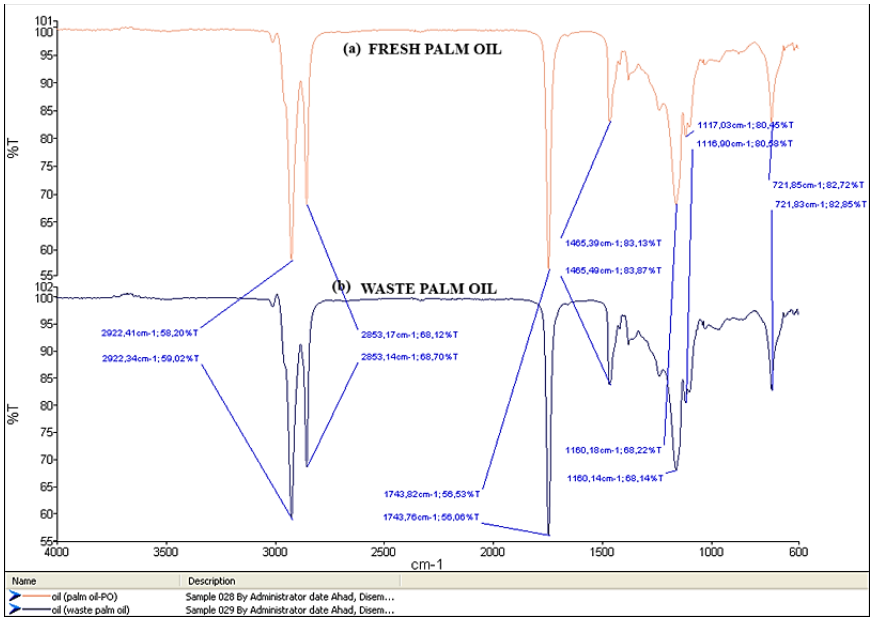


Fig. 4. FTIR spectra (a) Fresh palm oil (b) Waste palm oil

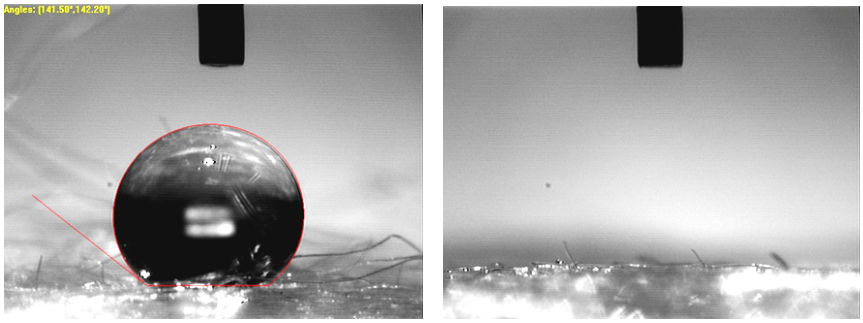
Table 3
Major peaks observed from the FTIR of palm oil and waste palm oil

Peak no	Wavelength (cm^{-1})	Functional group
1	2,922	Broad intermolecular H Bonds in O-H stretching vibration group
2	2,853	Asymmetric and symmetric aliphatic CH2 and CH3 stretching vibrations
3	1,743	Associated with the C=O stretching vibration group
5	1,465	Aromatic skeletal vibration
5	1,160	C–O–C stretching vibrations in Ester group

3.3 Surface Properties Analysis

The VCA Optima contact angle measuring instrument was employed to quantify the surface wettability, specifically focusing on the kapok fiber. This method is effective in determining the

hydrophilic or oleophilic nature of the fiber. The contact angle measurements for kapok fiber, recorded at 141.5° and 132.20° as illustrated in Figure 5(a), indicate its hydrophobic characteristics. This hydrophobicity makes kapok fiber conducive to floating in water environments and retains the adsorption of water, rendering it suitable for adsorption testing in the present study. In Figure 5(b), the lack of a noticeable water contact angle is evident as water forms a new layer above the kapok fiber sample. This observation implies full wetting of the oil and underscores the strong affinity of palm oil for kapok fiber, uniformly spreading across its surface.



(a) (b)

Fig. 5. Pure water on kapok fiber (a) Raw (b) After oil adsorption

3.4 Agitation Effect in Oil Adsorption

Figure 6 illustrates the significant role of agitation in influencing the adsorption dynamics. Notably, at all different temperatures (30°C, 60°C, and 90°C), the trend reveals that higher agitation speeds (200 RPM) result in superior oil adsorption capacity compared to lower speeds (125 RPM and 0 RPM). This observation suggests that the increase in agitation enhances the surface contact between the kapok fiber and waste palm oil. The higher rotational speed at 200 RPM likely leads to more effective interaction and contact between the adsorbent (kapok fiber) and the adsorbate (waste palm oil). This improved contact facilitates better utilization of the kapok fiber's surface area, allowing for a more efficient adsorption process. The finding that higher agitation speeds correlate with increased oil adsorption capacity is significant. It implies that optimizing agitation conditions, particularly at 200 RPM, could be a key factor in enhancing the overall effectiveness of kapok fiber in adsorbing waste palm oil from a water mixture.

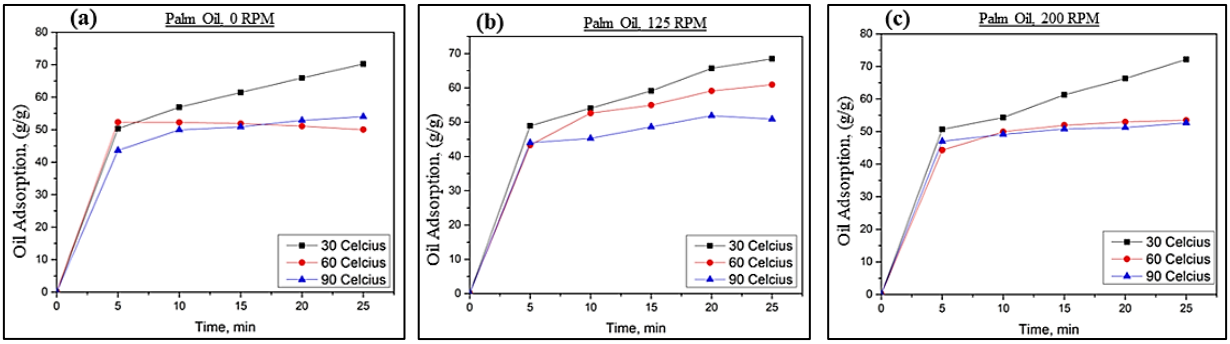


Fig. 6. Effect of temperature on adsorption in different agitation (a) 0 rpm (b) 125 rpm (c) 200 rpm

3.5 Temperature Effect in Oil Adsorption

Figure 7 shows the impact of temperature on oil adsorption capacity is evident across various agitation settings. Surprisingly, the lowest temperature setting at 30 °C outperforms both 60 °C and

90 °C under all agitation conditions. This observed trend can be attributed to the relationship between temperature and oil viscosity. The rise in temperature leads to a decrease in viscosity, influencing the ability of kapok fiber to retain oil. Specifically, as temperature increases, the reduction in viscosity diminishes the capacity of kapok fiber to effectively hold and retain the oil. This phenomenon results in a more efficient adsorption process at lower temperatures, such as 30 °C, where the viscosity is higher, promoting better retention of oil by the kapok fiber.

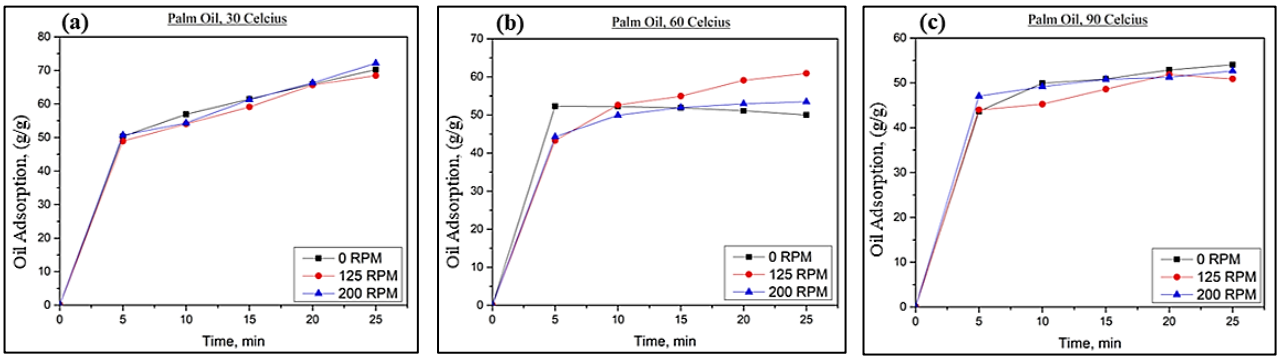


Fig. 7. Effect of agitation on adsorption in different temperatures (a) 30 °C (b) 60 °C (c) 90°C

Additional findings further support the notion that the observed outcome signifies an exothermic adsorption process; wherein elevated temperatures lead to the degradation of binding sites on the adsorbent [10]. Table 4 shows that the adsorption capacity of raw kapok in this study is significantly higher compared to the treated kapok fiber. The raw kapok demonstrated exceptional performance without requiring chemical treatment, emphasizing the potential as a cost-effective and eco-friendly solution for oil spill remediation.

Table 4
Comparison adsorption performance of kapok fiber

Adsorbent material	Adsorption capacity (g/g)	Optimal conditions (temperature, agitation)	Key observations	Study/reference
Raw kapok fiber	75.02	30°C, 200 RPM	High capacity attributed to hydrophobicity and porous structure without any surface treatment	Current study
Acid-treated kapok fiber	39.84	Room temperature, static	Improved sorption following acid surface treatment.	[11]
kapok fibre with $NaClO_2$	48.6	Room temperature, static	Modification surface for oil absorbing	[13]
Cast2-kapok fiber	68	Room temperature	Oleophilic calcium stearate-coated kapok fibers (CaSt2-KF) for improved hydrophobicity	[27]

3.6 Kinetic Study

The adsorption performance of kapok fiber with palm oil and waste palm oil further proceeded to analyze using pseudo-first order and pseudo-second order model to estimate the sorption mechanism. Figure 8 shows the experimental data obtained and both nonlinear fitted models. Notably, the R2 values for both models are significantly high and close to 1 as shown in Table 5, affirming the reliability of both pseudo-first order and pseudo-second order models in capturing the

kinetics of the adsorption process. The agreement to this model suggests that both physical and chemisorption are present during oil sorption [11].

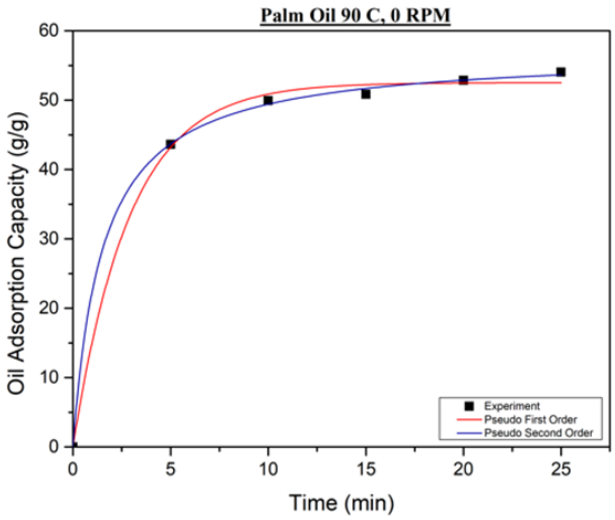


Fig. 8. The adsorption data fitted with Pseudo-First Order and Pseudo-Second Order

However, a closer examination of the results related to equilibrium adsorption capacity (q_e) reveals nuances. The pseudo-second order model is deemed more reliable in predicting the amount of oil adsorbed at the conclusion of the last measurement. The result from this study also proven by other studies that used coated kapok fiber that recorded pseudo-second order most fitted for oil in water adsorption [27].

Table 5
The kinetic model parameters related to oil adsorption under different temperature and agitation conditions

Type of oil	Temperature, ℃	Agitation speed, RPM	$q_{e,}$ Exp	Pseudo-first order			Pseudo-second order		
				K_1	q_e	R^2	K_2	q_e	R^2
Palm oil	30	0	70.22	0.254	65.99	0.98	0.0049	75.02	0.99
		125	68.46	0.242	64.585	0.973	0.00459	73.938	0.988
		200	72.15	0.236	66.694	0.962	0.00423	76.713	0.981
	90	0	54.04	0.345	52.52	0.996	0.011	56.88	0.999
		125	50.88	0.403	49.662	0.986	0.015	53.12	0.994
		200	52.68	0.490	51.144	0.996	0.026	53.398	0.999

3.7 Adsorption Mechanism

The adsorption mechanism of kapok fiber for oil is primarily influenced by its unique structural and chemical properties. The hollow lumen structure of kapok fiber provides a large surface area and internal volume for oil retention. The natural wax layer, composed of hydrophobic groups, plays a critical role in repelling water and facilitating the selective adsorption of oil. This characteristic is supported by the FTIR data, which reveal functional groups such as $-CH_2$ and $-CH_3$ that enhance the fiber's oleophilic properties [19]. Adsorption occurs predominantly through physisorption, as evidenced by the pseudo-second order kinetic model, which suggests the presence of van der Waals forces and other weak interactions [27]. At higher agitation speeds, the enhanced mixing improves contact between the fiber surface and oil, increasing adsorption efficiency. Similar findings have been

reported in studies attributing increased adsorption to improved surface utilization at higher agitation speeds [25]. Additionally, the low adsorption efficiency observed at higher temperatures (e.g., 90 °C) can be attributed to the reduced viscosity of oil, which diminishes its ability to adhere to the kapok fiber. This behavior aligns with other studies demonstrating the impact of temperature on adsorption performance, particularly for exothermic processes [13].

These findings demonstrate the combined effects of kapok fiber's morphology, chemical composition, and external conditions, such as agitation and temperature, in driving the oil adsorption process. Future studies incorporating advanced surface characterization techniques could further elucidate the specific contributions of each component to the adsorption mechanism.

4. Conclusions

The analysis of characterization unveiled the kapok fiber's lightweight nature, waxy surface, and porous structure, with recorded hydrophobic features. The distinct morphology of kapok fiber, as revealed in the SEM analysis presented earlier, distinguishes it from other natural fibers. The porous nature of kapok fiber not only facilitates convenient handling but also enables easy packing into various forms, such as mats or booms, for effective deployment in oil spill cleanup. The chemical structure remained unchanged before and after adsorption, underscoring its potential for reuse and making it an ideal, eco-friendly adsorbent. Both temperature and agitation rate play a significant role in the adsorption process. An elevated oil temperature alters its viscosity, while heightened agitation leads to increased surface contact between kapok fiber and oil.

Consequently, the study identifies the optimal conditions for oil adsorption as a combination of a low temperature (30 °C) and an agitation rate of 200 rpm. The hydrophobic sample ability of kapok fiber to retain water was evidenced by the analysis of water contact angle which recorded at 141.5°. The high retention of water making this fiber can easier to be reusing in subsequent cleanup operations. This hydrophobic nature material ensures minimal water retention, make it easier for extraction process and recovery process. To further delve into the adsorption kinetics, this study employed nonlinear pseudo-first order and pseudo-second-order models that fitted. Pseudo-second-order model fit well in this study that later through validation method by finding closest R2 to 1 and value qe in comparison with experimental data.

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