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Bleaching of crude palm oil using Kirfi adsorbent: Optimum conditions, adsorption isotherm, Thermodynamics, and Kinetics

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Abstract
Bleaching, a critical step that significantly reduces color impurities to improve palm oil quality, is essential for refining this globally produced edible oil. This study explores Kirfi adsorbent, a promising locally sourced alternative to conventional bleaching clays, for a more sustainable and cost-effective process. Several batch experiments were conducted to determine the optimum parameters for the adsorptive bleaching process. The variables considered were temperature, contact time, and adsorbent dosage, all under room pressure conditions. The optical absorbance of the oil samples, both crude and bleached palm oil, was measured at 445 nm using a UV-Vis spectrometer. The results revealed that the optimum conditions for achieving a bleaching performance of 95.61% were a contact time of 20 minutes, a temperature of 90 °C, and an adsorbent dosage of 1.5 g/10ml. Adsorption isotherms were plotted to evaluate the applicability of the Freundlich and Langmuir equations. The Freundlich isotherm, with a regression coefficient of 0.9556, provided a better fit for the isotherm data, suggesting a multilayer adsorption process. Thermodynamic analysis revealed that the process is endothermic and entropy-driven, with a rate constant of 0.0546 S^{-1}, an enthalpy change (ΔH) of 26.678 kJ/mol, and an entropy change (ΔS) of 92.260 J/molK. The Gibbs free energy (ΔG) values, ranging from -4447.0 J/mol to -9511.2 J/mol, increase negatively with temperature (70 ^{o}C—120 ^{o}C), indicating the process becomes more feasible at higher temperatures. These findings have potential implications for sustainable and cost-effective practices in the palm oil refining industry.

Keywords: Adsorbent, Adsorption isotherm, Adsorptive bleaching, Palm oil, Rate constant, Single factor optimum, Thermodynamic parameters

1. Introduction
Palm oil is a vegetable oil obtained from the fresh, fleshy fruits of oil palms through extraction. It is a vital ingredient for the food industry because of its superior characteristics (Chigozie & Stone, 2014; Raji et al., 2019). Oil produced by solvent or, most times, by mechanical pressing extraction of fresh ripe palm fruits, termed crude palm oil (CPO), contains impurities like organic pigments, oxidation metals, trace metals, and trace soaps (Raji et al., 2019; Usman et al., 2012). To achieve high-quality oil standards necessary for edible applications, these impurities, which negatively affect the qualities taste, smell, appearance, and Shelf life stability of the oil must be removed. Originally described as a process of mixing oil and clay adsorbent to remove color, bleaching, which usually comes after

degumming and refining processes, is needed to remove specific contaminants which are not effectively removed by the neutralization processes before the oil goes through the deodorization process (Brooks et al., 2019). Bleaching, which involves physical and chemical interaction of an oil with an adsorbent, occurs via adsorption of the impurities onto the surface-active adsorbent material or bleaching agent for some time (Brooks et al., 2019; Raji et al., 2019; Usman et al., 2013). Adsorptive bleaching remains a crucial step in the palm oil refining industry. Being the last operation in the oil refining process before deodorization, bleaching is considered by some people to be the "safety net" of the refining process (Brooks et al., 2019).

The major adsorbent used in the palm oil refining industry is bleaching earth; others include activated carbon and silica gel. The adsorptive capacity of adsorbents depends on factors such as mineralogical structure and adsorptive properties, like the surface area, particle size distribution, surface acidity, and porosity. Bleaching clays are generally composed of one or more calcium montmorillonite, attapulgite, and sepiolite (Brooks et al., 2019). Some clays, mostly bentonite minerals, have limited adsorptive properties in their natural states thus require chemical treatment by acids to improve their bleaching power, while others, mostly attapulgite, and sepiolite minerals, have naturally high bleaching power without any acid treatment (Brooks et al., 2019). Although bleaching improves the quality of palm oil by removing contaminants, it may also lead to the loss of valuable compounds like tocopherols and sterols, significant loss of oxidative stability, formation of trans fatty acids, and increase in free fatty acid (Naz et al., 2011; Škevin et al., 2012). The optimization of the bleaching process, mainly temperature, time, and clay content, is thus very important to both achieving high-quality products, the economic viability of the purification process, and minimizing undesirable oil changes (Brooks et al., 2019; Škevin et al., 2012). Optimization of CPO bleaching variables is achieved either through response surface methodology or one factor at a time methodology (Babayemi et al., 2021; Raji et al., 2019). The latter involves studying a process by maintaining other parameters at designated constant levels (Raji et al., 2019).

Adsorption isotherm describes the equilibrium of the adsorption of the palm oil impurities on the adsorbent surface at a constant temperature. Theoretical and empirical models that describe reversible adsorption have been developed based on a thermodynamic equilibrium (Nkeiruka et al., 2024; Nkeiruka et al., 2023; Babayemi et al., 2021). The bleaching of palm oil often adhere to the Freundlich adsorption isotherm, as they involve adsorption from liquids mediums (Nwabanne & Ekwu, 2013). Langmuir isotherm follows the assumption that the process happens on a uniform surface. Thermodynamic parameters like the standard Gibb's free energy change, the standard enthalpy change, and the standard entropy change indicate whether the adsorption process is spontaneous, non-spontaneous, exothermic, or endothermic (Nweke & Ajemba, 2022). Studying the optimum conditions, isotherm, thermodynamics, and kinetics of the bleaching activities of an adsorbent are necessary for the development of the bleaching process.

In recent years, the field of palm oil bleaching has seen significant advancements (Abdullahi et al., 2022; Mba et al., 2015). Researchers have delved into various adsorbents, process variables, and kinetic models to improve efficiency. While traditional adsorbents like activated carbons and activated bleaching earth have proven effective in removing impurities from crude palm oil (CPO), new challenges require innovative solutions. One promising avenue is the acid activation of clays. Despite Nigeria's abundant clay deposits, its potential for vegetable oil bleaching remains largely unexplored (Abdullahi et al., 2022). Kirfi Kaolin clay, found in Kirfi village of Bauchi state, Nigeria, is one such clay with untapped possibilities. Previous research by Abdullahi et al. (2022) explored Kirfi clay for bleaching purposes, creating several adsorbents through acid activation. However, a comprehensive study optimizing the bleaching process of vegetable oils using adsorbents prepared from this clay has not been reported. This study aims to fill this gap by optimizing the bleaching process for CPO using Kirfi adsorbent under ambient pressure conditions, determining the optimal parameters (temperature, time, and dosage), evaluating the applicability of Langmuir and Freundlich equations, and quantifying the thermodynamic parameters and rate constants associated with the bleaching process.

2. Materials and methods

2.1. Sample collection and treatment

The adsorbent (Kirfi Adsorbent) was prepared from a clay sample sourced from Kirfi village in Bauchi state of Nigeria. The optimal conditions for its preparation were established based on a study conducted by Abdullahi *et al*. (2022). CPO was procured from Yan Manja Market in Jos North Local Area of Plateau State, Nigeria. The CPO underwent degumming, a critical step in the refining process. Specifically, 0.1% phosphoric acid was added to the oil at 80 ^{0}C, followed by stirring for 15 minutes. Subsequently, the oil was washed with distilled water and centrifuged to remove the phosphatides. Table 1 outlines the key characteristics of the adsorbent.

Table 1. Characteristics of Kirfi adsorbent (Abdullahi et al., 2022)

Parameter	Adsorbent
Color	White
Particle size	200 mesh
pH (10% in water)	3.54
Apparent bulk density (g/l)	0.6007

2.2. Bleaching process

In this study, bleaching parameters (contact time, temperature, and adsorbent dosage) were optimized using a one-factor-at-a-time (OFAT) approach. Each variable was varied independently while keeping the others constant, and the optimum value for that factor was identified. The overall (combined) optimum bleaching performance was then obtained by applying these individually optimized values simultaneously.

To 10 ml of the degummed CPO at 70 ^{0}C, 0.5 g of adsorbent was added, and the temperature was raised to 90 ^{0}C on a magnetic hot plate and stirred for 30 minutes. The oil was cooled to 70 ^{0}C and filtered under suction with a sinter funnel to remove the adsorbent. This process was repeated with adsorbent dosages of 1.5 g, 2.0 g, 2.5 g, and 3.5 g; bleaching temperatures of 70 ^{0}C, 90 ^{0}C, 100 ^{0}C, and 125 ^{0}C, and contact times of 10 minutes, 20 minutes, 30 minutes, 40 minutes, and 60 minutes. Each experiment was repeated twice, and the mean of the results was computed.

The absorbance of the CPO and those of all the BPOs were measured using a UV-Vis spectrophotometer (UV-1700, Shimadzu, Japan). A solution of the CPO in acetone (LOBA Chemie PVT Ltd, India) was used to obtain the wavelength of maximum absorbance. This wavelength (445nm) was used to assess the absorbance of the CPO and the BPOs using acetone as a reference, and the percentage bleaching performance was calculated using Equation 1.

$$BP\ (\%)\ = \frac{Ao-A}{Ao} \times 100 \quad (1)$$

Where Ao and A are the absorbances of the CPO and BPO, respectively (Foletto *et al*., 2011; Usman *et al*., 2013). Finally, the optimum parameters were used simultaneously to determine the overall bleaching performance of the adsorbent.

2.3. Adsorption isotherms

To assess the nature of adsorption quantitatively, the experimental data obtained from the adsorption of pigments onto Kirfi adsorbent at 70 ^{0}C in the bleaching process were subjected to fitting with model isotherm equations, namely the Langmuir and Freundlich equations. The Langmuir and Freundlich equations for oil bleaching are expressed as equations 2 and 3, respectively (Nkeiruka et al., 2024; Nkeiruka et al., 2023; Nwabanne & Ekwu, 2013).

$$\frac{Xe}{(X/m)} = \frac{1}{a} + \frac{a}{b} Xe \quad (2)$$

$$log\frac{X}{m} = log\ K + n\ log\ Xe \quad (3)$$

Where X is the amount of substance adsorbed, X_e is the residual amount at equilibrium, m is the mass of adsorbent, a and b are Langmuir constants, and K and n are Freundlich constants. K is a system constant associated bonding and adsorption, or the distribution coefficient, which quantifies the adsorbate adsorbed onto the adsorbent at unit equilibrium concentration. Meanwhile, n corresponds

the adsorption driving force magnitude and the energy distribution adsorption sites (Nkeiruka *et al*., 2023; Nwabanne & Ekwu, 2013).

The amount of pigment adsorbed (X) and the residual amount at equilibrium (X_e) can be obtained using Equations 4 and 5 (Nkeiruka *et al*., 2023; Nwabanne & Ekwu, 2013).

$$X = \frac{Ao - At}{Ao} \quad (4)$$

$$Xe = \frac{At}{Ao} = 1 - X \quad (5)$$

Where Ao is the absorbance of the CPO and A_t is the absorbance of the BPO at time t. A plot of $X_e/(X/m)$ versus X_e and log X/m versus log X_e was done, and the least squares analysis was used to calculate the parameters (a, b) and (K, n) from the intercept and slope of straight lines of the isotherms.

2.4. Adsorption thermodynamics

Gibbs free energy (ΔG), enthalpy (ΔH), and entropy (ΔS) are thermodynamic parameters used to evaluate the thermodynamic behavior of the adsorption process. The thermodynamic properties were calculated using equations 6, 7, and 8 (Nkeiruka et al., 2024; Nkeiruka et al., 2023; Usman et al., 2013).

$$lnKe = -\frac{\Delta H}{RT} + \frac{\Delta S}{R} \quad (6)$$

$$\Delta G = -RTlnKe \quad (7)$$

$$Ke = \frac{Ao - Ae}{Ae} = \frac{X}{Xe} \quad (8)$$

Where X_e = the residual amount at equilibrium, R = gas constant (8.314 J/molK), T = absolute Kelvin temperature, and K_e = equilibrium constant.

2.5. Rate constant

The rate constant of the bleaching process, which measures adsorption speed, allowing the fine-tuning of the process parameters for desired efficiency and minimal processing time, was obtained using equation 9 (Nwabanne & Ekwu, 2013)

$$ln\frac{A}{Ao} = -k(t)^{0.5} \quad (9)$$

Where A is the absorbance of the oil bleached at time t(s), and A_o is the absorbance of crude or unbleached oil. From this equation, the linear regression between $\ln(A/A_o)$ and $t^{0.5}$ or $\ln X_e$ and $t^{0.5}$ is a straight line whose slope is equal to -k.

2.6. Characterization of oils

Free fatty acid (FFA) contents of the CPO and BPO were determined using the titration method outlined by Usman *et al*. (Nweke & Ajemba, 2022). Color and density assessments were conducted following the procedures outlined by Langkuk *et al*., 2021. Absorbance (Abs) was measured at 449nm using a UV-Vis spectrophotometer (UV-1700, Shimadzu, Japan). Additionally, specific gravity (sp gr) was determined by comparing the weight of a density bottle when filled with the oil with the weight when filled with water.

3. Results and discussions

3.1. Single factor optimum bleaching performance

3.1.1. Effect of contact time

Figure 1 shows the variation of the bleaching performance of Kirfi adsorbent with contact time. The results show that bleaching performance increased rapidly with contact time up to an optimum of 20 min, corresponding to a bleaching performance of 94.1184, beyond which there is no considerable increase in bleaching performance. Nwabanne & Ekwu (2013) and Usman et al. (2013) also observed an increase in color removal from palm oil with an increase in contact time.

The quick adsorption of pigment by the adsorbent during the first 10 to 20 minutes can be attributed to the availability of vacant surface sites at the initial stages. The observed optimal contact

time of 20 min for kirfi adsorbent correspond with the findings of Usman et al. (2013), who established that a contact time 15 to 45 minutes is necessary for effective bleaching

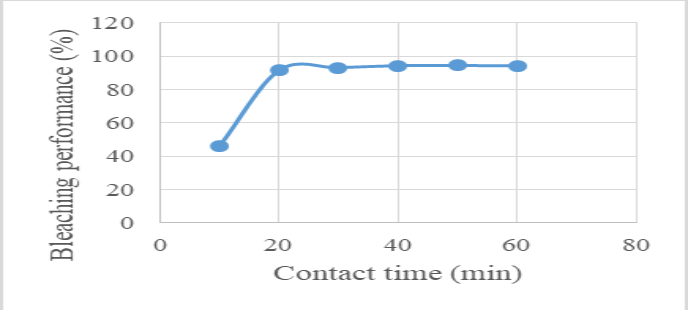


Figure 1: Bleaching performance (%) of Kirfi adsorbent at different contact times (minutes)

3.1.2. Effect of temperature

According to Figure 2, percentage bleaching performance increased with an increase in temperature to a maximum at 90°C, corresponding to a BP of 92.278%, beyond which there is no considerable increase in BP with temperature. This situation was also reported in similar studies by Langkuk et al. (2021) and Usman et al. (2013). The change in bleaching performance with an increase in temperature occurs as a result of an increase in the kinetic energy of the color pigment particles, which increases the frequency of collisions between the adsorbent and pigment particles (Ajemba & Onukwuli, 2012).

According to Usman et al. (2013), typical bleaching temperature ranges from 90 to 125°C. He also noted that oil viscosity decreases with increasing temperature, resulting in better dispersion of particles, improved clay oil interactions, and flow ability. Nwabanne and Ekwu (2013) reported that the optimum bleaching temperature is specific for a particular adsorbent and oil, which is between 100 – 120°C °C for palm oil.

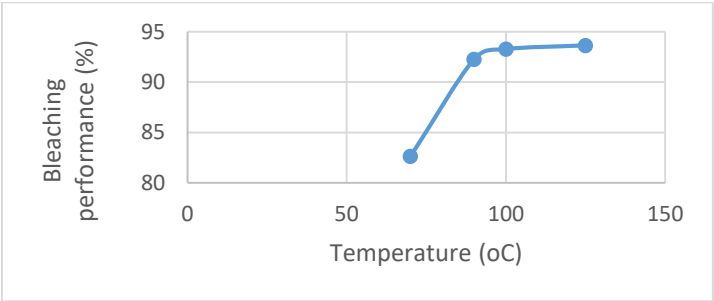


Figure 2: Bleaching performance (%) of Kirfi adsorbent at different temperatures

3.1.3. Effect of adsorbent dosage

Figure 3 is the graph of bleaching performance with clay dosage. The figure shows that there was an increase in bleaching performance with increasing adsorbent dose. This could be attributed to the complete adsorption of the color pigments to the available active sites on the adsorbent (Mustapha *et al*., 2013). The bleaching performance of the adsorbent increased from 40.332 % to an optimum of 94.525 %, corresponding to a clay dosage of 1.5g. Earlier reports showed that palm oil required as much as 2 to 4% or more to meet final color requirements (Valenzuela Díaz & Santos, 2001). Nwabanne and Ekwu (2013) have reported an increase of BP% with the increase in clay dosage for the decolorization of palm oil using local Nigerian clay.

The stabilization of bleaching performance beyond the optimum is due to the adsorption equilibrium established between the adsorbent and oil mixtures, limiting further pigment removal despite the excess adsorbent dosage (Usman et al., 2013).

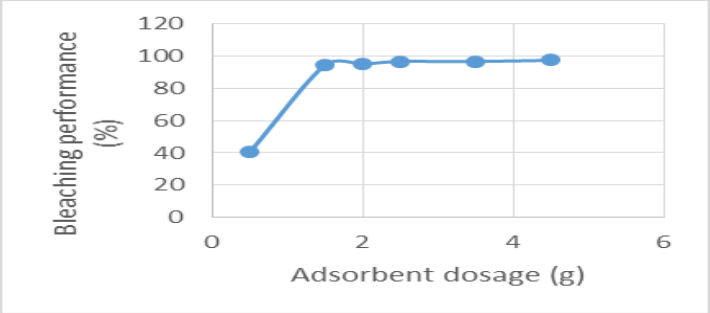


Figure 3: Bleaching Performance (%) of Kirfi adsorbent at Different dosages (g)

3.2. Combined optimum bleaching performance

The overall optimum bleaching performance determined by applying the individually optimized parameters obtained in Sections 3.1.1–3.1.3 (contact time: 94.1% at 20 min; temperature: 92.3% at 90 °C; dosage: 94.5% at 1.5 g/10 ml) simultaneously reached 95.61%, slightly higher than the single-factor optima.

This outcome reflects the one-factor-at-a-time (OFAT) optimization approach, where each parameter is optimized in isolation before combining them. The improvement in bleaching efficiency demonstrates the synergistic effect of simultaneously applying the best values of time, temperature, and dosage. Similar findings have been reported in recent studies: Başaran et al. (2025) showed that combining optimal bleaching earth content, temperature, and duration reduced glycidyl esters and improved palm oil quality; Nwabanne and Ekwu (2013) observed that Nigerian clays achieved higher bleaching efficiency when optimal parameters were applied together; and recent work by Yahya et al. (2026) using nano-SiO_2 bleaching earth confirmed that simultaneous optimization enhances both efficiency and sustainability.

Thus, the combined optimum not only validates the OFAT approach but also highlights the importance of parameter synergy in achieving maximum bleaching performance and oil quality improvement

3.3. Langmuir and Freundlich isotherms

Adsorption isotherm plays a pivotal role in our study by providing insights into the equilibrium behavior of Kirfi adsorbent during the bleaching process. It helps us understand how the adsorbent interacts with pigments and impurities at varying concentrations at a constant temperature (70 ^{0}C). To evaluate the nature of the adsorption, the experimental isotherm data were fitted by model isotherm equations, namely: the Langmuir equations and the Freundlich equations in terms of absorbance (X) and concentration (C), respectively (Foo & Hameed, 2010; Toor & Jin, 2012).

Plots of the Langmuir and Freundlich isotherms are shown in Figures 4 and 5, respectively. The Langmuir isotherm plots show randomly distributed data with a regression coefficient of 0.7463, whereas a better fit with a straight line was obtained in the Freundlich isotherm plots with a regression coefficient of 0.9556. A similar conclusion was made by Ajemba and Onukwuli (Ajemba & Onukwuli, 2012). The values of the constants calculated from the slopes and intercepts of the plots are presented in Table 2.

Table 1: Isotherm constants for Kirfi adsorbent

Isotherms					
Freundlich isotherm			Langmuir isotherm		
n	K	R^2	a	b	R^2
0.540	14.290	0.9556	1.1546	-0.695	0.7463

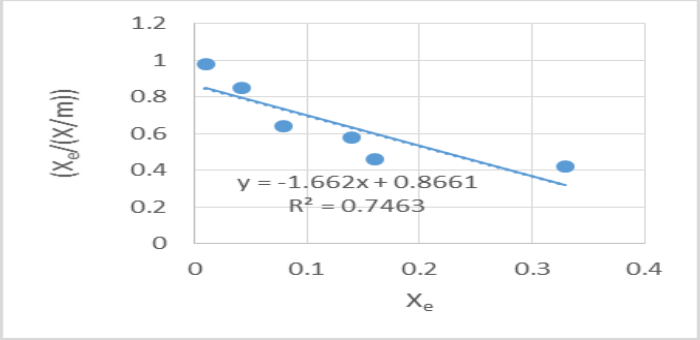


Figure 4: Langmuir Isotherm Plot for the bleaching process

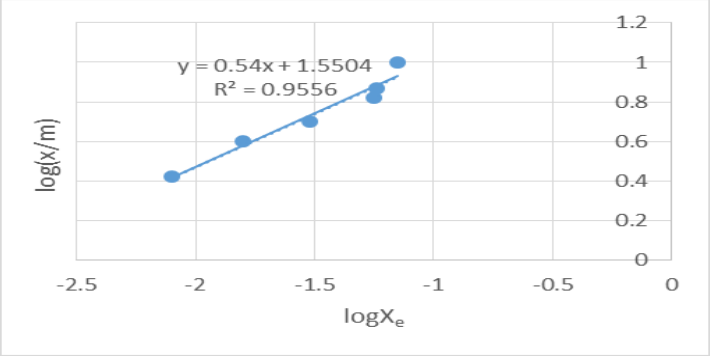


Figure 5: Freundlich Isotherm Plot for the bleaching process

3.4. Thermodynamic parameters of adsorption

Enthalpy change (ΔH) of 26.678 kJ/mol and entropy change (ΔS) of 92.260 J/molK were calculated from the slope and the intercept of equation 6 by plotting $\ln K_e$ against1/T (Figure 6), and ΔG was calculated at different temperatures using equation 7. Table 3 represents the thermodynamic parameters for the CPO bleaching by Kirfi adsorbent. The bleaching of CPO by Kirfi absorbent is driven by entropy. The positive values of enthalpy indicate that the bleaching process is endothermic, and also its magnitude gives information on the type of adsorption, which can be either physical or chemical. The enthalpy of adsorption (26.678 kJ/mol) corresponds to chemisorption (activated adsorption) (Nkeiruka et al., 2023; Nkeiruka et al., 2024). Chemisorption occurs when the intermolecular attractive forces between molecules of the adsorbate itself are greater than those between molecules of adsorbate and adsorbent (Usman et al., 2013). In this study, the positive value of ΔS suggests an increase in the randomness at the adsorbate-solution interface during the adsorption process. ΔH of the pigments are in the range of chemisorption, an indication that the adsorption of pigments on the adsorbent takes place via chemisorption. The increase in values of ΔG (-4447.0 J/mol to -9511.2 J/mol), as shown in Table 3, with increasing temperature (70 ^{0}C to 125 ^{o}C) means that the feasibility of adsorption increases with increasing temperature.

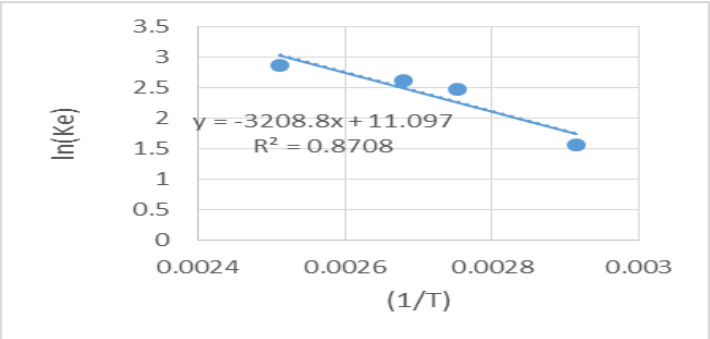


Figure 6: A plot of ln(Ke) against 1/T

Table 2: Thermodynamic parameters for CPO bleaching

T(°C)	70	90	100	120
ΔG (J/mol)	-4447.0	-7489.9	-8159.8	-9511.2

3.5. Rate constant

The rate constant (k =0.0546) obtained from the plot of $\ln(A/A_o)$ against $t^{0.5}$, as shown in Figure 7, is an indication that the adsorption of pigments of the oil is not very rapid. Joy et al. obtained similar values of k (0.031, 0.072, and 0.199) at temperatures of 65 ^{0}C, 80 ^{0}C, and 90 ^{0}C, respectively, using an adsorbate activated with 1M H_2SO_4 (Joy et al., 2007). On the other hand, Nwabanne and Ekwu (2013) obtained a value of 1.552 using an adsorbent prepared by activating a local clay obtained from Nteje town in Anambra State, Nigeria using 1 M sulphuric acid at 95^{0}C °C.

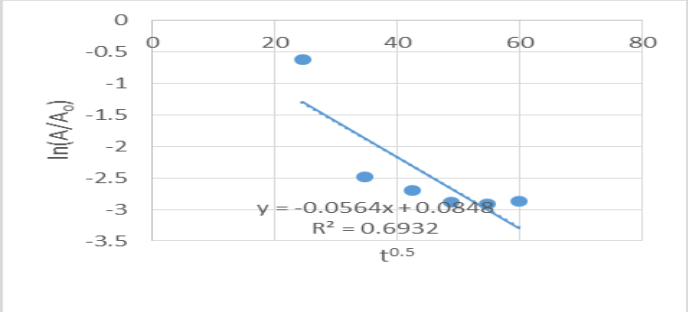


Figure 7: Plot of ln(A/A0) against $t^{0.5}$

3.6. Properties of oils

Table 4 shows the properties of palm oil before and after bleaching. The properties of the oil after bleaching with Kirfi adsorbent show a considerable difference from the properties of the crude oil. Free fatty acids are included among compounds naturally present in low amounts in vegetable oil; however, they can be produced during the storage or processing of edible oils. Thus, the quantity of FFA is a good measure of the quality of oil. The FFA content of the BPO was found to be lower than that of the CPO. This implies that Kirfi adsorbent can reduce not only pigment from oil but also FFA.

Table 3: Characteristics of CPO and BPO

Property	CPO	BPO
Color	Reddish	Yellowish brown
Odor	Palm oil odor	Palm fruit odor
Taste	Palm fruit taste	Blend taste
Abs (nm)	0.956	0.728
Sp gr	0.9377	0.9375
FFA (%)	3.8	2.8

4. Conclusion

The optimum bleaching condition of crude palm oil (CPO) (contact time,20 minutes; temperature, 90^{0}C, and adsorbent dosage, 1.5g/ 10ml of CPO) was equivalent to a bleaching performance of 95.61%, indicating that Kirfi adsorbent is a better adsorbent at the optimum condition than at the non-optimum condition (94.05%) (Abdullahi et al., 2022).

Langmuir and Freundlich's adsorption models were applied to describe the experimental equilibrium isotherms. The Freundlich isotherm model (R^2 = 0.9175) adequately described the adsorption data, indicating that the process is physical adsorption by weak bonds between the adsorbent and the adsorbate (Bakhtyar et al., 2011).

According to the thermodynamic parameters, the bleaching process is an endothermic process driven by increased randomness at the adsorbate-solution interface.

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