RESEARCH PAPER

Kinetic and equilibrium studies of bleaching used palm olein oil using alkaline-activated rice husk

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Abstract

In this study, the effectiveness of bleaching used palm olein oil was investigated using alkaline-activated rice husk as an adsorbent. The rice husk sample was activated with potassium hydroxide. Characterization of the raw and the alkaline-activated rice husk (AARH) was carried out using Brunauer-Emmett-Teller and scanning electron microscopy. The AARH shows an improvement in the total surface area (127.210 m²/g) compared to that of the raw rice husk (59.520 m²/g). Scanning electron microscopy micrograph investigation reveals an improvement in the morphological characteristics of the AARH. The examination of the bleached used palm olein oil using Fourier transform infrared spectroscopy reveals the existence of distinctive functional groups. The pseudo-second-order kinetic model, with a coefficient of determination, R² of 0.9848, provides the best description of the adsorption process at 80 °C. The Temkin isotherm model offered the best and most accurate fitting for the adsorption figures, with the highest values of R² (0.5395, 0.9967, 0.9650, and 0.9990) at temperatures of 60, 70, 80, and 90 °C, respectively. This investigation has proven that rice husk can be converted into an effective and efficient alkaline-activated adsorbent for vegetable oil bleaching.

Keywords: Alkaline-activation, Equilibrium study, Kinetic study, Rice husk, Used palm olein oil

1. Introduction

Waste vegetable cooking oil from industries and households is known as used cooking oil. They are categorized as wastes in the European Union waste catalog under the classification of edible oils and fats and thus may be employed as waste raw materials with additional value upon collection. During the preparation phase, good, fresh cooking oil is used to cook at temperatures between 160 and 180 °C more than three to four times. When cooking oil is exposed to either air or water during the frying process, the oil's quality degrades to the point that it can no longer be relied on for further frying and must be disposed of ² During frying, oxygen, moisture, high temperatures, and component leaching in the food cause vegetable oils to oxidize and hydrolyze.3 Numerous degradation byproducts, including volatile and nonvolatile compounds, are consequently created and accumulated. According to recent research, the production of used frying oils accounts for around 20-32% of total vegetable oil use. Thus, it is estimated that the world produces 42 million tons of frying oil annually.4 Many environmental and economic difficulties arise when used oils are discarded within solid wastes or via drains and sinks. This could result in increased operating costs for sewage and waste treatment facilities, hasten the development of pests and vectors, harm infrastructure, and harm the environment and public health. Additionally, pouring used oils into soil resources can damage the ecosystem since oil kills helpful living things like bacteria and worms by obstructing the soil's air and water supply. Used oil contamination in water bodies could obscure sunlight and limit oxygen levels, harming aquatic wildlife and plants.^{4,5}

Food processing industries produce waste when transforming unprocessed materials (raw) into

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valuable products. Researchers have focused on converting biodegradable waste from food, such as rice husk, groundnut hull, orange peel into a useful material. If there's no practical way to solve the problem, like turning it into biodiesel, adsorbent, or other useful resources, the growing amount of waste from food and other materials will become an important threat to the environment.^{6,7} Despite regulations and laws put in place by the government, there appears to be an upsurge in the disposal of waste-bleaching materials.8 In Nigeria, the majority of the bleaching materials employed are conventional, and a number of them nonreclaimable, causing environmental pollution. This can be mitigated to some extent by using renewable agricultural waste materials in bleaching processes.⁸ In general, soluble organic compounds can be extracted from agricultural wastes through the pretreatment process by applying a variety of modifying agents, such as base and acid solutions. This article provides the findings of a study that examined how chemical treatments affected the potential of an alkaline activated rice husk (AARH) to bleach used palm olein oil. The findings of the investigation will advance our understanding of alkaline activation and offer guidance for producing AARH on an industrial scale. Using rice husk that is locally sourced, this process would help to reduce importation and boost the Nigerian economy.

2. Materials and methods

A bag of rice husk was purchased from a nearby milling center in Kasuwan Gwari Market, Minna, Niger State, and a 51 gallon of used palm olein oil, sourced from Devon King's vegetable oil, Ikorodu, Lagos State, Nigeria, was purchased from an akara vendor in Kasuwan Gwari Market, Minna, Niger State. The high-quality analytical chemicals sourced from BDH chemicals, London, that were used had been purchased from Panlac Chemical Store, Keteren Gwari Minna, Niger State, Nigeria.

2.1. Adsorbent preparation

400 g of rice husk was weighed using the digital scale and placed into a 1000 ml conical flask. 500 ml of a 5.0 M potassium hydroxide (KOH) solution was poured into the flask to activate the pores of the rice husk. The mixture was stirred and allowed to rest for about 2 h at room temperature. Then, while being shaken occasionally, the solution was heated for 3 h at 100 °C. The neutrally activated rice husk was then dried for a further 12 h at 70 °C, and then sieve using 0.25 mm mesh size. ¹⁰

2.2. Acid degumming

500 ml of used palm olein oil were placed in a conical flask and placed in a thermostatic water bath shaker adjusted to 85 °C. 2 ml of phosphoric acid (H₃PO₄) was then added to the mixture. The mixture was agitated for 10 min so as to thoroughly degum the oil. To help dissolve the hydratable gums and make sure the palm olein oil is free of gums, 20 ml of distilled water were poured into the heated oil-acid solution that had been divided into portions so as to help remove the hydratable gums as well as ensure that the used palm olein oil is free of gums. This was achieved by using a centrifuge machine for 5 min at 4000 rpm. ¹¹

2.3. Neutralization

Used palm olein oil that had been degummed was neutralized by mixing 200 g of oil with 20 ml of sodium hydroxide (0.1 M) solution, producing stock soaps. After adding, stirring for 10 min, and letting the mixture split into two layers, a centrifuge machine was employed to separate the oil-stock soaps for 5 min at a speed of 4000 rpm. ¹¹

2.4. Bleaching process

The neutralized used palm olein oil was bleached using a glass beaker that served as the bleaching container. The bleaching container (250 ml) was filled with 35 g of neutralized used palm olein oil and placed on a hotplate with a magnetic stirrer. 3 g of AARH was then added to the container. At contact intervals of 15, 25, 35, and 45 min, the mixture was heated while being constantly stirred. After the bleaching process had been concluded, the AARH was separated from the oil by filtering using Whatman filter paper, and the absorbance of the bleached oil was determined at 450 nm wavelength using ultraviolet-visible spectrophotometer. The studies were conducted at 60, 70, 80, and 90 °C. Different doses of AARH (2.0, 4.0, 6.0, 8.0, and 10.0 g) were used in the equilibrium adsorption study. The bleaching process was completed with contact times of 15, 25, 35, and 45 min at an operating temperature of 80 °C.¹²

2.5. Adsorption kinetic study

To assess the principle governing the process of the adsorption and the impact of contact time on the effectiveness of bleaching used palm olein oil using AARH, two kinetic models¹³ were simulated in the equations in Table 1 below.

Kinetic model	Kinetic equation	Isotherm model	Isotherm equation
Pseudo-first-order	$\ln (q_e - q_t) = \ln q_e - k_1 t$	Langmuir	$\frac{X_e}{X_e} = \frac{1}{X_e} + \frac{1}{X_e}$
		Freundlich	$q_e q_m K_L q_m^{He}$ $\log q_e = \log K_f + \frac{1}{n} \log X_e$
Pseudo-second-order	$rac{t}{q_t} = rac{1}{K_2q_e^2} + tigg(rac{1}{q_e}igg)$	Temkin	$q_e = \frac{RT}{b_T} ln K_T + \frac{RT}{b_T} ln X_e$

Table 1. Adsorption kinetic and equilibrium isotherm models for the bleaching process.

2.6. Adsorption isotherm

Adsorption isotherm assists in the comprehension of the adsorption technique and offers information on the surface area, adsorption capacity, and the adsorbent and adsorbate's interactions. ¹⁴ The results of the experiment were fitted into the linearized form of Freundlich, Langmuir and Temkin isotherms. ¹⁵ as shown in Table 1 below.

Where the pseudo-first-order and pseudo-second-order constants are denoted by K_1 (min⁻¹) and K_2 (g/mg min), and the adsorption capacities at time t and equilibrium e, are q_t (mg/g) and q_e (mg/g) respectively. ' q_m "indicates the isotherm's theoretical saturation capacity (mg/g), and ' K_L "stands for the Langmuir isotherm constant, which represents the adsorptive capacity (mg/g). R is the universal gas constant (J/molK), T is the absolute temperature (K), A_T denotes the Temkin isotherm equilibrium binding constant (L/mol), and b_T is the Temkin isotherm constant (J/mol).

3. Results discussion

3.1. Physiochemical characteristics of palm olein oil

Experimental analysis in Table 2 above, revealed that the acid value of used palm olein oil to be 8.40 mgKOH/g Oil. The acid value was found to have decreased to a value of 1.34 mgKOH/g Oil upon bleaching with the AARH from its initial value but remained outside the permitted Codex standard. Additionally, based on the low acid value, the findings indicated that the quality of the oil is good

Table 2. Physicochemical properties of used and bleached palm olein oils.

Properties	S_0	S ₁	S ₂
Acid value (mgKOH/g oil)	≤0.6	8.40	1.34
Free fatty acid (%)	≤3	4.23	1.02
Colour	Light	Brownish	Light
	Yellow	Yellow	brownish
			Vallou

S₀: Codex alimentarius standard for palm olein oil.¹⁶

and consumable.¹⁷ The oil sample bleached with AARH shows a decreased free fatty acid value of 1.02%, which is within the permitted level. This was no surprise as the acid value (free fatty acid) was targeted specifically in refining via neutralization to reduce its toxicity to humans.¹⁸ Also, the bleached oil was significantly lighter in colour than the unbleached oil. This suggests that during the bleaching process, some of the pigment was lost. Following the refining processes, the crude oil's dominant, rich odour became less appealing.

3.2. Scanning electron microscopy (SEM) analysis

The surface features of the AARH were slightly altered following the chemical treatment (KOH) as against the raw rice husk shown in Fig. 1 above. According to Kaur et al., 19 pretreatment using base and acid improved the morphology of rice husks by removing lignin and hemicellulose, reducing cellulose crystallinity, as well as removing the inorganic substances from the surface. The raw rice husk and AARH images in Fig. 1 demonstrate that the constituent particles exhibited nonuniform sizes and shapes. The structures in the micrographs of the raw rice husk are sparse, rather big, and have some tiny agglomerates on their surface. However, AARH's micrograph reveals a smaller, greater number of structures that are smoother, flatter but irregular distributed throughout their matrix, producing a surface area that is significantly greater and an increase in micropore volume.¹⁹

3.3. Surface area analysis of the adsorbent using BET

The Brunauer-Emmett-Teller (BET) characterization of raw rice husk and alkaline-activated rice husk was done to determine their surface area. From the results in Table 3 above, noticed that the BET total surface area of the AARH (127.210 m²/g) was 53.24% higher than that of the RRH (Raw Rice Husk) (59.520 m²/g). Do et al.,²⁰ reported similar findings. Suggesting that AARH could possess the strongest physical adsorption capacity. Similarly, the increase in the total pore volume of the

S₁: Used palm olein oil (unbleached).

S2: Palm olein oil bleached at optimum values using AARH.

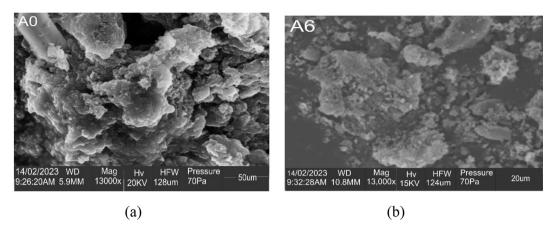


Fig. 1. Scanning electron micrographs of (a) raw rice husk, (b) 5 M KOH activated rice husk at 13 000 × magnification.

Table 3. Brunauer-Emmett-Teller total surface area and total pore volume of RRH and alkaline-activated rice husk.

Biomass	BET total surface area (m²/g)	Total pore volume (cm ³ /g)
RRH	59.520	0.006035
AARH	127.210	0.059330

adsorbent might also result from the activation process, activation temperature, and activation time. This is because the chemical agents or physical treatments can remove some impurities and volatile matter, causing an overall pore volume increase.²⁰

3.4. Fourier transform infrared (FT-IR) spectroscopy analysis of bleached and unbleached palm olein oils

The fourier transform infrared (FT-IR) were obtained for the used palm olein (crude) and bleached palm olein oil within the wavelength range of $350-4400 \,\mathrm{cm^{-1}}$ (350–1500 cm⁻¹ is known as the fingerprint region and 1600-4400 cm⁻¹ known as the functional group region). From Fig. 2a above, the used palm olein oil presented residue in a wavenumber 3781.82 cm⁻¹, this can be attributed to the carbon origin, which signifies the presence of high energy storage in the compound.²¹ The FT-IR analysis depictions (Fig. 2a and b) have slight changes between their spectra. The characteristic adsorption band for crude and bleached used palm olein oils at 3457.73 cm⁻¹ and 3457.00 cm⁻¹, respectively, signifies that O-H is present in the form of free carboxylic acids, confirming that both oils contain fatty acids.²¹ For peak 2934.47 cm⁻¹, Fig. 2a above is linked to the asymmetric stretching (C–H) of fatty acid hydrocarbon present in the oil, showing the existence of alkane functional group (-CH₃, -CH₂).²¹ Also, Fig. 2a shows absorption band of 2361.53 cm⁻¹, suggesting that the used palm olein oil

contained a sharp signal and weak intensity alkynes compound with a functional group of $-C \equiv C$ symmetric stretching. From Fig. 2a and b, broad signals and strong intensities absorptions at 1633.77 cm⁻¹ and 1632.42 cm⁻¹ suggested alkene compounds with functional group of -C=Cstretching, typical for esters carbonyl compounds.²¹ Similar ester carbonyl compound, -C=O stretching at a wavelength of 1712.70 cm⁻¹ was presented in Fig. 2b with sharp signal and strong intensity. It suggests that there are triglycerides in the oil. The wavenumbers at 1437.50 cm⁻¹ and 1445.78 cm⁻¹ in Fig. 2a and b respectively, shows the existence of hydrocarbon compounds (C-H) with a functional group of (CH₂, CH₃) asymmetric stretching. Absorption band of 1387.00 cm⁻¹ (Fig. 2b), suggest the existence of trimethyl or 'tert-butyl' (tertiary alcohol).²¹ The FT-IR spectra (Fig. 2a) shows the peaks at the finger print region (1276.00 cm⁻¹, 1114.33 cm⁻¹, and 1062.00 cm⁻¹), suggesting an ester group with an assigned functional compound (C-O, C-N) stretching. Also, peaks 663.57 cm^{-1} and 605.00 cm⁻¹) with an assigned alkane compound (C-H) bending out of plane. Similarly, for the bleached oil (Fig. 2b) the observed peaks at 1275.00 cm⁻¹, 600.00 cm⁻¹ and 370.17 cm⁻¹, which suggest the existence of skeletal -C-C- vibration, aromatic -C-H, and iron-oxide, Fe-O.²¹

3.5. Adsorption kinetic study

The linearized model equations in Table 1 above were used to analyze the experimental data.

As seen in Fig. 3a above, a plot of $\ln (A_0 - At)$ versus t was employed to show the pseudo-first-order at the temperature of $80\,^{\circ}\text{C}$; the slope and intercept were used to calculate A_0 and K_1 , respectively. From the results in Table 4, the adsorption

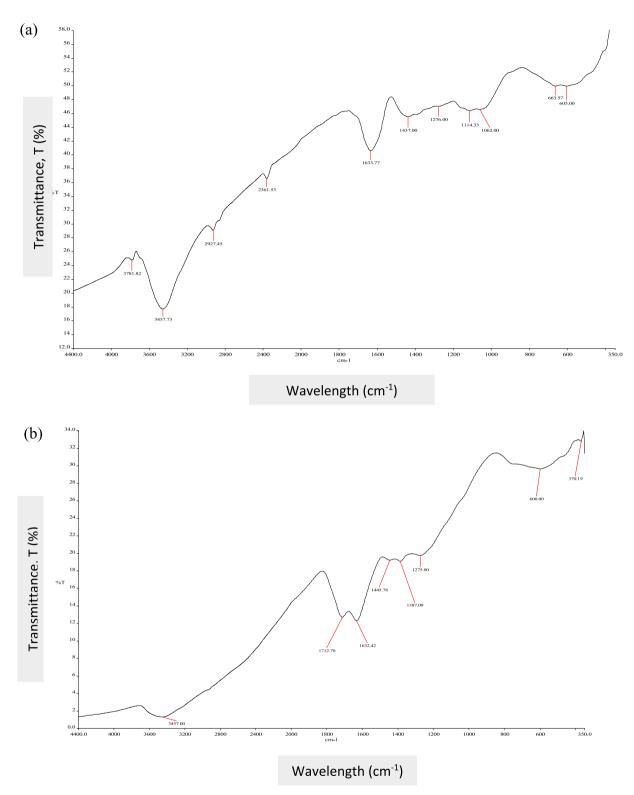


Fig. 2. a: FT-IR spectra of used palm olein oil. b: FT-IR spectra of bleached used palm olein oil.

experimental values do not fit into the pseudo-firstorder model, that is, is unable to describe the rate process as the regression coefficient R² value is ~0.1489.²² The pseudo-second-order rate constants K_2 and A_t for the adsorption at 80 °C were calculated from the plot of t/At versus t in Fig. 3b above. The adsorption experimental data (Table 4) fit into the pseudo-second-order model as the value of

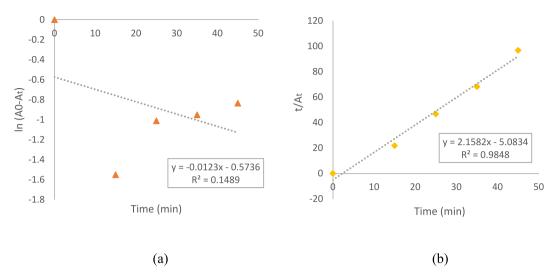


Fig. 3. Kinetic model plots of (a) Pseudo-first-order, and (b) Pseudo-second-order.

Table 4. Kinetic constants/parameters for the bleaching process.

Kinetic model	del Constant/parameters		
Pseudo-first-order	K ₁ (min ⁻¹)	$q_e \text{ (mg/g)}$	R ²
Pseudo-second-order	0.00027 $K_2 (g^{-1} mg min)$	0.5635 $q_e \text{ (mg/g)}$	0.1489 R^2
	0.9165	0.4633	0.9848

regression coefficient, R², is ~0.9848. Meaning that, the kinetic model was able to describe the rate process. With this high regression coefficient which is an indication that the rate-controlling step for the kinetic adsorption process is chemisorption. ^{14,22}

3.6. Equilibrium and isotherm study

To carry out the adsorption isotherm inquiry on alkaline-activated rice, we analyzed the equilibrium data by applying the Langmuir, Freundlich and Temkin models.

From Table 5 above at temperature 60 °C, the Freundlich plot gives n value of 0.6428 and 1/n value

Table 5. Adsorption isotherm parameters for the bleaching process.

Isotherm model	Parameter/constant	Temperature (°C)			
		60	70	80	90
Freundlich	K _F (mg/g)	0.0745	0.2505	0.8314	0.2550
	n	0.6428	2.1074	2.9373	2.2427
	1/n	1.5557	0.4745	0.3404	0.4459
	\mathbb{R}^2	0.5319	0.9908	0.1794	0.9857
Langmuir	$K_L (mg/g)$	1.0820	1.0683	1.0610	1.0294
	$q_m (mg/g)$	0.1500	0.0652	0.0760	0.0565
	R_L	0.5069	0.5101	0.5118	0.5194
	R^2	0.3120	0.9119	0.7849	0.8922
Temkin	$bT \times 10^3$ (J/mol)	15.9087	3.5054	3.4776	3.5712
	A _T (L/mol)	1.000	1.000	1.000	1.000
	\mathbb{R}^2	0.5395	0.9967	0.9650	0.9990

of 1.5557, indicating chemisorption and favorable or cooperative adsorption process, at temperature 70-90 °C, n values is between 2.1074 and 2.9373 and 1/n values between 0.3404 and 0.4745, indicating physisorption with a normal adsorption process. R^2 values of 0.5319, 0.9908, and 0.9857 corresponding to 60, 70, and 90 °C, respectively, demonstrate how well the Freundlich isotherm model fits the adsorption data. However, R² (0.1794) at temperature 80 °C shows the adsorption results cannot be explained by the Freundlich isotherm model. The K_f value increased from 60 to 80 °C (Table 5 above), which can be attributed to an increase in the rate at which contaminants and undesirable components diffuse into the pores of the adsorbent at higher temperatures. 24 Conversely, K_f values decrease as temperatures increase above 80 °C posing the possibility that a high temperature (90 °C) could be harmful to the adsorption sites. The used palm olein oil bleached with AARH has R_L values that are more than zero but less than one, suggesting a favorable Langmuir isotherm. The values of q_m (Table 5 above) is comparable to the value reported by,²⁵ where low values of q_m (<2) were also obtained after the calculation. Additionally, as the adsorption process's temperature increased, the values of the Langmuir constant (K_L) decreased. One possible explanation for this could be that the high temperature led to the destruction of some pore space on the adsorbent's surface. The Langmuir plot was rather accurate in depicting the adsorption processes at temperatures of 70, 80, and 90 °C, as shown by the plots and correlation coefficient (R²) values for the adsorption. For the Temkin isotherm, the Temkin constants A_T and b_T were calculated from the slope and intercept of the graph, which were obtained by plotting the values of x/m against Log X_e . The findings (Table 5) for this constant were positive, indicating that an exothermic reaction took place. Also, the fact that the value of the constant b_T is smaller than 16 kJ/mol suggests that physical adsorption is the mechanism taking place. In comparison to the other models that were used, the R^2 values (0.5395, 0.9967, 0.9650, and 0.9990) show that the relevant equation provides the best fit with the experimental data.

3.7. Conclusions

The bleaching of used palm olein oil was accomplished by using rice husk as an affordable, low-cost adsorbent. The scanning electron microscopy and BET analyses of the alkaline activated rice husk show morphological differences between the RRH and the AARH, and the BET study revealed an increase in the total surface area (127.210 m²/g) of the AARH. The existence of characteristic functional groups in the bleached oil was confirmed by FT-IR analysis. The experimental results for the adsorption process fit into the pseudo-second-order kinetic model, with chemisorption serving as the rate-controlling step, based on the coefficient of determination (R²) of 0.9848 obtained. The Temkin isotherm model suitably described the adsorption data, which revealed the highest values of R² (0.5395, 0.9967, 0.9650, and 0.9990) at temperatures of 60, 70, 80, and 90 °C, respectively.

This investigation has shown that AARH can be produced and used as a substitute for imported adsorbents for bleaching used palm olein oil at moderately higher temperatures.

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Author contributions

M.J.G.: conceptualization, formal analysis, investigation, methodology, writing-original draw, resources, writing-review and editing. A.O S.:

conceptualization, investigation, resources, validation, software, supervision, writing-review and editing. U.H.: resources, writing-review, validation and supervision. A.A.: resources, writing-review, validation and supervision. A.A.: resources, writing-review, validation and supervision.

Conflict of interest

There are no conflicts of interest.

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