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Bleaching of Palm Oil by Activated Local Bentonite and Kaolin Clay from Afashio, Edo-Nigeria

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Abstract
The bleaching potential of Afashio kaolin (AFK), from Edo state in Nigeria, for removal of pigments from palm oil was investigated in comparison with that of local bentonite (BN). Both AFK and BN were activated by acid treatment. Batch experiments were conducted to study the effects of adsorbent dosage (2 – 8 %), contact time (0 – 1.5 hr) and temperature (60 – 90^{o}C) on pigment adsorption. Four adsorption isotherms were used to model the equilibrium data for both adsorbents (AFK and BN), namely Langmuir, Freundlich, Sips and Redlich-Peterson models. The Sips model provides the best correlation and adsorption occurred in monolayer. Kinetic adsorption data were analyzed using the pseudo-first order, pseudo-second order, and intraparticle diffusion models. The regression results showed that the adsorption kinetics was more accurately represented by the pseudo-first order kinetic model. Intraparticle diffusion studies reveals that pore diffusion is the controlling mechanism. Thermodynamic parameters, ΔG, ΔH and ΔS, were calculated as a function of both temperature and adsorbent dosage. The values of enthalpy change were positive and greater than 40 kJ/mol in all cases, indicating that the bleaching process is endothermic and chemisorption in nature. The results suggest that AFK could be employed as low-cost alternative for removal of pigments from vegetable oil though BN is better.
Keywords: Afashio kaolin, bentonite, bleaching, equilibrium isotherms, kinetic models

1. Introduction

Palm oil in its raw form contains impurities such as organic pigments, oxidation metals, trace metals and trace soaps. For palm oil to be edible these impurities which negatively influence the taste and smell of the oil as well as its appearance and shelf life stability, thus reducing consumer acceptance and marketability have to be removed. Thus the refining of palm oil through adsorptive bleaching remains inevitable in the oil refining industry (Bockisch, 1997).

Bleaching remove pigments and other unwanted compounds which negatively influence the taste of the oil by bringing into contact with a surface-active substance that adsorbs the undesired particles. Bleaching of palm oil is done by the use of adsorbents; usually activated carbons and activated bleaching earths. These bleaching earths are mainly Aluminium silicates which are usually montmorillonitic clays that exhibit adsorptive properties in their natural and activated state (Oboh et al., 1987).

Natural clays are acquiring prominence as low-cost adsorbents over the last few decades due to their local and abundant availability and the capability to undergo modification to enhance the surface area, adsorption capacity, and range of applicability (Monvisade and Siriphannon, 2009). Therefore, in order to ameliorate the adsorption properties and range of applicability, a number of physical and chemical methods have been investigated to modify the clays which including heat treatment, acid activation, treating the cationic surfactants and polymer modification (Chaisena and Rangsriwatananoon, 2004; Al-Asheh et al., 2003; Motlagh et al., 2008; James et al., 2008).

Nigeria clays have been found competent in the adsorption bleaching of vegetable oil. In recent years researches have been carried out on clay activation and bleaching of palm oil using activated and non-activated Nigeria clay. Salawudeen et al. (2007) evaluated the effectiveness of clays from Oyo and Osun state with the view of comparing the adsorptive power to that found in other parts of Nigeria. Moreover, the study of activated bleaching power of some selected Nigerian clays found in Shagamu and Ewekoro both in Ogun state; Calabar in Cross River state and Nsukka, Enugu state were investigated by Oboh (1987) and reasonable percentage reduction around 80% was achieved. The bleaching performance of raw and acid activated Yola montmorillonite clay was studied by James et al. (2008). The best bleaching performance was obtained with clay sample activated with 5M H_2SO_4 at acid to clay ratio 0.7, bleaching temperature of 120 ^{o}C and at 10 minute stirring time. At these conditions the improved clay compares favourably well in terms of bleaching performance with the imported Fuller's earth.

Many other studies have reported successful bleaching of oil using acid activated clays from across the world (Christidis et al., 1997; Komadel et al., 1990; Rozic et al., 2010; Kirali and Lacin, 2006; Srasra et al., 1989;

Novakovic et al., 2008; Foletto et al., 2011; Azeez et al., 2011; Usman et al., 2012; Ajemba and Onukwuli, 2012).

To the best of our knowledge, the Afashio kaolin clay (AFK) in Edo State of Nigeria has remained unexplored for its bleaching potentials. In this study, therefore, AFK was activated by acid treatment and evaluated for the bleaching of palm oil in comparison with local bentonite (BN). The effect of such process parameters as contact time, adsorbent dosage and temperature on bleaching of palm oil using both AFK and BN were investigated.

2.0 Materials and methods

2.1 Materials

The local kaolin clay sample was obtained from Afashio, Etsako-West Local Government, Edo State, Nigeria while the local bentonite clay sample was got from FIIRO (Federal Institute of Industrial Research, Oshodi) in Lagos State, Nigeria. The crude palm oil was obtained locally from the Bariga, Lagos. Other materials used were hydrochloric acid, acetone (all analytical grades reagents) and distilled water.

2.2 Acid Activation

The clay samples obtained were sun-dried to make them amenable to grinding. They were then grinded with a local mortar and pestle, and sieved using a 150 µm mesh. 50 g of the clay samples were measured, and mixed with 250 mL of hydrochloric acid at a chosen concentration of 5 M to form slurry, and the slurry was heated at 105 ˚C for 30 minutes. After slow cooling in air at room temperature, the slurry was filtered and washed free of acid using distilled water as indicated by a pH meter. The clay was then dried at a temperature of 110˚C for 3 hours, ground using mortar and pestle, sieved to 150 µm, and stored.

2.3 Equilibrium adsorption studies

The bleaching vessels were four 250 ml conical flask which were immersed in a thermostatic shaker bath. The palm oil (20 cm^3) was heated to the desired temperature before adding the bleaching clay. The mixture continued to be heated and stirred for 1.5 hours at the desired temperature. The hot oil and clay mixture was filtered at the end of the experiment before measuring the absorbance. The concentration of the clay in the conical flasks was 2%, 4%, 6% and 8% by weight. The experiments were carried out at temperatures of 60, 70, 80 and 90 ^oC.

2.4 Adsorption Kinetics Experiment

For the adsorption kinetics experiment, 20cm^3 of crude palm oil was placed in four 250ml conical flask held in a thermostatic shaker bath to reach the desired temperature before adding 0.8g of the clay samples (using an adsorbent oil ratio of 4%) which was monitored for 1.5 hours. The samples as contained in the four conical flasks were taken out from the bath at a respective time of 15, 30, 60 and 90 min, and filtered to measure the absorbance. The experiments were carried out at temperatures of 60, 70, 80 and 90 ^oC.

2.5 Evaluation of the Bleaching Performance

At the end of each bleaching process, the content of the flask were filtered through a Whatman No.1 filter paper and the concentration in pigment of the filtrate was determined. The evaluation of the amount of pigment was made by UV visible spectroscopy. The samples were diluted in acetone to a concentration of 10% (v/v), and the absorbances of the samples were determined at a wavelength of 450 nm using the UV spectrophotometer.

To measure the absorbance of the unbleached palm oil, A_o, 1 cm^3 of the unbleached palm oil was diluted in acetone to a concentration of 10% (v/v), and its absorbance was determined at a wavelength of 450nm using the UV spectrophotometer.

The bleaching performance of the adsorbent was determined from the following equation (1):

$$BleachingPerformance = 100(A_o - A)/A_o \quad (1)$$

Where A_o and A are the absorbance of the crude and bleached palm oil respectively

3.0 Results and discussion

3.1 Adsorption Kinetics

3.1.1 Effect of Contact Time

The kinetic studies were carried out at 60, 70, 80, and 90 ^oC with an adsorbent oil ratio of 4%. The adsorbents used for kinetic studies were AFK and BN activated with 5M hydrochloric acid solution as reported in Figure 1 and Figure 2. It is observed that the bleaching efficiencies of the adsorbents vary with the temperature for all the bleaching efficiency curves i.e. the bleaching efficiency increases with the increase in temperature for all the adsorbent used herein. For the bleaching of the palm oil, both activated clays present similar evolution of the bleaching capacities versus time. The bleaching of oil is fast in the first 30 mins and progresses much more slowly thereafter. And towards approaching a contact time of 90 min (1.5h), the bleaching efficiency begins to stabilize or become steady as the medium approaches equilibrium.

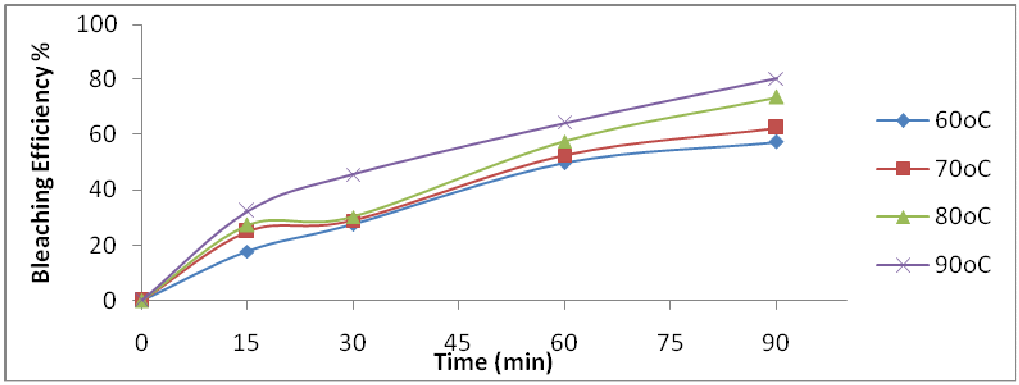

Figure 1: Kinetics of bleaching palm oil at different temperatures by BN

From Figure 1, unlike the bleaching efficiency curve of 60 ^{o}C which attained an optimum value after 60 mins, it can be deduced that a bleaching efficiency greater than 81.4% can still be obtained for the bleaching efficiency curve of 90 ^{o}C after a total contact time of 90 mins i.e. with an increase in the contact time, the optimum bleaching efficiency can be attained while that of local kaolin clay, likewise, as seen in Figure 2, gives a bleaching efficiency of 62.4% at a temperature of 90 ^{o}C and contact time of 90 min.

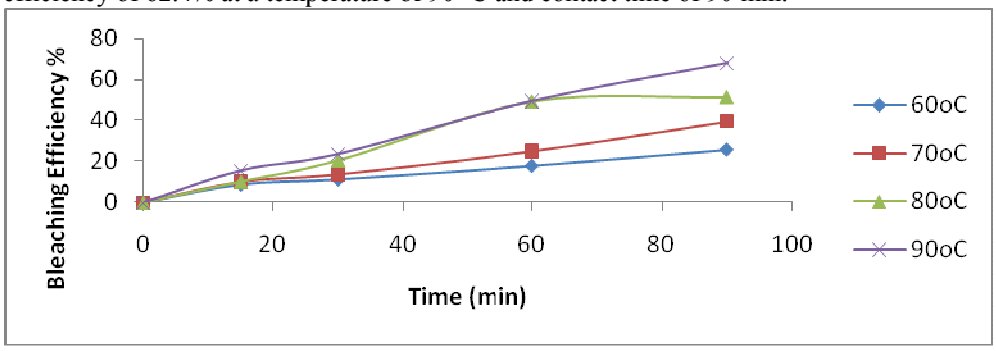

Figure 2: Kinetics of bleaching palm oil at different temperatures by AFK

3.1.2 Effect of variation in adsorbent dosage

Figure 3 and 4 represents the plot of bleaching efficiency versus adsorbent dosage. The clay dosage was varied from an adsorbent oil ratio of 2% to 8%.It was observed that increasing the clay dosage increased the bleaching efficiency for both AFK and BN; however, the results clearly indicate that the bleaching efficiency increases to an optimum value from which further increase in the adsorbent dosage has no significant effect on them. The attainment of a steady bleaching efficiency beyond the optimum as particular to both local clays can be explained by the fact that adsorption equilibrium has been reached between the adsorbent/oil mixtures, thereby, preventing further pigment removal by the excess adsorbent dosage.

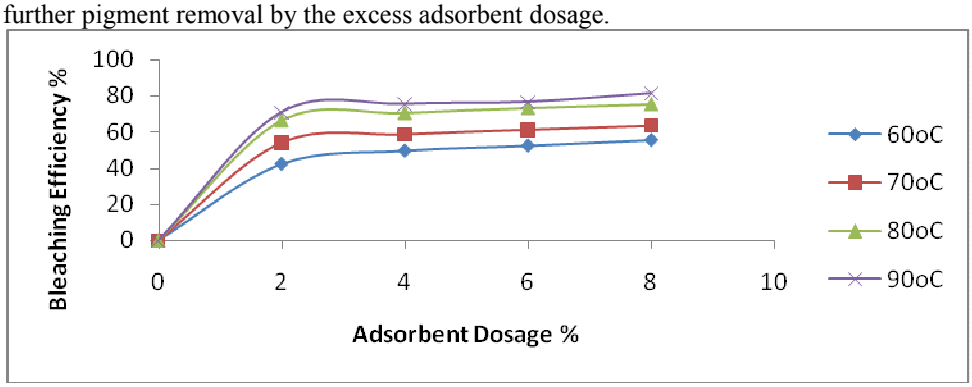

Figure 3: Variation of the bleaching efficiency of the adsorbent as a function of the adsorbent used for palm oil bleaching for BN

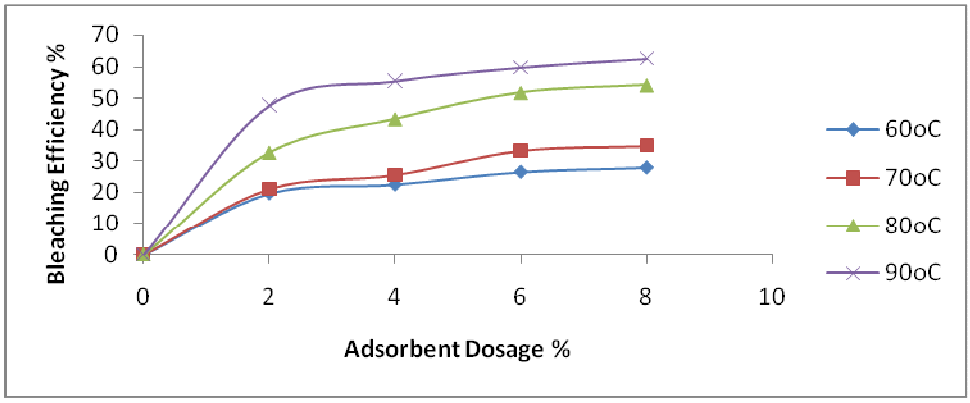

Figure 4: Variation of the bleaching efficiency of the adsorbent as a function of the adsorbent used for the bleaching of palm oil for AFK

3.2 Adsorption Isotherm

Adsorption isotherm describes the equilibrium of the sorption of the palm oil impurities at constant temperature. To evaluate the nature of adsorption, the experimental isotherm data were fitted by model isotherm equations namely; the Langmuir (1906) equations (2, 3), Freundlich (1918) equations (4, 5), Sips (Sips, 1948) equations (6, 7) and Redlich–Peterson (Redlich and Peterson, 1959) equations (8, 9) in terms of absorbance (X) and concentration (C) respectively (Foo and Hameed, 2010; Kumar and Porkodi, 2006; Toor and Jin, 2012).

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

Langmuir isotherm $\frac{C_e}{q_e}$

$$= \frac{1}{k_l q_l} + \frac{C_e}{q_l} \quad (3)$$

$$\log\frac{X}{m} = \log K + \frac{1}{n}\log X_e \quad (4)$$

Freundlich isotherm $\log q_e$

$$= \log k_f + \frac{1}{n}\log C_e \quad (5)$$

$$\frac{X_e^{1/m}}{q_e} = \frac{1}{b.a} + \frac{X_e^{1/m}}{a} \quad (6)$$

Sips isotherm $\frac{C_e^{1/m}}{q_e}$

$$= \frac{1}{k_s q_s} + \frac{C_e^{1/m}}{q_s} \quad (7)$$

$$\frac{X_e}{X/m} = \frac{1}{a.b} + \frac{X_e^{\alpha}}{a} \quad (8)$$

$$\text{Redlich–Petersonisotherm}\frac{C_e}{q_e} = \frac{1}{k_l q_l} + \frac{C_e^{\alpha}}{q_l} \quad (9)$$

Where X/m = q_e, and $X_e = C_e$

X is amount of adsorbed solute, Xe is amount of unadsorbed solute, m is grams of adsorbent, a and b are Langmuir isotherm constant and Freundlich isotherm constant are n and K. Figures 5, 6, 7 and 8 shows the Freundlich, Langmuir, Sips, Redlich-Peterson isotherm respectively for the bleaching of palm oil with the activated clays. The isotherm model parameters and the statistical fits of the adsorption data to these models as calculated are presented in Table 1. It was observed that Sips model adequately described the adsorption data with best linear fit and greater regression coefficient values for the AFK and BN.

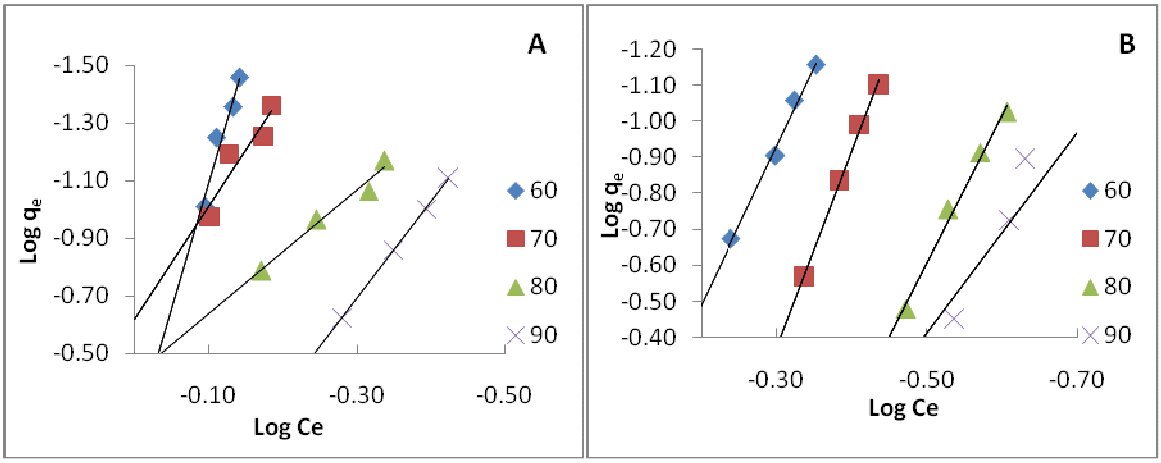

Figure 5: Freundlich isotherm for the bleaching of palm oil with [A] AFK and [B] BN at various temperatures

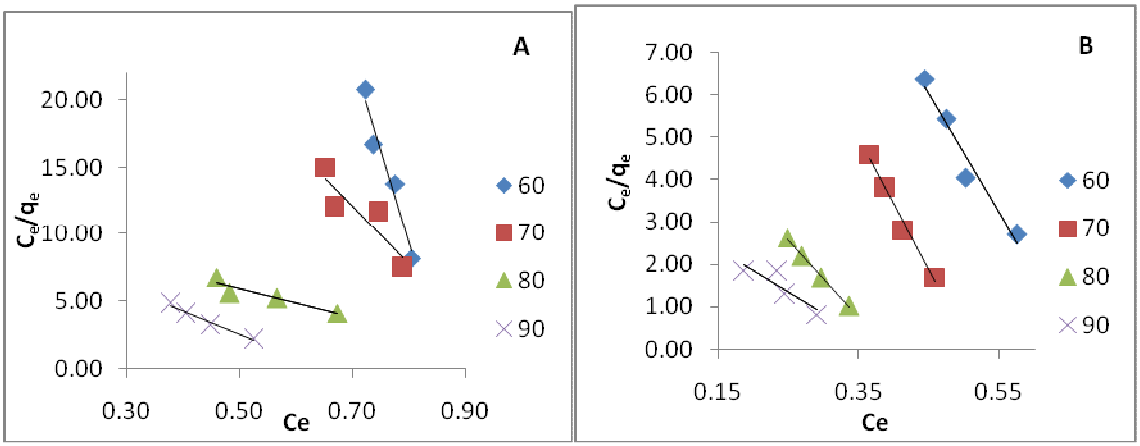

Figure 6: Langmuir isotherm for the bleaching of palm oil with [A] AFK and [B] BN at various temperatures

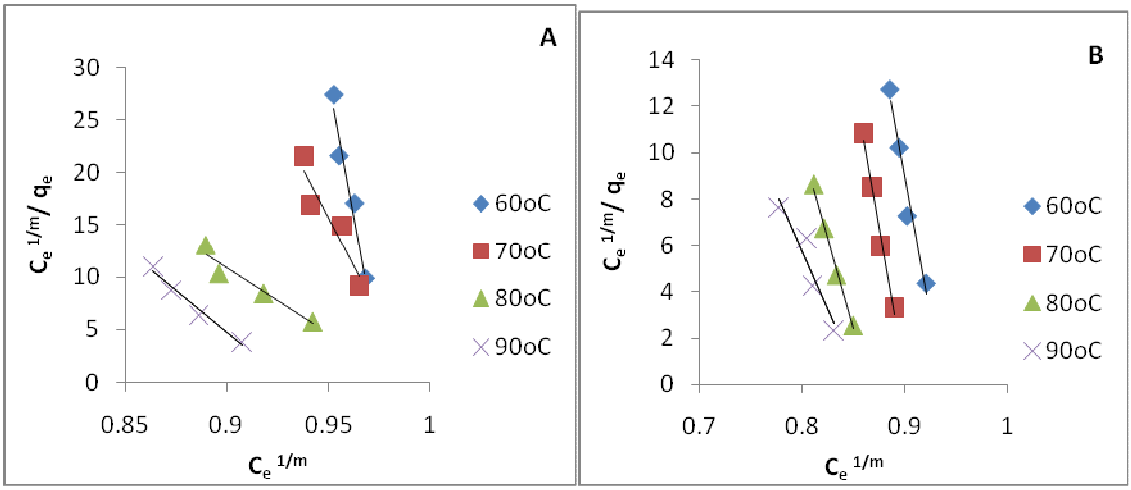

Figure 7: Sips isotherm for the bleaching of palm oil with [A] AFK and [B] BN at various temperatures

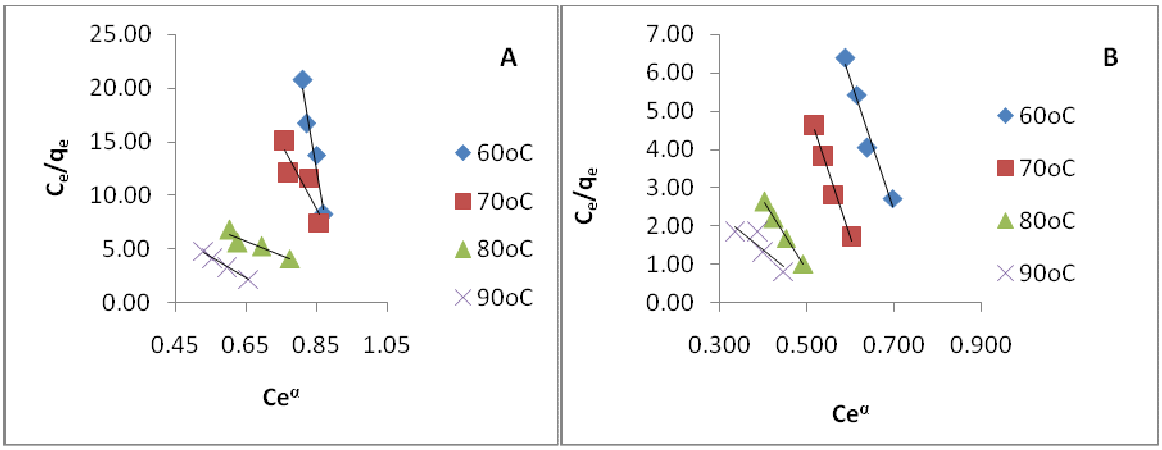

Figure 8: Redlich - Peterson isotherm for the bleaching of palm oil with [A] AFK and [B] BN at various temperatures

Table 1: Langmuir, Freundlich and Sips isotherm constants for the bleaching of palm oil with AFK and BN

		AFK				**BN**			
Isotherm Models	Parameter	Temperature(°C)				Temperature(°C)			
		60°C	70°C	80°C	90°C	60°C	70°C	80°C	90°C
Freundlich	K	0.6079	0.2396	0.379	2.063	2.3878	18.638	27.759	9.5719
	1/n	0.1143	0.2567	0.4671	0.2988	0.2289	0.1832	0.2436	0.3590
	R^2	0.9399	0.8714	0.9749	0.9999	0.9924	0.9953	0.9912	0.8684
Langmuir	a	-0.007	-0.023	-0.094	-0.058	-0.036	-0.032	-0.055	-0.099
	b	-1.153	-1.021	-0.947	-1.538	-1.501	-1.961	-2.555	-2.615
	R^2	0.9491	0.8057	0.8788	0.9751	0.9554	0.9887	0.9967	0.7766
Sips	$a*10^{-3}$	-9.8	-2.66	-7.9	-6.23	-4.2	-3.98	-6.43	-9.97
	b	-1.023	-1.008	-1.014	-1.076	-1.067	-1.109	-1.156	-1.167
	R^2	0.9558	0.8718	0.9329	0.9745	0.9620	0.9854	0.9937	0.9098
Redlich-Peterson	a	-0.005	-.0169	-0.075	-0.050	-0.030	-0.028	-0.055	-0.11
	b	-1.095	-1.003	-0.928	-1.312	-1.294	-1.543	-1.825	-1.819
	R^2	0.9488	0.8035	0.8825	0.9798	0.9598	0.9909	0.9983	0.7614

3.3 Adsorption Thermodynamics

Thermodynamic behavior of the adsorption process was evaluated by the following thermodynamic parameters including the change in free energy (ΔG), enthalpy (ΔH), and entropy (ΔS).These parameters were obtained using the following equations:

$$\ln k_d = \frac{\Delta S}{R} - \frac{\Delta H}{RT} \quad (10)$$

$$\Delta G = = RT \ln(k_d) \quad (11)$$

$$k_d = \frac{X}{X_e} \quad (12)$$

where R is the universal gas constant (8.314 (J/mole) K), T is temperature(K), and K_d is the distribution coefficient for the adsorption

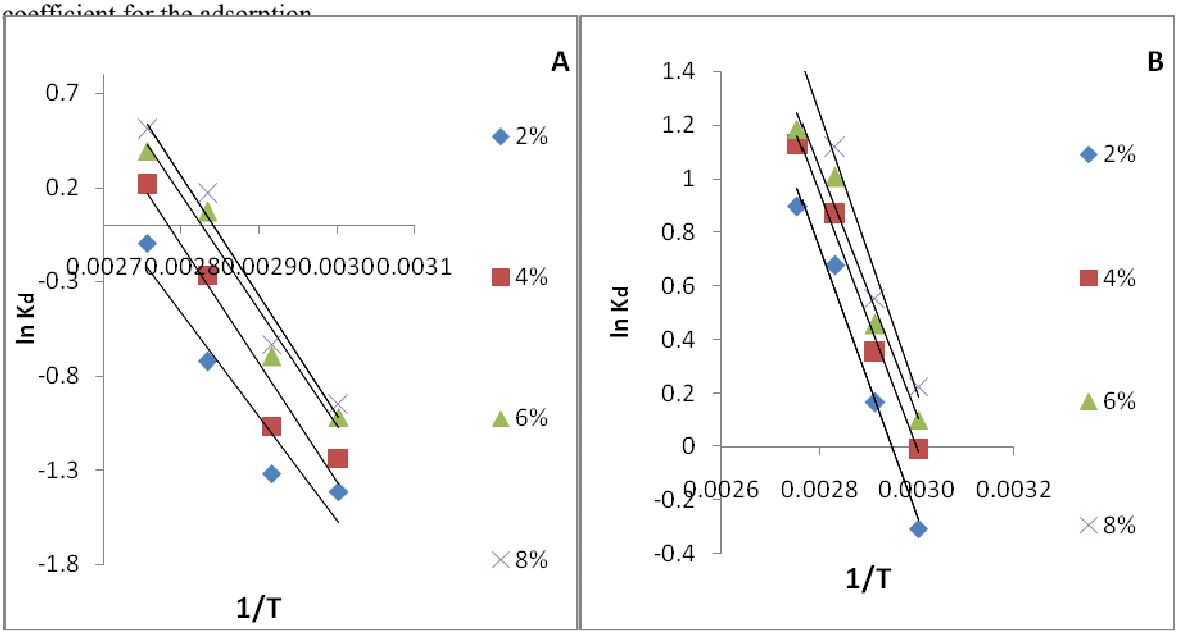

Table 2: Thermodynamic behavior parameter of the adsorption process for the bleaching of palm oil with AFK and BN

	AFK				**BN**			
	Adsorbent Dosage				Adsorbent Dosage			
Parameter	2%	4%	6%	8%	2%	4%	6%	8%
ΔH	45.3271	51.54846	50.28307	52.01155	41.50515	39.50647	39.53253	43.3201
ΔS	0.1230	0.1434	0.1420	0.1477	0.1224	0.1185	0.1160	0.1316
R^2	0.9129	0.943	0.9689	0.9675	0.9814	0.9882	0.9731	0.9882

		AFK				**BN**			
Thermo-dynamic	Parameter	Temperature(°C)				Temperature(°C)			
		60°C	70°C	80°C	90°C	60°C	70°C	80°C	90°C
ΔG	2%	848.2	-468.7	-1986	-2702.3	3907.8	3761.1	2118.3	289.9
	4%	33.2	-1002.6	-2542.8	-3396.6	3424.1	3042.4	779.3	-642.2
	6%	-277.1	-1299.6	-2948.9	-3578.9	2824.32	1980.93	-211.4	-1186
	8%	-622.8	-1579.1	-3271.4	-4455.2	2642.33	1803	-482.4	-1528

In this study, the positive ΔHis an indicator of endothermic nature of the adsorption and also its magnitude gives information on the type of adsorption, which can be either physical or chemical. The enthalpy of adsorption, ranging from 40 to 400 kJ/mole corresponds to chemisorption (activated adsorption). Chemisorption occurs

when the intermolecular attractive forces between molecules of the adsorbate itself are greater than those between molecules of adsorbate and adsorbent. In this study, the adsorption heat of the pigments is in range of chemisorption. Therefore, the ΔH values show that the adsorption of pigments on the local clays takes place via chemisorption. The positive range of ΔS value suggests an increase in the randomness at adsorbate-solution interface during the adsorption process. Also the decrease in ΔG values with increasing temperature shows a decrease in feasibility of adsorption at higher temperatures.

3.4 Adsorption kinetics models

The effect of contact time on the bleaching efficiency of AFK and BN activated with 5M hydrochloric acid as reflected in Figure 1 and 2, were tested with three kinetic models in order to examine the controlling mechanism of the adsorption process which are Pseudo-first order kinetic (Lagergren, 1898), pseudo-second order kinetic (Ho and McKay, 1999) and intraparticle diffusion model (Webber Jr and Morriss, 1963).

The respective differential and linear forms of the equations are as given below:

$$\frac{dq_t}{dt} = k_1(q_e - q_t) \qquad (13)$$

$$\log(q_e - q_t) = \log q_e - \frac{k_1 t}{2.303} \qquad Pseudo-first\ order\ Model \qquad (14)$$

$$\frac{dq_t}{dt} = k_2(q_e - q_t)^2 \qquad (15)$$

$$\frac{t}{q_t} = \frac{1}{k_2 {q_e}^2} + \frac{1}{q_e} t \qquad Pseudo-second\ order\ Model \qquad (16)$$

$$q_t = K_3 t^{1/2} + C \qquad \text{Intraparticle diffusion model} \qquad (17)$$

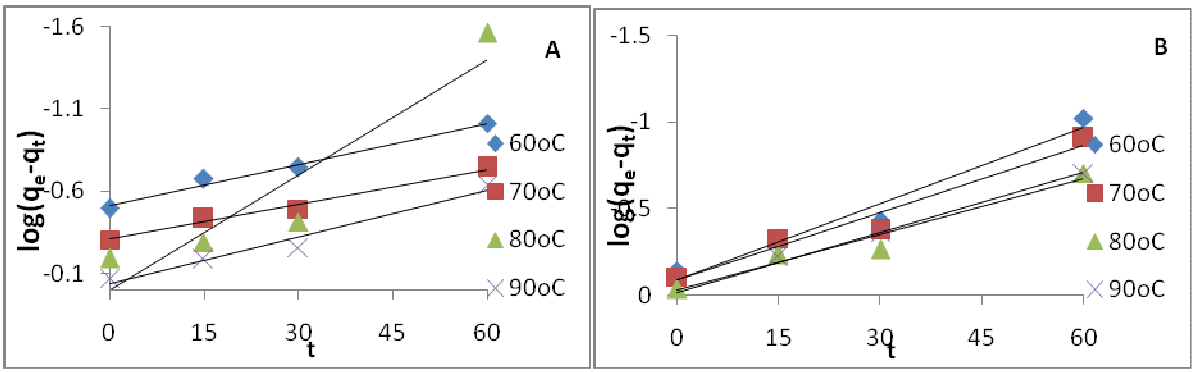

Figure 8: Pseudo-first order kinetics for the adsorption of palm oil pigments onto [A] AFK and [B] BN

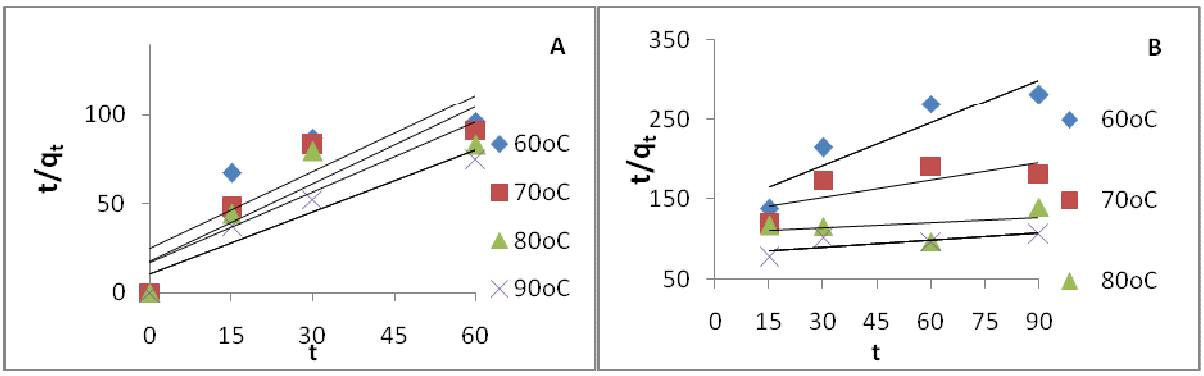

Figure 9: Pseudo-second order kinetics for the adsorption of palm oil pigments onto [A] AFK and [B] BN

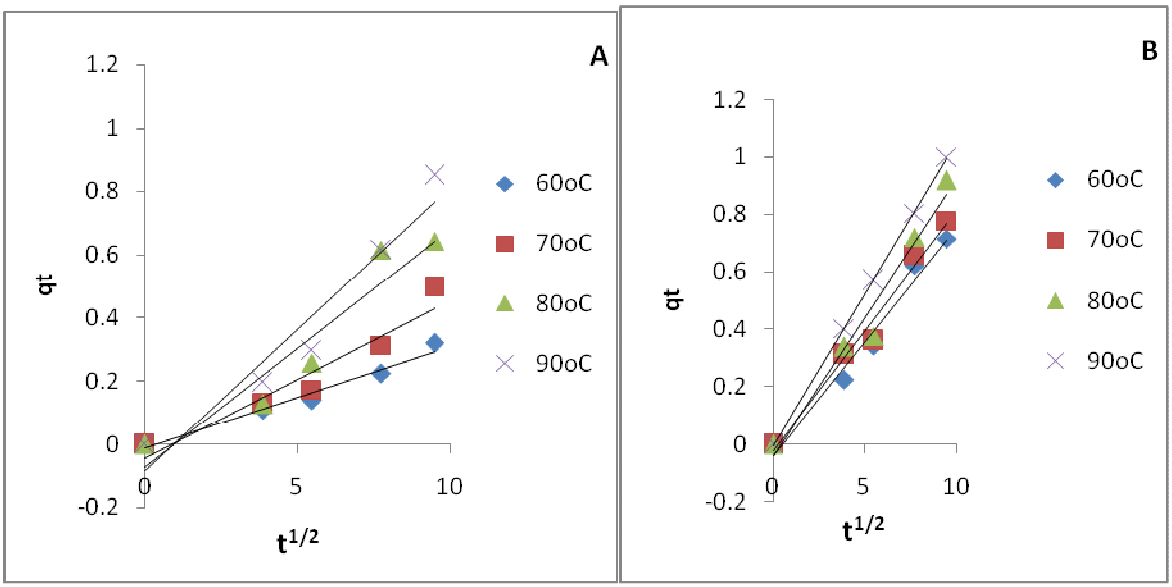

Figure 9: Intraparticle diffusion for the adsorption of palm oil pigments onto [A] AFK and [B] BN

Table 3: The kinetic parameters evaluated for palm oil pigment adsorption onto local clays

		AFK				BN			
Isotherm Models	Parameter	Temperature(^{o}C)				Temperature(^{o}C)			
		60^{o}C	70^{o}C	80^{o}C	90^{o}C	60^{o}C	70^{o}C	80^{o}C	90^{o}C
Pseudo-First-Order	$q_{e\,(exp)}$	0.32	0.495	0.6425	0.8488	0.7163	0.7788	0.9175	1.0013
	$q_{e\,(calc)}$	0.3064	0.4937	0.9112	0.997	0.8111	0.8089	0.9322	0.9521
	k_1	0.0191	0.0164	0.0216	0.053	0.0339	0.0297	0.0265	0.0246
	R^2	0.9851	0.9873	0.9653	0.8787	0.9636	0.9592	0.9946	0.9563
Pseudo-Second-Order	$q_{e\,(exp)}$	0.32	0.495	0.6425	0.8488	0.7163	0.7788	0.9175	1.0013
	$q_{e\,(calc)}$	0.6961	0.6877	0.7531	0.8564	0.6961	0.6877	0.7531	0.8564
	k_2	0.0822	0.1209	0.1045	0.1304	0.0822	0.1209	0.1045	0.1304
	R^2	0.7122	0.8009	0.771	0.9058	0.7122	0.8003	0.771	0.9058
Intraparticle Diffusion	$q_{e\,(exp)}$	0.32	0.495	0.6425	0.8488	0.7163	0.7788	0.9175	1.0013
	C	-0.0142	-0.0708	-0.0432	-0.0846	-0.0383	-0.0387	-0.0189	-0.0026
	K_3	0.0324	0.0751	0.0498	0.0896	0.0788	0.0959	0.0828	0.105
	R^2	0.9712	0.9063	0.9248	0.9333	0.9755	0.9689	0.9812	0.9998

The associated kinetic parameters have been evaluated from the slopes and intercepts of the respective linear plots of the pseudo-first order, pseudo-second order kinetic equation and intraparticle diffusion with the values as shown in Table 3. Comparison of the analyzed data based on the linear regression coefficient (R^2) values, showed that the experimental data is best described by the pseudo-first order kinetic model for both adsorbents. Intraparticle diffusion study show straight line passing through the origin with high correlation coefficient for both adsorbents. Therefore the intraparticle diffusion is the dominating mechanism for the adsorption process.

4.0 Conclusion

The kinetic and equilibrium studies of the adsorptive purification of palm oil using hydrochloric acid activated AFK and BN has been successfully investigated. The adsorption of pigments onto the clay surfaces increased with temperature, adsorbent dosage and contact time.

Langmuir, Freundlich,Sips and Redlich–Peterson adsorption models were applied to describe the experimental equilibrium isotherms. The Sips model agreed well with the equilibrium adsoption data with R^2 range of 0.9063 to 0.9998, while the Langmuir, Freundlich, and Redlich–Peterson equation gave a slightly poorer fit.The heat evolved during adsorption was recorded as 39.52 to 43.32 kJmol^{-1} and 45.33 to 51.55 kJmol^{-1} for the local activated bentonite and kaolin clay respectively. These values are greater than 40 kJmol^{-1} which is an indication of activated adsorption (chemisorption) between the adsorbate and adsorbent. The kinetic study performed based on pseudo-first-order, pseudo-second-order and intraparticle diffusion indicates that the pseudo-first order kinetic model better represents the bleaching process while intraparticle diffusion is the dominating mechanism for the adsorption process.

The thermodynamic parameters, namely ΔH, ΔS and ΔG showed that adsorption of pigments onto the local activated bentonite and kaolin clay respectively under the examined conditions was spontaneous and endothermic.

The optimum bleaching condition was equivalent to a bleaching efficiency of 81.4% and 62.4% for the local activated bentonite and kaolin clay respectively, consequently indicating that the clays have potential as bleaching earth/adsorbent under this condition. We infer that Afashio clay from Edo state in Nigeria can be converted into potential adsorbents for bleaching of palm oil, which can represent economy added value.

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