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Dynamics of M^{x+} Salts of Fatty Acids Adsorption onto Metallic Ores

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***Abstract*—Iron ore as possible adsorbent for metallic salts of fatty acid (M^{x+}-SFA) oils was investigated. Experimental constants from several kinetic models were used to interpret M^{x+}-SFA uptake. Mode of diffusion was also studied. Applicability tests for adopted models favors the Pseudo second order kinetics which presented high R^2 values > of 0.9; high precision or least qcal/qexp values of ratio within 1.0 for the K^+-SFA, Ca^{2+}-SFA and Al^{3+}-SFA sorption. The Pseudo second order equation also gave least values for the three error functions viz: 9.6×10^{-4} to 5.4×10^{-3}, 3.1×10^{-2} to 7.4×10^{-2} and 4.6×10^{-3} to $2.5\ \times10^{-3}$ for EABS, SSE and X^2 respectively. Multiple linearity presented by intra-particle diffusion plots is of three distinct stages of linear initial, linear intermediate and linear last portions. These stages are coupled with the non-passage of lines through the origin being an indication that other diffusion models such as mass transfer or film diffusion exists and that the intra-particle diffusion is not the only rate factor controlling M^{x+}-SFA sorption.**

***Index Terms*—Surfactants, ores, kinetics, diffusion, metallic soap, adsorption.**

I. INTRODUCTION

In recent years, management of pollution has been a universal challenge [1]-[5], especially in the developing countries. There are several technologies for the treatment of environmental pollutants. The technologies can be divided into biological, chemical and physical categories [6]. Among these methods, remediation via adsorption is widely used for its simplicity. It is sludge free [7]. The nature of the adsorbate and its surface functional or substituent groups greatly influenced the surface chemistry of which adsorption is listed. In other words, the presence and concentration of surface functional groups also play an important role in the adsorption phenomenon.

The most effective and known adsorbent in the adsorption process is activated carbon, mostly of plant origin. Activated carbon has become the adsorbent of choice for toxic metal and colour pollutants due to its high surface area and porosity that culminate into high adsorption capacity and intensities for these adsorbates [8]-[10].

Application of activated carbon is limited as a result of high production cost and minimal rate of decomposition which may pose environmental threats. Therefore, alternative low-cost adsorbents from substrates such as chitin, coffee, tea waste, orange peel, rice husk, bark, coir pith and metallic ores such as mixed iron ore (MIO) have been studied [11]-[19].

In principle, mineral flotation is an interfacial phenomenon brought about fundamentally by the selective adsorption of a surfactant onto a mineral surface; this term has been established by researchers who studied the adsorption of surfactants onto some mineral surfaces in aqueous medium as action that precedes selective flotation of the minerals from their ores. Factors like concentration, pH, temperature and the presence of inorganic ions in the ore pulp have been identified to influence adsorption process. Flotation technique is often employed in the separation of adsorbates or solids of similar densities which cannot be separated by gravity [20]-[23].

Shea butter oil botanically called *Butyrospermum parkii* is in solid form, a soft gelatin-like paste of melted fat with milky colour. It is brownish when melted and usually processed from *Vitellaria paradoxa* nuts/seeds. As an important source of fat, *Butyrospermum parkii* has a characteristic odour. It contains fatty acid triglyceride and a high amount of unsaponifiable matter, which ranges from 2.5 to 15% [24]-[26].

Adsorption kinetics or rate studies are of great significance to evaluate the performance of a given adsorbent and gives insight into the underlying mechanisms. When adsorption dynamics is concerned, transport or diffusion mode, thermodynamic and kinetic aspects should be involved. Generally, kinetic analysis unveil the solute uptake rate, which explain the residence time required for completion of adsorption reaction. In other words, adsorption kinetics is critical in the determination of the performance of fixed-bed in any flow-through or column systems [27].

The present work was to explore the possibility of finding adsorbent potentials of readily available and economically viable adsorbent; mixed iron ore (MIO). Its adsorption abilities upon interaction with M^{x+}-SFA were studied as a function of initial concentration and time. Batch mode adsorption process was adopted and reports of the applicability of various kinetic models and mechanism of transports was presented.

II. MATERIALS AND METHODS

A. Adsorbent-Mix Iron Ores

MIO randomly was procured from within Lokoja-Okene-Kabba triangle of Kogi state, Nigeria. Samples were pretreated as described elsewhere [28]. Solutions of reagents were prepared with distilled water. 0.1M NaOH and HCl

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solutions were used for pH adjustments. All Chemical reagents of analar grade were used as received except for their dilutions in preparations of solutions.

B. Metallic Soap (Adsorbate) Preparation

Metallic Surfactants, viz; Al^{3+}, Ca^{2+} and K^+-soaps of Shea butter oil were prepared by carefully following the method described elsewhere [29] with slight modification. 50 g of Shea butter oil was added to each 50ml of 30% potassium hydroxide, calcium hydroxide and aluminum hydroxide. 30 ml of ethanol was added into each of the 60ml beakers, heated at almost 90^0C for about 1 hour 10 minutes in a water bath with vigorous stirring until creamy pasty soaps are formed. About 50 ml hot saturated sodium chloride solution was then added to each of the pasty soaps with continuous stirring for 10 to 15 minutes and allowed to cool overnight. The soap cakes formed on the surface of the lye were removed, dried and stored in plastic containers. Working standard soap solutions was prepared in mg/L following standard procedures of serial dilutions.

C. Batch Mode Adsorption Test

The batch mode interaction for adsorbate/adsorbent labeled as Al^{3+} soap/MIO, Ca^{2+} soap/MIO and K^+ soap/MIO was set up. Respective formulations were obtained from collecting 0.2g of MIO into a 100cm^3 Erlenmeyer flask. 25 cm^3 of each aqueous soap solutions was added. The reaction was enhanced using a mechanical shaker at 120 oscillations per minute followed by centrifuge at 150 rpm. After filtration of the decanted samples, the filtrate was measured for equilibrium phase concentration (C_e) using the already designed calibration curve. Amount of surfactant adsorbed onto a unit mass of the adsorbent (q_e) in mg/g was calculated as [30]

$$q_e = (C_o - C_t)\frac{v}{w} \tag{1}$$

C_o and C_e stands for aqueous phase and equilibrium phase soap concentration in mg/L respectively. v is the volume of soap solution (L) while w is the mass of adsorbent in gram.

III. RESULTS AND DISCUSSION

A. Kinetic Equilibrium Study

Equilibrium was considered to be reached when at least two concentration measurements were constant [31]. Linear kinetic models and their respective plots are summarized in Table IV.

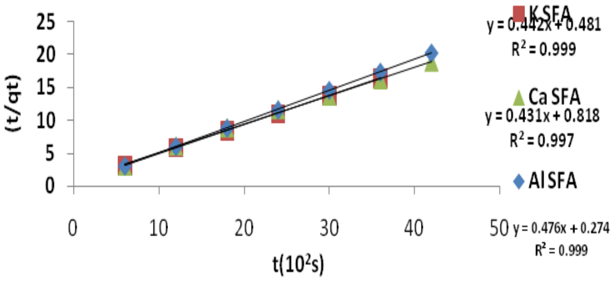

Fig. 1. Pseudo second order kinetic plots for M^+ salts of fatty acid Shea butter oil uptake onto MIO.

Fig. 1 is an insight into Sorption rate or kinetic model plot. Best fit model in this study was based on the chemisorptions kinetic rate equation simply expressed as pseudo second order kinetics. The plot is typical of experimental data presented for pseudo second order kinetics in Table I, wherein adsorption rate of K_2= 0.43, 0.25 and 0.85 was reported for K^+-SFA**,** Ca^{2+}-SFA and Al^{3+}-SFA sorption onto MIO. Second order rate constant, K_2 for Al^{3+}-SFA is high compared to those of K^+ and Ca^{2+}. This however does not justify ionic charge dependency of the speed at which cationic SFA is sorbed onto MIO. Value for Ca^{2+}-SFA is least (0.25). Rate constants for the other models falls within the range obtained for the pseudo second order kinetics. They include 0.03 -0.17, 0.02-0.17, 0.02-0.17, 1.70-1.82, 0.007-0.03 and 0.02-0.021 for k_1, k_m, k_{BV}, k_{FP}, k_{NK} and k respectively.

TABLE I: EXPERIMENTAL CONSTANTS FOR ADSORPTION OF M^{x+}-SFA ONTO METALLIC ORES

Kinetic model	**M^{x+}-SFA**	**R^2**	**constants**		
			$K(10^{-2})$	**q_{cal}**	**q_{exp}**
Linear 1st order	K^+-SFA	0.85	1.84	-	-
	Ca^{2+}-SFA	0.91	2.07	-	-
	Al^{3+}-SFA	0.81	1.84	-	-
			K_1		
Pseudo 1st order	K^+-SFA	0.98	0.17	6.48	2.19
	Ca^{2+}-SFA	0.87	0.03	13.96	2.23
	Al^{3+}-SFA	0.84	0.13	17.99	2.07
			K_m		
Modified pseudo 1st order	K^+-SFA	0.97	0.17	2.10	2.19
	Ca^{2+}-SFA	0.87	0.03	4.70	2.23
	Al^{3+}-SFA	0.838	0.02	4.48	2.07
			$K_{NK}(10^{-2})$		
N & K	K^+-SFA	0.85	1.84	-	-
	Ca^{2+}-SFA	0.91	2.07	-	-
	Al^{3+}-SFA	0.81	0.69	-	-
			K_2		
Pseudo second order	K^+-SFA	0.99	0.43	2.26	2.19
	Ca^{2+}-SFA	0.99	0.25	2.30	2.23

	Al^{3+}-SFA	0.99	0.85	2.10	2.07
			K_{BV}		
B & V	K^{+}-SFA	0.97	0.17	-	-
	Ca^{2+}- SFA	0.87	0.03	-	-
	Al^{3+}- SFA	0.83	0.02	-	-
			K_{Fp}		v
Fractional Power	K^{+}-SFA	0.95	1.70	-	0.07
	Ca^{2+}- SFA	0.80	1.70	-	0.07
	Al^{3+}- SFA	0.94	1.82	-	0.04
			B	Ln(αβ)	
Elovich	K^{+}-SFA	0.96	6.62	11.07	-
	Ca^{2+}- SFA	0.79	7.09	11.08	-
	Al^{3+}- SFA	0.94	13.70	24.80	-

Keys: N&K- Natarajan and Khalaf; B & V- Bhattacharya and Venkobachar

TABLE II: ERROR FUNCTIONS FOR SORPTION OF M^{x+}-SFA ONTO METALLIC ORES

Kinetic models	**Interaction**	**q_e, cal**	**q_e, exp**	**Error Functions**		
				SSE	**EABS**	**X^2**
Pseudo first order	K^{+}-SFA/MIO	6.48	2.19	18.44	4.30	8.43
	Ca^{2+}-SFA/MIO	13.96	2.23	137.71	11.74	61.81
	Al^{3+}-SFA/MIO	17.99	2.07	253.38	15.92	122.41
Modified Pseudo first order	K^{+}-SFA/MIO	2.10	2.19	0.01	0.09	0.00
	Ca^{2+}-SFA/MIO	4.70	2.23	6.13	2.48	2.7
	Al^{3+}-SFA/MIO	4.48	2.07	5.80	2.41	2.80
Pseudo second order	K^{+}-SFA/MIO	2.26	2.19	0.01	0.07	0.003
	Ca^{2+}-SFA/MIO	2.30	2.23	0.01	0.07	0.002
	Al^{3+}-SFA/MIO	2.10	2.07	0.00	0.03	0.001
Intraparticle Diffusion	K^{+}-SFA/MIO	1.78	2.19	0.17	0.41	0.08
	Ca^{2+}-SFA/MIO	1.76	2.23	0.22	0.47	0.10
	Al^{3+}-SFA/MIO	1.87	2.07	0.04	0.20	0.02

TABLE III: DIFFUSION MECHANISM CONSTANT FOR ADSORPTION OF M^{X+}-SFA ONTO METALLIC ORES

Kinetic model	**M^{x+}-SFA**	**R^2**		**Constants** ***Kp***	
Intraparticulate diffusivity	K^{+}-SFA	0.97	-	-	0.17
	Ca^{2+}-SFA	0.87	-	-	0.03
	Al^{3+}-SFA	0.08	-	-	0.02
			q_{cal}	q_{exp}	K_{id}
Intraparticle diffusion(Type 1)	K^{+}- SFA	0.90	1.78	2.19	0.07
	Ca^{2+}- SFA	0.88	1.76	2.23	0.07
	Al^{3+}-SFA	0.88	1.87	2.07	0.03
			a		K_{id}
Type II	K^{+}- SFA	0.98	0.09	-	1.32
	Ca^{2+}- SFA	0.93	0.11	-	1.49
	Al^{3+}-SFA	0.95	0.04	-	1.21
			D		K_{mt}
Mass transfer	K^{+}- SFA	0.82	15.49	-	0.004
	Ca^{2+}- SFA	0.92	15.27	-	0.003
	Al^{3+}-SFA	0.79	15.55	-	0.001

TABLE IV: LISTS OF (A) KINETIC MODELS AND (B) ERROR FUNCTION
(A) LISTS OF KINETIC MODELS

S/No	**Kinetic model**	**Linear form**	**Plot**	**Reference**
1	simple first order	$\log c_t = \frac{k_1}{2.303}t + \log c_o$	log c_t vs t	[32], [33]
2	pseudo first-order equation	$\log(q_e - q_t) = \log q_e - \frac{k_1}{2.303}t$	log ($q_e - q_t$) vs t	[35]
3	modified first order kinetic	$\ln\left(1-\frac{q_t}{q_e}\right)+\frac{q_t}{q_e} = k_m t$		
4	Natarajan and Khalaf first order kinetic	$\log\left(\frac{c_o}{c_t}\right) = \left(\frac{k}{2.303}\right)t$	log (C_o/C_t) against t	[39]
5	pseudo second-order rate expression	$\frac{t}{q_t} = \frac{1}{k_2 q_2{}^2} + \frac{1}{q_e}t$		[42]
6	Bhattacharya and Venkobachar first order kinetic	$\log\left[1-u(t)\right] = -\left(\frac{k}{2.303}\right)t$	plot $\log\left[1-u(t)\right]$ against t	[39]

		$u(t)=\left[\frac{(c_o-c_t)}{(c_o-c_e)}\right]$		
7	fractional power model	$q=kt^v$		
8	Elovich kinetic equation	$q_t=\frac{1}{\beta}\left[\ln(\alpha\beta)\right]+\ln\frac{t}{\beta}$	q_t against ln t	[43]

(B) LISTS OF ERROR FUNCTIONS

S/No	Error Function	Abbreviation	Expression	Reference
1	Sum square error	(SSE or ERRSQ)	$\sum_{i=1}^{n}\left(q_{e,cal}-q_{e,\exp}\right)_i^2$	[46]
2	Sum of absolute errors	(EABS)	$\sum_{i=1}^{n}\left(q_{e,\exp}-q_{e,cal}\right)$	[46]
3	Non-linear chi-square test	(X^2)	$\sum_{i=1}^{n}\frac{\left(q_{e,cal}-q_{e,\exp}\right)^2}{q_{e,\exp}}$	[46]

Besides the rate constant (k), several adsorption constants were also adopted in performance assessment. Among these constants include fractional power equation, v. Elovich plot's β and α (estimates the initial adsorption rate) [47].

The experimental and calculated adsorption capacity parameters, $q_{\exp}$ and q_{cal} were not presented for fractional power model. However, Value of its constant, v between 0.04-0.07 obtained in this research is positive and less than 1. This indicates favorable adsorption and this constant falls within ranges reported in published literature [48]. β(6.62-13.70) is related to surface coverage and measures heterogeneity. β value which is also a measure of desorption constant also conform to the empirical equation of Elovich. This is in supports the second order kinetic since Elovich applies to chemisorption. The Elovich equation is however with limited validity [49].

B. Error Functions

Table II shows estimated error functions. In error analysis, experimental and calculated adsorption capacities (q_e) are modeled with error function equations [46] for predicting model with minimized error distribution when experimental q_e is compared with calculated q_e values.

These three error models clearly show that the pseudo second order kinetic equation which presented the least error sums of SSE (9.6×10^{-4} to 5.4×10^{-3}), absolute error (0.031-0.074) and 0.00046-0.0025 in the chi-squared analysis. These values for second order kinetics are lower than those of the other models. Less error function value depicts better fitness.

C. Adsorption Diffusion Models

Table III represents data explaining the mode of transport or diffusion mechanism. According to Weber and Morris, the intra particle diffusion rate constant (K_{id}) is computed as,

$$q_t=k_i t^{\frac{1}{2}} \tag{2}$$

K_{id} (mg/g /min$^{1/2}$) can be determined from slope of the plot, q_t versus $t^{1/2}$.

That of particle diffusion controlled adsorption is a linear driving force concept p-resented as [50],

$$\ln(1-\theta)=-k_p t \tag{3}$$

K_p is rate coefficient for particle diffusion controlled process. It measures adsorbent's particle size. ln (1-θ) relates to the intraparticle diffusivity. A linear plot of ln (1-θ) versus t, is expected for adsorption that is particle–diffusion controlled. The K_P obtained in this work falls within the range of 0.02 to 0.17 (Table III), meaning that the diffusivity of M$^+$-SAF onto MIO does not depend on the extent of sorption.

The type II intraparticle diffusion plots (Fig. 2) gave the highest coefficient of determination value (R^2> 0.94) for adsorption of the entire M$^+$-SFA. However, the high positive R^2 value for mass transfer model is an indication that sorption mechanism is more than one process. Mass transfer diffusion plot was deduced from (4),

$$\ln\left(C_o-C_t\right)=\ln D+K_o t \tag{4}$$

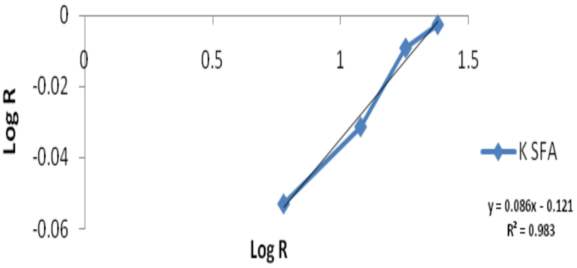

Fig. 2. Intraparticle diffusion plots (Type II) for K$^+$ salt of fatty acid Shea butter oil uptake onto MIO.

External surface adsorption (first linear portion in Fig. 2) as well as non-linear plot (stages at which the transport occurs) are clearly seen. Also observed is a second linear portion where intraparticle diffusion is the rate controlling step and finally, the third linear portion where transport by intraparticle diffusion slows down [50].

IV. CONCLUSION

Sorption affinity of mixed iron ore for metallic salts of fatty acids was evaluated. Equilibration time was attained at the 50th min for the selected M^+-soaps. Metallic surfactant uptake is independent on ionic charge. Results from this work shows that the several kinetic models, three diffusion models and the error functions utilized in this work are very essential for consideration. Best fit models based on three applicability tests are in support of the pseudo-second-order kinetics. Particle trasport is characterized by both surface reactivity as well as a multiple diffusion process. This generalization was drawn from a fact linked to the non passage of intraparticle diffusion plots through the origin.

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