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Adsorption isotherm and kinetics modeling of carotene and free fatty acids adsorption from palm oil onto montmorillonite

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Abstract

Adsorption of carotene and free fatty acid from palm oil onto raw and activated montmorillonite clay has been studied. Acid activation lead to the leaching of exchange cations (Ca^{2+}, K^+, Na^+, Mg^{2+}) and of ferric ions (Fe^{3+}). Acid activated clay has better carotene and free fatty acid adsorption capacities than raw clay. However, leaching of clay with high concentrated acid solution reduced its adsorption efficiency. The amount of carotene adsorbed per gram of adsorbent also increased with temperature, notwithstanding the acid treatment of clay. The kinetics of carotene and free fatty acid were best described by pseudo-second order and intraparticle diffusion models. The activation energies obtained by applying Arrhenius equation to the pseudo-second order kinetic constant were all lower than 24 $kJ.mol^{-1}$. It was concluded that there are different types of adsorption sites for carotene adsorption as the adsorption isotherms did not fit Langmuir equation, but they fit very well Freundlich and Temkin models. These observations were also valid for free fatty acids adsorption. The small values of Freundlich constant n, and of Temkin constant B, show the loose bonding of carotene and free fatty acids on clay surface. Hence the adsorption of these 2 compounds is a physisorption.

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Introduction

Palm oil is one main source of fat in the humid tropical zone all over the world. The native oil has an intensive red color due to its high content of carotene. In Africa, particularly in Cameroon palm oil is heated at temperature above 150°C before it is used for cooking. The heating procedure is intended to remove carotene, but it also lead to the destruction of the triglyceride and to the production of huge amount of smoke (Nnadozie *et al.*, 1989). The red color of the palm oil also limited it utilization in cosmetic and drug production. Owing to these limitations carotenes should be removed from palm oil before using it. (Nguetkam *et al.,* 2007; Ndé Aga *et al.*, 2007)

Adsorption on suitable material has been a preferred method for the removal of pigments from vegetables oils (Sabah *et al.*, 2007; Santhy and Selvapathy 2006; Bike Mbah *et al.*, 2005; Christidis and Kosiari, 2003; Sarrier and Güller 1988). The most effective adsorbent for pigment adsorption from vegetables oils are montmorillonite base activated clay and activated carbon (Ndé Aga *et al.*, 2007; Rossi *et al.*, 2003; Kamga *et al.,* 2000; Nnadozie *et al.*, 1989). However, montmorillonite clay is preferred to activated carbon by the vegetable oil refiner due the high cost of the former adsorbate. However, all these studies reported only on the removal of oil pigments during the adsorption process. The faith of other vegetable oil impurities during the adsorption process has not been presented. Only Bike Mbah *et al.,* (2005) and Ndé Aga *et al.,* (2007) have described the adsorption of free fatty acids during the decolorisation process of shea butter and palm oil respectively. However, these two authors limited their studies on the adsorption kinetics of free fatty acids but the adsorbents properties were not presented. Furthermore, only Langmuir and Freundlich equation were used to model pigment adsorption isotherms.

It is the purpose of this contribution to study the mechanism of simultaneous adsorption of carotene and free fatty acids of palm oil by means of modeling of kinetic and isotherms data's. Three kinetic models and three isotherms models are then used to analyze palm oil pigments and free fatty acid adsorption onto montmorillonite base local clay.

Material and methods

The adsorbent

The raw clay used in the present work was extracted from vertisol soil sample collected at Kaélé (10°08'N and 14°28'E) in the far north region of Cameroon. The 2µm fraction of the raw material was obtained by the sedimentation method according to the Stokes law. Acid activation of the clay was carried out by treating 50 g of clay with 250 ml solution of sulfuric acid at different concentrations. The resulting slurry was heated at 80°C in a shacked thermostated water bath for 2 hours. The mixture was then filtered over Watman N°1 filter paper and washed several times with distilled water until negative test for SO_4^{2-}. The solid material obtained was dried at 105°C in an air oven until constant weight was attained. The non activated clay was designated AF10N, while the clay activated with 1N, 2N and 3N sulfuric acid solution were designated AF11N, AF12N and AF13N respectively.

The chemical composition of the clay was determined by Induced Coupled Plasma Atomic Emission Spectroscopy (ICP-AES). X-ray diffraction patterns were obtained with a DRON 3 diffractometer using Cu-Kα radiation (λ=1.5405 $\overset{o}{A}$). The textural properties of sample out gassed at 120°C for 18h were studied by N_2 sorption measurement at 77K within P/P_o values of 10^{-5}-0.999 in an ASAP 2010 from Micrometric Instruments Co (USA). The total specific surface area was calculated by the BET method. The mesopore size distribution and the mesopore surface area were obtained by applying the numerical integration method of Barret, Joyner and Halenda (BJH) to the desorption isotherm data. The cation exchange capacity (CEC) was measured at neutral pH using cobaltihexammonium ions $[Co(NH_3)_6]^{3+}$ as the exchangeable probe. The equilibrium pHs of clay suspensions (1/2.5 w/w) were measured with a standard LPH 33OT electrode.

Bath adsorption

The olein fraction of palm oil provided by SOCAPALM Co (Cameroon) was used for the adsorption experiment. The adsorption experiments were carried out in 100 ml Erlenmeyer flask by mixing clay and 10 g of palm oil and agitating the mixture in a constant shaking (80 cycle/min) thermostated water bath for the desire time. The mixture was then filtered over Whatman N°1 filter paper and the remaining carotene and free fatty acid in the oil were determined. The amount of carotene adsorbed was determined by measuring the adsorption of cyclohexane diluted palm oil (0.1 g of oil in 5 ml of cyclohexane) at 452 nm, with a Varian Cary 100 spectrophotometer. Cyclohexane was used as reference. The optical path of quartz cell was 1 cm. The relative amount of carotene adsorbed per gram of adsorbent was expressed as:

$$q = \frac{A_0 - A}{A_0 m}$$

Where A_0 and A are the absorbance of the oil at time t=0 and at time t respectively; m the mass of adsorbent. The free fatty acids content were determined by the AFNOR standard methods.

Results and discussion

Adsorbent characteristics

The adsorbent composition is presented in table 1. The main oxides of the raw clay are SiO_2, Al_2O_3 and Fe_2O_3. These three oxides represent more than 80% of the total weight of the clay sample, while the weight percentage of other oxides is only 2%. The SiO_2/Al_2O_3 ratio of about 2, and the high loss on ignition (14.85%) shows that smectite clay is the major clay mineral of this clay. There is a continuous reduction of the exchangeable cation (Na^+, K^+, Ca^{2+}, Mg^{2+}) over treatment of the raw clay with H_2SO_4 solution of increasing concentration.

The X-ray diffraction patterns of raw clay (Fig. 1) shows the broad basal spacing d_{001} of smectite at 8.1-10.0 Å. Kaolinite are identified by its basal spacing at 13.0-14 0 Å and 22 Å. When the clay sample is treated with acid solution of increasing concentration the intensity of the smectite band progressively diminish. This is usually attributed to the partial destruction of the clay lattice (Ngeutnkam *et al.*, 2005).

Table 1. Chemical composition of the raw and activated clay.

Composition (%)	Adsorbent			
	AF10N	AF11N	AF12N	AF13N
SiO_2	46.87	51.33	53.56	59.26
Al_2O_3	23.56	20.00	19.98	17.03
Fe_2O_3	8.97	8.08	7.15	5.65
MnO	0.06	0.04	<Ld	<Ld
MgO	1.45	0.98	0.78	0.56
CaO	1.37	0.36	0.25	0.16
Na_2O	0.52	0.2	0.17	0.11
K_2O	1.29	0.81	0.78	0.71
TiO_2	0.97	1.01	1.31	1.2
P_2O_5	0.09	0.07	0.07	0.06
LI	14.85	14.12	13.95	13.26
Mass balance (%)	100	97	98	98

Textural characteristics of the adsorbent are presented in table 2. Acid activation lead to the increase of the surface area and pore volume. This was attributed to the formation of silica during acid activation. The reduction of the CEC during the acid activation is attributed to the leaching of exchangeable cations.

Table 2. Textural characteristics of the adsorbents

	AF10N	AF11N	AF12N	AF13N
S_{BET} (m^2/g)	90.1	134.5	181.2	201.3
Pore diameter (Å)	48.9	50.2	55.7	68.3
CEC (meq/100g)	72.7	23.8	19.3	14.0

Carotene adsorption

Kinetic

Kinetic curve for the adsorption of carotene on clays are presented in Fig. 2. It is observed that the relative amount of carotene adsorbed per gram of clay gradually increases and then become constant after a certain time. The time required to reach adsorption equilibrium are 105min, 85min, 70min and 60min at 60°C, 70°C, 80°C and 90°C respectively for all the adsorbents. This reduction of the time required to reach the adsorption equilibrium vs temperature is probably due to the reduction of the viscosity of the oil and activation of more adsorption site (Ahmad *et al.,* 2009; Hameed and El-Khaiary, 2008; Bera *et al.,* 2004; Langmaack and Eggers, 2002). The amount adsorbed at equilibrium increases with the concentration of acid solution used for activation up to 2N and then decreases. The initial increase of the amount of carotene adsorbed with acid concentration of acid solution is due to the removal of exchangeable cations which induce the increased of the specific surface area, and more adsorption sites are available for pigment adsorption

Table 3. Pseudo-second order and intraparticle diffusion parameters of the adsorption kinetic of palm oil pigments onto different clay fraction.

Clay fraction	**T(°C)**	**Pseudo-second order model**		**Intraparticle model**	
		k_2 (µmol/g.min)	R^2	k_{int1} (µmol/g.min$^{1/2}$)	R_1^2
AF10N	60	0.0121	0.991	0.201	0.958
	70	0.0045	0.973	0.316	0.985
	80	0.0054	0.989	0.308	0.999
	90	0.0059	0.989	0.348	0.998
AF11N	60	0.0023	0.955	0.478	0.993
	70	0.0033	0.968	0.613	0.971
	80	0.0021	0.970	0.700	0.997
	90	0.0040	0.955	0.717	0.966
AF12N	60	0.0041	0.989	0.507	0.981
	70	0.0050	0.966	0.675	0.955
	80	0.0031	0.962	0.781	0.987
	90	0.0083	0.976	0.689	0.918
AF13N	60	0.0025	0.968	0.400	0.980
	70	0.0025	0.957	0.440	0.982
	80	0.0031	0.962	0.495	0.996
	90	0.0031	0.965	0.558	0.998

The reduction of the amount of carotene adsorbed and high acid concentration is due to the destruction of clay lattice and formation of silica (Wu *et al.*, 2009; Sabah *et al.*, 2007; Nguetkam *et al.*, 2005). Silica is a poor adsorbent for vegetable oil pigment (Kamga *et al.*, 2000). The sorption data were analyzed using three kinetic models, manly intraparticle diffusion, pseudo-first order and pseudo-second order models. The respective linearised equation of intraparticle diffusion models pseudo-first order and pseudo-second order models are:

$$q_t = k_{\text{int}} t^{1/2} + \lambda$$

$$\log(q_e - q_t) = \log q_e - \frac{k_1}{2.303} t$$

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

where k_{int}, k_1 and k_2 are the respective intraparticle diffusion, pseudo-first and pseudo-second order kinetic constant, q_e and q_t are the respective amount of pigment adsorbed at equilibrium and at time t, λ is a constant.

Table 4. Langmuir, Freundlich and Temkin constants for adsorption of palm oil pigments by differents adsorbents at 90°C.

Adsorbent	Freundlich isotherm			Temkin isotherm		
	K_F	n	R^2	A	B $(J.mol^{-1})$	R^2
AF10N	0.201	1.305	0.990	0.636	2.012	0.947
AF11N	0.182	1.706	0.996	0.706	1.711	0.972
AF12N	0.146	1.636	0.977	0.712	2.035	0.896
AF13N	0.157	1.249	0.983	0.644	2.401	0.911

Table 5. Kinetic parameters of pseudo-second order and intrapartcle diffusion models for the palm oil free fatty acids adsorption on differents adsorbent.

	Pseudo-second order model		Intraparticle diffusion model	
Adsorbents	k_2 (µmol/g.min)	R^2	K_{int} (µmol/g.min)	R^2
AF10N	0.0048	0.987	0.305	0.992
AF11N	0.0055	0.970	0.454	0.972
AF12N	0.0065	0.982	0.488	0.979
AF13N	0.0045	0.955	0.441	0.958

Table 6. Freundlich and Temkin constants of free fatty acids adsorption onto differents adsorbent at 90°C.

Adsorbent	Freundlich isotherm			Temkin isotherm		
	K_F	n	R^2	A	B $(J.mol^{-1})$	R^2
AF10N	0.246	0.488	0.922	0.345	3.497	0.980
AF11N	0.175	0.760	0.916	0.509	3.109	0.974
AF12N	0.129	0.722	0.903	0.521	3.780	0.978
AF13N	0.165	0.622	0.920	0.456	3.676	0.980

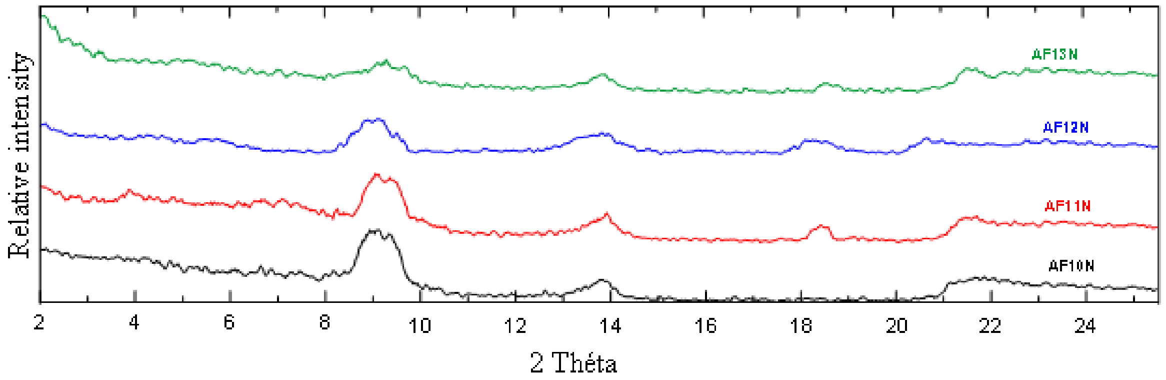

Fig. 1. X-rays diffraction spectra of the raw and activated clay.

The linear regression of $\log(q_e - q_t)$ vs t, have R^2 values lower than 0.800 for all the adsorbent used.

Table 3 present the kinetics constants k_{int}, and k_2, for all the adsorbents used herein. The R^2 values are greater than 0.955 for pseudo-second order plot (i.e. $\frac{t}{q}$ vs t) and intraparticle diffusion plot (q_t vs $t^{1/2}$) respectively. These results show that our experimental data fit very well the pseudo-second order and the intraparticle diffusion models.

The pseudo-second order kinetic constants were used to determine the activation energy for carotene adsorption. For this purpose the Arrhenius equation was used. This equation is generally described as

$$k_2 = k_0 \exp\left[\frac{-Ea}{RT}\right]$$

where k_0 is the Arrhenius constant, Ea is the activation energy, R is the perfect gas constant and T is the absolute temperature. The linear expression of Arrhenius equation is

$$\ln k_2 = \ln k_0 - \frac{Ea}{RT}$$

In order to obtained the activation energy, a straight line plots of $\ln k_2$ against 1/T were made (not shown here). The obtained activations energies are 9.8±0.2, 17.6±0.2, 23.9±0.2 and 10.8±0.3 kJ.mol^{-1} for AF10N, AF11N, AF12N and AF13N respectively. The evolution of the activation energies has the same trend as that of k_2. The activation energies for all the adsorbents are much lower than those obtained by Bike Mbah *et al.,* (2005). But Bike Mbah *et al.*, used kinetic constant of the first stage of intraparticle diffusion to evaluate the activation energy. The first stage of intraparticle diffusion model is associated to the transfer of adsorbate from solution to the adsorbent surface. The adsorbate-adsorbent interaction is not considered, therefore, this method is not appropriate to determine the activation energy. On the contrary pseudo-second order model take in account the diffusion of adsorbate from herein (results not show). These results show that the adsorption of palm oil carotenes on raw and activated clay is not pseudo-first order reaction. solution to the adsorbent surface and the interaction between adsorbate and adsorbent.

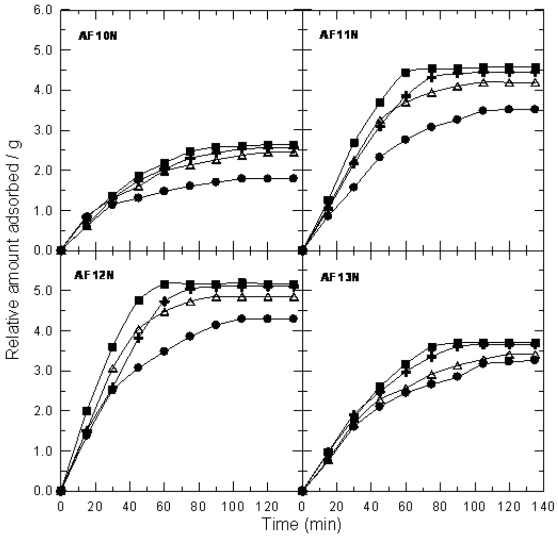

Fig. 2. Adsorption kinetics of palm oil pigments by adsorbents AF10N, AF11N, AF12N and AF13N at the temperatures of 90°C (■), 80°C (✚), 70°C (Δ) and 60° (●)

Isotherm

Adsorption isotherm is an invariable curve describing the phenomenon governing the retention (or release) of a substance from the solution to the solid phase at constant temperature. Fig. 3 shows the adsorption isotherm of palm oil carotene onto raw and various acids treated clays. It appears that acid treatment of clay sample improves their carotene adsorption up to concentration of 2N. Over this value there is a reduction of the amount of carotene adsorbed by the acid-treated clay. This trend is similar to that observe during kinetic study.

Using different adsorption model to analyze adsorption isotherm data could be an interesting approach to have more information on the carotene-clay interaction. Freundlich and Langmuir models are the most commonly used equation to describe adsorption from solution, but Temkin model is also used. In this study Langmuir, Freundlich and Temkin equation were used to model adsorption data. The respective linear forms of these equations are

$$\frac{X_e}{\frac{X}{m}} = \frac{1}{q_m K_L} + \frac{1}{q_m} X_e$$

$$Log\left(\frac{X}{m}\right) = LogK_F + \frac{1}{n} LogX_e$$

$$\frac{X}{m} = BLnA + BLnX_e$$

where X_e is the residual amount of carotene at equilibrium; X/m the relative amount of carotene adsorbed per gram of adsorbent, K_L is the Langmuir constant related to adsorption energy; K_F is a Freundlich constant, indicative of adsorption capacity of the adsorbent; q_m is the monolayer maximum adsorption capacity; n is a constant indicative of adsorption intensity; B is the Temkin constant indicative of adsorption energy and A is the Temkin isotherm constant.

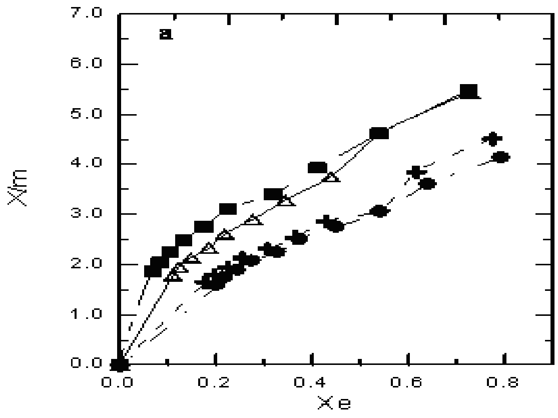

Fig. 3. Adsorption isotherms of palm oil pigments by adsorbents AF12N (■), AF11N (Δ), AF13N (✚) and AF10N (●)

The R^2 values for the linear plot of adsorption data at 90°C and the corresponding Freundlich and Temkin constant are reported in table 4. The R^2 values for Lamgmuir plot were lower than 0.900 for all the adsorbent and are not reported. The R^2 values are greater thant 0.970 and 0.900 for Freundlich and Temkin model respectively.

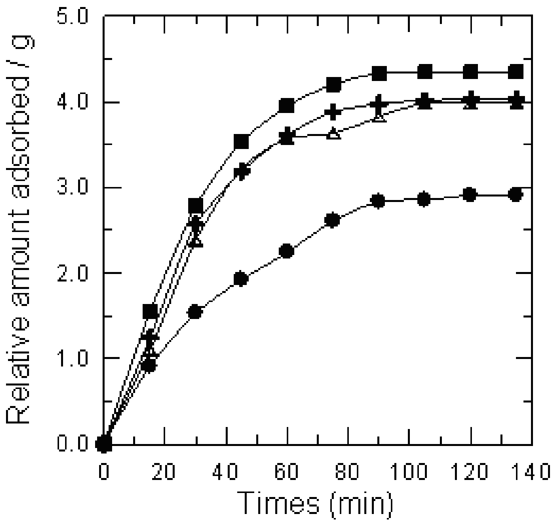

Fig. 4. Adsorption kinetics of palm oil free faty acid at 90°C by adsorbents AF12N (■), AF11N (✚), AF13N (Δ) and AF10N (●)

The Langmuir equation is based on the assumption of structurally homogeneous adsorbent where all sorption sites are identical. The poor correlation of our experimental data with the Langmuir equation shows that carotene adsorption sites are not identical. The best fit were obtained with Freundlich equation (R^2>0.970). This equation is an empirical equation employed to describe heterogeneous adsorption. We thus conclude that there are different carotene adsorption sites on the clays sample. The n values which lie between 1 and 10, are indicative of good affinity between carotene and clays. However, the low values of adsorption energies as shown by the B values are indicative of physical adsorption of carotene on clay adsorbents.

Free fatty acids adsorption

Kinetic

The kinetic curves for the adsorption of palm oil free fatty acids on clay at 90°C are presented in fig. 4. It comes out from this figure that the relative amount of free fatty acids adsorbed per gram of adsorbent increases with time and stabilizes after 100 min thus the time required to reach the free fatty acids adsorption equilibrium is much longer than those observed for carotene adsorption.

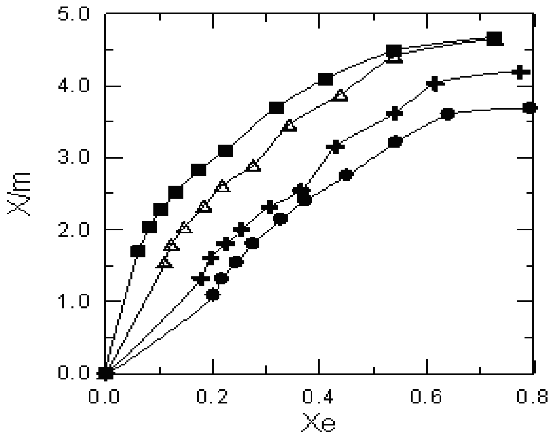

Fig. 5. Adsorption isotherms of palm oil free fatty acids onto adsorbents AF12N (■), AF11N (Δ), AF13N (✚) and AF10N (●)

Free fatty acids kinetic data were compared with the pseudo-first and pseudo-second order kinetic models and with intraparticle diffusion model. It comes out from the results obtained that the pseudo-first order kinetic model is not applicable for the palm oil free fatty acid adsorption due to the very low value of the correlation coefficients R^2 (lower than 0, 7). The results concerning this model were thus not shown. The R^2values and the kinetic constant for pseudo-second order and intraparticle diffusion models are presented in tables 5.

The values of the correlation coefficient R^2 are all higher than 0,95. This result suggests that the speudo-second order kinetic model is suitable to discribe the palm oil free fatty acids adsorption on natural clay and activated clay. The free fatty acids adsorption is thus done in two steps:first the diffusion of the free fatty acids towards the adsorbent surface followed by the interaction to the surface of the adsorbent.

Isotherm

The adsorption isotherms of the palm oil free fatty acids onto clay samples at 90°C are reported in Fig.5. It appears that acid treatment of clay sample improves their free fatty acids adsorption up to concentration of 2N.

The experimental data obtained were compared with Langmuir, Freundlich and Temkin models. Table 6 presents the results obtained. The R^2 values for Langmuir model are all lower than 0,60 and are not reported. This is an indication that this model is thus not adapted to describe the palm oil free fatty acid adsorption. We can thus deduce from it that, free fatty acids are adsorbed on the sites of different nature than those from the pigments. These sites can be on clays or oxides such as the iron oxide present in the various samples. Indeed the chemical composition of the various samples shows a proportion of Fe_2O_3 higher than 4,00%.

The heterogeneity of the adsorption sites is confirmed by the correlation coefficients R^2 of Freundlich model which are all higher than 0,90. The *n* constant are all lower than 1, this is an indication of weak affinity between the free fatty acids and the adsorbents surfaces (Adamson, 1990; Ho and McKay, 1998). The more adapted model for the palm oil free fatty acids adsorption onto clay is the Temkin model. Indeed the R^2 are all higher than 0,95. Taking into account the values of constant B (relating to the energy of interaction between adsorbent and adsorbate) obtained for our samples, we can conclude that the interaction between the free fatty acids and all the clay samples are weak. Thus adsorptions of free fatty acids on clay are probably a physical adsorption.

Conclusion

Acid activation of clay with sulfuric acid solutions enhanced their palm oil carotene and free fatty acid adsorption. However activation of clays with concentrated solution lead to the reduction of these adsorption capacities. Both pigment and free fatty acids intraparticle diffusion kinetic models.

Neither carotene adsorption nor free fatty acids follow Langmuir model. Thus there are different types of adsorption sites for these compounds. It comes out from Freundlich and Temkin modeling of the isotherm that there are weak interactions

between the adsorbates (carotenes and free fatty acids) and the clay surface.

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