

Adsorptive Removal of Carotene and Phosphorus from Palm Oil Using Natural and Modified Aguleri Clay

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Date of Submission: 10-09-2022

Date of Acceptance: 20-09-2022

ABSTRACT

Adsorptive removal of carotene and phosphorus from palm oil using natural and modified aguleri clay. The raw sample of palm oil were bought from a market at Umunze in Anambra State while the clay were collected from Aguleri to determine the kinetics and thermodynamics of adsorption of carotene and phosphorus using natural clay and modified clay. The clay was thoroughly washed with de-ionized water at pH 7.0, completely dispersed in water to remove extraneous materials and afterward was dried at 85°C. The chemical analysis of the clay were obtained using XRF, while their BET surface area, pore volume, average pore width, average pore diameter, porosity and solid density were determined using standard methods. Samples of same clays were made to pass thermal and acid activation method. The clay was crushed and sieved with 0.25mm mesh and 40g of the sieved sample was measured into a crucible and placed in a muffle furnace. The thermal activation was done over a temperature of 500-600°C for a fixed period of 120minutes. Thereafter, the samples were allowed to cool to room temperature in a desecrator for 4 hours. The samples were then packaged according to their activation temperature. The dried clay sample was ground into powder with mortar and pestle, and sieved through 1100um sieve, and 50g of the clay sample was introduced into 500ml beaker. 1M solution of H₂SO₄ of analytical grade was added with solid-liquid ration of 1:5(w/v). The resulting suspension was heated while being stirred using adjustable temperature magnetic stirrer set at 100°C for 90mins. The resulting mixture was filtered and washed with distilled water until neutral pH is obtained. The activated clay was dried in an electric oven at

105°C. There after the dried samples were sieved and stored for use. The three samples, raw clay (RC), thermally activated clay (TAC) and acid activated clay (AAC) were characterized using Fourier Transform Infra-Red (FTIR), Scanning Electron Microscope (SEM) and X-ray Fluorescence (XRF).

Keywords: Adsorption, Carotene, Clay, Kinetics, Palm Oil, Phosphorus and Thermodynamics.

I. INTRODUCTION

Palm oil is one of the various types of vegetable oils belonging to the group called lipids because of its fatty acid content. Indonesia is the most producer of palm oil (crude palm oil/CPO) in the world with palm oil production of 25.4 million metric tons in 2012. 65% of Indonesia CPO are still largely exported as raw material, the rest (35%) is used for domestic needs as raw materials for cooking oil, margarine and a small portion is used as a raw material for biodiesel and oleichemicals including fatty acids, fatty alcohol and glycerine (Kurniawan & Afriapollo, 2019). Parwez, 2006 defined palm oil as triglycerides extracted from plants. Crude oil is the world's richest natural plant source of carotene in terms of retinol.

The natural pigments present in vegetable oils are mainly the carotenoids, giving yellow and red colours and the chlorophylls which give green colours (Brooks, 1999). The orange-red colour of palm oil is due to the relatively high content in these carotenes. The major carotenes in palm oil are α and β carotenes, which account for 90% of the total carotenes (Nurmayanti, 2013). Carotenoids is not only needed for food industry and pharmaceutical, but also for cosmetic

industries. In the food industry, it is used as nutritional and colouring ingredients; in the pharmaceutical industry, it is used as a beauty ingredient. Raw palm oil, in addition to carotenoid pigments content, contains impurities like free fatty acid, fibre particles, chlorophyll, and phosphorus among others. Phosphorus in palm oil mostly occur as inorganic phosphates (Goh, Tong, and Gee, 1984)

Vegetable oils are important compounds of the human diet and they should be refined before consumption. Consumers demand for healthier products as well as stiff environmental legislation are forcing refining industries towards changes and improvement of processes. Several methods are utilized in purification of palm oil including adsorption (Hui, 2009).

Removal and reduction of colour and other pigmented components, otherwise called bleaching during refining according to Borner and Schneider (1999) is necessarily not only because a pale-coloured fat has an appeal of purity, but also because the colours of the fat can influence the appearance of prepared food and even more importantly, the pigment present may affect the flavor and stability of the fats and foods made from them. Unbleached oils contain pesticide residues such as cypermethrin and λ -cyhalothrin. They also contain oxidation products and heavy metals which can become harmful when consumed (Ma & Lin, 2004).

Clays are collectively called aluminosilicates as they contain aluminium oxide and silicon dioxide as universal minerals. Clay is a group of hydrous aluminium silicate minerals with particles that are either $< 2 \mu\text{m}$ or $< 5 \mu\text{m}$ in nominal spherical diameter. The actual size may be $100 \mu\text{m}$ in one dimension and very much less than $1 \mu\text{m}$ in another (Raftery, 2005). Commercial clays, or clays utilized as raw material in manufacturing, are among the most important non-metallic mineral resources. The value of clays is related to their mineralogical and chemical composition, particularly the clay mineral constituents, kaolinite, montmorillonite, illite, chlorite and attapulgite (McGraw-Hill Encyclopidia, 1987). According to Borner and Schneider (1999), clays were classified into phyllosilicates and layers silicates. A very important aspect in the field of clay minerals is its use in bleaching of vegetable fats and oils to improve its nature. Various studies have also been carried out with or without modification in the use of clays to purify edible oils by adsorption (Sarier & Guler, 1988). Clays has for long been used as clarifying agents for vegetable oils for human consumption (Egbuna & Aneke, 2005), filling white

paper, drilling fluids, making bricks, tiles, sanitary wares, cups and plates.

According to Taylor (2005), various naturally occurring clays have found significance for adsorptive purification in its most undesirable constituents and contaminants from fats and oils by adsorptive mechanism. It was shown that acid activated clay adsorbed carotenes on its active sites by formation of hydrogen bonding with the Broensted sites or co-ordination bonds with Lewis acid sites forming stable carbonium ions (Christidis & Kosiari, 2003; Chen, 1994). Bleaching removes phospholipids, inorganic phosphates and gums from oils.

The removal of pigments and several other traces of constituents by adsorption is one of the most important stages in the oil refining process. Activated clays with strong inorganic acids have been used as adsorbents. However, the used clays, impregnated with oil, correspond to about 30-40 % of the total weight and are discarded, causing problems of environmental pollution. The restrained unsaturated oils in the discarded clay oxidize quickly when in contact with air. This process causes strong odor and the material spontaneous combustion becomes possible (O'Brien, 1998).

II. METHODOLOGY

Materials

The materials used are palm oil, natural/raw clay, reagents: Methanol and H_2SO_4 . The clay were collected from Aguleri.

Method

Adsorbent Preparation

The clay was thoroughly washed with de-ionized water at pH 7.0 and was completely dispersed in water to remove extraneous materials and dried at 85°C . The chemical analysis of the clay were achieved using X-ray fluorescence (XRF), while their BET surface area, pore volume, average pore width, average pore diameter, porosity and solid density were determined using standard methods. Samples of same clays were made to pass thermal and acid activation.

Thermal activation

The prepared clay was crushed and sieved with 0.25mm mesh; and 40g of the sieved sample was measured into a crucible and placed in a muffle furnace. The thermal activation was done over a temperature of $50\text{-}600^\circ\text{C}$ for a fixed period of 120 minutes. Thereafter, the samples were allowed to cool to room temperature in a desecrator for 4 hours. The samples were then packaged according to their activation temperature.

Acid activation

Acid activation method according to Brooks et al (2012) and Ajemba (2012) was adopted. The dried clay sample was ground into powder with mortar and pestle, and sieved through 1100µm sieve. 50g of the clay sample was introduced into 500ml beaker. 1M solution of H₂SO₄ of analytical grade was added with solid-liquid ration of 1: 5(w/v). The resulting suspension was heated while being stirred using adjustable temperature magnetic stirrer set at 100°C for 90mins.

The resulting mixture was then filtered and washed with distilled water until neutral pH is obtained. The activated clay was dried in an electric oven at 105°C. There after the dried samples were sieved and stored for use.

Adsorbent Characterization

The three samples (RC, TAC and AAC) were characterized using Fourier Transform Infra-Red (FTIR), Scanning Electron Microscope (SEM) and X-ray Fluorescence (XRF).

Characterization of CPO (Crude Palm Oil)

The CPO was characterized using standard method adopted from AFNOR (1981). The moisture content, initial carotene value (UV at 446nm) measured using UV visible spectroscopy, initial phosphorus value, Deterioration of Bleachability Index (DOBI) etc. values were determined through chemical analysis of the CPO.

Determination of carotene content of palm oil

The carotene content of the palm oil is calculated as β-carotene expressed in terms of parts per million (ppm). The method is according to the PORIM Test Methods (1995). The β-carotene concentrations in the crude and bleached palm oil samples were measured by the UV-Vis spectrophotometer (Varian Cary Bio 100) at 446 nm. The β-carotene removal percentage in the bleached palm oil can be calculated using the following

$$\% \text{ Carotene removal} = \frac{\text{Abs}_{\text{int}} - \text{Abs}_{\text{fin}}}{\text{Abs}_{\text{int}}} * 100$$

Determination of phosphorous content of palm oil

This analysis determines the total phosphorus content by charring and ashing the oil in the presence of magnesium oxide followed by calorimetric measurement as phosphovanadomolybdc complex. The method used is according to the PORIM Test Methods (2005). The charring step is a critical and tedious

stage, precautions were required to ensure reproducibility of result. The yellow phosphovanadomolybdc complex between phosphate, vanadate and molybdate was formed and the colour was determined by the UV-Vis spectrophotometer (Varian Cary Bio 100) at 400 nm.

Actual Adsorption Experiment

Summary of batch adsorption experimental method

The adsorption studies were carried out in batches. The desired weight of CPO was weighed into 250ml conical flask. The temperature of the sample was raised to a desired temperature with the aid of adjustable temperature magnetic stirrer. A known quantity of the adsorbent (RC, TAC and AAC) was introduced into the system. The mixture was further sustained (heating and stirring) for 90mins.

The hot mixture was filtered under gravity using filter paper at the end of the heating time,. The absorbance of the resultant oil was measured with the aid of UV visible spectrophotometer. The percentage of carotene and phosphorous were determined. The percentage removed were analyzed based on the difference between the concentration of carotene and phosphorus remaining and the initial carotene and phosphorus content.

Effect of adsorbent weight/dosage

Different weight of RC, AAC and TAC (1g-5g) were used for the adsorption experiment. The various weights of the adsorbents were added into 100g of CPO in batches after the CPO samples have been thermally stabilized and brought to 90°C. The mixture was stirred while the temperature was maintained at 90°C for 60mins, thereafter, the quantity of carotene and phosphorus adsorbed were estimated.

Effect of adsorbent particle size

The RC, AAC and TAC samples were made to pass through different sets of sieves, and different particles size of the adsorbents were obtained. The best adsorbent dosage or weights obtained were used with different particle sizes. 3%w/w of adsorbent and CPO were used. The adsorption experiment was repeated at 90°C and 60mins, thereafter the percentage of carotene and phosphorus removed were estimated.

Kinetics of adsorption system

The best adsorbent dosage and particle size were used. The adsorption experiment was conducted at 90°C in batches at different times (20-160mins) and 3%w/w of adsorbent and CPO. The carotene and phosphorus content were established after each experiment.

Effect of temperature

The effect of temperature was studied at the best adsorbent dosage, best obtained particle size and at the best time. The temperature was varied from 30-100°C for RC, TAC and AAC. The system was kept stirred within the stipulated time. Thereafter, the carotene phosphorus content was calculated.

Adsorption Optimization

The combined effect or interaction of different independent variables such as time, temperature, adsorbent weight (or dosage) and adsorbent – oil ratio were investigated using response surface methodology. The design matrix generated for the experimental studies are shown below in table 1.

Table 1.1: Optimization Matrix for Carotene and Phosphorus Removal from CPO

StdOrder	RunOrder	PtType	Blocks	Time(Min)	Temp(K)	Adsorbent weight(g)	%Carotene removed	%phosphorus removed
11	1	-1	1	75	-342.372	3		
9	2	-1	1	-0.68068	625.5	3		
19	3	0	1	75	625.5	3		
12	4	-1	1	75	1593.372	3		
10	5	-1	1	150.6807	625.5	3		
6	6	1	1	120	50	5		
16	7	0	1	75	625.5	3		
3	8	1	1	30	1201	1		
13	9	-1	1	75	625.5	-0.36359		
7	10	1	1	30	1201	5		
2	11	1	1	120	50	1		
20	12	0	1	75	625.5	3		
4	13	1	1	120	1201	1		
18	14	0	1	75	625.5	3		
14	15	-1	1	75	625.5	6.363586		
8	16	1	1	120	1201	5		
1	17	1	1	30	50	1		
15	18	0	1	75	625.5	3		
5	19	1	1	30	50	5		
17	20	0	1	75	625.5	3		

Results and Discussion

Characterization of RC, TAC AND AAC

The physical and chemical properties of the raw clay (RC), thermally activated clay (TAC) and acid activated clay (AAC) are presented in table 2.1, 2.2 and 2.3 respectively.

Table2.1 Physical Characteristics of RC

SURFACE AREA(M2/G)	64.8
ORGANIC MATTER(%)	6.87
BULK DENSITY G/ML	1.99
POROSITY	0.502
CATION EXCHANGE CAPACITY(CEC)	0.1163
PH(H2O)	6.1
TOC(%)	12
PH(CACL2)	7.3
MOISTURE CONTENT	9.4

Table 2.2 Physical Characteristics of TAC

SURFACE AREA(M²/G)	90
ORGANIC MATTER(%)	5.1
BULK DENSITY	1.78
POROSITY	0.67
CATION EXCHANGE CAPACITY(CEC)	0.123
PH_(H2O)	6.5
TOC(%)	5
PH_(CACL2)	5.4
MOISTURE CONTENT	6.3

Table 2.3 Physical Characteristics of AAC

Surface area(m²/g)	82
Organic matter (%)	5.3
Bulk Density	1.5
Porosity	0.802
CEC (Meq/100g)	2.66
pH_(H2O)	2.4
TOC(%)	8
pH(cacl₂)	1.3
Moisture Content%	8.6

The pore size distribution results showed that natural clay is predominantly mesoporous. The specific surface area of the raw and modified clay samples are given in Tables 2.1, 2.2 and 2.3 for RC, TAC and AAC respectively. The effect of thermal and acid modification can be observed Tables 2.1, 2.2 and 2.3. In Table 2.1, RC has surface area of 62m²/g while the TAC has 90m²/g (Table 2.2) and the AAC has surface area of 82m²/g (Table 2.3). From the tables, it can be observed that the modification process increased the surface area of the clays. This improvement is desirable for adsorption because it creates more active sites for particles or color adsorption. These results are in good agreement with the work done by Valenzuela-Diaz and Souza-Santos (2001).

Moisture is considered undesirable in bleaching of palm oil, since it contributes to the free fatty acids content and increases the oil loss during oil refining (Ng and Gibon, 2010). Too much water in bleaching clay will tremendously reduce the ability of the bleaching clay to absorb colour. According to Oboh and Aworh (1988) bleaching clay normally contains between 10-18% free moisture. This study indicated that the moisture content of both RC, TAC and AAC were within the acceptable range for oil bleaching. RC contains 9.4 TAC and AAC contain 6.3 and 8.6 respectively. This means that the moisture content of clays investigated in this study is suitable for bleaching of the palm oil.

Increase in porosity was observed as a result of the modifications. The result of Table 2.1 showed the porosity of RC to be 0.52, as a result of thermal and acid activation, this porosity increased to 0.67 and 0.8 respectively, according to Welsh & Bogdanor (1993) high clay porosity results in high bleaching capacity for the adsorption of carotene and phosphorous from the oil. It is evident that the modification of the textural properties is related to the changes in the clay structure which occurred during thermal and acid activation. Therefore, it is important to monitor the structural and textural changes that occurred during the modification, this was done using the instrumental analysis.

Kinetics of removal of Phosphorus from crude palm oil using RC, TAC and AAC

The kinetics data of phosphorus removal using RC, TAC and AAC were analyzed using reversible first order, first order, Pseudo-first order kinetic model, Pseudo-second order kinetic model, Elovich kinetic model, Intra-particle diffusion model and Boyd kinetic model. The plots were made using the kinetic data obtained at the best observed clay or adsorbent dosage and 800C. These kinetics plots are presented in Figs 1.1 - 1.7. It was observed that removal of phosphorus followed the same trend as the removal of carotene.

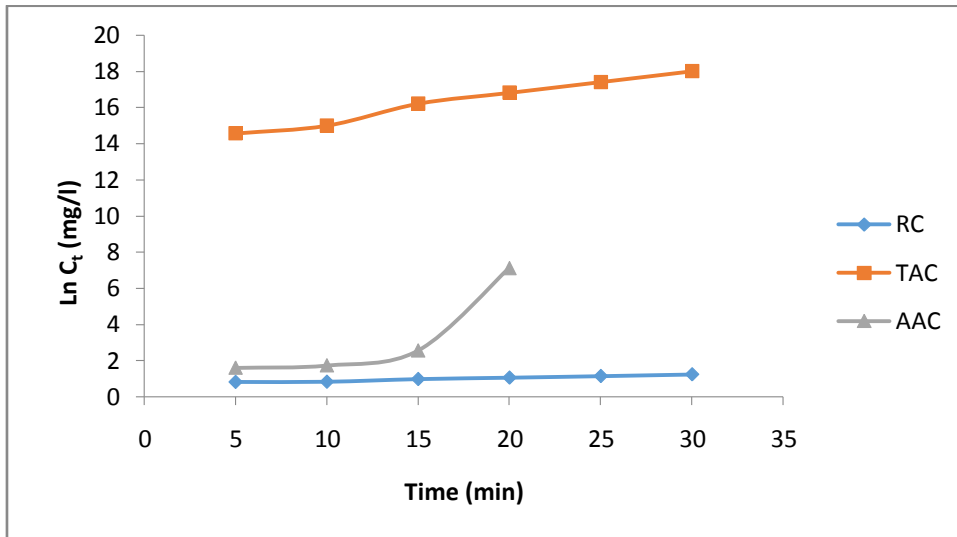


Fig 1.1: First order kinetics plot for phosphorous removal using RC, TAC and AAC

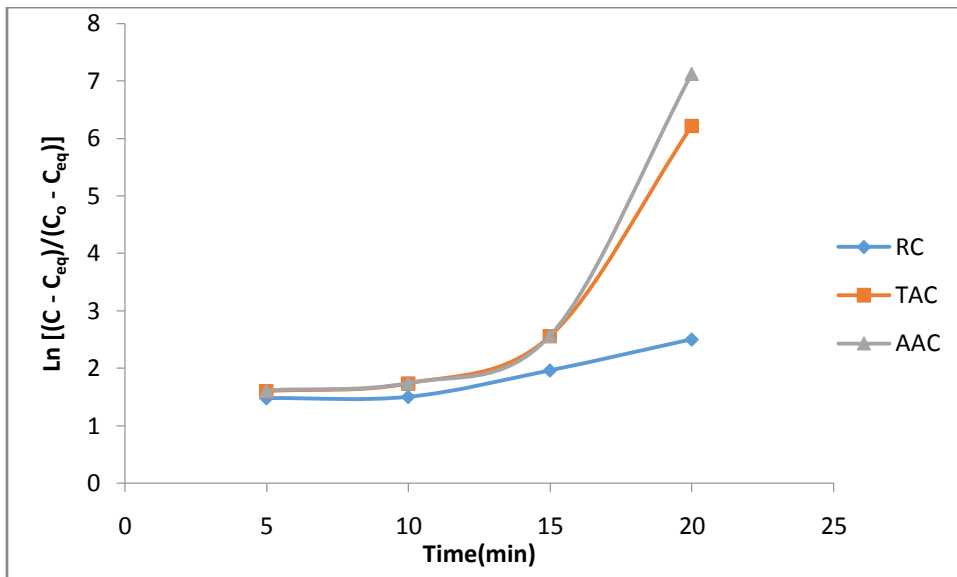


Fig 1.2: Reversible first order for phosphorous removal using RC, TAC and AAC

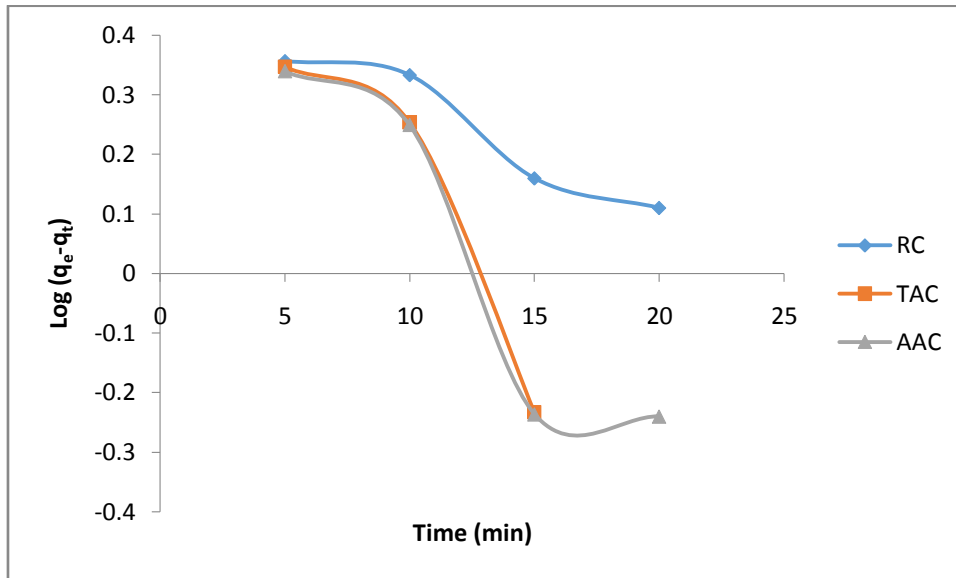


Fig 1.3: Pseudo first order for phosphorus removal using RC, TAC and AAC

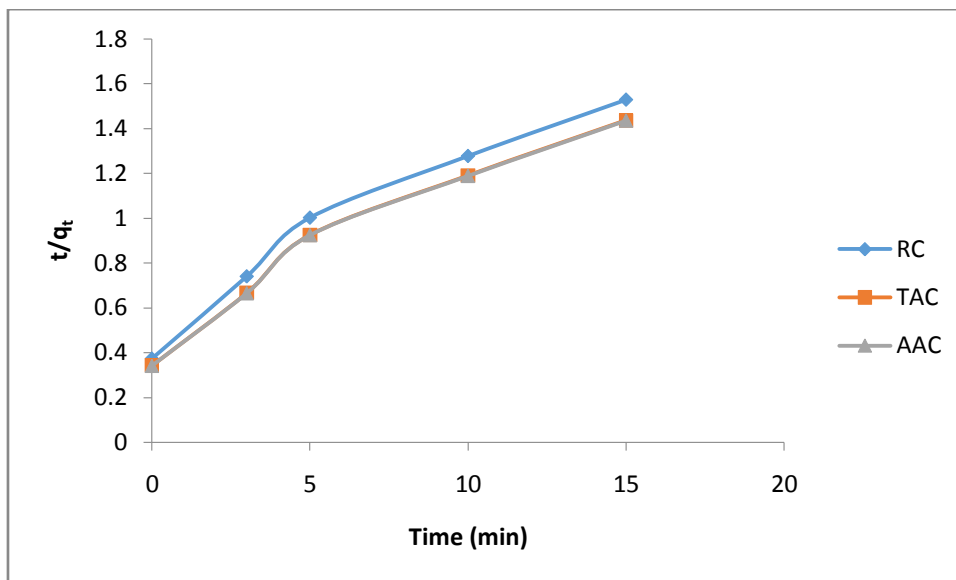


Fig 1.4: Pseudo second order for phosphorus removal using RC, TAC and AAC

Elovinch kinetics for phosphorus removal using RC, TAC and AAC

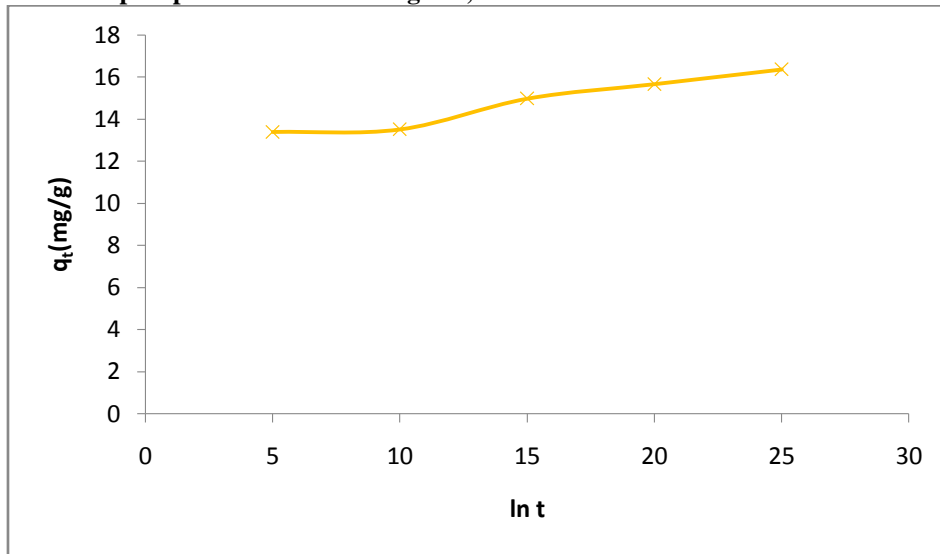


Fig 1.5: Elovinch kinetics for phosphorus removal using RC

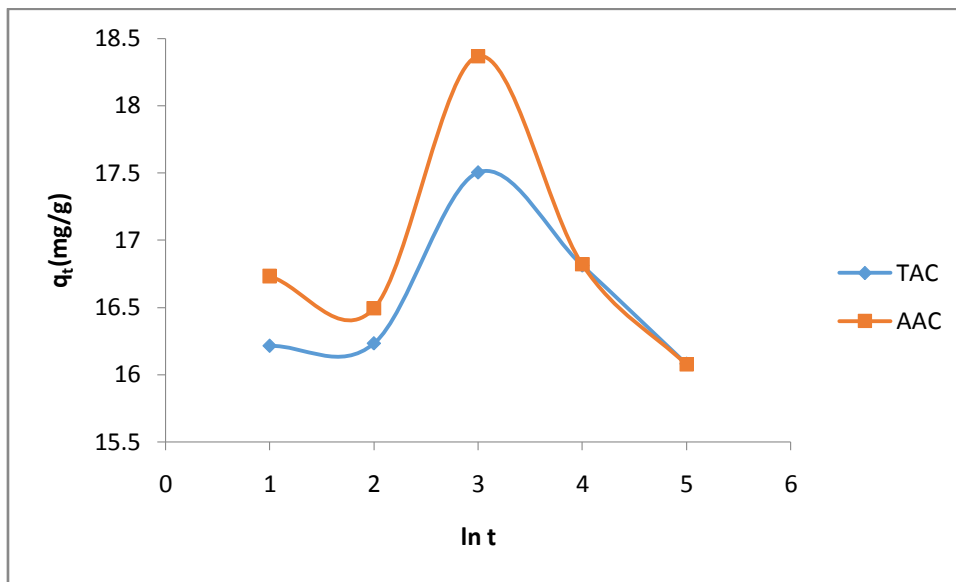


Fig 1.6: Elovinch kinetics for phosphorus removal using TAC and AAC

Intra-particle kinetics for phosphorus removal using RC, TAC and AAC

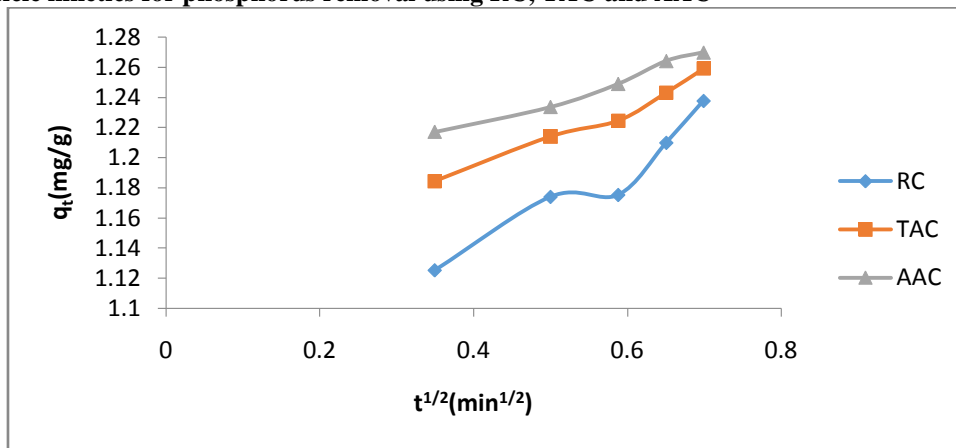


Fig 1.7: Intra-particle kinetics for phosphorus removal using RC, TAC and AAC

Thermodynamics of carotene and phosphorus adsorption from crude palm oil using RC, TAC and AAC

The thermodynamics parameters: the standard free energy (ΔG°), enthalpy change (ΔH°), and entropy change (ΔS°) were estimated to evaluate the feasibility and the exothermic nature of the carotene and phosphorus removal process. The Gibbs free energy change of the process is related to the equilibrium constant (K_L) by the following equation:

$$(\Delta G^\circ) = - RT \ln K_L$$

(eq. 1.1)

According to thermodynamics, the Gibb's free energy change is also related to enthalpy change and entropy change at constant temperature as follows:

$$\ln K_L = (\Delta S^\circ / R) - (\Delta H^\circ / RT)$$

(eq. 1.2)

The values of (ΔH°) and (ΔS°) were calculated from the slope and intercept of the plot of $\ln K_L$ against $1/T$ for RC, TAC and AAC studied for carotene and phosphorus removal at 80°C, 100°C and 120°C.

Thermodynamics plots of carotene removal from crude palm oil using RC, TAC and AAC

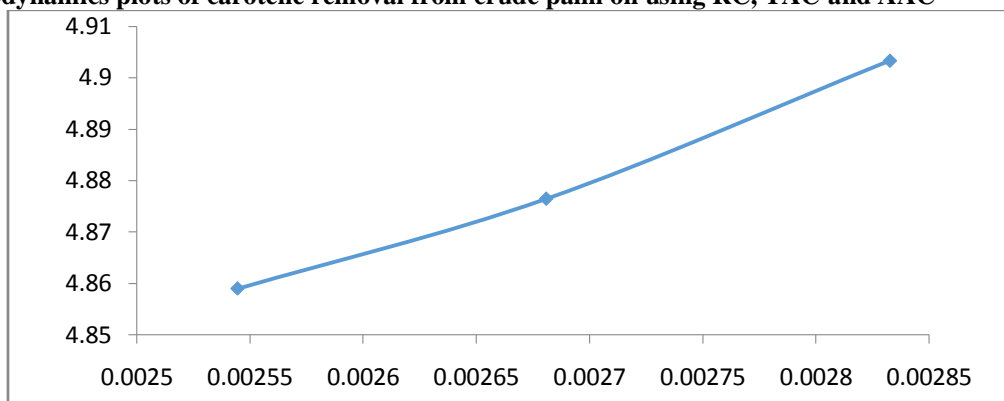


Fig 2.1: Thermodynamics plot for carotene removal using RC

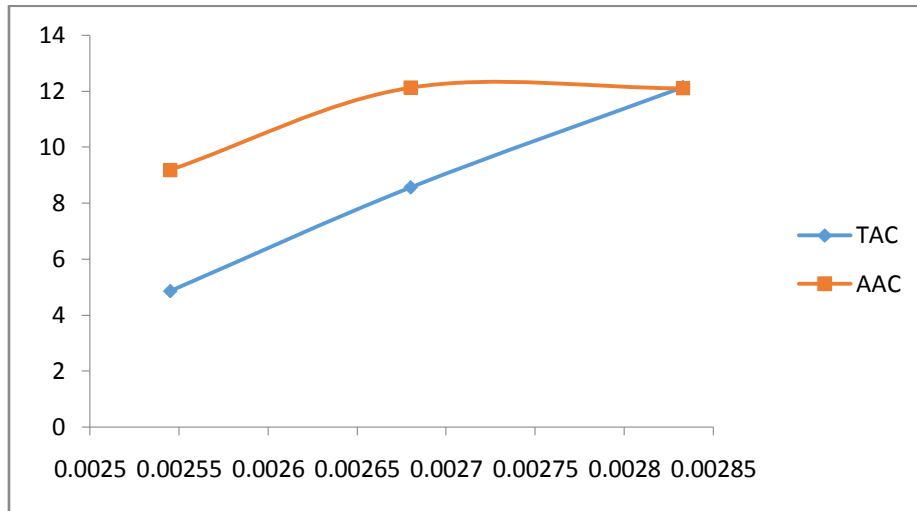


Fig 2.2: Thermodynamics plot for carotene removal using TAC and AAC

Thermodynamics plots of phosphorus removal from crude palm oil using RC, TAC and AAC

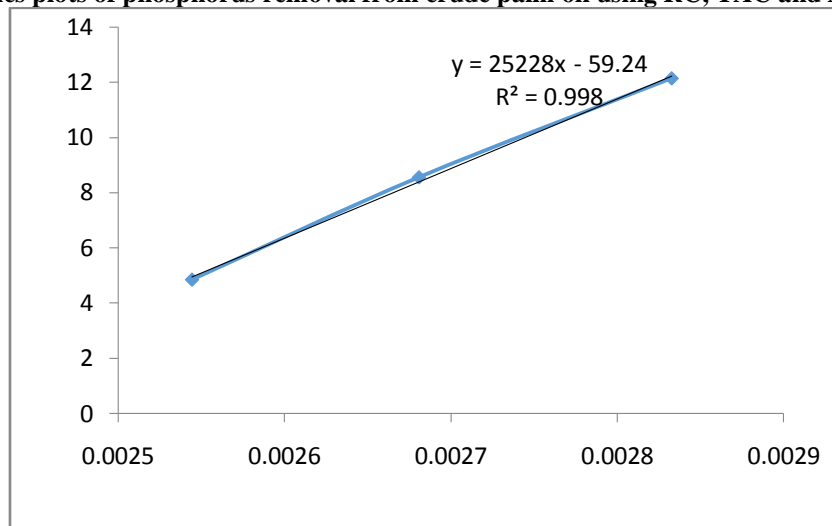


Fig 2.3: Thermodynamics plot of phosphorus removal from crude palm oil using RC

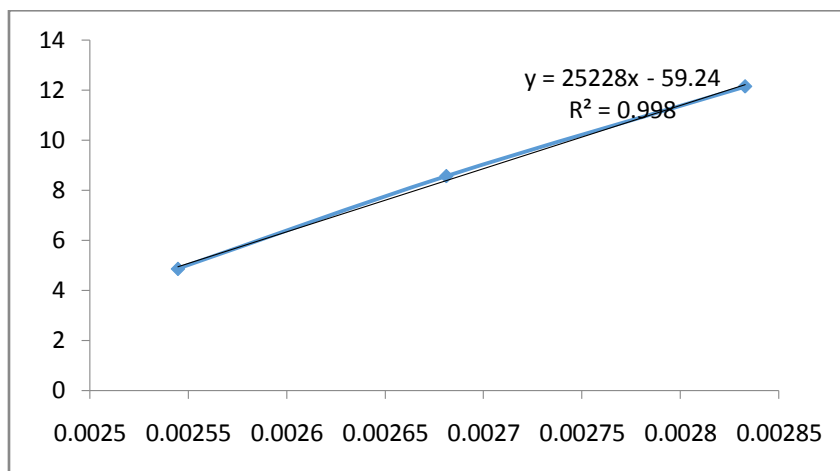


Fig 2.4: Thermodynamics plot of phosphorus removal from crude palm oil using TAC

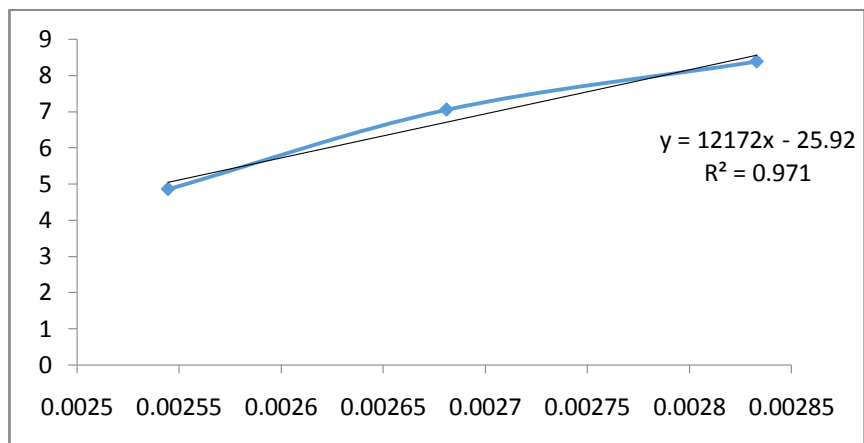


Fig 2.5: Thermodynamics plot of phosphorus removal from crude palm oil using AAC

The thermodynamics plots for carotene removal were presented in Fig 2.1 to 2.2 present, while Fig 2.3 – 2.4 showed the plots for phosphorous removal. The Gibb’s free energy (ΔG°) values were found to be negative in all the cases investigated.

Instrumental Characterization

Elemental analysis of RC, TAC and AAC

The elemental analysis of RC, TAC and AAC were obtained using X-ray Fluorescence Spectroscopy (XRF).The results indicated the chemical composition of RC, TAC and AAC in Table 3.1 and 3.2.

Table 3.1 Chemical Composition of Aguleri Raw Clay

ELEMENT NUMBER	Element Symbol	Element Name	Atomic Conc.	Weight Conc.
14	Si	Silicon	53.20	47.73
13	Al	Aluminum	27.92	24.06
26	Fe	Iron	11.60	20.69
22	Ti	Titanium	1.50	2.30
12	Mg	Magnesium	2.74	2.13
20	Ca	Calcium	0.93	1.19
11	Na	Sodium	1.41	1.04
19	K	Potassium	0.69	0.86

Table 3.2 Chemical composition of TAC

Element Number	Element Symbol	Element Name	Atomic Conc.	Weight Conc.
14	Si	Silicon	45.94	42.91
13	Al	Aluminum	39.83	35.74
26	Fe	Iron	6.75	12.54
22	Ti	Titanium	1.08	1.72
12	Mg	Magnesium	2.11	1.71

19	K	Potassium	0.76	0.99
40	Zr	Zirconium	0.32	0.97
20	Ca	Calcium	0.69	0.92
11	Na	Sodium	1.06	0.81
16	S	Sulfur	0.75	0.80
15	P	Phosphorus	0.44	0.46

Table 3.3 Chemical composition of AAC

Element Number	Element Symbol	Element Name	Atomic Conc.	Weight Conc.
14	Si	Silicon	50.35	45.27
13	Al	Aluminum	24.80	21.42
26	Fe	Iron	10.26	18.35
16	S	Sulfur	7.82	8.03
12	Mg	Magnesium	2.30	1.79
22	Ti	Titanium	1.16	1.78
20	Ca	Calcium	0.83	1.06
11	Na	Sodium	1.20	0.89
15	P	Phosphorus	0.73	0.72
19	K	Potassium	0.55	0.69

The elements, periodic number, symbol, atomic concentration and concentration by weight are shown in Table 3.1 to 3.3. The high content of Si, Al and Iron in Table 3.3 showed that these three elements were the major constituents of RC. S, Mg, Ti, Ca, Na, P and K were in trace values. The effect of modifications (thermal and acid modification) can be observed by increase in trace elements and variation in major components of RC. These changes must have resulted from bond breaking and bond formations that occurred during the thermal and acid interactions with the RC Table 3.2 and Table 3.3. The increase in Si content can be explained by the depletion of the interlayer and octahedral cations after acid activation (Siddiqui et al, 1996). The elemental make presented above supports the report of Richard, 2005, that majorly kaolinite are composed of silicate sheets bonded to aluminium oxide called gibbsite.

III. CONCLUSION

This study has demonstrated that the carotene and phosphorus content of crude palm oil can be successfully reduced using Aguleri clay as adsorbent. It was established that thermal and acid modification of the clay improved their adsorptive nature by increasing the clays surface area. Thermal and acid modification of Aguleri clay significantly improved its nature as adsorbent in removal of phosphorus. However, it did not show much significant effect on carotene removal. The phosphorus and carotene removal performance of acid activated clays (AAC) in general was found to be considerably better than that of the raw and thermal activated clays (RC and TAC). It was established that carotene and phosphorus removal depends on temperature, time and slightly on adsorbent dosage.

In addition, carotene and phosphorus removal experimental data accurately followed pseudo-first-order and the pseudo-second-

order kinetic models. Intra-particle diffusion was involved in carotene and phosphorus adsorption process, although it is not the sole rate limiting step. The equilibrium data were described more accurately by Langmuir and Freundlich models, for β -carotene and phosphorus, respectively. A thermodynamic study demonstrated that β -carotene and phosphorus adsorption is spontaneous, endothermic and an entropy-driven process. High temperatures are better for adsorptive removal of carotenes and phosphorus.

Adsorption of carotene and phosphorus from crude palm oil onto RC, TAC and AAC was successfully optimized using RSM. The optimal responses of 57.46%, 54.16% and 40.25%, for carotene removal were obtained for RC, TAC and AAC respectively. For phosphorus removal, 75.55%, 90.01 and 92.20 were obtained for RC, TAC and AAC respectively. Experimental validation had good correlation with the optimized values.

It can therefore be concluded that Aguleri clay can be a good adsorbent for carotene and phosphorus removal. Also, from the result in line with Gibon et al., 2007 that complete removal of carotenes during adsorption process is not possible and also not necessary since these components are not heat stable and can be degraded during high temperature deodorization.

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