

Optimization of Acid Activation Conditions for Athi River Bentonite Clay and Application of the Treated Clay in Palm Oil Bleaching

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Abstract: Athi River bentonite clay was activated using sulphuric acid at various acid concentrations, clay-acid ratio, temperature and contact times. Optimization was based on palm oil bleaching performance. The chemical compositions of the raw clay and clay activated at different acid concentrations were analysed to investigate the extent of cation dissolution and exchange. From the results, the clay was classified as calcium bentonite. Both cation exchange capacity (CEC) and the apparent bulk density were found to decrease with increase in acid concentration. When palm oil was bleached at 90°C for 30 minutes using 1% and 4% clay activated with 2M sulphuric acid, the bleaching performances obtained were 94.5 and 98% respectively. These results compared well with those of a commercial bleaching clay which was used as standard. The equilibrium data was analysed using Freundlich and Langmuir adsorption isotherms and the former was found to provide a better fit for the data. The calculated heat of adsorption indicated that palm oil bleaching by the activated clay was an exothermic reaction and a physico-chemical process. Sulphuric acid activation was found to be an effective method of improving adsorption performance of Athi River bentonite clay and the activated product is a viable alternative to imported commercial bleaching earth.

Keywords: Acid activation, Bentonite clay, Oil bleaching, Adsorption isotherm

I. Introduction

The term bentonite was first used to designate a particular highly colloidal, plastic clay found near Fort Benton in the Cretaceous beds of Wyoming, USA [1]. Later the term was attributed to every plastic clay generated by the alteration of organic ash or glass emitted from volcanoes [2, 3].

Bleaching is an adsorption process in oil refining and the most critical stage since it helps to improve the appearance, flavour, taste and stability of the final oil products by removing coloured and colourless pigments from the oil [4, 5, 6].

Activated bleaching earth (ABE) is the most common adsorbent for oil purification. Activated carbon and silica-based products have also been investigated for removal of pigment and impurities from oil [7]. Oil industries in Kenya use imported bleaching earth in spite of the fact that the raw material for this product is the abundantly available bentonite clay. This clay is found in many parts of the country including Athi River, Amboseli, Timau, Nanyuki, Meru and Namanga among others [8, 9]. Most bentonites found in nature need to undergo appropriate physical or chemical treatments such as acid activation, ion exchange and heating in order to increase surface properties, adsorption capacity, and meet a range of other application requirements [8, 10, 11, 12]. Ajemba and Onukwuli [13] observed that activated clay adsorb colour pigments more than inactivated clay and concluded that modified clay minerals have a high potential of serving as an alternative to the most widely used and high-cost activated carbon. Farihahusnah *et al.*, [6] reported that the high bleaching performance of bleaching earth depends on activation conditions such as acid concentration, contact time and the temperature of the activation process, all of which contribute to structural modification.

Many studies have reported successful bleaching of oil using acid activated clays from across the world [5, 14, 15] but few have been carried out using local Kenyan clay [16]. The purpose of this study was to characterize Athi River bentonite clay and to investigate optimum conditions for acid activation and subsequently compare the activated clay with commercial bleaching earth. Bleaching performance was based on palm oil because it is the most widely used vegetable oil in Kenya. Besides developing a cost effective method for improving adsorbent properties of local clay, commercial exploitation of the clay was expected to increase income, reduce imports and contribute towards food self-sufficiency.

II. Experimental Section

The clay was obtained from Athi-River Mining Co. Ltd. Kenya. Crude palm oil was bought from Kapa Oil Refineries Ltd. All reagents used were analytical grade and were used without further purification.

Experiments were performed in duplicate. The clay was washed with plenty of distilled water to remove contaminants. After drying in the oven at 105°C, it was ground to pass through 200 mesh sieve. To determine the appropriate acid concentration, 5g of clay was activated at 100°C for 3 hours under reflux with constant stirring using 50ml of 1M H₂SO₄. The mixture was filtered under vacuum and washed repeatedly with distilled water until the filtrate was free of sulphate ions [12]. The sample was dried at 105°C and ground to a particle size that passed through 200 mesh sieve. This procedure was repeated with 2M, 3M, 4M and 5M acid concentrations. The activated clay samples were labelled A1, A2, A3, A4 and A5 where the numbers in these labels referred to the molarities of the acid used. To study the effect of activation time and temperature, the above procedure was repeated with 5g clay in 50ml 2M sulphuric acid, varying time between 30 and 80 minutes and temperature between 100 and 120°C respectively. Clay-acid ratio was investigated with 5M sulphuric acid at 90°C. All samples of activated clay were stored in polyethylene bags for characterization and bleaching studies.

To evaluate the performance of the activated clays, the palm oil was degummed using 0.1% phosphoric acid at 80°C, stirred for 5 minutes, washed with distilled water and then centrifuged to remove the phosphatides. To 10ml of the degummed oil at 70°C, 0.1g activated clay was added and the temperature was raised to 90°C under a stream of nitrogen. The oil was cooled to 70°C and then vacuum-filtered. Bleached oil absorbances were read at 450nm using UV-Vis spectrometer (UV-100). Clay activation under conditions that gave best performance with 1% activated clay, was repeated with 0.05, 2, 3, and 4% clay. Similar experiments were carried out with same dosages of a commercial bleaching earth used locally. Bleaching temperature was also varied between 70 and 100°C. Raw and activated clays were analysed for chemical composition and cation exchange capacity (Varlan Spectra AA-10 spectrophotometer) [17], moisture [18], pH (pH 211, Hannah Instruments), apparent bulk density [19]. Bleached oil was analysed for free fatty acids [20, 21, 22], density (pycnometer method), oil retention [18], copper and lead (AA Spectroscopy).

III. Results and Discussion

3.1 Effect of acid concentration on clay composition and structure

Acid treatment was observed to modify the structure of bentonite as indicated by the change in the chemical composition (Table 1). Except for SiO₂ whose composition rose from 46.71% to 78.52 % as acid concentration was increased, the composition of other components decreased after activation. The increase in SiO₂ content and a concentration decrease for other oxides was in agreement with the results reported by Usman *et al* [18] and Arfaoui *et al* [23]. According to Diaz and Santos [15], the octahedral sheet destruction passes the exchangeable cations into the solution while the silica generated by the tetrahedral sheet remains in the solid phase due to its insolubility. The ratio of Na₂O to CaO was 0.92, a value less than 1, which is indicative of the absence of swelling bentonite [3, 18]. Therefore, the clay was classified as calcium bentonite.

Table 1: Chemical composition and loss on ignition (LOI) of raw and acid activated Athi River bentonite clay, and commercial bleaching earth (CBE)

% Oxide content	Raw clay	Athi River bentonite clay activated with H ₂ SO ₄					CBE
		1M	2M	3M	4M	5M	
SiO ₂	46.71	60.03	69.45	74.8	78.52	78.34	62.89
Al ₂ O ₃	15.96	13.75	9	6.8	5.31	4.13	12.68
CaO	4.09	0.57	0.58	0.6	0.83	0.63	0.457
MgO	1.53	0.4	0.33	2.7	0.5	3.6	1.212
Na ₂ O	3.8	0.98	1	1.09	1.12	1.17	0.397
K ₂ O	1.35	1.16	1.23	1.3	1.2	1.17	0.778
TiO ₂	0.65	0.49	0.12	LD	LD	0.82	0.492
MnO	0.6	0.4	0.2	0.2	0.3	0.2	LD
Fe ₂ O ₃	7.86	4.09	1.37	0.71	0.36	0.22	3.742
LOI	14.56	17.58	16.58	11.26	10.42	9.39	17.25

Note: LD means the oxide content was below the detection limit.

It was observed that at first, the moisture content increased with increasing acid concentration. The acid attack increased the capacity of the clay to hold water. Subsequently, the moisture content decreased as the acid concentration was further increased to 3M and above. This could be attributed to the increase in porosity after partial loss of the exchangeable metal ions by hydrogen ions which have smaller volumes than the metal ions. The cation exchange capacity (CEC) was decreased by acid treatment, but at 5M, the CEC was observed to

increase. A similar trend was observed by Ajemba and Onukwuli [13]. The apparent bulk density decreased to a minimum of 0.45 at 5M acid concentration. These results are shown in Table 2 and Fig. 1.

Table 2: Moisture, CEC and apparent bulk density of inactivated clay and clay activated at various acid concentrations

Molar concentration of sulphuric acid	Inactivated clay	1M	2M	3M	4M	5M
Moisture content (wt%)	5.69	7.58	7.18	3.45	3.21	2.38
CEC (10^2 cmol/Kg)	1.19	0.8735	0.5926	0.5846	0.3925	0.4717
Apparent bulk density (g/ml)	1.025	0.75	0.6	0.5	0.46	0.45

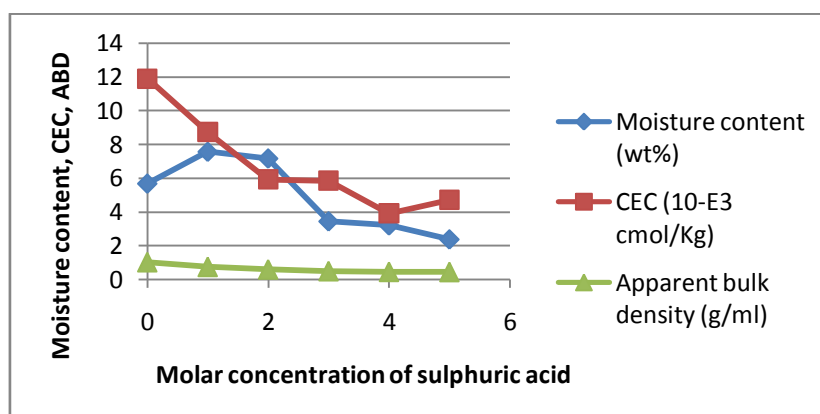


Figure 1. Effect of acid concentration on moisture content, cation exchange capacity and apparent bulk density of clay (bleaching conditions: 1% activated clay, 90°C, 30 minutes)

3.2 Effect of activation acid concentration on bleaching performance

The results are presented in Fig. 2. (A1 stands for Athi River clay activated using 1M sulphuric acid). The bleaching performance first increased with the concentration of acid used up to 2M H_2SO_4 and also with bleaching time up to 40 minutes. The maximum value of bleaching performance (94.54%) was attained at 2M acid concentration after 30 minutes. The initial increase in bleaching performance with increasing sulphuric acid concentration was probably due to formation of active sites on bentonite surface. Subsequent decline in bleaching performance could be due to passivation of the rest of the clay, thus protecting the clay layers from further acid attack [24].

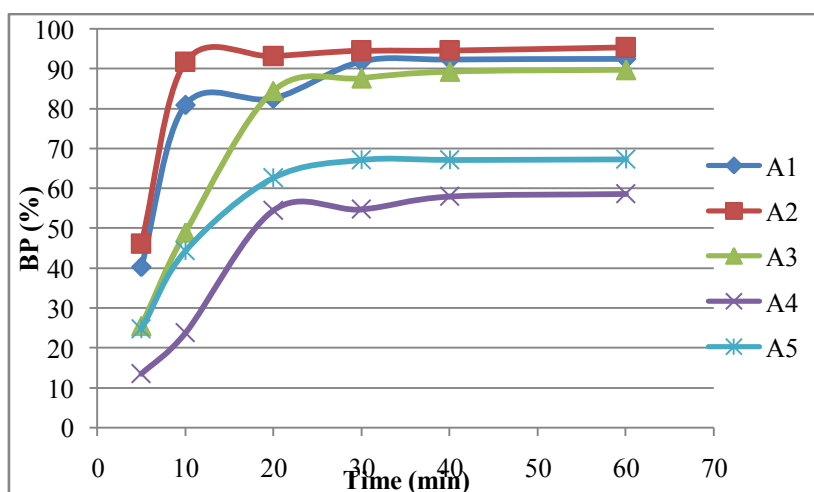


Figure 2. Effect of acid concentration and bleaching time on palm oil bleaching performance with 1% activated Athi River bentonite clay.

3.3 Effect of clay-acid ratio

The effect of clay-acid ratio was studied at 100°C, with 5M H_2SO_4 , for 3 hours. The results are shown in Fig. 3.

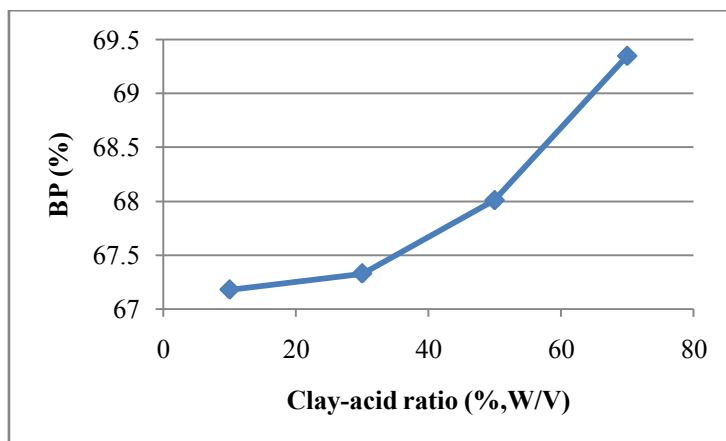


Figure 3. Effect of clay-acid ratio on bleaching performance of 1% activated Athi River bentonite clay

For this excess acid concentration found earlier on to perform poorly, bleaching performance was observed to slightly improve with increase in clay-acid ratio. When the ratio was increased from 10 to 70% w/v, bleaching performance increased from 67.18 to 69.35, an increase of about 2% implying a small effect on beneficiation of bentonite clay obtained at the expense of increased viscosity due to elevated high clay-acid ratio.

3.3 Effect of activation on temperature

The effect of temperature on activation of Athi River bentonite was investigated with 2M H₂SO₄, 1:10 clay-acid ratio (w/v). The results of bleaching oil at 90°C for 30 minutes using 1% clay from samples activated at 100°C and 120°C are shown in Fig. 4.

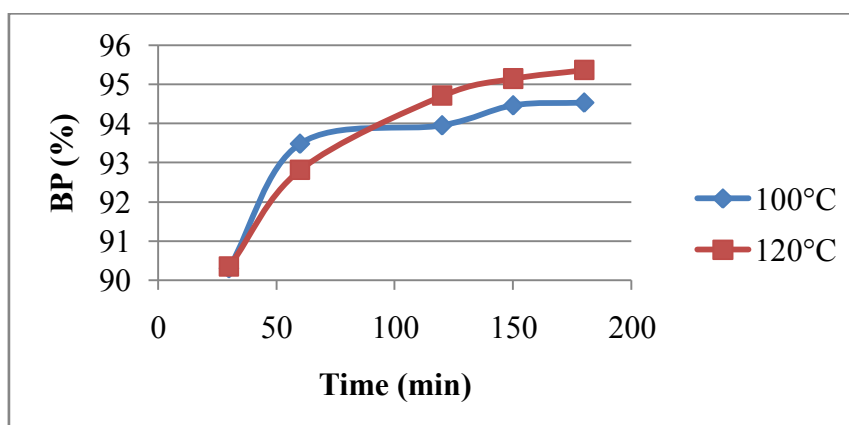


Figure 4. Effect of activation time and temperature on bleaching performance (1% clay at 90°C for 30 minutes)

The above figure shows that clay acid activation was improved by increasing temperature. This was probably due to increased speed of the molecules in the system which enhanced contact between clay surface and the acid.

3.4 Effect of acid activation on clay colour

As shown in Fig. 5, the colour of the clay changed from yellowish-brown to dirty white after activation at optimized conditions.



Figure 5. Raw and activated Athi River (A) bentonite clay

3.5 Optimum bleaching conditions

3.5.1 Bleaching temperature

As shown in Fig. 6, the % bleaching performance (BP) increased with temperature to a maximum at 90°C followed by a drop except for sample A1 which did not show any maxima. The highest bleaching performance was obtained at 90°C for clay activated using 2M acid (Sample A2). There was a significant drop in performance for A3 and A4 (3M and 4M acid). After the drop, the performance remained constant. For A3, performance rose to 82% at 90°C before dropping to 65% at 120°. Sample A2 reached a maximum bleaching performance at 90°C, thus performing better than commercial bleaching earth. The results are in agreement with those of Berbesi [25], who observed that bleaching temperature typically ranges between 90 and 125°C. He also noted that oil viscosity decreases with increasing temperature resulting in better dispersion of bentonite clay particles, improved clay-oil interactions and less resistance to flow. The improved percentage pigment removal with increase in temperature was probably because of increased kinetic energy of the molecules leading to more adsorption. Adsorption reactions are generally exothermic [26] hence subsequent drop in bleaching performance with increase in temperature.

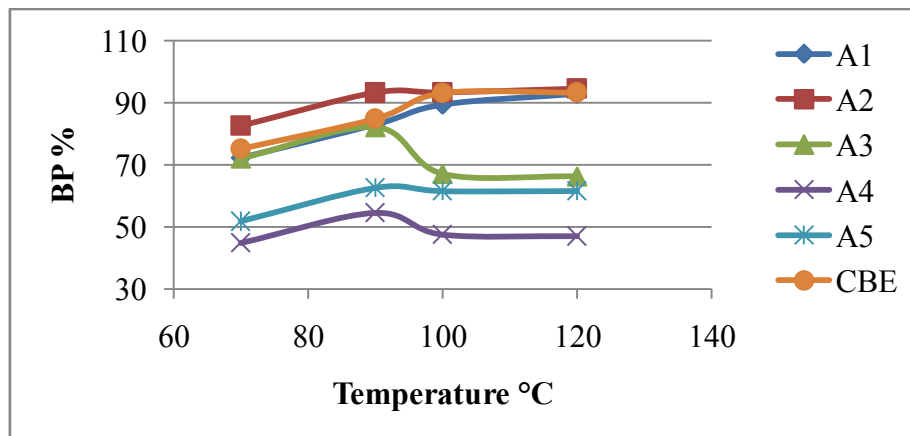


Figure 6. Effect of bleaching temperature on performance (palm oil, 1% clay at 90°C for 30 minutes, Athi River bentonite and commercial bleaching clay (CBE)).

3.5.2 Effect of contact time on bleaching performance

The results are shown in Fig. 7. Colour removal from palm oil increased with contact time. The rapid pigment adsorption in the first 5 to 20 minutes can be attributed to the increased availability of vacant surface sites at the initial stages. The optimal contact time was a maximum at 30 minutes for the various acid concentrations and this is in agreement with the results of other workers [5, 25, 27]. After 30 minutes, the adsorption was slow, implying equilibrium conditions had been attained.

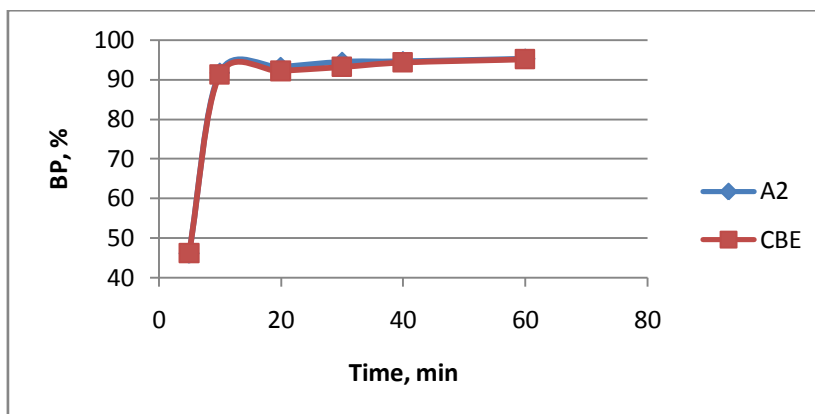


Fig. 7. Effect of bleaching time on bleaching performance (palm oil, 1% clay at 90°C, Activated Athi River bentonite (A2) and commercial bleaching clay (CBE))

The effect of contact time on bleaching of palm oil with commercial bleaching earth (CBE) was studied for comparison with the activated local bentonite clay. It is seen from Fig. 7 that although the bleaching curve for CBE is similar to that of activated Athi River bentonite sample A2 the latter performed slightly better. Therefore, this local activated clay can be used to bleach crude vegetable oil in place of imported bleaching earth.

3.5.3 Effect of dosage on bleaching performance

Fig. 8 shows that the bleaching performance increased to a maximum with increasing activated clay dosage and this can be attributed to the increase in available adsorption active sites. The dosage was varied between 0.5 and 4%. The bleaching performance of A2 increased from 40.32% to 98.49% and that of CBE from 46.8% to 97%. It increased to an optimum value with 1-2% adsorbent dosage after which further increase in dosage had only a little effect on performance. Diaz and Santos [15] observed that darker oils like palm oil require as much as 2-4% activated clay or more to meet the colour specifications. Nwabanne and Ekwu [28] reported the increase of BP with increase in clay dosage for decolourization of palm oil using some Nigerian local clay.

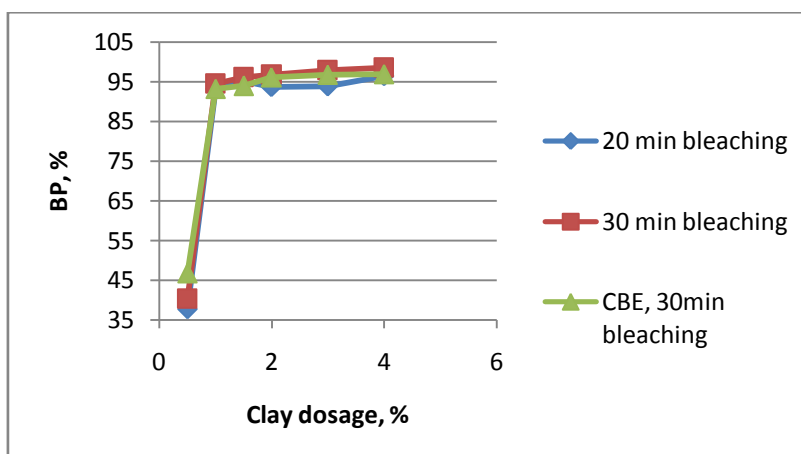


Figure 8. Effect of dosage on bleaching performance at 90°C

3.5.4 Adsorption isotherms

The adsorption process data were fitted to Freundlich and Langmuir isotherms (equations 1 and 2 respectively).

$$\log \frac{x}{m} = \log K + n \log c \quad (1)$$

where x is the amount of substance adsorbed, m is the quantity of adsorbent and c is the quantity of residual substance dissolved. K and n are constants unrelated to the amount of solute and adsorbent.

$$\frac{x_e}{x/m} = \frac{1}{a} + \frac{b}{a} x_e \quad (2)$$

where a and b are Langmuir constants, and are evaluated by plotting $x_e/(x/m)$ against x_e .

As has been discussed earlier, as temperature was increased, the amount of pigments adsorbed on clay surface increased. According to Ajemba *et al.* [28], this change occurs as a result of increased kinetic energy of the colour pigment particles, which in turn increases the frequency of collisions between the adsorbent and pigment particles and hence enhancement of adsorption on the surface of the adsorbent. Fig. 9 and 10 show the Freundlich and Langmuir isotherms for the bleaching of palm oil using the activated bentonite clay and commercial bleaching earth respectively. Freundlich isotherm gave a better fit than the Langmuir isotherm as can also be seen in Table 3.

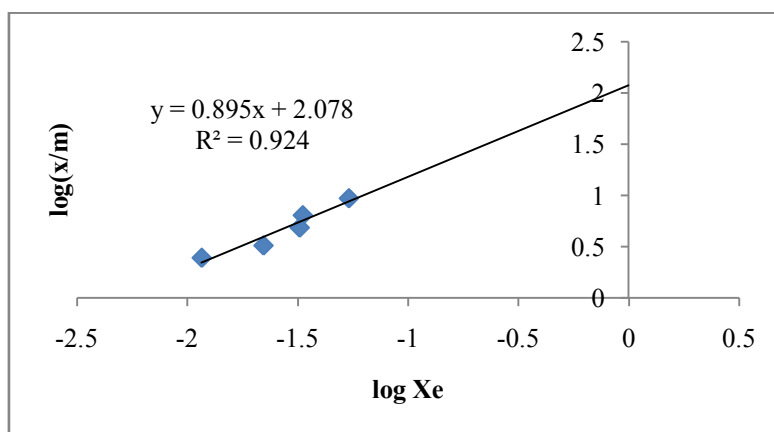


Figure 9. Freundlich adsorption isotherm for palm oil bleaching using clay activated with 2M sulphuric acid (Sample A2)

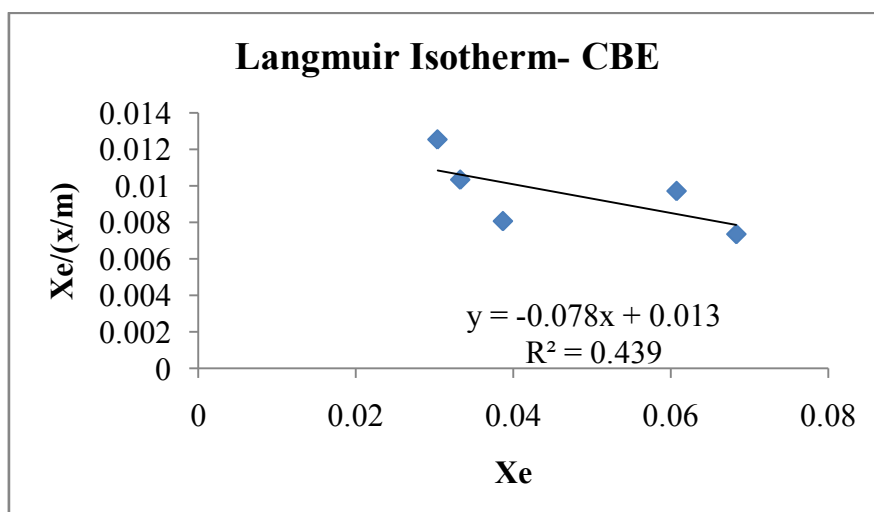


Figure 10. Langmuir adsorption isotherm for palm oil bleaching using commercial bleaching earth

Table 3: Freundlich and Langmuir isotherm constants for the bleaching of palm oil with activate Athi River clay and CBE

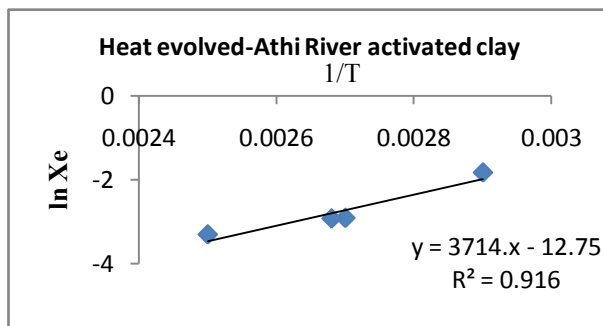
	Parameter	Athi River (A) bentonite clay	Commercial bleaching earth (CBE)
Freundlich	k	119.89	370.68
	n	0.8959	1.399
	R ²	0.9247	0.9175
Langmuir	a.	185.18	75.75
	b	2.35	-5.97
	R ²	0.0481	0.4393

3.5.6 Heat evolved during the bleaching process

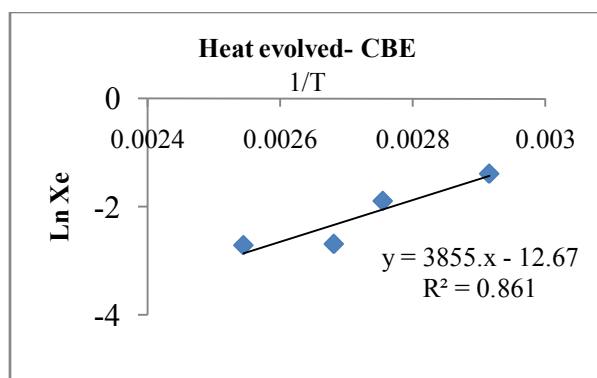
The heat evolved during the adsorption process was calculated using the following equation [29].

$$\ln X_e = \frac{-\Delta H_a}{RT} + C \quad (3)$$

where X_e is the residual amount of pigment at equilibrium, ΔH_a is the heat of adsorption (kJ/mol), R is the gas constant (8.314 J/mol K), T is the temperature (K), and C is the integration constant obtained from the plot of $\ln X_e$ against $1/T$ as shown in Fig.11.



(a)



(b)

Figure 11. Plot of $\ln X_e$ against $1/T$ for sample A2 (a) and CBE (b)

The heat evolved was evaluated from Equation 7 and Fig. 11 and found to be -30.881kJ/mol for A2 and -32.05kJ/mol for CBE. The negative value implies that the adsorption of pigments from palm oil onto bentonite clays is an exothermic process. ΔH_a value also indicates the type of sorption. The heat evolved during physical adsorption falls in the range of 2.1 to 20.9kJ/mol while that evolved during chemical adsorption is in the range of 80 to 200kJ/mol [29]. Therefore, since the values obtained lie between the upper value for physical adsorption and the lower value for chemical adsorption, the bleaching of palm oil by both activated Athi River clay and CBE is a physico-chemical adsorption process.

The specifications of the optimized activated bentonite clay and that of commercial bleaching earth are shown in Table 4. The oil retention for Athi River bentonite clay activated at 100°C with 2M H_2SO_4 and for CBE was determined, and found to be 27.73% and 27.04% respectively. This is in agreement with the observation made by Rich [19], who found that clay with higher apparent bulk density value retain less amount of oil. In addition, according to Usman *et al* [18], this value meets commercial standards. The free fatty acid content was significantly decreased by the activated clay from 6.68 to 0.268 % while commercial bleaching clay showed no effect on this parameter. Therefore the activated clay has capacity to not only bleach the oil but also to reduce the free fatty acids. In Fig. 12, the crude and bleached palm oils are shown and it is quite obvious that the bleaching process was successful.

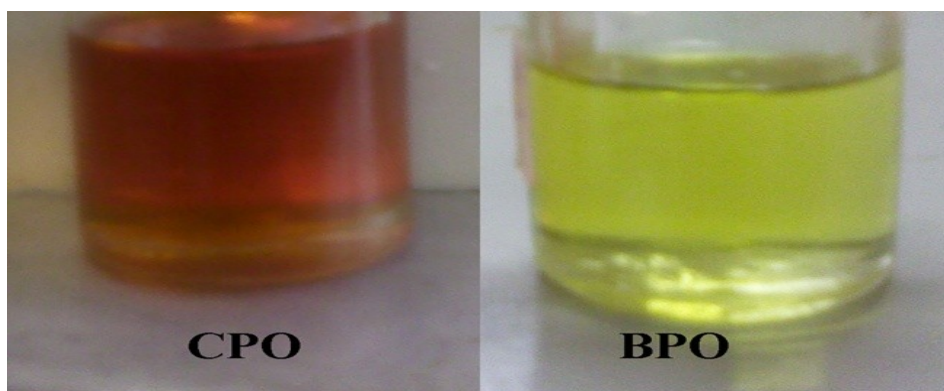


Figure 12. Unbleached (Crude palm oil) and bleached palm oil (BPO)

Table 4: Quality characteristics of the raw and activated Athi River bentonite clays, and commercial bleaching earth.

Clay analysis	Raw Athi River bentonite	Athi River bentonite activated with 2M H ₂ SO ₄	Commercial bleaching earth
Colour	Yellowish-brown	Dirty white	Dirty white
Particle size	200 mesh	200 mesh	200 mesh
pH (10% in water)	10.7	4.35	3.8
Apparent bulk density g/l	1024.97	600.68	715.81
CEC 10 ² cmol/kg	ND	0.5926	ND
Bleached Palm oil analysis			
FFA content %	6.68	0.268	6.68
Oil retention %	ND	27.73	27.04
Moisture content %	5.69	7.18	8.3
Cu ppm	ND	470	465
Pb ppm	ND	1.0	1.4

ND: not determined

IV. Conclusion

In this study, activated Athi River bentonite clay was assessed for its potential in bleaching of palm oil. Chemical composition allowed classification of the clay as ‘calcium bentonite’. Acid activation at optimized conditions was found to significantly alter the clay structure and consequently improve the bleaching performance of the clay which compared well with that of the commercial bleaching earth. Furthermore, the activated clay was found to reduce free fatty acids from crude palm oil, unlike the commercial clay which had no effect. This study has shown that Athi River bentonite clay is a viable source of adsorbent for palm oil bleaching and a feasibility study for its exploitation is hereby recommended.

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