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Research Note

Laboratory Trials on Bleaching Palm Oil with Selected Acid-activated Nigerian Clays

ABSTRACT

The optimum conditions for acid activation of some selected Nigerian clays were determined. The bleaching potential of the activated clays on crude palm oil relative to a commercial Fuller's earth (Fulmont 700C) were assessed.

Bleaching activity was highest when 1 part of clay of particle size < 180 μm was mixed with 3 parts of 1·4–2·7M HCl or 0·5–1·0M H_2SO_4, refluxed for 2–6 h, dried to between 5–15% moisture content, milled and sifted to obtain a particle size of <90 μm. Clays from Ewekoro and Shagamu in southern Nigeria, when properly activated using the wet slurry method, compared favorably with the commercial Fuller's earth in bleaching activity. The Nigerian clays, however, showed lower apparent bulk densities and, consequently, a corresponding higher oil retention.

INTRODUCTION

Palm oil is probably the most versatile of natural fats. Its wide range of food applications has recently been reviewed (Berger, 1986). Surpassed only by Malaysia and Indonesia, Nigeria is the third largest producer of palm oil, accounting for 13% of world production (Wood & Beattie, 1981). However, with current production at about 700 000 tonnes of palm oil per annum, there is still a wide gap between domestic vegetable oil demand and supply in Nigeria, resulting in yearly upward trends in vegetable oil imports, most of which is refined palm oil from the Far East (Ataga, 1984). In accordance with government policy to diversify export from its present heavy reliance on crude petroleum, local palm oil production is being stimulated in Nigeria.

Commercial vegetable oil refining depends on the use of activated clays

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(Rich, 1964; Siddiqui, 1968*a*). Their function in the processing of palm and palm kernel oils has recently been reviewed (Morgan *et al.*, 1985). Nigeria's increasing dependence on refined palm oil and the nation's declining foreign exchange earnings emphasize the need to exploit the country's numerous clay mineral deposits for the production of bleaching earths. Preliminary studies suggested that clays from Nkalagu, Nsukka, Ewekoro, Shagamu and Anaku, all in Southern Nigeria, with relatively high montmorillonite content were suitable for bleaching earth preparation (Oboh *et al.*, 1987). The present study was undertaken to determine the optimum conditions for activating these clays and to assess their performance, relative to Fuller's earth, when used to bleach palm oil.

MATERIALS AND METHODS

Crude palm oil was obtained from a modern palm oil mill at the Nigerian Institute for Oil Palm Research, Benin City. Fuller's earth (Fulmont 700C) was obtained from Laporte Inorganics, Cheshire, Great Britain.

Pilot experiments

A series of pilot experiments was conducted to determine the optimum conditions for activating clays from Nkalagu, Nsukka, Ewekoro, Shagamu and Anaku, all in Southern Nigeria. For each experiment, a 5-kg sample of each clay was collected as previously described (Oboh *et al.*, 1987). The clays were dried in an oven at 105–110°C for 1–2 h, milled and sifted with a 180 μm standard Canadian Tyler sieve No. 80. 50-g samples were mixed with various quantities (100–500 ml) of HCl (0·3–8·2M), H_2SO_4 (0·1–5·1M), acetic acid (0·2–8·3M) and refluxed by boiling for various durations (1–6 h). Following cooling, the samples were washed free of acid as indicated by litmus, filtered, dried to various moisture contents (0–30%), milled and sifted with standard Canadian Tyler sieves, Nos 80–250, to obtain particle sizes of <63–$180\,\mu$m.

The bleaching power of the activated clays was assessed by mixing 5 g of clay with 100 ml of crude palm oil and heating, with constant stirring, to 105°C and maintaining at this temperature for 1 h. The slurry was then filtered to remove the clay and the colour of the oil was measured with a Lovibond tintometer using a 1 in cell. Fulmont 700C, a commercial Fuller's earth, was used as reference. The per cent colour reduction was calculated using the equation of Krishnan (1975):

$$\% \text{ colour reduction} = \frac{100 \times (10R + Y) - (10R_1 + Y_1)}{(10R + Y)}$$

where $10R + Y$ = total colour of crude oil and $10R_1 + Y_1$ = total colour of bleached oil.

Physico-chemical analysis of clays

pH and titratable acidity were determined by soaking 5 g of clay in 100 ml of distilled water for at least 12 h. After thorough mixing, the pH of a 25 ml aliquot was measured with a Metrohm-Herisau pH meter model E-520, and the titratable acidity was estimated by titration against standard 0·1M NaOH using 1% phenolphthalein as indicator. Moisture was measured directly at 105°C with a Buhler moisture tester model HLT 400. Apparent bulk density (ABD) was estimated as described by Rich (1960). Oil retention was calculated from ABD: % oil retention (% basis dry cake) = ABD − 90 (Siddiqui, 1968*b*).

Assessment of bleaching power

Based on the results of the pilot experiments, Ewekoro brown, Ewekoro grey, Shagamu brown and Shagamu grey clays were selected for further studies. The bleaching power of the activated clays was assessed as described under 'pilot experiments' using various clay doses (2–10%), various temperatures (95–140°C) and duration (10–60 min).

RESULTS AND DISCUSSION

In general, irrespective of the source of clay, the best results, in terms of bleaching efficiency, were obtained when one part of clay of particle size $< 180\,\mu$m was mixed with three parts of 1·4–2·7M HCl or 0·5–1·0M H_2SO_4, refluxed for 2–6 h, dried to 5–15% moisture and sifted to obtain a particle size of $< 90\,\mu$m. Typical results on the effect of type and concentration of acid used for activation on bleaching activity are shown in Table 1. Nigerian clays treated with HCl or H_2SO_4, commonly used as activating agents (Mansfield, 1936; Siddiqui, 1968*a*), were more effective in bleaching palm oil than those treated with acetic acid. Acid treatment increases bleaching activity of clays by replacing exchangeable Al^+ and other metallic ions by H^+ in the interlamellar space. In addition, mineral salt constituents are leached from the lattice structure, thus rendering the clay physically more porous and electrochemically more active (Rich, 1964; Siddiqui, 1968*a*). Bleaching power of clays treated with HCl or H_2SO_4 increased with concentration of acid to a maximum and declined thereafter (Table 1). This is presumably because excessive acid treatment may result in excessive

TABLE 1
Effect of Type and Concentration of Acid Used for Activation on Bleaching Power of Ewekoro Grey Clay, Using 5% Clay at 105°C for 1 Hour.

Acid		*Clay moisture content (%)*	*Lovibond colour reading (1 in)*	*Total colour*	*% Colour reduction*
Type	*Conc.* (M)				
H_2SO_4	5·1	9·91 ± 2·20[a]	26·0R + 18·0Y	278·0	9·91 ± 3·92
	3·4	11·55 ± 4·63	24·3R + 17·0Y	260·0	16·07 ± 1·92
	2·0	11·55 ± 2·31	9·8R + 11·4Y	109·4	64·06 ± 4·28
	1·0	11·02 ± 1·38	1·1R + 10·4Y	21·4	93·07 ± 0·47
	0·5	12·43 ± 2·55	5·2R ± 16·1Y	69·1	77·43 ± 2·24
	0·1	11·11 ± 1·62	12·6R + 13·3Y	139·6	54·80 ± 4·54
HCl	8·2	11·30 ± 1·36	21·4R + 17·6Y	231·6	24·91 ± 0·34
	5·5	13·22 ± 2·47	18·5 ± 15·0Y	200·0	35·26 ± 0·96
	2·7	13·03 ± 2·20	6·4R + 18·3Y	82·3	73·23 ± 1·55
	1·4	13·37 ± 2·52	0·9R + 12·7Y	21·7	92·99 ± 0·83
	0·3	11·06 ± 1·29	9·5R + 13·7Y	108·7	64·59 ± 1·24
CH_3COOH	8·3	12·06 ± 2·06	15·0R + 19·1Y	169·1	45·59 ± 1·74
	5·0	12·87 ± 1·50	21·2R + 16·2Y	228·2	26·16 ± 3·61
	3·3	12·78 ± 2·08	17·1R ± 17·1Y	188·2	39·08 ± 2·83
	1·7	13·03 ± 2·25	18·7R + 21·2Y	208·2	32·41 ± 3·72
	0·8	13·49 ± 1·41	22·7R + 20·8Y	247·8	19·83 ± 5·28
	0·2	11·79 ± 2·88	24·4R + 17·2Y	261·2	16·22 ± 5·34

[a] Mean and standard deviation of triplicate samples.

leaching of basic clay constituents with consequent loss in selectivity (Siddiqui, 1968*a*,*b*).

Under optimum activation conditions, clays from Ewekoro and Shagamu produced a colour reduction of about 90% when used to bleach palm oil, whilst Nkalagu and Anaku clays were less effective. Consequently, further studies were restricted to Ewekoro and Shagamu clays. Laboratory-activated Nigerian clays were comparable with commercial Fuller's earth in pH (2·7–3·0), but lower in titratable acidity (0·22–0·48 mg NaOH/g for Nigerian clays; 0·68 for Fuller's earth) and moisture (9·0–11·2% for Nigerian clays; 14·8% for Fuller's earth). Relative to Fuller's earth, laboratory-activated Nigerian clays had lower apparent bulk densities (36·4–45·6 lb/cft for Nigerian Clays; 50·5 for Fuller's earth) and higher oil retention (45·1–53·6% for Nigerian clays; 39·5% for Fuller's earth). This is presumably because the wet activation procedure results in disintegration of clay minerals producing a light and fluffy product with a large amount of fines (Siddiqui, 1968*b*).

The bleaching activity of any clay is influenced by several factors, including the physico-chemical characteristics of the clay, the bleaching method, atmospheric versus vacuum, and conditions including clay dosage, bleaching temperature and time (Rich, 1964; Morgan *et al.*, 1985). Clay dosage, bleaching temperature and duration had marked effects on bleaching activity when crude palm oil was bleached with Fuller's earth or with laboratory-activated Nigerian clays. For each clay dosage, bleaching temperature and time, the data from all four Nigerian clays, Ewekoro brown, Ewekoro grey, Shagamu brown and Shagamu grey, were combined, since there were no significant differences amongst them. Bleaching activity increased with temperature from 95 to 140°C. At low bleaching temperature

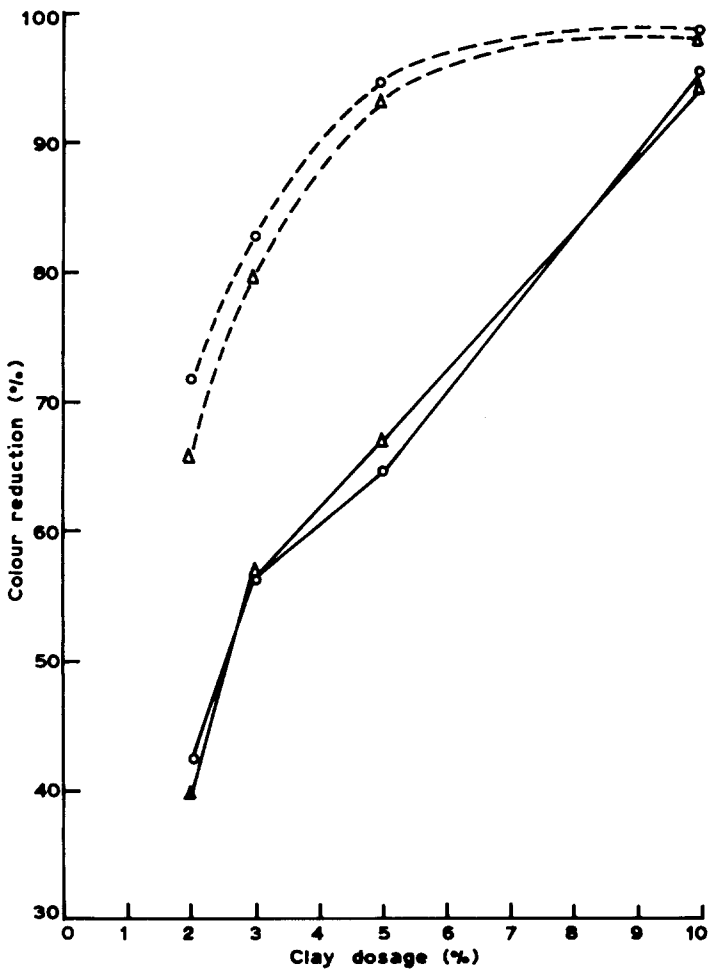

Fig. 1. Effect of clay dosage on the colour of palm oil bleached at 95°C (——) and 140°C (– – – –) for 30 min. ○, Fuller's earth; △, Nigerian clays.

(95°C), colour removal increased with time up to 60 min, whereas, at higher temperatures (110–140°C), it began to level off after 20–30 min, when a dose of 5% earth was used. For the same bleaching duration up to 30 min, larger clay doses were required to attain the same degree of colour removal at low temperatures as at higher temperature (Fig. 1). In general, laboratory-activated Nigerian clays were comparable with commercial Fuller's earth in bleaching activity. However, Fuller's earth was slightly more effective than the Nigerian clays in bleaching palm oil at higher temperatures, especially at low doses (Fig. 1). This presumably may be due to the fact that, in commercial practice, better control over activation conditions may be attainable than under laboratory conditions.

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