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## Assessing the bleaching capacity of some Cameroonian clays on vegetable oils

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## Abstract

Four Cameroonian clays, constituted mainly of smectites associated to minor amounts of kaolinite and quartz, have been used to bleach palm oil, cottonseed oil and maize germ oil. Kinetic studies show that the best temperature and contact time for optimum bleaching of vegetable oil varied with the type of oil; these are: 95 °C and 2.5 h for palm oil, 85 °C and 0.25 h for cottonseed oil, and 85 °C and 0.5 h for maize germ oil. The bleaching ability of natural clays was poor when compared with that of industrial adsorbents. Acid activation of Cameroonian clays with 1N sulphuric acid solution increased tremendously their adsorptive capacity, which therefore matches those of the industrial adsorbents. Further increase of the concentration of the acid solution used for clay activation did not induce a noticeable increase in the bleaching power of the adsorbents. This was attributed to the fact that the increase in the surface area is due to the formation of silica but the surface area of H-clays remains almost constant over strong acid leaching and silica has very poor bleaching power of vegetable oils. FT-IR spectroscopy studies of the adsorbents after bleaching suggest strong interactions between the clays and adsorbent surface. Acid and iodine values of bleached oils reveal that oils are not deteriorated by clays during the bleaching process. The overall results lead to the conclusion that Cameroonian clays can be converted into potent adsorbents for bleaching vegetable oils.

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Keywords: Cameroonian clays; Acid activation; Vegetable oils; Bleaching capacity; Adsorption kinetic

## 1. Introduction

Crude vegetable oil in addition to triglycerides contains impurities like free fatty acids, phospholipids, hydroperoxides, trace metals and pigments (carotenoids, chlorophylls, tocopherols, gossypol and it degradation

\* Corresponding author. *E-mail address:* frederic.Villieras@ensg.inpl-nancy.fr (F. Villiéras). products, etc.), (Occelli and Tindwa, 1983; Sarier and Güler, 1988; Nnandozie et al., 1989; Boki et al., 1992; Hymore, 1996; Wan et al., 1996; Falaras et al., 1999; Zschau, 2001). Those impurities limit the conservation and the uses of the oils. It is thus imperative to refine the oils to provide the consumers with attractive products having a long shelf life.

The bleaching operation is one of the most significant steps of any vegetable oil refining process. In this step,

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pigments and other unwanted compounds are adsorbed on solid materials. Many types of adsorbent have been tested for the removal of pigment from vegetable oil (Proctor and Synder, 1988; Proctor and Palaniappan, 1990; Habile et al., 1992; Kamga et al., 2000; Falaras et al., 2000). However the most common materials used nowadays for vegetable oil bleaching are activated montmorillonites (Srasra et al., 1989; Sarier and Güler, 1988; Christidis et al. 1997; Falaras et al., 2000; Christidis and Kosiari, 2003).

Vegetable oil refining factories are the main industries in Cameroon, but they cover their need for adsorbent by import. Previous works (Nguetnkam, 2004; Nguetnkam et al., 2005) showed that clays from extreme north of Cameroon contain mainly smectites. Moreover one of those clays activated with sulphuric acid solution was successfully used for removing pigments and free fatty acids from shea butter (Bike Mbah et al., 2005).

The aim of this work is to examine the respective influence of temperature and of mineralogical composition, chemical composition, and acid activation of clays from the extreme north Cameroon on their efficiencies in bleaching of palm oil, cottonseed oil and maize germ oil. Its scope encompasses the improvement of the quality of local vegetable oils as well as the development of local materials likely to be used for the decolourisation of vegetable oils.

## 2. Materials and methods

Palm oil used for this work was the olein portion of fractioned palm oil provided by SOCAPALM Co (Cameroon). Neutralized cottonseed oil was obtained from SODECOTON Co (Cameroon). Crude maize germ oil was supplied by MAISCAM Co (Cameroon).

Adsorbents used in the present study were industrial adsorbents and local clays. The three industrial adsorbents Enge, Fulmont AA, and FlorB80 are products of Engelhard Co (Netherland), Laporte Inorganic (England) and of South African Industrial Clay (South Africa), respectively. The local clay materials used were sampled from pellitic vertisols located in the extreme North of Cameroon. The localisation and the mineral composition of the natural clays are presented in Table 1.

Acid activations were carried out by mixing 50 g of clay with 250 mL of 0.5, 1, 2 and 4 M  $H_2SO_4$  solution. The suspensions were stirred at 80 °C for 2 h. After cooling in air at room temperature during 45 min, suspensions were filtered and the collected solid phases were washed several times with pure water (MiliQ ultrafiltration) until the silver nitrate test for sulphate ion was negative. The obtained activated clays were then dried at 110 °C and gently pulverized in an agate mortar.

Activated clays of the area of Kaélé will be referred to as Kaélé1N, Kaélé2N, Kaélé4N, and Kaélé8N respectively, those of the area of Kousseri as Kous1N, Kous2N, Kous4N and Kous8N, those of the area of Fortftourou as Fortf1N, Fortf2N, Fortf4N and Fortf8N, and finally those of the area of Maga as Maga1N, Maga2N, Maga4N and Maga8N. The initial clays will be referred to as Kaélé0N, Kous0N, Fortf0N and Maga0N. The characteristic of the adsorbents are presented in Table 2.

The cationic exchange capacities (CEC) were measured by using cobaltihexamine  $[Co(NH_3)_6Cl_3]$  as exchangeable ions. The amount of cobaltinexamine fixed by the solid phase was determined from concentration measurements using UV–vis spectroscopy.

Transmission infrared spectra were recorded using an IFS55 Bruker Fourier Transform IR spectrometer equipped with DTGS detector. Around 0.75 mg of each sample previously dried at 120 °C was mixed with 150 mg of KBr and pressed to obtain disks.

Nitrogen adsorption isotherms at 77K were recorded on a step by step automatic home built setup. Pressures were measured by using a 0-1000 Pa and a 0-100000 Pa Baratron type pressure sensors provided by Edwards. Nitrogen saturation pressure was recorded in situ by using an independent 0-100000 Pa Baratron type pressure sensor provided by Edwards. Prior to adsorption, samples were outgassed overnight at 120 °C and under a residual pressure of 0.01 Pa. Nitrogen N55 (purity>99.9995%) used for experiments was provided by Alphagaz (France).

Specific surfaces areas (Ssp) were determined from adsorption data by applying the Brunauer–Emmet–Teller (BET) equation (Brunauer et al., 1938) and using 16.3 Å<sup>2</sup> for the cross sectional area of nitrogen (Gregg and Sing, 1982). In the present study, the error in the determination of the Ssp was  $\pm 1 \text{ m}^2/\text{g}$ . Mean pore diameters were derived from mesopore

Table 1 Localisation and mineral composition of the natural clays

Elocalisation and mineral composition of the natural entrys					
Designation	Localisation		Mineral composition <sup>a</sup> (Wt %)		
	Latitudes	Longitudes	Smectites	Kaolinite	Quartz
Kaélé	10°06' North	14°27' East	$74\pm7$	$8\pm1$	15±2
Fortftourou	10°38' North	14°17' East	$85\pm9$	$9 \pm 1$	$6 \pm 1$
Maga	10°51' North	14°57' East	$79\pm8$	$12 \pm 1$	$8 \pm 1$
Kousseri	12°06' North	14°27' East	$76 \pm 7$	$19 \pm 2$	$5\pm1$

<sup>a</sup>The mineral compositions of the natural clays are from Nguetnkam et al. (2005).

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