Preparation of High Apparent Bulk Density Clays by Fluidized Activation

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Abstract

Fluidizing a free-flowing mixture of clay and concentrated sulfuric acid in a medium of hot air at 150 C yields an active product of high apparent bulk density (ABD) and consequent low oil retention unlike the conventional "wet" method of activation which yields a fluffy clay with high oil retention. Comparative data on bleaching capacity, ABD and oil retention of the products obtained by both methods are given.

Introduction

FOR ECONOMIC BLEACHING of oils, an activated clay possessing low oil retention has long been sought. Rich (3) observed that an inverse relationship exists between apparent bulk density (ABD) and oil retention of bleaching clays, but a process of activation known to yield an activated material of high ABD has yet to be devised. A "dry" method of activation employing concentrated sulfuric acid has been developed by Puddington and Farnand (2), but little is known about the properties of the material thus activated, such as ABD and oil retention capacity. The present studies deal with the fluidized activation of a clay responsive to acid treatment and subsequent examination of physico-chemical properties.

Experimental Procedures

The Fluidizer

A detailed account of the fluidization apparatus devised by the author is given elsewhere (4) and is shown in Fig. 1. It consists of a glass column of 1.5 in. diameter connected with a blower, a set of cyclone separators, a bag filter, a rotameter, an air distributor, a pre-heater and an arrangement for recirculation of hot air.

Bleaching clay from Korvi, Mysore (India), ground and sifted to −120 + 170 mesh B.S.S. was used in the experiments.

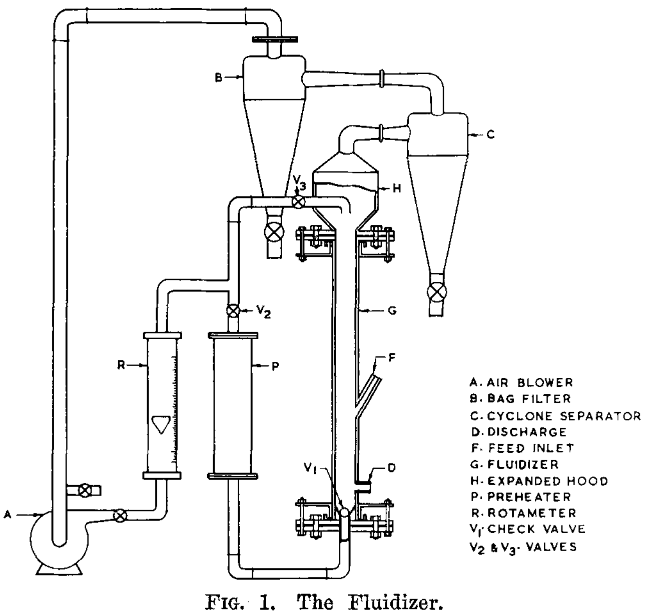

FIG. 1. The Fluidizer.

Concentrated sulfuric acid (96%) and the clay were mixed in varied proportion and a little moisture was added to the mix so as to ensure a thorough spread of the acid on to the surface of the clay. The free-flowing acid-clay mixture was fed in the fluidizer G through the feed inlet F, and preheated air was passed through at a rate slightly exceeding the incipient velocity of fluidization. Fluidization was continued at 150 C for 30 min. The change in color of the dried material from brown to dark grey could also be observed at the end of the operation. The product was drawn through a side outlet tube D, washed free of soluble sulfates, dried at 110 C and ground to pass a 200-mesh sieve.

The samples were evaluated for their physico-chemical properties such as, acidity, hydrated silica, ABD and decolorizing power. Alkali-refined soybean, cottonseed, sesame and safflower oils were used as substrates in the bleaching tests and color of the oils was measured in a Lovibond tintometer using 5¼ in. cell.

Results and Discussion

It is known that raw clays of poor decolorizing ability usually have a high bulk density and low oil retention compared to acid-activated decolorizing clays which have low bulk density and high oil retention. This is because clay materials treated with dilute mineral acids in wet activation disintegrate into finer particles, the amount of fines increasing with the dilution of the slurry and the length of

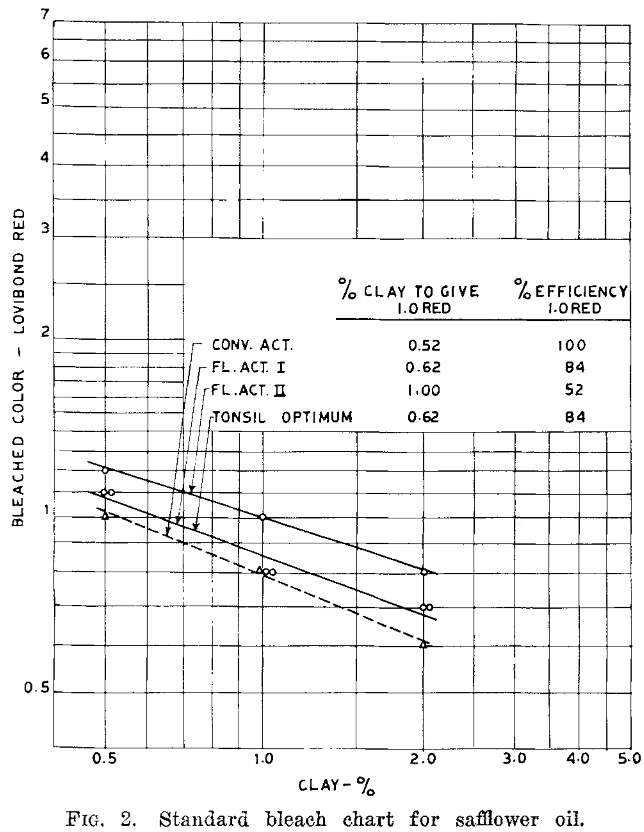

FIG. 2. Standard bleach chart for safflower oil.

TABLE I

Data on the Physico-Chemical Properties of the Raw, Conventionally Activated and Fluidized Activated Clays

	Raw clay	Conventionally activated[b]	Fluidized activated		Tonsil optimum
			Sample I[a]	Sample II[b]	
Moisture, %	13.5	6.5	6.0	6.5	5.5
Screen, % T-200/% T-325	100/98	100/98	100/94	100/98	96/76
pH	7.1	2.5	3.2	2.8	2.7
Acidity, as H_2SO_4, %	0.00	0.05	0.10	0.15	0.02
Hydrated silica (15% M.B.), %	1.6	12.6	14.2	20.2	16.5
ABD, lb/cft	54.3	41.0	51.8	48.0	36.7
Oil retention, % basis dry cake[c]	35.7	49.0	38.2	42.0	53.3

[a] Treated with 30 parts of acid by weight of clay.
[b] Treated with 50 parts of acid by weight of clay.
[c] Calculated from the ABD: % oil retention = 90 − ABD.

treatment. This method results in a light and fluffy product characterized by high oil retention. Since bleaching is essentially an intracrystalline property of clays whereas oil retention is the property of the bulk, the latter could be controlled by minimizing production of fines in the activation process. This has been achieved using the dry method of activation, which also carries other advantages inherent in the fluidization technique such as better heat transfer and homogenization of the product.

The clay activated by the conventional method using 50 parts of acid by weight of the clay, was found to possess an ABD of 41 lb/cft against 51.8 lb/cft for the fluidized activated product using 30 parts of acid. The corresponding values for the oil retention capacity were 49% and 38.2% by weight of the sample. The ABD of the unactivated clay was 54.3 lb/cft and its oil retention 35.7% (Table I). By a suitable modification of the process of activation and judicious control of particle size distribution, it is possible to obtain a product of high apparent bulk density approaching very near to that of raw clay. However, the products activated by the conventional and the fluidization methods would differ to some extent in their physical structure, hence in their selectivity for removing color from different oils. Therefore, the economic use of either activated clay with respect to a particular oil would largely depend on a compromise between the difference in bleaching and oil retention.

From the bleach charts for safflower oil (Fig. 2), the equivalent quantities of clays to reach the same extent of bleach, Lovibond color 1.0 red, are 0.52% conventionally activated clay and 0.62% fluidized activated clay I. This gives the bleaching efficiency of the fluidized activated clay I as 84%, taking the efficiency of the conventionally activated clay as 100%. Taking into account the equivalent quantities of clays and their oil retentions, the net oil loss occurring in bleaching safflower oil using either clay comes out to be almost the same. Similarly, on cottonseed oil (Fig. 3), the efficiency of the fluidized clay I is determined as 72%; the net oil loss with the two clays is also not found to be much different. It is thus obvious that on these oils the decrease in the bleaching efficiency of the fluidized activated clay I is compensated by the low oil retention of the clay due to its high ABD. Of course, for soybean oil (Fig. 4), the bleaching efficiency of the fluidized activated clay is more than offset by the oil retention advantage. The discrepancies in bleaching may be due to the difference in the hydrated silica content

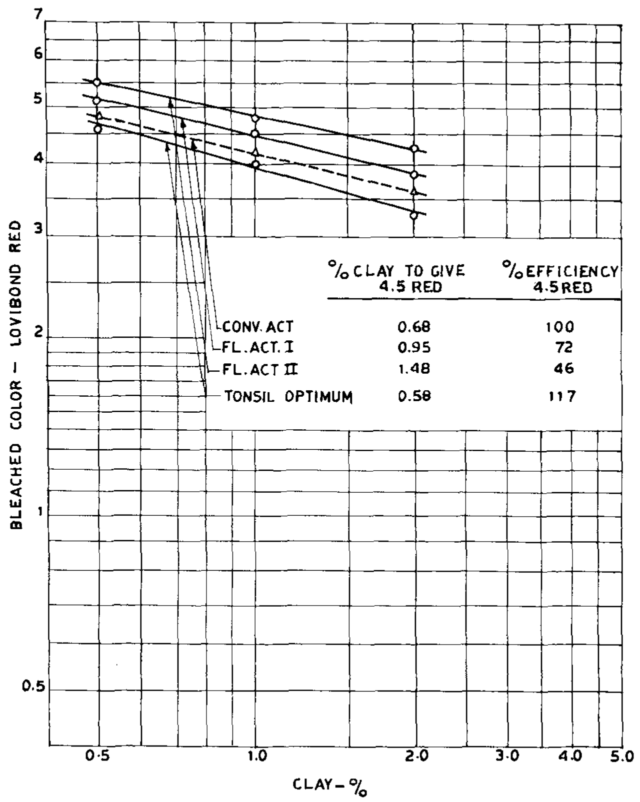

FIG. 3. Standard bleach chart for cottonseed oil.

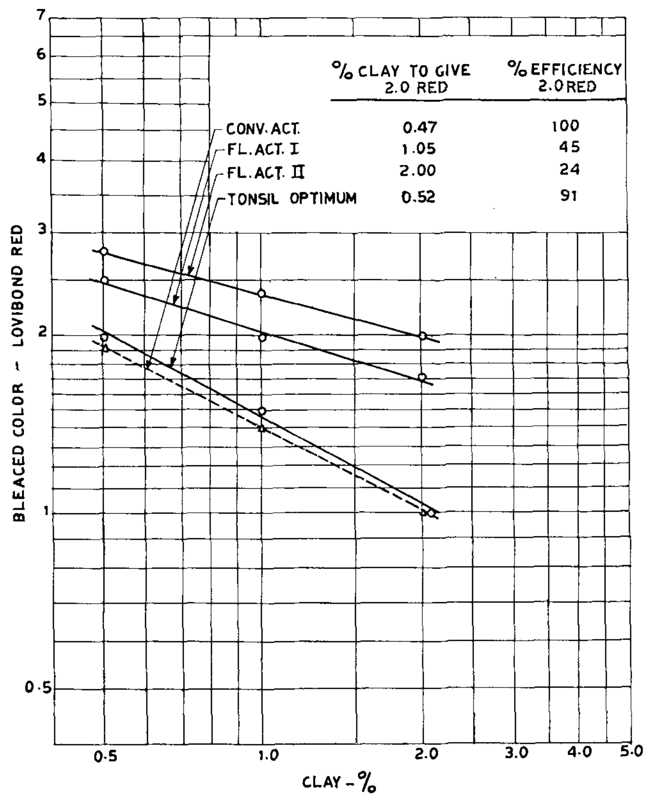

FIG. 4. Standard bleach chart for soybean oil.

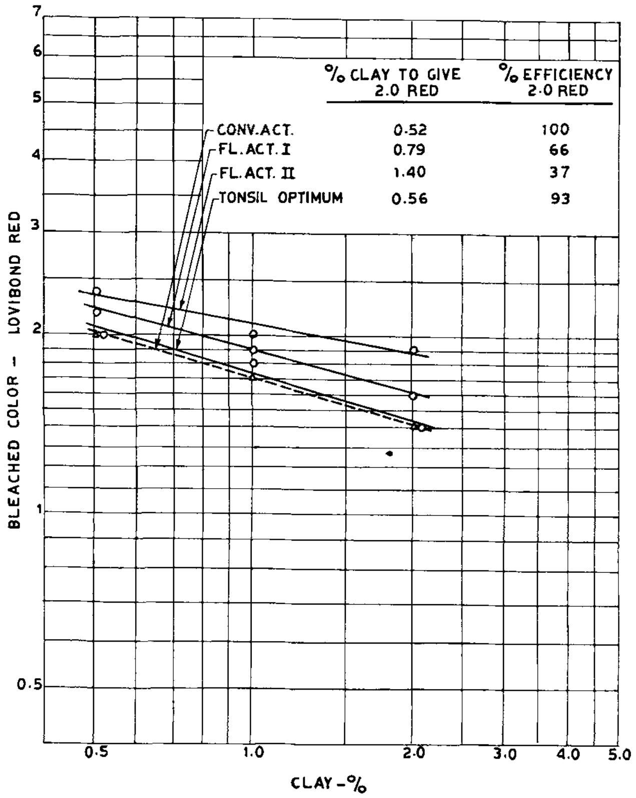

FIG. 5. Standard bleach chart for sesame oil.

of clays (Table I). Soybean oil seems to be more responsive to such effects than most other vegetable oils. Further, the product which was prepared by fluidization using 50 parts of acid by weight of clay (fluidized clay II), contained as much as 20.2% hydrated silica but exhibited very poor bleaching capacity when compared to the clay activated by 30 parts of acid (fluidized clay I), which has yielded 14.2% hydrated silica (Table I, Fig. 2–5). This is due to the fact that the bleaching activity increases with the level of acid treatment to a maximum and declines thereafter whereas free silica increases steadily even beyond the maximum in the bleaching activity (1).

This could also be seen from the data given in Table I. The fluidized activation has resulted in the separation of more hydrated silica from the clay than did the conventional activation on the same weight basis of acid, probably due to the higher concentration of acid and the higher temperature of activation employed in the former method. In other words, in the fluidization method a much lower proportion of acid-to-clay can be used than in the conventional method for achieving the same degree of activation. Besides, in the conventional "slurry" method, large quantity of mix is required to be heated and the time of activation is often prolonged. Both these factors are very much reduced with the dry method using fluidization technique. Hence, the new method predicts to low heat requirements. These aspects would, of course, have some bearing on the economic considerations of the process for the manufacture of bleaching clays.

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