Laboratory Bleaching Technique for Fatty Oils*

Control of Moisture Content of Vital Importance in Obtaining Results Comparable to Plant Operation

By J. T. R. ANDREWS and R. G. FOLZENLOGEN

THE fatty oil industry has long been cognizant of the fact that the bleaching of refined vegetable oils, such as cottonseed, with fuller's earth is considerably more efficient when performed on a factory scale than when carried out in the laboratory. The official bleaching test of the A.O.C.S., which is practically identical with that of the National Cottonseed Products Association (Rule 275, Sec. 2), makes allowance for this discrepancy between plant and laboratory by prescribing the use of 6.0% earth, an amount which is greater than that ordinarily used in the commercial bleaching of cottonseed and similar refined oils, but which is agreed to by oil technicians because they know that the laboratory bleached color can be duplicated in the plant with only about half of this earth usage. The official bleaching test was studied recently by Ma and Withrow[1]. In general, they found its main features justified. An excellent bibliography is appended to their paper.

THE official laboratory bleach test is made by heating 300 grams of refined oil in a refining cup to 120° C.; 6.0% of the official fuller's earth is added and the mixture is stirred mechanically at 250 r.p.m. for five minutes without allowing the temperature to drop below 105° C. The bleached oil is filtered through paper and the color of the clear oil is determined by matching a 5¼" column with Lovibond red and yellow type glasses.

In order to bring plant and laboratory conditions of time and temperature more closely together 105° C. and 15 minutes were selected for our first experiments. The official bleaching apparatus operated at 250 r.p.m. and 300 gram samples of oil were employed for most of our work. An open cup or beaker was used in all experiments except the few specifically described as made in covered vessels. We were fortunate in directing our early experiments to an investigation of the role of moisture in bleaching and the results obtained were so startling that the development of this idea constituted the remainder of our program.

Moisture Loss a Factor

IT WAS thought that in the laboratory, with only a 300 gm. sample in an open cup, drying would take place much more rapidly than in the plant where 50,000 to 80,000 lbs. of oil are agitated in a closed U-tank. Accordingly, comparative bleaches were made with and without addition of 1.0% of water (Table I) which showed that 3.0% earth + 1.0% of water is almost, but not quite, equal in bleaching power to 6.0% earth without water. Further experiments (Table II) showed that the optimum amount of water to be added is not proportional to the quantity of earth. For most efficient bleaching on a 300 gram scale,

TABLE I

Bleaching Refined Cottonseed Oil With and Without Added Water

(105°C.—15 minutes—open cup—original color 6.2 R) Color, Lovibond Red

% English Earth	No Water	1.0% Water
1.0	5.0	4.1
2.0	4.2	3.3
3.0	3.4	2.8
4.0	3.1	2.5
5.0	2.9	2.3
6.0	2.7	2.2

TABLE II

Bleaching Refined Cottonseed Oil With Varying Amounts of Added Water

(105°C.—15 minutes—open cup—original color 7.5 R) Color, Lovibond Red

% English Earth	1.0	2.0	4.0	6.0
% Water				
0.00	4.5	3.1	2.3	2.0
0.20	4.1	2.9	2.1	2.1
0.50	3.7	2.7	2.0	2.0
0.75	3.4	2.4	2.0	1.7
1.0	3.3	2.3	2.0	1.8
1.5	3.3	2.4	2.0	1.9
2.0	3.4	2.4	2.1	1.9
3.0	4.7	3.8	2.3	2.1
4.0	5.4	3.8	3.1	2.3

*Presented at Fall Meeting, American Oil Chemists' Society, Chicago, Nov. 14, 1930.

[1] Ind. & Eng. Chem. Analytical Ed. Vol. 2, pp. 374-77 (October, 1930).

the water added should be about 1.0% of the weight of oil taken. Without pausing to exhibit data, some of our other findings may be summarized as follows:

1. There apparently exists an optimum intensity of agitation above and below which poorer bleaching results. Speed is not a critical factor between 150 and 250 r.p.m.

2. Best results are obtained by mixing the earth with the cold oil and adding the water before heating is begun.

3. Added moisture improves the bleaching efficiency of earth and carbon mixtures, e.g. English + Darco.

4. Oil may be humidified during bleaching by use of steam which is effective but difficult to control.

5. Free fatty acid percentage does not increase during bleaching by the moisture method with English earth or Carlton earth.

6. A blanket of inert gas (CO_2) is not beneficial and when circulated over the surface of the oil it apparently defeats in part the object of added moisture.

7. Filtration of the refined oil through paper removes excessive moisture and conditions it for bleaching by the moisture method.

8. On freshly refined oils, no difficulty was experienced in getting a smooth and apparently homogeneous suspension of earth in oil after 1.0% of water had been added, but on oils which had been dried and filtered through kieselguhr in a press a considerable tendency for the earth to agglomerate into small balls was noted. This condition was attributed to removal of soap from the oil by adsorption on the guhr and, as the trouble was prevented entirely by the addition of from 0.125 to 0.25% of 10% soap solution, this surmise was probably correct.

9. Dehydration, either by oven drying or by desiccation over H_2SO_4, lowers bleaching power to a marked degree in the case of English earth. By humidification, bleaching efficiency can be completely restored. The same observations, to a somewhat less extent, are true of all other earths which we have studied over a period covering the past three years. It should be noted that maximum bleaching efficiency with English, Carlton and Filtrol is reached at about 20% moisture content. (Tables III-A, B & C.)

TABLE III-A

Bleaching Refined Cottonseed Oil
With English Earth of Variable Moisture Content
(105°C. — 15 minutes—open cup — no added water — 2.68% dry earth)

Earth Treatment	% Moisture in Earth	Color, Lovibond Red Oil No. 1	Oil No. 2
Color before Bleaching		8.3	8.8
Dried at 105°C. 16 hrs.	0.0	4.9	5.2
Over conc. H_2SO_4 16 days	6.1	4.2	
Earth as received	10.8	3.7	3.8
Over 50% H_2SO_4 16 days	13.8	3.4	
Over water 16 days	19.8	3.1	3.4
Moistened with water, mixed and allowed to stand several days	25.6		5.4
	31.7		6.6
	36.3		7.9

TABLE III-B

Bleaching Refined Cottonseed Oil
With Carlton Earth of Variable Moisture Content
(105°C.—15 minutes — open cup — no added water — 2.75% dry earth)

Earth Treatment	% Moisture in Earth	Color, Lovibond Red
Color before bleaching		8.8
Dried 105°C. 16 hrs.	0.0	4.2
Earth as received	8.3	3.4
Moistened with water, mixed and allowed to stand several days	19.3	3.3
	26.2	3.6
	31.7	4.5
	38.2	6.3

TABLE III-C

Bleaching Refined Cottonseed Oil
With Filtrol of Variable Moisture Content
(105°C.—15 minutes—open cup—no added water)

Earth Treatment	% Moisture in Earth	Color, Lovibond Red	
% Dry Earth used in Bleach.		1.28	2.57
Color before bleaching		8.8	8.8
Dried 150°C. 16 hrs.	0.0	4.3	2.6
Earth as received	14.4	3.9	2.4
Moistened with water, mixed and allowed to stand several days	19.4	3.8	2.5
	26.0	4.2	3.1
	31.8	4.4	3.3
	37.4	5.3	3.6

10. With proper moisture control, laboratory bleaching with English earth is equal to or better than plant bleaching in efficiency (Table IV-A).

TABLE IV-A

Plant vs. Laboratory Bleaching of Refined Cottonseed Oil with English Earth

Color Refined Oil	% English Earth	Color Plant Bleached	Laboratory Color No Water	1.0% Water
8.5	2.4	3.9	4.7	3.6
6.2	2.1	3.4	4.2	3.1
8.6	2.3	3.8	4.4	3.3
10.0	2.3	3.6	4.4	3.6
9.0	2.3	3.5	4.3	3.4
8.3	1.9	3.2	4.4	3.3
8.6	2.9	3.1	3.5	3.0
8.0	1.9	3.4	4.2	3.3
7.9	1.8	4.0	4.1	3.4
Average Color		3.5	4.3	3.3

With due precaution to prevent loss of moisture during laboratory bleaching, the efficiency obtained is approximately the same as that of the plant but not quite as high as that obtainable in the laboratory when optimum moisture conditions prevail (Table IV-B).

All of the conclusions herein presented were drawn from experimental bleaches on plant refined cottonseed oil. A few experiments on refined tallow and coconut oil indicate that similar results may be expected on other fatty oils. It should be noted that these conclusions apply primarily to laboratory bleaching under the conditions described.

At this point, the question may be asked "Cannot plant as well as laboratory bleaching be improved by proper moisture control?" Our observations have led us to the opinion that under ordinary conditions this is unlikely. The optimum moisture for plant scale bleaching is much lower than 1.0%; probably nearer 0.1-0.3% and most plant refined oil direct from the settling tanks is of practically optimum moisture content for bleaching on a factory scale. In a few cases, perhaps, such as in the case of oils which have been dried for storage, moisture control may have a plant application, but we are not optimistic over the possibility of its general extension into plant practice.

Effect of Temperature

ON BLEACHES made below 100° C. there is apparently no benefit derived from added moisture. With increasing temperature, the

TABLE IV-B
Plant vs. Laboratory Bleaching of Refined Cottonseed Oil with English Earth

Color Refined Oil	% English Earth	Color Plant Bleached	Laboratory Color No Water	Covered	1.0% Water
8.2	2.3	3.5	4.1	3.4	3.3
7.7	1.8	3.3	4.6	3.8	3.7
7.9	1.8	3.8	4.7	3.8	3.5
6.4	1.5	4.0	4.4	3.7	3.3
Average Color		3.7	4.5	3.7	3.5

moisture method improves in efficiency, reaching a maximum at about 135° C., where 5 minutes has been found ample time. At either 135° C. for 5 minutes or at 105° C. for 15 minutes, the results obtained by the moisture method using 3.0% English earth are only slightly inferior to bleaches made by the A.O.C.S. official method using 6.0% earth (Table V).

TABLE V
Comparison with A.O.C. S. Official Bleach Test

Color Refined Oil	Official Bleach 6% Earth; 120°-105° C. 5 minutes	Color, Lovibond Red 3% Earth; 1% Water; 105° C. 15 minutes	3% Earth; 1% Water; 135° C. 5 minutes
10.4	4.1	4.4	4.4
16.8	5.4	5.7	5.2
8.2	2.1	2.3	2.1
6.9	2.1	2.2	2.1
6.4	2.1	2.2	2.3
33.0	10.2	11.2	10.8
8.2	3.2	3.6	3.6
Average Color	4.2	4.5	4.4

General Conclusions

IN EXAMINATION of a considerable number of bleaching earths, we have found very few laboratory bleach tests which are not improved by the addition of water and practically none which are injured unless the refined oil is already excessively wet. In order to make an intelligent comparison of the bleaching efficiency of earths, the laboratory test should simulate the essential conditions prevailing in actual plant practice and we claim that no test which ignores the important function of moisture will meet this requirement. By the official test, or any test in which the laboratory moisture loss is not kept at a minimum or replaced by added water, positively erroneous conclusions can be drawn regarding the relative bleaching efficiencies of two earths under factory conditions and this is especially true in case one of these earths is more sensitive to moisture control than the other.

In conclusion, it is suggested that the official bleaching method of the A.O.C.S. be modified in the way we have demonstrated as effective in putting laboratory and plant bleaches on a more comparable basis. The modified method, making use of moisture control, will enable laboratory tests on both earths and oils to be translated directly into an accurate forecast of plant scale bleaches.

Report of the Committee to Rewrite the Methods

THE CHANGES made last year in the methods, and new methods covering cottonseed made necessary the reprinting of eighteen pages of the methods. This work was done by the Lefax Company at a total cost to the Society of $82.50 for two thousand sets.

Our methods, as now being sent out, are revised to August 1, 1930.

The only recommendation this committee has to offer is that the methods covering Soap also be printed in the Official Methods.

W. H. IRWIN, *Chairman.*

Chicago, April 13, 1931.

Laboratory Construction Company of Kansas City, Missouri, reports the sale of nitrogen apparatus to Wilson and Company of Oklahoma City, and Cudahy Packing Company of Kansas City, Kansas. Also, the complete installation of Laboratory Equipment Tables and Kjeldahl Nitrogen Apparatus for the three laboratories of the Food and Dairies Division, State of Illinois at Springfield, Illinois.

The Research Association of British Paint, Colour and Varnish Manufacturers have recently issued a Review, giving abstracts of current literature relating to the paint, color and varnish industries.