Determining Bleaching Loss Coefficients

A Laboratory Test for Oil Retention of Bleaching Earths and Carbons

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ADSORPTION bleaching materials are not evaluated solely on a basis of bleaching efficiency and cost per ton. An important consideration is the loss or degradation of the oil retained by the filter cake. Frequently this factor outweighs the original cost of the earth or carbon to such an extent that the higher priced material may be actually the cheaper to use.

Admittedly plant experience is the best method of determining oil retention, but plant scale experiments are costly and are inapplicable to small samples. Bailey and Allen[1] have devised a comparative laboratory test which may be employed when only a few grams of material are available. This method possesses the advantage of requiring very simple equipment, but involves considerable personal equation. There is need of a laboratory method which, in the hands of different operators at different times, will give correct comparative values for the oil retention of bleaching materials. To meet this need, the method described below is proposed, being based on the gain in weight of the bleaching material after an excess of oil has been removed in a stream of hot, inert gas.

The test has been applied to about 500 earths and carbons and has given satisfactory comparative results which approximate those obtained in plant practice. It requires less than 10 grams for duplicate determinations.

Apparatus

THE equipment is shown in Figure 1. To a liter suction flask is fitted a Gooch crucible of 35 mm. outside diameter and 40 mm. height. A disk of muslin is cut to fit into the bottom of the Gooch crucible. Any convenient apparatus for delivering an excess of dry, hot carbon dioxide into this crucible may be provided. For heating the gas, we have used a piece of standard 2 inch pipe about 8 inches long with closed ends pierced by holes through which is fitted a ¼-inch copper tube of 10 inches length. The iron pipe is filled with dry sand packed closely around the copper tube, in order to minimize temperature fluctuations. Inside the copper tubing is placed a piece of

[1] Cotton Oil Press, Vol. 7, No. 8 pp. 36-37 (Dec. 1923).

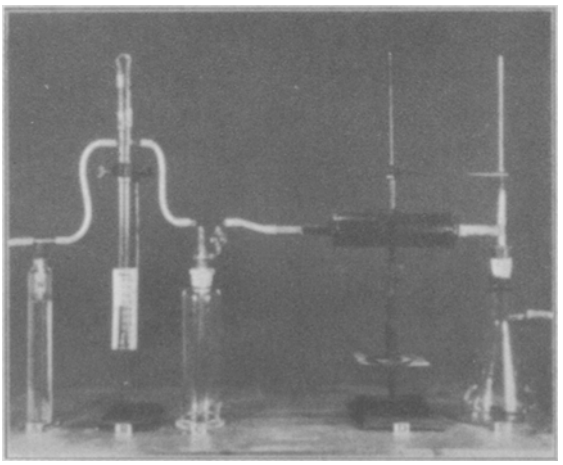

FIG. 1 *Apparatus*
A, bottle for relief of gas pressure. B, flowmeter. C, liquid trap. D, furnace. E, suction flask with crucible containing bleaching material.

Pyrex glass tubing which fits snugly and is about 14 inches in length. On one end is sealed a vertical T tube in which is held a thermometer for measuring the temperature of the carbon dioxide. The lower end of the T tube holds a cork stopper designed to fit loosely into the top of the crucible. The furnace is heated by means of a wing top gas burner. Carbon dioxide from an ordinary cylinder is passed through a T tube connected with a safety bottle or other suitable means for emergency relief of excess pressure, through a flow meter followed by a trap of size sufficient to hold any liquid which might accidentally be blown over from the manometer of the flow meter, and thence to the furnace already described. Vacuum is produced in the suction flask by use of a water pump.

Method of Operation

THE flow of carbon dioxide is adjusted to 4-5 cubic feet per hour and the heating of the furnace regulated so that the temperature of the carbon dioxide is 145-155°C. at the position of the thermometer. The Gooch crucible is fitted on to the filter flask, the muslin disk is placed in the bottom of the crucible, and the suction pump is started. About 5 cc.

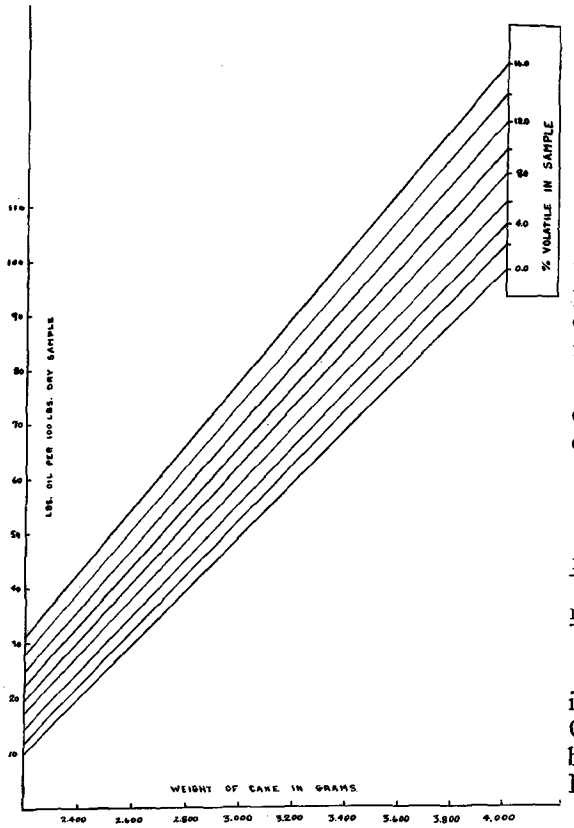

FIG. 2
Chart for calculating oil retention from weight of blown cake and % volatile in original sample.

of dry, clear cottonseed salad oil heated to 105-10°C. is poured over the muslin disk in order to cause it to adhere properly to the bottom of the crucible. Next 2.000 (±.001) grams of the earth or other bleaching material is transferred from a glazed paper to the crucible, with use of a camel's hair brush. The powdered material is packed down well with a flattened stirring rod. Then 10-15 cc. of the hot oil is poured over the sample, which is again packed down with a stirring rod to a level surface. The stirring rod is washed off and the crucible filled with the same hot oil to within ¼-inch of the top. The crucible is then placed under the cork stopper carrying the T tube through which the hot carbon dioxide is flowing.

The suction and the hot gas flow are continued one hour after the oil level has reached the surface of the cake. Then the crucible is removed, the excess of oil wiped off from the outside and from the inside surface above the cake, and the crucible cooled in a desiccator. The contents of the crucible are transferred into a tared dish; for this purpose, a light puff of air applied from any convenient source is very useful. The muslin disk is removed and any particles of cake adhering to the sides of the crucible or to the muslin are easily scraped off. The cake is weighed to ±.001 gram.

Bleaching carbons are likely to show a tendency to pass through the muslin disk. When this happens, the first portion of the filtrate is collected in a test tube of suitable size suspended inside the suction flask. The oil thus collected is returned to the crucible until it is no longer cloudy.

The moisture in the original sample of earth or other bleaching material is determined by drying 5 grams to constant weight at 105-10°C.

Calculation

$$2.000 - \frac{2\ (\%\ \text{Moisture})}{100} = \text{Wt. dry sample.}$$

$$\frac{100\ (\text{Wt. cake} - \text{Wt. dry sample}}{\text{Wt. dry sample}} = \text{Lbs. oil per 100 lbs. dry sample}$$

$$\frac{100\ (\text{Wt. cake} - \text{Wt. dry sample})}{\text{Wt. cake}} = \%\ \text{Oil in cake (dry basis)}$$

Duplicate determinations should check within one pound oil per 100 pounds dry sample. Calculation of the results may be facilitated by the use of a chart constructed as shown in Figure 2.

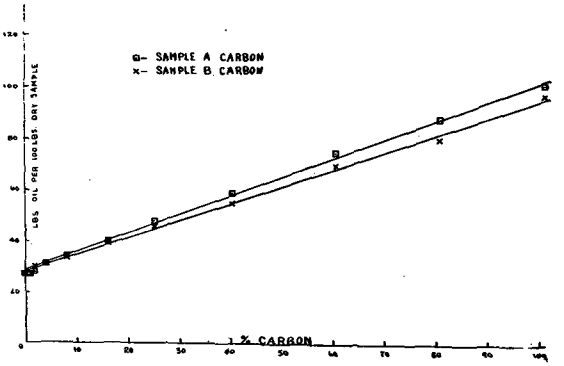

FIG. 3
Oil retention of mixtures of English earth with carbon.

Discussion

TYPICAL results from the above method are shown in Figure 3, which incidentally brings out the fact that the oil retention of earth-carbon mixtures has been found to be a straight line function.

It is not claimed that the method has been reduced to its simplest possible terms. We undertook to develop a procedure for determining in the laboratory reproducible values for oil retention which would fall within the range normally observed in plant practice for oil

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