and the Buckeye Cotton Oil Company for the oils used in this investigation.

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Solvent Extraction of Cottonseed and Peanut Oils. IX. Determination of Fines in Miscella

A. V. GRACI JR., A. J. CROVETTO, J. S. PARKER,[1] and C. G. REUTHER JR.,
Southern Regional Research Laboratory,[2] New Orleans, Louisiana

IN the continuous solvent extraction of oilseeds the quantity of fine meal solids (fines) that will be carried out from the extractor with the oil-solvent mixture (miscella) depends upon the oilseed, the method of preparation, and the extractor used. Fines (1) create a problem. This problem is in proportion to the quantities involved. In soybean solvent extraction, for example, the miscella usually contains fines less than 0.2% by weight of the extractor feed material. With quantities as small (3) as these the clarification step, or the removal of the fines before evaporation and stripping, can be easily achieved by direct filtration. The problem is different in extracting cottonseed, peanuts, okraseed, and rice bran. With these materials the fines in the miscella may be as high as 5% by weight of the feed material, and settling or centrifugation (3) of the miscella is required before filtration.

It is desirable therefore to control as far as possible the amount of fines produced. A rapid, on-the-spot method for the determination of fines in miscellas has been developed and is presented here. The method is volumetric and replaces the slower gravimetric method, which consists of weighing the filter cake after a complete cycle and calculating what the average fines content of the miscella had been. The volumetric determination is converted to weight values by the use of an appropriate curve. The construction of the curve and its application in pilot-plant operations to cottonseed, peanuts, rice bran, and okraseed are described.

Experimental Data

The miscella samples used for the construction of the curves in Figures 2 and 3 were obtained from a continuous solvent-extraction pilot plant (2), which had a capacity of approximately 150 lb./hr. of flakes. In all runs commercial hexane was used. Miscella samples were taken before filtration.

The solids content of the unfiltered miscella samples was determined both gravimetrically and volumetrically. To determine gravimetrically the grams of solids per liter of clear miscella in a sample the following data were recorded: weight in grams of unfiltered miscella (slurry) sample and of solids from slurry after filtering and the specific gravity of the filtered miscella. These data can be substituted in the following formula to calculate the points along the ordinate of the chart:

$$\frac{(\text{wt. solids})(\text{sp. gr.})(1{,}000)}{\text{wt. slurry—wt. solids}} = \frac{\text{grams solids}}{\text{liter filtered miscella}}$$

The units involving filtered miscella were chosen for the ordinate of the plot because most solvent-extraction plants meter the clear miscella flow from the filter(s).

To determine volumetrically the solids content, duplicate samples of the miscella were spun for 10 min-

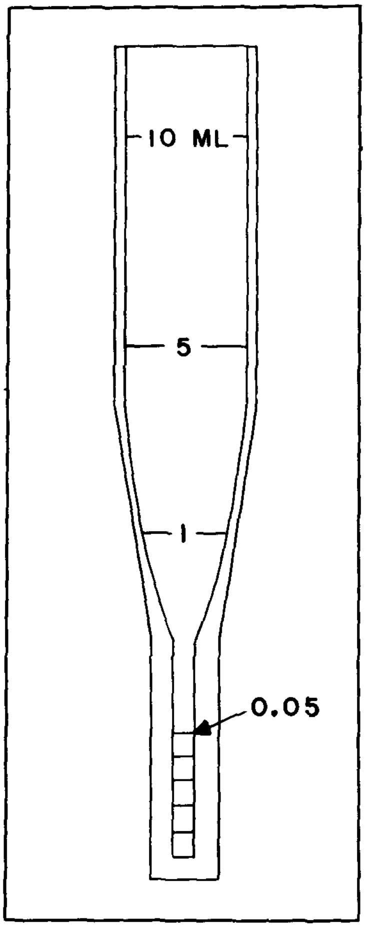

FIG. 1. Centrifuge tube used for experiments.

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[1] Present address: Wesco Water Paints, Inc., Goodhope, La.

[2] One of the laboratories of the Bureau of Agricultural and Industrial Chemistry, Agricultural Research Administration, U. S. Department of Agriculture.

TABLE I

Experimental and Calculated Data for Plot of Okraseed Curve on Figure 2

Sample number	Dilution ratio slurry to hexane	Dilution factor	Tube reading, ml. solids per 10 ml. slurry	Weight slurry, grams	Weight solids, grams	Sp. gr. miscella, grams per ml.	Weight solids per liter filtered miscella, grams	Corrected tube reading, ml. solids per 10 ml. slurry
1	None	1	0.022	238.6	0.226	0.687	0.652	0.022
2	None	1	0.022	282.4	0.273	0.687	0.665	0.022
3	1:1	2	0.022	130.3	0.261	0.702	1.408	0.044
4	1:1	2	0.023	118.2	0.233	0.702	1.386	0.046
5	1:1	2	0.045	314.0	1.782	0.698	3.985	0.090
6	1:1	2	0.046	291.7	1.414	0.695	3.384	0.092
7	1:1	2	0.043	297.7	1.415	0.695	3.318	0.086
8	1:1	2	0.048	332.7	1.616	0.698	3.408	0.096
9	1:4	5	0.030	315.5	2.074	0.702	4.648	0.150
10	1:4	5	0.030	294.9	1.933	0.702	4.629	0.150
11	1:7	8	0.032	332.7	3.658	0.705	7.840	0.256
12	1:7	8	0.032	320.1	3.520	0.705	7.835	0.256
13	1:7	8	0.038	321.8	4.440	0.705	9.860	0.304
14	1:7	8	0.038	345.8	4.819	0.705	9.970	0.304
15	1:14	15	0.048	80.4	2.547	0.710	23.23	0.720
16	1:14	15	0.050	66.8	2.077	0.710	22.80	0.750
17	1:24	25	0.050	47.0	2.579	0.708	41.15	1.25
[illegible]	1:25	26	0.050	62.3	3.540	0.708	42.62	1.30

utes in an International Clinical Centrifuge[3] using head No. 213, which develops a force approximately 900 times that of gravity. The milliliters of solids packed at the bottom of the centrifuge tube per 10 ml. of sample are the units of the abscissa.

The centrifuge tube, Figure 1, was chosen because of its graduations between 0.01 and 0.05 ml., the range in which all readings of milliliters of solid in a sample should fall for the greatest accuracy. If the volumetric determination is above 0.05 ml. of solids, the slurry must be diluted to fall within this range. The approximate volume percentage of solids and the proper dilution may be determined by centrifuging a sample for 4 or 5 minutes in a tube with graduations along its entire length.

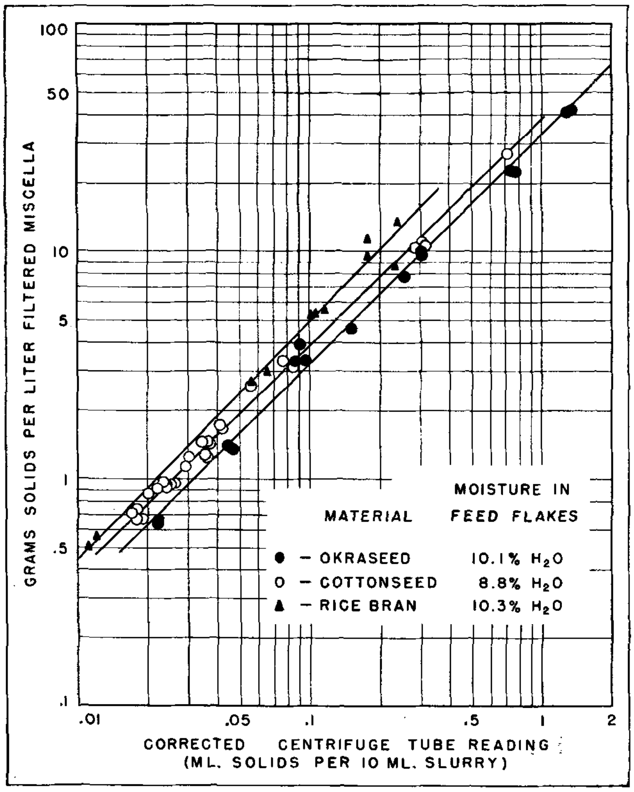

FIG. 2. Curves for determining fines in miscella

This dilution to the range just mentioned results in a straight line plot. When samples which fall outside the range are not diluted, the use of centrifuge tubes with variously shaped tips results in plots deviating from the straight line; the degree of deviation is dependent upon the concentration of the slurry and the particular centrifuge tube used.

The corrected abscissas for the curves are obtained by multiplying the tube readings by the dilution factor. The dilution factor is equal to the total volume to which the slurry was diluted. For example, if 1 part of slurry were diluted with 10 parts (by volume) of commercial hexane, the dilution ratio would be 1 to 10, the dilution factor would then be 11, or the total volume. Expressed algebraically, the dilution factor is calculated as follows:

$$\frac{\text{parts slurry} + \text{parts hexane}}{\text{parts slurry}} = \text{dilution factor.}$$

Table I shows the experimental data and the calculations required for the construction of the okraseed curve (Fig. 2).

The effect of the moisture contents of the peanut flakes used in three solvent-extraction runs, respectively, on the slope of the curves is pronounced (Fig. 3). The particle sizes of the fines from the three runs were of the same magnitude, 98.4 to 99.7% through 300-mesh, hence any effect due to particle size differences was not ascertainable.

The cottonseed curve in Figure 2 was checked against unfiltered miscella samples received from three commercial solvent-extraction plants. Moisture contents of the feed flakes from the three plants were 8.6, 10.5, and 12.9%; the particle size distribution, determined by wet-screening, was 75.4, 83.7, and 97.6% through 300-mesh. In spite of the differences in moisture contents and particle sizes the curves for the commercial cottonseed miscella samples coincided with the curve in Figure 2. This indicates that cottonseed fines were not noticeably influenced by a variation of approximately 4% in moisture or a 22% change in the through 300-mesh material while peanut fines packing characteristics were greatly affected by moisture.

Construction of a Working Curve

It is desirable to have the working curve plotted on rectilinear paper to facilitate its use. The experi-

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[3] The mention of trade products does not imply their endorsement by the Department of Agriculture over similar products not mentioned.

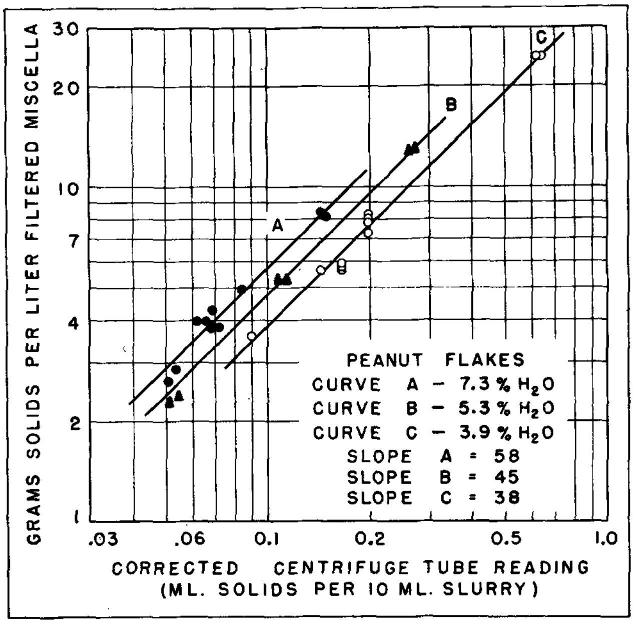

Fig. 3. Effect on slope of curve due to moisture content of feed flakes.

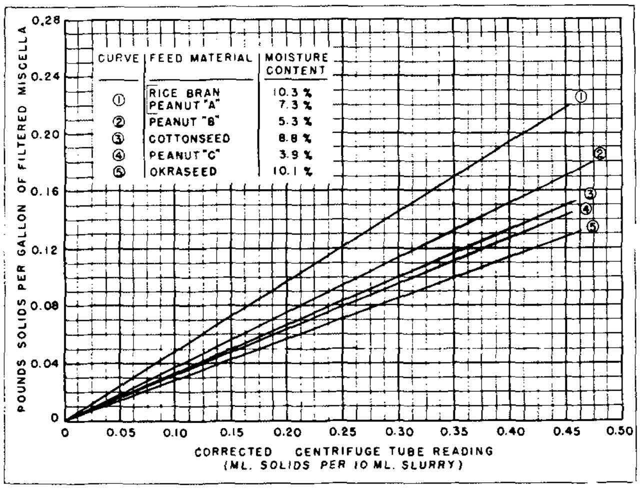

Fig. 4. Working curves plotted on rectilinear paper.

mental curves, Figures 2 and 3, were plotted on log-log paper to spread out the experimental points. Since on rectilinear paper the curve must pass through the origin (0-0), if the curve is first plotted on log-log paper and its slope determined, it may be replotted without the aid of the points on the more practical rectilinear paper and extrapolated, if necessary, to cover the range of fines anticipated.

Figure 4 is a replot of the six curves shown in Figures 2 and 3. The rice bran curve and peanut curves, A and B, were extrapolated approximately 50%, while only parts of the cottonseed and okraseed curves and the peanut curve, C, derived from experimental points are shown. It will be noted that the units of the vertical axis have been changed from "grams of solids per liter of filtered miscella" to "pounds of solids per gallon of filtered miscella." Figure 3, for example, shows values of slope for curves A, B, and C, as 58, 45, and 38, respectively. Had not the units of the vertical axis on Figure 4 been changed, the slope of peanut curves, when replotted on rectilinear paper, would still have been 58, 45, and 38. The change in slope however is in the same proportion as the change in units.

Applications of the Curve

The value of the method lies in the comparative ease and rapidity of the determination once a curve or family of curves has been drawn for a particular oilseed. Where a variation in moisture and particle size in the ranges investigated did not cause the experimental points to deviate appreciably from the curve, as for cottonseed, the one curve is adequate.

Where moisture content greatly affects the packing characteristics, as for peanuts, a family of curves or a nomograph can well be drawn to cover the anticipated seasonal moisture range.

The units chosen as the ordinate of the plot may vary depending upon the information desired. For example, the plotted data can be used to calculate the filtering cycle time, the quantity of fines produced daily, or the percentage of fines produced based on weight of seed handled, amount of meal extracted, or miscella produced. Such calculations have value in the control of operations and for accounting purposes.

Summary

Four oil-yielding materials, cottonseed, peanuts, okraseed, and rice bran, were used to develop a rapid method of determining the solids content of miscellas. In each case straight-line plots of the gravimetric determination vs. the volumetric determination were obtained when samples were diluted to fall within the proper range.

Acknowledgment

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