A Laboratory Method for Studying Basket-Type Extraction

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SOLVENT extraction of prepressed oleaginous materials has been practiced for a long time in Europe and is becoming increasingly important for recovering oil from comparable products in this country. In Germany countercurrent batchwise extraction of expeller cakes (1) has been favored because of its extreme adaptability, but continuous extraction is used almost exclusively in the United States because of lower labor costs associated with continuous operations and because most domestic plants are devoted to the extraction of a single oil-bearing material.

The basket-type extractor yields an essentially meal-free miscella and is therefore well adapted to the extraction of prepressed or scalped materials. However operation of a basket-type extractor on feed of this nature introduces certain problems, notably the preparation of flakes which, while facilitating good extraction of the oil, at the same time retain sufficient porosity to permit ready percolation of solvent through a considerable layer. This paper describes a simple laboratory extractor from which data may be obtained on the rate of flow of solvent through extractor feed as well as on the rate and completeness of extraction. Correlation between these results and the operation of a plant scale basket-type extractor is shown. Most of the information has been obtained on flaked expelled corn germ, but this laboratory extractor can also be adapted for studies on any material to be extracted in a basket-type extractor.

Corn germ obtained from the wet milling process (2) contains about 55% oil (d.b). The dry germ is expelled to a cake containing 15 to 30% oil. This expeller cake is then cracked, steamed to adjust the moisture content to 10%, flaked, and extracted in a basket-type extractor. During the initial operation of this extractor, which is designed to handle 225 tons of flakes per day, certain problems arose relative to the completeness of extraction, rate of extraction, and rate of miscella drainage. Although a great deal of work has been done previously on this and similar problems, there is no literature reference to a laboratory method for obtaining information on a) rate of flow of miscella through a thick bed of flaked expeller cake and its relation to particle size distribution, b) rate of extraction, c) practical limits of extractability, and d) effects of moisture and fines on both extraction of oil and flow rate of miscella through the flakes.

Descriptions have been published of several laboratory extractors which have been used to obtain useful data on the solvent extraction of oil seeds. A Butt extractor and a batch extractor fitted with an agitator have been employed (3) for determination of rate of extraction. Neither method can be utilized to obtain the flow rate data that is of prime importance in the extraction of scalped products. A laboratory basket-type extractor has been described (4), but it was felt that this was too complicated and expensive to be of general utility. Another laboratory continuous extractor (5) was not considered for the same reason.

Experimental

A diagram of the apparatus used to obtain the data to be presented is shown in Figure 1. A 5-gallon can supported by a steam cone constitutes the hot solvent reservoir which is fitted with a reflux condenser, an opening for filling, and an outlet near the bottom. The outlet is controlled by a ¾-inch gate valve, and a 14-mm. glass tube connects the valve to the extraction column through a large Neoprene stopper. The column consists of a 3⅜-inch I.D. glass tube 26 inches long. A Buchner funnel fitted with a 26-mesh stainless steel screen supported ½ inch above the funnel plate by a ring of a large diameter glass tube forms the bottom of the extractor. A Neoprene tube controlled with large pinch clamp is fitted to the bottom of the funnel.

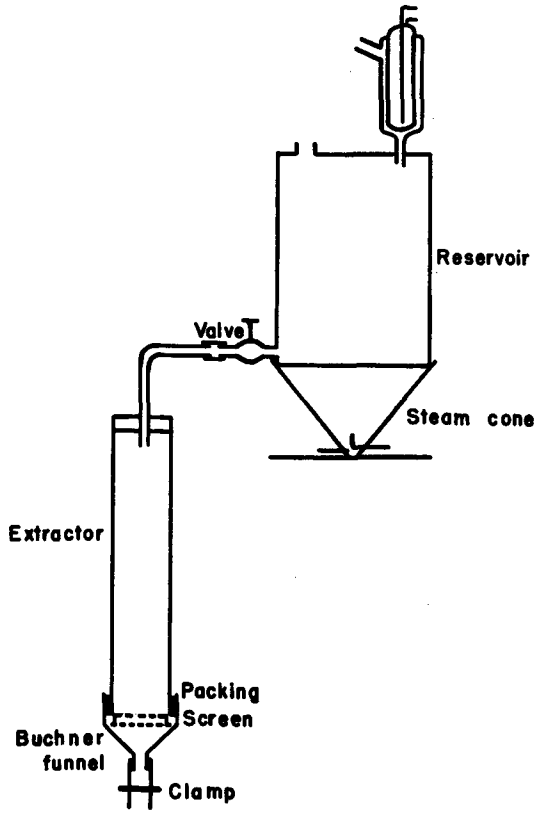

FIG. 1. Diagram of laboratory column extractor.

The extraction procedure is relatively simple. About 5 gallons of hexane in the solvent reservoir is brought to a gentle boil. The stopper and tube are removed from the top of the extractor, and the sample to be extracted and boiling hexane are introduced into the tube simultaneously. Flakes and solvent levels are kept as nearly equal as possible until the flakes reach a height of 18 inches, which is the average depth in the baskets of the plant extractor. For a typical flaked expelled corn germ this charge amounts to about 1 kg. The extractor is then connected to the hot solvent reservoir and the gate valve is opened. This automati-

cally holds the solvent level about 6 inches above the original flake level. After 5 minutes 2 liters of miscella are withdrawn from the bottom of the extractor and measured exactly; and an aliquot is analyzed for oil content. Withdrawal of 2 liters of miscella gives a complete change of solvent over the depth of the bed. Additional 2-liter portions of miscella are withdrawn after 10, 15, 20, and 30 minutes. At this time the reservoir is disconnected and the final 2 liters of boiling hexane at 50 minutes total extraction time are poured through the column by hand at such a rate that the solvent level is maintained at 6 inches above the bed while the solvent drains freely from the bottom. During this withdrawal the flow rate of the miscella is measured. The extraction temperature is about 145°F. A total extraction time of 50 minutes was chosen since this represents the average time in the commercial extractor. At the end of the extraction period the extractor is allowed to drain and the spent flakes are air-dried for analysis.

Throughout this paper the oil contents of the various materials are reported on a dry basis. Oil in corn germ has been measured by 8-hour Soxhlet extraction of ground flakes (20-mesh) with carbon tetrachloride. There is no official A.O.C.S. method for the determination of oil in corn germ, but if the method for soybean flakes is used, the apparent oil contents are considerably lower than those found by carbon tetrachloride extraction.

Sieve analysis of the flakes also yields useful information. A sample of about 100 g. is placed on a 16-mesh screen, below which there is a 30-mesh screen and a pan. The screens are shaken vigorously for about 90 seconds and the contents of each weighed to the nearest gram. The material through 30-mesh is referred to as "fines." This size was chosen because the bottom of the commercial extractor baskets is a 30-mesh screen.

The method of loading the laboratory extractor is not critical. Flakes should be poured in rapidly (30 seconds), but the rate at which boiling solvent is poured into the extractor is not important. For example, in one series of experiments with flakes containing 22% fines, a) the extractor was filled with dry flakes and the hot solvent poured over it, b) the extractor was filled with hot solvent and the required amount of flakes was poured in rapidly, and c) the flake and solvent levels were kept about equal while the extractor was being filled. Resultant flow rates were 11.8, 13.9, and 13.1 gal./sq. ft./min., respectively. There is however considerable evidence to indicate that when large amounts of fines (about 40%) are present, the method of loading the extractor is critical. From a practical viewpoint there is no need to measure flow rates precisely on high fines content material; it is sufficient to know that the flow rate is very low.

Some idea as to the duplicability of the results obtainable by this method can be seen from the data in Table I. The degree of extraction was found to be reproducible. The error in measurement of flow rates at low values is greater than at higher rates.

It was of interest to observe that during the first portion of the extraction the addition of hot (145°F. or higher) solvent to the extractor causes some evolution of gas and agitation of the top inch or two of flakes. This is believed due to the formation of an azeotrope of n-hexane and water (5.6% water), which boils at 143°F. Formation of this low boiling azeotrope probably accounts for the collection of considerable amounts of water on the upper portion of the shell of basket-type extractors.

TABLE I

Duplicability of Extraction Results

Sample No.	Fines[a]	Residual oil in meal[b]	Flow rate
	%	%	*gal./sq. ft./min.*
1	50	0.8	1.6
1	50	0.7	4.2
2	24	2.3	13.8
2	24	2.3	13.1
2	24	2.3	13.1
2	24	2.6	13.1

[a] Material through 30-mesh screen.
[b] Analyzed by extraction of the spent flakes with CCl_4 for 8 hours.

The particular schedule for the withdrawal of miscella from the extractor was chosen so as to obtain data roughly in accord with that obtained by extracting with a continuous flow of hexane and still avoid handling the large volume of solvent required for continuous flow experiments. A comparison between these two extraction methods can be made by inspection of the data in Figure 2. Flow of solvent was 4.2 gal./sq. ft./min. (1 liter/min.) for the first 21 minutes then 1.1 gal./sq. ft./min. for the remainder of the experiment. The total amount of oil extracted was about the same by both methods despite the fact that in the continuous method a much larger volume of solvent was used.

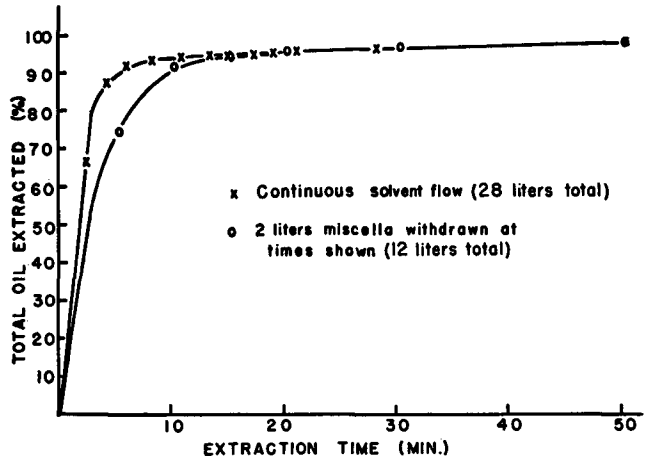

FIG. 2. Effect of rate of miscella withdrawal on amount of oil extracted.

A question that arises during operation of an extractor on scalped material is the degree to which the material should be expelled. The practical answer to this must depend to a large extent on economic factors, but laboratory extraction data from a number of plant and laboratory prepared samples of corn germ indicate that the amount of residual oil in the extracted flakes may be related directly to the amount of oil left in the expeller cake (Figure 3). Correlation is highly significant with a correlation coefficient of 0.93, but an equation for the curve is not presented since it lacks general applicability.

This laboratory extraction apparatus is particularly useful for determining rate of flow of miscella through a bed of flakes equivalent in thickness to that in a

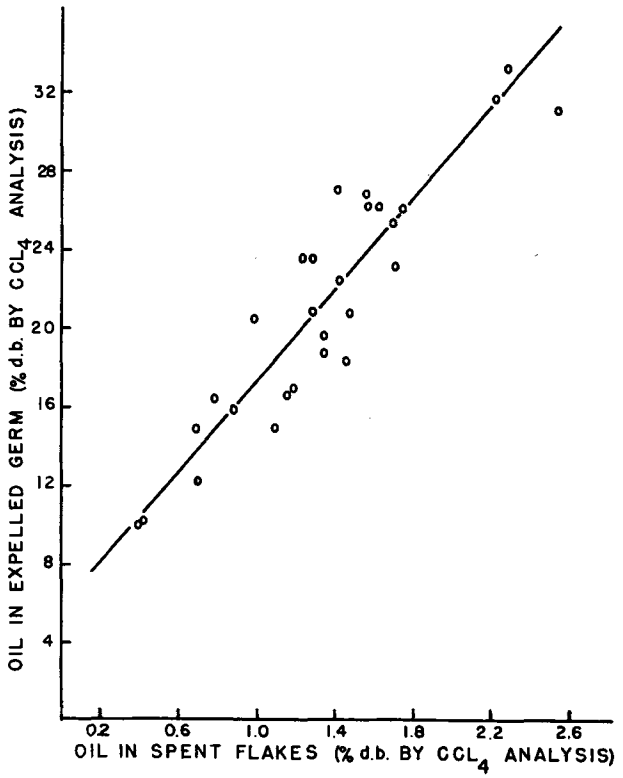

FIG. 3. Relationship between oil in expelled germ and oil in spent flakes.

basket. Data in Figure 4 indicate the inverse relationship between fines content and flow rates through flaked expelled corn germ.

Moisture in the hexane greatly decreases the flow of solvent through the bed. When water equivalent to 0.5% by weight of the hexane was added in the laboratory extractor, the flow rate was reduced by about 50%. Water absorbed on the surface of the bed causes the formation of a dense layer, which impedes solvent flow. Moisture content of this upper layer may be as high as 50% on a solvent-free basis.

Flow rates through beds of various depths can be measured in the laboratory extractor. This information is important for evaluating the applicability of extractors, such as the Rotocel, which require greater flake depths. The flow rate through a bed of flaked expelled corn germ (32% fines) 53 inches deep was found to be 6.4 gal./sq. ft./ min. while the rate through an 18-inch bed of the same material was 9.4 gal./sq. ft./min.

To demonstrate the utility of this apparatus for the extraction of other oil bearing materials, samples of flaked expelled cottonseed and flaked soybeans were extracted with the results shown in Table II (oil content by A.O.C.S. method). Both these materials were more readily extractable than flaked expelled corn germ.

Discussion

The completeness of extraction as measured in this laboratory apparatus has been found to be a standard by which the operational efficiency of basket extractors can be judged. The oil content of spent flakes from the laboratory extractor represents the practical limit of extractability in the large-scale extractor. It is also believed that this laboratory evaluation may be

TABLE II

Laboratory Extraction

Flaked material	Oil content of flakes[a]	Average flake thickness	Fines	Oil content of extracted meal[a]
	% d. b.	*mils.*	*%*	*% d. b.*
Soybean	22.2	14.8	0	0.4
Expelled cottonseed	14.7	14.4	9	0.3
Expelled corn germ	17.9	8.8	21	0.7

[a] By A.O.C.S. Official Method Ba 3-38

applied to any material which can be handled in such plant equipment.

Evaluation of the flow rate data in terms of operation of a large-scale basket-type extractor requires calculation of the minimum flow of solvent through the flakes that may be expected to give successful operation. The plant extractor commonly runs at a 1.5 solvent to flake ratio. The need for this high solvent ratio is related to the very considerable retention of solvent by flaked expelled corn germ. With a knowledge of drainage rate and amount of liquid held in the germ when flooded with solvent, an estimate can be made as to the rate at which solvent must pass through the meal. This minimum flow rate was estimated at 9 gal./sq. ft./min. Correctness of this figure is supported by general plant observations. Poor drainage has been noted on flakes containing 30-35% fines whereas the drainage is nearly always good when the fines content is less than 30%. These limitations are in good agreement with laboratory flow rates. Routine determination of the fines in flaked expeller cake should therefore prove of considerable aid in improving the efficiency of extractions. When the fines content approaches the maximum calculated for satisfactory drainage, corrective measures could be taken

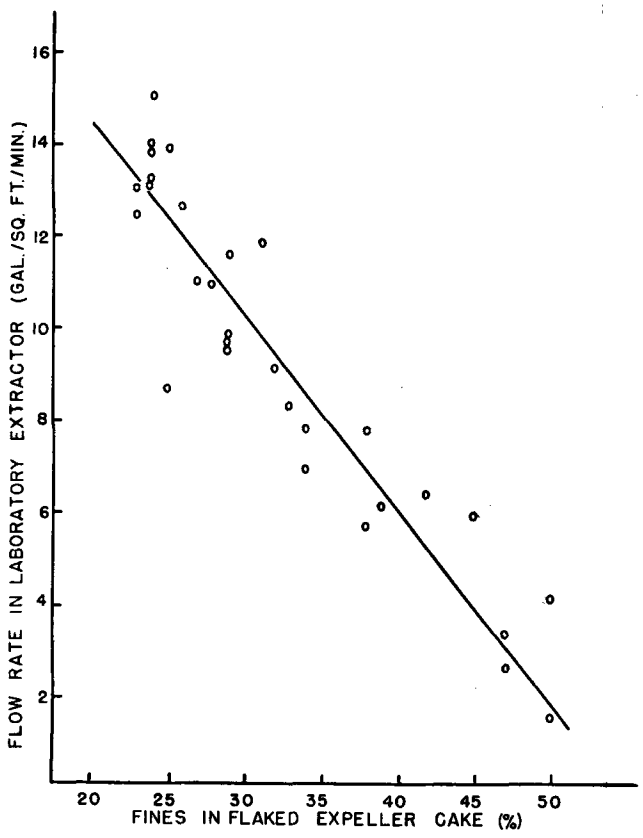

FIG. 4. Effect of fines in flaked expelled germ on flow rate.

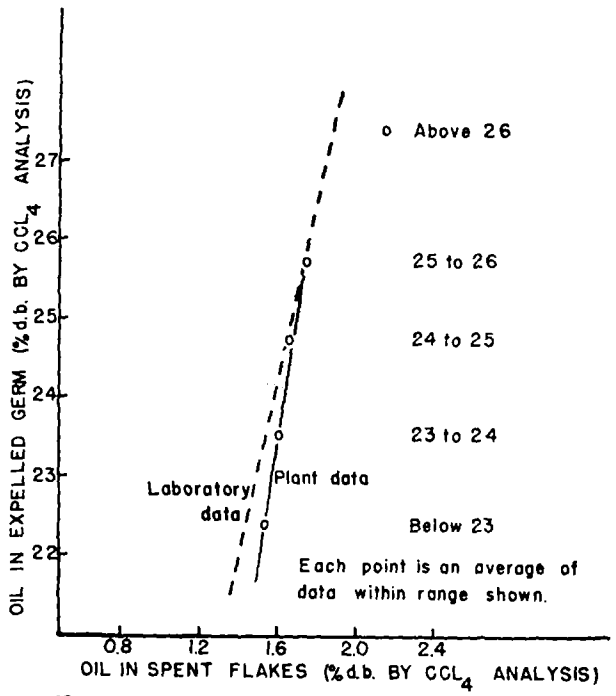

FIG. 5. Plant extraction of flaked expelled germ.

immediately, and well ahead of the time that any sizable quantity of high fines content material enters the extractor.

Occasionally, during the operation of our plant extractor, the moisture content of the hexane pumped to the extractor has been 0.5% by weight. During these periods the drainage of the solvent through the baskets was poor unless the fines content was very low. The reduced flow of solvent through the flakes is in line with operation of the laboratory extractor in the presence of water.

Figure 5 shows data obtained during 3 months of plant operation on flaked scalped corn germ. Each point represents the average of values in the range of oil contents shown. The largest standard deviation (σ) of any of the points (at 2.5% oil in cake) was ± 0.42. Agreement between laboratory and plant data appears to be good. The point representing extraction of highest oil content germ does not fall in line with the rest of the data. This deviation may be due to the fact that high fines content flakes may result on flaking high oil content germ. The subsequent poor drainage explains the unexpectedly high oil content of the extracted meal.

The applications of this method of laboratory evaluation to problems involved in the operation of basket-type extractors are numerous. It is possible to measure the effects of fines, moisture, and bed depth on flow rates, the relationship between oil contents of expeller cakes and spent flakes, the effect of flake preparation on extractability, and the practical limit of extraction of a particular product.

Summary

A simple laboratory extractor is described which gives results that can be correlated with the operation of a basket-type extractor. With this apparatus the flow rate of solvent through the meal can be determined, as well as the rate and completeness of extraction. Using flaked expelled corn germ, simple relationships were found to exist between oil content of the expelled meal and completeness of extraction, and between fines content of the flaked meal and flow of solvent through it.

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A Note on the Preparation of Methyl Esters of Fatty Acids*

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FRACTION distillation of the methyl esters of fatty acids is a very important analytical procedure in determining the composition of fats and oils. Conversion of the glyceride oils is usually effected by saponification followed by hydrolysis of the soaps with a mineral acid. Excess mineral acid is removed by washing, and the fatty acids are esterified by refluxing with an excess of alcohol in the presence of a catalyst, such as sulfuric acid (1). This method is time-consuming and subjects the material to prolonged heating. The yields of esters are from 90-95% with a free fatty acid content of 1-2%.

In work concerned with the composition of rapeseed oil, dimethyl sulfate was used for preparing the esters directly from the soaps in yields of over 99%. This procedure eliminates conversion to the free fatty acids, and the esterification is carried out under nearly neutral conditions with a shorter reflux time. Details of procedure are given below.

One hundred grams oil were saponified by refluxing for one-half hour with 25 g. of potassium hydroxide in 400 ml. methanol. The soap solution was cooled to

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