Improved Extraction Efficiency by Double-Soak, Filtration-Extraction Process

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A simple and inexpensive modification of the filtration-extraction process has been described which permits improved extraction efficiency and reduced solvent requirements over that presently obtained. It comprises an additional soaking step, which can be readily incorporated.

Bench-scale data are presented on three different oil-bearing materials which demonstrate that, at equivalent solvent-meats ratios, the percentage of residual lipids in the extracted meal products can be reduced about 50% or more by use of the double-soak over the single-soak procedure or that, for equivalent percentages of residual oil in extracted meals, the solvent-meats ratio can be reduced significantly.

A laboratory method is described which is recommended for use in the design of commercial-scale installations employing the double-soak, filtration-extraction process.

FILTRATION-EXTRACTION is an established commercial process for the continuous direct extraction of cottonseed, soybeans, sunflower seed, and rice bran (1,2,7,8) and has also been applied successfully on a bench- or pilot-plant scale to peanuts (3), flaxseed (4), sesame (5), castor beans (6), safflower, wheat and milo germs, sugar cane mud, press-cake, and other materials (9).

During the course of the research on filtration-extraction a number of modifications and new techniques were investigated for the purposes of reducing solvent requirements and to improve extraction efficiency and capacity. "Solvent Cooking," previously described (10), offers possibilities of combining the operations of cooking, crisping, and slurrying in a single vessel; reduction of solvent ratio; lower temperature of operation; and improved protein solubility of the final meal product. Another approach, described herein, utilizes "Double-Soak" extraction.

The solvent-processing phase of filtration-extraction consists essentially of a single co-current soaking of the prepared material in the most concentrated or product miscella, which contains about 20–40% oil by weight. Here substantially all of the lipids are extracted. This has been corroborated by Kulkarni (13), Coats (11), and Karnovsky (12) in their recent studies on the mechanism of solvent extraction of oil-bearing materials. The slurry formed is then drained to remove the bulk of the product miscella, and the resulting cake is countercurrently washed and drained three times in a period of 3–5 min., using progressively weaker miscellas and finally oil-free hexane.

It had been observed that some materials exhibited poor washability characteristics and that some allowed more channeling of liquids in the washing operations and thus could not be consistently extracted to a residual lipids content of about 1% in the final meal. It was also observed that the standard displacement washes were not completely effective in replacing or washing out all of the product miscella originally entrained in the filter-cake after the initial drain. To improve this step of the process, the use of an additional soaking step was explored.

A series of laboratory experiments was designed to include a second agitated soaking or slurrying step which would allow the drained marc after either the first or second wash to soak for a short time with one of the countercurrent wash miscellas in order further to extract, by a second equilibration, the more concentrated miscella (static hold-up) still retained or trapped between the cake particles and in the air and oil voids within the solid matrix.

It is the purpose of this report to demonstrate that the incorporation of an additional soaking step in the filtration-extraction process presents the advantages of higher extraction efficiency and low-solvent requirement. Bench-scale data are presented to show the extent to which the claimed improvements can be realized in the processing of three typical high-oil-content crops, namely, cottonseed, sesame, and peanuts. The study was completed in 1957; however publication of the results is believed warranted by the increasing trend among oilseed processors towards more exhaustive extraction.

Raw Materials and Equipment

The cottonseed was of prime quality and was representative of current oil mill receipts. The sesame was domestically produced seed obtained from Clemson Agricultural College, Clemson, S.C. The peanuts were U.S. No. 1 grade shelled Spanish peanuts from that year's crop, purchased from a local peanut butter manufacturer.

Equipment for preparation of the different materials comprised the following pilot-plant size units: Carver 12-in.-wide bar huller; 18-in.-wide double-shaker screener; 18-in.-wide meats purifier; French 12-in.-wide, 5-high roll stand, all rolls of standard diameters; Allis-Chalmers 1-pair high, 12-in.-in-diameter cracking and flaking rolls; Evarts G. Loomis 2-cu.-ft.-capacity, steam-jacketed, mixer-cooker; steam-heated, forced-draft cabinet dryer. All of these units have been described in previous reports (5,14).

Apparatus used for conducting the extraction was a 5¼-in.-in-diameter, bench-scale, filter test unit (15).

Experimental

Preparation of Materials for Extraction. The three raw materials were prepared in accordance with the specific procedures recommended for each in past reports (3,5,16,17). However, in order more effectively to demonstrate the improved extraction efficiency obtainable by the double-soak process, preparations were purposely selected, which when extracted by the regular single-soak process, gave residual lipids somewhat higher than 1.0%.

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Extraction. The single- and double-soak processes were compared with respect to extraction efficiency, mass velocity (filtration rate in pounds of liquids passing through the filter bed per hour per square foot of filter screen area), and solvent-meats ratio (referred to hereinafter as solvent ratio), using the bench-scale test method described by Graci *et al.* (15). It is the standard procedure employed to evaluate the filtration and extraction characteristics of oil-bearing materials for single-soak filtration-extraction, and results obtained correlate well with commercial-scale performance. This procedure is referred to as Method A and is illustrated in Figure 1.

Filtration by the double-soap process was carried out in accordance with the flow diagram shown in Figure 2. This procedure, referred to as Method B, differs from Method A in that the once-washed cake is given a second soaking or slurrying in weak miscella, followed by one wash with weaker miscella, and a final wash with oil-free hexane. Double-soak extraction was also carried out by another method, C, in which the filter-cake is given two washes and is resoaked in a weaker miscella than that for Method B, followed by one wash with oil-free hexane.

Eleven extraction experiments were conducted, in which each of the three freshly-prepared materials was processed by Method A and by Method B; and in the case of cottonseed, by Method C also. Detailed operating conditions employed are listed in Table I.

The extractions with cottonseed and peanuts were conducted by using miscellas for slurrying and washing which were specially adjusted in oil concentraing which were specially adjusted in oil concentraction to levels calculated to correspond, respectively, to those which would obtain under steady-state conditions in a truly continuous operation. In Experiments 4, 5, and 6 with sesame seed, oil-free hexane was used for slurrying and washing.

In all the experiments employing Method A, a soaking period of 30 min. was used. In Experiment 5 a soaking period of 60 min. was used to observe the effect of soaking time. In the experiments employing Methods B and C slurrying periods were 15 min. for the initial slurrying and 30 min. for the reslurrying.

In Experiments 7 through 11 with peanuts the solvent ratio was varied from 1.5 to 0.8 to observe the effect on residual lipids in extracted meal and on the rate of filtration.

In each of the experiments the average filtration rate was determined to observe if any reduction occurred because of the formation of additional fines by mechanical action in the reslurrying operation.

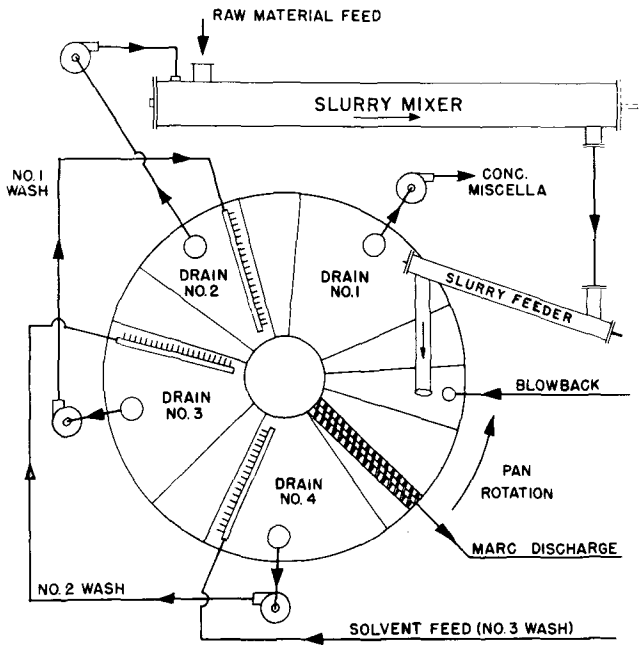

FIG. 1. Flow diagram of commercial-scale filtration-extraction process (extraction phase), employing standard "single-soak" procedure (Method A).

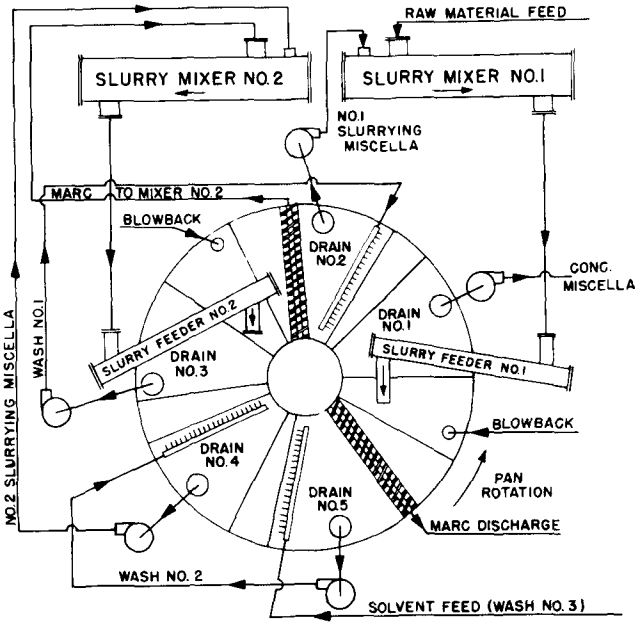

FIG. 2. Flow diagram of commercial-scale filtration-extraction process (extraction phase), employing modified or "double-soak" procedure (Method B).

Results and Discussion

Table I compares the results obtained in bench-scale processing of the three oil-bearing materials by the single- and double-soak filtration-extraction procedures.

In the experiments with cottonseed all three methods are compared. The data show that double-soak Methods B and C gave improved extraction over Method A by a margin of about 0.8% residual lipids content. It is noted that there was no significant difference in the extraction results obtained with Methods B and C. This may be explained on the basis that, whereas the resulting concentration of oil in the miscella in the soaking vessel is somewhat lower (1.5–2.0%) for Method C than that (3–5%) for Method B, the latter has had the benefit of an additional wash after the reslurrying step.

In the experiments with sesame seed the extracted meal produced by Method B was substantially lower (0.5%) in residual lipids than that (1.2%) by Method A. In Experiment 5 the increasing of the soaking time from 30 to 60 min. yielded a final meal somewhat lower in lipids content, but it was still considerably higher than that obtained by Method B. It is obvious that by using Method B the solvent ratio could have been reduced substantially without exceeding 1.0% residual lipids content in the extracted-meal product.

TABLE I

Comparison of Bench-Scale Extraction Results for Single- *versus* Double-Slurrying

Material processed	Cottonseed			Sesame			Peanuts				
Moisture, %[b]	10.6			5.3			4.0				
Lipids, %[b]	28.7			56.1			43.6				
Extraction method[a]	A	B	C	A	A	B	A	A	B	B	B
Experiment No.	1	2	3	4	5	6	7	8	9	10	11
Operating conditions:											
Hexane-meats ratio, lb./lb.	1.1	1.1	1.1	1.7	1.7	1.7	1.5	1.2	1.5	1.2	0.8
Cake thickness, in.	2	2	2	1¾	1¾	1½	2	2	2	2	2
Vacuum, in. mercury	4	4	4	4	4	4	4	4	4	4	4
Slurrying time, min.	30	30	15	30	60	15	30	30	15	15	15
Slurrying temp., °F.	135	135	135	135	130	130	140	140	140	140	135
Reslurrying time, min.	.....	30	30	.....	.....	30	.....	.....	30	30	30
Reslurrying temp. °F.	.....	135	135	.....	.....	130	140	140	140	140	135
Slurrying miscella, % lipids	10.0	10.0	10.0	0	0	0	10.0	12.5	10.0	12.5	19.0
Wash No. 1, % lipids	5.0	5.0	5.0	0	0	0	5.0	6.3	5.0	6.3	9.5
Reslurrying miscella, % lipids	.....	1.5	.....	.....	.....	.....	.....	.....	1.5	1.9	2.4
Wash No. 2, % lipids	0.5	0.5	1.5	0	0	0	1.5	1.9	0.6	0.6	0.8
Reslurrying miscella, % lipids	.....	.....	0.5	.....	.....	.....	.....	.....	.....	.....	.....
Wash No. 3, % lipids	0	0	0	0	0	0	0	0	0	0	0
Washes, temp., °F.	135	140	135	135	130	130	140	140	140	140	135
Results:											
Mass velocity (lbs./hr./sq.ft.)	3100	3300	3300	3200	3400	3800	5400	4100	4800	4500	3300
Extracted desolventized meal:											
H_2O %[b]	6.9	7.2	7.2	7.4	7.6	7.2	7.6	7.5	7.6	7.8	7.4
Lipids, %[b]	1.80	1.00	0.98	1.21	0.92	0.52	1.77	2.72	0.72	0.89	1.12

[a] Method A = slurrying, followed by three washes; B = slurrying and one wash, followed by reslurrying and two washes; C = slurrying and two washes, followed by reslurrying and one wash.
[b] Analysis of Official and Tentative Methods of American Oil Chemists' Society.

The experiments with peanuts provide a comparison of Methods A and B at two levels of solvent ratio and show, in each case, a significant increase in the degree of extraction in favor of the double-soak method. The data also point out that the difference in percentage lipids obtained by Methods A and B is inversely related to the solvent ratio. It is noted that the meal produced in Experiment 11 by Method B, using a solvent ratio of 0.8, was only slightly higher in residual lipids content than that for experiment 10, where a 1.2 ratio was employed, and was considerably lower than the meals produced by Method A, where the respective ratios used were 1.5 and 1.2. The use of such low solvent-ratio levels to remove substantially all of the oil from oil-bearing materials at relatively unimpaired mass velocity rates, together with the high product miscella concentrations obtainable, is believed to be unprecedented in the solvent-extraction industry.

The above results demonstrate that double-soak extraction Method B or C is capable of extracting oil-bearing materials to a substantially lower content of residual lipids in meal, equivalent to about half that obtainable by the conventional single-soak procedure, Method A. The data also show that equivalent extraction efficiency or degree of extraction can be obtained by double-soak extraction at a greatly reduced solvent ratio and that filtration rates for double-soak extraction are not measurably reduced as a result of the additional soaking step. All values for mass velocity are sufficiently high to fall within the range of 2,000–3,000 recommended for commercial feasibility (15).

In discussing the apparent superiority of double-*versus* single-soak extraction, no attempt will be made to discuss the theoretical physical-chemical aspects of solvent extraction. However it is postulated that the attainment of lower residual lipids content in meal by Methods B and C is attributed primarily to the fact that the additional soaking in weak miscella is more effective than displacement washing in removing the concentrated miscella entrained within the pore space of the solid particles of the filter-cake and only secondarily to intrinsic extraction (13), *per se*, of undissolved residual oil and of the more difficultly-soluble nonoil lipids, which are the last components to go into solution (12).

Another feature of double-soak extraction, aside from providing an additional step for thoroughly equilibrating the partially extracted material with weak miscella, is that the reforming of the filter-cake makes possible a repositioning of the fines within the cake, which in turn results in an increased filtration rate.

Reduction in the solvent ratio required to extract an oil-bearing material to any given level of residual lipids content permits the use of smaller-size equipment units for extraction and miscella concentration. The extent to which the above features can be exploited on a commercial scale of operation is discussed below.

Practical Application

Figure 1 is a flow diagram of the extraction phase of the commercial-scale, filtration-extraction process employing the standard single-soak procedure. Figure 2 depicts the process as it would be practiced commercially, using the modified or double-soak procedure. Comparison of the two figures shows that only minor equipment additions and modifications would be required to convert from the single- to the double-soak arrangement. The additional equipment would comprise a second slurry feeder, second slurry mixer, second marc discharge scroll with separate drive, one filtrate receiver, and one pump. The two slurry mixers could be combined into a single vessel with a dividing plate and could be operated with a single drive. The bridges and partitions in the filter valve would be repositioned to correspond with the required rearrangement of the filter screen area segments and to provide for the additional areas required for deposition, drainage, and blowback of the No. 2 slurry.

It is apparent that double-soak extraction can be utilized advantageously by a commercial processor in the following three ways, depending on the particular objective to be accomplished.

1. *Lower Residual Lipids at Equal Solvent Ratio.* For a 200-ton/day plant processing cottonseed and producing about 177,000 lbs. of meal per day, a reduc-

tion in lipids content of 0.1% would represent an increased daily yield of oil product of 177 lbs. This amount of oil priced at 10¢ per pound would be valued at $17.70. A reduction of 0.8% in residual lipids would represent an increased daily oil recovery worth $141.60.

It is important to emphasize that where the double-soak process would be practiced principally to achieve greater lipid recovery, without reducing solvent ratio, a slightly larger filter unit would be required to provide additional screen area for depositing and draining the No. 2 slurry and to accommodate the additional scroll. Thus the additional oil removed would have to be balanced on an economic basis against slightly increased capital investment and operating costs. However it may be reasonably assumed that the added investment would be amply justified in instances where a sizable reduction in lipids content of meal could be achieved.

2. *Equal Residual Lipids at Lower Solvent Ratio.* The prepared material would be extracted by Method B or C to a residual lipid content comparable to that normally obtained by using the conventional process, but the solvent ratio would be substantially reduced. The volume and flow rate of liquids to be handled would be lower, and thus somewhat smaller equipment units (and pumps) could be used to carry out the operations of filtration and miscella concentration. The product miscella concentration would be increased proportionally, and steam costs for evaporating same would be lowered proportionally. Where the solvent ratio would be lowered to a point below that required to produce a flowable slurry in the No. 1 slurry mixer, a portion of the product miscella would be recycled to the No. 1 vessel to dilute the slurry to the desired consistency.

3. *Equal Residual Lipids at Equal Solvent Ratio.* A processor could employ less severe and meticulous conditions for flaking, cooking, rerolling, and any combination of these and still achieve equal extraction performance at equal plant capacity. This would permit the use of smaller and less costly equipment units for preparation and lower power required for their operation; or greater capacity could be obtained for units of the present size. Other benefits that would accrue from not having to comminute an oil-bearing material as severely are that higher filtration rates and higher filter unit capacity would be obtained and the meals produced would be less dusty, thus more easily marketable.

The modified bench-scale method as described herein can be advantageously used to determine necessary data for the design of a commercial plant for the double-soaked, filtration-extraction of any prepared oil-bearing material, at any required capacity rate, and for any percentage of residual lipids desired in the meal product. It is simple to carry out and should prove equally as accurate and reliable as the standard bench-scale method which has proved invaluable for establishing the size of equipment units in the design of commercial plants employing the conventional filtration-extraction process.

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REFERENCES

1. Haines, H.W. Jr., Perry, G.C., and Gastrock, E.A., Ind. Eng. Chem., *49*, 920–929 (1957).
2. Furman, L.J., Amadori, M.E., and Guzman, F.O., J. Am. Oil Chemists' Soc., *36*, 454–457 (1959).
3. Pominski, Joseph, Knoepfler, N.B., Graci, A.V. Jr., Molaison, L.J., Kulkarni, B.S., and Vix, H.L.E., J. Am. Oil Chemists' Soc., *32*, 361–364 (1955).
4. Knoepfler, N.B., Spadaro, J.J., McCourtney, E.J., and Vix, H.L.E., J. Am. Oil Chemists' Soc., *33*, 272–276 (1956).
5. Graci, A.V. Jr., D'Aquin, E.L., Paredes, M.L., and Vix, H.L.E., J. Am. Oil Chemists' Soc., *33*, 645–650 (1956).
6. D'Aquin, E.L., Pominski, Joseph, Vix, H.L.E., Knoepfler, N.B., Kulkarni, B.S., and Gastrock, E.A., "Direct Solvent-Extraction of Castor Yields High Grade Oils," accepted for publication in J. Am. Oil Chemists' Society.
7. Gastrock, E.A., Vix, H.L.E., D'Aquin, E.L., Spadaro, J.J., and Graci, A.V. Jr. (to Secretary of Agriculture), U.S. Patent 2,726,253 (Dec. 6, 1955).
8. Gastrock, E.A., Vix, H.L.E., D'Aquin, E.L., Graci, A.V. Jr., and Spadaro, J.J. (to Secretary of Agriculture), U.S. Patent 2,727,914 (Dec. 20, 1955).
9. Gastrock, E.A., Spadaro, J.J., Gardner, H.K., Knoepfler, N.B., and Molaison, L.J., Oil Mill Gaz., *59*, No. 2, 40–41 (1954).
10. Spadaro, J.J., and Vix, H.L.E., J. Am. Oil Chemists' Soc., *33*, 177–180 (1956).
11. Coats, H.B., and Karnofsky, George, J. Am. Oil Chemists' Soc., *27*, 51–53 (1950).
12. Karnofsky, George, J. Am. Oil Chemists' Soc., *26*, 564–569 (1949).
13. Kulkarni, B.S., D'Aquin, E.L., and Graci, A.V. Jr., J. Am. Oil Chemists' Soc., *32*, 691–697 (1955).
14. King, W.H., Knoepfler, N.B., Hoffpauir, C.L., and McCourtney, E.J., J. Am. Oil Chemists' Soc., *34*, 27–30 (1957).
15. Graci, A.V. Jr., Spadaro, J.J., Paredes, M.L., D'Aquin, E.L., and Vix, H.L.E., J. Am. Oil Chemists' Soc., *32*, 129–131 (1955).
16. D'Aquin, E.L., Vix, H.L.E., Spadaro, J.J., Graci, A.V. Jr., Eaves, P.H., Reuther, C.G. Jr., Molaison, L.J., McCourtney, E.J., Crovetto, A.J., Gastrock, E.A., and Knoepfler, N.B., Ind. Eng. Chem., *45*, 247–54 (1953).
17. D'Aquin, E.L., Spadaro, J.J., Graci, A.V. Jr., Eaves, P.H., Molaison, L.J., Knoepfler, N.B., Crovetto, A.J., Gardner, H.K., and Vix, H.L.E., J. Am. Oil Chemists' Soc., *31*, 606–613 (1954).
18. D'Aquin, E.L., Vix, H.L.E., Graci, A.V. Jr., Gastrock, E.A., and Spadaro, J.J. (to Secretary of Agriculture), U.S. Patent 2,857,411 (Oct. 21, 1958).

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