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Filtration-Extraction of Flaxseed as Affected by Preparation Variables¹

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THE VERSATILITY of the filtration-extraction process has been established previously by its adaptation to the extraction of oil from cottonseed (1, 4, 13), rice bran (7), soybeans (2, 6), peanuts (11), and sesame seed (15). The sequence of operations for the filtration-extraction process is outlined in flow sheet, Figure 1, using flaxseed as the oil-bearing material. The process is similar for all oleaginous materials, but the inherent characteristics of each specific oil-bearing material necessitate the determination of a set of optimum processing conditions (5) for meats preparation, rolling or flaking, cooking, and crisping. Unlike cottonseed and soybeans, flaxseed is not hulled before rolling. The efficiency of extraction of the prepared material is directly related to the particle size or degree of comminution, time and temperature of cooking, the moisture during cooking, the degree of crispness (incompressibility), time and temperature of slurring, and the number and temperature of the washes. It is the purpose of this paper to present data showing the material preparation and extraction conditions required for the efficient removal of oil from flaxseed by the filtration-extraction process.

At the present time there are 32 commercial mills (9) located in 11 states engaged in the extraction of oil from flaxseed. Approximately 60% of the mills use screw-pressing, and 30% use screw-pressing followed by solvent extraction. The remainder use either hydraulic pressing or direct solvent-extraction.

Direct solvent-extraction of flaxseed has not proved successful in the past since the solvent tends to dissolve the connective mucilaginous material which holds the flakes together (10, 14), consequently the flakes disintegrate and become powdery and caky, making the process inefficient primarily because of channeling. Prepressing flaxseed overcomes this difficulty by imparting a rigid structure to the connective mucilaginous material and thus minimizing the in-

herent characteristic of this material to dissolve in the solvent. In filtration-extraction the particular method of preparation, *i.e.*, rolling, mild cooking, and crisping by evaporative cooling, yields a material in which the mucilaginous material becomes hardened, and the fines are minimized by agglomeration during the crisping operation. This preparation produces a material which can be extracted directly and efficiently by the filtration-extraction process.

Material and Equipment

The flaxseed used in this study was obtained from a commercial processor in Illinois and analyzed 6½% dockage, 35% oil, and 9.7% H₂O, and had a test weight of 49 lbs. per bushel.

Pilot-plant equipment used in the preparation of the material for this investigation consisted of: smooth dual flaking rolls; a five-high stand of rolls, the top two rolls of which are corrugated, and the bottom three rolls of which are smooth (12); and a jacketed Loomis³ mixer-cooker (155 sq. in. heat transfer surface area), equipped with a spray nozzle and a steam ejector for the direct addition of moisture during cooking.

Bench-scale equipment shown in Figure 2 and previously described by Graci *et al.* (8) was used to evaluate the filtration-extraction characteristics of the prepared materials.

Experimental Procedure

The general procedure in preparing flaxseed for these experiments consisted of rolling by either the single-pass dual rolls, or through the five-high rolls, cooking in the mixer-type cooker, screening through a 4- or 8-mesh screen, and crisping by aeration on open trays for approximately 20 min., followed by an evaluation of the prepared material for filtration-extraction characteristics. Experiments were designed to investigate the effect of the two principal phases of material preparation, that is, rolling and cooking

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³ The mention of trade names should not be construed as an endorsement by the Department of Agriculture over similar products.

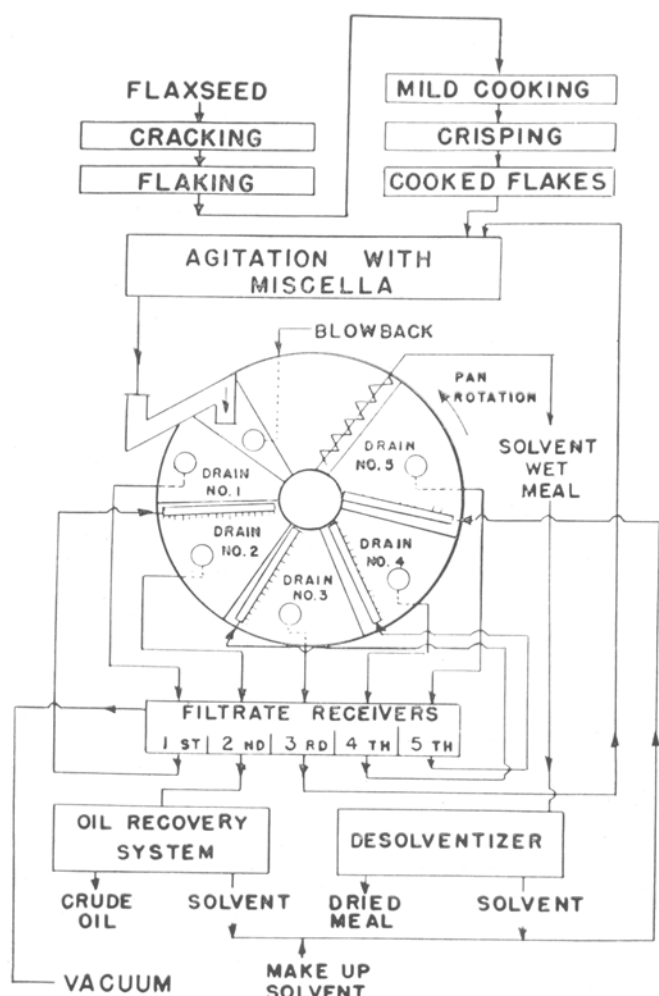


Fig. 1.

and their effects upon crisping, and to establish the conditions necessary for efficient extraction of the prepared materials.

Rolling. To determine the effect of rolling upon extraction characteristics, the following preparations were evaluated:

- (A) One pass thru the dual rolls with 0.002-in. clearance between the rolls.
- (B) Two passes thru the dual rolls with 0.002-in. clearance between the rolls.
- (C) One pass thru the dual rolls with 0.0005-in. clearance between the rolls.
- (D) Two passes thru the dual rolls with 0.0005-in. clearance between the rolls.
- (E) One pass thru the five-high rolls with 0.002-in., 0.002-in., 0.002-in., and 0.000-in. clearance between respective rolls from top to bottom.

Feed rates for all rolling settings were maintained at 300 lbs. per hour.

Cooking. The cooking procedure was standardized as follows. The flakes were charged to the mixer-type cooker, which had been preheated to 170–180°F., and the charge was brought up to 170°F. over a 10-min. period. Sufficient water was added through the spray nozzle over a 3-min. period to bring the moisture of the material to 20–24%. The moisture content of the material was maintained constant, and the temperature was increased to 200°F. over the next 7-min. period. Cooking was continued for an additional 10 min., during which time the moisture content was

reduced gradually to a predetermined level for each experiment. The cooked material was screened and crisped at ambient temperature and was then evaluated by the bench-scale filtration-extraction method at 80°F. (8).

To determine the effect of cooking conditions a second group of experiments were conducted in which the initial cooking moisture content was varied from 12 to 24% and the moisture content of the material at discharge was varied from 6 to 17%. Cooking time was 20 min. and maximum temperature averaged $202 \pm 5^\circ\text{F}$. The cooked material was screened and crisped and evaluated as previously described. Rolling was standardized as described in preparation (E) above, using one pass through the five-high rolls with 0.002-in., 0.002-in., 0.002-in., and 0.000-in. clearance between rolls.

Extraction. To ascertain the optimum conditions for filtration-extraction the following studies were made. Slurrying times were varied from 15 to 60 min. for one series of experiments, and the temperature of slurrying was varied from 84 to 135°F. (the time of slurrying being held constant at 15 min.) for another series of experiments.

Mass velocities and residual lipides of resulting materials were determined. Mass velocity is defined as pounds of miscella through the bed of material per hour per square foot of filter area. The extraction of oil from flaxseed is considered efficient when the mass velocity is above 2,000 (8) and the residual lipides in the extracted cake are below 1%. These two factors are the criterion for practical filtration extraction operations.

Discussion of Results

Table I illustrates the effect of rolling conditions upon mass velocity for the prepared material and upon the residual lipides of the extracted cake. Comparisons should be made only between one and two passes at the same roll clearance setting since the moisture contents of the materials after cooking and crisping were significantly different in various experiments. No significant differences in mass velocity exist between the materials prepared by one and two passes through the rolls at a given roll clearance setting. Mass velocity values for all experiments are in the range considered optimum for continuous filtration-extraction operations (8).

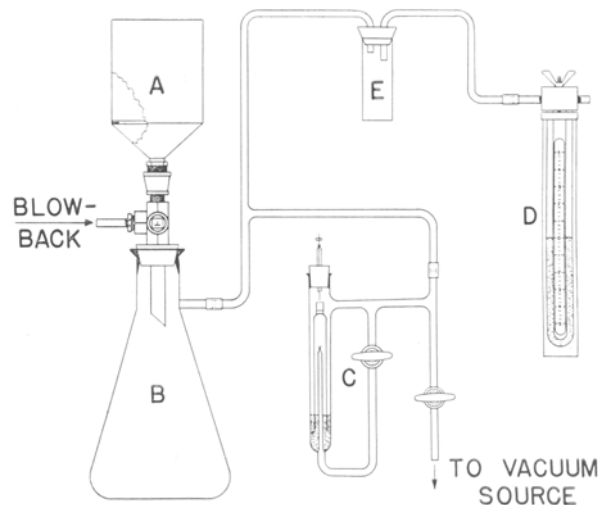


Fig. 2.

TABLE I
The Effects of Rolling

Processing Factors	Single Pass (Dual Rolls)				Five-High Rolls
	1 pass 0.002-in. clearance	2 passes 0.002-in. clearance	1 pass 0.0005-in. clearance	2 passes 0.0005-in. clearance	0.002, 0.002, 0.002, 0.000- in. clearances
Cooking time, min.	20	20	20	20	20
Moisture content of material, maximum during cooking, %	20.5	20.5	20.0	20.0	23.8
Out of cooker, %	16.3	15.6	12.7	13.6	19.8
After crisping, %	13.7	14.8	9.8	10.9	16.4
Slurrying time, min.	15	15	15	15	15
Slurrying temperature, °F.	80	80	95	95	82
Temperature of washes, °F.	80	80	95	95	82
Cake thickness, in.	2½	2½	3	3	2
Mass velocity, lbs./hr./sq. ft.	2910	3141	3312	3312	4052
Residual lipides in cake, %	17.5	6.42	6.35	3.65	3.14

An increase in the number of passes through the rolls resulted in lower residual lipides. For example, the residual lipides were reduced from 17.5% to 6.42% by repassing the material through the rolls at the 0.002-in. clearance, and from 6.35 to 3.65% by repassing the material through the rolls at the 0.0005 in. clearance.

The effects of rerolling through single-pass dual rolls at 0.002-in. clearance after cooking are shown in Table II. Cooking time, temperature, moisture content, and extraction conditions were comparable for all four experiments. Although rerolling decreased the residual lipides, the reduction fell short of the desired maximum residual lipide of 1% or below. Mass velocities, although slightly reduced, remained satisfactorily high.

On the basis of the reduction of residual lipides in the extracted cake that can be expected when the material is subjected to multiple passes through the rolls and/or rerolling after cooking (Tables I and II), an experiment using the five-high rolls was planned and conducted. The results shown in the last column of Table I verified the prediction that the residual lipides decrease with increasing severity of rolling.

It should be noted that these tests were made primarily to compare the effects of different types of rolling conditions, and other conditions were not necessarily optimum. The results reported for residual

lipides are all considered high, but they established a pattern upon which further work was based.

Table III gives typical wet screen analyses (3) of materials produced by the various methods of rolling. It will be seen that the comminution achieved in the five-high rolling is much more severe than that achieved by any of the other preparations studied. For example, 42.5% and 13.0% of the material passed through the 60- and 300-mesh screens, respectively, for the five-high rolling as compared to 12.3% and 5.9% of the material through these respective screens for the next most severe rolling conditions (two passes through the dual rolls at 0.0005-in. clearance).

Table IV indicates that different degrees of cooking, as reflected by the maximum initial cooking moisture content, the amount of moisture removed during cooking, and the moisture level of material as discharged from the cooker, resulted in significant variation in the filtration-extraction characteristics of the materials produced. Four series of experiments in which maximum cooking moisture contents were 23, 17, 14, and 12% are compared at various moisture contents at discharge from the cooker.

On the basis of the resulting mass velocity and residual lipides contents, efficient oil extraction was obtained over the range of initial cooking moisture levels, 17 to 23%, but over a narrower range of moisture levels, 8 to 11%, as discharged from the cooker.

When the maximum initial moisture content during cooking was 23% and the material at discharge from the cooker was 17.2%, a mass velocity of 1267 and residual lipides of 1.79% were obtained. When the 23% moisture content material was discharged from the cooker at 11.3%, a mass velocity of 3726 and residual lipides content of 0.87% were obtained; and when the 23% moisture content material was discharged from the cooker at 10%, the mass velocity was 1932 and the residual lipides content was 0.53%.

TABLE II
Effects of Rerolling After Cooking

Passes through rolls before cooking ¹	One pass		Two passes	
	No	Yes	No	Yes
Rerolling after cooking (one pass) ¹				
Moisture after crisping, %	9.8	9.6	11.0	10.9
Mass velocity, lbs./hr./sq. ft.	3212	2782	3312	2788
Residual lipides, %	6.35	4.14	3.65	2.49

¹ Single pass dual rolls were used.

TABLE III
Typical Wet Screen Analysis of Rolled Materials

Screen mesh	1 pass (dual rolls) 0.0005-in. clearance		2 passes (dual rolls) 0.0005-in. clearance		1 pass (dual rolls) 0.002-in. clearance		2 passes (dual rolls) 0.002-in. clearance		Five-high rolls 0.002, 0.002, 0.002, 0.000-in. clearances	
	% on	% thru	% on	% thru	% on	% thru	% on	% thru	% on	% thru
5	0.4	99.6	0.2	99.8	0.2	99.8	0.4	99.6	0.0	100.0
8	5.1	94.5	5.2	94.6	6.5	93.3	6.2	93.4	0.0	100.0
14									3.8	96.2
16	35.1	59.4	20.9	73.7	51.9	41.4	33.9	59.5		
20	26.7	32.7	26.2	47.5	12.4	29.0	22.6	36.9	19.1	77.1
40	16.6	16.1	21.2	26.3	16.0	13.0	15.4	21.5	20.0	57.1
60	7.5	8.6	14.0	12.3	5.8	7.2	9.9	11.6	14.6	42.5
80	2.3	6.3	2.7	9.6	1.9	5.3	3.5	8.1	11.0	31.5
120	1.0	5.3	1.6	8.0	1.2	4.1	1.7	6.4	7.0	24.5
170	0.9	4.4	1.5	6.5	0.5	3.6	0.9	5.5	6.7	17.8
200	0.2	4.2	0.2	6.3	0.3	3.3	0.4	5.1	1.6	16.2
300	0.4	3.8	0.4	5.9	0.3	3.0	0.6	4.5	3.0	13.0
Thru 300	3.8		5.9		3.0		4.5		13.0	

TABLE IV
Effects of Maximum Moisture During Cooking¹

Experiment No.....	1	2	3	4	5	6	7	8	9	10	11
Moisture content of material, maximum during cooking, %.....	23.0			17.0			14.0			12.0	
Out of cooker, %.....	17.2	11.3	10.0	11.2	10.7	8.1	11.6	7.9	6.5	7.6	5.7
After crisping, %.....	16.0	10.3	9.0	8.7	9.7	6.6	11.4	7.9	6.5	7.3	5.3
Mass velocity, lbs./hr./sq. ft.....	1267	3726	1932	3582	3075	2058	718	3199	2116	434	436
Cake thickness, in.....	1.75	2.25	1.75	2.00	2.25	1.87	1.50	1.87	1.75	1.50	1.50
Moisture in extracted cake, %.....	11.1	10.8	9.5	9.3	9.9	9.2	10.7	11.6	9.8	10.3	9.9
Lipides in extracted cake, %.....	1.79	0.87	0.53	1.62	1.48	0.71	0.48	0.97	0.91	0.42	0.46

¹ Five-high rolls—0.002-in., 0.002-in., 0.002-in., and 0.000-in. clearances used for rolling.

A similar result is reported for the 17% maximum initial cooking moisture level series. The 14% maximum initial cooking moisture level series presents a slightly different picture in that at the 11.6% and 10.1% moisture levels at discharge from the cooker, very low mass velocities and exceptionally low lipides contents were obtained. These low values indicate that the connective mucilaginous material was being dissolved by the solvent, which in turn caused caking and thus slowed down the rate of miscella passage through the bed. These factors can be attributed to insufficient cooking because the mass velocity was significantly increased when these materials were given additional cooking, that is, being discharged from the cooker at 7.9 and 6.5% moisture contents. Residual lipides in the latter cases were acceptable at 0.97 and 0.91%, respectively.

At the 12% maximum initial cooking moisture level the materials produced at 7.6 and 5.7% moisture contents at discharge from the cooker were unacceptable due to the extremely low mass velocities even though the residual lipides were also very low at 0.46 and 0.42%, respectively.

Crisping by evaporative cooling is not too effective where maximum initial cooking moisture contents are 14% and below. Average reduction in moisture during this phase of the processing at the 17 to 23% maximum initial moisture level was from 1 to 2.5% whereas it was only from 0.2 to 0.4% in all but one case where the maximum initial cooking moisture levels were 12 to 14%.

Table IV also shows that the effect of the moisture content of the crisped material in itself is not a criterion. However, when considered in conjunction with the maximum cooking moisture content, the relationship of these two factors has a pronounced effect upon the characteristics of the resulting materials.

This relationship can be noted by comparing the initial cooking moisture content and final moisture content after crisping with their respective mass ve-

locities and residual lipides. For example, with the 23% maximum moisture level it was necessary to decrease the moisture content to approximately 10% to obtain a mass velocity above 2,000 and residual lipides content below 1%. In the case of the 17% maximum moisture level it was necessary to have a moisture content of 6.6% after crisping to achieve the acceptable mass velocity and residual lipides. At the 14% maximum initial moisture content a similar relationship is shown. However at the 12% maximum initial moisture level a decrease in moisture content to as low as 5.3% after crisping failed to produce a material having satisfactory characteristics.

Table V demonstrates the effect of temperature of extraction (slurrying and washing) upon the residual lipides content of the extracted cakes from the various preparations. In all cases the lipides contents of the extracted cakes were significantly reduced by carrying out the extraction at elevated temperature.

The mass velocities however for the experiments in which the maximum initial cooking moisture contents were 17, 14, and 12% were essentially unchanged by extraction at elevated temperatures whereas, in the experiments with the 23% maximum initial cooking moisture content, the mass velocity was reduced by 30% by extraction at elevated temperatures.

The effect of slurrying time is shown in Table VI. Slurrying times of from 15 to 60 min. had negligible effect upon residual lipides content and mass velocity. Based upon these data and data from previous work on other oil-seeds, a 30-min. slurrying time was used as a standard throughout this work unless otherwise noted.

Summary

The data presented show that the filtration-extraction process can be applied to the extraction of oil from flaxseed. General conditions for preparing the material and for extraction have been established. Adequate comminution, cooking, and crisping yielded

TABLE V
The Effect of Temperature of Extraction Upon Residual Lipides¹

Moisture Content of Material			Temperature		Mass velocity	Cake thickness	Moisture of extracted cake	Residual lipides of extracted cake
Maximum during cooking	Discharge from cooker	In slurry	Maximum during cooking	Slurrying and washing				
%	%	%	°F.	°F.	lbs./hr./sq. ft.	(in.)	%	%
23.8	11.3	10.3	198	82 134	3726 2633	2.25 2.00	10.8 10.7	0.87 0.51
17.0	11.2	8.7	202	86 133	3582 3760	2.00 1.75	9.3 9.1	1.62 0.87
14.0	7.9	7.9	198	82 133	3199 3325	1.87 1.87	11.6 10.4	0.97 0.82
12.0	5.7	5.3	204	80 130	436 558	1.50 1.50	9.9 9.8	0.46 0.32

¹ Five-high rolls—0.002-in., 0.002-in., 0.002-in., and 0.000-in. clearances used for rolling.

TABLE VI
Effects of Slurrying Time Upon Residual Lipides and Mass Velocities

Slurrying time, min.	15	30	60
Slurrying temperature, °F.....	130	130	130
Wash temperature, °F.....	130	130	130
Mass velocity, lbs./hr./sq. ft.....	2217	2372	2612
Residual lipides, %.....	1.44	1.28	1.20

materials having necessary characteristics for the successful application of the filtration-extraction process. Mass velocities in excess of 2,000 and residual lipides contents below 1% were obtained when these materials were slurried and extracted at slightly elevated temperatures (130°F.).

The conditions of rolling had an appreciable effect upon the efficiency of extraction of flaxseed. One pass through five-high rolls with clearances of 0.002 in., 0.002 in., and 0.000 in. between respective rolls proved satisfactory. Apparent optimum cooking moisture levels for efficient oil extraction fall between 17 and 24% maximum initial, and 8.0 to 10.5% as discharged from the cooker. Mass velocities were significantly lowered when maximum initial cooking moisture contents were below 14% since at these moisture levels it becomes necessary to reduce the moisture content to below 8%, as discharged from the cooker, to achieve suitable cooking. At these low moisture levels crisping by evaporative cooling is not effective.

The versatility of the filtration-extraction process, which had been previously adapted to the extraction of oil from cottonseed, soybeans, sesame seed, peanuts, and rice bran, has been extended, with some minor but important modifications in the preparation

and extraction conditions, to the extraction of oil from flaxseed.

Acknowledgment

The authors wish to express their appreciation to L. J. Molaison and P. H. Eaves for their assistance in making some of the lipides and mass velocity determinations reported herein.

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Report of Smalley Committee—1955-56

THIS SEASON eight different types of samples were distributed by six subcommittees. These included cottonseed, peanuts, soybeans, meal, vegetable oils, tallow and grease, glycerine, and drying oils. In all, 3,533 samples were distributed to 398 collaborators and more than 13,000 results were tabulated. The distribution and participation are given in Table I.

From this table it can be seen that the preparation and distribution of samples together with the tabulation of the results has become a sizeable task.

The cost of preparation, packaging, and mailing has reached such a level that your chairman feels that an accounting should be given to the Society. In this report we are merely listing the receipts from the collaborators and the expenditures made. These were:

Total receipts.....	\$5195.23
Total expenses.....	5152.59
Net.....	42.64

A detailed accounting will be given to the Governing Board. It has not been the policy of the Smalley Committee to attempt to bring actual revenues to the Society, nor have we wanted the Society to subsidize the program. We attempt to adjust the charge for participation so that the program is self-supporting, with some surplus for the Society's overhead.

TABLE I

	Number of collaborators	Number of samples	Number of determinations per sample
Cottonseed.....	47	10	5
Peanuts.....	10	7	4
Soybeans.....	22	10	2
Meal.....	115	15	3
Vegetable oils.....	92	6	3
Tallow, grease.....	73	5	7
Glycerine.....	24	6	8-3
Drying oils.....	15	6	4

For brevity this year the details of the activities of the various subcommittees will not be discussed in detail. In each case a complete report has been given to the collaborators by the chairmen. Several unusual items or features will be discussed however:

1. The subcommittee on meal voted to present the Smalley Cup, beginning this season for combined proficiency on moisture, oil, and nitrogen. No certificate will be given for proficiency on combined oil and nitrogen. This deviation from precedent had the unanimous approval of the Governing Board.

2. At Mr. Doughtie's suggestion the method of calculating the grades in the Meal Series was changed. The method described here, for the record, gives a more realistic numerical grade as formerly most grades ran 99% and up. The method of grading is as follows:

$$\text{Grade equals } 100\% - \frac{\text{points off} \times 100 \times 0.392}{\text{no. of samples} \times \text{no. of determinations}}$$