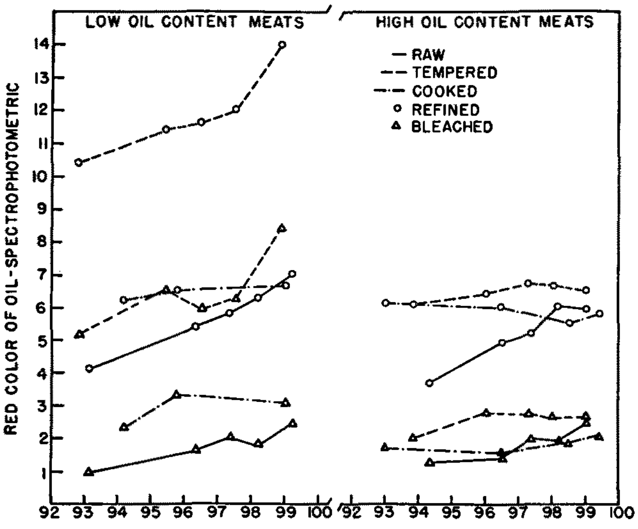

FIG. 2. Relationship of refined and bleached oil color to degree of extraction and method of meats preparation.

The oils obtained by most exhaustive extraction of the raw and the tempered meats exceeded the corresponding oils from the cooked meats in red color. Other than this no consistent correlation between the method of meats preparation and the red color of the oils was found.

The red colors of the bleached oils tended to follow the same pattern as the refined oils, generally increasing with the degree of total oil extraction, although this relationship was not absoiute. The oils from the raw meats tended to produce lighter-colored bleached oils than that from the cooked meats while the cooked meats oils bleached to lighter color than those from the tempered meats.

Summary and Conclusions

Crude lipides fractions were produced from raw, tempered, and cooked meats from two lots of cottonseed by a series of successive stepwise extractions, designed to obtain fractional portions of the total lipides in the order of the difficulty of their extraction. The proximate composition of the crude lipides fractions was determined. It was found that the composition of successive lipides fractions varied with the degree of exhaustiveness of extraction. The fractions obtained by more exhaustive extraction contained greater amounts of undesirable non-neutral oil material and lesser amounts of desirable neutral oil. It was also established that the method used in preparing meats for extraction was of paramount importance in its effect on the composition of the crude lipides obtained. The crude lipides fractions from raw and tempered meats contained large amounts of impurities while the crude lipides fractions similarly obtained from cooked meats were relatively low in impurities.

Crude oils equivalent to varying degrees of total lipides extraction were reconstituted from the crude lipides fractions and evaluated for refining characteristics. The impurities content of the reconstituted oils varied as the degree of total lipides extraction and increases in the impurities content of the oils were generally reflected in disproportionate increases in refining losses and/or refined oil color. The oils obtained from the cooked meats at all degrees of extraction were outstandingly low in refining losses as compared to the oils from the raw and the tempered meats.

Acknowledgment

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Filtration-Extraction of Sesame Seed on a Bench Scale[1]

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THE HIGH QUALITY of the oil and meal from sesame seed and the high oil content of the seed, together with the high seed yield per acre and the plant's adaptability to sub-tropical climates have stimulated increasing interest in commercial cultivation of sesame in the Cotton Belt of the United States. Although sesame has been grown experimentally in limited quantities in this country for many years, profitable production has been seriously hampered by the uneven ripening of the seed pods and by the tendency of the common varieties to shatter. These inherent characteristics have prevented adaptation of this crop to mechanized harvesting methods, and the solution to this problem has been the object of considerable agronomic research

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in the United States in the past 10 years by a number of experiment stations and other groups (8).

Keeping pace with the recent agronomic research on sesame have been the fundamental investigations recently conducted by the Southern Regional Research Laboratory on the chemical and physical properties of the oil and meal components. These findings are reported in a comprehensive article by Budowski and Markley (2) on the chemical and physiological properties of sesame oil, which includes a list of over 254 literature references on the subject.

Since sesame seed has been grown principally for edible consumption rather than as an oil crop, relatively little information has been published dealing with the economic processing of the seed into oil and meal products. It is the purpose of this paper to present bench-scale test data showing the material preparation and extraction conditions required for the efficient direct extraction of sesame seed by the filtration-extraction process, which process has been successfully applied to cottonseed, soybeans, and other oil-bearing materials (3, 5).

Materials and Equipment

Two lots of sesame seed were used. Lot A was a domestically grown seed that had been on hand for several years. This lot was used in the preliminary work and in some of the tests reported herein. Lot B was procured from the Clemson Agricultural College. This seed had been harvested during the previous crop year and was seven months old. Most of the experimental work was conducted with the latter seed.

TABLE I

Screen Analysis of Sesame Seed

Seed identification	A	B
Sieve No.[a]	% On	% On
10	0.0	0.2
12	10.6	51.3
14	70.0	42.0
16	18.0	5.7
18	0.5	0.1
20	0.7	0.0
200	0.1	0.5

[a] U. S. Sieve Series.

Hull content of the seed was approximately 38–40% by weight. The size distribution analysis for the two seeds, given in Table I, shows that 98% or more by weight of the seeds was retained on the 12- to 16-mesh screens. Weight per 1,000 seeds averaged 3.25 g. The chemical analysis on an "as is" basis of each of the two seed lots is given in Table II.

A 5-high roll stand and a set of single-pass smooth rolls were used for size reduction. The 5-high rolls were 12 in. long, the top 4 being 14 in. in diameter and the bottom 16 in. Lower three were smooth, the upper 2 were corrugated; the topmost having 8 cuts per inch on a ½-in. spiral, and the second, 16 cuts per inch on the same spiral. Roll speed was 178 r.p.m. The one-pass rolls are 12 in. in diameter by 12 in. long and revolve at 220 r.p.m.

TABLE II

Chemical Composition of Sesame Seed

Seed identification	A	B
Moisture,[a] %	6.3	6.2
Oil,[a] %	54.0	52.8
Nitrogen,[b] %	3.6	3.8
Protein (N × 6.25), %	22.5	23.7
F.F.A. of oil,[c] %	3.2	6.4

[a] Analysis by method of Stark and Hoffpauir (9).
[b, c] Official A.O.C.S. methods Ba 4-38 and Ab 5-49, respectively.

Cooking was conducted in a vapor-tight, steam-jacketed vessel of adequate capacity for cooking batches of up to 15 lbs. of material. The vessel was equipped with steam-heated meshing agitators, with a spray nozzle and a steam ejector for direct introduction of water and/or steam or air, and with means for measuring any water removed.

A pilot-plant size, steam-heated forced-draft tray dryer was used to dry the seeds.

Filtration-extraction of the variously prepared materials was carried out, using the bench-scale test unit previously described (6).

Investigation of Processing Variables

Systematic experiments were designed to establish the optimum conditions for the preparation and for the efficient extraction of the seed.

I. PREPARATION. Variables investigated were: particle size reduction or degree of comminution of sesame seed necessary to achieve high extraction efficiency at a satisfactorily high filtration rate; effect of seed moisture content upon effectiveness of comminution; time, temperature, and moisture level during cooking; and final moisture content after cooking and crisping.

Particle Size Reduction. It was ascertained in the initial experiments that, in order to attain thorough extraction of sesame seed at acceptably high filtration rates, the seed must be sufficiently comminuted so that about 2% of the total weight is retained on a 20-mesh screen or coarser, and about 50% is fine enough to pass through 300-mesh. It was also observed that to achieve the necessary high degree of comminution the seed of either of the two lots tested should preferably be partially dried prior to the rolling operation. Grinding *versus* rolling of the dried seed was not explored, but mill grinding of the undried seed resulted in buttering. The seed was dried at about 175°F. for up to 2 hrs., depending on the moisture reduction desired. The need for pre-drying would of course have to be determined for any particular lot of seed.

Table III shows the effect of moisture content of the seed upon the degree of comminution by rolling. Roll settings and rolling rates for both tests were

TABLE III

Effect[a] on Particle Size Distribution of Drying Sesame Seed Prior to Rolling

Material, Lot B	Undried seed	Dried seed
Moisture, %	6.0	2.4
Sieve No.[b]	% On	% On
5	0.0	0.0
8	0.0	0.0
14	0.4	0.0
20	3.6	0.8
40	14.7	6.3
60	12.6	11.4
80	4.1	4.5
120	9.0	7.1
170	10.4	3.9
200	0.8	0.8
300	5.7	4.4
Bottom	38.7	60.8

[a] Determined by wet screen analysis method (4) using commercial hexane.
[b] U. S. Sieve Series.

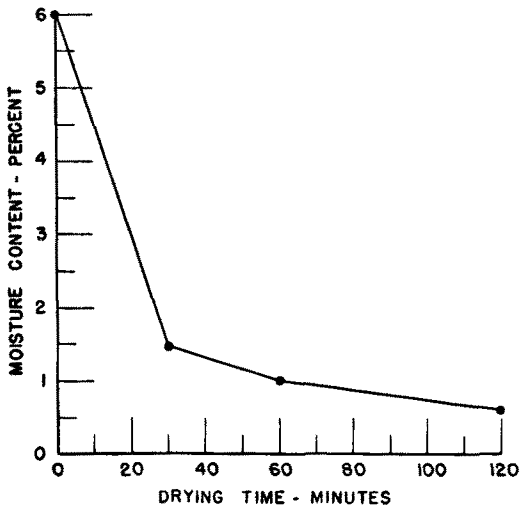

FIG. 1. Drying rate curve for whole sesame seed heated at 101°C. in forced-draft oven.

identical. The second and third columns compare the particle size distribution as determined by wet screen analysis (4) of undried seed (Lot A) of 6% moisture content with the seed dried down to 2.4% moisture content. It is apparent that drying before rolling was effective in increasing the percentage passing through 300-mesh to 61% as compared to 39% for the undried seed.

Figure 1 is a typical rate curve for the drying of sesame seed at 101°C. in a forced draft oven. This curve, plotted from the data of Stark and Hoffpauir (9), shows that, under the stated drying conditions, moisture is removed from the whole seed at a relatively rapid rate down to the level of about 1.5% moisture content but at a relatively slower rate below 1.5%. Based on these data, a moisture content of 2.5% prior to rolling, as is recommended herein, falls within the more economic range of drying conditions.

Cooking and Crisping. The cooking operation serves two principal functions. First, by the action of heat and moisture, it renders the oil more easily removable during the extraction phase of the process. Secondly, under proper conditions of moisture, temperature, and time, it causes the fine particles to agglomerate into larger ones. The hot, moist material discharging from the cooker was cooled and crisped by shaking it through a 1/4- or 1/8-in. mesh hardware cloth. This screening step, aside from breaking up any "moisture balls" which might have formed in cooking, allows the material to decrease in temperature to 110–130° F. and uniformly lose about 1 to 4% in moisture content. This combined effect yields a crisp, porous material which is relatively granular and incompressible, possessing characteristics favorable for rapid filtration.

The cooking vessel was operated in a manner which simulated the operation of a conventional stack-type of cooker. Accordingly the cooking cycle was carried out in the following successive stages: material heating, moisture addition, cooking, and moisture removal or drying.

The following operating conditions for cooking and crisping were investigated. The cooking period was varied between 15 and 45 min. Maximum moisture content reached in cooking was varied within the range 8 to 18% by the addition of water. In all cases the temperature of the rolled seed was allowed to rise to at least 175°F. before addition of moisture was begun. In the final stages of cooking the material was dried to between 4 and 12% moisture content. Temperature conditions employed in any one cooking experiment ranged from 175 to 212°F. at the start, to 190 to 230°F. at the end of the cook. Moisture content after crisping was varied between 4 and 9%.

Figure 2 shows the agglomeration accomplished by cooking and crisping of the rolled seed. Curve A is a graphical representation of the particle-size distribution of the material prior to cooking; curve B gives comparable data on the material after it had been subjected to the cooking and crisping steps.

II. EXTRACTION. Extraction variables studied included solvent to prepared feed material ratio, slurrying time, filter-cake thickness, and temperature of extraction (slurry and washes). All of the above variables were evaluated with respect to extractability (extraction efficiency) and mass velocity (filtration rate expressed as pounds of filtrate per hour per square foot of filter screen area).

The bench-scale unit for conducting the filtration-extraction tests was specifically designed to simulate the operating conditions of slurrying, slurry filtration, countercurrent cake washing, blowback, and vacuum drainage to which the prepared material would be subjected on a standard continuous 3-ft. in diameter, pilot-plant size, rotary, horizontal, vacuum filter (3). It has been pointed out (6) that results obtained with this unit, in the extraction of a wide variety of oil-bearing materials, have been closely correlated with pilot-plant scale performance and demonstrated for cottonseed and soybeans (1) to be readily translatable to commercial-scale application.

From a practical standpoint it was deemed necessary in the processing of this seed that extraction efficiencies be attained of at least 99.0%, which cal-

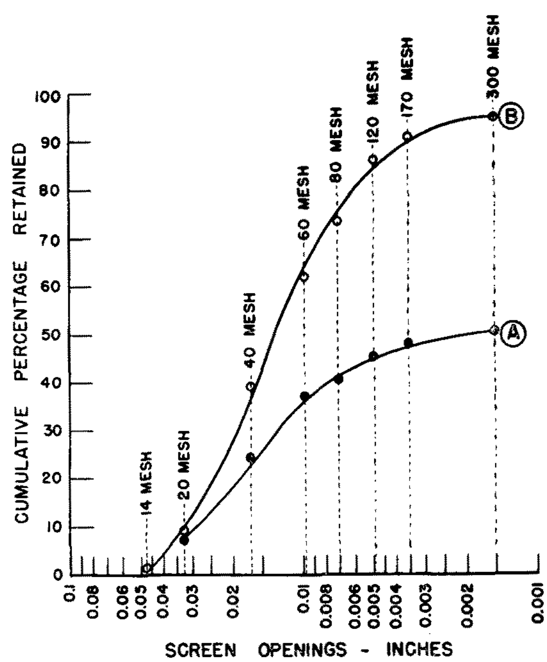

FIG. 2. Graphical comparison of particle-size distribution of comminuted sesame seed before (A) *vs.* after (B) cooking and crisping.

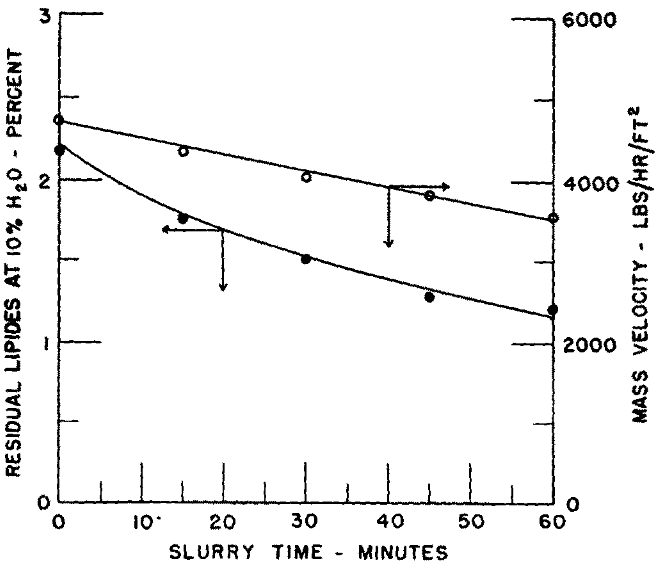

FIG. 3. Effect of slurry time on mass velocity and residual lipides content of desolventized extracted cake.

culates to be about 1.25% of oil or lipides remaining in the desolventized cake (7). To insure this high level of extraction efficiency with sesame it was necessary to employ a higher solvent ratio (1.7 to 1.0) than that (1.2 to 1.0) recommended for cottonseed because of the higher content of oil in sesame seed. Moreover, because of the higher ratio required, it was necessary to attain a higher mass velocity than that required for cottonseed. Calculation of the recommended mass velocity for sesame was based on a rather extensive backlog of filtration-extraction tests with prepared cottonseed material, which indicated that a cottonseed preparation must have a mass velocity of about 2,000 (6), as measured by the bench-scale test equipment, to insure trouble-free continuous operation of the 3-ft. in diameter (3.5 sq. ft.) pilot-plant filter at a handling capacity of about 9 tons of whole cottonseed (5.6 tons of meat-hulls mixture) per 24 hrs.—a rate considered satisfactorily high for commercial feasibility. The operating conditions under which this bench-scale, mass velocity was correlated with pilot-plant operation were a 2-in. filter cake thickness, recycling of the full miscella for fines removal, a 1.2 to 1.0 ratio of solvent to prepared material, 3 cake washings, and a 7.5-lbs.-per-minute feed rate.

On the basis of the above a mass velocity of around $2{,}000 \times \frac{1.7}{1.2}$ or 2,900 lbs./hr./sq. ft. would be required to insure trouble-free operation of the pilot-plant continuous filter with prepared sesame seed at a processing rate equivalent to that considered satisfactorily high for cottonseed.

Commercial hexane was used for both slurrying and cake washing. Concentration of the resultant product miscella at the 1.7 solvent ratio using washes adjusted in oil content to simulate countercurrent operation, calculated approximately 29%, based on approximately 40% solvent holdup in the marc.

Solvent Ratio. Solvent ratio is defined as the weight of hexane used per unit time for the final-cake wash, divided by the weight of oil-bearing material entering the slurry mixer per unit time. Solvent ratios as low as 1.3 and as high as 2.1 were employed. The detailed results of these tests are not included, but the residual lipides showed a continuing decrease with increase in the solvent ratio as would be expected. However the reduction rate at ratios higher than about 1.7 was not appreciable enough to be justified. The results also demonstrated that mass velocities tended to decrease at solvent ratios in excess of 1.7.

Slurry Time. Slurry time is the length of time the prepared material is soaked under agitation with solvent or miscella prior to filtration and washing. During this period the bulk of the oil (lipides) contained in the material goes into solution. Figure 3 shows the effect of time of slurrying under normal agitation upon final residual lipides and upon mass velocity. The lower curve indicates that the cooked and crisped sesame material extracted rapidly to about 2.3% residual lipides content but relatively slowly from 2.3% to 1.2% residual lipides content. The upper curve reveals the effect of slurry time upon mass velocity. It is apparent that the effect of increased slurry time up to 60 min. is to reduce the mass velocity at a uniform but only nominal rate.

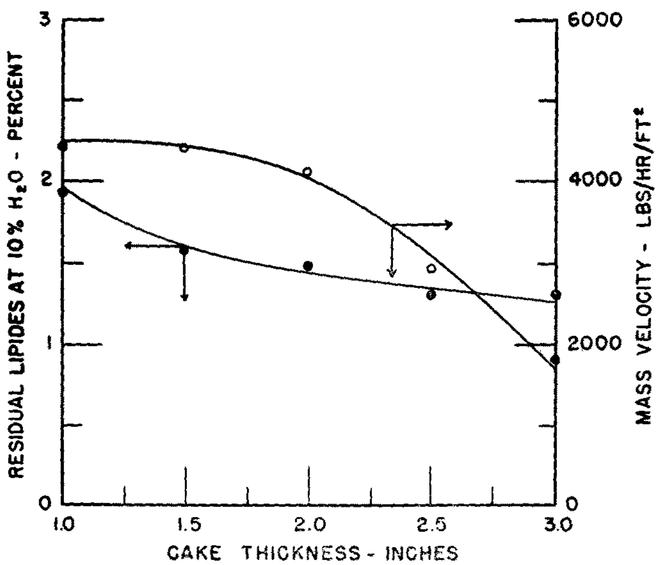

FIG. 4. Effect of cake thickness on mass velocity and residual lipides content of desolventized extracted cake.

Cake Thickness. The curves in Figure 4 illustrate that increase in depth of cake up to 3 in. on the filter resulted in some decrease in both residual lipides and mass velocity. This has been the experience with most oil-bearing materials tested to date. The bottom curve indicates a gradual but slight reduction in residual lipides with increase in cake thickness, for cake thickness above 2 in. A somewhat greater reduction occurs for cakes less than 2 in. thick. These results reflect improved effectiveness of the displacement type of wash as cake thickness is increased. The top curve shows that the mass velocity drops off sharply at cake thicknesses in excess of 2 in.

Extraction Temperatures. Experiments to determine the effect on extraction efficiency of increase in the temperature of extraction, *i.e.*, temperature of slurry and washes, showed that only a slight advantage with respect to lipides reduction was realized at temperatures above 120°F. Moreover, at temperatures of 140°F. and higher, mass velocity began to fall off, especially at cake thickness above 2 in. This

TABLE IV

Material Preparation Data

Experiment No.	1	2	3	4	5	6
Seed identification	A	A	A	B	B	B
Seed drying						
Moisture before, %	5.0	5.0	5.0	6.0	6.0	6.0
Moisture after, %	1.3	8.7 [a]	2.2	1.4	2.7	1.4
Seed rolling						
Rolls, type	1-pr. high	1-pr. high	1-pr. high	5-high	5-high	5-high
Roll spacings, in.	.002	.002	.002 [b]	.... [c]	.... [c]	.... [c]
Moisture after, %	1.4	8.7	2.2	1.7	2.7	1.7
Fraction through 300 mesh, %	38.9	12.7	30.7	54.3	49.6	54.3
Cooking						
Cycle time, min.	....	30	30	15	33	30
Temperature range, °F.	....	180–215	191–215	200–216	190–229	200–225
Moisture, maximum in cooking, %	....	14.0	14.4	9.6	17.4	14.8
Moisture out of cooker, %	....	9.3	7.5	9.6	9.2	6.2
Crisping						
Screen mesh, in.	....	¼	¼	⅛	⅛	⅛
Moisture before, %	....	9.3	7.5	9.6	9.2	6.2
Moisture after, %	....	7.3	6.6	7.1	6.2	5.3
Fraction through 300 mesh, %	....	2.1	9.0	20.0	5.1	6.4

[a] Moisture added. [b] Material repassed through rolls. [c] Roll spacings, inch: .008, .003, .001, .001.

is attributable to the greater degree of particle-size reduction of the solids in the slurry mixer, which occurs as a result of lowered viscosity of the mixture.

Integrated Bench-Scale Runs

In Tables IV and V is presented a detailed tabulation of experimental operating conditions and extraction results obtained in six typical integrated runs, in which the various preparation and extraction variables were studied and evaluated. The selected runs differ from each other mainly in the conditions used for preparation of the seed for extraction. The extraction conditions employed were those found best in the studies reported above.

In Run 1 the seed was oven-dried and severely rolled. No cooking was employed. As noted this material upon extraction exhibited a very low mass velocity and extracted very poorly despite its small average particle-size, as indicated by the fact that 38.6% passed 300-mesh. These results indicated the need for cooking to agglomerate the fine particles in order to improve the filtration rate and to render the oil more easily extractable.

In Run 2 the seed was moistened and equilibrated for 24 hrs. before rolling. Upon rolling, the percentage passing 300-mesh was only 12.7% compared to the 38.6% obtained in Run 1, in which the same roll settings and feed rate were employed. The rolled material was given a moderate moist cook, and after crisping it was subjected to the bench-scale filtration-extraction test. Mass velocity for this material was relatively high, and extraction was poor, both of which were attributed to insufficient comminution.

Run 3 denotes the effect on oil extractability of more severe comminution of the rolled seed prior to cooking. Percentage through 300-mesh was increased over Run 2 to 30.7 by drying ahead of rolling. This preparation showed improved filtration-extraction characteristics in that the filtration rate was acceptably high and extraction was improved over Run 2. These results indicated that, all other factors being equal, extraction efficiency is mainly a matter of particle size prior to cooking. This is illustrated more pointedly in Run 4 as explained below.

Run 4 shows the importance of moist cooking. In this experiment the dried seed was more severely comminuted than in Run 3 so as to yield a fraction through 300-mesh of 54.3%. The rolled material was subjected to a short, relatively dry cook. Wet screen analysis of this cooked and crisped product showed that, for lower moisture levels in cooking, only partial agglomeration of the very fine particles is accomplished. The cooked and crisped material upon extraction exhibited a low mass velocity. The lower residual lipides content also reflects the high proportion of fines associated with poor agglomeration in cooking. It is pointed out that high extraction efficiency so obtained is of little practical importance where mass velocity values are impractically low. The above data point up specifically the dependence of filtration rate upon cooking to achieve adequate agglomeration of the very fine particles.

Runs 5 and 6 incorporated all of the recommended conditions for preparation and extraction that had been tentatively established. The seed was dried further for Run 6 (1.4%) than for Run 5 (2.7%), which resulted in a somewhat smaller average particle size after rolling, as would be expected. Moderate cooking temperatures, times, and moisture levels were employed.

Two bench-scale extraction tests (see columns A and B—Table V), one at 30 min. and the other at 60 min. slurry time, were made on the cooked and crisped product from each of the runs. Solvent ratio throughout was 1.7 to 1. The residual lipides contents of the desolventized cakes indicated that high extraction efficiency was obtained in each of the four tests and that the best extraction, 0.92% residual lipides, was obtained in Run 6-B, using the stated combination of severe rolling, moist cooking, crisping, and a slurry time of 60 min. Mass velocity values in all cases exceeded the 2,900 lbs. per hour per square foot rate which, as explained earlier, is considered sufficiently high to insure that sesame seed can be handled on a standard horizontal filter at a satisfactory capacity rate per square foot of filtering area.

Summary and Conclusions

Filtration-extraction has been successfully applied to the processing of sesame seed on a bench scale, in which various procedures for preparation and solvent extraction were evaluated.

Conditions recommended for preparation of this seed for extraction are as follows: crushing through 5-high rolls set to yield a comminuted material having not more than 2% of its weight on 20-mesh or

TABLE V
Bench-Scale Filtration-Extraction Data

Experiment No.	1	2	3	4	5A	5B	6A	6B
Solvent/seed ratio	1.5	1.5	1.7	1.7	1.7	1.7	1.7	1.7
Slurry time, minutes	30	30	30	30	30	60	30	60
Extraction temperature, °F.								
Slurry[a]	85	135	140	135	140	140	135	130
Washes[a]	85	135	140	135	135	135	135	135
Vacuum, in. Hg	4	4	4	4	4	4	4	4
No. of washes	3	3	3	3	3	3	3	3
Cake thickness, in.	2.0	2.0	1⅝	1⅝	2.0	2.0	1¾	1¾
Mass velocity, lbs./hr./ft.[2]	120	5,420	2,810	520	4,150	3,660	3,230	3,350
Desolventized meal analysis								
Moisture, %	5.6	9.4	7.8	8.0	8.1	8.0	7.4	7.6
Residual lipides, %	5.24	6.27	2.47	0.90	1.56	1.27	1.21	0.92
Extraction efficiency, %	95.3	94.6	98.0	99.2	98.6	98.9	99.0	99.2

[a] Commercial hexane used for slurry and washes.

coarser, and not less than 50% passing through 300-mesh screen (predrying of the seed to 1.5–2.5% moisture content will increase degree of comminution at the same rolls settings and rate of throughput); preheating to 170 to 180°F.; addition of moisture to about 16 to 18%; cooking, followed by drying up to 225 to 230°F. to a moisture content of the discharged material of about 7 to 9%, and a total cycle time of about 30 min.; crisping by evaporative cooling to a temperature of about 130° F. and a final moisture content for extraction of about 5–7%. Conditions found adequate for efficient extraction are: slurrying for 45 to 60 min., solvent ratio of 1.7 to 1.0, three washes, cake thickness of 1¾ to 2 in., and temperature of extraction of about 130°F.

Based on the close correlation obtained to date between bench- and pilot-plant scale, filtration-extraction results for a wide variety of vegetable oil-bearing materials and between pilot-plant and industrial scale in the case of cottonseed and soybeans, it is anticipated that no serious difficulties should be encountered in the commercial processing of sesame seed by filtration-extraction.

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Seasonal Variation in Character of Lipides in Pure Lines of Spanish Peanuts

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SOME YEARS AGO Spanish peanuts were reported to be slightly more susceptible to oxidative rancidity development than either Runners or Virginias (4, 7). This characteristic is associated with a slightly higher percentage of linoleic glycerides in Spanish oils (4).

The results reported here are incidental to an investigation which has as its primary objective the selection of Spanish strains less susceptible to the above-mentioned oxidative rancidity development than those now in production. As data began to accumulate in this general investigation of rancidity, it became evident that these Spanish oils were not showing the variation in unsaturation which the literature shows is the case with oils from other plants. It was then decided that the seasonal variation in composition of Spanish oils should be investigated further, even though only a limited number of samples were available.

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Experimental

Nuts were obtained over a period of four seasons from the Botany Department of the Georgia Experiment Station, which has conducted a peanut breeding program for many years. Most of the strains examined resulted from crosses made at least 15 years previous to this investigation. The others were selected from old, standard types. Usually peanuts are self-pollinated, and genetic character is well maintained under field conditions. However the Spanish peanut is subject to some crossing, and for that reason the Spanish strains were grown in isolated fields and line-selected each year in the field to maintain the character of each strain. Inasmuch as changes have not been observed over a period of several years, genetic character must have been fixed in these strains long before the present study was undertaken.

Peanuts grown on sandy loam soil near Tifton in the Coastal Plain area of Georgia were obtained during the 1952, 1953, and 1955 seasons. Nuts were also obtained in 1954 and in 1955 from Experiment in the