with erucic and elaidic acids) suggests that stearolic acid is somewhat more polar than the other two compounds.

Table IV gives solubility data for stearic, oleic, and linoleic acids in certain hydrocarbon solvents. The differences in solvent properties of the various hydrocarbons were not great, but solubilities were significantly higher in methyl cyclohexane than in the other solvents and were lowest in neohexane.

The solubility of stearic acid was also studied in dimethyl formamide. The data obtained were as follows:

Temp.	Solubility (g./100 g. solution)
10°	1.15
0°	0.38
−10°	0.102
−20°	0.024

The solubilities are higher than for most of the other solvents. Dimethyl formamide was not further studied because of its high toxicity and its low volatility, which made solvent removal difficult. It was not considered a convenient medium for use in the low temperature crystallization procedure.

For purpose of comparison Table V has been prepared. This table compares certain solubility determinations made during this study with values which have been reported by other investigators. In general, the agreement is good. However several discrepancies are apparent. The solubilities in ethyl acetate, for example, tend to be considerably lower than those of Hoerr and Harwood (5). The value listed for diethyl ether, on the other hand, is significantly higher than that of these investigators.

Attempts were made to prepare pure linolenic acid, but a product of sufficient purity for solubility measurements was not obtained in time to be included in this study. This is unfortunate since linolenic is one of the most important of the unsaturated fatty acids. Solubility studies with mixtures of linoleic and linolenic acids would also be of great practical value as our work has indicated that these compounds exhibit marked mutual solubility effects.

Summary

A number of highly purified fatty acids have been prepared and their solubilities determined in six common organic solvents within the temperature range from 10° to −70°. The acids studied were palmitic, stearic, oleic, elaidic, petroselinic, petroselaidic, linoleic, stearolic, arachidic, eicosenoic, behenic, erucic, and brassidic. The solvents used were methanol, ethyl acetate, diethyl ether, acetone, toluene, and n-heptane, representing six different solvent types. A limited study was also made with a series of hydrocarbon solvents in order to note any effects of solvent structure on fatty acid solubility. Data are discussed with respect to their application in separating various fatty acid mixtures by low temperature crystallization.

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Filtration-Extraction of Peanuts on a Bench Scale[1]

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IN THE UNITED STATES in 1952, from the 685,000 tons of peanuts produced, approximately 100,000 tons were processed to yield oil and meal products (13, 14). Most of this processing was hydraulic and screw pressing with only an insignificant portion processed by solvent extraction. Should the cost of peanut production in the United States be reduced to a level where peanut oil and meal can compete with corresponding products of cottonseed and soybeans, the amounts of peanuts grown and processed would probably increase substantially.

Peanut oil is considered one of the better quality vegetable oils and sells at a premium price. The solvent-extracted meal, besides being a cattle feed, is an excellent source of industrial and edible protein (1). The removal of oil by solvent extraction from an oilseed of high fat content, such as peanuts, poses many technical problems (5). As a solution to some of these problems prepressing is currently used prior to solvent extraction in some instances. A new direct solvent-extraction process developed at this Laboratory and called Filtration-Extraction makes prepressing unnecessary. The filtration-extraction process has been applied on a pilot plant scale to cot-

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tonseed, rice bran, soybeans, flaxseed, and milo germ (3, 4, 6, 7, 8) and on a bench scale to sesame seed (10). Successful filtration-extraction of an oil-bearing material depends primarily upon meat preparation prior to extraction (12). The raw materials are conditioned by flaking, cooking, and crisping so that a relatively incompressible material having a proper particle-size distribution with a minimum of fines is formed (8, 12). The criteria for such prepared materials are high mass velocities during filtration and low residual lipids in the extracted meals (8, 12). In addition to these properties it is desirable for industrial uses that extracted peanut meals should have high protein solubility (1). Application of the filtration-extraction process to peanuts on a bench scale is reported in this paper. The work shows that flaked peanuts can be cooked at relatively low temperatures with little moisture addition to obtain a crisp material which gives high filtration rates, low residual lipids, and high protein solubility in extracted meals.

Materials Used

The peanuts selected for this work were shelled Spanish U. S. Nos. 1 and 2 grades with an oil content of 46-48% and moisture content of 6-7%.

Equipment and Methods

Peanuts for extraction were prepared with pilot plant equipment which included Allis Chalmers [4] single-pass cracking and flaking rolls, French 5-high cracking and flaking rolls, a French 5-high jacketed stack cooker, and an Evarts K. Loomis mixer. The extraction equipment used was either a vacuum crock filter (12) or a metal filter cylinder (9) with a removable screen.

The French cooker and the Loomis mixer were used for cooking peanut meats in their preparation for extraction. The Loomis mixer was used for the small-size cooking experiments and the French cooker for larger size cooks. Data from the smaller cooks can be generally translated to the larger cooks with minor changes in the variables involved.

In the French cooker 50-lb. batches of peanut flakes were cooked in the first ring after moisture addition, and then dried in the second and third rings. In the Loomis mixer 10-lb. batches of peanut flakes were cooked after moisture addition with the cover closed and then dried with the cover open. The cooked materials were cooled prior to screening or rerolling.

For laboratory filtration-extractions using the metal filter cylinder the prepared peanuts were slurried with approximately 10% oil-hexane miscella, and the washes contained approximately 5%, 1.5%, and 0% oil concentration. Where the vacuum crock filter was used, initial slurrying was made with pure hexane, and all other washings and slurryings were made with actual miscella washings from the previous cycle.

Experimental Results

To find the optimum conditions of preparation the following variables were studied: rerolling, moisture addition during cooking, temperature of cooking, and preheating prior to cooking. Peanut kernels with skins were cracked and flaked to approximately 0.010-in. thickness before cooking. Prior to extraction the cooked materials were cooled and then either screened, or rerolled, or both screened and rerolled.

Effects of Screening and Rerolling

The data in Table I show that filtration-extraction of materials screened only gives extracted meals of high residual lipids whereas lipids are reduced considerably when these same screened materials are rerolled through the flaking rolls. Other tests indicate that screening of cooked materials, followed by rerolling shows no improvement over rerolling. The subsequent investigations refer to cooked peanut meats that have been rerolled through the flaking rolls after cooking and screening.

TABLE I

Effects of Rerolling After Screening Cooked Peanuts

Description	Experiment A[a]		Experiment B[b]	
	Screened ¼″ mesh	Screened and rerolled	Screened ⅛″ mesh	Screened and rerolled
Cooker	French	French	Loomis	Loomis
Mass velocity, lbs./ft.²/hr.	6972	5420	3824	2829
Temperature of extraction, °F.	80	80	130	115
Desolventized meal analyses				
Lipids, %	4.83	1.9	1.57	0.48
H_2O, %	7.8	9.2	6.3	6.2

[a] Experiment No. 5 in Table II.
[b] Experiment No. 3 in Table III.

Effects of Moisture Addition in First Stage of Cooking Without Preheating of Peanuts

In Table II Experiments 1 through 5 show the effects of moisture addition without preheating in the first ring of the French cooker. For moisture levels ranging from 10.8% to 16.0% at temperatures from 190°F. to 220°F. crisped cooked materials were obtained which gave high mass velocities during filtration-extraction. With a higher moisture, as shown in Experiment 1, oil separated from the peanuts, and a low mass velocity was obtained during the filtration-extraction of these cooked flakes. In only one of these experiments (Experiment 4) were the residual lipids in the extracted meal less than 1%; in the others the residual lipids were approximately 2%.

Effect of Low Temperature Cooking Without Preheating of Peanuts

Experiment 6 of Table II shows that at the relatively low temperature of 182°F. a crisp peanut material was obtained which gave high mass velocities during filtration-extraction. Residual lipids in the extracted meal were 2.27%.

Preheating of Peanut Flakes Prior to Moisture Addition in Cooking

Data in Table III indicate that preheating of peanuts prior to cooking is a factor in the reduction of lipids during filtration-extraction to 1% or less. In Experiments 1 through 5 of Table III materials were preheated prior to moisture addition and cooking at temperatures from 190°F. to 220°F. Experiments 2 and 4 were conducted in the French cooker and Experiments 1, 3, and 5 in the Loomis mixer. Experiment 1 shows that peanut flakes can be cooked with 20% or more moisture without oil separation. This

[4] In using the names of equipment manufacturers, it should be understood that we are not recommending the products of one manufacturer over similar products of other manufacturers.

TABLE II

Filtration-Extraction of Peanuts, No Preheating

Experiment No.	1	2	3	4	5	6
Type peanuts	U. S. No. 2	U. S. No. 1	U. S. No. 1	U. S. No. 1, 2	U. S. No. 1	U. S. No. 1
Cooking						
Cooker	French	French	French	French	French	French
Batch wt., lbs.	50	50	48	50	50	50
H_2O, in feed, %	....	6.7	6.4	6.9	6.7	6.7
H_2O, 1st stage, %	17.5[a]	16.0	14.7	13.4	10.8	11.9
H_2O, at discharge, %	....	11.6	11.2	11.8	7.4	9.6
Time in cooker, min.	60	24	24	24	24	24
Temperature, 1st ring, °F.	205	208.5	217.5	203	210	182
Temperature, 2nd ring, °F.	240	200	198	202	190	193
Temperature, 3rd ring, °F.	270			1 & 2 only		
Screening						
Mesh, in.	¼	¼	¼	¼	¼	¼
Rerolling at 0.003 in.	Yes	Yes	Yes	Yes	Yes	Yes
Filtration-Extraction						
Feed analysis						
H_2O, %	6.1	10.4	9.5	8.5	6.7	8.3
Type extractor	Lab.	Lab.	Lab.	Lab.	Lab.	Lab.
Screen size	24 × 100	24 × 110	24 × 110	24 × 110	24 × 110	24 × 110
Solvent to meal ratio	1.5:1	1.5:1	1.5:1	1.5:1	1.5:1	1.5:1
Slurrying time	15	15	15	15	15	15
Total number washes	3	3	3	3	3	3
Mass velocity, lbs./ft.²/hr.	Too slow	6387	4384	1836	5420	7071
Temperature of extraction, °F.	80	80	80	80	80	80
Cake thickness, in.	2	2	2⅛	2	2	2
Vacuum, in. Hg.	4	4	<4	4	4	4
Desolventized meal analyses						
Lipids, %	....	2.1	2.23	0.52	1.9	2.27
H_2O, %	....	8.2	9.8	7.7	9.2	7.9
Protein solubility,[b] %	....	79.3	76.1	84.2	82.6	84.2

[a] Oil separated from meats.
[b] Protein solubility determined at 7.5 pH with NaOH.

is apparently due to preheating the flakes before the moisture addition and to the high rate of heat transfer resulting from better mixing and a larger area of heating surface per pound of material heated than those obtained in the French cooker. In Experiment 3 peanuts were cooked with a moisture content of 10.1%. The mass velocity obtained during filtration was 2,829 lbs./sq. ft./hr. and the residual lipids 0.48%. In Experiment 5, though the cooked peanuts were not rerolled and the residual lipids were high, the data show that a material suitable for filtration-extraction can be prepared with a cooking moisture as low as 9.3%. In Experiments 2 and 4 the peanuts were cooked with moisture contents of 12.5% and 9.9%, respectively. A maximum oil concentration of 40.5% in the miscella was reached in the slurrying operation of Experiment 4. Experiment 2 gave mass velocities (laboratory) of 4,790 lbs./sq. ft./hr. and residual lipids in the meal of 1.55% at an extraction temperature of 80°F. Extraction at a higher temperature should reduce the lipids approximately 0.5% with little change in mass velocity. With natural drainage (no vacuum), a mass velocity of 1,520 lbs./sq. ft./hr. was obtained with residual lipids of 0.84% at an extraction temperature of 130°F. For Experiment 4 filtration mass velocities (laboratory) at 80°F. were 1,100 lbs./sq. ft./hr. with residual lipids of 0.67% in the extracted meal.

Table IV shows wet screen analyses of raw and cooked peanuts for Experiments 2 and 4 in Table III. The cooked material prepared in Experiment 4 had more fines than the materials prepared in Experiment 2. These fines, in Experiment 4, were apparently tempered and crisped enough to permit filtration; however the amount of fines probably reduced the rate of filtration.

TABLE III

Filtration-Extraction of Peanuts, Preheating

Experiment No.	1	2		3	4	5	
Type peanuts	U. S. No. 1	U. S. No. 1		U. S. No. 1	U. S. No. 1	U. S. No. 1	
Cooking							
Cooker	Loomis	French		Loomis	French	Loomis	
Batch wt., lbs.	10	50		10	50	10	
H_2O, in feed, %	6.6	6.5		6.4	6.5	6.4	
H_2O, 1st stage, %	20.0	12.5		10.1	9.9	9.3	
H_2O, at discharge, %	18.9	10.1		4.8	6.2	5.1	
Time in cooker, min.	24	25		30	24	30	
Temperature, 1st stage,[a] °F.	212	205		208	218	208	
Temperature, 2nd stage,[b] °F.	191	194		200	192	197	
Screening							
Mesh, in.	¼	⅛		⅛	⅛	None	⅛
Rerolled at 0.003 in.	Yes	Yes		Yes	Yes	No	No
Filtration-Extraction							
Feed analysis							
H_2O, %	16.4	6.8	6.8	3.4	5.1	4.4	4.3
Type extractor	Lab.	Lab.	Lab.	Lab.	Crock	Lab.	Lab.
Screen size	24 × 110	24 × 110	24 × 110	60 × 60	24 × 110	60 × 60	60 × 60
Solvent to meal ratio	1.5:1	1.5:1	1.5:1	1.5:1	1.5:1	1.5:1	1.5:1
Slurrying time	15	15	15	30	15	30	30
Total number washes	3	3	3	3	3	3	3
Mass velocity, lbs./ft.²/hr.	2334	4790	1520	2829	1100	5836	5369
Temperature of extraction, °F.	130	80	130	115	80	140	140
Cake thickness, in.	1⅝	1¾	1¾	1⅞	2	1⅞	1⅞
Vacuum. in. Hg.	<4	4	0[c]	<4	4	4	4
Solvent in marc, %	43.8	39.5	49.1	....	41.2	....	....
Desolventized meal analyses							
Lipids, %	2.21	1.55	0.84	0.48	0.67	2.04	1.77
H_2O, %	8.6	7.8	4.5	6.2	8.9	7.4	7.6

[a] Cooking in 1st ring of French cooker or cooking in Loomis mixer.
[b] Drying in 2nd ring of French cooker or in Loomis mixer.
[c] Natural drainage.

TABLE IV
Wet Screen Analyses

Material	Table III				
	Experiment No. 2			Experiment No. 4	
	Uncooked	Cooked		Uncooked	Cooked, screened and rerolled
		Screened	Screened and rerolled		
Screen mesh	%	%	%	%	%
On 5	1.7	0.0	0.0	0.7	0.0
8	6.0	0.7	0.0	4.5	0.0
14	14.4	8.9	1.0	11.9	0.5
20	24.1	19.9	9.0	26.2	4.2
40	16.4	36.1	32.8	15.8	16.2
60	9.0	20.9	31.1	9.3	27.1
80	3.3	4.6	8.8	4.3	11.3
120	2.7	4.0	6.2	3.9	13.4
170	3.3	2.0	3.5	3.9	7.6
200	1.0	0.2	1.0	0.9	1.9
300	3.0	0.7	2.1	1.6	5.7
Through 300	15.1	2.0	4.5	16.6	12.1

Discussion

Preheating. The data indicate that preheating of the flaked peanuts before moisture addition is an important factor in preventing oil separation during cooking and in obtaining low residual lipids in extracted meals. In the Skipin process the oilseed meats are actually moistened before heating to promote oil flow (2). Preheating of the material to temperatures in excess of 170°F. prior to the addition of moisture has been shown to be effective in circumventing the Skipin range of conditions and preventing oil flow during cooking. This procedure appears to be highly important in the meat preparation, where filtration-extraction only is to be applied without recourse to prepressing, since in this process it is desirable to have the oil readily available to the solvent but is undesirable to have a sticky or oily meal mass which is easily compressible.

Rerolling Flakes After Cooking. Tables I, II, and III show that cooked peanut flakes must be rerolled to obtain low residual lipids in extracted meals. Only cooked flakes which had been rerolled gave lipids of less than 1% in the extracted meals. In actual practice screening would not be necessary before rerolling. Rerolling rates are high; in Experiment 3 of Table III a rate of 540 lbs./hr./ft. was obtained by using the Allis Chalmers smooth rolls.

Filtration-Extraction. Slurrying times of 15 min. are ample; however 30 min. give a greater margin of safety. A solvent to meal ratio of 1.5 to 1 is satisfactory; lowering the ratio to 1.2 to 1 leaves higher residual lipids. An increase in temperature from approximately 80° to 130°F. decreases lipids approximately 0.5%. The finer the material extracted, the greater is the solvent hold up in the marc. Where vacuum was applied, solvent in the marc varied from 34 to 44%. Miscella concentrations vary with the solvent to meal ratio and with the solvent hold up in the extracted meal. The higher the solvent meal ratio, the lower the oil miscella concentration; and the greater the hold up of solvent in the marc, the more concentrated is the oil miscella.

Meal. All solvent-extracted meals obtained from cooking the peanuts with 16.0% or less mositure at 217°F. or below had high protein solubilities ranging from 76.1 to 84.2%. In Experiment 1 of Table III, where 20% moisture was used in the cooking stage, the extracted meal had an unacceptable color and a low protein solubility of 57%. The off-color was probably due to the effects of the high moisture on the color pigments of the peanut skins.

Summary and Conclusion

Successful filtration-extraction of peanuts on a bench scale indicates that there should be little difficulty in conducting this process on a pilot plant or commercial scale.

Data indicate that the optimum conditions for preparing peanut flakes of approximately 0.010 in. thickness for filtration-extraction are as follows: preheating to 170°F. (approximately), moisture addition of 10 to 12.5%, cooking and drying at 190° to 220°F., crisping, and rerolling through rolls set at 0.003 in. and ending with a final moisture of about 7%. In the filtration-extraction of the peanut flakes a slurrying time of 30 min. and a solvent to meal ratio of 1.5 to 1.0 are adequate. Mass velocities of 2,800 to 4,800 lbs./sq. ft./hr. are obtained, and residual lipids in the extracted meal are approximately 1%. These mass velocities are suitable for commercial use. The extracted meals have a high protein solubility of about 80%.

Peanut flakes can be prepared for filtration-extraction by cooking at moistures ranging from 9.9 to 16.6% in the French cooker. At higher moistures with no preheating the oil will separate from the peanuts. Data using the Loomis mixer show that higher moisture can be used. Indications are that preheating and high rates of heat transfer prevent oil separation. Apparently preheating of peanut flakes before moisture addition is also a factor in lowering the lipids of extracted meals to or less than 1%. A low cooking temperature of 182°F. can be used to prepare a crisp material for filtration-extraction. Indications are that final moistures of cooked peanut flakes prior to extractions as they affect mass velocity are not critical. Large amounts of fines in the cooked peanut materials will reduce mass velocities during filtration-extraction.

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