The correlation coefficient between viability after 170 days and FFA after 171 days was only slightly higher than that between viability after six days and FFA at 171 days or 522 days. In this experiment germination tests made immediately after harvest would appear to provide a method for estimating the potential FFA of sesame seed at later dates.

Although correlation coefficients between FFA after 32 days and at later dates were high, it is unlikely that FFA values determined immediately after harvest would be of any predicative value since FFA increased throughout the storage period.

Present data do not provide sufficient evidence upon which to base an accurate prediction of potential FFA of oil from sesame seed under all conditions. A more reliable germination test should be developed since duplicate determinations sometimes showed significant variations in viability. More varieties should be studied to determine whether they all react alike. The effect of different conditions of seed production and storage should be further investigated. If later studies show that the relation between viability at harvest and FFA remains linear, a mathematical formula or a nomograph to estimate FFA at any given date from a knowledge of germination at harvest time may be developed.

Summary

Damage to sesame seed by mechanical threshing equipment resulted in loss of seed viability immediately. FFA content of oil in damaged seed increased gradually after harvest. High correlation coefficients were obtained between viability immediately following harvest and free fatty acid content of oil in sesame seed at various dates after harvest. The results of the analyses of the data indicate that viability of the seed at harvest may be of value in estimating the free fatty acid content of oil for various storage periods.

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The Difficultly Extractable Lipides of Cottonseed Meats, Their Composition and Effect on the Refining Characteristics of the Crude Oils[1]

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THE TREND IN RECENT YEARS to the use of solvent extraction for processing cottonseed has been pronounced. About 21% of the cottonseed produced in 1952–53 were processed by mills using the solvent extraction process in some form, as compared to only 11.5% in 1951–52 (6); and a number of mills have converted to solvent extraction since 1953.

Incentive for the more extensive use of the solvent-extraction processes has been that of obtaining greater yields of crude oil, as about 96–98% of the total lipides can be recovered by the solvent-extraction processes as compared to 83–93% by the mechanical expression methods.

Many mill operators, as well as personnel engaged in cottonseed processing and utilization research, have suggested that solvent extraction for maximum crude oil recovery has been over-emphasized at the expense of product quality and value. This viewpoint appears to be supported by the fact that solvent-extracted meals tend to be dusty and to have poor pelleting characteristics (8) and that many solvent-extraction plant operators have encountered oil-color problems.

Bull and Hopper (2) demonstrated that the lipides extracted from soybeans by successively more exhaustive extractions increased in impurities content with an increasing degree of total lipides extraction. Pons, Hoffpauir, and Thurber (12) have shown that the lipides extracted from cottonseed cake by the solvent-extraction step of the combination screw-press, solvent-extraction process is high in impurities and of lower quality than that obtained by the screw-pressing step. However no systematic study of the composition of the difficultly extractable lipides of cottonseed, or of their effect on the refining characteristics of the crude oils in which they are included, has been reported in the literature. It is the purpose of this paper to report the results of such a study.

In this work cottonseed meats from two different lots of seed were prepared for extraction by three different methods. The prepared meats were extracted with hexane by a series of successive stepwise extractions, and the fractional portions of the total meats-lipides so obtained were quantitatively isolated and analyzed. Crude oils corresponding to various percentages of total lipides extraction were then reconstituted from the crude lipides fractions and evaluated for refining characteristics.

Materials

The cottonseed meats used in this study were from prime delintered 1953 crop seed, hulled as required for use, and were essentially whole meats as nearly free from hulls as could be produced without hand-picking. Two lots of seed, one from the vicinity of Greenwood, Miss., and one from the El Paso, Tex., area were used. The whole meats from the two lots of seed contained, on a moisture-free basis, 35–36% and 39–40% of oil, respectively, and are designated

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hereafter as low and high oil-content meats. The solvent used was commercial hexane boiling at 68–70° C. and containing less than 0.0004% of non-volatile residue.

Equipment

The seed were hulled and the meats separated, cleaned, cracked, and flaked in mill-type, pilot-plant scale equipment. All cooking and tempering was done in a 22-in. diameter, five-high, stack-type cooker.

The extractions were conducted in an all-stainless-steel batch extractor (4) adapted for semi-Soxhlet operation.

The major portion of solvent was evaporated from the miscellas in a laboratory-size, rising film evaporator constructed of stainless steel and glass. Final desolventization of the lipides was done in glass laboratory equipment.

Experimental

Meats Preparation. Six extraction runs were made, one each with flaked raw meats, flaked tempered meats, and cooked flaked meats from the low oil-content seed, and one each with similarly prepared meats from the high oil-content seed.

Raw flakes were prepared for extraction by cracking and flaking the purified meats through a set of one-pair-high, Allis-Chalmers cracking and flaking rolls to produce flakes of 0.008 to 0.010 in. in thickness. The low oil-content seed were hulled and the meats flaked "as is." Excessive dryness of the high oil-content seed required that their moisture be increased before hulling to avoid shattering the meats and to obtain stable flakes.

The tempering procedure followed was that in general use (13), which requires adjusting the moisture of the cracked meats to *ca.* 13% in the top ring of the cooker, tempering the meats for about 23 min. by heating at temperatures below the boiling point of water, then flaking the meats while still warm. The tempering temperature range used with the low-oil meats was 110°F. to 170°F. and that for the high-oil meats was 105°F. to 200°F. Flake thickness was 0.008 to 0.010 in.

The cooking method employed was that used with the recently developed and commercialized filtration extraction process (3, 5). In this method of cooking the flaked meats are fed to the top ring of the five-high cooker and their moisture increased there to *ca.* 22–23%. The moistened flakes are then cooked through the remaining four rings over a temperature range of 195°F. to 235°F. with total residence time in the cooker of *ca.* 50 min. Residence time and temperatures in the successive rings are as follows: ring 1—7 min., maximum temperature 190°F.; ring 2—12 min., 220°F.; ring 3—12 min., 200°F.; ring 4—12 min., 215°F.; ring 5—7 min., 235°F. On discharge from the cooker the meats are screened to break up any lumps, then cooled and crisped by aspiration, followed by additional drying on open trays.

Extraction. The procedure employed for extraction was simple. The meats were covered with solvent and drained for each extraction pass. Fresh solvent was used for each pass, and the resulting miscella was collected individually in a separate container. Extraction was by diffusion only, with no agitation of the meats. A partial displacement wash was provided by solvent supernatant above the flake bed for each pass.

For each of the six extraction runs a weighed charge of 80 lbs. of prepared meats was put into the previously heated extraction-cell. The meats were then saturated and covered to a depth of 5–6 in. with warm solvent by spraying the solvent evenly over the meats. The meats were steeped in the solvent for a timed period, and the solution of lipides in solvent (miscella) was then drained away as rapidly and completely as the physical structure of the prepared meats permitted and was collected separately in tared glass containers. This procedure was repeated until the residual lipides in the meats had been reduced to about 0.50% as shown by analysis of the extracted meats.

The quantity of solvent required to saturate and cover each batch of prepared meats for the initial extraction passes varied with the bulk density and porosity of the prepared meats. About 10 gal. were required for cooked flaked meats and about 15 gal. for raw and tempered flakes. The quantity of solvent required for the succeeding extraction passes was the same for all types of prepared meats, 6.3 gal. per pass.

The temperature for all extractions was 120° ± 2° F., with the exception that the temperature dropped to about 80°F. during the over-night steeping periods employed in the later stages of each of the extraction runs. Solvent for all extraction passes was heated to 120° ± 2°F. before addition to the meats. The extractor cell was maintained at approximately the same temperature, with the exception noted above, by circulating hot water through the jacket.

On completion of an extraction run the miscellas from each of the individual extraction passes were weighed and the lipides content of each determined. The miscellas from the individual extraction passes were then combined in the order required to produce a series of miscellas containing successively extracted portions, or fractions, of the total lipides of the prepared meats.

The extraction data, the order in which the miscellas from the individual extraction passes were combined to contain the respective crude lipides fractions, the weight of the successive fractions, and other pertinent data are shown in Table I. The percentages of total lipides given in this table and subsequent tables were based on the weight of the crude lipides recovered as such plus the weight of the lipides remaining in the meals as determined by analysis.

The extracted meals were desolventized by air-drying without the use of heat and analyzed for moisture, oil, nitrogen and free and total gossypol. These analyses are shown in Table II.

The combined miscellas containing the desired crude lipides fractions were desolventized by evaporation followed by steam-stripping to obtain the solvent-free crude lipides fractions. Evaporation and stripping were conducted under reduced pressure at temperatures of not more than 170°F.

Samples of the crude lipides fractions were analyzed for moisture and volatiles, free fatty acids, neutral oil, gossypol, phosphorus, oxidized fatty acids, and unsaponifiable matter. In addition, the iodine value of the crude and neutral oils were determined. The analytical data for the crude lipides fractions are shown in Table III.

Reconstitution of Crude Oils. Crude oils equivalent to varying percentages of total lipides extraction

TABLE I

Extraction Data

Meats	Method of meats preparation	Crude lipides fractions	Extraction time per fraction	Extraction passes combined in fraction	Crude lipides in fractions	Total lipides extracted	Residual lipides in meal[a]
		No.	*Hours*	*Nos.*	*lbs.*	%	%
Low oil content	Flaking	1	0.5	1–3	25.25	93.17	3.77
		2	0.8	4–7	0.86	96.35	2.06
		3	1.1	8–12	0.28	97.38	1.39
		4	16.7	13–14	0.23	98.22	1.00
		5	65.6	15	0.28	99.26	0.41
	Tempering	1	1.2	1–6	23.72	92.84	3.84
		2	1.9	7–13	0.67	95.46	2.46
		3	15.5	14	0.27	96.52	1.89
		4	19.4	15	0.27	97.57	1.33
		5	41.3	16–17	0.34	98.90	0.60
	Cooking	1	0.9	1–6	23.73	94.20	3.01
		2	1.0	7–13	0.39	95.81	2.20
		3	15.4	14–15	0.78	99.05	0.52
High oil content	Flaking	1	2.6	1–5	28.78	94.39	3.92
		2	1.9	6–8	0.66	96.56	2.43
		3	3.5	9–13	0.26	97.41	1.84
		4	69.2	14–17	0.25	98.23	1.27
		5	143.5	18–21	0.25	99.05	0.68
	Tempering	1	3.9	1–7	27.41	93.90	4.04
		2	2.4	8–9	0.64	96.09	2.63
		3	2.6	10–11	0.36	97.33	1.81
		4	20.3	12–16	0.22	98.08	1.31
		5	132.5	17–22	0.28	99.04	0.66
	Cooking	1	1.0	1–3	26.15	93.09	4.00
		2	0.3	4	0.97	96.55	2.04
		3	2.0	5–9	0.56	98.54	0.86
		4	28.8	10–14	0.25	99.43	0.34

[a]Moisture-free basis.

were reconstituted from the crude lipides fractions obtained from each of the six batches of prepared meats. Reconstitution was effected by combining and thoroughly mixing weighed portions of the crude lipides fractions in the proper order and proportions. The analyses of the reconstituted crude oils, as calculated from their make-up of the respective successive crude lipides fractions, are shown in Table IV.

The reconstituted crude oils were evaluated for refining loss, refined oil color, and bleached oil color. These data are shown by Figures 1 and 2, respectively. In addition, the refining losses of the oils as determined by the chromatographic method are shown by Figure 1.

Methods of Analysis. Official methods of the American Oil Chemists' Society (1) were used to determine moisture and volatile matter, oil (lipides), nitrogen, and free gossypol of the meats and meals, and moisture and volatile matter, free fatty acids, oxidized fatty acids, unsaponifiable matter, and iodine number of the oils. The methods proposed by Pons, Hoffpauir, and O'Connor were employed for determination of total gossypol in the meats and meals (9) and in the oils (10). Phosphorus determinations in oils were by the method of Pons, Stansbury, and Hoffpauir (11). The crude lipides fractions were analyzed for neutral oil by use of a modification of the method of Linteris and Handschumacher (7).

Refining, bleaching, and color determinations employed the methods of the American Oil Chemists' Society (1). Official method Ca 9a-52 was followed in the refining loss tests, and bleaching was done in accordance with official method Cc 8a-52. Oil colors were determined photometrically, following official method Cc 13c-50.

Results and Discussion

The extraction data, Table I, show that the number of extraction passes and the time required to accomplish the desired degree of lipides extraction varied widely with the method of meats preparation employed, and to some degree between the similarly prepared meats from the two different lots of seed. Extraction times shown in the table are the sums of the times required to fill, steep, and drain. The prolonged extraction times for the raw and the tempered meats are largely attributable to the slow rate of extraction of these materials, particularly after extraction of the major portion of the lipides, but are also caused in part by difficulty encountered in draining. These materials exhibited a tendency to form large quantities of "fines," which tended to pack and prevent percolation of the solvent. The cooked meats offered no such difficulties but proved to be readily extractable and drained rapidly and easily because of their porous granular structure. The fact that only three and four crude lipides fractions were obtained from the respective batches of cooked meats was attributed to the comparatively rapid and complete extraction of their lipides by the first few extraction passes.

Each batch of material was prepared from meats freshly milled from the seed at the time of use, and consequently there was some variation in hull content. To permit direct comparison of the lipides content of the differently prepared meats and of the corresponding meals, correction was made for the differences in hull content by calculation to comparable nitrogen bases. As shown in Table II, the lipides found by batch-extraction of the variously prepared high oil-content meats exceeded those found by analysis by about 1% while the lipides found in the low oil-content meats material, with the exception of the cooked meats, agree fairly closely. This discrepancy was probably caused by sampling error as the cooking method results in virtually complete cell rupture and releases free oil, which tends to settle out and adhere to vessels and causes difficulty in obtaining reprseentative samples. Loss of oil by adherence to the walls and sides of preparation equipment may

TABLE II

Analyses of Prepared Meats and Extracted Meals [a]

Material	Meats	Lipides [b]		FFA of oil (as oleic)	Nitrogen	Gossypol	
		Batch extraction [c]	Analysis			Free	Total
		%	%	%	%	%	%
Raw flakes	Low oil content	35.56	35.68	0.50	6.51	1.12	1.14
Meal		.....	0.40	.....	10.07	0.96	1.53
Temp. flakes		36.57	36.67	1.13	6.23	0.72	1.02
Meal		.....	0.61	.....	9.55	0.82	1.14
Cooked flakes		35.05	33.99	0.79	6.28	0.03	0.99
Meal		.....	0.52	.....	9.31	0.06	1.35
Raw flakes	High oil content	41.72	40.73	0.77	5.56	1.17	1.14
Meal		.....	0.67	.....	9.09	1.22	1.67
Temp. flakes		41.05	39.86	1.72	5.50	0.90	1.18
Meal		.....	0.67	.....	8.92	0.87	1.37
Cooked flakes		40.49	38.41	1.07	5.46	0.06	1.08
Meal		.....	0.36	.....	9.02	0.07	1.55

[a] All analyses on a moisture-free basis.
[b] Lipides on a common nitrogen in materials basis. Basis: low oil content meats—6.34% nitrogen in meats, 9.643% in meals; high oil content meats—5.506% nitrogen in meats, 9.01% in meals.
[c] Lipides by batch extraction calculated from total amount of lipides recovered in miscella plus that remaining in meal, and moisture-free weight of meats extracted.

have contributed to a minor extent to the lower yields of oil from the cooked material noted in the succeeding paragraph.

The analyses of the prepared meats and their corresponding meals indicate that the method used in preparing cottonseed meats for extraction has a significant effect on the nature and quantity of the extractable materials. The more severe conditions of temperature, time, and moisture employed in cooking as compared to the relatively mild conditions used in tempering resulted in lower yields of extractable lipides from the cooked materials. The contrast between the two methods of preparation on the free gossypol of the meats was pronounced; tempering effected a reduction in the amount of free gossypol of only 25% to 35% as compared to about 95% for the cooking preparation. Both tempering and cooking resulted in increasing the free fatty acids of the oil in the meats, tempering by about twice as much as cooking.

Comparison of the analyses of the crude lipides fractions from the differently prepared meats, Table III, points to the reasons for the differences in the yields of crude lipides from the differently prepared materials noted in the preceding paragraph. The crude lipides fractions obtained from the raw and tempered meats were heavily loaded with non-neutral oil impurities and were correspondingly lower in neutral oil. The fractions from the cooked meats, on the other hand, were uniformly high in neutral oil and low in objectionable impurities. Apparently the effect of cooking cottonseed under the conditions used was to render a substantial portion of these impurities insoluble in hexane and oil so that they were not extracted but remained with the meal, resulting in lower crude lipides yields. The impurities most profoundly affected were gossypol and the phosphatides. There appears to be a consistent parallel relationship in the amounts of these components in the crude lipides fractions which suggests that they may be interactive with each other and/or with other meats components, such as protein.

For all methods of material preparation the percentages of most of the non-neutral oil impurities in the crude lipides fractions tended to increase with an increasing percentage of total lipides extraction, accompanied by a corresponding decrease in neutral oil content. The lipides fractions representing the final 4% or 5% of the total lipides extracted from both the raw and the tempered meats were exceptionally high in phosphatides, gossypol, and the material determined as oxidized fatty acids. However these constituents were more evenly distributed among the successive fractions from the tempered meats than among the fractions from the raw meats, tending to be concentrated in the final fractions from the raw meats. The amount of gossypol found in these fractions was surprisingly high, being over 6% and almost 12%, respectively, for the final fractions from the raw flakes of the low and high oil-content meats. The phosphatide contents of the respective final fractions from the two batches of raw flakes were also very high, 25.5% and 17.8%, respectively.

The fractions from the cooked meats were outstanding in their high content of neutral oil and their low content of non-neutral oil impurities. The final fraction from the low oil content meats contained almost 97% neutral oil, 1.59% oxidized fatty acids, only 0.58% phosphatides, and 0.03% gossypol. The corresponding fraction from the high oil content meats, representing a much smaller percentage of the total crude lipides, contained 93.9% neutral oil, 0.70% oxidized fatty acids, only 1.25% phosphatides, and 0.08% gossypol.

The free fatty acid values found for the fractions are not attributable solely to their actual content of free fatty acids but resulted in part from the large amounts of phosphatides and gossypol, both of which react with alkali on titration. The order of increase in free fatty acids determined in the fractions roughly parallels the order of increase in phosphatides and gossypol. The iodine values of the crude lipides and the neutral oils show a definite decrease with an increasing percentage of total oil extraction, indicating that the more saturated glycerides are the more difficultly extractable.

The composition of the crude oils reconstituted from the crude lipides fractions is given in Table IV. These data were calculated from the analyses of the crude lipides fractions and the amounts of each used in reconstitution. The data indicate that for all of the reconstituted oils the amount of impurities increased approximately as a direct function of the degree of total lipides extraction. However the amounts and rate of increase of the different components of the oils showed substantial variations, a fact which is not readily apparent from the analyses of the crude lipides fractions.

In general, the amounts of impurities in the oils from the raw flakes tended to increase rapidly from

TABLE III

Analyses of Crude Lipides Fractions

Material	Crude lipides fraction	Portion total lipides	Analyses [a]								
			F.F.A. (as oleic)	Neutral oil	Gossypol	Phosphorus	Phosphatides (P x 25)	Oxidized F.A.	Unsaponifiable matter	Iodine values	
										Crude oil	Neutral oil
	No.	%	%	%	%	%	%	%	%		
Low oil raw flakes	1	93.17	0.71	96.07	0.13	0.037	0.93	1.26	0.59	102.1	103.4
	2	3.17	2.43	89.36	1.37	0.250	6.25	2.57	1.12	100.3	100.4
	3	1.03	6.16	76.56	4.25	0.500	12.50	6.04	1.32	99.2	100.6
	4	0.85	4.35	73.63	3.50	0.620	15.50	5.94	1.19	98.3	101.7
	5	1.03	6.52	56.19	6.25	1.020	25.50	6.52	1.13	84.6	91.0
Low oil tempered flakes	1	92.84	1.58	96.41	0.25	0.075	1.88	0.56	0.67	104.1	104.6
	2	2.62	3.09	92.76	0.22	0.146	3.65	1.83	0.78	102.9	103.6
	3	1.06	2.50	93.88	0.32	0.190	4.75	1.08	0.92	101.6	103.1
	4	1.06	2.67	91.44	0.70	0.199	4.98	0.87	0.55	99.9	102.6
	5	1.33	8.28	81.27	1.77	0.418	10.45	3.68	1.06	91.5	92.9
Low oil cooked flakes	1	94.20	0.89	98.21	0.05	0.0009	0.02	0.33	1.07	103.3	104.1
	2	1.62	2.03	96.41	0.04	0.010	0.25	0.49	1.75	101.3	102.3
	3	3.23	1.77	96.66	0.03	0.023	0.58	0.29	1.59	102.3	103.3
High oil raw flakes	1	94.39	1.15	97.11	0.16	0.060	1.51	0.73	0.76	105.2	106.4
	2	2.16	3.19	84.10	1.41	0.420	10.52	2.77	1.72	101.8	104.4
	3	0.85	5.76	81.81	1.89	0.490	12.29	2.96	2.96	100.0	102.4
	4	0.82	6.05	83.10	1.15	0.440	11.03	2.12	2.06	99.8	102.1
	5	0.82	23.09	58.02	11.76	0.710	17.80	15.39	3.34	91.8	81.2
High oil tempered flakes	1	93.90	1.90	96.69	0.40	0.065	1.63	0.70	0.61	104.6	105.6
	2	2.19	2.14	95.79	0.34	0.100	2.50	0.68	0.47	103.1	104.6
	3	1.23	2.87	93.77	0.36	0.160	4.00	0.85	0.47	102.7	104.1
	4	0.75	5.15	90.53	0.72	0.200	5.00	1.45	1.20	100.1	101.8
	5	0.96	7.30	90.04	0.58	0.100	2.50	1.14	1.17	96.4	97.3
High oil cooked flakes	1	93.09	1.00	99.03	0.03	0.0024	0.06	1.55	0.73	104.7	105.2
	2	3.45	1.80	98.14	0.03	0.0088	0.22	0.39	0.94	104.9	104.8
	3	1.99	4.37	93.61	0.07	0.038	0.95	0.71	1.07	102.0	102.8
	4	0.89	3.57	93.88	0.08	0.050	1.25	0.70	1.88	102.4	103.1

[a] Moisture- and volatiles-free basis.

relatively low values in the oils by least exhaustive extraction, to high values in the oils by more exhaustive extraction. The gossypol content of the oils by most exhaustive extraction of raw flakes of both low and high oil-content meats, for instance, was approximately double that of the oils by least exhaustive extraction. Phosphatides distribution showed a similar trend, increasing by about one-third in the oil by most exhaustive extraction as compared to that by least exhaustive extraction. The oils from the tempered flakes prepared from both the low and the high oil-content meats showed only slight increases in impurities content between the oils by least and most exhaustive extraction; the percentages of impurities in all of the oils were uniformly of a high order. Gossypol in the oils by least and most exhaustive extraction of tempered flakes was virtually the same while phosphatide content increased only slightly with an increasing degree of extraction.

The oils extracted from the cooked materials contrasted sharply with the oils from both the raw and tempered flaked meats, being comparatively very low in impurities and correspondingly high in neutral oil. The percentages of gossypol in these oils were of a very low order, only 0.03% to 0.05%, as compared to 0.13% to 0.40% for the oils from the raw and tempered meats. Phosphatides in the oils from the cooked meats were also very low; the maximum for the most exhaustively extracted oils was only about 0.04% and 0.09% as compared to 1.6% to 2.1% in the corresponding oils from the raw and tempered meats.

The amount of unsaponifiable matter in all of the respective series of oils showed only slight increases with increasing degree of total oil extraction. The unsaponifiable-matter content of the oils did not correlate with the methods of meats preparation. The oils produced from the cooked, low oil-content meats were higher in this component than the oils from the raw and tempered meats while of the oils produced from the high oil-content meats that from the raw meats was highest.

A similar lack of correlation with the method of meats preparation exists with respect to the material determined as oxidized fatty acids. It would be expected that oxidation would be most evident in oils extracted from cooked meats resulting from the fact that the meats are necessarily exposed to air while hot. This expectation was found to hold for the oils extracted from the cooked high oil content meats; oxidized fatty acids material was approximately double that in the oils from the raw and tempered meats. However this situation was reversed with the oils from the materials prepared from low oil-content meats. The oil from the raw meats contained up to 1.45% of oxidized fatty acids, oil from the tempered material up to 0.68%, and oil from the cooked meats a maximum of only 0.33%.

The amount of material determined as free fatty acids in the reconstituted oils, while varying slightly between the successively extracted oils from the differently prepared materials, appears to have been more strongly affected by the method of meats preparation than by the degree of lipides extraction. The free fatty acid content of the oils extracted from the material prepared by tempering was substantially higher than in the oils from either the raw or the cooked meats while that in the oils from the raw meats exceeded that in the oils from the cooked meats.

The curves obtained by plotting the alkali and the chromatographic losses of the reconstituted crude oils against percentages of total lipides extraction (Figure 1), show that the refining losses by both methods tended to increase with an increasing degree of extraction. The losses by the alkali refining greatly exceeded those by the chromatographic method in magnitude. As increases in the free fatty acids content

TABLE IV

Analyses of Reconstituted Crude Oils Corresponding to Various Degrees of Total Lipides Extraction

Crude oil [b]	Total oil extraction	F.F.A (as oleic) [c]	Analyses [a]				
			Neutral oil	Phosphatides	Gossypol	Oxidized F.A.	Unsaponifiable matter
	%	%	%	%	%	%	%
LR1	93.17	0.75	96.07	0.93	0.130	1.26	0.59
LR2	96.35	0.82	95.85	1.11	0.171	1.30	0.61
LR3	97.38	0.84	95.65	1.23	0.214	1.35	0.62
LR4	98.22	0.85	95.47	1.35	0.243	1.39	0.62
LR5	99.26	0.90	95.05	1.60	0.305	1.45	0.63
LT1	92.84	1.37	96.41	1.88	0.250	0.56	0.67
LT2	95.46	1.30	96.31	1.93	0.249	0.59	0.67
LT3	96.52	1.31	96.28	1.96	0.250	0.60	0.68
LT4	97.57	1.44	96.23	1.99	0.255	0.60	0.67
LT5	98.90	1.49	96.02	2.11	0.275	0.64	0.68
LC1	94.20	0.89	98.21	0.023	0.050	0.33	1.07
LC2	95.81	0.83	98.18	0.024	0.050	0.33	1.08
LC3	99.05	0.74	98.13	0.042	0.049	0.33	1.10
HR1	94.39	1.03	97.11	1.51	0.160	0.73	0.76
HR2	96.56	1.13	96.82	1.71	0.188	0.71	0.78
HR3	97.41	1.13	96.69	1.80	0.203	0.80	0.80
HR4	98.23	1.15	96.57	1.88	0.211	0.81	0.81
HR5	99.05	1.31	96.25	2.01	0.307	0.93	0.83
HT1	93.90	1.70	96.69	1.63	0.400	0.70	0.61
HT2	96.09	1.70	96.67	1.65	0.399	0.70	0.61
HT3	97.33	1.71	96.63	1.68	0.398	0.70	0.61
HT4	98.08	1.76	96.58	1.70	0.401	0.71	0.61
HT5	99.04	1.78	96.52	1.71	0.402	0.71	0.62
HC1	93.09	1.00	99.03	0.060	0.030	1.55	0.73
HC2	96.55	1.01	99.00	0.066	0.030	1.51	0.74
HC3	98.54	1.08	98.89	0.084	0.031	1.49	0.74
HC4	99.43	1.07	98.84	0.094	0.031	1.49	0.75

[a] Analyses of the reconstituted oils calculated from the analyses and amounts of the crude lipides fractions making up the oils, with the exception of free fatty acids.

[b] Reconstituted oil identification. First letter indicates type of meats—low (L) or high (H) oil content; second letter meats preparation—raw flaking (R), tempering (T), or cooking (C); digit indicates number of crude lipides fractions making up oil.

[c] Determined by titration of reconstituted oil with 0.1 N. NaOH.

of the oils with increasing degree of extraction were relatively slight (Table IV), the disproportionate increases in refining losses with an increasing degree of extraction must be attributable to some other component. There appears to be a relationship between the phosphatide and gossypol contents of the oils and their refining losses which suggests that the major portion of the refining losses may be attributed to the effects of one or both of these components, probably to the phosphatides.

The refining loss of the oils extracted from the raw flakes, prepared from both the low and high oil-content meats, exhibited rapid increases with an increasing degree of total oil-extraction. The difference in the magnitude of the losses of the oils from the high oil-content meats as compared to those from the low oil-content meats no doubt results from the combination of relatively high free fatty acid and relatively high phosphatide content of oil from the high oil-content meats.

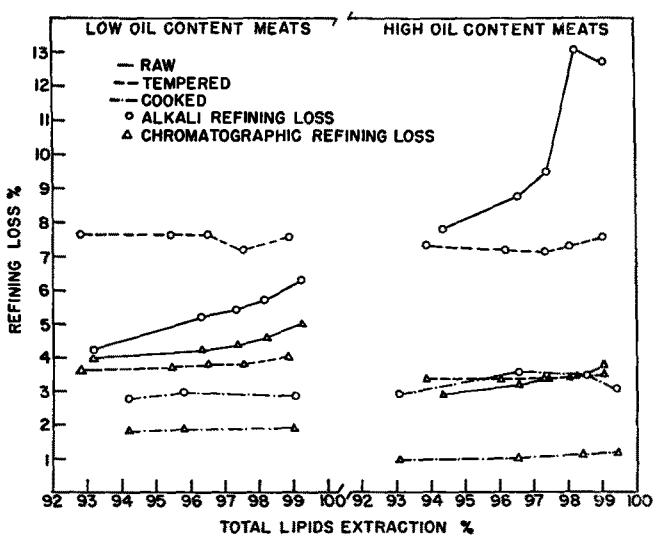

FIG. 1. Relationship of refining loss to degree of lipides extraction and method of meats preparation.

The oils from both batches of tempered meats gave refining losses of approximately the same order of magnitude at all degrees of total oil extraction, tending to show some slight decrease in loss with an increasing degree of extraction, a trend which may have been caused by experimental error. Examination of the compositional data of these oils shows that there was no very great difference in the composition of the oils by most and least exhaustive extraction, a condition which would be expected to result in relatively uniform refining losses for these oils.

The refining losses of the oils from the meats prepared for extraction by cooking were outstandingly low. The low losses of these oils are probably largely attributable to their low content of phosphatide materials, with the consequent absence of neutral oil entrainment due to the emulsifying effect of phospholipides.

The curves obtained by plotting the red color of the refined and the bleached oils against the percentages of total lipides extraction to which the oils correspond (Figure 2) show that the red color of all of the refined oils, with the exception of that from the cooked high oil-content meats, tended to increase with an increasing degree of total lipides extraction. It is noteworthy that no consistent correlation between the red color of the refined oils and the gossypol content of the crude oils can be discerned in the present data. It can only be concluded that other factors in addition to gossypol as such, are contributory to the red color of the refined oils.

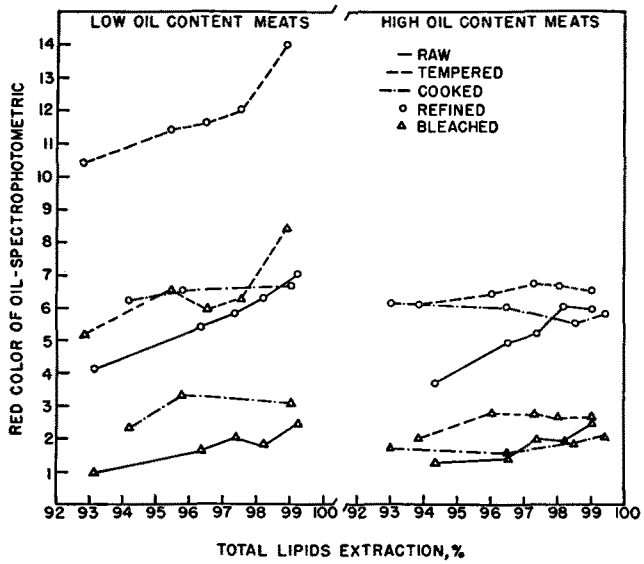

FIG. 2. Relationship of refined and bleached oil color to degree of extraction and method of meats preparation.

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.

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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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