Comparative Absolute Yields of Crude and Neutral Oils from Variously Prepared Cottonseed Meats[1]

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THE NEED for these studies became evident as a result of reports from numerous mill operators to the effect that they were encountering difficulties in obtaining oil balances for their operations (2). In addition, it had been observed in connection with some of the studies at this laboratory (4) that the yields of crude oil obtained from differently prepared meats by extraction with hexane varied appreciably with the method used in preparing the meats for extraction.

Failure to obtain oil balances in mill operation could conceivably result from a variety of causes, such as sampling errors, inaccurate weighing of materials into and out of process, flaws in analytical techniques and methods, and failure to take in consideration such possible sources of oil loss as meats lost in motes, flue bran, linters, and hulls. In addition, as previously noted, the method used in preparing the meats for extraction could be a major contributing factor.

The first four possible causes of oil balance failure can only be investigated at the mill during operation. However the fifth possible source of oil loss, the effect of the method of meats preparation, is well suited to laboratory investigation, and it was this factor which was evaluated in the present study.

The procedures employed in this study were designed to eliminate or reduce to a minimum any errors caused by sampling or loss of materials in process. Every precaution was taken to insure that the samples analyzed were representative of the materials from which they were taken. The data represent the effects of the method of meats preparation on the yields of crude and neutral oil within the limitations of experimental error and the methods of analysis employed.

Experimental

Solvent. Commercial hexane containing no nonvolatile residue and boiling at 68–70°C. was used as the oil-extraction solvent. All extractions were with solvent from the same drum.

Cottonseed and Meats. Meats from three lots of seed were used in this study. The meats used in Experiment 1 were from a prime 1953 high oil content seed, those for Experiment 2 were from a prime 1953 medium oil content seed, and those for Experiments 3 and 4 were from a prime 1954 seed of lower oil content. Experiments 1, 2, and 3 utilized essentially hull-free whole meats. The meats used in Experiment 4 contained an amount of hulls approximately equal to that of mill-run meats.

Meats Preparation. Three different methods of meats preparation were employed for each "experiment" in this study. The first, simple flaking of the meats "as is," was used to serve as a control since extraction of raw meats is not practiced commercially. The second, known as "tempering" (12), consists of heating cracked meats in the presence of added moisture at temperatures below the boiling point of water prior to flaking and is a well known and widely used commercial process. The third, embodying thorough cooking of the meats, employed the conditions of temperature, cooking time, and moisture addition, retention, and removal developed for use with the filtration-extraction (3, 6) process. This method, while similar to the cooking procedure used in conjuction with hydraulic pressing, embodies important modifications such as lower temperatures, shorter cooking time, higher moistures, and evaporative cooling and crisping, which yield material differing significantly from hydraulic-cooked meats in physical characteristics.

Equipment. The seed were hulled, and the meats were separated and cleaned in mill-type, pilot-plant scale equipment, and the meats were cracked and flaked, respectively, in corrugated single-pass dual rolls and in smooth single-pass, dual rolls.

Cooking and tempering operations were conducted in a specially designed, batch-type, all-stainless-steel bench-scale cooker which has been described in a previous publication (5). This cooker, which had a capacity of 1,000 g. of flaked meats, permitted satisfactory replication of mill-type cooking and tempering operations. A feature of the cooker, a removable, stainless-steel liner, enabled quantitative handling and recovery of materials and avoided the small losses occasioned by transfers from one container to another.

The extractions were conducted in modified Soxhlet type 2.5-liter-capacity glass extractors, fitted with glass wool filter pads to minimize carry-over of fine meal particles with the miscella.

Procedure. A 2,500-g. portion of the meats used for each of the series of the experiments was thoroughly mixed, heaped into an even conical pile, and then vertically divided into six approximately equal portions. The oppositely positioned sixths were then combined and mixed to give three equal portions of meats of virtually identical composition which were designated as A, B, and C. Portions A and B were combined and mixed for use in the raw flaking and cooking preparations, respectively. Portion C of the meats, for use in the tempering preparation, was sealed in a metal container and stored at approximately 40°F. for a short time prior to use.

Combined portions A and B were flaked through clean flaking rolls to produce flakes of 0.008-in. thickness. These flakes were thoroughly mixed, and one 500-g. portion was weighed into the extractor and covered with solvent. A second 500-g. portion was weighed directly into the tared, removable, stainless-steel liner cup of the cooker, and the cooking operation was begun immediately.

Representative samples of the flakes were weighed out for determination of moisture and oil when the flakes were weighed into the extractor and into the cooker. An additional sample was taken at the same

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TABLE I
Analyses of Flaked, Raw, Whole Meats Used as Starting Material[a]

Experiment	Oil	F.F.A. of oil	Free gossypol	Total gossypol	Phosphorus		Nitrogen
					Total	Inorganic	
	%	%	%	%	%	%	%
1	39.92	1.38	0.95	1.12	1.01	0.05	5.47
2	37.12	1.10	0.91	1.06	1.06	0.03	6.10
3	34.83	0.71	0.72	0.93	1.03	0.02	6.51

[a] Moisture- and volatiles-free basis.

time to be analyzed for free fatty acids of the oil, free and total gossypol, total and inorganic phosphorus, and nitrogen.

When cooking was completed, the liner cup containing the cooked flakes was weighed back to determine the moisture gain of the meats. The cooked meats were then transferred quantitatively to an extractor and covered with solvent. Any oil and meats adhering to the cooker cup and agitator blades were rinsed into the extractor with solvent.

Portion C of the meats was cracked through clean cracking rolls and thoroughly mixed after cracking; 750 g. of the cracked meats were weighed into the liner cup of the cooker and tempered. The warm, tempered meats were then flaked through clean rolls. The flaked, tempered meats were thoroughly mixed, and 500 g. of the flakes were weighed into an extractor and covered with solvent.

Samples of the tempered flakes were weighed out for moisture determination at the time the flakes were weighed into the extractor.

The flaked, raw meats, the cooked meats, and the flaked, tempered meats were exhaustively extracted, with not less than 30 solvent-extraction passes for each batch of prepared meats.

The miscellas were collected in tared, 3-liter, single-neck, round-bottom flasks and were desolventized in the same flasks. Desolventization was done by distilling off the major portion of the solvent under vacuum, followed by stripping with nitrogen under high vacuum to remove the last traces of solvent. (This procedure permitted direct weighing of the extracted crude oils without loss of oil by adherence to containers.) The moisture and volatiles and meal content (some of the oils contained traces of meal despite the glass-wool filter pad in the extractors) were determined, and the crude oil weights were corrected accordingly.

The meals were quantitatively removed from the extractors, spread on tared sheets of aluminum foil, permitted to air-equilibrate, and weighed. Their moisture, volatiles, and residual oil contents were determined.

Methods of Analysis. Analyses of the materials for moisture and volatiles, oil, free fatty acids, free gossypol, nitrogen, oxidized fatty acids, and unsaponifiable matter were done as prescribed by the Official and Tentative Methods of the American Oil Chemists' Society (1). Total gossypol in cottonseed meats and gossypol in crude oils were determined by the methods of Pons, Hoffpauir, and O'Connor (8, 9). Neutral oil determinations utilized a modification of the chromatographic method of Linteris and Handschumacher (7). The methods of Pons, Stansbury, and Hoffpauir were followed in determining phosphorus (10).

Results

The analyses of the flaked, raw, whole meats for oil, free fatty acids of their oils, free and total gossypol, total and inorganic phosphorus, and nitrogen are given in Table I.

Table II shows the results of the analyses of the crude oils for neutral oil, free fatty acids, oxidized fatty acids, gossypol, phosphorus, and unsaponifiable matter. Phosphatides content was approximated by multiplying phosphorus by the factor 25. The factor 25 was obtained by calculations based on the assumption that all of the phosphorus in hexane-extracted cottonseed oils occurs as lecithin and cephalin and that the ratio of lecithin to cephalin is about 2.5 to 1 (11). The approximate correctness of the value so arrived at for phosphatides in these oils is attested by the fact that the sum of the constituents determined in the oils (omitting unsaponifiables which are assumed to remain with the neutral oil) approach 100% very closely.

The material balance data for each of the four experiments are given in Table III. These data are shown on a moisture-free basis throughout, with the yields of meal solids, residual oil in meals, and crude oils expressed as percentages of the moisture-free weight of the original meats used in the experiment. The results show recoveries of the original meats charge, ranging from 99.54% to 100.15% and averaging 99.86%. These results are within the accuracy of most analytical methods.

The yields of crude and neutral oil obtained are given in Table IV. In order to show the ultimate, or absolute yields of oil, the percentages of crude oil include the residual oil remaining in the meals. Yields of crude and neutral oils were calculated on the basis of the moisture-free weight of the meats

TABLE II
Analyses of Crude Oils[a]

Experiment	Method of meats preparation	Neutral oil	Free fatty acids	Oxidized fatty acids	Gossypol	Phosphorus	Phosphatides (P×25)	Unsaponifiable matter
		%	%	%	%	%	%	%
1	Raw	94.7	1.70	1.48	0.388	0.063	1.58	0.70
	Tempered	95.9	1.58	0.78	0.135	0.062	1.55	0.68
	Cooked	97.9	1.64	0.29	0.046	0.017	0.43	0.67
2	Raw	95.3	1.50	1.12	0.348	0.078	1.95	0.71
	Tempered	95.1	1.30	1.02	0.235	0.080	2.00	0.72
	Cooked	97.9	1.05	0.34	0.057	0.026	0.65	0.76
3	Raw	95.9	0.83	0.67	0.400	0.082	2.05	0.62
	Tempered	96.2	0.78	0.58	0.225	0.092	2.30	0.74
	Cooked	98.4	0.75	0.35	0.030	0.030	0.75	0.66
4	Raw	95.6	1.21	1.16	0.380	0.073	1.83	0.68
	Tempered	96.1	1.05	0.86	0.188	0.087	2.18	0.66
	Cooked	97.9	0.94	0.59	0.037	0.041	1.03	0.66

[a] Moisture- and volatiles-free basis.

TABLE III

Material Balance [a]

Experiment	Method of meats preparation	Meats into process	Recovery of prepared meats			
			As meal		As crude oil	Total recovery
			Solids	Residual oil		
		gm.	%	%	%	%
1	Raw	465.2	58.92	0.10	40.94	99.96
	Tempered	450.3	58.89	0.29	40.62	99.80
	Cooked	467.9	60.25	0.14	39.58	99.97
2	Raw	464.9	62.03	0.13	37.51	99.67
	Tempered	450.2	62.06	0.21	37.27	99.54
	Cooked	464.9	62.85	0.14	36.74	99.73
3	Raw	464.0	64.14	0.15	35.56	99.85
	Tempered	452.4	64.15	0.18	35.46	99.79
	Cooked	464.0	64.98	0.08	34.87	99.93
4	Raw	462.0	71.34	0.11	28.61	100.07
	Tempered	447.3	71.27	0.24	28.39	99.90
	Cooked	462.0	72.12	0.15	27.88	100.15

[a] Moisture- and volatiles-free basis.

charge. In addition, the yields of crude and neutral oil from the tempered and the cooked meats are given on the basis of the oil yields from the raw meats as 100%.

TABLE IV

Comparative Yields of Crude and Neutral Oil

Experiment	Method of meats preparation	Crude Oil [a]		Neutral Oil [a]	
		Prepared meats basis	Crude oil from raw flakes basis	Prepared meats basis	Neutral oil from raw flakes basis
		%	%	%	%
1	Raw	41.04	100.00	38.86	100.00
	Tempered	40.91	99.68	39.22	100.93
	Cooked	39.73	96.81	38.90	100.10
2	Raw	37.64	100.00	35.88	100.00
	Tempered	37.47	99.55	35.63	99.30
	Cooked	36.87	97.95	36.09	100.58
3	Raw	35.71	100.00	34.25	100.00
	Tempered	35.63	99.78	34.28	100.09
	Cooked	34.96	97.90	34.40	100.44
4	Raw	28.72	100.00	27.47	100.00
	Tempered	28.64	99.72	27.52	100.18
	Cooked	28.03	97.60	27.44	99.89

[a] Yields based on crude oil recovered as such, plus that remaining in the meals.

Discussion

The data show clearly that there are significant differences in the composition and in the yields of crude oil which can be obtained from differently prepared cottonseed meats by extraction with commercial hexane. These differences are attributable solely to the methods employed in preparing the meats for extraction. The greatest yields of crude oil were obtained from meats prepared by simple flaking of the raw meats "as is." Slightly lower yields were obtained from meats which had been cracked and tempered prior to flaking. The lowest yields were obtained from thoroughly cooked meats.

The yields of crude oils from the differently prepared meats however do not give a clear picture of the relative merits, with respect to oil yields, of the three methods of meats preparation. Neutral oil is the valuable constituent of crude oils. Any constituent of the crudes, other than neutral oil, must be removed by refining, and the value of a crude oil is determined by its refining loss and the color of the refined oil produced from it. The data show that the method of preparing cottonseed meats for extraction does not significantly affect the yields of neutral oil obtained by extraction with commercial hexane. The small differences in the neutral oil yields from the differently prepared materials which do appear are within the limits of accuracy of the methods used in determining neutral oil.

It is important to note that the yields obtained from meats prepared by all of the three methods by extraction with commercial hexane exceeded the amounts of oil found by analysis of the raw flakes used as starting material. (Table I, col. 2), also that the percentages of free fatty acids found in the hexane-extracted oils exceeded the percentages found in the oil of the raw flakes by analysis (Table I, col. 3). These differences are attributable to the fact that the commercial pentane used in analyzing for oil content and in extracting the oil of the raw flakes on which free fatty acids were determined, has less solvent power for the non-oil materials of cottonseed meats, such as gossypol, phospholipides, and the materials determined as oxidized fatty acids, than does commercial hexane. Actually the analytical determination of oil in raw cottonseed meats closely approximates the sum of the neutral oil and free fatty acids of the oil of the meats, as is shown by the data in Tables I and IV.

The causes of the differences in the yields of crude oil obtained from the differently prepared meats are clear when the data on the analytical composition of the crude oils from the differently prepared meats are examined. The crudes from the raw flakes were consistently higher in gossypol and oxidized fatty acids than were the oils obtained from the corresponding tempered and cooked meats. The oils from the tempered meats tended to be significantly lower in these constituents than those from the raw flakes. The phosphatides content of the oils from the raw and tempered flakes were of essentially the same order, with that of the tempered flakes tending to be slightly higher. The crude oils obtained from the cooked flakes contained substantially lower amounts of these non-neutral oil components than did the crude oils from the raw and the tempered meats.

The unsaponifiable matter contents of the crudes from the differently prepared meats were essentially the same. There is a tendency towards lower free fatty acids content for the crudes from the cooked meats as compared to those from the tempered meats, and for the crudes from the tempered meats as compared to those from the raw flakes. This tendency however may be discounted in view of the fact that the free fatty acid values of the various crudes reflect their content of acidic components, such as gossypol and phosphatides. These components react as acids on titration and tend to give values which are higher than the true free fatty acid content of the oils. On the basis of the compositional data for the crude oils it appears that the method of meats preparation has little, if any, effect on the actual free fatty acids content of the oils.

Summary

Experiments utilizing cottonseed meats of diverse origin and composition were conducted for the purpose of determining the effect of the method of meats preparation on the yields of crude and neutral oil obtainable from differently prepared, comparable meats by solvent extraction. Three methods of meats preparation were employed, *i. e.*, simple flaking of raw meats "as is," tempering of cracked meats

prior to flaking, and cooking by the modified hydraulic method developed for use with the filtration-extraction process. Commercial hexane was used as the extraction solvent. The experiments were carried out by procedures which eliminated the effects of any variables other than the method of preparing the meats for extraction.

The results of the studies showed that the method used in preparing cottonseed meats for extraction had a significant effect on the yields of crude oil obtained but that the yields of neutral oil, the valuable constituent of crude oils, were virtually unaffected. Analyses of the crude oils showed that the differences in crude oil yields were caused by the relative amounts of non-neutral oil materials in the crudes from the differently prepared meats. The greatest yields of crude oil were obtained from raw flakes, intermediate yields from tempered flakes, and the smallest yields from cooked flakes. The impurities content in the respective crude oils followed the same order, *i.e.*, crudes from raw flakes were highest in impurities and lowest in neutral oil, crudes from tempered flakes were lower in impurities and higher in neutral oil, and the crudes from the cooked meats were outstandingly low in impurities and high in neutral oil. Virtually equal amounts of neutral oil were obtained from equivalent quantities of comparable meats regardless of the method used in preparing the meats for extraction.

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Procedure and Apparatus for Plasticizing Fats in the Laboratory[1]

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THE POUND-CAKE VOLUME BAKING TEST is an important initial guide for evaluation of a new shortening. Variations of this test also are valuable production control tools in the manufacture of shortenings.

The eight ounces of shortening used in this bake test must be plasticized and tempered unless some other consistent means of obtaining a satisfactory physical state is known. A fast, dependable procedure for plasticizing such a small amount of shortening has been a problem in the research laboratory.

In the plant the shortening is chilled rapidly with sufficient agitation to accumulate 10–15 cc. of air per 100 g. of fat. The air content is controlled, principally to improve the appearance of the shortening. The plasticized shortening is then tempered at least 48 hrs. at 80–90°F. (room temperature).

We have found a procedure whereby shortenings can be so plasticized in the laboratory as to give pound-cake volume values closely approximating those given by the same fats plasticized in plant equipment. The products of our process contain more air than those produced in plant equipment, and consequently they are not comparable in texture or appearance. Our process must be considered strictly as a method of preparing the shortening for the pound-cake volume test.

[1] Presented at the fall meeting, American Oil Chemists' Society, Philadelphia, Pa., Oct. 10–12, 1955.

Apparatus

The process is carried out in a Hobart Kitchen Aid Mixer (Model K-4-B), which is of the planetary type, and the bowl is cooled in a water bath in which the entire assembly is set as shown in Figure 1.

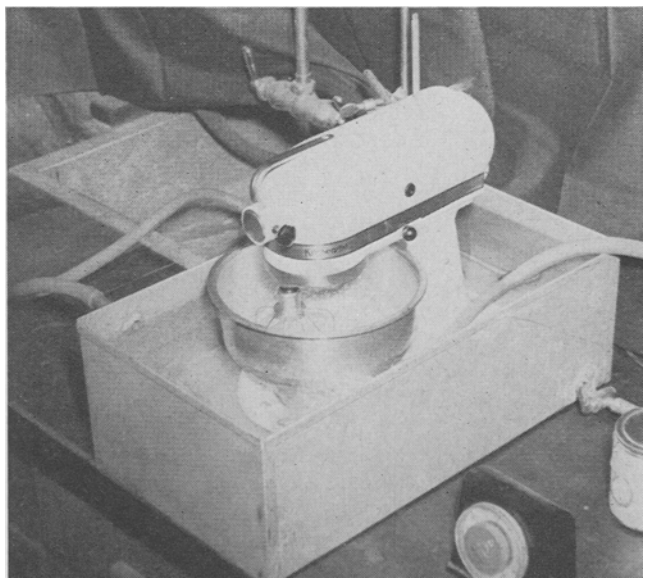

FIG. 1. Apparatus used for plasticizing fats.