ples of cooked cottonseed meats (1.8 to 13.9% water). Statistical treatment of the data yields the following results: (1) equation of the regression line,

$$\%\ \text{water} = (82.31)(\text{millimhos}) - 22.53;$$

(2) correlation coefficient, 0.998; (3) standard deviation from the regression line, 0.27% water.

Ten replicate determinations made on each of two samples containing 2.7 and 7.7% water showed standard deviations of 0.07 and 0.08% water.

These results show that the accuracy and precision of the modified indirect conductivity method are satisfactory for practical applications. For a consecutive series of determinations the average time required per determination was about 9 min. This time could be reduced to about 5 min. by using duplicate sets of stirring apparatus.

Acknowledgment

The authors are grateful for the cooperation and assistance of A. C. Wamble of the Cottonseed Products Research Laboratory of the Texas Engineering Experiment Station. Also the technical assistance of J. W. Jernigan is acknowledged. Except as noted, the experimental work was performed in the Laboratories of the Department of Chemistry, A. & M. College of Texas.

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Solvent Cooking of Cottonseed Meats for Extraction[1]

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Since 1946 the cottonseed processing industry has undergone considerable technological change. The conventional hydraulic process has been replaced in many oil mills by screw pressing (3, 8), direct solvent extraction (9), or a combination of the two generally referred to as prepress solvent extraction (9, 12). One of the recently commercially developed processes, which solved many of the problems encountered in applying direct solvent extraction to cottonseed, is filtration-extraction (1, 2, 5). An important feature of this process is the cooking and crisping of the raw, rolled meats prior to extraction which results in the granulation of the meat particles and reduction of gossypol (0.03–0.05% in the final meal) while maintaining a reasonably high protein solubility. The cooking-crisping operation is conducted in the same type of conventional equipment as is used for preparing cottonseed for hydraulic pressing. This method of preparation also makes possible the rapid solution of the oil while slurrying the rolled, cooked, crisped meats in the extractor (2) and the efficient removal of the resulting miscella on a horizontal rotary vacuum filter (2).

"Solvent cooking," as described in this paper, combines the cooking, crisping, and the slurrying steps in a single operation. The raw, rolled cottonseed meats are mixed in the solvent, together with sufficient moisture (containing added chemicals), and then the resulting slurry is subjected to a combined heating and azeotropic distillation, with further mixing, to reduce the moisture content to an optimum value.

Data are presented to show the influence of various conditions and chemicals in the "solvent cooking" operation on the granulation and extraction of the oil in the prepared meats and the reduction of free gossypol and maintenance of high alkali protein solubility in the final meals.

Experimental

Material and Equipment. One prime lot of cottonseed was used to obtain the data reported in this paper. The solvents used were commercial grades of hexane and heptane.

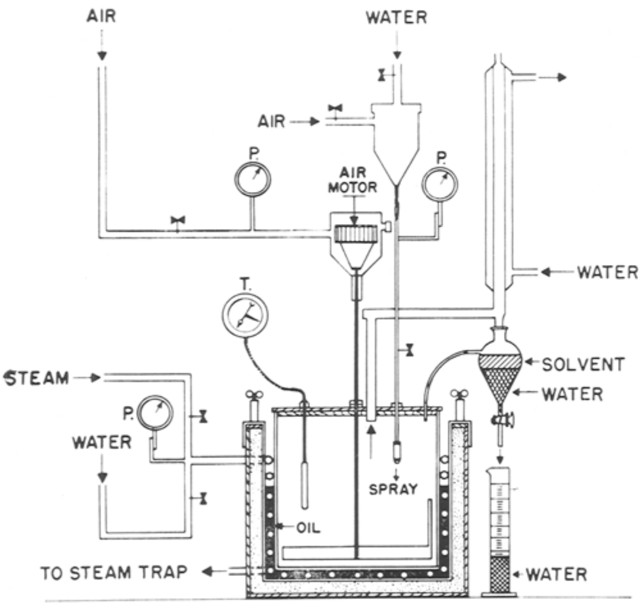

Fig. 1. Solvent cooker.

Figure 1 shows a drawing of the apparatus in which the solvent-cooking of cottonseed flakes was conducted. A coil immersed in an oil bath and surrounding the outside and bottom of the cooking container is connected to both steam and water lines for heating and

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cooling the container and its contents. A condenser and separator were installed as shown to condense the azeotropically distilled vapors and to remove water and to return the condensed solvent to the system. The equipment used for the pilot plant preparation of flakes has been described previously (13). Bench-scale filtration-extraction equipment, also previously described (6) and shown in Figure 2, was used in part of the evaluation of the extraction and filtration characteristics of the solvent-cooked material.

Procedure. The following procedure was used for solvent-cooking and chemically treating cottonseed flakes, which were prepared by rolling the raw meats to 0.008–0.010 in. in 5-high rolls. The required amount of solvent (usually 0.8:1 ratio of solvent to flakes) was added to the cooking unit and heated to the boiling point before adding the flakes. The calculated amount of water necessary to increase the moisture content of the flakes to the desired level (20 to 40%) was sprayed onto the slurry. Chemicals were added with the water. When more than one chemical was used, each was dissolved in a portion of the water before spraying into the cooker. The portion with the alkali or ammonia was always added first, followed by the second portion about 10 min. later. The slurry was agitated sufficiently to attain thorough mixing during the addition of flakes and water with chemicals, and during the solvent-cooking operation. Cooking temperature was maintained at the boiling point of the solution. Cooking and azeotropic distillation were continued until the amount of water removed was that calculated to decrease the moisture in the meal to 10%.

The resultant slurry was subjected to a wet screen analysis (4) and to a filtration-extraction test (6) to obtain mass velocity data and extraction characteristics. Mass velocity is the pounds of filtrate per square foot per hour that passes through the filter medium. It has been established previously (6) that a material with a mass velocity of 2,000 or over and a residual lipides content of 1% or less would be considered suitable for practical operations. In some tests, as indicated, a sample of the cooked material was also subjected to a modified filtration-extraction test (pre-extraction), which consisted of a reslurry step after the first wash. All extracted meals were analyzed for residual lipides, free (10) and total (11) gossypol, alkali protein solubility (7), nitrogen, moisture content, and dry screen analyses.

Results and Discussion

Evaluation of several factors during the initial study of the application of solvent-cooking to the processing of cottonseed served as a basis for outlining the experiments reported in the tables. This early work showed that the addition of less than 1% each of NaOH and $CaCl_2$ resulted in decreasing the free gossypol in the meal to 0.14% as compared to 0.36% for a test run in which no chemicals were used. The use of $CaCl_2$ in combination with NaOH showed an improvement in both the agglomeration of meal particles and in the mass velocity as compared to tests in which no chemicals or only NaOH was used. The increasing of the initial moisture content of the flakes from 20 to 25% also improved agglomeration and further decreased the free gossypol content to 0.105%.

Based on the above initial results a series of seven experiments were planned and conducted. The solvent-cooking conditions for this series are shown in Table I, and the results are given in Tables II, III, and IV.

TABLE I
Solvent Cooking Data

Experiment No.	1	2	3	4	5	6	7
Added to cooker:							
Flakes, lbs	6.0	6.0	6.0	6.0	6.0	6.0	6.0[a]
Hexane, lbs	4.8	4.8	4.8	4.8	4.8	....	4.8
Heptane, lbs	....	....	....	....	....	4.8	....
Moisture, incr. to, %	25	25	25	30	30	30	22[a]
NaOH, gm	25	25	....	25	....	25	25[a]
$CaCl_2$, gm	20	....	....	20	....	20	....
NH_3, gm	....	....	25	....	25	....	....
NaCl, gm	....	20	20	....	20	....	....
Cooking time, min	85	93	85	89	96	62	66
Cooking temp., °F	146	146	146	146	146	181	146

[a] NaOH was added to the meats with part of the water before flaking, and the flakes were heat-treated, with moisture content increased to 22%, for 15 min. at 200°F.

Only for Experiment 7 were the prepared flakes partially pre-cooked for 15 minutes at 180–200°F. in a 5-high stack cooker. The only chemical used was 1% NaOH. Part of it was added to the meats before rolling, and the remainder in the water was used to raise the moisture content of the rolled meats to 22% during the pre-cooking operation. These prepared meats were solvent-cooked without further addition of chemicals or water.

The effects of using NaCl instead of $CaCl_2$ are shown in Experiments 1 and 2. The mass velocity was improved appreciably, from 185 to 2833, and the free gossypol decreased to approximately one-half, from 0.18 to 0.091%. The nitrogen solubility remained unchanged. The residual lipides were higher for the meal from the NaCl-treated material, and only by using a reslurrying step after the first wash in the bench-scale filtration-extraction test was it possible to reduce the residual lipides to below 1.0%.

Substituting ammonia for the caustic, as shown in Experiment 3, improved the nitrogen solubility to a high of 80.4% and residual lipides below 1%, but the mass velocity was decreased to a low of 198 and the free gossypol to a high of 0.255%. Experiment 5, in which ammonia was also used but moisture content increased to 30%, showed some improvement in that the mass velocity increased to over 3800, and the gossypol decreased to 0.15%. The high nitrogen solubility remained about the same, but the residual lipides were above 1.0% for all tests made. It may be noted that the pH of the slurries were lower for the two experiments in which ammonia was used in place of caustic.

The effects of moisture content are further shown in Experiments 1 and 4, in which the moisture content was increased from 25 to 30% for the NaOH-$CaCl_2$ combination. Mass velocity increased to 4360, free gossypol decreased to 0.064%, and the nitrogen solubility was raised to a 73.4%. Again the residual

TABLE II
Product Evaluation

Experiment No.	1	2	3	4	5	6	7
Slurry, pH	8.3	8.4	7.2	8.4	7.6	8.3	7.5
Mass velocity lbs./sq. ft./hr	185	2933	198	4360	3805	3895	3344
Extracted meal:							
Residual lipides:							
After 3rd wash, %	0.62	3.6	0.88	4.4	4.6	2.6	3.11
After 4th wash, %	0.48	2.6	0.59	3.2	4.0	1.5	1.3
By pre-extraction, %	......	0.83	......	1.6	1.5	0.70	0.89
Gossypol:							
Free, %	0.18	0.091	0.255	0.064	0.15	0.057	0.043
Total, %	0.93	1.00	1.12	0.96	1.03	1.06	1.10
Nitrogen solubility, %	71.0	71.8	80.4	73.4	80.6	62.4	58.6
Nitrogen, %	6.48	6.33	6.87	6.15	6.43	6.23	6.17
Moisture, %	10.3	8.2	8.7	8.9	8.4	7.9	10.8

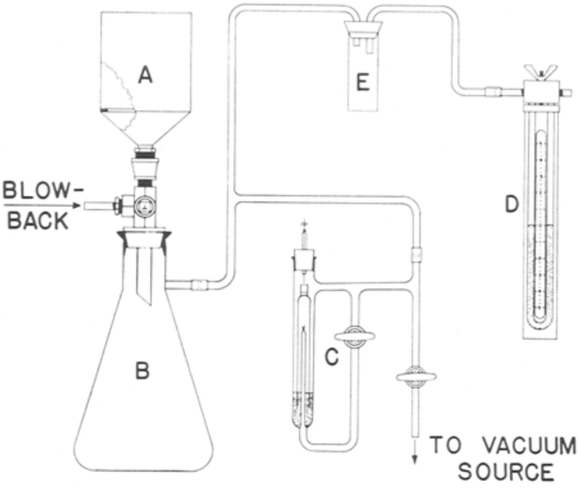

FIG. 2. Bench scale test unit for filtration-extraction.

lipides, even with pre-extraction, were considered high. One experiment was tried in which the moisture content of the material was increased to 40%. The mass velocity was very high (6000) and the free gossypol low (0.036%), but the residual lipides were also considered too high to have practical application.

The effect of using heptane as the cooking and extracting solvent is shown in Experiment 6. Experiment 4 can be used as comparison since the moisture content and the chemicals added were similar. The azeotropic distillation temperature was 181°F. as compared to 146°F. for hexane. Consequently the cooking temperature was higher, and the cooking time required to remove a similar quantity of water by the azeotropic distillation was less (62 min. as compared to 89 min.). The higher temperature apparently had the effect of lowering the nitrogen solubility to 62.4%, but the other factors were considered to be favorable. The mass velocity was 3895, the free gossypol 0.057%, and the residual lipides 1.5% after the fourth wash and 0.70% by pre-extraction.

The results of solvent-cooking flakes prepared by a different procedure, as previously described, are shown in Experiment 7. The nitrogen solubility, free gossypol, and mass velocity (58.6%, 0.043%, and 3344, respectively) were slightly lower than for those obtained in Experiment 6. Residual lipides were comparable.

Table III shows the wet screen analyses of the solids in the slurries after cooking, and Table IV the dry screen analyses of the extracted meals after evaporation of solvent. Screen analyses for Experiment 1 and 3, in which extremely low (below 200) mass velocities were obtained, showed the highest percentage of fines as noted by the through 300 mesh and through 200 mesh for the wet and dry screen analyses, respectively. This is also reflected in the decreased amount of the larger than 20-mesh particles shown in both screen analyses. For the other experiments the screen analyses in both tables indicate that a much coarser and granulated material was produced than in Experiments 1 and 3. This is further reflected by the relatively high mass velocities (2933–4360) obtained.

TABLE III

Wet Screen Analyses of Solids in Slurries

Mesh, %		Experiment Number						
		1	2	3	4	5	6	7
On	5	1.2	3.2	0	3.3	1.8	1.3	1.4
	8	0.9	5.1	2.4	2.8	10.6	5.8	7.8
	14	12.9	19.9	16.7	26.0	35.3	19.1	36.5
	20	7.9	9.2	10.6	12.6	14.0	9.4	14.4
	40	20.6	22.3	19.6	27.1	20.1	21.4	14.1
	60	14.1	16.9	13.9	14.9	10.0	16.0	9.1
	80	8.8	8.3	6.5	4.9	2.8	8.8	3.7
	120	7.2	7.1	7.3	2.8	1.8	7.9	4.4
	170	6.0	3.9	5.3	1.5	1.0	5.1	3.0
	200	0.9	0.5	1.2	0.7	0.4	0.6	1.2
	300	6.5	1.6	6.3	1.1	0.2	1.3	1.7
Through	300	13.0	2.0	10.2	2.3	2.0	2.4	2.7

TABLE IV

Dry Screen Analyses of Meals

Mesh, %		Experiment Number						
		1	2	3	4	5	6	7
On	10	5.2	7.9	3.5	0	9.6	4.2	15.4
	20	18.4	23.3	22.0	33.5	46.6	23.0	58.8
	40	21.3	24.3	23.3	34.6	24.0	25.1	15.0
	60	13.8	16.0	12.1	20.2	12.1	19.2	5.1
	80	7.5	10.4	7.9	6.0	4.4	10.9	2.3
	100	3.1	7.1	6.0	1.6	0.6	4.1	1.1
	200	12.7	7.4	8.4	3.3	2.2	10.8	1.9
Through	200	18.0	3.6	16.8	0.8	0.5	2.7	0.4

The screen analyses for Experiment 7 showed the coarsest particle size distribution which can be attributed to the method of material preparation. Experiments 4, 5, and 6 in general showed an overall coarser particle size distribution than the first three experiments, which can be attributed to the higher initial moisture content during the cooking operation. Experiment 5 showed the coarsest particle size range (aside from Experiment 7), which would indicate that the combination of NH_3 and NaCl had better agglomeration characteristics than the other chemicals tried.

Summary

Data have been presented for a solvent-cooking method of preparing cottonseed meats for extraction. The study includes experiments in which the flake moisture contents were increased to as high as 40% during the initial solvent cooking phase and decreased to about 10% by the end of the cooking cycle, and in which hexane and heptane were used as the cooking solvent in the presence of chemicals such as NaOH, NaCl, $CaCl_2$, and NH_3.

Results showed specific trends with varying conditions. Examples are: increase in the initial moisture content during cooking increases granulation and consequently the mass velocity; increase to 30% moisture content is sufficient to result in mass velocities above the desirable 2,000 lbs. per square foot per hour; free gossypol content of the extracted meals is lower with the higher initial cooking moisture contents and with the addition of certain chemicals ($NaOH$-$CaCl_2$); and the addition of the combination of NaOH and NaCl resulted in better overall filtration-extraction characteristics than either the $NaOH$-$CaCl_2$ or the NH_3-NaCl combinations.

On the basis of the above results the following general conclusions can be made:

1. Solvent cooking offers the possibility of combining cooking, crisping, and slurrying (extraction) steps into a single operation in a process, such as filtration extraction for the direct solvent extraction of cottonseed.
2. Chemicals can be introduced as needed to promote free gossypol reduction and possibly maintain a high alkali protein solubility in the solvent extracted meal.
3. Agglomeration and extractability of the solvent-cooked cottonseed meats can be controlled.

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ABSTRACTS . . . . R. A. Reiners, Editor

• Oils and Fats

S. S. Chang, Abstractor
Sini'tiro Kawamura, Abstractor
Dorothy M. Rathmann, Abstractor

Development of rancidity in lard. J. M. R. Borbolla y Alcalá de la, A. Vargas Romero, and R. Vázquez Ladron(Inst. grasa, Servilla, Spain). *Grasas y aceites*(Seville, Spain) **6**, 74-80 (1955). Development of rancidity of lard in sealed tin cans was studied. By means of the Kreis test and peroxide value, it was shown that the rancidity developed only at the surface of the lard in contact with air in the can. Rancidity was not caused by the metal of the can. (*C. A.* **50**, 1228)

Experiments on the decoloration of cotton-seed oil with anionic-exchange resin like Duslite A. L. Ninni and M. Ninni. *Prakt. Akad. Athenon* **28**, 285-93(1953). Efficient bleaching was obtained. (*C. A.* **50**, 1339)

Reprocessing of soybean seeds for production of edible cake and oil. V. P. Rzhekhin, N. I. Pogonkina, and V. N. Chukaeva. *Masloboino-Zhirovaya Prom.* **21**(6), 9–13(1955). Data are presented to show that in order to obtain high-quality soybean cake and oil, it is necessary to raise moisture content of crushed seed meats to 12.5–13% and age the meats for 4 hours. The temperature of the meat during cooking and pressing should be 95 and 105°, respectively. (*C. A.* **50**, 1228)

Glycerol. Brit. Standards Inst., London, S. W. 1. *Brit. Standard* 2621–5: 1955, 58 pp. Specifications for 5 grades are given. The characteristics and test methods are for the glycerol content, specific gravity, apparent density, refractive index, acidity or alkalinity, ash, total and nonvolatile residues, water, alkali absorption, As, chloride, Cu, Fe, Pb, fatty acid and ester content. (*C. A.* **50**, 1339)

The effect of salt and of antioxidants on the keeping quality of butter. A. K. R. McDowell(Dairy Research Inst., Palmerston North, New Zealand). *J. Dairy Research* **22**, 349–64(1955). Butterfat oxidation occurred in both salted and unsalted butters during storage at 14°F. Butterfat oxidation values, after storage, were much lower in unsalted and in highly salted butters than in butter of normal salt content. Butterfat oxidation in salted butters during storage was retarded by treatment with antioxidants. These included fat-soluble and water soluble substances, surface active agents, and copper-complexing compounds. Unsalted butter, salted butter, and salted butter treated with antioxidants all developed "storage" flavor during storage for 8 months. Many of the antioxidants added to salted butter were effective in retarding further fat oxidation and further increase in intensity of "storage" flavor when the butters were transferred to a temperature of 60–65°F. after storage. (*C. A.* **50**, 1226)

Properties, technology, and economy of melted alimentary fats of southeastern Turkey. R. C. Adam. *Univ. Ankara Fac. Agr. Pub.* **67**, 99 pp.(1955). The people of southeastern Turkey use mixtures of melted milk fats from different pasture animals. Fats from the provinces of Urfa, Diyarbakir, and Kars have the following properties: m.p. 35.8°(34.2–37.4°), 35.5°(31.4–38.4°), and 33.4°(29–37°); f.p. 20.5°(18.3–22.3°), 20.4°(16.5–22.1°), and 17.9°(13.7–20.0°); *n* 1.4585(1.4561–1.4595), 1.4591 (1.4580–1.4600), and 1.4594(1.4559–1.4605); saponification no. 232.6(227.9–236.6), 231.4(226.1–237.8), and 229.7(228.3–231.5); Reichert Meissl no. 27.82(25.19–30.96), 28.13(25.74–34.37), and 28.74(27.39–29.81); Polenske no. 5.48(4.10–8.00), 4.86(2.70–6.55), and 3.57(2.70–4.00); iodine no. 33.42(29.58–37.82), 35.59(32.54–39.58), and 38.47(37.01–40.42), resp. The values in the parentheses are the limit values for unadulterated fats. Urfa fats contain mainly sheep- and goat-milk fat. Diyarbakir fats contain cow butter also, and Kars fats contain mainly cow butter. (*C. A.* **50**, 582)

Hydrogenation of shad oil. F. Waisgluss and R. R. Brenner. *Iudustria y quimica* (Buenos Aires) **17**(1), 13–18(1955). The hydrogenation of shad oil to produce an edible hydrogenated oil was studied at 180° and 1 atm. pressure with 2% nickel catalyst. By using no. 3 oil (m.p. 26.0°) the iodine no. decreased 27.1 units as the m.p. rose to 40.0°. The odor was removed and the oil was almost colorless. The highest m.p. obtained was 56.3°. (*C. A.* **50**, 502)

Chemical study of the progressive development of off-flavor in frozen raw vegetables. F. A. Lee, A. C. Wagenknecht and J. C. Hening(New York Agr. Expt. Sta., Geneva). *Food Research* **20**, 289–97(1955). Unblanched peas, sweet corn, and snap beans showed development of off-flavor in frozen storage in from 2 to 4 weeks. The crude lipide matter extracted from frozen unblanched vegetables showed a marked increase in acid after the vegetables were stored for a week, and showed a positive test for peroxides after the vegetables had been held in frozen storage as follows: peas 3 weeks, snap beans 1 month, sweet corn 2 months. The presence of peroxides in the crude lipide matter extracted from the blanched sweet corn after the vegetable was held in frozen storage for 18 months was confirmed.

The composition of Dutch butter fat. I. Seasonal variations in the unsaturated fatty acid composition of butter fat. J. Stadhouders and H. Mulder(Agr. Univ., Wageningen, Neth.). *Neth. Milk Dairy J.* **9**, 182–93(1955). The respective minimum and maximum contents of the unsaturated fatty acids in butter fat were: oleic acid 28.1 and 40.1, conjugated dienoic acids 0.58 and 2.81, unconjugated dienoic acids 0.73 and 1.90, conjugated trienoic acids 0.015 and 0.026, unconjugated trienoic 0.35 and 0.96, conjugated tetraenoic acids 0 and 0.008, and unconjugated tetraenoic acids 0.27 and 0.60%. The iodine value of summer butter was higher than that of winter butter and was correlated with the refractive index of the butter. The conjugated dienoic acids varied with iodine values, but the unconjugated dienoic acids did not. The seasonal variation of trienoic acids was small and that of the tetraenoic acids was within the limit of experimental error. (*C. A.* **50**, 501)

The melting point and the structure of cocoa butter. S. V. Vaeck(Dept. econ. affairs, Brussels, Belg.). *Zucker u. Süsswarenwirtsch.* **8**, 718–21(1955). The m.p. of a sample of cocoa butter stored for over a year at 17° did not rise above 34.5°. Crystals isolated from the melt at 32° showed m.p. 45°, although the m.p. of the whole was 35–36.5° when these crystals were left in the sample. A sample heated at 50°, mixed with freshly calcinated kieselguhr, filtered through very fine filter paper at 50°, and energetically mixed at 32° remained perfectly clear after 6 hours. When this experiment was repeated with addition of 0.1 g. solid cocoa butter to the molten mix-