All snakes used in this project were in excellent condition and were captured during the season when the deposit of fats are at a maximum.

The methods employed in the analyses are the Official and Tentative Methods of Analysis of the Association of Official Agricultural Chemists. Analytical data for the characteristics of the cold-pressed oil are shown in Table I. No nitrogen was found in any of these snake oils, and the amount of moisture and volatile matter was found to be less than 0.1%. Because the amount of highly unsaturated fatty acids present in snake oil is quite large, it was not considered advisable to attempt the use of iodine and thiocyanogen values in calculating the percentages of monoethenoid and polyethenoid acids.

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Solvent Extraction of Cottonseed and Peanut Oils. VII. Effect of Drying Flaked Prime Cottonseed on Color of Oil and Meal Properties

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OIL color is an important problem to the solvent extraction of cottonseed. Previous work by Vix *et al.* (5) has shown that the heating of cottonseed oil miscellas to various temperatures ranging from a control temperature up to 240°F. for periods of 15 minutes to 3 hours increased the color of the final refined and bleached oils from prime colors for the control to 35Y-32R for those heated at 240° for 3 hours. Colors were determined in a 5¼-inch cell using the Lovibond color system. In view of these results it was believed that the heat used in flake drying might also have an appreciable effect upon the color of the extracted oil. Also, other investigations (1, 2) have shown that heat, together with other conditions such as moisture and pressure, may affect the protein solubility and free gossypol content of the meal. Consequently this investigation was undertaken to obtain data regarding the relationship between flake drying temperatures and color of the oil obtained from the flakes and properties of the resulting meals. Moreover flake preparation, especially moisture control of flakes, is proving more and more important in the current industrial development of the cottonseed solvent extraction process.

The cottonseed used for this study was a prime lot from the 1948 crop of New Roads, La. The seed had been in storage about 8 months under favorable conditions. Flakes prepared from this seed had initially the relatively low moisture content of 6.9%. It must be emphasized that flakes from seed that were not prime or that had a high moisture content or were stored under unfavorable conditions would in all probabilities have given different results.

Seven 14-lb. samples of these prime flakes were dried, each under different conditions, as follows: 120°, 150°, 180°, 210°, and 240°F. all for 3 hours, and 180° for 6 hours. A run with undried flakes was conducted as a control. The dried flakes were extracted with hexane at room temperature, and the resulting miscellas concentrated, steam stripped, and vacuum-dried at temperatures below 120°F. [previous work (5) having shown that color fixation of miscellas occurred at temperatures above 150°F.]. The colors of the refined and bleached oils obtained were determined by the Lovibond colorimeter.

Results showed that, although the colors increased slightly with increased drying temperatures of the flakes, all of the oil colors were prime.

Flake Preparation

Three hundred pounds of prime cottonseed from the 1948 crop of New Roads, La., were cleaned, delinted, and dehulled in standard pilot-plant cottonseed flake preparation equipment. The whole meats containing a hull content of 1.4% were cracked and flaked to an average thickness of 0.009 in. To minimize any increase in their free fatty acid value of 1.2%, the flakes were kept in sealed cans placed in cold storage until just before drying each batch.

Flakes were dried in a forced draft electric oven having 36- x 48-inch trays which were loaded with approximately 3 lb. of flakes per tray. Each batch was dried at the specified temperature and time just prior to extraction. Table I shows the drying data for the seven experiments. Moisture content of the dried flakes varied from 4.5 to 0.3%.

Extraction

Each batch of dried flakes was immediately extracted with commercial hexane at room temperature in a group of six Soxhlet extractors having a total capacity of about 6,000 grams of flakes. A "countercurrent-batch" procedure was used in which the oil miscella from one extractor was used in the

TABLE I

Flake Drying Data and Meal Properties

Exp. No.	Weight of flakes	Drying		Moisture of dried flakes	Extracted meal	
		Temp.	Time		Protein solubility[a]	Free gossypol
	lb.	*°F.*	*hrs.*	*%*	*%*	*%*
1	13.0	Control	Control	6.9	81.9	1.05
2	13.0	120	3	4.5	84.4	1.13
3	13.0	150	3	2.6	82.4	1.23
4	14.5	180	3	1.2	83.3	1.23
5	14.5	180	6	0.8	83.3	1.10
6	14.5	210	3	0.6	81.6	1.13
7	14.5	240	3	0.3	78.7	1.06

[a] Protein solubility in 0.5 N NaCl solution.

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TABLE II

Refining and Bleaching Data

Exp. No.	Flake drying		F.F.A. of oil	Refining lye	Method of break	Refining loss	Lovibond color–5¼" tube	
	Temp.	Time					Refined oil	Bleached oil
	°F.	hrs.	%	°Bé		%	Y R	Y R
1	Control	Control	0.31	14° (max.) 14° (80%)	Modified[a] Regular	2.0 1.7	35 - 3.7 35 - 4.1	10 - 0.7 10 - 1.1
2	120	3	0.39	14° (max.) 14° (80%)	Modified[a] Regular	2.3 1.9	35 - 3.6 35 - 4.5	10 - 0.5 10 - 0.9
3	150	3	0.36	14° (max.) 14° (80%)	Modified[a] Regular	2.3 2.1	35 - 3.7 35 - 4.2	10 - 0.4 10 - 0.9
4	180	3	0.26	14° (max.) 14° (80%)	Modified[a] Regular	2.4 1.8	35 - 3-6 35 - 4.3	10 - 0.6 10 - 1.1
5	180	6	0.26	14° (max.) 14° (80%)	Modified[a] Regular	1.9 1.6	35 - 3.6 35 - 4.0	10 - 0.8 10 - 0.9
6	210	3	0.25	14° (max.) 14° (80%)	Modified[a] Regular	1.4 1.4	35 - 3.8 35 - 4.5	10 - 0.9 10 - 1.2
7	240	3	0.31	14° (max.) 14° (80%)	Modified[a] Regular	2.3 2.1	35 - 4.0 35 - 5.0	15 - 1.1 15 - 1.4

[a] Regular A.O.C.S. refining procedure modified by stirring 90 minutes in cold bath instead of 15 minutes after the addition of alkali.

adjacent extractor except that the initial miscella from each extractor was withdrawn. The procedure was continued until a total of 9 complete passes was attained for each extractor. A contact period of 15 minutes for each pass gave a total of 2¼ hours that the solvent was in contact with the flakes. The residual oil in the flakes averaged 0.80%.

The resulting dilute miscella of 5.0 to 9.0% oil by weight was filtered on Büchner funnels to remove any fine particles of meal in the miscellas.

Oil Recovery

Concentration. The dilute, filtered miscellas were concentrated to 84 to 96% oil by weight in a laboratory model rising film evaporator.

The evaporator was operated under average reduced absolute pressures of 74 mm. of mercury automatically controlled with a manostat, and at miscella temperatures not exceeding 120°F. The dilute miscella was drawn into the reduced pressure system from a 5-gallon bottle through a feed control valve. Feed rates of up to 301 ml. per minute were attained with a 5% oil miscella as feed and with a resulting concentrated miscella of 90% oil. The concentrated miscella was periodically discharged from the bottom of the main evaporator body.

Stripping. The apparatus used for steam stripping the remaining solvent from the concentrated miscella and its operation are described in detail in a previous publication (5).

The solvent was stripped at low temperatures under reduced absolute pressures of 88-mm. mercury in a glass column 36 inches high and 3⅝ inches in diameter. Steam under reduced pressures was introduced at the bottom of the column, and the concentrated miscella preheated to 115°-120°F. was introduced at the top of the column at an average feed rate of 30-35 ml. per minute. The oil-water mixture was collected in a flask at the bottom of the column. The bulk of the water was removed by decanting and centrifugation.

Vacuum Drying. The excess moisture in the resulting stripped oil was removed by vacuum drying in a heating apparatus similar to the one described in a publication by Pollard *et al.* (4).

Stripped oil was poured in batches of 1,400 ml. into the boiler which contained a motor-driven stainless steel stirrer to minimize foaming and to maintain a homogeneous temperature throughout the oil. A reduced absolute pressure of 20 mm. of mercury was applied to the system and the temperature gradually increased until the oil temperature reached 120° F., at which time the oil was immediately removed from the boiler and cooled. The desired temperature increase was attained through an interval time switch which controlled the electrical input to the hot plate and indirectly to the oil bath. The boiler temperatures were measured with a single junction thermocouple of standard copper-constantan wire and a potentiometer with 5 microvolt graduations. The temperature differential between the oil bath and boiler contents was about 10°F. A batch of 1,400 ml. of oil-water mixture required approximately 1 hour to remove 60 ml. of water with a resulting crude oil containing only 0.07 to 0.10% water.

Refining and Bleaching

The crude oils were refined and bleached according to the official American Oil Chemists' Society methods (3) for hydraulic oils since official methods for solvent-extracted cottonseed oils are not yet available. In addition, a sample of each oil was refined by modifying the official hydraulic method (5) by using a cold stir period of 90 minutes in place of the 15 minutes required by the official method. Data regarding the refining, bleaching, and colors of the oils are given in Table II. Using the regular refining method, the color of the refined oil increased from 35Y-4.1R to 35Y-5.0R with increase in flake drying temperatures of from the control (no drying) to 240° F. for 3 hours. Similarly, for the bleached oils the colors increased from 10Y-1.1R to 15Y-1.4R.

Meal Properties

The extracted meals from each of the experiments were tested for protein solubility and for free gossypol. As noted in Table I, the highest drying temperature used (240°F.) decreased the protein solubility slightly to 78.7% and had negligible effect on the free gossypol content.

Conclusions

Data obtained show a negligible effect on meal properties and on color of the refined and bleached oils obtained from flakes dried at temperatures up to

240°F. for 3 hours. These results were obtained with flakes from prime cottonseed with a moisture content of 6.9% and a free fatty acid of 1.2%. Flakes from seed that have been stored under unfavorable conditions and that are not prime or have a high moisture content will in all probabilities give different results.

On the basis of these experiments and on those of a previous study on the effect of heat on cottonseed oil miscellas (5) it can be concluded that dark oils produced from prime seed of low moisture content by solvent extraction methods are due to conditions other than flake drying temperatures; for example, improper treatment of seeds and flakes, tempering of meats, or high temperatures during miscella concentration. Data also show that prime flakes can be dried at relatively high temperatures without affecting the protein solubility of the meal which is desirable. Proper drying of flakes will aid in obtaining optimum solvent extraction conditions, such as percolation rate, extractability of flakes, and minimum fines.

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ABSTRACTS . . . . . Don Whyte, Editor

• Oils and Fats

R. A. Reiners, Abstractor

POLYMORPHISM OF UNSATURATED C_{18} FAT ACIDS. G. B. Ravich, V. A. Vol'nova, and T. N. Kuz'mina (Inst. Gen. and Inorg. Chem., Acad. Sci. U.S.S.R.). *Izvest. Sektora Fiz.-Khim. Anal., Inst. Obshchei i Neorg. Khim., Akad. Nauk S.S.S.R.* **15**, 47-57 (1947). Crystallization and melting curves were obtained for oleic acid under conditions of very slow cooling and heating. The thermograms were recorded with a recording pyrometer and a differential thermocouple. The results seem to indicate the existence of a modification m. 20-20.5°C. (*Chem. Abs.* **44**, 6250)

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